



XXIX ENCONTRO LUSO-GALEGO DE QUÍMICA

» 10-12 NOV '25
FORUM BRAGA
PORTUGAL



»» LIVRO DE RESUMOS

ORGANIZAÇÃO



Sociedade Portuguesa
de Química



Colegio Oficial de
Químicos de Galicia



ASOCIACIÓN DE
QUÍMICOS DE GALICIA



XXIX
ENCONTRO
LUSO-GALEGO
DE QUÍMICA

LIVRO DE RESUMOS

FICHA TÉCNICA

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Este livro foi produzido a partir dos resumos submetidos diretamente pelos autores, de acordo com o modelo publicado. Apenas foram introduzidas pequenas alterações de formatação, que não alteraram o conteúdo científico, que é da exclusiva responsabilidade dos autores.

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PREFÁCIO

É pela curiosidade, pela observação rigorosa e pela busca incessante do conhecimento que compreendemos o mundo e contribuímos para o avanço da ciência. Cada descoberta científica representa não apenas um avanço técnico, mas também um passo em direção a uma sociedade mais informada, sustentável e justa.

A **Química**, em particular, desempenha um papel central, encontrando respostas para os grandes desafios do nosso tempo: o desenvolvimento de novos materiais que impulsionam a transição energética, a descoberta de novos fármacos para combater doenças e bactérias multirresistentes, a busca por processos mais sustentáveis e pela gestão responsável dos recursos naturais. Em domínios como o ambiente, a energia, a alimentação, a saúde e a água, os químicos contribuem diariamente para um futuro mais equilibrado, seguro e sustentável.

O verdadeiro espírito científico ultrapassa fronteiras. A troca de ideias e a cooperação entre investigadores de diferentes países constituem o alicerce de uma ciência global, aberta e colaborativa. Quando mentes de diversas origens e culturas se encontram, a investigação ganha novas perspetivas, a inovação torna-se mais rica e as soluções tornam-se mais abrangentes. É nesta partilha de saberes e experiências que a ciência encontra a sua maior força transformadora.

Neste contexto, o **Encontro Luso-Galego de Química** mantém-se fiel ao seu espírito original, sendo um modelo de cooperação transfronteiriça e acolhendo uma visão ampla da investigação científica ao serviço da sociedade. É um espaço de reflexão e de debate, motor de inovação e elo de ligação entre o conhecimento fundamental e a sua aplicação prática. Acima de tudo, é um ponto de encontro entre diferentes gerações e nacionalidades, onde o entusiasmo dos jovens investigadores se cruza com a experiência dos mais seniores, fortalecendo laços académicos, científicos e humanos entre as duas regiões.

Este livro é testemunho deste compromisso conjunto com o conhecimento e o progresso.

O Fórum Braga será o palco do **XXIX Encontro Luso-Galego de Química**, entre os dias 10 e 12 de novembro de 2025, numa organização conjunta da Delegação de Braga da Sociedade Portuguesa de Química (SPQ), do Colégio Oficial de Químicos da Galiza (COLQUIGA), da Associação de Químicos da Galiza e do Departamento de Química/Centro de Química da Escola de Ciências da Universidade do Minho.

A todos os participantes, damos as boas-vindas ao Encontro, esperando que estes dias sejam de partilha, inspiração e construção de novos caminhos para a ciência, a cooperação e o progresso comum, pilares fundamentais de um futuro melhor.

Susana Costa
(Coordenadora do XXIX ELGQ)

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INFORMAÇÕES GERAIS

LOCAL DO ENCONTRO

O Encontro decorrerá no FORUM BRAGA, em Braga.

O Forum dispõe de um parque de estacionamento exterior **gratuito** com capacidade para 620 viaturas, com entrada pelas traseiras do Forum:

Rua Monsenhor Airosa, 4705-102 Braga

<https://maps.app.goo.gl/VyJqQCDTjPTw7M8R8>

O acesso às salas onde decorre o Encontro só será permitido aos participantes que apresentem o respetivo crachá de identificação.

EQUIPAMENTO DE PROJEÇÃO

O auditório e as salas estão equipadas com computador com sistema operativo Windows e *software* Microsoft Office, sistema de projeção e apontador laser.

Cada orador deverá contactar o *staff* da respetiva sala para transferir o ficheiro da sua comunicação (em PowerPoint) para o computador da sala **até ao final da manhã ou tarde, anterior à sua comunicação**. A organização não se responsabiliza por questões de incompatibilidade de *software* e/ou sistema operativo.

COMUNICAÇÕES ORAIS

As lições plenárias terão duração de 50 minutos, a que se segue um período de discussão não superior a 10 minutos.

As comunicações orais terão duração máxima de **12 minutos**, seguidas de 3 minutos de discussão. Atendendo ao número elevado de comunicações orais, pede-se a todos os oradores que cumpram o tempo estipulado de forma a evitar atrasos no programa.

COMUNICAÇÕES POSTER

Dimensão máxima: tamanho A0 com orientação vertical.

Os posters estarão expostos em permanência, devendo ser afixados no primeiro dia do encontro e removidos no terceiro dia do encontro, após o último *coffee break*.

A discussão de posters decorre no horário dos *coffee breaks*.

BANQUETE

O jantar do encontro será no dia 11 de novembro, pelas 20:30, no Hotel Vila Galé Collection Braga.

ENTIDADES ORGANIZADORAS



APOIO INSTITUCIONAL



APOIO DE OUTRAS ENTIDADES



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PROGRAMA CIENTÍFICO

10 nov (segunda-feira/lunes)				
Auditório	Sala I	Sala II	Sala III	
09:00	Registo			
09:15				
09:30				
09:45				
10:00				
10:15				
10:30	Abertura/Sesión de apertura			
10:45	PL1 - Nicola Armaroli (Auditório)			
11:00				
11:15				
11:30				
11:45				
12:00	QS1	QP1	QF1	S1
12:15	QS2	QP2	QF2	S2
12:30	QS3	QP3	QF3	S3
12:45	QS4	QP4	QF4	S4
13:00	Almoço/Comida			
13:15				
13:30				
13:45				
14:00				
14:15				
14:30	QS5	NN1	BB1	S5
14:45	QS6	NN2	BB2	S6
15:00	QS7	NN3	BB3	S7
15:15	QS8	NN4	BB4	S8
15:30	QS9	NN5	BB5	S9
15:45	QS10	NN6	BB6	S10
16:00	QS11	NN7	BB7	S11
16:15	QS12	NN8	BB8	S12
16:30	Coffee-Break + Posters			
16:45				
17:00				
17:15				
17:30	QS13	NN9	ED1	S13
17:45	QS14	NN10	ED2	S14
18:00	QS15	NN11	ED3	S15
18:15	QS16	NN12	ED4	S16
18:30	QS17	NN13	ED5	S17

20:30

11 nov (terça-feira/martes)			
Auditório	Sala I	Sala II	Sala III
QS18	QSU1	QAM1	QAA1
QS19	QSU2	QAM2	QAA2
QS20	QSU3	QAM3	QAA3
QS21	QSU4	QAM4	QAA4
PL2 - Pastora Bello Bugallo (Auditório)			
Coffee-Break + Posters			
QS22	QSU5	QAM5	QAA5
QS23	QSU6	QAM6	QAA6
QS24	QSU7	QAM7	QAA7
QS25	QSU8	QAM8	QAA8
QS26	QSU9	QAM9	QAA9
QS27	QSU10	QAM10	QAA10
Almoço/Comida			
QS28	QSU11	QAM11	QAA11
QS29	QSU12	QAM12	QAA12
QS30	QSU13	QAM13	QAA13
QS31	QSU14	QAM14	QAA14
PL3 - Nuno Mateus (Auditório)			
Coffee-Break + Posters			
QS32	QSU15	QAM15	QAA15
ED6	QSU16	QAM16	QAA16
ED7	QSU17	QAM17	QAA17
QT1	QSU18	QAM18	QAA18

Banquete/Cena de Gala

12 nov (quarta-feira/miércoles)			
Auditório	Sala I	Sala II	Sala III
QIE1	QSU19	QI1	QA1
QIE2	QSU20	QI2	QA2
QIE3	QSU21	QI3	QA3
QIE4	QSU22	QI4	QA4
PL4 - Antonio Echavarren (Auditório)			
Coffee-Break + Posters			
QO1	QSU23	QI5	QA5
QO2	QSU24	QI6	QA6
QO3	QAM19	QAA19	QA7
QO4	QAM20	QAA20	QA8
QO5	QAM21	QAA21	QA9
QO6	QAM22	NN14	QA10
Almoço/Comida			
QO7	CAT1	QIE5	QA11
QO8	CAT2	QIE6	QA12
QO9	CAT3	QIE7	QA13
QO10	CAT4	QIE8	QA14
QO11	CAT5	QIE9	QA15
	QV1	QA17	QA16
PL5 - Verónica Bermudez (Auditório)			
Encerramento/Clausura			

BB - Bioquímica e Biotecnologia
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 QIE - Química Industrial e Engenharia
 QO - Química Orgânica
 QP - Química dos Polímeros

QS - Química e Saúde
 QSU - Química Sustentável
 QT - Química Teórica e Modelação Molecular
 QV - Química Verde
 S - Simpósio

PROGRAMA DO SIMPÓSIO

“Produção e Armazenamento de Energia”

10 de novembro de 2025



	<i>Chair: Clara Rodrigues Pereira</i>
12:00	Emma López (Red CRUSOE) <i>Apresentação da Red CRUSOE</i>
12:15	S1: Félix Nieto (Centro de Innovación Tecnológica en Edificación e Enxeñaría Civil, Univ. Coruña) <i>Deployment of an energy harvester prototype in an urban environment location</i>
12:30	S2: André Pereira (Inanoenergy & IFIMUP, Faculty of Sciences, University of Porto) <i>Empowering the Internet of Things through sustainable energy harvesting: the inanoEnergy approach</i>
12:45	S3: Cátia Rodrigues (IFIMUP, Faculty of Sciences, University of Porto) <i>Triboelectric nanogenerators for energy harvesting in remote and harsh environments</i>
13:00	S4: Pedro Fernandez (Centro de Innovación Tecnológica en Edificación e Enxeñaría Civil, Univ. Coruña) <i>Piloto virtual GIS de la cadena de valor de la eólica offshore de Galicia y Norte de Portugal</i>
	Almoço/Comida
	<i>Chair: Félix Nieto</i>
14:30	S5: Joana Príncipe (LEPABE, ALiCE, Faculty of Engineering, University of Porto) <i>Inverted flexible perovskite solar modules for sustainable urban integration</i>
14:45	S6: Joana S. Teixeira (LAQV-REQUIMTE, Faculty of Sciences, University of Porto) <i>Flexible thermally-chargeable supercapacitors with a flavylum derivate solid-gel electrolyte: thermoionic & energy storage performance evaluation</i>
15:00	S7: Rúben Feiteira (LAQV-REQUIMTE, Faculty of Sciences, University of Porto) <i>New carbon/mixed-valence metal oxide nanohybrids for thermal energy harvesting and electrochemical energy storage</i>
	<i>Chair: Manuela Silva</i>
15:15	S8: Salomé Soares (LSRE-LCM, ALiCE, Faculty of Engineering, University of Porto) <i>Boosting CO₂ hydrogenation to methanol efficiency through carbon-metal oxide synergy</i>
15:30	S9: Murilo Alcântara (CICECO, University of Aveiro) <i>COSMO + AI for screening of solvents: case study of methanol synthesis</i>
15:45	S10: Rafael G. Morais (LSRE-LCM, ALiCE, Faculty of Engineering, University of Porto) <i>Designing Fe-N-C single-atom catalysts using carbon black for efficient oxygen reduction reaction</i>
16:00	S11: Caroline Araújo (Chemistry Centre, University of Minho, CQ-UM) <i>Carbon-based nanomaterial in solid biopolymer electrolytes: effect of graphene oxide on methylcellulose films</i>

16:15	S12: Liliana Truta (CeNTI) <i>Para um futuro mais verde: células flexíveis com materiais sustentáveis</i>
	Coffee-Break + Posters
	<i>Chair: Salomé Soares</i>
17:30	S13: Renato Gonçalves (Chemistry Centre, University of Minho, CQ-UM) <i>Cathodes manufacturing strategies for improved efficiency, security and sustainability in lithium-ion batteries</i>
17:45	S14: Carlos Costa (CF-UM-UP and LaPMET, University of Minho) <i>Next generation solid polymer electrolytes based on ternary components for high-performance room-temperature solid-state lithium-ion batteries</i>
18:00	S15: Inês F. Monteiro (CF-UM-UP & Chemistry Centre, University of Minho CQ-UM) <i>Advancing safety and performance in next-generation smart batteries</i>
18:15	S16: Tiago Salgueiro (FEUP, FCUP & LAETA) <i>Solid-state Na-metal batteries with improved performance via facile liquid electrolyte wetting at NaSICON interface</i>
18:30	S17: Gabriel L. Camilo (CICECO, University of Aveiro) <i>Biphasic aqueous electrolytes combining TFSI and salting-out agents for stable and selective membraneless redox systems</i>

LISTA DE COMUNICAÇÕES

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PL1	The great energy transformation – Challenges for science and beyond	Nicola Armaroli
PL2	Systems analysis for a regenerative growth: the role of Chemistry	Pastora Bello Bugallo
PL3	Exploring the colour and bioactivity of anthocyanin derivatives	Nuno Mateus
PL4	Gold-catalyzed molecular gymnastics	Antonio Echavarren
PL5	Emerging materials and technologies for smart window systems in buildings and vehicles	Verónica Bermudez

Orais

	<i>Bioquímica e Biotecnologia</i>	
BB1	Valorização de biorresíduos de chia (<i>Salvia hispanica</i> L.): perfil fenólico e potencial bioativo	Maria Eduarda Silva
BB2	Green Extraction of Antioxidant Compounds from Propolis	Nicolas Justus
BB3	2,4,5-Triaminopyrimidines: a new class of fluorescent nucleobase analogues for cell viability studies	João Gonçalves
BB4	Synergistic effects of physical pretreatments and fermentation on the nutritional value of lentils	Ângela Liberal
BB5	Hemolytic activity of biosurfactant extracts obtained in corn steep liquor for further inclusion in antibiotic formulations	Noelia Gonzalez
BB6	Advancing monoclonal antibody purification: the application of supported ionic liquids (SILs)	João Vasco Valente
BB7	Sustainable biosurfactant extract from corn steep water: characterization and multifunctional applications in cosmetic, pharmaceutical and agrochemical formulations	Andrea Martinez
BB8	Emulsifying potential of a biosurfactant extract obtained from dairy by-products	Nicolas Ruso
	<i>Catálise e Fotocatálise</i>	
CAT1	Role of nitrogen doping and carbon support structure in the oxidation of 5-hydroxymethylfurfural to Furans over Pt/C catalysts	Katarzyna Eblagon
CAT2	Structure-performance relationships of carbon-based supports in Fischer-Tropsch catalysts	Thais Berberich
CAT3	Sustainable magnetic catalysts for glycerol valorization into solketal	Fátima Mirante
CAT4	A novel lipase form with enhanced activity across diverse substrates	Jennifer Noro
CAT5	Challenges in the synthesis of substituted aminobenzaldehydes via green and auto-catalytic approaches	André Lopes
	<i>Educação e Ensino da Química</i>	
ED1	Integração da Química numa disciplina STEAM: potencial e desafios da modelagem tridimensional de moléculas no 8.º ano	Isabel Saúde
ED2	Práticas supervisivas na formação inicial de professores de Química: uma revisão integrativa	Avelina Filipe

ED3	O Jogo do Equilíbrio: uma experiência de aprendizagem baseada em jogos no ensino do Princípio de Le Châtelier	José Luís Araújo
ED4	Del átomo al algoritmo: transformando la enseñanza de la Química com Inteligencia Artificial	Elisabeth Lucero
ED5	A Química no 1.º ciclo do ensino básico: um programa de formação personalizado de professores sobre práticas <i>hands-on</i>	Isabel Saúde
ED6	Gamificación como recurso para el fortalecimiento del aprendizaje a través del 2º Concurso de Conocimientos en Química Analítica 2025: "Dr. Gaston Charlot"	Elvis Tovar Facundo
ED7	Museu da Escola Sá de Miranda: um património científico ímpar	Paula Cristina Silva
	<i>Nanoquímica e Nanotecnologia</i>	
NN1	Dehydridiketopiperazines: minimalist peptide blocks for self assembled materials	Carlos Oliveira
NN2	When biomass-based Carbon Quantum Dots meet photocatalysis	Helena Sousa
NN3	PC-based liposomal formulations for efficient delivery of a novel highly potent anticancer drug	Maria Alice Carvalho
NN4	Computer modelling, one tool to rule them all? From carbon nanotubes to biomolecules, covalent organic frameworks and moiré synthetic systems	Manuel Melle Franco
NN5	Surface Chemistry-driven control of magnetic interactions in iron oxide nanoparticles for MRI and Hyperthermia applications	Pelayo García
NN6	Hardy kiwi loaded liposomes: nano-delivery of natural antioxidants	Filipa Teixeira
NN7	Nanocarrier encapsulation of quaternary phosphonium salts	Joana Moreira
NN8	Lipid-polymer hybrid nanocarriers for intranasal administration of entacapone	Cláudia Sampaio
NN9	From sequence to gel: structure-property insights into isoleucine-containing dehydropeptides	Valéria Gomes
NN10	Synthesis of carbon nanotubes from polyolefin waste: mass balance and gas phase composition	Fernanda Roman
NN11	Size-dependent eryptotic effects of citrate-coated silver nanoparticles on human erythrocytes and protection by quercetin	Inês Alexandre Santos
NN12	Formación <i>in situ</i> de nanopartículas de oro y plata en films líquidos para la detección colorimétrica de formaldehído	Nerea Villarino
NN13	Non-lamellar lyotropic liquid crystalline lipid nanoparticles for brain drug delivery	Ana Amorim
NN14	Peptide-functionalized SPIONs for dual-mode imaging and drug delivery in COPD	Cátia Martins
	<i>Química Analítica</i>	
QA1	Miniaturized assay for monitoring sulfite preservatives in food samples	Vítor Hugo Costa
QA2	Point-of-care detection of soybean Gly m TI in complex foods: immunosensor vs MIP biosensor	Catarina Ramos Dias
QA3	Determinación de THC mediante microextracción líquido-líquido dispersiva asistida por aire (AALLME)	Laura Blanco Garcia
QA4	Optical chemosensor based assay for measuring tributyltin in environmental samples	Inês Nunes Vilhena
QA5	Innovative sampling strategies for trace metals analysis in water monitoring	Tânia Vaz Pedro
QA6	DNA-based electrochemical biosensors for honey authentication	Stephanie Morais
QA7	Sequential injection lab-on-valve with bead injection for iron speciation in waters	Mafalda Pereira
QA8	Enantioseparation of methylone by chiral liquid chromatography and metabolite synthesis for further metabolomic studies	Ana Sofia Almeida

QA9	Uso da sililação na caracterização de used cooking oil por cromatografia gasosa mono e bidimensional	Pedro José Filho
QA10	A public health tool for monitoring aluminium levels in environmental and biological samples	Juliana Aguiar
QA11	Determination of Cu(II) in Mozambican artisanal spirits by square-wave anodic stripping voltammetry	Gercílio Chichava
QA12	Point-of-care determination of free chlorine in swimming pools waters as a public health issue	Pedro Almeida
QA13	Mechanical vs. manual harvesting: effects on phenolics and antioxidant potential in Fernão Pires, Loureiro, and Alvarinho wines	Mafalda Cunha
QA14	Estrategias analíticas para el control de contaminantes en aguas mediante enfoques miniaturizados y de bajo coste	Vanessa Romero
QA15	Dispositivo microfluídico tridimensional para la determinación colorimétrica multianálito de analgésicos en muestras clínicas	Alberto Abalde
QA16	Análise isotópica de Sr mediante ICP-TOF-MS para a verificación da orixe de produtos agroalimentarios	Ainhoa Collazo
QA17	Turn-off/on fluorescence-based detection of SARS-CoV-2 using a carbon dots optical biosensor	Ricarda Torre
	<i>Química Agro-Mar-Alimentar</i>	
QAA1	Unveiling the impact of cadmium on silica-based food additive (E551): insights into concentration-dependent interactions	Cláudia Lopes
QAA2	Removing paralytic shellfish poisoning (PSP) toxins: what are the solutions?	Joana Ferreira Leal
QAA3	Evaluation of the seasonal variability in the phytochemical and bioactive composition of the alga <i>Sargassum muticum</i>	Adela López-Vizcaino
QAA4	Anthocyanins-proteins interactions for the development of natural blue colorants	Elsa Martins
QAA5	Pão enriquecido com <i>Chlorella vulgaris</i> Honey: avaliação nutricional e determinação de luteína	Rossana Cardoso
QAA6	From waste to taste: sustainable seasoning production from atlantic mackerel by-products	Francisca Santos
QAA7	Integrating edible insects and spirulina as a nutritive and sustainable food solution: consumer acceptance, sensory analysis, proximate composition and immunoreactivity	Rita Biltes
QAA8	Structural and matrix effects on astringency in butterfly pea (<i>Clitoria ternatea</i> L.) tea	Inês Silva
QAA9	Design and application of antioxidant surfactants to preserve Omega-3 rich foods	Fátima Paiva-Martins
QAA10	Decoding astringency: linking oral interactions to sensory perception	Inês Ferreira
QAA11	Yeast protein extracts and hydrolysates production as a novel source of umami flavour enhancers	Bernardo Almeida
QAA12	Efeito do regime hídrico e de culturas intercalares na qualidade e produtividade de <i>Prunus dulcis</i> Mill. em três anos de produção	Bruna Moreira
QAA13	Caracterização bioquímica e potencial de valorização de perdas pós-colheita de <i>Daucus carota</i> L.	Joana Rodrigues
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QAA18	<i>Prunus lusitanica</i> L. subsp. <i>lusitanica</i> fruits and by-products as a novel source of added-value metabolites	Natacha Rodrigues

QAA19	Tratamientos hidrotérmicos sucesivos del bagazo de uva tinta: estrategia para maximizar la recuperación de polifenoles bioactivos	Alba Pérez Pérez
QAA20	The volatile profile of beer: a review of chemical diversity and analytical perspectives	Clarisse Lopes
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QAM3	Optical chemosensory studies of novel D-A- π -A benzothiadiazoles for cyanide detection in aqueous media	Mathilde Boland
QAM4	A comparative framework for evaluating energy security in Europe using a multi-criteria approach	Danial Abdolmaleki
QAM5	Hybrid organic-inorganic materials for rare earth elements removal: unravelling the role of physicochemical properties	Joana Sousa
QAM6	Análisis y simulación dinámica del comportamiento energético en edificios: aplicación a la selección de aislamientos sostenibles para optimizar la eficiencia energética	Khalid Azizi
QAM7	Designing functional COFs for water purification: towards stable and efficient adsorbents	Uxía Gómez-Bouzó
QAM8	From mineral water to E-waste: rare-earth removal by a microporous niobium-iron silicate	M ^a Eduarda Pereira
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QAM10	CNT-embedded PVP/PVDF membranes for CWPO-intensified filtration of sulfamethoxazole and paracetamol	Adriano Silva
QAM11	Benign by Design of new depigmenting agents	Abigail Rodrigues
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QI2	Iron(III) helicates with the ability to interact with DNA	Isabel Velo Heleno
QI3	Coordination modes of carbonyl phosphine-iminophosphorane ligands: from the unusual to the unprecedented	Irene Vázquez
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QI5	Biological evaluation of Cu(I) phosphino-azine mesocates	Uxia Barreiro Sisto
QI6	A mono-silanolate Dy-N ₆ macrocyclic complex with SMM behavior	Paula Oreiro
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QS7	Design and synthesis of novel potential inhibitors of p52 dimerization	Sofia Sarabando
QS8	Spectroscopic insights into polyamide 6 microplastics after simulated gastrointestinal digestion	Mariana Lamas
QS9	Development of a grape stem polyphenol-based mouthwash for periodontal health: from byproduct valorization to therapeutic application	Sofia Pires da Silva
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QS19	Parabens' exposure modulates inflammatory mediators in HepG2 cells and human neutrophils	Ana Ramalho

QS20	Development of a scaffold-free 3D osteosarcoma model uncovers selective drug responses	M ^a Isabela Santos
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QS22	Insights into the development of anticancer agents targeting the spindle assembly checkpoint	Inês Lima Carvalho
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QS25	Discovery of potent modulators of the dopamine D ₂ receptors with enhanced biochemical stability for Parkinson's therapy	Xavier Correia
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QS31	From diagnosis to treatment: the impact of light in biomedical science	Sara Gamelas
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QAA23	Study of the biological activity of <i>Fucus vesiculosus</i> : aqueous extractions and potential food application	Marcelo Catarino

QAA24	Scaffolds de proteína de soro do leite para encapsulação de vitamina A	Manuel Figueiredo
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QIE14	Estudo dun deseño de biorrefinería integrada con peles de pataca: Análise de ciclo de vida (ACV)	Beatriz Rodríguez Martínez
QIE15	Autohidrólise asistida por microondas como estratexia sostible para a produción de oligosacáridos e bioetanol a partir de madeira de <i>Acacia dealbata</i>	Álvaro Lobato Rodríguez
QIE16	Green solvent and microwave-assisted strategies for efficient conversion of white grape pomace into 5-hydroxymethylfurfural	Cristiana Martins
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QIE18	Predicción del número de cetano del gasóleo a partir del índice de cetano calculado y el contenido en mejorador de ignición usado en su formulación obtenido por FTIR	M ^{ra} Mercedes Fernández Feal
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QV7	Phytochemical analysis of <i>Camellia japonica</i> husks and leaves by GC-MS: exploring bioactive compounds for future applications	Francisco Díaz
QV8	Removal of additives from plastic to enhance recycling	Marco Prazeres
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S22	Advanced smart textiles integrating hybrid carbon/mo-v sulfide nanomaterials for energy storage	Marta Nunes
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S26	Cork-based carbon electrodes for sustainable supercapacitors	Raquel Rocha
S27	Safety and accident analysis in small modular reactors (SMRs) for controlled nuclear fission	Miranildo Silva



RESUMOS PLENÁRIAS



The great energy transformation Challenges for science and beyond

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Human-induced global warming demands a profound transformation of the global energy system, which must largely be achieved by mid-century. This represents an immense technological, economic, and societal challenge. A key feature of this transition will be the increasing electrification of energy end uses, with solar and wind power—supported by storage technologies—playing a central role due to their unique combination of technical features and economic viability. The lecture will touch on a range of critical topics, including: the role of renewables in enhancing energy security; the decarbonization of the transportation sector; the challenges associated with hydrogen as an energy carrier; the complexities of hard-to-abate sectors, including the chemical industry; the availability of mineral resources needed for manufacturing renewable energy converters and storage systems. Throughout, special attention will be given to the pivotal role of chemistry in enabling and accelerating the energy transition.

Systems analysis for a regenerative growth: the role of Chemistry

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Managing humanity's relationship with the environment in which we live is a fundamental component of any activity. Environmental degradation resulting from decades of abuse, ignorance, frivolity, ambition, and ultimately a lack of respect for nature requires continuous and demanding governance actions. The management of any activity should be previously analysed with regenerative and resilient objectives in mind. In this last decade, the idea of **Regenerative Growth** has emerged with strength and optimism, a few years later the media-driven approach of the "circular economy".

Designing strategies that seek regeneration and resilience, that restore damage while enhance the vitality of nature, appears to be a refreshing and optimistic approach that can be applied to systems when pursuing any type of activity.

The success of these strategies in achieving goals beyond sustainability requires a prior understanding of the characteristics of these systems, their dynamics, internal interactions, and their relationship with their surroundings, including physical, chemical, biological, and other interactions. Systems analysis, as a discipline that traditionally uses mathematical methodology to obtain the details of the systems being analysed, can be complemented with other analytical methods to incorporate the ideas of regenerative growth for innovative pre-management activities.

Systems Analysis using this approach for specific cases that represent a challenge for humanity has provided new evidence and novel results, both from a methodological perspective and by obtaining new key parameters and indicators for each type of system. Furthermore, the study of hypothetical scenarios based on the results of the analysis constitutes a very valuable tool for the design of policies that transcend sustainability and enable a future of symbiotic coexistence between humans and nature. Some of the systems studied include fires, plastics and microplastics, global sustainability, buildings, industry, electricity management, demand-side management, and grid stability, among others. In all cases, the importance and consideration of **Chemistry** appear to be a fundamental aspect for achieving valid and realistic results.

Exploring the colour and bioactivity of anthocyanin derivatives

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Anthocyanins are a naturally occurring class of polyphenolic compounds widespread in plant-derived foodstuffs and therefore abundant in human diet. These natural pigments are responsible for different colour hues displayed by many fruits and flowers. On the other hand, there is evidence regarding the positive association of their intake with healthy biological effects displayed in vivo.

Therefore, both colour features and putative bioactivities make these natural pigments appealing targets in different fields such as food, cosmetic and pharmaceutical industries.

However, there are stability issues linked to the challenging chemistry of these molecules. Their occurrence in red wines and interaction with other phenolic compounds yielding new anthocyanin derivatives with unusual colours led to the finding of new chemical pathways. Naturally inspired by red wine chemistry, new synthetically produced anthocyanin-derived pigments may be obtained.

This presentation will revisit the main chemical features beyond the colour and bioactivity of these molecules as well as highlight how anthocyanin derivatives may be explored for some technological applications.

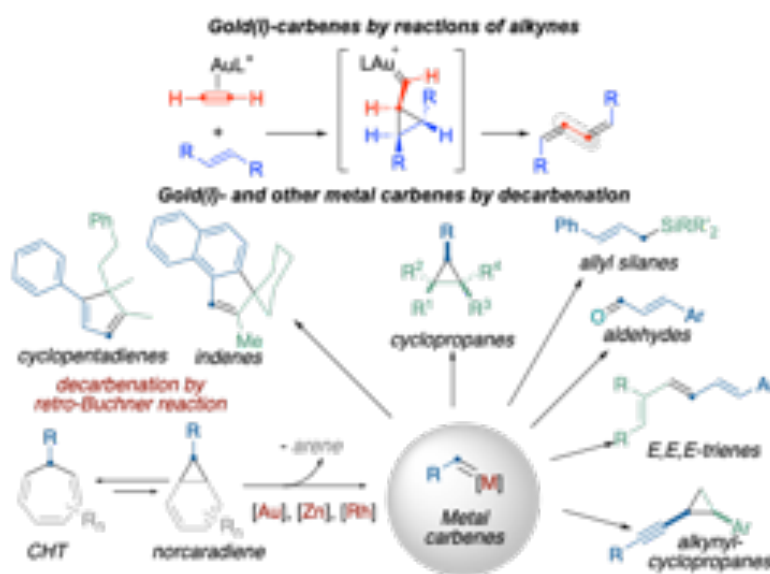
Gold-catalyzed molecular gymnastics

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Many gold(I) catalyzed reactions take place through gold(I) carbenes as intermediates [1,2], Gold(I) carbenes, as well as other metal carbenes or carbenoids, can also be generated in solution by retro-Bucher reaction of cycloheptatrienes (formal decarbenation) [3]. This lecture will focus on reactions developed in our group that proceed via gold(I) carbenes and related intermediates, leading to the construction of complex carbon skeletons.



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Emerging Materials and Technologies for Smart Window Systems in Buildings and Vehicles

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Smart cities depend on the development of intelligent, sustainable, and energy-efficient buildings. Within the European Union, the building sector constitutes the largest consumer of energy and one of the primary contributors to CO₂ emissions, accounting for approximately 40% of total energy consumption and 36% of greenhouse gas emissions. The recognition of this sector as a critical component of the global energy transition has led to the establishment of the Nearly Zero Energy Building (NZEB) concept.

The building envelope—which comprises external walls, floors, roofs, ceilings, windows, and doors—represents more than 30% of total energy consumption. Among these components, windows exert a particularly significant influence on a building's overall energy performance. The transition from conventional static glazing systems to dynamic, or smart, windows has the potential to decrease a building's energy demand by up to 40%.

Smart windows enable real-time regulation of sunlight (visible radiation) and solar heat gain (near-infrared, NIR) radiation) according to the occupant's preferences, regardless of geographic location, climatic conditions, seasonal variations, or room orientation. Smart windows offer a plethora of advantages, enhancing the building's energy efficiency by reducing heating and cooling loads, while simultaneously improving indoor visual and thermal comfort and maintaining clear outdoor visibility. Given that ca. 44% of solar energy is contained within the visible spectrum and 49% within the NIR region, the integration of both visible and NIR control functionalities in smart window systems is imperative to achieve optimal energy efficiency. These principles are equally applicable to the design and optimization of windshields within the automotive industry.

This presentation will address the major challenges currently faced in the field, highlighting the key scientific and technological barriers that hinder further progress. It will explore the main endeavors undertaken to overcome these limitations, including recent innovations, emerging strategies, and interdisciplinary approaches aimed at advancing the development of sustainable and energy-efficient smart window technologies. Potential directions for future research and implementation will be outlined.

Acknowledgments

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RESUMOS COMUNICAÇÕES ORAIS



Valorização de biorresíduos de Chia (*Salvia hispanica* L.): perfil fenólico e potencial bioativo

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O aproveitamento de subprodutos agroindustriais representa uma estratégia sustentável para o desenvolvimento de ingredientes funcionais inovadores [1]. Embora a *Salvia hispanica* L. (chia) seja amplamente reconhecida pelo elevado valor nutricional das suas sementes, as suas estruturas vegetativas e reprodutivas, como folhas e flores, permanecem subexploradas, sendo frequentemente descartadas como resíduos [2]. Em alinhamento com os Objetivos de Desenvolvimento Sustentável (ODS) 3, 12 e 13 da Agenda 2030, este estudo investigou o potencial de valorização dos bioprodutos obtidos das partes vegetativas (CHPV) e reprodutivas (CHPR) de *S. hispanica*, através da análise do perfil individual de compostos fenólicos e respetivo potencial bioativo.

O perfil individual de compostos fenólicos foi determinado através de um sistema HPLC-DAD-ESI/MS e a ação bioativa foi testada por ensaios *in vitro* quanto à atividade antioxidante (ORAC, DPPH e TBARS) e atividade antimicrobiana (método da microdiluição contra estirpes bacterianas e fúngicas). A citotoxicidade foi determinada em culturas celulares de fígado (AML12) e intestino (Caco-2).

A análise fitoquímica revelou que CHPR é significativamente mais rica em compostos antioxidantes, com teores de compostos fenólicos totais (3,41 mg/g) e, particularmente, flavonoides totais (2,89 mg/g) cerca de três vezes superiores aos de CHPV. Essa diferença refletiu-se numa atividade antioxidante mais elevada, com CHPR a exibir mais do dobro da capacidade ORAC (1376,4 vs. 634,7 $\mu\text{mol/g}$ Trolox), eficiência aproximadamente quatro vezes maior no DPPH (EC_{50} : 0,020 vs. 0,084 mg/mL) e quase cinco vezes maior no TBARS (EC_{50} : 0,030 vs. 0,142 mg/mL). Nenhum dos extratos apresentou atividade antimicrobiana relevante. Nos ensaios de citotoxicidade, CHPV manteve viabilidade celular superior a 70% até 1250 $\mu\text{g/mL}$, enquanto CHPR não apresentou toxicidade até 625 $\mu\text{g/mL}$.

Concluiu-se que os bioresíduos de chia estudados, especialmente da parte reprodutiva, são fontes seguras e promissoras de antioxidantes naturais, que agregam valor à cadeia produtiva e incentivam as estratégias europeias de economia circular, alinhando-se às metas globais de sustentabilidade e saúde.

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Green extraction of antioxidant compounds from Propolis

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Propolis is a complex resinous material produced by honeybees from plant exudates, recognized for its exceptional content of bioactive compounds including flavonoids, phenolic acids, and their esters, which provide potent antioxidant, antimicrobial, and anti-inflammatory properties [1,2]. Traditional extraction methods using organic solvents present environmental concerns and processing limitations, compelling for sustainable alternatives. This study investigates the application of Natural Deep Eutectic Solvents (NADES) for bioactive compound extraction from propolis from the Montesinho Natural Park, Portugal. A biphasic NADES system combining Menthol:Lauric acid (4:1) with Proline:Citric acid (1:1) and Menthol:Lauric acid (4:1) and ultrapure water was developed using ultrasound-assisted extraction (UAE) with a ultrasound probe UP400S (400 W, 24 kHz). The extraction parameters were optimized through L-Optimal experimental design, evaluating the effects of time (15-30 min), temperature (35-55 °C), water content (20-40%), and amplitude (60-100%). Among the three systems, the apolar phase composed of Menthol:Lauric acid (4:1) demonstrated the best selectivity for antioxidant compounds.

The antioxidant activity was evaluated using DPPH and ABTS radical scavenging assays, with results expressed as ascorbic acid equivalents (AA). The Menthol:Lauric acid (4:1) system achieved antioxidant activities values: DPPH of 41-46 mg AA/mL of extract and ABTS of 45 mg AA/mL of extract. These results represent an enhancement compared to conventional ethanol extraction 1:10 (w/w) (DPPH: 24-33 mg AA/mL of extract; ABTS: 25-33 mg AA/mL of extract). Statistical analysis revealed no significant differences between the conditions (80% amplitude, 22.5 min, 45 °C) and simplified parameters (60% amplitude, 15 min, ambient temperature), demonstrating robustness and scalability potential. Also, the kinetic curve for the system Menthol:Lauric acid (4:1) and ultrapure water showed that after 14 minutes, with 60% amplitude and at 31-35 °C, no considerable increase in the antioxidant capacity was observed. The biphasic nature of the NADES system enables selective compound separation. This eliminates toxic organic solvents, reduces extraction time from days to minutes, and its extracts can be directly incorporated into formulations. This research demonstrates that Menthol:Lauric acid (4:1) NADES represents a sustainable and efficient alternative for propolis bioactive compound extraction.

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2,4,5-Triaminopyrimidines: a new class of fluorescent nucleobase analogues for cell viability studies

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Fluorescent probes remain indispensable tools for studying biological processes and cell status, which includes cell viability. Nonetheless, the development of robust, sensitive, and versatile small molecules, combining easy and accessible synthesis with optimal photophysical properties, is still challenging. While monitoring cell viability is a cornerstone across biological sciences and drug discovery, most technologies are based on colorimetric assays and are often destructive, time-consuming, or rely on dyes with limited sensitivity [1,2].

In this work, we showcase a new class of fluorescent nucleobase analogues (FNAs) based on the 2,4,5-triaminopyrimidine scaffold that combine efficient synthetic routes with improved photophysical properties enabling their use in bioimaging and flow cytometry techniques. Moreover, the blue emission of these molecules is particularly useful for techniques using multiplexing by circumventing spectral overlap hindrances than can occur with the most common green- and red-emitting dyes.[3]

The staining capabilities of the new fluorophores were assessed using the eukaryotic cell model organism *Saccharomyces cerevisiae* (baker's yeast) as a model organism. Notably, these molecules were found to be virtually non-toxic to the model organism which is an important factor to consider when measuring cell viability. Interestingly, these molecules were found to specifically stain dead yeast cells with bright blue fluorescence, as demonstrated by fluorescence microscopy experiments. This was further validated by co-staining with propidium iodide, a commercially available fluorophore known to only penetrate cells with a compromised plasma membrane.[4] Moreover, dsDNA interaction studies paired with molecular docking strongly suggest that these new FNAs bind to the grooves of dsDNA molecules. In addition to this, preliminary flow cytometry experiments show their ability to effectively discriminate live and dead yeast populations, producing distinct and quantifiable populations.

Taken together, the photophysical properties and the performance of the new 2,4,5-triaminopyrimidines in bioimaging and flow cytometry techniques presents a significant advancement within the FNA class. These results underscore the potential of these molecules to putatively serve as accessible, versatile, and effective indicators of cell viability. Their successful application in fluorescence microscopy and preliminary flow cytometry studies with yeast further highlight the potential of these fluorophores to be used in multicolour imaging and high-throughput studies.

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Synergistic effects of physical pretreatments and fermentation on the nutritional value of lentils

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Lentils are one of the oldest leguminous crops consumed and cultivated around the world, and although they are well established as a nutritious food, their composition and nutritional value can differ among different varieties. Various processing methods have been explored to improve legumes' nutritional value, enhancing the concentration of micro and macronutrients, and improving the digestibility and bioavailability of proteins, fibers, and minerals in the grains [1].

This study aims to evaluate the influence of different physical processing methods (germination and cooking), both individually and in combination with lactic acid (*Furfurilactobacillus rossiae* and *Lactobacillus brevis*) and yeast (*Saccharomyces boulardii*) fermentation, on the nutritional profile of two lentils varieties (Beluga and Du Puy), with the goal of producing nutritionally richer flours. Germination was carried out for 72 hours in the dark at room temperature, while in parallel, the grains were soaked cooked in distilled water for 12 hours and then cooked for 20 minutes [2]. Fermentation was carried out by inoculating lentil flour doughs with starter cultures of the selected microorganism and allowing then to ferment for 24 hours at 30°C [3]. The fermented doughs were subsequently freeze-dried and analyzed for their nutritional composition following AOAC procedures.

The study revealed significant nutritional changes in lentils following germination, cooking, and fermentation. Carbohydrates levels consistently decreased, whereas protein content increased, especially in the Beluga variety after germination and fermentation with specific microorganisms. Fiber content varies by variety and treatment, increasing in some cases and decreasing in others. Fat content declined after germination and cooking but increased following fermentation. Ash content rose significantly in germinated and fermented Beluga lentils.

Briefly, all processing methods influenced the nutritional profile of both lentil varieties, with combined treatments showing the greatest potential to improve the nutritional properties of lentil flours, particularly by increasing proteins and fibers content.

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Hemolytic activity of biosurfactant extracts obtained in corn steep liquor for further inclusion in antibiotic formulations

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Agri-food residual streams serve as a source of relevant bioactive compounds. Corn steep liquor (CSL), a secondary stream from the corn-milling industry, was proved to be a source for the extraction of biosurfactants [1] and microorganisms able to produce antibiotics such as Gramicidin S [2]. Among these biosurfactant extracts, it is important to highlight the presence of both extracellular and cell-bound surfactants, which can be obtained according to patents WO 2014/044876 [3] and WO 2020/234501 [4], respectively. Given their demonstrated industrial potential, it would be of interest to evaluate the hemolytic activity of these extracts on eukaryotic erythrocytes, to assess their biocompatibility for the possible inclusion in different formulations for the pharmaceutical, cosmetic or agri-food industries.

In this work, extracellular and cell-bound biosurfactant extracts were produced at pilot scale following the patents abovementioned [3,4] and compared to other commercial biosurfactants and surfactants in terms of hemolytic activity. The optimized protocol was based on the hemolysis method of van der Knaap et al. [5] using sheep red blood cells. For this, different dilutions of the samples were tested by triplicate, and the percentages of hemolysis were calculated.

The results show that both extracellular and cell-bound biosurfactant extracts do not present hemolytic activity in concentrations at 10 g/L, showing higher biocompatibility than other biosurfactants tested at the same concentration. This opens the way for their potential incorporation into commercially relevant formulas for the pharmaceutical industry containing Gramicidin S, which is an antibiotic poorly soluble in water.

In future studies, additional cytotoxicity and biodegradability tests should be performed on these biosurfactant extracts in combination with antibiotics like Gramicidin, to determine their suitability for incorporation into pharmaceutical formulations.

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Advancing monoclonal antibody purification: the application of supported ionic liquids (SILs)

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The global prevalence of non-treatable/hard-to-treat diseases has been rising and is projected to escalate, driven by the growing of an aging population. In this way, novel biopharmaceuticals, including monoclonal antibodies (mAbs), have been arisen to overcome this problem. Unlike chemical drugs, mAbs are derived from living cells and therefore more complex and less stable. This, presents significant challenges in their production (upstream) and subsequent stages (downstream), driving up costs and timelines [1]. Supported Ionic Liquids (SILs), formed by the solid immobilization of ionic liquids (ILs), offer a promising solution due to the unique designer-level tunability, thereby facilitating the precise tailoring of mAbs' physicochemical properties [2].

In this scenario, the present study investigated a novel approach for the purification of anti-hepatitis C virus (anti-HCV) mAbs from Chinese Hamsters Ovary (CHO) cell culture supernatants using SIL-packed chromatographic columns.

The purification of anti-HCV mAbs was accomplished using two carefully chosen SILs: 1-methyl-3-propylimidazolium-based supported silica with chloride ([SSi][C₃mim]Cl) and propyltrioctylammonium-based supported silica with chloride ([SSi][N₃₈₈₈]Cl). This process successfully employed two distinct chromatographic approaches: (i) flowthrough mode – with protein impurities being adsorbed to the column, while mAbs were eluted; and (ii) bind-and-elute mode – where the opposite behavior occurred. The successful implementation of these contrasting separation mechanisms highlighted the inherent tunability conferred by the incorporated ILs within the SIL matrix. Afterwards, the anti-HCV mAbs' protein profile and integrity were confirmed by SDS-PAGE (Sodium Dodecyl Sulfate Polyacrylamide Gel Electrophoresis).

In conclusion, the results obtained in this study demonstrates that SILs allowed the development of a promising and cost-effective downstream strategy for mAbs. This shows promise as a substitute for existing downstream processing of mAbs within the industry.

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Sustainable biosurfactant extract from Corn Steep water: characterization and multifunctional applications in cosmetic, pharmaceutical and agrochemical formulations

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There is a growing demand for sustainable and compatible ingredients and additives across the cosmetic, pharmaceutical, and agri-food sectors. In this context, biosurfactants have emerged as a promising alternative to conventional chemical surfactants, which can be harmful to the environment, offering advantages such as biocompatibility, high efficiency, easy biodegradability, and production from renewable resources [1].

In this study, a biosurfactant (BS) extract was obtained from a naturally fermented stream in the corn wet-milling industry (corn steep water, CSW) using a dialysis-based process as a green alternative to conventional organic solvent extraction, previously optimized in earlier works [2]. The BS extract was thoroughly characterized and evaluated to determine its effectiveness as a solubilizing, emulsifying, spreading, and wetting agent [3].

The results showed that the BS extract obtained from CSW via the dialysis process was able to reduce the surface tension (ST) of water by 20 units, with a critical micellar concentration (CMC) of 0.14 ± 0.05 g/L, and is mainly composed of glycopeptides and peptides, like those produced by lactic acid bacteria. The extract was non-irritating (Mucosal Irritation Score < 0.07) and demonstrated promising potential as a formulation enhancer in Pickering emulsions with tocopherol and mica ($70.7 \pm 1.7\%$ EV, $89.1 \pm 2.3\%$ ES). In terms of spreading capacity, the BS extract effectively removed the essential oil layer on the water surface, creating a clear area (18.8 ± 2.0 cm²). Although its solubilization of chicken broth cubes was limited ($16.1 \pm 1.9\%$), the extract significantly enhanced the solubility of Cu-Oxy (17%) and moderately improved ZnP solubility (0.80%), outperforming conventional surfactants and providing a clear booster effect in formulations. Moreover, the BS extract displayed exceptional wettability, drastically reducing water contact angles on glass ($10.7 \pm 1.6^\circ$), steel ($13.5 \pm 1.1^\circ$), and polyethylene ($17.1 \pm 1.4^\circ$), surpassing most conventional surfactants. These multifunctional properties, combined with its biocompatibility, highlight its potential as a sustainable ingredient for cosmetic, pharmaceutical, and agrochemical formulations, offering an eco-friendly alternative to chemical surfactants.

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Emulsifying potential of a biosurfactant extract obtained from dairy by-products

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The large-scale production of dairy products inevitably leads to significant waste generation [1], a situation that must be addressed through the development of alternative applications aimed at enhancing the intrinsic value of these by-products. On the other hand, the extraction of natural compounds in compliance with the Cosmos (Cosmetic Organic Standard) framework requires the use of natural and organic solvents approved under this regulation. In addition, in previous works, it has been proven that corn mill wastewater sludge (CMWS) is an interesting source of biosurfactants [2], which opens the door to evaluate other food industry residues, rich in nutrients and spontaneously fermented, as a direct source of bioactive compounds. In this study, the objective was to obtain a biosurfactant-rich extract from the microbial biomass present in the residual stream generated during buttermilk production. For that, the waste stream was centrifuged, and the lactic acid bacterial biomass was subsequently washed with deionized water. The extraction process was carried out under Cosmos-compliant conditions using phosphate-buffered saline (PBS), following the protocol described by Bustos et al. [3]. Surfactant properties of the biosurfactant extract under evaluation were measured in terms of surface tension reduction and emulsifying capacity with olive oil, peppermint oil, tea tree oil and paraffin in aqueous systems. It was observed that the biosurfactant extract under evaluation was able to reduce the surface tension of water in 22 units with a percentage of emulsion higher than 50 % (see Figure 1), being in consonance with the surfactant and emulsifier properties of biosurfactant extracts produced by lactic acid bacteria [4]. Therefore, demonstrating its potential as emulsifier and surfactant agent for cosmetic and pharmaceutical industries.

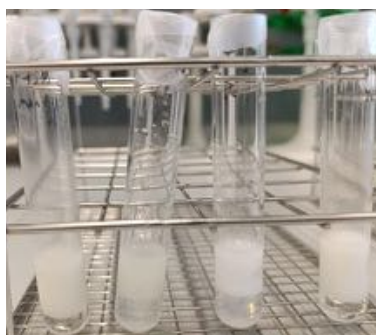


Fig.1. Aqueous emulsion systems of biosurfactant extract solutions with oils after 30 days.

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Role of nitrogen doping and carbon support structure in the oxidation of 5-Hydroxymethylfurfural to furans over Pt/C catalysts

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The oxidation of biomass-derived 5-hydroxymethylfurfural (HMF) produces several important furan derivatives that serve as key precursors for bio-renewable polymers and chemicals, such as 2,5-diformylfuran (DFF), 2,5-furandicarboxylic acid (FDCA), 5-hydroxymethyl-2-furancarboxylic acid (HMFCa), and 5-formyl-2-furancarboxylic acid (FFCA). The fully oxygenated product, FDCA, has garnered particular attention due to its role in the synthesis of PEF (polyethylene furanoate), a biobased alternative to PET (polyethylene terephthalate); however, the other furans also have important applications in various biorefinery processes [1]. Controlling the selectivity toward a specific product in HMF oxidation remains a major challenge, which can be addressed through catalyst tuning. Accordingly, this work combined the high catalytic activity of Pt with the beneficial properties of carbon supports, focusing on the impact of (i) the structure of the carbon support (carbon nanotubes (CNT) vs. carbon xerogels (CX)) and N-doping of the carbon on the selectivity of Pt/C in this process.

Technical-grade carbon nanotubes (CNTs) and carbon xerogels (CX) were functionalized with nitrogen using ball milling with melamine (CNT_M) [1], in situ doping with urea (CX_U) [2], or via treatment with gaseous NH₃ (CX_NH₃) [3]. Subsequently, 2 wt% Pt was deposited on pristine and N-doped supports via wet impregnation, followed by activation in a H₂ flow. Finally, the catalysts were tested in HMF oxidation using a Par-type batch reactor under mild reaction conditions (2.5 bar of oxygen at 90 °C for 20 hours). The obtained reaction mixtures were analysed using High-Performance Liquid Chromatography (HPLC).

Various types of nitrogen functionalities, including graphene-like nitrogen (N-Q), pyrrole-like nitrogen (N-5), and pyridine-like nitrogen (N-6), were successfully incorporated into the carbon supports, as confirmed by XPS and elemental analysis. Catalytic tests showed that Pt supported on pristine CNT delivered the best performance, producing 78.1% FDCA, followed by the benchmark catalyst (Au/TiO₂), which achieved a 70% FDCA yield under the same conditions. In contrast, Pt/CX favoured the partial oxidation of HMF, generating a mixture of HMFCa (22.1%), FFCA (33.6%), and FDCA (46.2%). Meanwhile, Pt supported on N-doped carbons hindered HMF conversion and increased selectivity toward intermediate furans, such as FFCA or HMFCa, depending on the carbon structure. Additionally, it was found that Pt supported on carbons with a higher N6 to N5 ratio mainly produced FFCA (over 60%) with only traces of FDCA (1-2% yield). This result was linked to the electronic interaction between Pt and N-doped carbon, which affected the adsorption and activation of reaction intermediates. Overall, N-doping of carbon supports in Pt catalysts hindered the final oxidation step of FFCA to FDCA, regardless of the support structure. Larger average Pt particle sizes were observed on the N-doped supports compared to pristine ones. The superior performance of Pt/CNT was attributed to smaller, more uniform Pt nanoparticles and the electronic interaction between Pt and the support, which facilitated the rate-determining final oxidation step.

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Structure–performance relationships of carbon-based supports in Fischer–Tropsch catalysts

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The Fischer–Tropsch synthesis (FTS) constitutes a pivotal catalytic route for the transformation of synthesis gas into liquid hydrocarbons [1], and the selection of an appropriate catalyst support plays a decisive role in dictating both selectivity and overall process efficiency. Among the various candidates, carbon-based supports are particularly attractive due to their high surface area, tuneable porosity, excellent chemical stability, and the ability to tailor their structural and electronic properties, all of which can enhance metal dispersion and catalytic performance [2]. In the present study, the effect of carbon-based supports with distinct structural and electronic characteristics was systematically examined for cobalt-based catalysts under industrially relevant operating conditions (220 °C and 20 bar) in a fixed bed reactor. The carbon materials investigated in this study included: carbon nanotubes (CNTs), nitrogen-doped carbon nanotubes (N-CNTs), carbon nanofibers (CNFs), and graphene. These supports exhibit pronounced differences in surface area, defect density, degree of graphitization, and propensity for metal–support interactions, all of which significantly influence cobalt dispersion and, consequently, catalytic performance. Catalysts with comparable cobalt loadings were synthesized, activated, and subsequently evaluated with respect to CO conversion and product distribution.

The experimental results revealed clear correlations between support nature and catalytic behaviour. Graphene exhibited the lowest selectivity toward methane. Catalysts supported on CNTs preferentially produced hydrocarbons within the gasoline-range fraction (C₅–C₁₁), whereas N-CNTs enhanced the formation of heavier hydrocarbons in the diesel-range fraction (C₁₂–C₂₀). In contrast, CNF-supported catalysts were distinguished by their notably low selectivity toward CO₂, indicating a reduced propensity for secondary reactions such as the water–gas shift.

This study underscores the critical influence of support nature and architecture on product distribution in FTS over cobalt catalysts. The distinct behaviours observed across the studied materials demonstrate that both structural morphology and surface chemistry govern the balance between activity, chain-growth probability, and secondary reactions. These findings not only confirm the pivotal role of carbon-based supports in steering hydrocarbon selectivity but also provide a framework for the rational design of next-generation FTS catalysts. Strategic tailoring of support properties can steer product selectivity toward targeted hydrocarbon fractions, unlocking more efficient and sustainable routes for syngas-to-liquid fuel conversion.

Acknowledgements

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Sustainable magnetic catalysts for glycerol valorization into Solketal

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The rapid growth of biodiesel production in recent years has led to the generation of large amounts of glycerol as a by-product, accounting for about 10% of the total biodiesel produced, with a typical purity of 50–55% [1]. The oversupply of crude glycerol exceeds market demand, and large-scale purification is economically unfeasible [2,3]. Nevertheless, glycerol represents an abundant and versatile raw material with strong potential for conversion into value-added products. Among the different catalytic strategies investigated for glycerol valorization, acetalization has emerged as a particularly promising pathway. The resulting products not only hold industrial relevance, but also contribute to advancing circular economy models closely linked to the biodiesel sector.

Keggin-type heteropoly acids (HPAs) are highly efficient catalysts for glycerol acetalization to produce solketal [4]. However, their solubility limits recovery and reuse, making immobilization on inert supports a practical solution. Phosphotungstic acid (HPW₁₂) was incorporated into magnetic nanoparticles (NiFe₂O₄@PW₁₂ and Fe₃O₄@PW₁₂) and immobilized on low-cost natural supports, biochar from invasive plants and cork stoppers. The four magnetic composites were characterized by FTIR-ATR, SEM-EDS, ICP-OES, BET, XRD, and potentiometric titration. Their catalytic activity was evaluated in the acetalization of glycerol with acetone. Remarkably, the cork-supported composite Fe₃O₄@PW₁₂@B2 achieved 87% glycerol conversion with 97% solketal selectivity after only 5 min at 60 °C. Magnetic composite catalytic stability of Fe₃O₄@PW₁₂@B2 was studied during 4 consecutive reutilization cycles, without loss of catalytic performance (Figure 1).

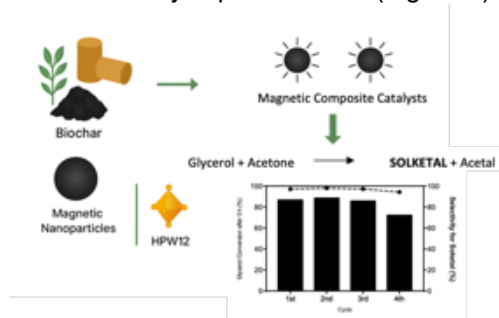


Fig. 1. Scheme of preparation of magnetic composite catalysts to use in acetalization reaction. Glycerol conversion results and selectivity obtained at 3 h of glycerol acetalization with acetone, using Fe₃O₄@PW₁₂@B2 composite catalyst, over the four consecutive reutilization cycles. Reactions were performed using a ratio of 1:15 glycerol/acetone, 24 mg of catalyst and 60 °C.

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A novel lipase form with enhanced activity across diverse substrates

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Biocatalysis has become one of the most promising strategies, aiming for environmentally friendly and sustainable processes. The faster reaction rates and the use of mild temperature conditions are among its many advantages [1]. However, limited substrate specificity remains a main barrier to its broader application [2].

This work presents several strategies to overcome a specific limitation of one of the most studied industrial lipases (Lipolase®). The chemical modification of lipase from *Thermomyces lanuginosus* with small or large molecules has shown a notable enhancement in its esterification and transesterification activities [3-5]. The data indicated an expansion of the enzyme's active site, which directly influenced the range of substrates that could be processed in the reaction. Additionally, the chemical modification increased the enzyme's tolerance, enabling it to operate under solvent-free conditions without reducing the reaction yield.

This pioneering work demonstrates the promising potential for more reactions where biocatalysis can be employed, paving the way for a cleaner and more environmentally friendly industry.

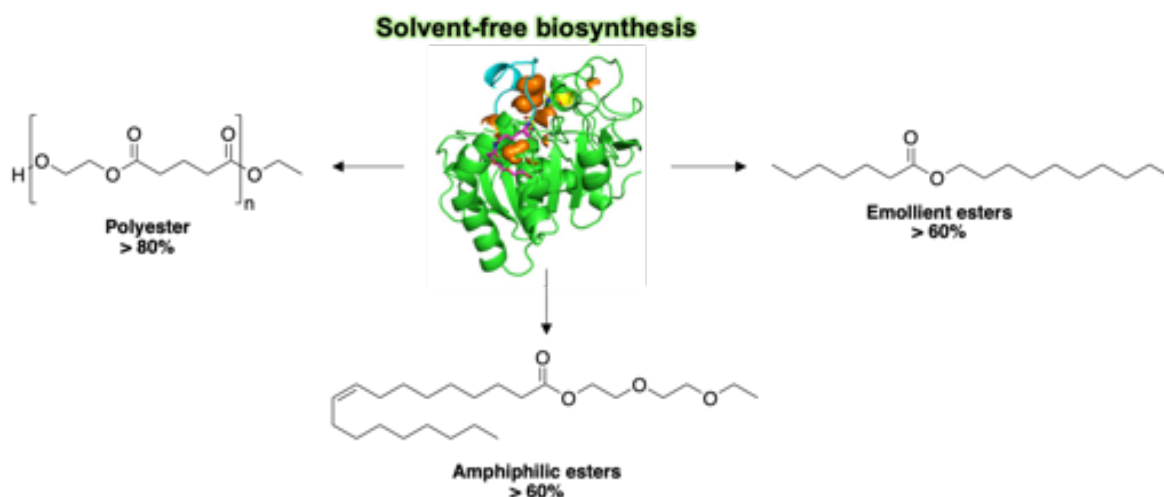


Fig. 1. Esters that are biosynthesised under solvent-free conditions by chemically modified lipases.

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Challenges in the synthesis of substituted aminobenzaldehydes via green and auto-catalytic approaches

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Aminobenzaldehydes are key intermediates in pharmaceuticals, dyes, fragrances, and fluorescent probes, yet their preparation often requires multi-step sequences or harsh reaction conditions and limiting functional group tolerance [1,2]. More recently, transition-metal-catalysed C–N bond formation has emerged as a direct and modular alternative, particularly using palladium (Buchwald–Hartwig) or copper (Ullmann-type) catalysis [3–5].

Copper-catalysed systems, especially those employing CuI and amino acids, offer a cost-effective and sustainable platform and have been widely applied to C–N bond formation between aryl or vinyl halides and amines or N-containing heterocycles [6,7]. However, *meta*-halogenated benzaldehydes remain largely excluded from this chemistry. Reports on the amination of 3-bromobenzaldehyde are limited to harsh, ligand-free conditions [8], and no general *L*-proline-based protocol has been established.

In this work, we studied the reactions of halo-benzaldehydes with secondary amines, aiming to generate substituted aminobenzaldehydes. The reactions were carried out under heat with and without a catalyst (Fig. 1). *Para*-aminobenzaldehydes were accessed via a solvent-free, autocatalytic transformation. *Meta*-aminobenzaldehydes were obtained under mild CuI/*L*-proline catalysis, delivering the products in moderate yields. The catalytic Cu-*L*-proline protocol expands the scope of Ullmann-type couplings to aldehyde-containing substrates and provides streamlined access to functionalized aminobenzaldehydes for downstream synthesis.

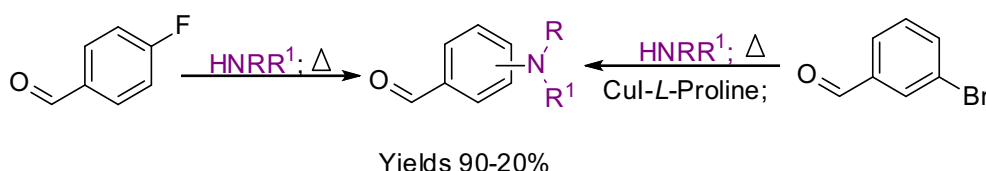


Fig.1. Synthesis of substituted aminobenzaldehydes

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Integração da Química numa disciplina STEAM: potencial e desafios da modelagem tridimensional de moléculas no 8.º ano

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A atividade “Modelagem tridimensional de moléculas à escala” foi desenvolvida no âmbito da disciplina STEAM junto de alunos do 8.º ano do 3.º Ciclo do Ensino Básico, tendo como principal objetivo a construção de modelos moleculares tridimensionais com recurso ao software MS Paint 3D. O estudo centrou-se na implementação e avaliação de práticas STEAM e visou consolidar conteúdos da disciplina de Química, especificamente no domínio das Reações Químicas. A literatura recente mostra que a integração de abordagens STEM em Química potencia não apenas a compreensão conceptual mas também competências transversais como o pensamento crítico e criativo [1] [2] [3].

Para além da consolidação de conteúdos disciplinares, a atividade procurou fomentar a interdisciplinaridade característica das práticas STEAM, promovendo a articulação entre ciência, tecnologia e representação digital. A modelagem tridimensional permitiu aos alunos transitar de uma visão abstrata da constituição da matéria para uma representação concreta e manipulável, explorando dimensões de escala atómica difíceis de apreender em representações tridimensionais tradicionais.

A amostra incluiu 51 alunos de uma escola privada de contexto socioeconómico acima da média, sendo o desenho metodológico de natureza qualitativo-quantitativa. A atividade decorreu durante duas semanas, em regime individual. Os alunos foram desafiados a representar moléculas à escala, respeitando raios atómicos e a geometria molecular, incluindo a construção de legendas descritivas no trabalho.

Os resultados quantitativos evidenciaram uma aceitação reduzida, com uma minoria dos alunos a valorizar a atividade e uma proporção significativa a manifestar desagrado. A análise qualitativa revelou que as principais dificuldades se prenderam com a manipulação do software e a aplicação rigorosa das escalas. Foram identificados erros recorrentes, mas também trabalhos de boa qualidade, incluindo representações corretas de geometrias moleculares. Apesar da baixa popularidade, a atividade revelou potencial pedagógico ao permitir a visualização tridimensional de moléculas e ao favorecer a compreensão de conceitos abstratos. Alguns alunos reconheceram a utilidade da modelagem para perceber a escala atómica no entanto, destacaram a necessidade de abordar primeiro os conceitos teóricos na aula de química antes da construção dos modelos 3D.

Conclui-se que, embora a receptividade dos alunos tenha sido reduzida devido às dificuldades encontradas na manipulação da ferramenta digital utilizada, a atividade alcançou os objetivos pedagógicos, mostrando-se eficaz no apoio à aprendizagem de conceitos abstratos de Química. Sugere-se a introdução de formação prévia em ferramentas digitais ou a utilização de softwares mais intuitivos, de modo a potenciar a aceitação de futuras atividades STEAM.

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Práticas supervisivas na formação inicial de professores de Química: uma revisão integrativa

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A presente revisão da literatura tem como objetivo contribuir para a compreensão das práticas supervisivas na formação inicial de professores de Química em Angola, que se revela pertinente, cuja pertinência se deve à escassez de estudos sobre o tema no referido contexto. Esta revisão foi orientada pela seguinte questão de investigação: Que práticas supervisivas são desenvolvidas pelos professores orientadores de Química durante a formação inicial de futuros professores?

Trata-se de uma revisão integrativa da literatura, conduzida nas bases Scopus e Web Of Science, seguindo a metodologia PRISMA. Esta opção metodológica, permitiu a inclusão de artigos, revisões e capítulos de livros, considerando o reduzido número de estudos sobre supervisão em Química e possibilitando uma compreensão mais ampla do fenómeno. Foram aplicados como critérios de inclusão: recorte temporal (2019–2024) e idioma (inglês). Do total de 346 publicações identificadas, sete cumpriram os critérios de elegibilidade, tendo sido analisadas em profundidade.

Após a leitura integral dos artigos, procedeu-se à análise de conteúdo, identificando categorias emergentes que refletem diferentes dimensões. Dessa análise destacaram-se três categorias: a primeira refere-se aos contextos laboratoriais e experimentais, onde a orientação sistemática é essencial para o desenvolvimento de competências técnicas, a compreensão dos fenómenos científicos e a motivação dos estudantes, reforçando o papel central do supervisor no trabalho experimental [1,2]. A segunda categoria centra-se na formação inicial de professores, evidenciando a supervisão como mediadora entre teoria e prática, sendo o feedback pedagógico determinante para o desenvolvimento do conhecimento profissional e para a consolidação da identidade docente [3]. A terceira categoria relaciona-se com a integração de tecnologias digitais e práticas de ensino a distância, em que a supervisão desempenha uma função estruturante, assegurando a qualidade da aprendizagem e o apoio necessário em contextos virtuais e híbridos [4,5].

Os resultados sugerem que estas dimensões são determinantes, tanto para a aprendizagem em Química, como para a profissionalização dos docentes e futuros professores em diferentes contextos educativos. Todavia, a produção científica nesta área revela limitações, nomeadamente a escassez de estudos comparativos em diferentes realidades internacionais e a ausência de investigações longitudinais que permitam avaliar de forma consistente o impacto da supervisão na formação inicial de professores de Química e nos resultados de aprendizagem.

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O Jogo do Equilíbrio: uma experiência de aprendizagem baseada em jogos no ensino do Princípio de Le Châtelier

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Pela sua natureza intrínseca, a Química é muitas vezes considerada pelos alunos como uma ciência de difícil aprendizagem. Este facto exige que os professores integrem nas suas práticas abordagens inovadoras que promovam envolvimento ativo dos alunos na construção de uma aprendizagem significativa. O *Game-Based Learning* (GBL) tem-se destacado neste âmbito, ao potenciar a motivação e o envolvimento dos alunos na aprendizagem, a interatividade e o desenvolvimento de competências cognitivas [1] [2]. Revisões recentes apontam que os jogos podem facilitar a compreensão de conceitos complexos, como os de equilíbrio químico, mas também revelam desafios quanto à sua aplicação prática e à adequação para introdução de novos conteúdos [3] [4] [6].

Este estudo, desenvolvido no âmbito da formação inicial de professores de Física e de Química, pretendeu analisar o impacto da utilização do GBL no ensino do equilíbrio químico, explorando o potencial e as limitações desta abordagem na introdução do Princípio de Le Châtelier.

A intervenção decorreu numa aula de 90 minutos com 25 alunos do 11.º ano de uma escola pública da região de Aveiro. Os estudantes, organizados em grupos de 4 elementos, participaram no *O Jogo do Equilíbrio*, um jogo de cartas que envolvia a análise de reações químicas e a previsão das possíveis alterações a esse equilíbrio quando algumas variáveis do sistema eram alteradas (temperatura, concentração, pressão, volume e presença de catalisador). Foram recolhidos registos escritos no *Padlet*, observações diretas e feedback verbal dos alunos, posteriormente analisados de forma descritiva.

Os alunos demonstraram elevada motivação e envolvimento, confirmando a relevância do GBL na promoção da participação ativa [5] [3]. Contudo, dificuldades emergiram na gestão do tempo, na interpretação das instruções e, numa menor extensão, no domínio das ferramentas digitais utilizadas. Os registos mostraram que alguns alunos sentiram frustração por não terem conseguido ser bem sucedidos no jogo. Os dados sugerem ainda que a estratégia poderia ter alcançado maior sucesso se explorada durante um intervalo de tempo maior e se utilizada para consolidar conteúdos já lecionados ou aplicá-los a novos contextos, em vez de introduzir novas aprendizagens.

O GBL apresenta elevado potencial no ensino da Química, ao promover motivação, colaboração e aprendizagem ativa. Todavia, a sua utilização para introduzir novos conteúdos deve ser repensada, pois a sua eficácia foi limitada. Este estudo reforça a pertinência de integrar metodologias inovadoras no ensino, destacando simultaneamente os desafios da sua implementação em contextos reais.

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Del átomo al algoritmo: transformando la enseñanza de la Química con inteligencia artificial

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La enseñanza de la Química enfrenta el reto de hacer comprensibles conceptos abstractos y procesos invisibles para el estudiante. En este contexto, la Inteligencia Artificial (IA) emerge como una herramienta transformadora que permite rediseñar las experiencias de aprendizaje desde lo molecular hasta lo computacional. Diversos estudios han demostrado que la IA puede facilitar el aprendizaje autónomo y significativo mediante simulaciones, asistentes virtuales y algoritmos que modelan propiedades moleculares [1].

Este trabajo explora cómo la IA puede integrarse en la didáctica de la Química, tanto en educación secundaria como superior. Por ejemplo, Baidal Castillo (2025) [2] destaca cómo el uso de plataformas inteligentes mejora la comprensión de reacciones químicas complejas en estudiantes de bachillerato, mientras que Pérez Rodríguez, Herráez Aguilar y Montalvo Quirós (2024) [3] analizan el impacto de herramientas como Bing y Wolfram Alpha en la enseñanza universitaria, promoviendo una mayor interacción y personalización del aprendizaje.

Además, se discuten los desafíos éticos, técnicos y pedagógicos que implica su implementación, así como las competencias docentes necesarias para su uso efectivo. Este enfoque propone una transición desde la enseñanza tradicional hacia un modelo híbrido, donde el pensamiento químico se potencia con herramientas algorítmicas, promoviendo una educación más inclusiva, personalizada y crítica.



Fig.1. Del Átomo al Algoritmo

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A Química no 1.º ciclo do ensino básico: um programa de formação personalizado de professores sobre práticas *hands-on*

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O ensino das ciências nos primeiros anos de escolaridade é essencial para o desenvolvimento de competências fundamentais, como o pensamento crítico e o raciocínio científico, preparando os alunos para os desafios futuros e promovendo uma sociedade cientificamente literada [1]. As atitudes dos alunos em relação às ciências são moldadas desde cedo pelas experiências educativas, sendo as abordagens de ensino ativas e envolventes cruciais para fomentar o interesse e o sucesso futuro [2] [3] [4]. No caso da Química, a complexidade conceptual, o grau de abstração e a quimiofobia frequentemente associada a esta ciência/disciplina reforçam a importância de atividades práticas e *hands-on* capazes de tornar a aprendizagem significativa e motivadora [5].

Neste enquadramento surge o programa ExperimentaQuímica no 1.º ciclo do ensino básico (1.º CEB), um modelo de formação personalizado que se distingue pela flexibilidade de implementação, apoio individualizado aos professores deste nível de escolaridade, autonomia na escolha e adaptação das atividades e uso de materiais seguros, mas semelhantes aos de um laboratório. O estudo apresentado teve como objetivo compreender em que medida a participação nesta formação promove mudanças nas atitudes e práticas pedagógicas dos professores do 1.º CEB, bem como avaliar de que forma este modelo os capacita e motiva para integrar mais práticas de ensino de Química e, em particular, com forte componente experimental.

A investigação envolveu 18 professores de escolas públicas e privadas da região do Grande Porto, Portugal. Foram disponibilizados materiais, vídeos de apoio, documentos norteadores e acompanhamento direto em sala de aula. Os instrumentos de recolha de dados incluíram questionários e entrevistas, previamente validados por especialistas em Educação em Química.

Os resultados apontam para melhorias significativas nas práticas letivas, com aumento da confiança dos professores, diversificação de estratégias pedagógicas e valorização da prática experimental. Foram também relatadas mudanças positivas nos alunos, nomeadamente maior envolvimento, interesse e compreensão dos conceitos de Química. Contudo, subsistem desafios relacionados com a continuidade das práticas após o término do programa, devido a limitações institucionais e de recursos.

Em termos de significância, este estudo demonstra a eficácia de um modelo de desenvolvimento profissional centrado em atividades *hands-on* com apoio individualizado no 1.º CEB, colmatando lacunas da formação inicial generalista e produzindo ganhos significativos na autoconfiança, no domínio de recursos e no entusiasmo para ensinar ciências. O modelo revelou-se uma via eficaz para aproximar teoria e prática, reforçando a importância da formação personalizada e ativa.

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Gamificación como recurso para el fortalecimiento del aprendizaje a través del 2º Concurso de Conocimientos en Química Analítica 2025: “Dr. Gaston Charlot”



Elaborado por E.A. Tovar F. (2025) en Microsoft Power Point

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El proceso de enseñanza-aprendizaje de la Química Analítica (QA) implica un desafío constante de innovación en la Facultad de Química de la UNAM. La implementación de la investigación documental y la gamificación en el proceso de enseñanza-aprendizaje han demostrado motivar la formación académica de los estudiantes [1, 2]. Con base en lo anterior, se presenta el desarrollo de la segunda edición del Concurso de Conocimientos en QA 2025 “Dr. Gaston Charlot”, realizado el 12 de septiembre del 2025 en instalaciones de la FQ, UNAM, en el marco de los objetivos del proyecto DGAPA-UNAM PAPIIME PE201324 [3]. La estrategia didáctica incluyó la elaboración previa de un banco de preguntas (incluyen respuestas) obtenidas de 44 referencias especializadas en el área (libros y manuales de prácticas de laboratorio) con una cantidad de 386 preguntas. Se abordaron los programas de estudio (considerando los objetivos de aprendizaje) de las asignaturas impartidas en el Departamento de Química Analítica (DQA) correspondientes a los planes de estudio de las cinco de seis licenciaturas de la FQ [4]: Q. Analítica I, II y III (Claves 1402, 1504, 1604 y 1507/1602), Analítica Experimental I, II y III (1607/1700, 1802) y Q. Analítica Instrumental I y II (1612, 1803).

La segunda edición complementó la dinámica base del primer concurso de conocimientos: “100 Químicos Analíticos Dijeron” (20 de Septiembre 2024) [5], a través de una ruleta en línea con la adición de preguntas en las modalidades siguientes: “interpretar el diagrama”, “completar la frase”, además de preguntas conmemorando la Celebración de los 60 Años del Posgrado en la FQ, UNAM (que incluyen los 50 Años del DQA). Los alumnos participantes fueron evaluados por el Jurado integrado por Académicos y Jefatura del DQA, para elegir los tres primeros lugares de acuerdo al desempeño durante las dos fases del concurso: “Helmuth Pitsch” (primera, por equipos) y “Alain Quére Thorent” (segunda, individual). Los reconocimientos para los alumnos ganadores corresponden a estímulos económicos procedentes de proyecto, y de patrocinadores (Jaru [6], Merck [7] y ARP Soluciones). La estrategia educativa surge de actividades de investigación formativa a través del Programa de Servicio Social SS-2025-12/157-1011 y Programa de Estancias Cortas de Investigación.

En los siguientes enlaces del sitio institucional AmyD [8], se puede visualizar el material del concurso: Proyecto PAPIIME201324: <https://amyd.quimica.unam.mx/course/view.php?id=459&section=2>. Exposición del concurso: https://amyd.quimica.unam.mx/pluginfile.php/19628/mod_resource/content/2/expos%20In%20Situ%20o%20Concurso%2020250925.pdf. Memorias Fotográficas: https://amyd.quimica.unam.mx/pluginfile.php/19630/mod_resource/content/1/fotos%20ext%20concurso%202025.pdf (extracto) y <https://drive.google.com/drive/folders/1RuEEqR490NqPBAY0a-8aeQWs6bz4JEAL> (completo).

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Museu da Escola Sá de Miranda: um património científico ímpar

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A instituição do Ensino Liceal em 1836, no quadro da Reforma de Passos Manuel, constituiu um marco da modernização educativa em Portugal. Entre 1836 e 1905, a Física e a Química adquiriram progressiva relevância curricular, mas o ensino experimental permaneceu constrangido pela insuficiência de laboratórios e de equipamentos, realidade exemplarmente ilustrada pelo Liceu de Braga, atualmente designado Escola Secundária Sá de Miranda. A reforma de 1914 reforçou a valorização do trabalho prático e a atualização dos recursos pedagógicos, mas no século XX, sob o Estado Novo, o programa de construções liceais veio evidenciar a discrepância entre a retórica modernizadora e a persistente escassez de meios, tornando esta escola um caso paradigmático dessa tensão entre ideologia e prática escolar.

Ao longo da sua trajetória, a Escola Secundária Sá de Miranda reuniu um espólio de excecional valor histórico e científico, composto por aparatos laboratoriais, mobiliário, coleções pedagógicas e documentação, atualmente preservado no seu museu escolar. Criado após a requalificação do edifício em 2010, este espaço museológico afirma-se como núcleo privilegiado de memória, investigação e divulgação, dinamizado através de exposições, conferências, aulas práticas e ações de formação. Entre o acervo, destacam-se dois quadros parietais recolhidos do antigo Gabinete de Química, que exemplificam a continuidade e a atualização dos recursos didáticos em diferentes contextos históricos: a tabela dos pesos atômicos aproximados de 1912 (inv. ME/402849/65) e a tabela periódica dos elementos de 1955 (inv. ME/402849/66). A figura 1 apresenta os quadros parietais incluídos na exposição permanente do museu, os quais plasman testemunhos eloquentes da evolução pedagógica da Química e da relevância patrimonial da instituição. Assim, a Escola Secundária Sá de Miranda não se limita a ser herdeira de uma tradição liceal centenária, assumindo-se igualmente como guardião de um património educativo ímpar, singular na cidade de Braga e de inegável valor no panorama nacional, essencial para a compreensão crítica da história da educação em Portugal.



Fig.1. Tabela Periódica dos Elementos (1955) e Tabela de Pesos Atômicos Aproximados (1912).

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Dehydrodiketopiperazines: minimalist peptide blocks for self-assembled materials

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Cyclic dipeptides, diketopiperazines (DKPs), emerged in the last decades as chemically stable multifunctional structural building blocks for development of supramolecular complex materials, *e.g.*, nanoparticles, nanovesicles, hydrogels. Dehydrodiketopiperazines (Δ DKP) - diketopiperazine derivatives featuring a dehydroamino acid residue, appear in many natural products that display antibacterial, anticancer and other biological activities. Synthetic Δ DKP are under development as promising anticancer agents. Enhanced conformational rigidity and ability to form directional 2D hydrogen bonding patterns, π - π stacking, hydrophobic and van der Waals interactions, endow Δ DKPs with superb self-assembly propensity [1,2].

In this work we disclose the synthesis, characterisation and the self-assembly behaviour of a focused library of Δ DKP. A cost-effective, sustainable and versatile synthetic route was devised for Δ DKP using methodologies developed by the research group. The Δ DKP library was characterised by ¹H and ¹³C NMR spectroscopy and mass spectrometry. The biocompatibility of the Δ DKP library was established using the MTT assay with the HaCaT keratinocyte and Caco-2 intestinal epithelial cell lines. The self-assembly behaviour of the Δ DKP library was characterised by UV-VIS spectrophotometry, fluorescence spectroscopy and circular dichroism. Gelation tests identified several Δ DKPs that formed self-assembled hydrogels by different gelation methodologies. Some Δ DKPs formed self-assembled aggregates in aqueous solution under the tested gelation conditions. Others, originated crystalline solids in organic solvents. The gelation kinetics and the mechanical and viscoelastic properties of the hydrogels was probed by oscillatory rheology. The microscale morphology of the self-assembled aggregates and the crystalline solids was studied by microscopy techniques (Figure 1).

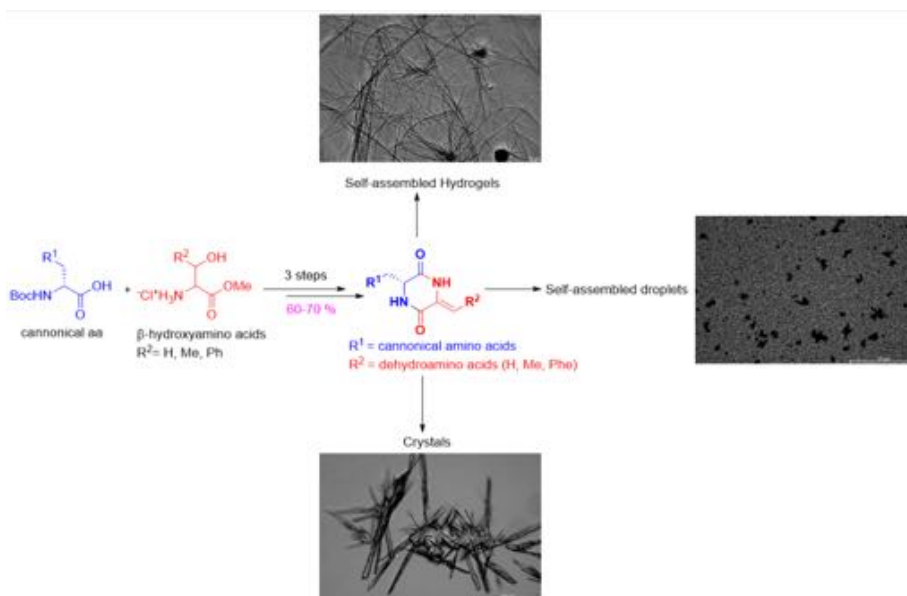


Fig.1. General structure of the synthesised Δ DKPs and their self-assembly pathways.

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When biomass-based Carbon Quantum Dots meet photocatalysis

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As technology progresses and societies grow, so does grow the production of waste and pollution. Thus, searching for innovative and green solutions to reuse unwanted materials and convert them into valuable and exploitable resources has become a pressing scientific endeavor. Approaches taken by the scientific community revolve around the reduction of waste production and/or the degradation of already established pollutants. In both problems, nanomaterials have proven to be helpful, with Carbon Quantum Dots taking a leading role. This is attributed to their easy synthesis from waste materials and their potential use as photocatalysts for the degradation of pollutant compounds, due to their photo- and chemical stability, low toxicity and easy surface modification [1].

Carbon Quantum Dots (CQDs) are spherical nanoparticles with size below 10 nm, consisting of a mixture of sp^2 and sp^3 carbons arranged in an amorphous to crystalline structure [2]. CQDs can be produced by either a top-down or a bottom-up approach, depending on whether the synthetic precursors utilized during synthesis are complex resources or small molecules. Considering that top-down approaches allow for the reduction of waste products through their use as carbon sources for the synthesis of CQDs, it is of great interest to develop a rational design strategy for their sustainable synthesis. Additionally, due to their diverse surface chemistry and optical properties, CQDs have proven their worth in the photocatalytic degradation of pollutants, from dyes to metals [3], thus closing the life-cycle of these nanomaterials in environmental protection.

This work focuses on the optimization of the synthesis of CQDs *via* microwave irradiation, using a CEM Discover microwave synthesizer. The selected precursor is coffee silverskin (CS), a significant waste product generated during the roasting of coffee. Disposal protocols for CS are oftentimes challenging because of the high percentage of compounds such as chlorogenic and caffeic acids, as well as caffeine, in its rich chemical composition [4]. However, for the synthesis of CQDs, those compounds provide a source for diversity of surface functional groups, including carboxylic acids, amides and phenolic hydroxyl groups. With the objective of obtaining CQDs with maximized photocatalytic efficiency, the impact of synthesis variables such as temperature, reaction time, pH, and coffee silverskin concentration was evaluated within a design of experiments (DoE) framework. Characterization of the prepared CQDs suspensions was achieved *via* UV-Vis spectrophotometry and fluorometry analysis, as well as methylene blue degradation assays. For the later, an original design for the photocatalytic reactor was developed *in lab*, focusing on utilizing 96-well plates to maximize the number of conditions tested by the irradiation cycle. Additionally, relative fluorescence quantum yield values were determined resorting to quinine sulphate as a standard reference, as well as the determination of particle size and zeta potential through zetasizer analysis. Preliminary results revealed a pronounced impact of the reaction temperature and pH value in modulating the CQDs photocatalytic efficiency and quantum yield, both considering the role of each variable individually or when in interaction with the other studied variables.

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PC-Based liposomal formulations for efficient delivery of a novel highly potent anticancer drug

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Cancer is one of the deadliest diseases worldwide. Overall, in 2020, approximately 7% and 9% of the deaths were related to breast and colorectal cancers, respectively [1]. Despite the existing treatments, the adverse side effects and the increasing drug resistance to the current therapies lead to a reduced quality of life for patients and poor prognosis [2,3].

In response, the scientific community has been developing new strategies [4,5] and new cytotoxic agents [6] more targeted to the molecular characteristics of these cancers. Still, during the process of drug discovery and development, aqueous solubility issues and toxicity often arise while trying to increase the potency of the new compounds, preventing their progress in the pipeline [7].

In our research group, a new class of pyrimidine compounds with anticancer activity was discovered. One of the derivatives, exhibited excellent *in vitro* anticancer activity in triple-negative breast and colorectal cancer cell lines, but displayed poor solubility in aqueous medium and low safety profile. To tackle these issues, the compound was encapsulated in liposomal formulations based on phosphatidylcholines (PC). The development, characterization, anticancer activity, toxicity evaluation, and cell uptake of drug-loaded liposomal formulations will be presented [8].

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Computer modelling, one tool to rule them all? From carbon nanotubes to biomolecules, covalent organic frameworks and moiré synthetic systems

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Computer modelling is arguably the most prevalent tool in physical sciences today to predict and rationalize materials properties. From my career spanning over 25 years, I will didactically discuss key choices in modelling and its strengths and limitations when honestly addressing real-world problems. Also, I will discuss the explosive development of the field in recent years, **illustrated, in a non-technical way, by specific examples from my research** [1-9]. Namely, from my early studies in CO and CO₂ adsorption on metal oxides to functionalized **carbon nanotubes** [1], comparing 20 years ago and now! In addition to the modelling of inorganic **and biomolecular** graphene **composite systems** [2] and organic Li cathodes [3]. Special relevance will be given to the key role played by modelling in Covalent Organic Frameworks (COFs), as a fundamental characterization tool able to predict structures [4], rationalize polymerization on surfaces [5] and the formation of **moiré 2D materials** [6]. Last but not least, we will discuss the on-surface formation of **nanographenes** [7], magnetic nanographenes properties such as uthrene and **triangulene** [8], as well as the synthesis and **computational characterization of very large systems** as monodisperse ultralong **graphene nanoribbons** [9].

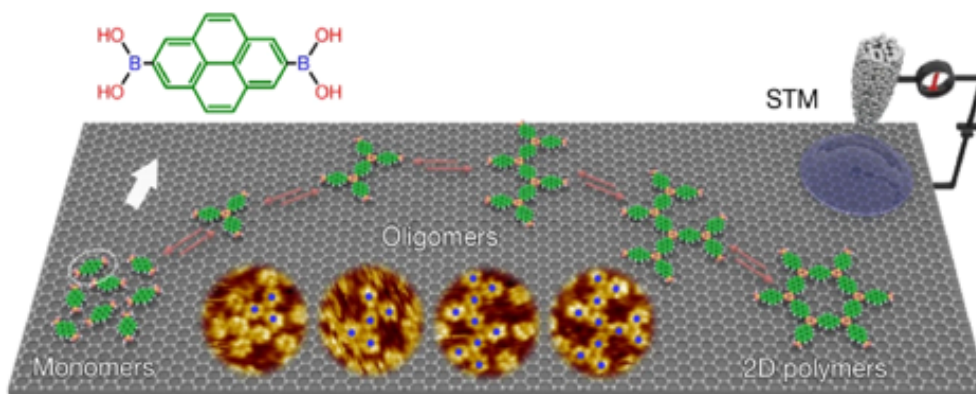


Fig. 1. Real-time observation of 2D dynamic covalent polymerization [5].

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Surface Chemistry-driven control of magnetic interactions in iron oxide nanoparticles for MRI and hyperthermia applications

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Theranostics, which integrates diagnostic and therapeutic functions into a single platform, has emerged as a promising strategy to enhance the efficacy and precision of treatments for complex diseases such as cancer. In this context, iron oxide magnetic nanoparticles (MNPs) have attracted considerable attention due to their ability to improve magnetic resonance imaging (MRI) contrast, while also enabling therapeutic applications such as magnetic hyperthermia and controlled drug release [1]. Nevertheless, the translation of MNPs-based systems into biological settings faces significant challenges, including nanoparticle aggregation and loss of functional efficiency [2]. These limitations highlight the need for further optimization of MNP design and stability to fully realize their clinical potential.

In this work, we explore how chemical surface modulation of iron oxide nanoparticles enables the design of distinct experimental models, including both single-core and multi-core architectures, with controlled interparticle distances and aggregation states (Fig 1). Magnetic cores were synthesized via thermal decomposition, followed by surface functionalization through ligand exchange and the formation of tunable silica (SiO₂) shells via microemulsion. These strategies allow the study of dipolar interactions and their influence on magnetic properties, such as effective anisotropy [3] and relaxation behavior, and also mimicking intracellular-like environments [4]. This approach provides insights into how nanoscale organization affects MRI contrast and magnetic hyperthermia performance, guiding the rational design of iron oxide nanoparticle-based theranostic systems.

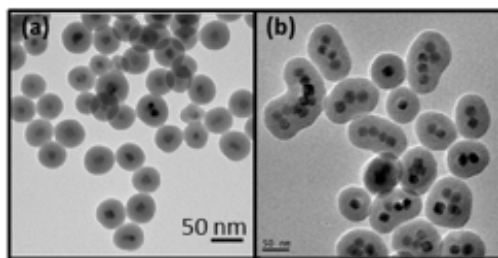


Fig.1. Transmission electron microscopy (TEM) of iron oxide@SiO₂ NPs showing individual (single-core) and collective (multi-core) structures, synthesized via thermal decomposition and microemulsion methods.

Agradecimientos

This work has been partially supported by Spanish Ministry of Science and Innovation (PDC2021-121455-I00, PID2022-141258OB-I00, PID2023-150379OB-C22, PLEC2022-009217), Xunta de Galicia (Axencia Galega de Innovación: IN607A2022-03), Axencia Galega para a Xestión do Coñecemento en Saúde (SA304D-PRIS-T-2024/03), Instituto de Salud Carlos III (ISCIII/PI21/01256/ PI24/00813) and by the European Union (DTS23/00103 and AC21_2/00014) under the frame of EuroNanoMed. R. Iglesias-Rey (CP22/00061) from the Miguel Servet Program of Instituto de Salud Carlos III. P. García-Acevedo thanks to Axencia Galega de Innovación for his Posdoctoral Grant ("Axudas de apoio á etapa de formación posdoutoral" - IN606B-2024.1).

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Hardy kiwi loaded liposomes: nano-delivery of natural antioxidants

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The therapeutic and cosmetic potential of natural bioactive compounds is often limited by their intrinsic instability, poor bioavailability, and low skin permeability [1]. Liposomes, owing to their biocompatibility and versatility, represent an effective strategy to overcome these limitations [1]. Therefore, the aim of the present study was to design liposomal carriers to enhance the delivery of bioactive compounds from *Actinidia arguta* fruit (known as hardy kiwi), rich in antioxidant phenolics. The extract was obtained by ultrasound-assisted extraction [2] and incorporated into phospholipid vesicles prepared through thin-film hydration followed by probe sonication [3]. A response surface methodology using central composite design was employed to optimize three formulation parameters (lecithin concentration, extract concentration, and sonication amplitude) seeking to minimize the vesicle size (VS) and polydispersity index (PDI), while maximizing the encapsulation efficiency (EE). The optimized system, containing 167.9 mg/mL lecithin, 42.5 mg/mL extract, and 28% sonication amplitude, produced nanosized liposomes (108.9 ± 1.4 nm) with narrow PDI (0.177 ± 0.014), a highly negative surface charge (-46.9 ± 1.1 mV), and an EE of $68.9 \pm 3.5\%$. HPLC-DAD-ESI-MS analysis confirmed the presence of chlorogenic acid, neochlorogenic acid, catechin, and epicatechin as major compounds, in concordance with a previous study of Macedo *et al.* [2] for the free extract. The formulation demonstrated stability for up to 90 days, with only minor increases in VS (108.9 to 132.6 nm) and minimal variation in PSI (0.177 to 0.196) and ZP (-46.9 to -48.8 mV), supporting a robust colloidal integrity. Regarding biological evaluations, all formulations demonstrated good biocompatibility in normal cell lines (HaCaT and HDFa), with cell viability values remaining within the non-cytotoxic range ($> 80\%$) (**Figure 1**). The free extract showed dose-dependent effect, especially in HaCaT cells, while unloaded liposomes reduced HDFa viability at higher doses, indicating some intrinsic liposome-related toxicity. Encapsulation of the extract mitigated these effects in healthy cells, suggesting improved safety and suitability for topical antitumor delivery. Ongoing investigations in A375 melanoma cells aim to further confirm the potential of the liposomal formulation as a promising vehicle for targeted antitumor therapy.

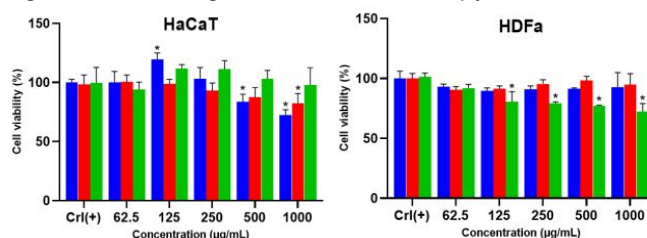


Figure 1. Effect of *A. arguta* extract (blue), loaded (red) and unloaded (green) liposomes on the viability of HaCaT and HDFa cell lines after 24h exposure by an MTT assay ($n = 3$), at different concentrations (62.5 – 1000 µg/mL). * $p < 0.05$

Acknowledgments

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Nanocarrier encapsulation of quaternary phosphonium salts

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Quaternary phosphonium salts (QPS) exhibit broad-spectrum and potent antibacterial and antifungal activity, making them promising candidates as antimicrobial agents. Their bioactivity is strongly influenced by the alkyl chain length, which can be optimized to enhance efficacy [1]. Compounds with intermediate aliphatic chain lengths (C14–C16) demonstrate particularly high antimicrobial activity against microorganisms such as *Staphylococcus aureus*, *Escherichia coli*, and *Candida albicans*. However, this increased potency is often accompanied by higher cytotoxicity in human embryonic kidney (HEK293) and hepatocellular carcinoma (HepG2) cell lines [2]. Encapsulating QPS within nanocarrier-based drug delivery systems offers a potential strategy to mitigate these drawbacks by reducing toxicity, improving stability, enabling targeted delivery, and allowing controlled release [3].

In this study, two delivery platforms—nanostructured lipid carriers (NLCs) and liposomes were developed for the encapsulation of QPS with varying alkyl chain lengths (C8, C10, C12, C14, and C16). Following successful synthesis, the QPS-loaded nanosystems were characterized for their physicochemical properties, morphology, and time-dependent stability using dynamic light scattering (DLS). Encapsulation efficiency was determined by high-performance liquid chromatography (HPLC). Finally, their cytotoxicity was evaluated in human hepatocellular carcinoma (HepG2) and normal human dermal fibroblast (NHDF) cell lines by measuring their metabolic activity and cell mass. For NLCs, both NLC-C14 and NLC-C16 produced monodisperse populations with sizes below 200 nm. A similar outcome was observed for liposomes encapsulating C-12, C-14, and C16-QPS. Beyond yielding nanoparticles of an appropriate size for tissue delivery, the nanostructures incorporating C-16-QPS also demonstrated enhanced cell viability in HepG2 and NHDF cells compared with QPs alone. In liposomes, this effect was particularly pronounced in NHDF cells. Overall, these results indicate that an intermediate chain length not only enhances antimicrobial activity but also improves the stability and biocompatibility of the nanostructures.

Agradecimentos

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Lipid-polymer hybrid nanocarriers for intranasal administration of entacapone

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Entacapone is a peripheral catechol-O-methyltransferase inhibitor (iCOMT) widely used for the treatment of late-stage Parkinson's Disease (PD). However, it presents several setbacks, such as small oral bioavailability (around 35%) and quick elimination after oral administration [1]. The emerging nanomedicine allows the development of sustainable nanocarriers with well-defined properties to overcome these problems, being Lipid-polymer hybrid nanoparticles reported as promising tools for increasing drugs' stability and permeability as well as reducing their cytotoxic effects [2]. With this in mind, the aim of this project was the development of new entacapone-loaded lipid-polymer hybrid nanocarrier based on poly(lactic-co-glycolic acid) (PLGA) and different natural oils, such as olive and coconut oils, functionalized with d- α -tocopheryl polyethylene glycol 1000 succinate.

These nanoformulations presented antioxidant properties against H₂O₂ and iron (III) insults, while preserving their physicochemical properties at serum-like solutions. These formulations did not present cytotoxic effects in hepatocarcinoma and dopaminergic neuroblastoma cells (HepG2 and SH-SY5Y, respectively), and improved entacapone's permeability in *in vitro* intranasal cellular (RPMI cells) monolayer. Moreover, in an *in vivo* *C. elegans* model of PD, the nanoformulations loaded with entacapone cause an improvement of mobility when compared with free entacapone.

Agradecimentos

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From sequence to gel: structure-property insights into isoleucine-containing dehydropeptides

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Peptide-based hydrogels have gained much attention as versatile materials. However, the molecular rules linking peptide sequence to macroscopic properties remain poorly understood.

Here, we present a new family of minimalist isoleucine-containing dehydropeptides designed to probe the balance between two important factors in self-assembly process: conformational rigidity and hydrophobic drive [1,2]. In fact, these molecules undergo spontaneous self-assembly into nanostructured networks that evolve into hydrogels under remarkably mild conditions. By combining supramolecular, mechanical, and biological characterisation, we have established clear relationships between subtle variations in sequence and material properties. This approach reveals how the strategic incorporation of dehydroamino acids and hydrophobic residues can dictate aggregation efficiency, pH responsiveness, viscoelasticity, and even biocompatibility. Besides introducing a new set of ultra-short hydrogels, our results highlight the power of the structure-property relationship as a design principle for peptide-based biomaterials.

These insights pave the way for engineering the next generation of adaptive, injectable and functional hydrogels for biomedical applications.

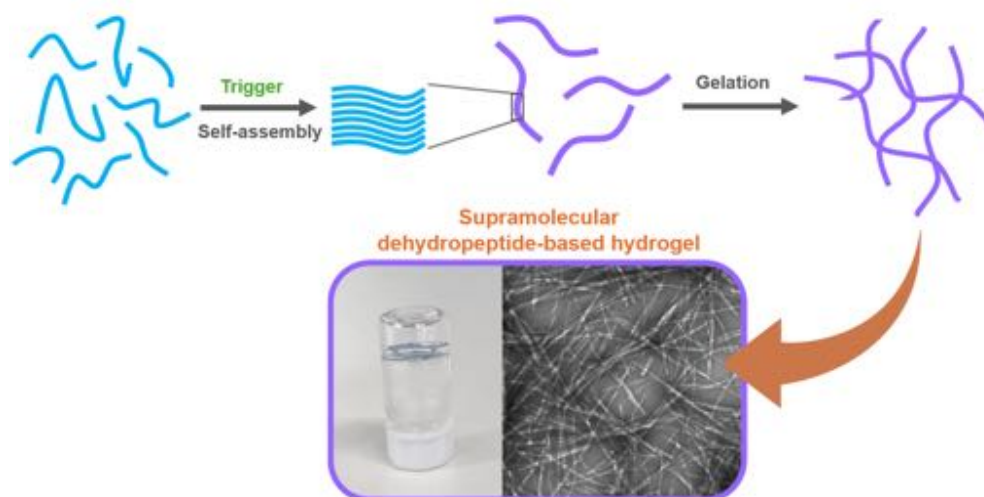


Fig.1. Generic supramolecular gelation mechanism and illustrative example (TEM micrograph) of the network formed by an isoleucine-containing dehydropeptide.

Acknowledgements

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Synthesis of carbon nanotubes from polyolefin waste: mass balance and gas phase composition

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The valorization of plastic solid waste (PSW) through thermochemical routes has emerged as a sustainable strategy to address the growing accumulation of these materials. Among the most attractive products, carbon nanotubes (CNTs) stand out due to their high added-value and potential applications in catalysis, sensors, and environmental remediation. CNT synthesis from plastics typically involves thermal decomposition of the polymer into a carbon-rich gas fraction, followed by its deposition on metallic nanoparticles to form CNTs. Optimizing this process requires a detailed understanding of both the mass balance and gas composition. In this work, CNTs were synthesized by chemical vapor deposition (CVD) from low-density polyethylene (LDPE), high-density polyethylene (HDPE), and polypropylene (PP) using a nickel-iron (NiFe) catalyst (synthesis details presented elsewhere [1]). The experiments were performed in a one-chamber reactor, with 1 g of plastic loaded in the upper zone (400–450 °C) and 0.2 g of NiFe in the lower zone (850 °C). The system was heated for 1 h and then held for 0.5 h under a N₂ flow (50 cm³ min⁻¹). Generated gases were analyzed *in situ* by gas chromatography with flame ionization detection (GC-FID). After reaction, solid and liquid fractions were collected and weighed. Characterization of similar CNTs can be consulted in previous publications [1–3]. The results are summarized in Fig. 1. As shown in Fig. 1a, the solid fraction yields were similar for all polymers (35–38%), while HDPE produced a higher liquid fraction than LDPE and PP. The gas fraction dominated in all cases, suggesting catalyst deactivation or an inadequate catalyst-to-plastic ratio. Three major gases were detected (Fig. 1b): methane >> propylene > ethylene. LDPE and PP showed comparable gas distributions, whereas HDPE generated markedly less methane and propylene, consistent with the mass balance trends.

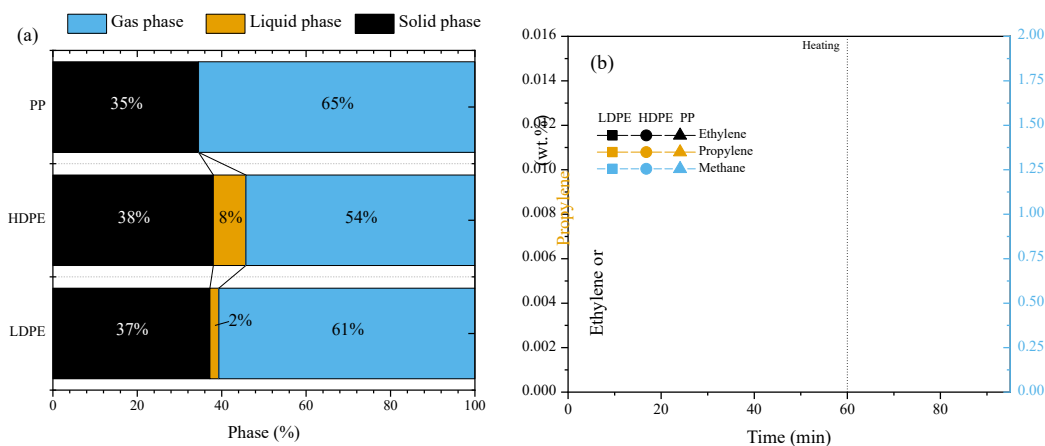


Fig.1. (a) Mass balance and (b) gas phase composition.

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Size-dependent eryptotic effects of citrate-coated silver nanoparticles on human erythrocytes and protection by quercetin

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Silver nanoparticles (AgNP) are widely incorporated into everyday products, resulting in inevitable human exposure and potential entry into the bloodstream. Once in circulation, AgNP can interact with blood cells, namely erythrocytes, raising important safety concerns [1]. Natural compounds with antioxidant activity have attracted considerable attention as potential protective agents. Among them, quercetin, a dietary flavonoid abundant in fruits and vegetables, has been extensively studied for its strong antioxidant properties [2]. In this context, this study aimed to investigate the size-dependent cytotoxic potential of citrate-coated AgNP of three different sizes (5, 10, and 50 nm) on human erythrocytes and evaluated the potential protective role of quercetin.

To this end, haemolysis, reactive species (RS) generation, total glutathione (GSH) levels, adenosine triphosphate (ATP) content, and intracellular calcium concentrations were assessed. The results showed that AgNP of all tested sizes induced haemolysis, markedly increased RS levels, and reduced ATP content. Pre-treatment with quercetin effectively prevented haemolysis and depletion of RS levels; however, it further exacerbated ATP depletion. Moreover, 10 and 50 nm AgNP caused significant GSH depletion, which was not prevented by quercetin. Interestingly, 5 and 10 nm AgNP induced an increase in intracellular calcium, an effect counteracted by quercetin pre-treatment.

In conclusion, citrate-coated AgNP trigger key hallmarks of eryptosis in human erythrocytes. Quercetin exerted a partial protective effect, mitigating some cytotoxic outcomes while aggravating others. These findings highlight the need for careful evaluation of natural compounds such as quercetin as protective agents against nanoparticle-induced toxicity and may contribute to the development of targeted strategies to mitigate the health risks associated with AgNP exposure.

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Formación *in situ* de nanopartículas de oro y plata en films líquidos para la detección colorimétrica de formaldehído

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Las estrategias integradas de enriquecimiento mediante microseparación y detección *in situ* han logrado mejorar significativamente la sensibilidad y selectividad de los métodos analíticos basados en nanopartículas (NPs) de metales nobles [1]. En estos sistemas, las soluciones coloidales actúan simultáneamente como fasesceptoras y elementos de reconocimiento óptico [2]. Sin embargo, los microsistemas más comúnmente empleados presentan geometría esférica, la cual lleva asociada una mínima relación superficie-volumen (S/V) que dificulta la transferencia de masa en la fase aceptora y provoca una disminución del rendimiento del método [3].

En este trabajo se propone el uso de films líquidos con NPs plasmónicas como alternativa para mejorar la transferencia de masa sin aumentar su complejidad. Para ello, se emplearon secciones circulares de soportes de acero inoxidable para formar films líquidos estables con mayor relación S/V. Como prueba de su aplicabilidad, se utilizaron films líquidos de NPs esféricas de oro con reactivo de Tollens para la detección colorimétrica de formaldehído basada en la formación *in situ* de nanopartículas bimetálicas de oro y plata con estructura núcleo-corteza [4].

La propuesta descrita en este trabajo permitió alcanzar factores de enriquecimiento entre 2,2 y 3,0 veces superiores a los obtenidos con técnicas convencionales de microextracción en fase líquida, alcanzando un límite de detección de 56,7 nM y una repetibilidad del 6,8 % (N=8). El método fue finalmente aplicado al análisis de muestras reales de agua y textiles, obteniendo resultados comparables a los obtenidos con el método de referencia.

Agradecimientos

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Non-lamellar lyotropic liquid crystalline lipid nanoparticles for brain drug delivery

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Glioblastoma Multiforme (GBM) is the most common malignant tumor found in the central nervous system [1]. Classified as a grade IV tumor by the World Health Organization, GBM is characterized by its high malignancy, rapid growth, and aggressive nature [2]. Treatment options are limited and the prognosis remains poor: patients usually have a median survival time of below 15 months and a 5-year survival rate of less than 5% [1,3,4]. Nanotechnology can arise as an alternative to conventional GBM treatments. Cubosomes, a new generation of lipid nanoparticles and a type of non-lamellar lyotropic liquid crystalline nanoparticles (LCNPs), have emerged as promising drug carriers due to their unique properties, such as biocompatibility, complex internal structure, high surface area, and stability [5,6].

This research aimed to produce biomimetic cubosomes for brain drug delivery with tumor homing abilities. To achieve that goal, cubosomes were prepared using the thin film hydration method, a top-down approach. Mitoxantrone (MITO) was selected as a model anticancer drug for proof-of-concept studies. Three types of formulations were produced: unloaded cubosomes, MITO-loaded cubosomes, and MITO-loaded biomimetic cubosomes. The produced nanoparticles were characterized in terms of size and polydispersity by dynamic light scattering (DLS) and nanoparticle tracking analysis (NTA). The surface charge (zeta potential) was evaluated by electrophoretic light scattering (ELS). The assessment of entrapment efficiency (EE%) and loading capacity (LC%) was performed using UV-Vis Spectrophotometry. Storage stability studies were performed by monitoring the size, polydispersity, zeta potential, EE% and LC% for 12 weeks (unloaded cubosomes and MITO-loaded cubosomes) or 3 weeks (MITO-loaded biomimetic cubosomes). The stability of the nanoparticles in protein-rich biological media, their drug release profile in a blood-mimicking fluid, and their hemolytic activity were also evaluated. Cell assays included the assessment of metabolic activity and cell uptake in both non-cancer and cancer cell lines upon incubation with the nanoparticles, and permeation across an *in vitro* Blood-Brain Barrier (BBB) model.

The sizes obtained were around 190 nm, which is adequate for brain delivery. All formulations showed polydispersity index (Pdl) values below 0.3, indicative of monodisperse solutions. These findings were corroborated by the NTA results. MITO EE% was higher than 70% for all cubosomes. The formulations remained stable upon storage at 4 °C for at least 3 weeks. Additionally, the stability was maintained when the cubosomes were incubated in protein-rich biological media. Cubosomes showed a sustained and controlled release of MITO when compared to a drug solution and did not exhibit hemolytic activity. Unloaded cubosomes demonstrated high cytocompatibility in L929 fibroblast and bEnd.3 brain epithelial cells. Moreover, MITO-loaded cubosomes exhibited dose-dependent cytotoxic activity in all concentrations tested in U251 glioblastoma cells, which was similar and in some of the concentrations tested, significantly higher than free MITO. The cell uptake assay demonstrated that biomimetic cubosomes exhibited higher cellular internalization in U251 glioblastoma cells than L929 fibroblast cells, in a manner dependent on both time and cellular content, demonstrating the tumor homing ability of the developed nanoplatform. Lastly, biomimetic cubosomes did not affect the permeation capacity across an *in vitro* BBB model. To summarize, these results demonstrate that the produced cubosomes are promising delivery vehicles for targeted GBM delivery.

Agradecimentos

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Peptide-functionalized SPIONs for dual-mode imaging and drug delivery in COPD

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Inflammatory airway and lung diseases, such as chronic obstructive pulmonary disease (COPD), result from oxidant/antioxidant imbalance and are characterized by chronic inflammation of the airways [1]. The prevalence and rapidly growing burden of such diseases is a major health challenge. In COPD, the expression of several matrix metalloproteinases is elevated, especially MMP-9 which acts as downstream effector and upstream mediator of growth and inflammation pathways, thereby becoming a promising therapeutic target and prognostic biomarker [2,3].

Here, we report an innovative nanoengineered delivery system based on superparamagnetic iron oxide nanoparticles (SPIONs) coated with a polymer (Fig.1A). These nanoparticles were further functionalized with an MMP-9-specific FRET-labelled peptide (Fig.1B) and loaded with an anti-inflammatory drug (Fig.1C). The delivery system, based on magnetic nanoparticles, allows coupling of magnetic resonance imaging (MRI) and fluorescence imaging (FI), providing dual-mode non-invasive imaging of MMP-9 activity, as well as the use of magnetic hyperthermia for release of the anchored drug.

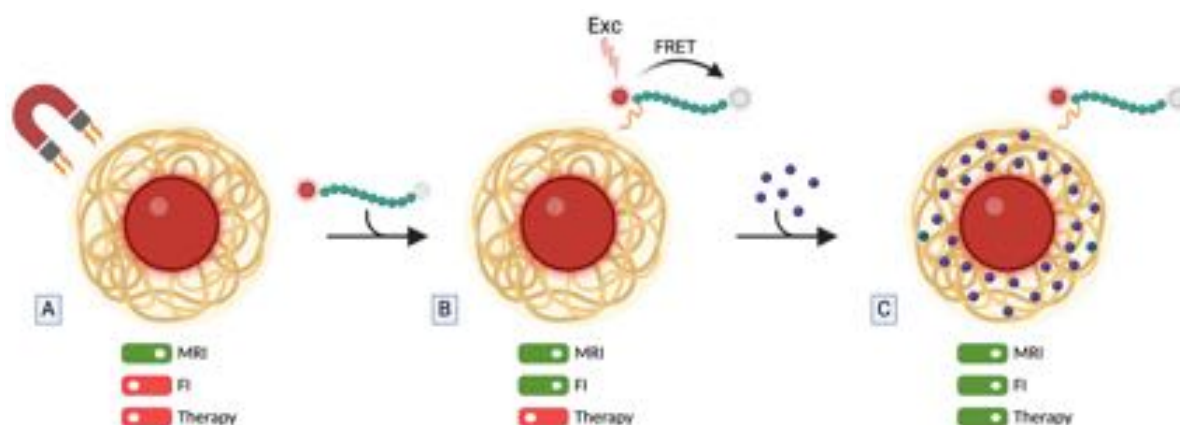


Fig.1. Schematic representation of the peptide-functionalized SPIONs platform.

Acknowledgments

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Miniaturized assay for monitoring sulfite preservatives in food samples

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Sulfites are a group of chemical compounds that can emit sulfur dioxide (SO_2), which is the active element that aids in the preservation of food and are amongst the many additives utilized in the food and beverage industry due to their antimicrobial, antioxidant and preservative properties [1]. Sulfites, even though they are recognized as safe, can cause skin rashes and irritations in susceptible individuals. Sensitive individuals may also experience anaphylactic episodes in severe circumstances. Given the adverse effects that sulfites can cause on sensitive individuals, it is extremely important the use of sensitive and trustworthy methods for measuring sulfites in food products [1-2]. Currently, sulfite monitoring methods are still complex and labor-intensive (e.g. Monier-Williams method), which makes them costly, with lengthy analysis time and a high level of control required in some steps of the analytical procedure [3].

Therefore, this work aimed to develop a simple, fast and low-cost method to determine sulfite levels in food samples, combining gas-diffusion extraction with optical detection under microplate format. Gas Diffusion Microextraction (GDME), which is based on the extraction of analytes from the sample solution (donor) via a gas-permeable membrane into a common liquid receiver solution (acceptor), made it possible to extract from the target matrix [4]. To this end, a GDME device was designed and manufactured by SLA 3D printing (Figure 1). The device was then mounted onto a 6-well microplate that was inserted into a microplate reader at 37 °C for the extraction (up to 15 min). Our preliminary results showed a linear range from 0 to 45 mg/L of sulfite concentration using a solution of Malachite Green as the acceptor solution. Other pH sensitive optical probes solutions, prepared from fluoresceine and a quinoline-based hydrazone have also been tested as acceptor phase. A critical comparison between the different detection strategies will be presented.

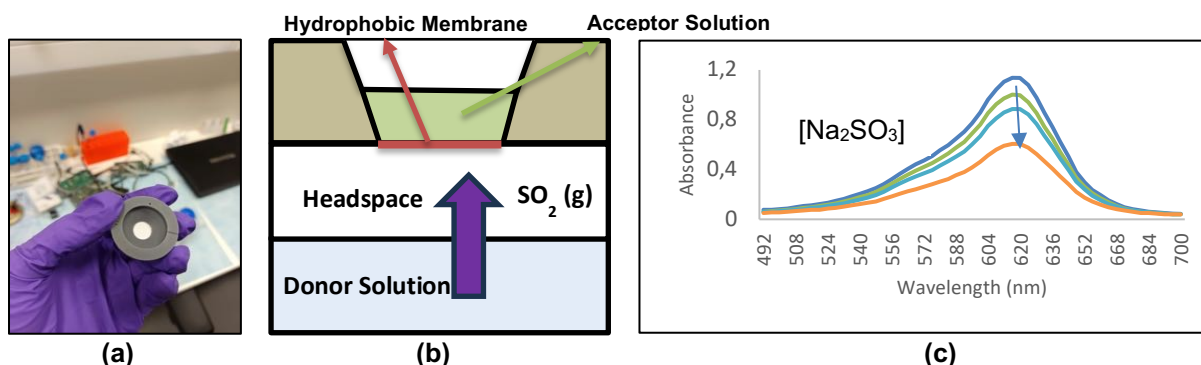


Fig. 1. (a) GDME device. (b) Schematic illustration of the GDME procedure in a single microplate well (c) Absorbance spectra of Malachite Green 20 mg L⁻¹ with increasing amounts of Na₂SO₃ (0, 15, 30, 45 mg L⁻¹).

Acknowledgements

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Point-of-care detection of soybean Gly m TI in complex foods: Immunosensor vs MIP biosensor

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The rising incidence of food allergies highlights the need for advanced, cost-effective allergen detection methods, with biosensors offering a promising solution due to their simplicity, sensitivity, and automation potential. This study reports an electrochemical immunosensor for the quantitative detection of the soybean allergen Gly m TI in complex food matrices, including sausages, hams, and biscuits, contributing to advancing biosensor-based methods for reliable allergen detection. The immunosensor exhibited high specificity, minimal cross-reactivity, a linear response ranging from 1 ag mL⁻¹ to 0.1 mg mL⁻¹, and a limit of detection (LOD) below 1.0 ag mL⁻¹, successfully quantifying 0.1 mg kg⁻¹ of soybean protein in all tested matrices, thereby protecting most of the soybean-allergic individuals [1]. Although antibody-based immunosensors provide exceptional selectivity, their practical implementation can be hindered by issues related to cost, storage stability, and reproducibility. Molecularly imprinted polymers (MIP) technology was incorporated to offer a synthetic recognition approach capable of replicating the binding characteristics of antibodies with enhanced chemical robustness [2].

Accordingly, this work explores MIP-based detection strategies as a sustainable and efficient alternative to traditional immunosensors for soybean Gly m TI quantification. The Gly m TI-MIP biosensor demonstrated high specificity, a broad linear range, from 1 ag mL⁻¹ to 10 µg mL⁻¹, and a LOD below 1 ag mL⁻¹, detecting 0.1 mg kg⁻¹ of soybean Gly m TI in biscuits, hams, and sausages. This innovative platform enhances allergen traceability in foods, supporting rigorous safety control, compliance with international standards, and increased consumer confidence. Both biosensing systems proved suitable for detecting trace amounts of soybean in processed and complex foods, protecting 95-99% of food-allergic individuals [2]. Both the electrochemical immunosensor and the MIP biosensor effectively detected Gly m TI in complex food matrices, confirming their applicability for real-world food analysis, thus helping to protect at least 99% (eliciting dose of 1%) of soybean allergic patients.

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Determinación de THC mediante microextracción líquido-líquido dispersiva asistida por aire (AALLME)

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El pelo es una muestra alternativa en toxicología cuya principal ventaja es su amplia ventana de detección, por lo que es útil para la realización de análisis retrospectivos. El desarrollo de la química analítica verde ha fomentado el uso de técnicas de microextracción como alternativa a las extracciones convencionales. En este campo, el pretratamiento de muestras de pelo también ha experimentado un desarrollo exponencial en los últimos años, pero aún sigue siendo escaso [1].

En este contexto, se desarrolló un método para la determinación de THC en pelo. El cannabis continúa siendo la droga de abuso ilegal con mayor incidencia en el mundo y por ello, ha sido objeto de este trabajo con la intención de estudiar la aplicabilidad de novedosas técnicas de microextracción. En este caso se estudió la aplicación de una variante de la microextracción líquido-líquido dispersiva (DLLME), que evita el uso de disolventes orgánicos clorados, la microextracción líquido-líquido asistida por aire (AALLME) [2,3].

La determinación del tetrahidrocannabinol (THC) se realizó mediante GC-MS y la validación del método se llevó a cabo siguiendo las directrices de validación de la FDA. Los resultados obtenidos demostraron una linealidad en el intervalo de 0,01 a 4 ng/mg, con un límite de detección (LOD) de 0,008 ng/mg. Además, se obtuvieron resultados satisfactorios en el resto de los parámetros estudiados: selectividad, exactitud, precisión y recuperación. Por último, el método se aplicó a 20 casos reales demostrando su aplicabilidad.

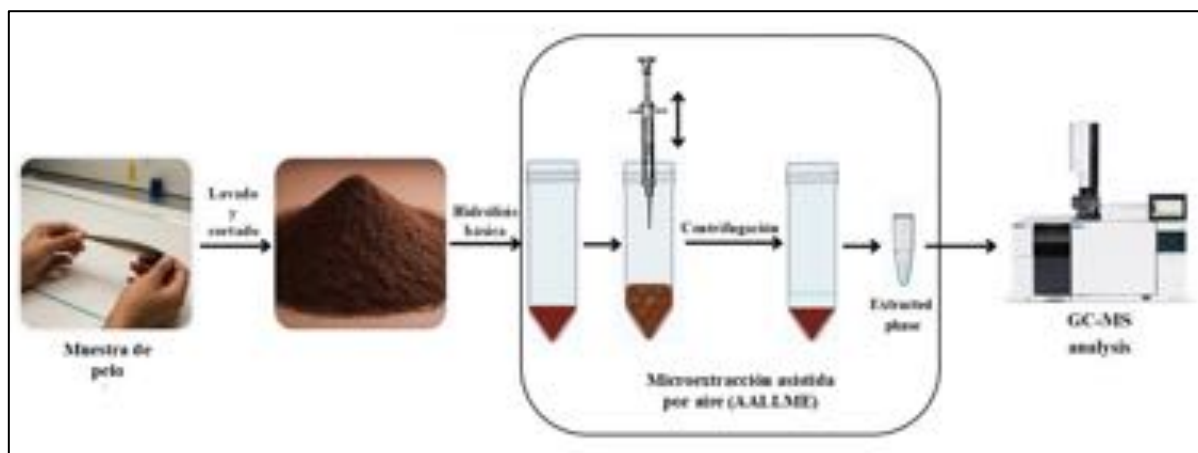


Fig.1. Esquema de la metodología analítica desarrollada.

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Optical chemosensor based assay for measuring tributyltin in environmental samples

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Biofouling occurs when algae and marine microorganisms accumulate on ships, boats, and coastal structures. Tributyltin (TBT) is a well-known, highly effective antifouling component used in most anti-fouling paints for boats since the 1980s. TBT was discovered to be severely toxic to the aquatic environment, and its use was banned in 2008 under the Rotterdam Convention. Even with the ban in place, TBT still affects aquatic environments worldwide [1]. Currently, TBT monitoring methods are essentially based on the use of either gas or liquid chromatography coupled to mass spectrometry [2]. These methods are costly, time-consuming, and labor-intensive, which creates a quest for simple and streamlined analytical methods.

Therefore, this work aimed to develop a simple and low-cost method to screen TBT levels in water samples using fluorescent probes [1]. Previous research by the authors synthesized hydrazone derivatives that worked as optical chemosensors when in contact with TBT [1], [3]. The analytical performance of the different hydrazone derivatives was investigated, and the most promising one was selected. Therefore, 2-((2-(pyridin-2-yl)hydrazineylidene)methyl)quinoline (hydrazone **1**) was able to directly measure TBT in the concentration range 10 – 150 mg L⁻¹ in acetonitrile (Fig. 1).

To detect low concentrations of TBT, a pre-concentration method based on solid-phase extraction is also under development. The preliminary results (based on LC-MS detection) showed that weak-cation exchange (WCX) sorbent could retain TBT from water samples, with recoveries between 61 – 34%. Future work will be focused on the improvement of the extraction procedure, as well as in the detection of TBT from the eluate with the selected fluorescence probe.

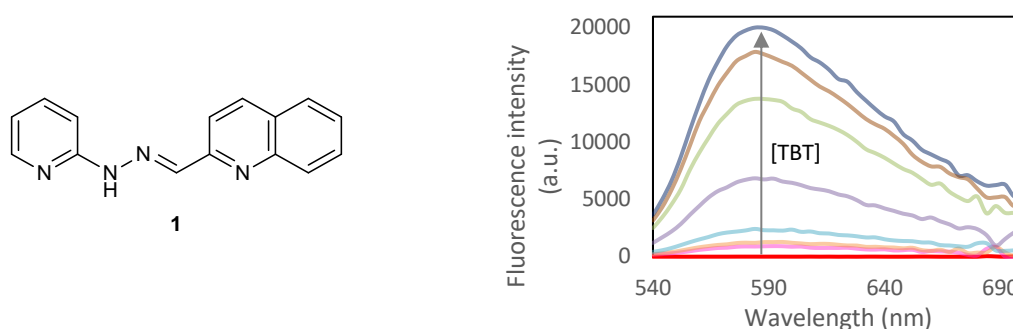


Fig. 1. (left) Chemical structure of hydrazone **1**. (right) Emission spectra of hydrazone **1** 100 mg L⁻¹ in ACN (red) with increasing amounts of TBT (0, 10, 15, 25, 50, 100, 150 mg L⁻¹) in ACN.

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Innovative sampling strategies for trace metals analysis in water monitoring

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The presence of metal ions in aquatic ecosystems has become a major environmental and public health concern due to their toxicity, persistence, and bioaccumulative properties. Metal ions, such as lead, zinc, mercury, cadmium, copper, chromium, and arsenic, pose significant risks even at trace levels, potentially leading to severe health issues, which highlights the need for regular monitoring of water bodies.

Conventional water sampling methods for metal ions analysis typically involve bottle sampling, the addition of preservatives, and, afterwards, at the laboratory, the samples usually undergo a treatment procedure prior to the determination. At this point, procedures like liquid-liquid extraction or solid phase extraction are applied to the samples in order to remove interferences and/or enrich the analyte before the analyte quantification, usually through atomic absorption spectrometry (AAS) or inductively coupled plasma optical emission spectrometry (ICP-OES). These steps, from sampling to quantification, present several concerns/challenges that may affect sample integrity and, therefore, the accuracy of the determination. Alteration of the sample can occur due to temperature variation, UV irradiation, microbial activity, oxygen exposure, changes in pH, and the use of organic solvents that might alter chemical equilibrium and the sample composition.

To overcome these limitations and, within the scope of the project Aqua_Smart, novel water sampling strategies based on solid phase extraction were designed. This approach could be an advantageous alternative to the conventional sampling, transport and storage of a high volume of water samples. In this context, the objective was to develop miniaturized extraction procedures metal ions sampling at the water source for subsequent transport to the laboratory for quantification. An additional goal is to integrate sampling and sample pre-treatment in a single in-situ step and, this way, simultaneously enable the cleanup of the sample matrix and analyte enrichment. These innovative strategies not only enhance analytical performance but also support environmental sustainability, making it a valuable tool for water quality monitoring and metal pollution management. Furthermore, the ability to reuse the solid phase extraction sorbent material further improves the method's cost-effectiveness and sustainability.

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DNA-based electrochemical biosensors for honey authentication

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The increasing number of reported fraudulent natural food products detected in the international market has led to awareness on the issues of food fraud and authenticity. These fraudulent actions not only threaten consumer health and trust but also damage the reputation and integrity of the food industry [1]. Among the natural products, honey is particularly vulnerable to adulteration due to its high market value and complex botanical composition [2].

In this study, two disposable electrochemical DNA-based biosensors were developed and optimized to detect the presence of the residual botanical DNA from *Erica arborea* (heather flower) and *Castanea sativa* (chestnut flower) in commercial honey samples. The methodology behind these devices stems from the enzymatic hybridization reaction of a heather- and chestnut-specific DNA-target probe to their complementary sequences on screen-printed gold electrodes. To increase both sensors' selectivity, their complementary probes were split into two shorter sequences: a fluorescein isothiocyanate-labelled DNA-signaling probe and a thiol-DNA-capture probe. Additionally, a DNA-capture and mercaptohexanol mixed self-assembled monolayer was utilized to improve hybridization efficiency. All surface alterations were characterized by cyclic voltammetry and electrochemical impedance spectroscopy.

The enzymatic amplification of the electrochemical signal, obtained using chronoamperometry, revealed a linear response within a 0.03 to 2.00 nM and 0.03 to 1.00 nM concentration range for *E. arborea* and *C. sativa*, respectively. These biosensors were then successfully applied for the detection of the residual botanical DNA of both species in locally commercialized honeys, confirming their botanical composition. These results were validated by conventional PCR and real-time quantitative PCR, with the genosensor demonstrating a superior sensitivity over the traditional methods. Therefore, DNA-based biosensors are a promising sensitive and cost-effective analytical approach for honey authentication, contributing to the prevention of food fraud and the enhancement of food safety and quality for the honey industry.

Acknowledgements

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Sequential injection lab-on-valve with bead injection for iron speciation in waters

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The current increasing focus on analytical analysis, is resulting in the development of innovative techniques to handle sample preparation [1]. These efforts aim to minimise sample manipulation, while developing innovative methodologies, and reducing the environmental impact associated with various analytical techniques. At the same time, green analytical chemistry is an emerging concept in the last years. The main goal consists in the development of sustainable analytical techniques while still maintaining high analytical performance. Efforts are being made to reduce the environmental impact caused by toxic reagents and the large waste that can be created in each technique, on top of reducing the handling of samples. By setting these goals, green analytical chemistry intends to develop safer procedures, for the environment and the analysts, but still maintaining accurate results.

As a consequence, flow-based methods became an important tool to achieve the current goals in analytical chemistry. Application of flow-based analytical systems offers significant benefits, given their miniaturised characteristics, allowing the reduction of sample volume and reagents required, thereby lowering waste generation and analysis costs. Moreover, these systems are automated, contributing to an increase in analytical performance. Monitoring iron concentrations in natural waters is essential due to its influence on aesthetic qualities, such as water colouration, and the potential impact on structures situated within these aquatic environments. Iron levels vary considerably among different water bodies, with concentrations ranging from millimolar values in estuarine regions to nanomolar levels in open ocean areas. For trace-level measurement of metal ions in natural waters, atomic absorption or emission spectroscopic techniques are frequently employed. However, these techniques constitute relatively costly approaches and limit the feasibility of *in-situ* analysis, which does not contribute to green analytical goals.

In this work, a sequential injection lab-on-valve (SI-LOV) system coupled with a bead injection approach was developed for iron determination in water samples. NTA beads were kept in contact with the water sample for a predetermined period to preconcentrate iron at the sampling step. Afterwards, the beads suspension was transported to the laboratory and introduced into the SI-LOV system. Within this system, the iron retained on the beads reacted with the ligand MRB13, alongside a buffer solution, resulting in the formation of a coloured complex. This methodology enables accurate quantification of iron while employing minimal sample volumes, reducing reagent consumption, and environmentally friendly monitoring of water quality.

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Enantioseparation of methylone by chiral liquid chromatography and metabolite synthesis for further metabolomic studies

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Synthetic cathinones, such as methylone, are new psychoactive substances with psychostimulant effects which represent a major public health challenge due to their easy accessibility and limited toxicological data [1]. It is of pivotal relevance to understand their toxicological effects. In this context, the emerging field of metabolomics holds great promise as a tool to elucidate mechanisms of action and identify biomarkers of exposure [2].

Additionally, synthetic cathinones are chiral compounds, and in previous studies we demonstrated that their enantiomers exhibit distinct toxicological properties [3]. Although metabolomics studies have been conducted for some synthetic cathinones, including methylone [4], no investigations have yet addressed their enantioselectivity effects using this approach. To explore their enantioselectivity in further metabolomic studies, it is necessary to obtain the individual enantiomers which can be achieved by enantioseparation using chiral liquid chromatography (cLC) [5].

Furthermore, it is essential to consider not only the parent compounds but also their metabolites, since they may contribute to the overall biological impact or toxicity. Although the metabolism of synthetic cathinones is well studied [6], the specific effects of their metabolites remain largely underexplored. The synthesis of these metabolites is a crucial step to evaluating their effects.

In this work, the semi-preparative enantioseparation of methylone and evaluation of enantiomeric purity was successfully performed by cLC using amylose-based columns. Enantiomers with high enantiomeric ratios and good recovery rates were obtained. Additionally, several synthetic methodologies were employed to obtain the major metabolites of methylone. Intermediates and metabolites were obtained with high yields and purity, being the structure elucidation carried out using spectroscopic techniques, including IR, and ¹H and ¹³C-NMR.

The isolated enantiomers and synthesized metabolites will serve, in future studies, to understand the enantioselective effects and the role of metabolites in the metabolomic profile of synthetic cathinones.

Acknowledgements

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Uso da silição na caracterização de used cooking oil por cromatografia gasosa mono e bidimensional

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Amostras de resíduos derivados de biomassa são extremamente complexas. A cromatografia gasosa mono e bidimensional (GCxGC) são uma estratégias que permitem resolver este desafio. A presença de grupos funcionais, como -COOH e -OH, diminuem a volatilidade, estabilidade térmica, assim como interações com a fase estacionária, implicando numa baixa resposta e redução da vida útil da coluna cromatográfica. Este trabalho teve como objetivo usar o processo de derivatização por silição e otimizar métodos cromatográficos em 1D e 2D para a caracterização (qualitativas e quantitativas) do UCO, do inglês, *Used Cooking Oil*. As amostras foram analisadas, num cromatógrafo LECO Pegasus BT 4D GC x GC quadjet, com uma coluna Rxi-5ms (30m, 0,25 mm de diâmetro interno e 0,25 µm de espessura de filme) no forno primário (1ª dimensão), no forno secundário uma coluna Rxi-17 Sil MS (1,6 m, 0,25 mm de diâmetro interno e 0,25 µm de espessura de filme). A derivatização foi conduzida por silição com MSTFA (N-methyl-N-(trimethylsilyl)trifluoroacetamide) (30 minutos a 80°C, num banho de areia)¹. As amostra foram analisads também por GPC e FTIR. A aplicação do método de silição melhorou a resolução e fatores de resposta, sendo os metodo cromatográfico quantitativo considerado exato (erro < 10%) e preciso (RSD <10%) com resposta linear (r>0.99). As análises por GC x GC permitiram identificar e quantificar em torno de 500 compostos, classificados como, mono e diacilglicerois, ácidos, diácidos, ésteres, aldeídos e hidrocarbonetos, entre outros. Os compostos maioritários foram ácidos Oleico, linoleico e palmítico. A identificação segura dos compostos presentes nestes materiais é de fundamental importância para compreensão das possíveis rotas de transformação destas matérias-primas complexas.

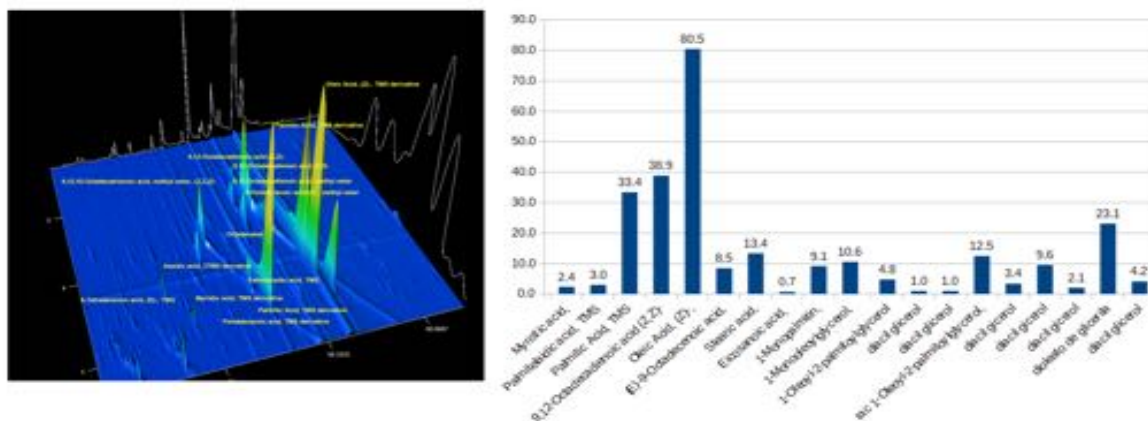


Fig.1. Cromatogramas GCxGC-TOFMS (a). Distribuição g.kg⁻¹ dos compostos no UCO

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A public health tool for monitoring aluminium levels in environmental and biological samples

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Aluminium is an element with potential toxic effects, particularly when exposure exceeds safe limits. Drinking water is a key matrix for assessing environmental contamination and human exposure, since long-term intake of high aluminium concentrations has been associated with several health issues, including Alzheimer's disease. Additionally, monitoring aluminium in urine provides an important measure of cumulative exposure.

In this work, we report the development of a microfluidic paper-based analytical device (μ PAD) for the determination of aluminium in both water and urine samples. The device relies on colorimetric detection using Chrome Azurol S (CAS), which forms a blue complex with aluminium ions. The intensity of the blue colour increases proportionally with the aluminium concentration, as illustrated in Fig. 1. Several physical and chemical parameters were optimized to achieve the best analytical performance, including the type and porosity of the filter paper, reagent volumes and concentrations, and sample volume. The final design consists of two paper layers: the top layer contains the CAS solution and acetate buffer solution, while the bottom layer is used as a reservoir to allow a higher sample volume. Aluminium determination in drinking water was successfully achieved, and the μ PAD was further adapted for urine analysis, with ongoing studies addressing potential interferences.

This paper-based platform provides a simple, low-cost, and rapid analytical tool that does not require sophisticated instrumentation and shows strong potential for environmental monitoring and human biomonitoring.

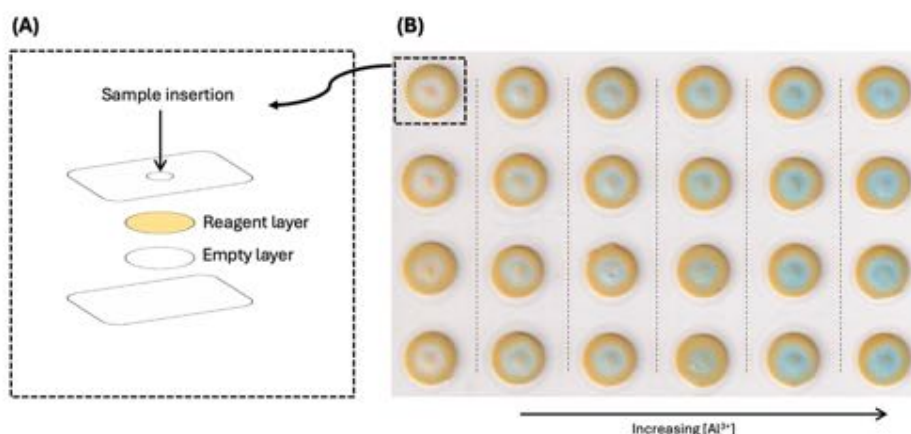


Fig.1. Schematic representation (A) and a real image (B) of the developed μ PAD for aluminium quantification.

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Determination of Cu(II) in mozambican artisanal spirits by square-wave Anodic stripping voltammetry

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The production and commercialization of artisanal spirits (*Nipa*) in various regions of Mozambique constitute an important source of income for certain households. Nevertheless, the lack of national regulations establishing production standards and quality control for these beverages [1], combined with public health concerns related to their consumption, raises questions regarding potential chemical contamination.

In this context, the present study aimed to determine the concentration of Cu(II) in twelve *Nipa* samples produced in three Mozambican provinces: Sofala, Manica, and Gaza, using square-wave anodic stripping voltammetry (SWASV). The analytical method was optimized with respect to key parameters, including deposition potential and deposition time, stripping technique, and other experimental conditions, to improve sensitivity and reproducibility. The optimized method exhibited good linearity within the concentration range of 0.025–1.2 ppm Cu(II), with a slight deviation from linearity above 1.5 ppm (Figure 1), and yielded a correlation coefficient of 0.997, thus demonstrating its reliability. The limits of detection and quantification were 0.12 ppm and 0.38 ppm, respectively, values considered adequate when compared with the maximum allowable limit established by Mozambican legislation for drinking water (1 ppm) [2], which was adopted as a reference in this study. Despite the complex nature of the matrix, the voltammetric method enabled the quantification of Cu(II) in three *Nipa* samples, two of which presented concentrations exceeding 1 ppm, thereby indicating potential health risks for consumers.

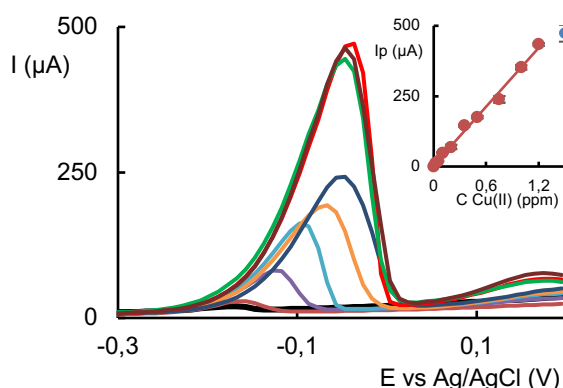


Fig.1. Square-wave anodic stripping voltammograms of Cu(II) in 0.1 M HCl, with the calibration curve shown as an inset, covering the concentration range 0.025–1.5 ppm. Experimental conditions: $t_d = 90$ s; $\Delta E_s = 10$ mV; $f = 80$ Hz; $\Delta E_p = 50$ mV. — 0.025 ppm — 0.05 ppm — 0.1 ppm — 0.2 ppm — 0.35 ppm — 0.5 ppm — 0.75 ppm — 1.0 ppm — 1.2 ppm — 1.5 ppm.

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Point-of-care determination of free chorine in swimming pools waters as a public health issue

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Sodium hypochlorite (free chorine) is widely recognized as one of the most effective and user-friendly disinfectants used in water treatment. However, at high concentrations it has been reported to react with organic compounds, thus producing toxic by-products [1] that can be hazardous. To safeguard public health, international health organizations such as the world health organization have set the limit for allowable concentration (5 mg/L) in drinking water. In this context, many colorimetric methods are used to monitor hypochlorite content in water samples, but most of these methods employ toxic reagents [2].

Microfluidic paper-based analytical devices (μ PAD's) have recently emerged as a fast, in-situ, alternative to the conventional methods and focus on minimizing waste production and reagent consumption in a "green chemistry" approach. Furthermore, as the μ PAD's methodologies follow the ASSURED criteria: Affordable, Sensitive, Specific, User-friendly, Rapid and Robust, Equipment-free and Deliverable to end-users [3].

In this work, a μ PAD device was developed to assess free chlorine concentration in swimming pool waters. The developed μ PAD relies on the oxidation of *o*-dianisidine by hypochlorite, in acidic medium, forming a colored product. The color intensity of the formed product is measured by scanning the μ PAD and a calibration curve is established between the hypochlorite concentration and the calculated absorbance from the measured color intensities. The scanning is made 6 min after introducing standards/samples in the μ PAD and the measurements correspond to an average of at least #3 readings. After optimization, the method enabled a quantification range from 2-10 mg/L, which is suitable for testing swimming pool waters. The proposed μ PAD offers a simple and fast alternative to conventional methods, avoid the use of hazardous reagents, and showing potential to become an everyday tool.

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Mechanical vs. manual harvesting: effects on phenolics and antioxidant potential in Fernão Pires, Loureiro, and Alvarinho wines

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The impact of harvesting method (manual vs. mechanical) on the phenolic composition and antioxidant capacity of white wines from three Portuguese grape varieties (Fernão Pires, Loureiro, and Alvarinho) from the Vinho Verde region, produced at Casa de Villa Maria in Penafiel, was investigated. Phenolic characterization was performed using colorimetric assays to assess ortho-diphenols, total phenolics, and flavonoids. Antioxidant activity was evaluated using ABTS, DPPH, and FRAP assays.

Across all varieties, mechanical harvesting consistently led to significantly higher concentrations of phenolic compounds. In Fernão Pires, mechanical harvest wines exhibited elevated levels of *ortho*-diphenols (163.6 vs. 113.6 mg GAE/L), total phenolics (294.1 vs. 90.8 mg GAE/L), and flavonoids (26.1 vs. 13.8 mg CE/L) compared to manual harvest. Loureiro followed a similar trend, with higher contents of *ortho*-diphenols (295.4 vs. 196.1 mg GAE/L), total phenolics (95.1 vs. 80.7 mg GAE/L), and flavonoids (36.0 vs. 22.9 mg CE/L) under mechanical harvesting. Alvarinho showed the most pronounced differences, with mechanical harvesting yielding the highest levels of *ortho*-diphenols (315.8 vs. 176.4 mg GAE/L), total phenolics (101.9 vs. 75.8 mg GAE/L), and flavonoids (41.6 vs. 27.2 mg CE/L).

Antioxidant capacity results mirrored these findings. Mechanical harvest wines displayed superior ABTS (up to 1.041 mmol TE/L in Alvarinho vs. 0.468 in manual), DPPH (0.992 vs. 0.536 mmol TE/L), and FRAP (1.024 vs. 0.619 mmol TE/L) values, particularly in Loureiro and Alvarinho. In contrast, Fernão Pires showed less marked differences, except in the ABTS assay.

Overall, the results highlight mechanical harvesting as a promising enological strategy to enhance phenolic extraction and antioxidant potential in white wines, particularly in Loureiro and Alvarinho. These findings also raise questions about potential sensory implications, such as changes in astringency, color, and oxidative stability, warranting further research.

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Estrategias analíticas para el control de contaminantes en aguas mediante enfoques miniaturizados y de bajo coste

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La disponibilidad de recursos hídricos limpios juega un papel crucial no solo para la salud pública, sino también para el crecimiento económico y la sostenibilidad del medioambiente. Se establece que solamente alrededor del 0,01 % del agua terrestre es potable, cantidad que se reduce año tras año por causa de la contaminación antropogénica. La creciente preocupación por la contaminación de los recursos hídricos está generando una demanda urgente de propuestas analíticas rápidas, eficientes y accesibles que permitan el control continuo de contaminantes medioambientales. Tradicionalmente, la determinación de contaminantes en muestras medioambientales se realiza empleando técnicas analíticas centralizadas que, aunque ofrecen alta sensibilidad en general implican altos costes operativos y de mantenimiento, así como una generación de residuos considerable. En este contexto, el desarrollo de metodologías analíticas miniaturizadas y que puedan adaptarse a la portabilidad es de gran interés para reducir el consumo de reactivos, minimizar la generación residuos y mejorar la eficiencia energética [1,2].

La combinación de nanomateriales y/o dispositivos analíticos basados en papel (*paper-based analytical devices*, PADs) junto con estrategias de microextracción ofrecen una potente herramienta para realizar el enriquecimiento y/o detección de los contaminantes objetivo. En particular, cabe destacar el desarrollo de una red orgánica covalente (*covalent organic framework*, COF) que demostró ser eficaz para la preconcentración selectiva de tonalide y galaxolide, fragancias sintéticas clasificadas como contaminantes emergentes, en agua de mar [3]. Bajo condiciones óptimas, se lograron eficiencias de adsorción superiores al 90% para ambas fragancias, lo que permitió su detección fluorimétrica a niveles de concentración de µg/L. Otro enfoque interesante es la integración de la derivatización, separación/preconcentración y cuantificación en una sola etapa reduciendo el tiempo de análisis. En este sentido, recientemente se desarrolló una nueva plataforma/sensor de preconcentración utilizando un sustrato de celulosa funcionalizado con nanoclústers de plata (Ag NCs) para la detección rápida y accesible de N-nitrosaminas en agua [4]. La microextracción y la detección, asociada a la desactivación de luminiscencia de los Ag NCs, pueden realizarse en el mismo dispositivo en menos de 4 minutos.

Asimismo, los estudios actualmente en curso dentro de la iniciativa *AQUASENSE* buscan maximizar la eficiencia analítica mediante la miniaturización de la etapa de preparación de muestra empleando PADs e integración con técnicas de bajo consumo energético y menor generación de residuos, con el fin de ofrecer alternativas sostenibles, de menor coste y menos dependientes de infraestructuras analíticas centralizadas, para el análisis de contaminantes de interés en muestras de aguas naturales.

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Dispositivo microfluídico tridimensional para la determinación colorimétrica multianalito de analgésicos en muestras clínicas

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La fácil accesibilidad a analgésicos de venta libre como el paracetamol (PRL) y la aspirina (AAS) ha generado una creciente preocupación debido a los posibles riesgos para la salud asociados a su uso indebido o a casos de autointoxicación deliberada. La demanda creciente de herramientas analíticas rápidas y fiables pone de relieve la importancia de las estrategias de diagnóstico en el lugar de atención (Point-of-Care Testing, PoCT) en entornos clínicos y de emergencia [1]. En este contexto, los dispositivos analíticos microfluídicos basados en papel (μ PADs) han surgido como sistemas muy prometedores gracias a su simplicidad, bajo coste y capacidad para realizar análisis multiplexados con un requerimiento mínimo de muestra [2].

En este trabajo, se presenta un novedoso dispositivo analítico microfluídico tridimensional basado en papel (3D μ PAD) diseñado para la detección colorimétrica simultánea de PRL y AAS en muestras sintéticas de saliva y orina [3]. El dispositivo fue fabricado utilizando sustratos de celulosa hidrofílica, lo que permite un transporte eficiente de fluidos y la integración de múltiples zonas de reacción en una plataforma compacta. Se optimizaron los parámetros experimentales involucrados en la etapa de fabricación y detección, obteniéndose límites de detección de 0,004 mM para PRL y 0,013 mM para AAS. Los límites de cuantificación obtenidos fueron de 0,013 mM y 0,045 mM, respectivamente. El método demostró una excelente repetibilidad y reproducibilidad, con desviaciones estándar relativas inferiores al 6% en varios niveles de concentración. Los estudios de recuperación en matrices clínicas sintéticas arrojaron valores entre 98–103%, confirmando la robustez del enfoque.

Es especialmente relevante que la integración de la adquisición de imágenes y procesamiento de datos empleando el espacio de color RGB mediante *smartphone* permite la cuantificación rápida de ambos fármacos en menos de 4 minutos, subrayando el potencial del dispositivo para aplicaciones PoCT portátiles y no invasivas [4]. Este innovador 3D μ PAD representa un avance significativo en el monitoreo dual de fármacos, ofreciendo una herramienta fiable, económica y fácil de usar para el cribado en tiempo real de PRL y AAS en el diagnóstico clínico y la vigilancia en salud pública.

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Análise isotópica de Sr mediante ICP-TOF-MS para a verificación da orixe de produtos agroalimentarios

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A composición isotópica do estroncio (Sr) proporciona información asociada á procedencia xeográfica, xa que o Sr encóntrase biodispoñible na terra e é asimilado por organismos e plantas, polo que o estudo das variacións naturais da relación isotópica $^{87}\text{Sr}/^{86}\text{Sr}$ é unha ferramenta útil para establecer ou verificar a orixe de produtos agroalimentarios. [1] Para isto, requírese a medida da relación isotópica do Sr con elevada precisión, é por iso que a espectrometría de masas con fonte de plasma acoplado indutivamente de multicolelector (MC-ICP-MS) é a técnica por excelencia. Sen embargo, é unha técnica custosa e o tratamento da mostra precedente as medidas é laborioso. [2] É por iso, que se opta por avaliar o uso de técnicas máis accesibles coma a espectrometría de masas con fonte de plasma acoplado indutivamente de tempo de voo (ICP-TOF-MS). Esta técnica permite a medida

cuasi-simultánea de todos os isótopos de forma rápida e sensible. Aínda que a precisión que ofrece é inferior á obtida co MC-ICP-MS, pode ser suficiente para discernir entre mostras. [3]

Neste traballo, desenvólvese e aplícase un método analítico para a medida das relacións isotópicas de Sr mediante ICP-TOF-MS. Estudáronse as principais variables instrumentais que afectan á precisión na relación isotópica $^{87}\text{Sr}/^{86}\text{Sr}$ e unha vez optimizado o método, empregouse para a análise isotópica de i) viños de Galicia con denominación de orixe (Rías Baixas, Ribeira Sacra, Ribeiro, Monterrei e Valdeorras) e ii) plantas procedentes de rexións próximas ós viñedos coa finalidade de obter a composición isotópica do Sr biodispoñible nestas zonas e relacionala coa dos viños.

O método baseouse na dixestión ácida das mostras e a purificación do analito de interese empregando unha resina específica para Sr. Avaliouse a eficacia da separación cromatográfica, onde se obtiveron recuperacións entre o 91 e o 110%. As mostras de Ribeiro presentaron a composición isotópica máis pesada (relación $^{87}\text{Sr}/^{86}\text{Sr}$ máis alta) mentres que as de Monterrei mostraron a composición isotópica máis lixeira. Na rexión costeira, non se detectou o efecto do asperxo mariño (*sea spray effect*). Facendo unha comparativa entre viños e plantas, non se observaron diferenzas significativas, agás no Ribeiro, posiblemente debido, o uso de aditivos, fertilizantes ou vertidos. Os datos obtidos avaliáronse respecto a modelos predictivos construídos a partir de datos doutras rexións de España e Norte de Portugal. As relacións isotópicas de $^{87}\text{Sr}/^{86}\text{Sr}$ obtidas, atopáronse dentro do rango de valores esperados. Comparando a composición isotópica do Sr biodispoñible en Galicia con diferentes zonas de Europa, obtívose unha relación isotópica máis pesada debido principalmente o leite de rochas antigas (graníticas e metamórficas) que caracteriza as zonas de estudo.

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Turn-off/on fluorescence-based detection of SARS-CoV-2 using a carbon dots optical biosensor

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The COVID-19 pandemic has underscored the urgent need for rapid, accurate, and accessible diagnostic tools for infectious diseases. Beyond clinical testing, the detection of SARS-CoV-2 in wastewater has emerged as a powerful strategy for population-level surveillance, providing early warning of outbreaks and enabling the monitoring of viral circulation in communities. This approach is particularly valuable for tracking asymptomatic carriers and capturing trends that may not be visible through clinical testing alone. However, conventional methods such as RT-PCR, while highly sensitive and specific, are constrained by the need for centralized laboratories, trained personnel, and long turnaround times, often exceeding 24 hours [1,2]. Point-of-care (POC) diagnostic systems that deliver rapid and reliable results are therefore critically needed, as they enable timely decision-making, reduce dependence on laboratory infrastructure, and facilitate large-scale wastewater epidemiology programs.

In this work, we developed a fluorescence-based optical biosensor for SARS-CoV-2 detection using waste-based carbon dots (CDs) synthesized from citric acid, sawdust, and ethylenediamine in sodium hydroxide. Citric acid serves as the primary carbon precursor, sawdust provides a sustainable biomass-derived carbon source with additional surface functionalities, ethylenediamine introduces amino groups to enhance solubility and fluorescence, and NaOH facilitates carbonization and nanostructure formation [3]. The sensing mechanism is based on a first turn-off process: Fe^{3+} ions, which are known quenchers [4] are added to CDs' solution, leading to fluorescence quenching. Subsequent addition of single-strand DNA target sequences, specific to SARS-CoV-2 virus, leads to recovery of the signal (fluorescence enhancement) by interaction of the target analyte with Fe^{3+} quencher ions. This turn-off/on process enables sensitive detection through fluorescence intensity changes upon target addition. The sensor exhibits a linear detection range from 0.1 to 1.5 μM , with a limit of detection (LOD) of 0.060 μM and a limit of quantification (LOQ) of 0.20 μM . The coefficient of variation of the method (V_{x_0}) was 2.97%, demonstrating good precision and reproducibility.

This work demonstrates the potential of biomass-derived carbon dots combined with fluorescence biosensing as a robust platform for SARS-CoV-2 detection, with applications in clinical testing, wastewater monitoring, and pandemic preparedness.

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Unveiling the impact of cadmium on silica-based food additive (E551): insights into concentration-dependent interactions

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The increasing use of nanomaterial-based food additives has improved the quality and functionality of foods but also raised concerns about their interactions with co-existing chemical contaminants. Such interactions may alter the additives' properties and, consequently, their behaviour and bioavailability.

In this study, conducted within the MixIng-Tox project (2022.03491.PTDC), we investigated how cadmium affects the physicochemical properties of the silica-based food additive E551. Four experimental scenarios were tested, combining two concentrations of E551 (300 and 3000 mg·kg⁻¹) with two concentrations of Cd²⁺ (1.0 and 3.0 mg/kg). Stable suspensions were prepared and characterised by dynamic light scattering, zeta potential and electron microscopy.

At low E551 concentration (300 mg·kg⁻¹), Cd²⁺ reduced the zeta potential magnitude, leading to increased particle size and polydispersity index (Pdl), consistent with charge neutralisation and aggregation. Conversely, at high E551 concentration (3000 mg·kg⁻¹), zeta potential remained strongly negative and unaffected by Cd²⁺, while both particle size and Pdl decreased, indicating enhanced colloidal stability.

These results highlight a concentration-dependent interplay between E551 and Cd²⁺, controlled by surface charge and ionic interactions. Understanding such mechanisms is essential for a realistic assessment of nanomaterials behaviour and stability in complex food matrices, and key to support the correct assessment of dietary exposure risks to these additives and coexisting chemical contaminants.

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Removing paralytic shellfish poisoning (PSP) toxins: what are the solutions?

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Paralytic shellfish toxins (PST) comprise dozens of analogues with different toxic potentials. All molecules in this group share the same core structure (Fig. 1), consisting of a 3,4-propinoperhydropurine tricyclic system including two guanidine groups, which confer high polarity and hydrophilicity [1]. These toxins are produced by toxic microalgae in marine systems, but also in freshwater, and can be directly ingested by organisms that inhabit these environments, particularly bivalves. Human poisoning, which can be fatal, occurs when organisms with high concentrations of paralytic shellfish toxins are consumed. Despite the severity of the problem, viable and effective solutions have not yet been found to eliminate these toxins from organisms, or to convert them into less toxic analogues. The suitability and feasibility of the methods proposed and described in the literature has been discussed [2].

In recent years, our group has carried out work aimed at the optimization of adsorbent materials, based on ion exchange resins [3] and modified clays. This communication aims to present the potential uses of these adsorbents for removing PST from marine environments, discussing their advantages as well as their limitations. Our studies were conducted in aqueous solutions, in cultures of PST-producing marine microalgae, and in live bivalves, using mussels *Mytilus edulis*. PST were quantified in the different matrices according to the current official method, using high-performance liquid chromatography with fluorescence detector (HPLC-FLD) with pre-oxidation, and the materials were characterized by FTIR, X-ray diffraction and optical microscopy. The results showed different efficiencies of the materials in the different matrices tested, revealing that the natural constituents of marine systems exert a strong influence on PST removal, which is largely explained by various competitions, and interactions between the molecules in the systems.

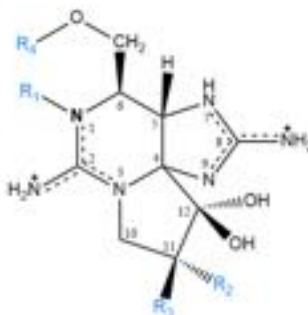


Fig.1. Core structure of PSP toxins.

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Evaluation of the seasonal variability in the phytochemical and bioactive composition of the alga *Sargassum muticum*

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Sargassum muticum, an invasive brown in European coasts, disrupts ecosystems. However, its abundant biomass also represents a sustainable and accessible source of nutrients and high-value bioactive compounds. Since its biochemical profile is shaped by environmental factors that vary throughout the year, understanding seasonal fluctuations is key to optimizing its valorization. To address this, monthly samples were collected during 2024 from a single location (in Vigo, NW Spain) under identical processing conditions, enabling seasonal comparisons [1-2].

Proximal analysis revealed the highest protein content in March (114 mg/g), while polysaccharides, mainly fucoidans, peaked in July (63 mg/g). To assess variations in key compounds (polysaccharides, proteins, and phenolics), maceration extractions were carried out using two solvents of different polarity (water and 70% ethanol). Aqueous extracts yielded higher concentrations of the target compounds, reaching maximum values in March for proteins (43 mg/g), in June for fucoidans (7 mg/g), and from June to August for phenolics (43.5 mg GAE/g algae).

Aqueous extracts also exhibited the strongest antioxidant activity (DPPH and FRAP assays) from June to August. Regarding antihypertensive potential (ACE inhibition), the August extract showed the highest activity (IC₅₀ = 0.69 mg/mL), whereas the March extract presented the lowest (IC₅₀ = 4.80 mg/mL). For antidiabetic activity (α -glucosidase inhibition), June samples were the most active, contrasting with the weaker effect observed in March. In addition, antimicrobial assays revealed that aqueous extracts from summer months displayed the strongest inhibitory effect against both Gram-positive and Gram-negative bacteria, while lower activity was observed in spring samples. These findings suggest that summer months enhance the bioactivity of *S. muticum*, likely due to increased levels of phenolics and fucoidans.

This study provides key insights into the optimal harvesting periods of *S. muticum* depending on the compounds of interest, supporting its potential applications in the food and pharmaceutical industries.

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Anthocyanins-proteins interactions for the development of natural blue colorants

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The increasing consumer demand for natural and health-promoting food ingredients has intensified the search for alternatives to synthetic colorants, which remain under scrutiny due to potential health risks. Among natural pigments, anthocyanins, a group of water-soluble flavonoids responsible for the red, purple, and blue hues of many fruits, vegetables, and flowers, have emerged as particularly promising candidates. In addition to their broad chromatic versatility, anthocyanins exhibit strong antioxidant capacity and have been linked to a range of health benefits. This dual role positions them not only as attractive natural colorants but also as functional food ingredients capable of enhancing the nutritional and therapeutic value of food products [1,2].

Despite their potential, the practical application of anthocyanins is constrained by their limited stability under external conditions. These pigments are highly sensitive to pH, temperature, light, and oxygen, factors that can significantly compromise their color expression and stability. Recent studies have highlighted protein-anthocyanin interactions as a promising stabilization strategy, with such complexes shown to improve not only anthocyanin stability but also their solubility and bioavailability [3,4].

Taking advantage of anthocyanins' characteristics as potential natural colorants and their well-documented interaction with proteins, this study aimed to modulate the color of different anthocyanin extracts to develop a novel natural blue colorant. Plant- and yeast-based proteins (pea, rice, and YBP) were combined with two anthocyanin-rich extracts with different structural properties, namely i) polyacylated anthocyanins; and, ii) mono- or non-acylated anthocyanins. Protein-anthocyanin interactions were evaluated under various pH conditions, with a particular focus on stabilizing the characteristic blue hue. In all conditions tested, protein complexation intensified the blue coloration, with polyacylated anthocyanins showing the highest degree of stabilization. Overall, these findings provide valuable insights into the protein-mediated stabilization of anthocyanins and highlight their potential for the development of stable blue food colorants.



Fig.1. Interaction of polyacylated anthocyanin-rich extract with different proteins at pH 8.0.

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Pão enriquecido com *Chlorella vulgaris* Honey: avaliação nutricional e determinação de luteína

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Nas últimas décadas, o aumento da incidência de doenças crónicas não transmissíveis tem sido associado a hábitos de vida pouco saudáveis e ao consumo excessivo de alimentos ultraprocessados. Neste contexto, o desenvolvimento de produtos que promovam uma alimentação equilibrada e que sejam enriquecidos com ingredientes funcionais representa uma tendência no setor alimentar, sendo o pão um dos veículos mais promissores devido à sua ampla aceitação e versatilidade [1]. De entre os ingredientes promissores e de base biológica, destaca-se a microalga *Chlorella vulgaris* Honey, reconhecida pelo seu elevado teor de proteínas, vitaminas, minerais, bem como por ser uma fonte de luteína – carotenóide associado a diversos benefícios para a saúde macular [2]. Assim, o presente estudo teve como objetivo desenvolver um pão inovador enriquecido com 5 e 10% de *C. vulgaris* Honey e avaliar o impacto da incorporação nas propriedades nutricionais e no teor de luteína no produto final. A caracterização nutricional do pão foi realizada de acordo com os métodos oficiais de análise de alimentos AOAC. O teor de luteína foi determinado por cromatografia líquida de alta eficiência (HPLC) acoplado com detetor de díodos, após extração exaustiva do composto em etanol, assistida por ultrassons. Foi também desenvolvido um pão sem a adição de *C. vulgaris* (controlo). Ensaios preliminares indicam que o pão enriquecido com 5% de *C. vulgaris* não alterou significativamente o sabor do produto, enquanto a adição de 10% resultou num sabor mais intenso e perceptível. O pão fortificado com 5% apresentou um teor de humidade de 33,87 % de massa fresca (mf), cinzas de 2,30 g/100g mf, lípidos de 0,35 g/100g mf, proteínas de 8,73 g/100g mf, fibras de 3,96 g/100g mf e hidratos de carbono de 50,78 g/100g mf. Em comparação, o pão controlo apresentou teores de humidade de 27,81 %, cinzas de 1,64 g/100g mf, lípidos de 0,20 g/100g mf, proteínas de 8,07 g/100g mf, fibras de 3,62 g/100g mf e hidratos de carbono de 62,28 g/100g mf. A análise de ácidos gordos revelou que o pão enriquecido com 5% de microalga apresentou um perfil predominantemente insaturado, destacando-se pela presença de ácidos gordos ómega-3, nomeadamente o EPA (ácido eicosapentaenoico), que não foi detetado no pão controlo. A quantificação de luteína revelou um teor de 1,25 µg/g massa seca (ms) no pão enriquecido com *C. vulgaris*, enquanto no pão controlo não foi detetada a presença deste carotenoide. Estes resultados reforçam o potencial da microalga *C. vulgaris* como ingrediente inovador na formulação de produtos alimentares sustentáveis e nutritivos.

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From waste to taste: sustainable seasoning production from atlantic mackerel by-products

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In Portugal, approximately 1.93 million tons of food are wasted annually [1]. This large-scale loss leads to significant environmental, social, and economic consequences, including an increased carbon footprint, enlarged overload on waste management systems, and the depletion of essential natural resources. Reducing food waste is therefore central to fostering a circular economy and improving food system resilience.

Against this backdrop, the present study explores a sustainable approach to utilize fresh Atlantic mackerel (*Scomber scombrus*) by-products generated during the processing of canned fish. These by-products, comprising primarily heads, bones, and skin, are often underutilized despite their nutritional potential. The overarching goal of the study was to transform these materials into a low-sodium, mineral-rich natural seasoning, thereby addressing both food waste reduction and the demand for healthier, functional food ingredients.

To this purpose, a nutrient-rich broth was prepared using a combination of mackerel by-product and selected vegetables, aromatics [2,3], and wine. The inclusion of wine played a dual role: enhancing the solubility of minerals and facilitating their extraction from the bones and tissues [4]. This broth was boiled for two hours, strained, and subsequently frozen for freeze-drying. Subsequent analytical characterization focused on several key parameters, including mineral composition, oxidation indicators, sodium content, macronutrients, and the free amino acid profile.

Conceived as a natural and versatile seasoning, this broth exemplifies the potential of by-product valorization in the seafood sector. Its development not only contributes to healthier dietary practices by offering a low-sodium alternative to conventional seasonings but also aligns with broader sustainability strategies, promoting resource efficiency, circular economy principles, and the responsible use of marine resources.

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Integrating edible insects and spirulina as a nutritive and sustainable food solution: consumer acceptance, sensory analysis, proximate composition and immunoreactivity

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The consumption of insects – entomophagy – is gaining attention as a viable and sustainable alternative to traditional meat and plant protein sources. Likewise, spirulina is a widely cultivated cyanobacteria, accounting for over 30% of global 'microalgal' biomass production, which is recognised as a highly nutritive protein source. In response to the growing demands for high quality nutrients relying on sustainable protein sources, we propose four novel food formulations/products incorporating insects (*Tenebrio molitor* larvae) and spirulina to enhance protein and nutritional value. With this aim, a consumer acceptance survey was first conducted to assess market viability, evaluating awareness, interest and acceptance of insect and/or spirulina-based products. Due the encouraging results, several foods were prepared with addition of insect+spirulina (crackers, bread, pancakes, pasta) and their organoleptic characteristics, proximate composition and immunoreactivity were evaluated. A CATA (Check-All-That-Apply) analysis was conducted to assess the sensory attributes of the different food formulations, comparing with respective control products. The formulated foods were also characterised regarding their fatty acid and amino acid contents. In addition, their immunoreactivity was evaluated by immunoblotting and ELISA using sera of patients allergic to crustaceans. The results of sensory/organoleptic evaluation showed that neither insect nor spirulina affected the texture attributes. Notably, the products' green colour influenced their acceptability since some participants described it as "vegetal" or "algae" attributes. Regarding the compositional analysis, the formulated products showed higher protein, amino acid and fatty acid contents, without significantly affecting both amino acid and fatty acid profiles. The results show some cross-reactivity for individuals allergic to crustaceans, as their IgE reacted with insect proteins. Considering the encouraging acceptability results and their enhanced nutritional value, the new insect+spirulina formulated products present an opportunity for food innovation, with potential for further industrial improvement/development and commercialisation, towards the rising demand of sustainable protein rich foods.

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Structural and matrix effects on astringency in butterfly pea (*Clitoria ternatea* L.) tea

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Astringency is perceived as a tactile sensation characterized by roughness, puckering, and tightening in the mouth [1]. It is a key sensory attribute in polyphenol-rich foods and beverages such as tea and red wine, resulting from interactions between polyphenols and oral constituents. Beyond their intrinsic chemical structure, these interactions may be modulated by the food matrix. *Clitoria ternatea* L., commonly known as butterfly pea flower, is particularly rich in flavonols and polyacylated anthocyanins (ternatins) [2], which contribute to both its sensory attributes and its characteristic blue color. Known for its herbal flavor, often compared to black tea and coffee [3], butterfly pea tea can also induce astringency, which is commonly mitigated by the addition of lemon or honey [3].

This study explored the molecular basis of astringency in the butterfly pea flower tea using a cell-based oral model comprising human saliva, oral mucosal pellicle, and HSC3 oral epithelial cells under acidic conditions. To evaluate the contribution of structural features of phenolic compounds and to assess potential matrix effects, an equimolar mixture of flavonols present in the extract and their respective aglycones was compared with the whole butterfly pea extract (PBE).

Adsorption experiments revealed that p-coumaroyl residues enhanced oral interactions, whereas rhamnosyl residues reduced them. Furthermore, quercetin-3-O-rutinoside showed significantly higher binding in the PBE matrix compared to equimolar mixtures, suggesting that the matrix enhances oral interactions. These findings underscore the importance of considering matrix effects, including flavonoid–flavonoid or flavonoid–anthocyanin interactions, when investigating the mechanisms underlying astringency perception.

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Design and application of antioxidant surfactants to preserve Omega-3 rich foods

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Clinical trials have demonstrated that omega-3 fatty acids (FAs) can reduce the risk of various diseases. However, the use of omega-3-rich oils, such as fish and flaxseed oil, in supplements poses several challenges due to their low water solubility, unpleasant taste, and poor oxidative stability. Although emulsions and liposomal formulations can offer some advantages, their limited oxidative stability has hindered broader application. Similarly, the low oxidative stability of many frozen fish products and by-products has restricted their commercial potential, and the addition of conventional antioxidants (AOs) has proven largely ineffective.

In this study, we developed novel antioxidants with enhanced surfactant properties by incorporating a highly active radical-scavenging molecule designed to optimize interfacial positioning in multiphase systems.[1] Specifically, we synthesized a series of cationic caffeic acid-based phenolipids by using serine as the central scaffold, to which both the caffeic acid and various long-chain fatty alcohol moieties (methyl, octyl, lauryl, palmitoyl, stearyl, oleyl, and phytyl) were covalently attached. The cationic nature of these compounds may contribute to improved antioxidant efficiency and potential antimicrobial activity, making them particularly suitable for stabilizing fish-based food products.

All synthesized caffeic acid cationic phenolipids were obtained in good yields (over 70%). Preliminary voltammetric studies showed that increasing the alkyl chain length did not significantly affect the anodic peak potential (*E_{pa}*), which remained around 235 mV—higher than that of α -tocopherol (209 mV). However, the increase in chain length appears to be a key factor in shifting from diffusion-controlled to adsorption-controlled behavior, indicating a stronger interfacial affinity. This suggests greater effectiveness in protecting surfaces or membrane structures. Additionally, their radical scavenging activity against DPPH exceeded that of α -tocopherol.

The antioxidant performance of these compounds in model fish oil emulsions and liposomes was evaluated, revealing that they exhibited superior antioxidant capacity compared to α -tocopherol. Preliminary toxicological evaluation indicates that the introduction of the amino acid moiety reduces the toxicity compared to the corresponding caffeic acid phenolipid.[2]

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Decoding astringency: linking oral interactions to sensory perception

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Astringency is a multifaceted oral sensation, typically described as tightening, puckering, or epithelial roughness [1]. It constitutes a critical sensory attribute influencing consumer acceptance of plant-based foods and beverages. While traditionally linked to salivary protein precipitation, recent evidence shows it arises from multiple mechanisms, including disruption of the salivary film, epithelial adsorption, and trigeminal activation [2]. Beyond intensity, astringency presents distinct *subqualities* (such as puckering, velvety, rough) that remain poorly understood at the molecular level [3,4].

This study dissected the mechanisms of three representative astringent agents: black tea extract (catechin-rich, associated with puckering), alum (multivalent salt, eliciting aggressive dryness), and quercetin (flavonol linked to velvety smoothness). Using cell-based oral models (human tongue-derived epithelial cells, mucosal pellicle, and human saliva in separate and integrated whole systems), adsorption was quantified by UV-Vis assays and complemented by atomic force microscopy (AFM). AFM enabled direct mapping of surface roughness and nanotribological (friction forces) changes upon exposure.

Distinct compound-model interactions emerged. Alum and quercetin strongly increased roughness and friction on epithelial cells surfaces, reflecting preferential epithelial adsorption. In contrast, black tea extract exerted major effects only in the whole model, highlighting the critical role of matrix complexity in polyphenol-driven astringency. These molecular fingerprints aligned with distinct sensory subqualities, namely aggressive puckering for alum, velvety smoothness for quercetin, and pronounced puckering for BTE.

This work demonstrates that astringency results from convergent molecular pathways shaped by both chemical structure and oral environment. The combination of AFM with cellular and salivary models offers a powerful platform to link nanoscale interactions to perceptual outcomes. Such mechanistic insights pave the way for rational design of plant-based foods with optimized mouthfeel and improved consumer acceptance.

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Yeast protein extracts and hydrolysates production as a novel source of umami flavour enhancers

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Consumer concern and demand for health and environmental sustainability have motivated the development of vegan and eco-friendly food alternatives. Yeast protein has emerged as a promising ingredient, combining both nutritional value and functional versatility. This work explores the potential of multiple yeast protein extracts (YPE) as natural sources of umami and how hydrolysis can boost this potential to produce a novel flavour enhancer suitable for industrial production.

Both chemical and enzymatic hydrolysis were performed and optimized on YPEs to industrially viable conditions, with efforts to balance efficiency, cost, and final product quality. Reaction progress was monitored through techniques like SDS-PAGE, HPLC-SEC (high-performance liquid chromatography – size exclusion chromatography), while peptide characterization was achieved through high-resolution mass spectrometry. Predictive bioinformatic tools, in this case UMPred-FRL [1] and iBitter-Fuse [2], were employed to estimate the umami and bitter potential of the identified peptides profile. Finally, sensorial analysis was conducted to assess consumer acceptance, comparing against existing commercial flavour enhancers produced from similar matrices.

Optimization of the hydrolysis process successfully produced flavour enhancers with high amounts of umami peptides. Analytical and predictive results confirmed strong umami flavour potential with limited bitterness. These results were confirmed by sensorial analysis trials, with competitive performance when compared to commercial products.

Yeast protein hydrolysates present a sustainable and consumer-accepted solution for umami flavour enhancers. Analytical, computation and sensory methodologies were successfully employed to provide a comprehensive characterization of the produced hydrolysates. This work highlights the effectiveness of the collaboration between academia and industry in driving sustainable innovation within the food sector.

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Efeito do regime hídrico e de culturas intercalares na qualidade e produtividade de *Prunus dulcis* Mill. em três anos de produção

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Uma das principais prioridades da agricultura é assegurar a produção de alimentos seguros e nutritivos, atendendo às necessidades alimentares da população e promovendo sistemas produtivos mais sustentáveis [1]. Nesse contexto, a diversificação das culturas em amendoais busca melhorar a saúde do ecossistema, prevenir a erosão e mitigar os impactos das mudanças climáticas, além de favorecer a fertilidade do solo [2,3]. Este estudo teve como objetivo avaliar as propriedades físicas e nutricionais das amêndoas, bem como o rendimento da produção, em diferentes condições hídricas (de sequeiro e regadio) ao longo de três anos.

Para a análise física das amêndoas, recorreu-se a um colorímetro para a avaliação da cor e, de um texturómetro TA.XT plus para avaliar a textura. A composição nutricional foi determinada através das Metodologias Oficiais da Análise (AOAC), incluindo teores em proteínas, gorduras, cinzas, hidratos de carbono e humidade. No primeiro ano não foram observadas diferenças significativas entre as amostras, enquanto nos anos seguintes as amêndoas de sequeiro apresentaram coloração mais escura, além de diferenças de dureza e gomosidade. Quanto à composição nutricional, verificou-se que as amêndoas cultivadas em sequeiro apresentaram maiores teores de proteína no primeiro ano, com destaque para os consórcios com trevo e grão-de-bico, que apresentaram valores de 18 ± 1 g/100g pf e $15,8 \pm 0,3$ g/100g pf, respetivamente. Já o sistema de regadio apresentou maiores teores de gordura ao longo dos três anos.

Em relação ao rendimento, observaram-se diferenças marcantes entre os sistemas. No sequeiro, a produção variou de 18,2 a 65,0 kg/15 árvores, dependendo do ano e do consórcio, enquanto no regadio os valores foram consistentemente superiores, variando de 27,1 a 129,1 kg/15 árvores. Destacaram-se, sobretudo, os consórcios de grão-de-bico em regadio, que apresentaram as maiores produtividades em todos os anos avaliados. Em suma, este estudo demonstra que os sistemas de culturas intercalares podem contribuir para a sustentabilidade da produção, preservando a qualidade nutricional e física das amêndoas, ao mesmo tempo que favorecem maior rendimento sob condições de regadio.

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Caracterização bioquímica e potencial de valorização de perdas pós-colheita de *Daucus carota* L.

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A cenoura (*Daucus carota* L.) é amplamente consumida devido ao seu valor nutricional e presença de fitoquímicos tais como vitaminas, minerais, carotenoides e compostos fenólicos. Em 2022, a sua produção global ultrapassou as 36 milhões de toneladas [1]. No entanto, uma parte significativa da sua produção acaba sendo descartada por não satisfazer os padrões estéticos exigidos pelo consumidor, nomeadamente o formato irregular, tamanho ou pequenas imperfeições na casca. Estas perdas pós-colheita contribuem para o impacto ambiental e económico, sobretudo devido à geração de resíduos orgânicos [2]. Assim, a sua utilização constitui uma via sustentável de valorização agroalimentar, uma vez que apresentam elevados teores de compostos bioativos. Este trabalho teve como objetivo, caracterizar as propriedades químicas e bioativas das perdas pós-colheita de cenoura, explorando o seu potencial de reaproveitamento. Amostras de cenoura laranja, amarela e roxa foram fornecidas por uma empresa local de Bragança, Portugal. As amostras foram liofilizadas e mediante técnicas cromatográficas foram identificados e quantificados os ácidos orgânicos (UFLC-PDA), os tocoferóis e os folatos (HPLC-FL), os minerais por espectrofotometria de absorção atômica e os carotenoides totais por espectrofotometria. Para a avaliação do potencial bioativo, as amostras foram submetidas à extração por decocção e maceração etanol/água (80:20, v/v). A atividade antioxidante foi avaliada através do ensaio de TBARS (inibição da peroxidação lipídica), DPPH (captação de radicais livres) e Poder Redutor, enquanto a atividade antibacteriana foi avaliada pelo método da microdiluição em caldo contra estirpes bacterianas alimentares. A cenoura de variedade laranja destacou-se com o maior teor de ácidos orgânicos (7,3 g/100g massa seca (ms)) e de folatos totais (634 µg/100g ms), a variedade roxa apresentou o maior teor de tocoferóis (14,17 mg/100g ms) e carotenoides (0,096 mg/g ms). Em relação aos minerais, todas as amostras revelaram teores elevados de potássio (9,5-14,8 g/kg ms). Os extratos de decocção da variedade laranja e roxa apresentaram melhor atividade antioxidante nos ensaios realizados. O extrato hidroetanólico da cenoura amarela apresentou a melhor atividade, com uma concentração mínima inibitória (CIM) de 1,25 mg/mL contra a estirpe *Yersinia enterocolitica*. Este estudo evidencia o potencial destes resíduos como uma fonte de compostos bioativos, destacando a sua aplicação na criação de soluções sustentáveis e inovadoras, e reforçando o papel da economia circular.

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Métodos emergentes de extração como estratégia de valorização de subprodutos de frutas

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A recuperação eficiente de extratos ricos em compostos fenólicos de subprodutos de frutas está relacionada com o procedimento de extração, visto que a variação dos métodos aplicados e respetivos parâmetros podem afetar significativamente a qualidade e a quantidade desses compostos [1]. Nesse contexto, considerando a importância de valorizar subprodutos de frutas, este estudo teve como objetivo otimizar três métodos diferentes de extração: extração assistida por microondas (MAE), extração com líquido pressurizado (PLE) e extração assistida por calor (HAE), a fim de maximizar a recuperação de compostos fenólicos totais de três subprodutos de frutas: anona (*Annona cherimola* Mill.), ameixa (*Prunus domestica* L.), e carambola (*Averrhoa carambola* L.). A otimização das três técnicas foi realizada utilizando uma metodologia estatística contemplando a avaliação de diferentes variáveis: para PLE, tempo (t, 1-25 min), temperatura (T, 50-200 °C) e percentagem de etanol (S, 0-100%); para MAE, (t, 5-25 min, T, 50-180 °C, S, 0-100%); e HAE, (t, 5-60 min, T, 30-90 °C, S, 0-100%) num delineamento composto central circunscrito com cinco níveis, resultando em 28 ensaios independentes. As respostas avaliadas foram rendimento, atividade antioxidante (através do método de captação de radicais DPPH) e compostos fenólicos totais (analisados por HPLC-ESI-QqQ-MS/MS). Os resultados mostraram que as três amostras responderam de maneira distinta às diferentes técnicas de extração estudadas. Para a ameixa, MAE e PLE apresentaram desempenho superior ao método convencional HAE, proporcionando tempos de extração reduzidos e maior eficiência na recuperação de compostos fenólicos (134,2±5,8 mg/g e 136,8±5,8 mg/g, respetivamente), além de melhor atividade antioxidante. Na anona, PLE apresentou maior recuperação de compostos fenólicos utilizando apenas água, mas com menor rendimento e atividade antioxidante, enquanto MAE (5 min, 50 °C, 100% etanol) mostrou melhor desempenho global. Para a carambola, o tempo de extração ideal foi de 5 min para MAE e HAE, e 25 min para PLE, destacando-se MAE como a técnica mais eficiente (46,1±3,4 mg/g de compostos fenólicos totais e atividade antioxidante de 0,40 mg dw/mL). De forma geral, a MAE evidenciou vantagens significativas em eficiência e sustentabilidade, ao associar menor tempo de extração, maior concentração de compostos fenólicos totais e atividade antioxidante superior nos três subprodutos analisados. A ameixa apresentou maior concentração de compostos fenólicos totais, destacando o seu potencial bioativo. A valorização sustentável desses subprodutos poderá contribuir para a redução do desperdício alimentar e apoiar os Objetivos de Desenvolvimento Sustentável da Agenda 2030, promovendo inovação e sustentabilidade na cadeia agroalimentar.

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Circular Economy in Tomato Processing: From By-products to Value-Added Ingredients

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The valorisation of by-products from the tomato processing industry offers a sustainable strategy to reduce waste and recover functional ingredients with potential applications in food, nutraceutical, and antimicrobial formulations. Rich in bioactive such as polyphenols and carotenoids these by-products present a unique resource aligned with circular economy principles. This study, conducted in collaboration with a multinational tomato processing company based in Portugal, aimed to valorise these by-products by characterizing their biochemical composition and assessing their antioxidant and antimicrobial potential, including their role as natural photosensitizers in photodynamic inactivation (PDI).

This research focused on optimizing the extraction and quantification of these bioactive compounds from by-products of standard and high-lycopene tomato varieties. Polyphenol content was quantified using both colorimetric methods (Folin–Ciocalteu assay) and HPLC-DAD-ESI-MS/MS, providing comprehensive insights into composition and concentration. Total polyphenol content was comparable between varieties and 28 polyphenols were tentatively identified. These included flavonoids in high concentrations such as rutin and naringenin chalcone, along with phenolic acids such as *p*-coumaric acid and chlorogenic acid. Carotenoids were also extracted and quantified through HPLC and spectrophotometric methods, as well carbohydrates quantified through colorimetric methods.

Antioxidant capacity was measured via DPPH and FRAP assays, with both varieties showing high activity. Antimicrobial testing of the polyphenol and carotenoids extracts against bacteria strain models commonly associated with tomato spoilage was performed both in the absence and presence of irradiation, highlighting the potential of these compounds as natural antimicrobials and potential photosensitizers.

This work demonstrates that tomato by-products can be transformed into high-value ingredients with antioxidant and antimicrobial properties. The optimized extraction and quantification strategies developed in this study support future applications of these by-products as functional ingredients as well as creating possible sustainable food preservation systems.

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Extração de proteína da *Ulva rígida* e *Sargassum spp.* – comparação de métodos

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A valorização de recursos marinhos como fonte de biomoléculas com elevado valor acrescentado tem despertado crescente interesse na investigação e na indústria alimentar. As macroalgas apresentam elevado teor proteico e diversidade de aminoácidos essenciais, configurando-se como matérias-primas promissoras para aplicação em produtos alimentares [1]. No entanto, a elevada concentração de minerais, estando alguns acima dos limites máximos admissíveis, limita a aplicação direta das macroalgas na alimentação. Uma alternativa é a extração das proteínas para enriquecimento de alimentos, como por exemplo pão sem glúten. Neste contexto o objetivo do trabalho é estudar a eficiência e seletividade de métodos de extração de proteína e a sua estabilidade [2].

O presente estudo avalia a extração de proteínas a partir de duas espécies de macroalgas recolhidas na costa litoral de Viana do Castelo: *Sargassum spp.* (alga castanha) e *Ulva rígida* (alga verde). Foram comparadas metodologias de extração: hidrólise enzimática com Viscozyme®, ruptura mecânica com bead beating [3] e cavitação induzida por ultrassons, e comparados com a extração aquosa em pH alcalino. A eficiência dos métodos é analisada através da quantificação de proteínas solúveis. A análise comparativa permitiu identificar a metodologia mais eficaz, considerando o rendimento proteico, e a potencial aplicação no enriquecimento de pão sem glúten. Na espécie *Sargassum spp.*, o teor de proteína no extrato variou entre 2,76% (Bead beater) e 3,89% (hidrólise enzimática com Viscozyme®), correspondendo a 13,3% e 18,8% da proteína no resíduo seco respetivamente. Os hidratos de carbono oscilaram entre 3,85% e 16,6%, resultando numa extração de 14,4% no método bead beater e 62,2% com hidrólise enzimática. Para *Ulva rígida*, os valores de proteína variaram entre 3,94% (Bead beater) e 4,25% (hidrólise enzimática), equivalendo a 15,3% e 16,4% da proteína no resíduo seco, respetivamente. Os hidratos de carbono apresentaram variações entre 7,65% e 14,82%, correspondendo a 26,4% e 51,2% da extração.

Os resultados mostram que existiu uma rutura da matriz estrutural com libertação de proteínas e de açúcares. As interações químicas entre hidratos de carbono e proteínas revela-se promissora para o enriquecimento de pão, permitindo melhorar simultaneamente o valor nutricional e a funcionalidade do produto na indústria alimentar. O estudo evidencia a importância da química aplicada à extração e caracterização de biomoléculas de macroalgas, destacando o seu contributo para o desenvolvimento de estratégias inovadoras e sustentáveis.

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Cyclodextrins as modulators of black tea astringency: impact on polyphenol–oral component interactions

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Black tea, a plant-based beverage widely consumed worldwide, is one of the largest dietary sources of polyphenols with recognized health benefits and unique organoleptic properties. Regular black tea consumption has been associated with multiple protective effects, including antioxidant and anticancer activities, reduction of blood pressure and sugar levels, improvement of gut health, and a decreased risk of cardiovascular and neurodegenerative diseases. These benefits are mainly due to its polyphenol content, especially flavan-3-ols. During enzymatic fermentation, these catechins are oxidized into theaflavins, theasinensins and thearubigins, which contribute to black tea's colour, flavour and bioactivity [1]. However, its consumption is often limited by sensory challenges, particularly astringency, a complex oral sensation perceived as dryness, puckering, or tightening, traditionally linked to interactions between polyphenols and salivary proteins, oral epithelial cells and/or mucosal pellicle, forming insoluble aggregates that reduce lubrication [2]. Recent studies suggest that polysaccharides could modulate these interactions offering promising potential to improve sensory experiences and consumer acceptance of healthy diets [3]. Cyclodextrins, cyclic oligosaccharides widely approved as food additives, are commonly used in the food industry for the encapsulation of compounds to improve organoleptic quality, enhance stability/solubility and extend shelf life [4]. However, their potential to mitigate interactions between tea polyphenols and oral components, thereby reducing the perception of astringency remains unexplored.

This work aimed to explore the capacity of three types of cyclodextrins (α -, β -, and γ -) to modulate the interactions between black tea polyphenols and key oral components involved in astringency perception. Their effect was studied using an advanced/representative quaternary cell-based oral model, comprising human tongue epithelial cells (HSC-3 cell line), human saliva, and mucosal pellicle. Black tea extract (BTE) was incubated with each cyclodextrin in the presence of individual oral components as well as in the complete model (HSC-3–mucin–salivary proteins). The experimental approach focused on evaluate the polyphenol binding to the oral constituents in the absence and presence of cyclodextrins by UV-Vis colorimetric detection assay. In addition, the polyphenol and salivary protein profile was analysed by HPLC, to assess the efficacy of cyclodextrins in reducing specific BTE–oral component interactions.

All cyclodextrins significantly decreased polyphenol-oral components interactions, demonstrating their potential as modulators of astringency. β -Cyclodextrin was the most effective, achieving the greatest reduction of polyphenol adsorption and protein precipitation mainly in the complete oral model, likely due to its optimal cavity size and balanced hydrophobic/hydrophilic properties that limit polyphenol binding to oral constituents.

These findings provide molecular evidence that cyclodextrins modulate black tea astringency with β -cyclodextrin showing the highest efficacy. Beyond their established roles as food additives, cyclodextrins offer a promising strategy to reduce astringency and improve sensory quality and consumer acceptance of polyphenol-rich products without compromising bioactive potential.

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***Prunus lusitanica* L. subsp. *lusitanica* fruits and by-products as a novel source of added-value metabolites**

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Prunus lusitanica L. subsp. *lusitanica*, commonly known as 'azereiro' or Portuguese laurel, is a rare and native species of Portugal. As a relic of the laurel forest, it holds significant ecological and ornamental value, yet it is currently endangered within Portuguese territory [1]. Hence, bioprospecting specialized metabolites could support the development of strategies to promote the conservation of this species and its wider cultivation.

UHPLC-ESI-DAD-MSⁿ and GC-MS analyses were conducted to assess the chemical profile of ethanol and hexane extracts of fruits, seeds, and leaves collected from three distinct geographic locations and plants representing different developmental stages (young and adult trees). The antioxidant capacity of the polar extracts was evaluated using the DPPH assay. Additionally, total reducing sugar content, total phenolic content (TPC) and total flavonoid content (TFC) were quantified spectrophotometrically. To the best of our knowledge, this is the first study to unveil the lipophilic composition of fruit extracts and the chemical profile of seed extracts, as well as to provide a detailed characterization of the leaf extracts.

Hexane extracts comprise several compound classes, including fatty acids and derivatives, sugars and derivatives, terpenoids, aldehydes, alcohols and hydrocarbons. Leaves stood out as a notable source of sorbitol (1,37 – 9,57 mg g⁻¹ DW), rutinose (2,25 – 15,31 mg g⁻¹ DW), and friedelin (4,54 – 7,35 mg g⁻¹ DW). Regarding fatty acid content and nutritional indices, seed extracts exhibited the best nutritional quality, particularly in terms of the PUFA/SFA ratio, desirable fatty acid (DFA) index, and the hypocholesterolemic/hypercholesterolemic ratio (H/H ratio).

UHPLC-ESI-DAD-MSⁿ analysis uncovered the predominance of phenolic acids and flavonoids in the polar extracts. Leaf extracts exhibited significantly higher antioxidant potential compared to fruit and seed extracts, likely due to their higher total phenolic content and total flavonoid content. In addition, fruits showed the highest concentration of reducing sugars, averaging 257,31 ± 4,56 mg g⁻¹ DW.

In-depth knowledge of the chemical profile of *P. lusitanica* subsp. *lusitanica* fruits and by-products was acquired, highlighting it as a novel source of added-value metabolites with broad applications in the food industry. These insights will be presented and discussed in detail in this communication.

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Tratamientos hidrotérmicos sucesivos del bagazo de uva tinta: estrategia para maximizar la recuperación de polifenoles bioactivos

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El bagazo de uva constituye el subproducto mayoritario generado por las bodegas durante el proceso de elaboración del vino. A nivel mundial, se producen anualmente alrededor de 4 millones de toneladas de este residuo, lo que equivale aproximadamente a 1 kg por cada 6 L de vino producidos. Esta biomasa está compuesta por raspones, piel, pulpa y semillas de la uva obtenidos tras la etapa de prensado. En los últimos años, el bagazo de uva ha despertado un creciente interés en sectores como el farmacéutico, cosmético y alimentario, debido a su elevada riqueza en polifenoles con reconocidas bioactividades beneficiosas para la salud humana, entre ellas propiedades antioxidantes y antiinflamatorias [1,2].

La recuperación de compuestos bioactivos procedentes de residuos de la industria agroalimentaria continúa representando un reto para la comunidad científica, especialmente cuando se emplean tecnologías innovadoras [3]. En este contexto, la extracción asistida por microondas (MAE) se considera una tecnología medioambientalmente sostenible, gracias a su bajo consumo energético y de disolventes, así como a los menores tiempos de procesamiento y elevados rendimientos de extracción que ofrece [4]. No obstante, para maximizar la recuperación de antocianinas, uno de los grupos fenólicos más abundantes en el bagazo de uva, resulta necesario aplicar un pretratamiento menos severo, dada la elevada termo-sensibilidad de estas moléculas.

El objetivo principal de este trabajo fue el evaluar la idoneidad de dos tratamientos hidrotérmicos secuenciales para el aislamiento de antocianinas (mediante tratamiento convencional) y de otros polifenoles complejos (mediante MAE) a partir del bagazo de uva tinta. La eficiencia de extracción de cada tratamiento se determinó mediante la cuantificación del contenido en fenoles, flavonoides y antocianinas monoméricas totales (TPC, TFC y TMAC, respectivamente), así como a través de la evaluación de su actividad antioxidante mediante los ensayos DPPH, ABTS, FRAP, CUPRAC y TAA. Por otro lado, se llevaron a cabo análisis de TBARS, citotoxicidad, actividad antibacteriana y antifúngica, y se caracterizó el perfil fenólico de los extractos obtenidos bajo las condiciones óptimas de cada tratamiento por HPLC-MS-ESI.

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The volatile profile of beer: a review of chemical diversity and analytical perspectives

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Beer is a chemically complex matrix, rich in volatile organic compounds (VOCs) originating from raw materials, fermentation, and maturation. These molecules collectively define its aroma, flavor, and stability, making their characterization crucial for monitoring and improving beer quality throughout production [1]. The work presented here compiles and critically reviews the main VOCs identified in beer, providing an updated overview of their chemical diversity and the analytical methods currently employed for their determination.

Across recent studies, nearly one thousand distinct VOCs have been reported, reflecting an exceptional chemical diversity and reactivity [2]. Esters, alcohols, and terpenoids dominate this volatile landscape (Figure 1), whereas other groups, such as thiols and amines, are less frequently detected. The most commonly identified compounds — isoamyl acetate, 2-phenylethanol, and ethyl hexanoate — highlight the link between fermentation pathways and aromatic expression. The sporadic occurrence of molecules such as 2-nonen-1-ol, 3-acetoxypyridine, and 4-hexene-3-one further demonstrates the remarkable chemical diversity of beer and how variations in raw materials or processing conditions can reshape its volatilome [3].

Analytical strategies to characterize beer's volatilome have evolved considerably. Headspace extraction (HS), headspace solid-phase microextraction (HS-SPME), and liquid–liquid extraction (LLE) remain the most widely employed techniques for VOC isolation, balancing recovery efficiency with sample integrity [4]. Gas chromatography coupled to mass spectrometry (GC–MS) continues to be the principal tool for compound identification and quantification, while complementary detectors — such as FID, PFPD, and olfactometric interfaces — enhance detection selectivity. Nevertheless, variations in extraction selectivity, derivatization protocols, and instrument calibration often limit cross-study comparability. Most analyses are still targeted, focusing on specific compound classes, although untargeted and metabolomics-based workflows are rapidly gaining prominence.

Collectively, these findings highlight both the remarkable chemical complexity of beer and the analytical challenges inherent to its characterization. The vast majority of existing studies remain targeted, focusing on predefined compound groups, which — given the intricate matrix and synergistic interactions among volatiles — can be restrictive and potentially overlook relevant components. To address these limitations, future work will apply an untargeted mass spectrometric approach, aiming to detect compounds that have so far escaped identification and to broaden our understanding of beer's volatilome.

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New plant-based beverages from Galician Chestnut and Faba de Lourenz : ultrasound-assisted process optimization using DoE

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The development of sustainable plant-based beverages represents a growing research area offering alternatives that combine nutritional quality with local identity [1]. In this context, two emblematic Galician raw materials, *Cast a de Galicia* and *Faba de Lourenz * (both protected by geographical indication, IGP) [2,3], were selected as innovative substrates for the preparation of plant-based beverages.

This study aimed to design and optimize an ultrasound-assisted (UA) process for beverage preparation, accelerating extraction and improving the nutritional and functional properties of the final products. Critical technological parameters (soaking ratio, ultrasound time and temperature, cooking time, and soaking) were optimized by an asymmetrical 2²-3³ screening design. Responses included antioxidant capacity (DPPH, Folin-Ciocalteu), protein content (BCA), and colloidal stability. Simultaneously, phenolic compounds profiles of the raw materials were characterized by HPLC-QTOF-MS/MS after UAE with acidified methanol (pH 3), for comparative with beverages [4]. Beverages prepared under optimal conditions were then assessed for their physicochemical properties, nutritional composition, and bioactive content, with special focus on phenolic and protein fractions. In addition, a preliminary sensory evaluation was conducted to evaluate consumer perception.

The results highlight ultrasound-assisted processing with experimental design as a sustainable strategy to develop innovative functional beverages from Galician IGP raw materials, uniting food innovation with the valorization of regional agri-food heritage and their natural richness in bioactive compound.

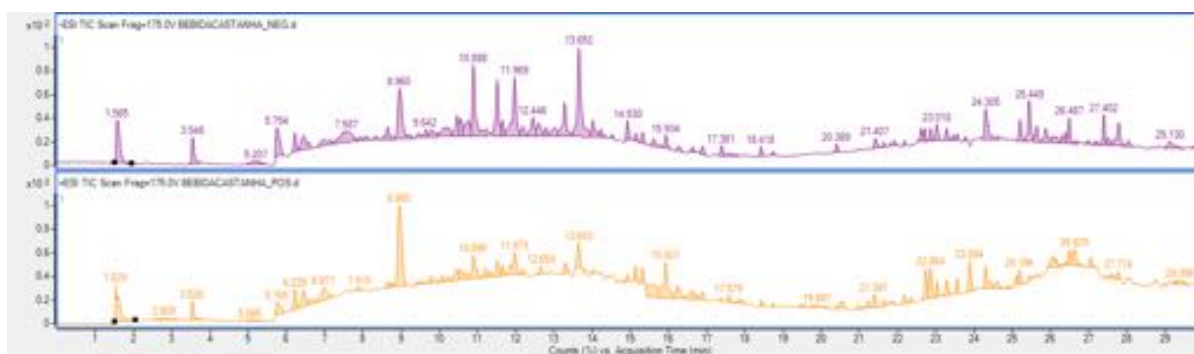


Fig.1. Total ion chromatogram of phenolics in the Galician chestnut-based beverages.

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Study of different anodes in microbial fuel cells for an efficiency industrial effluent wastewater treatment

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Water pollution from industrial effluents requires the development of innovative technologies. Microbial Fuel Cells (MFCs) is a promising solution, capable of treating organic matter and sustainably generating energy. This study explored the application of novel carbonaceous electrodes in MFCs (Fig. 1a) for the treatment of industrial effluents [1]. Paper recycling facility wastewater was collected and used in three identically constructed MFCs, varying only in the anode material. Simple graphite rod anode (A1) was used as a control (Fig. 1b). Second anode (A2) was prepared by 3D printing and impregnated by dip coating with carbon black (CB) (Fig. 1c). Third electrode (A3) was fabricated via sequential photopolymerization and carbonization enriched with both CB and iron nanoparticles (iNPs) (Fig. 1d). Photopolymerization was performed in a UV oven (405 nm) using the resin described in [2]. The current generation capacity of the MFCs was assessed for approximately 3 months, while organic matter removal efficiency was evaluated over various cycles (26 days each). The peaks voltage generation observed for the MFCs were 1.1 mA (320.8 mW/m²), 0.9 mA (180.1 mW/m²) and 2.2 mA (3755 mW/m²) using A1, A2 and A3 anodes, respectively. 48%, 56% and 80% Chemical Oxygen Demand (COD) removals were achieved in MFCs using the using A1, A2 and A3 electrodes, respectively, whereas 84%, 95% and 96% of total phenolic compounds removal were obtained in the same order, respectively. The results demonstrate a significant performance for our electrode prepared by photopolymerization with CB and iNPs compared to CB coated electrodes or traditional graphite rods.

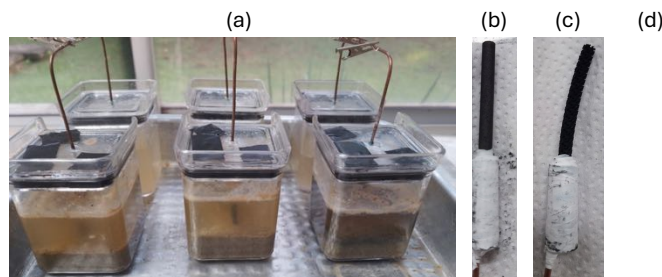


Fig.1. Photographs of (a) MFCs assembly, (b) graphite rod, (c) CB-coated electrode and (d) CB and INPs-doped photopolymer-based electrode.

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La Química implícita en el uso de las tecnologías de recuperación mejorada de hidrocarburos en la Faja Petrolífera del Orinoco

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La Faja Petrolífera del Orinoco (FPO) es el yacimiento de petróleo pesado y extrapesado más grande del mundo. Un mega yacimiento de más de 55000 Km², tan grande que en ella pueden convivir numerosas empresas de diferentes países en una serie de áreas, bloques o campos que la subdividen. Esta visión comercial disgregada, sin una visión holística y sostenible, hace que cada empresa operadora actúe de manera independiente o aislada en su respectiva área de concesión, lo cual podría desembarcar en impactos socio-ambientales no contemplados hasta ahora, sobre todo si se masifican las técnicas de Recuperación Mejorada de Hidrocarburos (RMH), en su gran mayoría técnicas que suelen aumentar Temperatura en los yacimientos para disminuir la viscosidad del petróleo crudo y hacer más fácil su desplazamiento hasta la superficie. Con un marketing que ha logrado penetrar con la idea de que se producirá mayor cantidad en menor tiempo, que no necesariamente es la mayor cantidad en el tiempo.

El presente trabajo intenta describir los diferentes procesos químicos vinculados a las tecnologías de RMH y todo lo que eventualmente pudiera ocurrir en la eventual masificación de estas Técnicas a lo largo de toda la FPO, usando información de subsuelo y de agentes contaminantes medidos en campo, tales como CO₂ o H₂S generados en las actividades típicas de la RMH. El CO₂ expulsado a la atmosfera por las locaciones de RMH si cada campo aplicara técnicas RMH como el Drenaje Gravitacional Asistido por Vapor (SAGD por sus siglas en inglés) superaría los límites acordados internacionalmente, no puede haber explotación de este tipo al unísono, debe ser programada. La velocidad de aparición y los niveles de H₂S que se presentan con el uso del agua y aumento de temperatura en la recuperación térmica son muy superiores a la producción en frío y generan peligro latente de toxicidad para las personas operadoras y los ecosistemas y/o poblaciones circundantes.

Se proponen medidas preventivas y correctivas a los fines de mitigar el impacto socio-ambiental, así como identificar y valorizar sus efectos potenciales y al mismo tiempo crear planes, programas o acciones siguiendo los parámetros establecidos en las normas ambientales, o inclusive para reescribir la normativa. [1],[2],[3],[4],[5],[6],[7]

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Optical chemosensory studies of novel D-A- π -A benzothiadiazoles for cyanide detection in aqueous media

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Donor- π -acceptor benzothiadiazole molecules have recently gained considerable interest because of their adaptable optical and electronic properties. These derivatives of benzothiadiazole are especially appealing in bioimaging and optical chemosensing applications, due to their straightforward functionalization, excellent photostability, and biocompatibility [1-5].

Cyanide ions pose severe toxicity risks to both humans and the environment by disrupting oxygen use. This interference can lead to cellular death and negatively impact several physiological systems, particularly the vascular system. Symptoms of cyanide poisoning include vomiting, and in extreme cases, it can be fatal. Despite its dangers, cyanide is used in various industries, including plastic manufacturing [6-8].

Here we present the synthesis, structural and photophysical characterization, and optical chemosensory studies of a positively charged benzothiadiazole derivative (**2**) with a D-A- π -A structure prepared from precursor **1**, which was already described elsewhere [9] (Figure 1). The optical chemosensory capabilities of this benzothiadiazole were assessed in acetonitrile (ACN) and in ACN/water mixture (75:25) against various ions of biological and environmental relevance. Spectrophotometric titrations of compound **2** were performed. The results showed a decrease in the absorption band at 460 nm, reaching the lowest intensity with 30 equivalents of CN^- . These results highlight the potential of this benzothiadiazole as an effective cyanide-selective optical chemosensor in aqueous media.

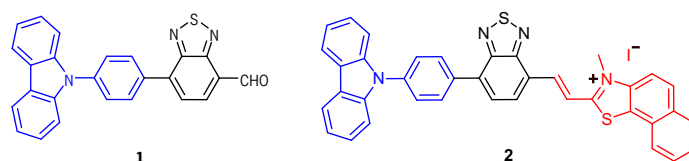


Fig.1. Structures of benzothiadiazole derivatives **1** and **2**.

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A comparative framework for evaluating energy security in Europe using a multi-criteria approach

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In the context of escalating geopolitical tensions, climate objectives, and the ongoing energy transition, energy security has emerged as a strategic imperative for European nations [1]. The vulnerability of energy systems to both internal and external shocks—such as supply disruptions, market volatility, or policy shifts—has underscored the need for robust evaluation tools that go beyond traditional metrics. Addressing this complexity requires multidimensional assessment frameworks capable of capturing the dynamic interplay between economic, environmental, and geopolitical factors [2].

This research proposes a comprehensive evaluation model grounded in Multi-Criteria Decision-Making (MCDM) techniques to assess the energy security status of selected European countries. The framework is designed to support policy planning by integrating expert-based insights with structured analysis. Twenty domain specialists were consulted to validate the selection of core indicators and to assign relative weights based on perceived importance, reflecting both qualitative judgment and technical expertise.

A key methodological feature of this model is its network-based structure, which accounts for the interdependence among criteria—a limitation often overlooked in linear decision-making tools. By adopting this approach, the model offers a more accurate reflection of real-world complexity and supports more nuanced interpretations of national performance in terms of energy security [3,4]. The findings aim to assist policymakers, analysts, and regional stakeholders in designing adaptable and forward-looking energy strategies aligned with Europe's shifting geopolitical context and sustainability goals.

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Hybrid organic–inorganic materials for Rare Earth Elements removal: unravelling the role of physicochemical properties

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The increasing demand for rare earth elements (REEs), essential to modern technologies such as magnets, catalysts, batteries, and renewable energy devices, has resulted in their accumulation in aquatic ecosystems, raising environmental and public health concerns [1]. This highlights the pressing need for efficient and sustainable strategies to reduce REEs pollution. This study investigates organic–inorganic hybrids based on pyrrolic macrocycles and silica for the removal of REEs from water, with particular focus on how variations in the macrocycle core structure influence sorption performance. Four materials (SiCor, SiPor, SiPen, and SiHex) were synthesised [2,3] and characterised by UV-Vis spectroscopy, FTIR, and BET analysis, confirming successful functionalisation and distinct physicochemical properties.

Under controlled conditions — 50 and 500 mg/L of sorbent, a mixture of six REEs (La, Nd, Gd, Dy, Ce, and Eu) at an initial concentration of 1 $\mu\text{mol/L}$, pH 6, and contact times of 0, 1, 6, 24, and 48 hours — the sorption efficiency and selectivity of each hybrid material were evaluated. The results revealed that SiPor and SiPen exhibited low sorption efficiency, SiHex demonstrated a significant sorption capacity, and SiCor achieved outstanding performance, removing over 90 % of most REEs at a sorbent dose of 500 mg/L. The robustness and exceptional thermal stability of these hybrids reinforce their suitability for sustainable water remediation applications [2,3]. This presentation will focus on the experimental details used to evaluate the performance of pyrrolic macrocycle–silica hybrids for the efficient and selective removal of REEs from water, confirming that these materials are highly promising for mitigating REE contamination in aquatic systems, combining efficiency, stability, and cost-effectiveness, while also enabling recovery of critical raw materials.

Acknowledgments

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Análisis y simulación dinámica del comportamiento energético en edificios: aplicación a la selección de aislamientos sostenibles para optimizar la eficiencia energética

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La urbanización acelerada de las últimas décadas ha provocado un aumento significativo del consumo energético en el sector de la construcción, generando impactos negativos en el medio ambiente y en la salud humana (Khrissi & Tilioua, 2025; Vilaboa Díaz et al., 2022). Este trabajo tiene como objetivo reducir la demanda energética de los edificios y, al mismo tiempo, mejorar el confort térmico mediante el uso de materiales de aislamiento sostenibles. La metodología adoptada se basa en simulaciones térmicas dinámicas de un edificio situado en una zona climática específica. Se realizó una simulación inicial sin ningún tipo de aislamiento térmico para representar un edificio convencional. Posteriormente, se simularon varios escenarios utilizando diferentes tipos de materiales de aislamiento sostenibles, seleccionados según su rendimiento térmico y su disponibilidad local. Cada material fue evaluado en términos de confort térmico durante el verano y el invierno, así como por la cantidad de potencia térmica ahorrada en comparación con el edificio sin aislamiento. Además, se incluyó un análisis económico para evaluar la viabilidad y la asequibilidad de los materiales estudiados.

Los resultados mostraron que el cáñamo y el corcho expandido ofrecieron el mejor compromiso entre eficiencia térmica, confort y sostenibilidad, mientras que el Isotex y los ladrillos de origen biológico proporcionaron mejoras moderadas pero valiosas. El poliuretano alcanzó la mayor reducción en la demanda de calefacción; sin embargo, su naturaleza sintética y no reciclable limita su idoneidad para la construcción sostenible. Este estudio demuestra, por tanto, que la adaptación de los materiales de aislamiento a las condiciones climáticas locales puede mejorar significativamente la eficiencia energética y el confort térmico, al mismo tiempo que promueve soluciones constructivas respetuosas con el medio ambiente.

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Designing functional COFs for water purification: towards stable and efficient adsorbents

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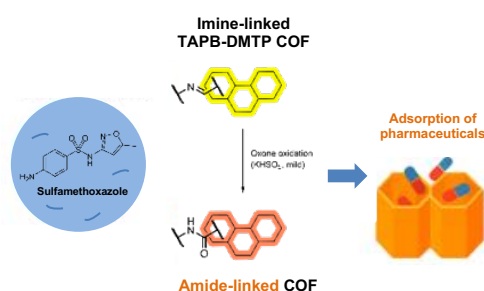
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In recent years, emerging contaminants such as pharmaceuticals have received increasing attention due to their widespread occurrence in aquatic environments and their potential impact on ecosystems and human health [1–3]. The persistence of these compounds underscores the urgent need for efficient and reusable adsorbent materials capable of capturing them under realistic environmental conditions. Covalent Organic Frameworks (COFs) have emerged as a new generation of crystalline porous materials with high surface area, low density, and uniform pore architecture, whose properties can be precisely tuned through the rational design of organic linkers [4,5]. However, the hydrolytic instability of imine-linked COFs limits their applicability in water, as framework degradation and leaching of building units.

In this work, TAPB–DMTP COF was post-synthetically transformed through an Oxone®-mediated oxidative conversion of imine into amide linkages under mild conditions [6]. This modification markedly enhanced the chemical stability of the framework without compromising its crystallinity or porosity. The resulting amide-linked COF preserved its structural integrity in various organic solvents, showing no evidence of leaching or loss of order, unlike its imine precursor. Beyond stability, the amide transformation increased the overall hydrophobic character of the framework, improving its affinity toward nonpolar pharmaceutical pollutants and enhancing its suitability for adsorption in complex or organic-rich matrices. Overall, this study demonstrates that the post-synthetic imine-to-amide transformation mediated by Oxone® provides an efficient route to overcome one of the key limitations of conventional COFs, yielding robust and durable materials with high potential for the selective adsorption of hydrophobic pharmaceutical contaminants in realistic environments.



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From mineral water to E-waste: rare-earth removal by a microporous niobium-iron silicate

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The global transition towards low-carbon technologies has intensified demand for rare-earth elements (REEs), raising concerns over supply security and environmental burdens associated with their extraction [1,2]. Efficient recovery from secondary sources such as electronic waste and industrial effluents has thus become a critical challenge [1,2].

This work investigates the sorption capacity of a microporous niobium-iron silicate, $\text{Na}_{2.9}(\text{Nb}_{1.55}\text{Fe}_{0.45})_2\text{Si}_2\text{O}_{10}\cdot x\text{H}_2\text{O}$ [3], for REE removal from complex aqueous matrices, including spiked mineral water and simulated electronic waste leachates derived from permanent magnets and lamp phosphors. Response Surface Methodology was employed to optimise the combined effects of pH, initial REE concentration, and sorbent dosage in mineral water spiked with nine REEs.

The optimal conditions (pH 4.2, 179 mg/L sorbent dose, and 1.8 $\mu\text{mol/L}$ REEs concentration) achieved >80 % removal of neodymium (Nd) and >60 % of yttrium (Y) after 6 h. In simulated e-waste solutions, the sorbent exhibited high efficiency, with removal exceeding 95 % for Nd and Y at initial concentrations of 5 and 10 $\mu\text{mol/L}$. Kinetic modelling revealed chemisorption as the dominant mechanism, with Nd uptake best fitted by the Elovich model and Y by the pseudo-second-order model, suggesting heterogeneous sorption sites and ion-exchange-driven processes. These results confirm the robustness of NbFe silicate across different matrices, bridging the gap between laboratory-scale optimisation and real-world conditions. The material demonstrates not only high affinity for multiple REEs but also rapid sorption kinetics, making it a promising candidate for integration into wastewater treatment and recycling systems [2,3,4]. These findings establish microporous niobium-iron silicates as promising candidates for future REE recovery technologies, contributing to more resilient and sustainable supply chains.

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Natural tannin-based coagulants as green alternatives for arsenic and antimony removal

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Arsenic (As) and antimony (Sb) are toxic metalloids commonly found in contaminated waters, posing serious risks to human health and the environment. This study investigated the efficiency of a tannin-based coagulant derived from chestnut shell residues (CC) for removing As(V) and Sb(V) from simulated groundwater. Its performance was compared with Tanfloc (TF), a tannin coagulant derived from *Acacia mearnsii*, and with conventional chemical coagulants, $\text{Al}_2(\text{SO}_4)_3$ and FeCl_3 . The influence of coagulation on pH, conductivity, dissolved total organic carbon (TOC), total nitrogen (TN), and turbidity was also examined. Metallic coagulants exhibited the highest removal efficiencies, reaching up to 100% for As(V) and 60-90% for Sb(V), although they caused notable changes in pH and conductivity, particularly the FeCl_3 . Natural coagulants demonstrated lower performance, achieving approximately 15% removal of As(V). For Sb(V), the chestnut shell-derived coagulant maintained removal rates around 15%, while Tanfloc reached nearly 50%, as presented in Figure 1. For turbidity, among the natural coagulants, only the CC was effective, achieving approximately 90% reduction. In contrast, Tanfloc, at a dosage of $12.5 \text{ mg}\cdot\text{L}^{-1}$, increased the turbidity to nearly twice the initial value. Among the chemical coagulants, the aluminum-based coagulant reduced turbidity by about 70%, while the iron-based one achieved only around 20% reduction. The CC coagulant led to an increase in organic matter with rising dosage, whereas the other three coagulants maintained TOC levels close to the initial values. Regarding nitrogen removal, all tested coagulants kept concentrations close to the initial values, which is a positive outcome—particularly for the natural coagulants, since an increase in nitrogen content is commonly reported in the literature for such materials. Despite the superior results of conventional coagulants, the findings highlight the potential of natural, sustainable alternatives as viable options for treating As(V) and Sb(V) contaminated waters.

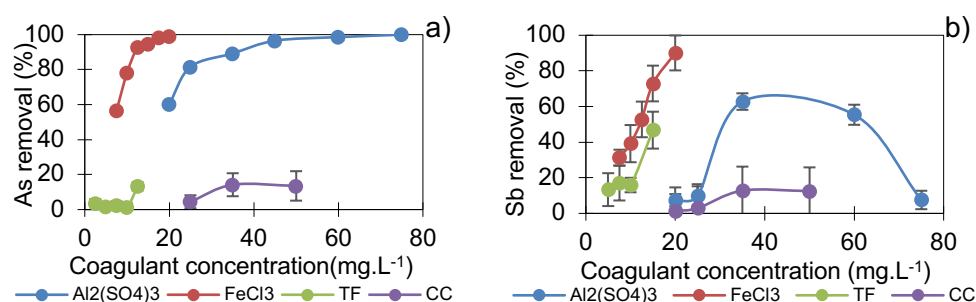


Figure 1. Removal of (a) As(V) and (b) Sb(V) from synthetic groundwater as a function of coagulant dosage.

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CNT-embedded PVP/PVDF membranes for CWPO-intensified filtration of sulfamethoxazole and paracetamol

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Water contamination with pollutants has been recognized as a major issue, posing threats to riparian ecosystems and human health. A potential solution under investigation is the use of catalytic wet peroxide oxidation (CWPO), in wastewater treatment plants, to improve the quality of treated waters [1]. In this work, polymeric composite PVP/PVDF (Polyvinylpyrrolidone/Polyvinylidene fluoride) membranes were prepared with 1.5 wt.% of as-synthesized carbon nanotubes (PVP/PVDF@CNT) and with acid-washed CNTs [2] (PVP/PVDF@CNTW). These membranes were applied in CWPO-intensified filtration using sulfamethoxazole (SMX) and paracetamol (PCM) as model pollutants, using 10 mg L⁻¹ as initial concentration in influent for both SMX and PCM. Continuous-flow experiments were carried out at 80 °C, initial pH 3.5, and with the stoichiometric amount of H₂O₂ for complete oxidation of SMX and PCM. The results (Figure 1) revealed that the best-performing membrane, prepared with as-synthesized CNTs, maintained SMX and PCM degradation levels of up to 80% over 8 h experiments (Figure 1a). Control experiments of filtration-only showed removals below 10% for both pollutants (Figure 1b), confirming that degradation was the main removal pathway in the CWPO-intensified process.

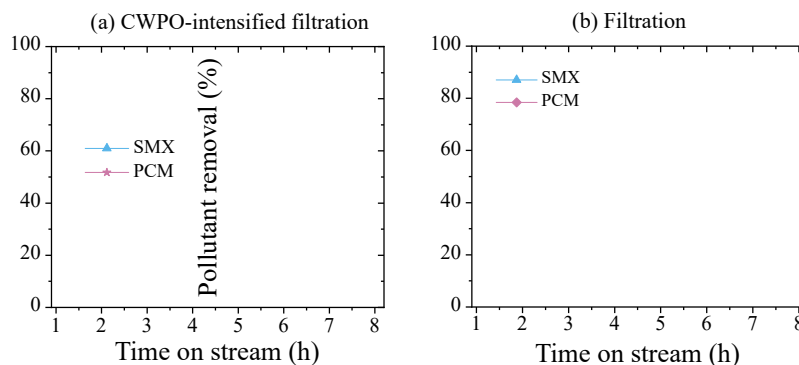


Figure 2 - Removal of SMX and PCM by (a) CWPO-intensified filtration and (b) filtration. $Q = 1 \text{ mL min}^{-1}$, $T = 80 \text{ }^{\circ}\text{C}$, initial $\text{pH} = 3.5$. SMX: $C_{\text{SMX},0} = 10 \text{ mg L}^{-1}$, $C_{\text{H}_2\text{O}_2,0} = 44.3 \text{ mg L}^{-1}$; PCM: $C_{\text{PCM},0} = 10 \text{ mg L}^{-1}$, $C_{\text{H}_2\text{O}_2,0} = 47.4 \text{ mg L}^{-1}$.

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Benign by Design of New Depigmenting Agents

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The benign-by-design (BbD) concept combines degradability, safety and functionality into molecules and process from the earliest design stages, aiming to prevent pollution rather than managing it afterwards [1]. In previous studies, 4-butylresorcinol was identified as a widely used depigmenting agent in anti-aging cosmetic products, but it is considered hazardous due to environmental toxicity [2]. To address this, BbD strategies were set to obtain a new library of resorcinol derivatives with potential tyrosinase inhibitory activity. A series of twelve aminated and imine derivatives were virtually screened for docking affinity against tyrosinase, biodegradability, aquatic toxicity, and ADMET properties, enabling the selection of the most promising candidates for synthesis. Guided by Green Chemistry principles, six derivatives were successfully obtained through a simple, one-pot reductive amination protocol depicted in Figure 1 [3], using green solvents.

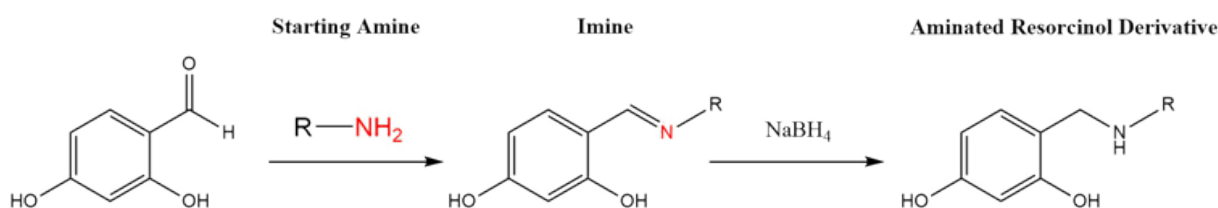


Fig. 1. Reductive amination reaction.

Moreover, the compounds were tested for biological activity, including mushroom tyrosinase inhibition and DPPH radical scavenging assays. The results suggest that introducing amine functionalities can enhance tyrosinase inhibition while improving biodegradability and safety compared to reference agents. Further studies are required to evaluate the *in vitro* biodegradability, aquatic toxicity and skin depigmenting efficacy of the synthesized derivatives.

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Valorisation of Kraft lignin as a sustainable sorbent for mercury removal from water

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Mercury (Hg) is listed by the European Environment Agency as one of the most critical industrial contaminants in Europe. Even at trace levels, it poses severe risks to aquatic ecosystems and public health. In response, recent EU regulations have imposed its complete removal from liquid effluents and long-term stabilization [1]. However, achieving these requirements remains a significant technological challenge. Lignin is a low-value by-product from the pulp and paper industry and 2G ethanol biorefineries, whose valorisation is essential within the circular economy. Its application as a sorbent offers an environmentally friendly, low-cost, and sustainable alternative for water treatment [3].

This work evaluates the potential of Kraft lignin (KL) for the removal of mercury (Hg) from contaminated water. Sorption performance was assessed under different initial Hg concentrations (25–1500 µg/L), in two water matrices of distinct complexity (ultrapure and bottled mineral water), and in competitive conditions with other priority contaminants (As, Cd, Pb). A Box–Behnken experimental design was applied to optimise key operational parameters, namely pH (4–8), sorbent dosage (20–200 mg/L), and initial Hg concentration (50–550 µg/L), using Response Surface Methodology [3].

Sorbent dosage and initial concentration were identified as the main factors influencing Hg removal, whereas pH showed only a minor effect. High removal efficiencies (up to 97 % within 6 h) were achieved at low dosages (10 mg/L) and an initial concentration matching the former EU discharge limit of 50 µg/L Hg, reducing residual levels to ~1 µg/L, close to the drinking water threshold. Under optimized conditions, at ten times higher concentrations (~500 µg/L Hg), the model predicted 98 % removal with only 170 mg/L of Kraft lignin (KL). Sorption kinetics were rapid (92 % in 2 h) and fitted well to pseudo-first order, pseudo-second order, and Elovich models, while SIPS isotherm analysis indicated a maximum sorption capacity of 31,040 µg Hg/g KL. Although efficiency decreased in complex matrices and under competitive conditions, removals of up to 66 % and 54 % were still obtained.

These results highlight KL as a low-cost, widely available, and robust sorbent with strong potential for industrial water treatment. Ongoing work is focused on material immobilization and process scale-up.

Acknowledgments

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ETS-4 as an efficient sorbent for neodymium removal from contaminated waters

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Rare earth elements (REEs) are critical components of modern technologies, with neodymium (Nd) being essential for high-performance permanent magnets used in wind turbines, electric vehicles, and electronics [1]. The escalating global demand for Nd, together with the concerns about its presence in industrial effluents and toxic effects on aquatic organisms [2], emphasizes not only the need to mitigate its environmental impact but also the strategic importance of recovering Nd from secondary sources to ensure a more sustainable supply chain [3]. Sorption has proven to be a cost-effective and environmentally friendly approach for REEs removal. Among different materials, microporous titanosilicates such as ETS-4 stand out due to their high ion-exchange capacity, chemical stability, and tunable “molecular gate” effect, making them promising sorbents for REE remediation and recovery [4]. This study describes the use of ETS-4 titanosilicate for the removal of Nd from water. The sorption tests focused on evaluating the capacity of ETS-4 to remove Nd from different contaminated waters and on optimizing the process by analyzing the effect of sorbent concentration, initial Nd concentration, and water salinity on the removal percentage. A Response Surface Methodology (RSM), consisting of 3 factors at 3 levels (sorbent dose: 20, 250, and 480 mg/L; solution concentration: 1, 25.5, and 50 μM ; salinity: 0, 17.5, and 35 g/L), was employed. From these conditions and the methodology used, it was observed that the optimal conditions for Nd removal, achieving approximately 100 % removal rates, correspond to the highest sorbent concentration, with the remaining parameters adapted to the experimental conditions. It was also noted that the majority of Nd removal occurs within the first hour of contact, as can be seen from the response surfaces in Figure 1. After 1 hour of contact (A) and for higher ETS-4 doses, removal percentages of approximately 95 % can be achieved, with no significant increase in Nd removal when the contact time is extended to 6 (B) and 24 hours (C).

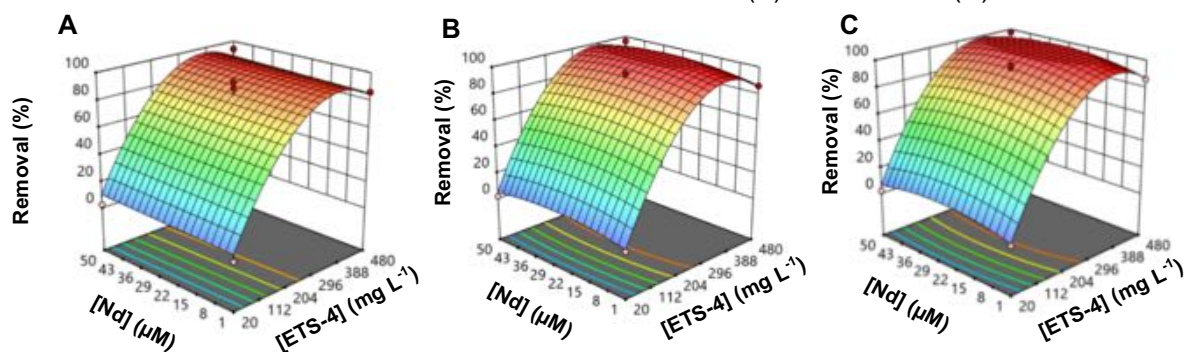


Fig 1. 3-D response surfaces illustrating the interaction between the initial concentration of Nd and the sorbent dose on Nd removal after 1 h (A), 6 h (B), and 24 h (C) of contact time.

Acknowledgments

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Monitoring effluent water quality parameters in two wastewater treatment plants in northeastern Portugal

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Monitoring water quality is key to evaluate the performance of wastewater treatment systems through physical, chemical, and biological parameters [1]. Under the project INOVÁGUA, a partnership was established to monitor effluents from an urban wastewater treatment plant (WWTP) and from a landfill leachate treatment plant (LTP) in northeastern Portugal. Effluent samples were analyzed within 48 hours after collection for twelve key parameters: pH, dissolved oxygen (DO), conductivity, salinity, total dissolved solids (TDS), total organic carbon (TOC), total nitrogen (TN), chemical oxygen demand (COD), 5-day biochemical oxygen demand (BOD₅), turbidity, chlorides, and phenolic compounds. The objective of this monitoring campaign is to evaluate the temporal behavior of effluent quality and to assess potential improvements regarding future installation of new treatment unit operations. **Figure 1** presents pH, DO, and COD values (determined according to *Standard Methods for the Examination of Water and Wastewater* [2]) collected over seven months. The LTP showed lower COD and higher DO values compared to the WWTP. This difference is likely related to the fact that LTP is treated by reverse osmosis, which is associated with a higher efficiency in producing high-quality effluent [3]. WWTP showed stable pH, but higher COD and lower DO.

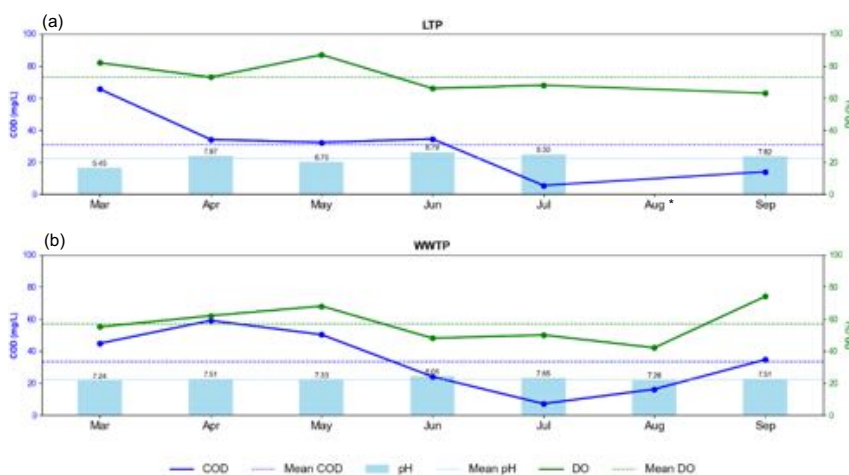


Fig.1. Water quality parameters (pH, COD, and DO) in (a) LTP and (b) WWTP over seven months. *sample not collected.

Acknowledgements

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Synthetic antifouling agents and somatosensory transient receptor potential (TRP) ion channels

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Marine biofouling, characterized by the adhesion of organisms to submerged surfaces, poses a major challenge to the maritime industry. The search for harmless antifouling agents emerges as an alternative to conventional coatings based on toxic metals and biocides.[1] In this context, understanding the mechanism of action (MoA) of novel compounds is essential, since eco-friendly alternatives cannot reach the market without clear evidence of their MoA. Studies suggest that transient receptor potential (TRP) ion channels play a key role in the settlement process of marine invertebrates, being responsible for substrate detection and adhesive secretion by larvae.[2] Notably, marine xanthenes (chrexanthomycins – 4-substituted xanthenes) have recently been identified as inhibitors of the human TRPV1 channel.[3] Moreover, certain xanthenes patented by our research group demonstrated the ability to interfere with the settlement behaviour of marine species.[4, 5] These observations supported the hypothesis that the antifouling properties of our synthetic xanthenes may be linked to their capacity to modulate TRP channels.

The objective of this work was to synthesize 4-hydroxyxanthone and different 4-substituted derivatives as simplified analogues of marine xanthenes and to investigate their structure–activity relationship (SAR) toward TRP channels. The synthesis of 4-hydroxyxanthone was achieved via the diaryl ether route with several optimized steps. Nine 4-substituted derivatives were obtained through reactions with different reagents and subsequently characterized by IR and NMR techniques.

In parallel, a calcium-based fluorescence assay was developed in HEK293T cells expressing human TRPV1 channels to evaluate the activity of two series of compounds: i) our library of antifouling xanthenes and ii) synthesized 4-substituted xanthenes. Four derivatives displayed antagonist profiles on human TRPV1, reducing the capsaicin response. This preliminary assay with human TRPV1 will be the foundation for future assays with mussel TRP channels and the positive correlation of antifouling activity and antagonism of mussel TRP channels can be the final confirmation of the MoA and the first step to change the phenotype-based antifouling discovery to a target-based approach.

Keywords: organic synthesis, TRP, xanthenes, antifouling, fluorescence

Acknowledgments

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Ocorrência e distribuição de contaminantes orgânicos em sedimentos costeiros do norte de Moçambique

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Existe ainda uma lacuna significativa de conhecimento científico sobre a presença e distribuição de contaminantes orgânicos em países africanos, nomeadamente em Moçambique. Este estudo constitui a primeira avaliação sistemática de uma ampla gama de poluentes orgânicos persistentes (POPs) e contaminantes emergentes na região costeira do norte do país, uma área sujeita à pressão de substâncias químicas prioritárias, como pesticidas organofosforados (OPPs), bifenilos policlorados (PCBs) e retardadores de chama organofosforados (OPFRs), entre outros [1,2].

Foram recolhidas amostras de sedimentos em oito praias localizadas na Baía de Pemba e no Porto de Nacala. As análises revelaram concentrações relevantes de ésteres organofosforados (OPEs) e retardadores de chama bromados (BFRs), destacando-se o tricresilfosfato (TCP) como composto predominante (0,075–0,176 µg/g), identificado em todas as oito amostras. O BDE-47 foi detetado em quatro amostras (0,014–0,054 µg/g), juntamente com o TToP3. Relativamente aos POPs, registaram-se níveis baixos, sendo o PCB-153 detetado apenas na praia de Sousa (0,008 µg/g).

Este estudo pioneiro demonstra a presença de contaminantes orgânicos persistentes e emergentes nos sedimentos costeiros do norte de Moçambique, fornecendo uma base de referência essencial para futuras ações de monitorização ambiental e estratégias de mitigação.

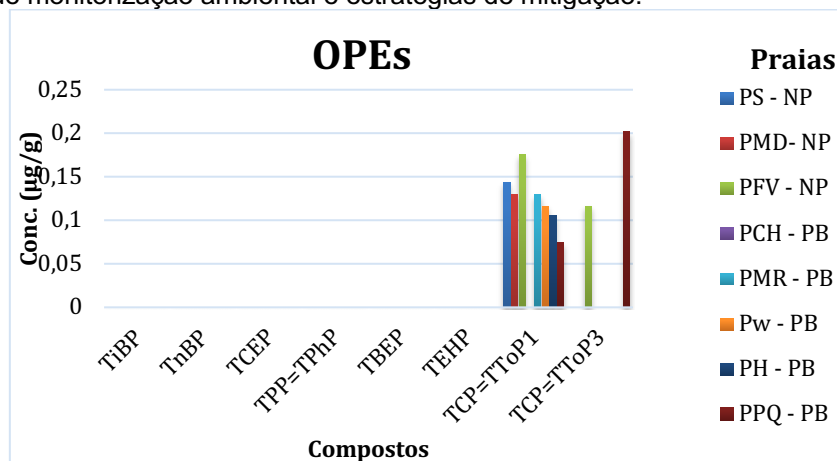


Fig.1: Concentrações médias em (µg/g), OPEs das praias de Pemba e Nacala Porto, em Moçambique.

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Disinfection of aquaculture water by Ga₂O₃/UV-C photocatalysis

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Introduction

The rising global population and increasing food demand have promoted the expansion of recirculating aquaculture systems (RAS) as sustainable alternatives for fish production [1]. These systems, however, rely on intensive water recirculation and thus require strict water quality management. Advanced Oxidation Processes (AOPs) have gained attention as effective water disinfection strategies, since they generate highly reactive species capable of inactivating waterborne pathogens [2]. Among them, photocatalysis stands out as a promising heterogeneous AOP to provide the comprehensive water treatment that RAS demand. In this context, the present study evaluates Ga₂O₃/UV-C photocatalysis and its intensification with peracetic acid (PAA) for the disinfection of *Enterococcus faecalis*, selected as a model microorganism due to its resistance and environmental relevance. Ga₂O₃, an emerging semiconductor with a wide bandgap and high chemical stability, offers great potential as a photocatalyst for UV-driven disinfection processes in aquaculture systems.

A UV-C batch system (PhotolabLED265-0.1er/cb, Apria Systems, Ltd.), operating at 0.5–2.5 W/m² irradiance, was used to treat simulated aquaculture water spiked with *E. faecalis* (10⁶ CFU/mL; ATCC 29212, Scharlab). Ga₂O₃ (Sigma-Aldrich) and peracetic acid (C₂H₄O₃, Panreac) served as photocatalyst and oxidant, respectively, without further purification.

The disinfection performance of Ga₂O₃/UV-C was first assessed under varying catalyst dosages. At an optimal concentration of 0.025 g/L Ga₂O₃, *E. faecalis* inactivation reached detection limit (DL = 10 CFU/mL) within 7.5 minutes of treatment, significantly outperforming UV-C alone. Increasing the Ga₂O₃ dosage beyond this point did not yield further improvements, likely due to light scattering or catalyst agglomeration. To enhance the process, PAA was introduced. The Ga₂O₃/UV-C/PAA system demonstrated synergistic effects. In particular, the combination of 0.025 g/L Ga₂O₃ and 0.05 mM PAA led to complete inactivation of *E. faecalis* in under 2.5 minutes, with all tested improving the Ga₂O₃/UV-C disinfection kinetics. Complementary studies were carried out, highlighting the reusability of the catalyst over at least 4 cycles of operation, the validation of the disinfection potential in real water matrices, the role of ¹O₂ and HO[•] radicals on the disinfection of *E. faecalis*, the robustness of the system in a wide (5–11) pH range and the low toxicity of the studied treatments.

The Ga₂O₃/UV-C and Ga₂O₃/UV-C/PAA systems demonstrated strong potential for advanced water disinfection, enabling rapid and efficient inactivation of *E. faecalis* under mild operating conditions. The PAA-enhanced process achieved significant disinfection with minimal oxidant dosage and short reaction times. Future research will focus on the validation of the process for practical application in continuous configurations, laying the groundwork for future scale-up for industrial aquaculture systems treatment.

Acknowledgements

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Adsorption meets biocatalysis: synergistic biochar-laccase composites for advanced water treatment

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The widespread occurrence of contaminants of emerging concern (CECs) in aquatic environments has become a major challenge for water quality management. Among them, pharmaceutical compounds are especially relevant because of their persistence, biological activity, and potential ecotoxicological effects. In particular, diclofenac (DCF), a widely prescribed non-steroidal anti-inflammatory drug, is one of the most frequently detected pharmaceuticals in surface and wastewaters worldwide. This ubiquity arises both from its extensive consumption and from the limited efficiency of conventional wastewater treatment processes, which are not designed to effectively remove microcontaminants. As highlighted by the recent European Directive 2024/3019/EU on urban wastewater treatment, developing more effective and sustainable treatment solutions is therefore urgent. In response to this challenge, adsorption and enzymatic degradation have emerged as two particularly effective approaches. Therefore, in this work, a synergistic strategy was explored through the development of a biochar-laccase composite material. Specifically, biochar (BC) was produced from spent brewery grains (SBG), a major by-product of the brewing industry, through microwave-assisted pyrolysis (800 °C, 20 min, N₂ atmosphere). The resulting material (SBG-MW-BC) was subsequently functionalized with laccase (Lac) from *Trametes versicolor* via physical adsorption, yielding the composite SBG-MW-BC-Lac. Both materials were extensively characterized to confirm their structural, physicochemical, and catalytic properties. Following this, DCF removal was assessed under batch conditions, through both adsorption and enzymatic degradation, in ultrapure water and buffered aqueous media (pH 5.0 and 7.0). Remaining DCF concentrations were quantified by capillary zone electrophoresis (CZE) and HPLC-UV, while negative-mode electrospray ionization mass spectrometry (ESI-MS) and LC-ESI-MS were employed to identify potential transformation products (TPs). The results showed that SBG-MW-BC achieved up to 94 % removal of DCF, with rapid adsorption kinetics which was best described by the pseudo-second order model. In parallel, free Lac systems confirmed strong catalytic activity, achieving complete DCF degradation within 24 h at 0.1 mg mL⁻¹. Notably, the composite material integrated both mechanisms, progressively removing DCF and reaching near-complete degradation after 120 h at 300 mg L⁻¹, without enzyme leaching. Moreover, MS analyses revealed a major TP at *m/z* 362, structurally related to DCF, underlining the role of advanced analytical tools in elucidating degradation pathways. Overall, the engineered SBG-MW-BC-Lac composite outperformed previously reported literature systems, enabling efficient removal under milder operating conditions and reduced enzyme dosages. These findings confirm its promise as a sustainable and effective candidate for advanced water treatment, particularly for pharmaceutical microcontaminants such as DCF.

Acknowledgements

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Pine bark-derived biocoagulant for sustainable textile wastewater treatment

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The discharge from untreated textile effluent generates large environmental issues owing to its high contents of dyes and toxic chemicals. We require sustainable and cost-effective alternatives to conventional chemical coagulants to minimize the environmental effect from wastewater treatment. The present work endeavored to synthesize and characterize a biocoagulant based on tannins extracted from pine bark, byproduct rich with natural chemicals [1].

The biocoagulant was synthesized by a two-stage process. In the first part, powdered pine bark was submitted to extraction with water at 90 °C with stirring at 250 rpm for 10 min. After filtration, the extracted solution was further optimized by Mannich reaction with formaldehyde and an amine to perform the necessary changes to promote the production of a coagulating agent [2]. The final coagulant was tested with simulated textile wastewater prepared synthetically. The performance was compared with Tanfloc (commercial tannin-based coagulant) and two standard chemical coagulants—aluminum sulfate and ferric chloride. At an optimal dosage of 10 mg/L and pH 5, the pine bark coagulant achieved ~93% color removal, comparable to Tanfloc (95%) and superior to traditional coagulants, which required 3–4 times higher dosages. Turbidity reductions of 60–70% and conductivity reductions of 15–25% were observed across all tested coagulants, with minimal impact on final pH. Total organic carbon (TOC) decreased from 39 mg/L to 30 mg/L after treatment with the pine bark coagulant.

The research opens up prospects for using modified pine bark to act as a green and nonharmful coagulant when it comes to textile wastewater treatment. As a green technology alone, it eliminates excessive reliance on commercial chemicals while it encourages natural resource reuse to provide a valuable solution to industrial effluent management issues.

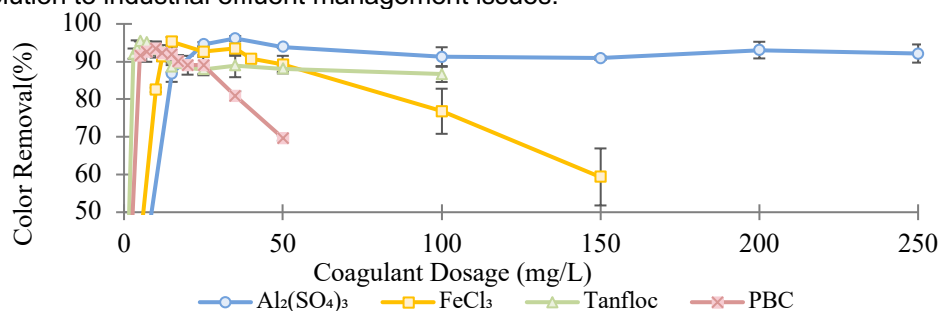


Fig.1. Efficiency of coagulants in removing color (absorbance measured across the entire visible wavelength range) from synthetic effluent as a function of dosage.

Acknowledgements

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Waste-derived nanocatalysts for catalytic ozonation of textile dyes

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Industry, especially the textile sector, plays a crucial role in economic development, but it is also one of the main contributors to water pollution, due to its high water consumption and the generation of large amounts of contaminated effluents [1]. Among organic pollutants, textile dyes, which are widely used and highly resistant to degradation, stand out. The improper discharge of these dyes compromises water quality, posing risks to human health and the integrity of ecosystems, which highlights the need for more efficient and economically viable treatment solutions [2]. Advanced oxidation processes (AOPs) are emerging as a competitive technology for wastewater treatment, with ozonation being one of the most promising approaches for the degradation of organic pollutants. However, several groups of pollutants are difficult to degrade via ozonation alone. Recently, nanomaterials have proven to be excellent nanocatalysts in ozonation, promoting the decomposition of ozone and the formation of highly reactive free radicals, which are responsible for increasing the degradation and mineralization of pollutants [3].

In this sense, a copper(II)-doped nanocatalyst was synthesized via hydrothermal synthesis, using corn straw and polyethylene glycol as carbon precursors and passivation agents. The objective of this work was to evaluate the application of this nanocatalyst in ozone-based AOPs for the degradation of different textile dyes (Methyl Orange, Orange II sodium salt, Reactive Black 5, and Remazol Brilliant Blue R). The use of corn straw in the synthesis of nanocatalysts not only valorizes abundant agricultural waste and promotes the circular economy but also contributes to the sustainability of the process without compromising catalytic efficiency.

The synthesized nanocatalyst demonstrated high efficiency in ozonation, promoting an enhancement of approximately 200% in the degradation rate constant of textile dyes, allowing complete degradation of the dyes in considerably shorter average times than the conventional process. In the case of methyl orange dye, simple ozonation took 23 minutes (36.8 Wh), while the catalytic route promoted complete degradation of the dye in just 8 minutes (12.8 Wh), corresponding to a reduction of about 65% in energy consumption. This highlights the positive impact of the nanocatalyst on the efficiency and energy sustainability of the treatment. Oxidation-reduction potential (ORP) measurements showed a significant increase in ORP values in the presence of the nanocatalyst, indicating intensified generation of reactive oxidative species during ozonation and directly correlating with enhanced dye degradation and discoloration. Recycling tests demonstrated that the synthesized nanocatalyst maintained high catalytic efficiency after 7 cycles of use, confirming its stability and potential for practical applications.

Acknowledgements

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Determinación del perfil de hidrocarburos ligeros (C₁-C₇) en muestras de gasolina por cromatografía de gases-ionización de flama

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El objetivo de este trabajo fue desarrollar un método analítico para la determinación del perfil de hidrocarburos ligeros en muestras de gasolina de alto octanaje. Las condiciones cromatográficas se basaron en la recomendación ASTM D2163-23^{e1} y se modificaron algunos parámetros instrumentales y operativos para disminuir la complejidad metodológica. Las muestras de gasolina se diluyeron con metanol y se analizó la fase de vapor de cada disolución. Los resultados muestran que la selectividad y la resolución cromatográfica permiten identificar a los hidrocarburos ligeros (metano, etano, propano, n-butano, n-pentano, n-hexano y n-heptano), los isómeros (isobutano, isopentano) y algunos compuestos insaturados (eteno, propileno, trans-2-buteno, cis-2-buteno, isobuteno, 1-buteno y 1,3-butadieno) en un tiempo total de análisis de 12 min. La metodología desarrollada es una alternativa analítica rápida y sencilla para determinar el perfil de hidrocarburos ligeros en el vapor de las gasolinas, utilizando un cromatógrafo de gases convencional y sin dispositivos neumáticos para la inyección de las muestras. La aportación de este desarrollo tiene implicaciones analíticas, comerciales y ambientales, porque elucida de manera precisa la composición de la fracción ligera de los hidrocarburos de un combustible. De manera adicional, este estudio corrobora que los hidrocarburos ligeros como el propano, isobutano y n-butano forman parte de los compuestos orgánicos volátiles (COVs) que se emiten a la atmósfera por la evaporación de las gasolinas. Por lo anterior, esta propuesta analítica es una herramienta que puede contribuir en el estudio de los hidrocarburos ligeros en muestras de aire ambiental.

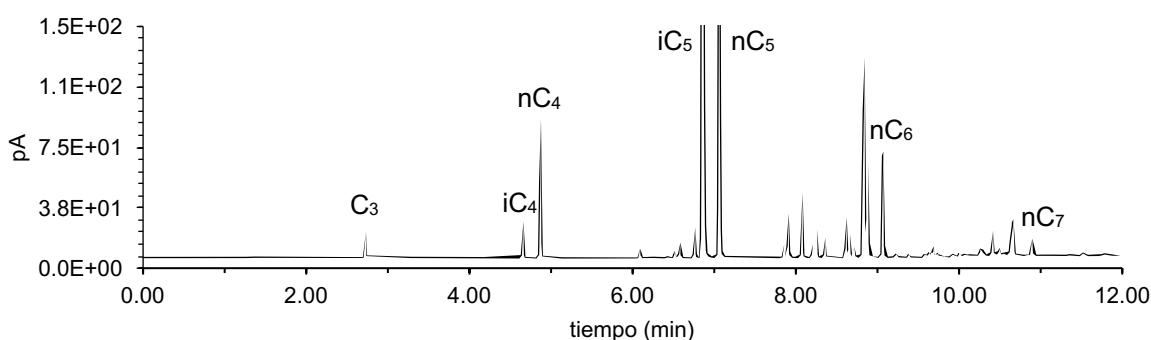


Fig.1. Cromatograma por GC-FID del análisis de la fase de vapor de una muestra de gasolina. Identificación de propano (C₃), isobutano (iC₄), n-butano (nC₄), isopentano (iC₅), n-pentano (nC₅), n-hexano (nC₆) y n-heptano (nC₇).

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Herramienta para sostenibilidad, el Análisis de Ciclo de Vida

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El Análisis de Ciclo de Vida (ACV) se utiliza para cuantificar e identificar el impacto ambiental de un producto durante toda su vida, desde el origen, que es la extracción de materias primas, el proceso de producción, el reciclado y el final, que son los residuos. Se pretende detectar puntos críticos como emisiones, residuos y consumo para implementar mejoras y proceder a buscar alternativas más sostenibles.

Son destacables las fases del ACN porque es un proceso complejo que necesita técnicos especializados, se aborda en cuatro etapas fundamentales guiadas por normas internacionales ISO 14040 e ISO 14044:

1. Definición de Objetivos y Alcance: Se establecen claramente los motivos del estudio y se delimita qué etapas del ciclo de vida se incluirán en el análisis.
2. Inventario del Ciclo de Vida (ICV): Se recopilan y cuantifican todas las entradas (consumo de recursos) y salidas (emisiones al aire, suelo y agua, y residuos) que podrían causar un impacto.
3. Evaluación de Impactos: Se relacionan las entradas y salidas del inventario con posibles efectos ambientales (salud, ecosistemas), se clasifican en categorías de impacto y se evalúa su importancia.
4. Interpretación de Resultados: Es la fase crítica, donde se identifican las fases con las mayores cargas ambientales. Se extraen conclusiones y recomendaciones para la toma de decisiones, por ejemplo, comparando productos para determinar cuál tiene un mejor desempeño ambiental.

Finalmente, la aplicación del ACV es esencial para la Economía Circular y la sostenibilidad, porque permite calcular el perfil ambiental de productos y servicios. Sus aplicaciones incluyen mejorar el diseño, establecer prioridades estratégicas, y facilitar el acceso a ecoetiquetas o Declaraciones Ambientales de Producto (DAP), mejorando el posicionamiento en el mercado.

Debido a su complejidad, la Organización Internacional de Normalización (ISO) ha desarrollado normas como la ISO 14040 [1] y ISO 14044 [2] para guiar su realización. Además, el manejo de datos y cálculos se facilita enormemente mediante software especializado basado en la metodología ACV, que incluye bases de datos ambientales para configurar el inventario y obtener resultados precisos.

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How to measure enthalpy of extraction by isothermal titration calorimetry

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The growing demand for metals in modern lifestyles and low-carbon technologies requires the development of more efficient and sustainable extraction methods. Solvent extraction (SX) is widely used for metal recovery through selective ion partitioning between immiscible phases. The enthalpy of extraction, which quantifies the energetic balance between these partitions, is often the focus of attention in attempts to enhance separation efficiency [1]. Even in scenarios where enthalpy of extraction is not the primary driving force, the accurate determination of this property is crucial for correctly determining entropic contribution to the metal partitioning at different temperatures.

Calorimetric techniques are the techniques to measure heat. Early studies employed isoperibol calorimeters, but required complex methodologies with multiple experimental steps [2]. In 1982, a pioneering approach enabled direct two-phase measurements [3], which subsequently evolved into the actual methodologies employed with isothermal titration calorimetry (ITC) [1,4]. These approaches were nevertheless developed to study enthalpy of extraction of a metal from an aqueous phase to an organic phase being constituted by a neat extractant or, more commonly, an extractant diluted in an “inert” solvent (e.g. dodecane, toluene). Nowadays, hydrophobic eutectic solvents (HES) present a promising alternative to conventional organic phases in SX, eliminating the need for diluents and mitigating third-phase formation [5].

In this study, we have built upon previous calorimetric methodologies to investigate the biphasic transfer of trisnitrato complexes of lanthanides ($M(NO_3)_3$) using tri-*n*-octylphosphine oxide (TOPO) as the extractant, incorporated within a hydrophobic eutectic solvent (HES) with decanoic acid as a phase former, using ITC. The experimental setup was optimized to ensure an efficient phase contact to attain a reaction equilibrium, without requiring extensive energies for phase agitation. Experiments involving the titration of aqueous metal solution of different concentrations, as well as, different aqueous to organic ratios were performed, leading to consistent results. Additionally, a performance test (water + propanol mixing) was conducted to confirm the calorimeter's sensitivity within the experiment's heat range, ensuring the absence of potential deviations due to the low thermal powers involved (as noticed by Lars Wadso [6]).

Overall, this study provides an accurate methodology to give valuable insights into the enthalpic contributions that drive metal ion partitioning and therefore on the extraction mechanisms of metal complexes in eutectic solvents. Furthermore, by advancing calorimetric methodologies for SX analysis, this work aims to support a robust framework for designing efficient and sustainable metal recovery processes with minimal waste generation.

Acknowledgments

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Rastreabilidade de dispositivos médicos de medição usando filtros líquidos de absorbância óptica

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A absorbância óptica é uma mensuranda utilizada em muitos dispositivos médicos de medição (DMM). De acordo com a legislação europeia [1] e os documentos normativos internacionais [2], [3], os DMM têm de ser calibrados ou, no mínimo, verificados. Todavia, observa-se uma falta de rastreabilidade metrológica devido a diferença de resultados de medição e de fabrico dos padrões utilizados para as calibrações ou verificações desses DMM. Ora, por um lado, a absorbância óptica nos DMM é medida em microvolumes, de acordo com as normas de qualidade farmacêutica, o que incentiva a utilizar preferencialmente, como padrões, filtros líquidos de absorbância óptica (FLAO). Além disso, alguns DMM devem ser calibrados ou verificados em comprimentos de onda específicos, devido à sua construção. Assim, um projeto europeu, 23RPT02 ETraceAbs [4], entre diferentes Institutos Nacionais de Metrologia começou em junho de 2024, para atualizar as capacidades de medição dessas instituições, através do fornecimento de FLAO para garantir a sua rastreabilidade ao Sistema Internacional de unidades.

Esta comunicação apresenta os objetivos concretos do projeto 23RPT02 ETraceAbs e o estado de avanço do mesmo. Assim, pretende-se garantir a rastreabilidade dos resultados de medição de absorbância óptica de filtros líquidos, entre 220 nm e 780 nm, para absorbâncias entre 0.001 e 3.000 (o que corresponde a um fator de transmissão entre 99.77 % e 0.1 %, respetivamente), na pressão atmosférica, temperaturas entre 15 °C e 40 °C, e para incertezas padrão relativas entre 0.3 % to 1.0 %. Critérios físicos e químicos mínimos para escolha dos FLAO foram escolhidos para permitir as diluições. Após um exercício de comparação de medição de absorbância óptica de FLAO entre os laboratórios das instituições europeias de metrologia, um guia de boas práticas de fabrico e utilização dos FLAO estão previstos terminar o projeto. O impacto sobre os laboratórios de espectrofotometrias do Instituto Nacional de Metrologia de Itália, o Istituto Nazionale di Ricerca Metrologica (INRiM) e de Portugal, o Instituto Português da Qualidade (IPQ) é apresentado.

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Smart surfaces based on poly(L-lactic acid)/ionic liquid hybrid materials

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The development and implementation of polymeric materials incorporating ionic liquids (ILs) has grown significantly in various fields, including energy generation and storage, biomedicine, environmental remediation, sensors and actuators [1]. Among the wide range of polymers available, poly(L-lactic acid) (PLLA) stands out as a non-toxic biopolymer with piezoelectric properties, making it particularly attractive for a variety of applications [2]. The inclusion of ILs, defined as molten salts at room temperature, into the polymer matrix allows to develop polymer composites with specific functional properties, such as thermochromic [3]. The ability of thermochromic ILs to change colour with temperature makes them highly suitable for sensing applications [4]. Thermochromic sensors are relevant in a large variety of area, such as to monitor and prevent overheating in battery systems, avoiding additional electronic systems [1].

This study reports the development of PLLA-based matrices incorporating different concentrations of the thermochromic IL bis(1-butyl-3-methylimidazolium) tetrachloronickelate ([Bmim]₂[NiCl₄]) (10, 20, and 40% wt.%) produced by solvent casting. The addition of IL resulted in the formation of porous structures, increased the electrical conductivity, and induced a plasticising effect on the polymer matrix, as evidenced by the decrease in Young's modulus. However, the presence of [Bmim]₂[NiCl₄] did not affect the thermal stability of the composite materials.

The developed materials exhibit a colour change upon heating, displaying a blue colour at 50°C, and the process is reversible. Additionally, inks based on the PLLA/IL solutions can be successfully screen-printed onto different substrates, including polyethylene terephthalate (PET), textiles, wood, and paper (Figure 1), demonstrating the versatility and suitability of the developed ink for a wide range of smart surfaces, from the textile industry to the field of sensors.

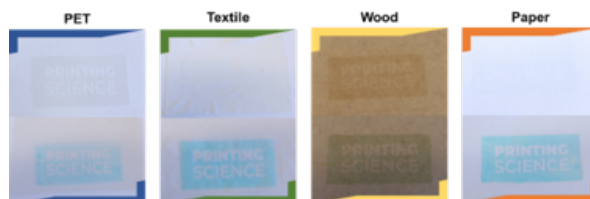


Fig.1. Images of PLLA/[Bmim]₂[NiCl₄] incorporating 40 wt.% IL content screen-printed in different substrates: PET, textile, wood, and paper.

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Alkylating potential of nitrosocompounds obtained from dietary supplements. Risk of DNA mutations

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The aim of this work is to study the nitrosation reaction of amino acids and to evaluate the alkylating potential of their nitrosation products, since the formation of alkylation adducts may pose adverse effects on human health.

L-Cysteine (Cys), L-Serine (Ser), and L-Threonine (Thr) are amino acids present in many foods consumed daily, but their supplementation may be recommended under specific conditions. For instance, Cys has shown benefits in the treatment of anxiety disorders [1], Ser promotes the neurological development and longevity [2], and Thr supports gastrointestinal mucosal integrity [3]. However, potential adverse effects of supplementation must also be taken into account. Indeed, some studies restrict the use of Ser in cancer patients, while others highlight the neurotoxicity of Cys, underscoring the need for further research to establish safe doses and conditions of use.

Amino acids (AAs) ingested orally can react under the acidic conditions of the stomach with nitrite (Nit), which is commonly added to cured and processed meats as a preservative against *Clostridium botulinum*. Their nitrosation products are unstable and may evolve into lactones capable of alkylating biomolecules such as DNA. In this study, 4-(4-nitrobenzyl)pyridine (NBP) was used as a model nucleophile to mimics the reactivity of DNA bases, allowing prediction of the harmful potential of nitrosated products in the body [4]

The kinetics of the nitrosation reaction were studied by UV-Vis spectroscopy, monitoring either the disappearance of nitrite or the formation of nitrosation products, with the aim of determining the experimental rate equation, and the activation parameters via the Eyring equation. To assess the alkylation potential of the nitrosation products, nitrosation mixtures were prepared under acidic pH, and aliquots were withdrawn at different reaction times (t_{nit}) and transferred to a neutral medium containing NBP to initiate the alkylation reaction. After a given time (t_{alk}), 2.4 mL of the mixture was added to the cuvette containing 0.6 mL of triethylamine to stabilize and quantify the final adduct, whose absorbance was measured at its maximum absorption wavelength. Experiments were conducted at 25 °C and 37 °C, and the kinetic behavior of both the formation and stability of alkylation adducts was analyzed.

The following conclusion were drawn: i) The experimental rate equation for the nitrosation of Ser and Thr was: $r = k [\text{AA}][\text{Nit}]^2$; the second-order dependence on nitrite concentration indicates that N_2O_3 is the effective nitrosating agent; ii) Nitrosation products of Ser and Thr were capable of alkylating NBP; iii) In contrast, the nitrosation of Cys followed a more complex mechanism, involving the rapid formation of an initial unstable intermediate that subsequently evolved into a more stable product. The experimental rate equation was $r = k [\text{AA}][\text{Nit}]$; the first-order dependence on nitrite concentration indicates that the effective nitrosating agent are $\text{NO}^+/\text{H}_2\text{NO}_2^+$; iv) The nitrosation products of Cys did not exhibit significant alkylating activity toward NBP. In conclusion, this study highlights the potential risk of generating alkylating agents –species that may contribute to tumorigenesis or cancer– resulting from simultaneous intake of amino acid and foods containing nitrite as preservative.

Acknowledgements

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Metal-driven radical divergence: Cu and Co as directors of redox pathways in bioinorganic models

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Metal-centered radicals play a key role in biological oxidation processes, such as those catalyzed by galactose and catechol oxidases. The metal identity determines both the nature and stability of the radical species. Two families of biomimetic first-row complexes were electrochemically generated from the same bis(aminobisphenolate) ligand (H_2L) but with different metals, Cu and Co, enabling comparison of their divergent redox behavior.

Controlled electrolysis of H_2L using metallic copper as a consumable anode affords a mononuclear $[Cu^{II}(\text{aldehyde})(\text{diaminophenolate})]$ complex derived from oxidative ligand cleavage. Spectroscopic data suggest transient formation of a $Cu(II)$ –phenoxyl radical ($Cu-O\bullet$) analogous to that in galactose oxidase (GO). Outside the protein matrix, this radical is unstable and undergoes disproportionation and oxidative fragmentation, producing aldehydic species and structural rearrangements characteristic of highly reactive $Cu(II)$ – $O\bullet$ intermediates.

Anodic oxidation of cobalt first yields $[Co^{II}(\text{CH}_3\text{CN})(L)]$, which upon exposure to O_2 evolves into a $Co(III)$ species featuring a ligand-centered semiquinone radical stable in solution. The higher redox rigidity of $Co(III)$ stabilizes the organic radical, preventing disproportionation and allowing persistence of the semiquinone state.

This Cu–Co divergence underlines the decisive role of the metal in defining radical fate. $Cu(II)$ promotes metal–ligand redox coupling and rapid radical deactivation [1], whereas $Co(III)$ stabilizes a persistent ligand-centered semiquinone [2]. From a biomimetic viewpoint, this contrast illustrates how different metals can modulate redox activity and radical stability according to their catalytic role.

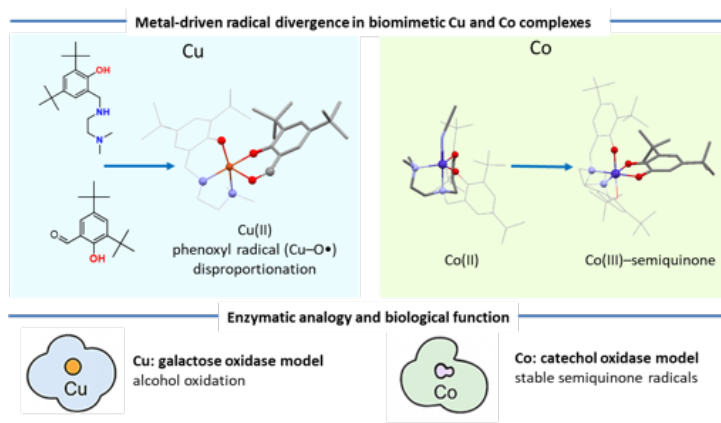


Figure 1. Schematic representation of radical divergence: (a) formation and decomposition of the $Cu-O\bullet$ phenoxyl radical; (b) stabilization of the ligand-centered semiquinone in the $Co(III)$ complex

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Iron(III) helicates with the ability to interact with DNA

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The design of artificial helical compounds derived from metal ions, known as helicates, represents a fundamental objective within the field of Metallosupramolecular Chemistry. Helicates have recently attracted considerable interest due to the intriguing characteristics they display in various fields, including Biology, Pharmacology and Material Chemistry [1]. In this context, a growing body of literature surrounds the use of iron(II) helicates, which can interact with DNA and have anticancer and antimicrobial properties [2].

In consideration of the preceding results, we have decided to extend these studies to iron(III) helicates. Thus, the present work reports the synthesis and characterisation of nine iron(III) helicates derived from a family of *bisthiosemicarbazone* and *bishydrazone* ligands with terminal groups of a different nature.

These neutral $[\text{Fe}_2(\text{L}^n)_2]$ helicates were obtained by an electrochemical methodology (Fig. 1a) and have been fully characterised by a range of techniques in solution and in solid state, including X-ray diffraction studies. The interaction of these helical complexes with DNA was studied using UV-Vis spectroscopy and fluorescence with ethidium bromide (EB, see Fig.1b).

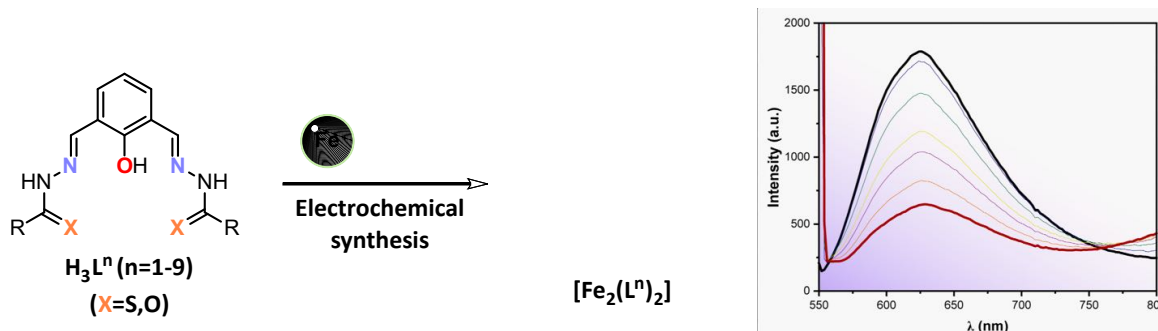


Fig.1. Synthesis of Fe(III) helicates derived from *bisthiosemicarbazone* and *bishydrazone* ligands H_3L^n (a) and Fluorescence spectra of EB bound to ct-DNA in the presence of the Fe(III) complex (b).

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Coordination modes of carboranyl phosphine-iminophosphorane ligands: from the unusual to the unprecedented

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This communication presents a series of new carboranylphosphine-iminophosphorane ligands. These feature a flexible three-atom spacer between the donor atoms (P, N), derived from *dppe* diphosphine. The ligands include examples of C-carboranyl derivatives (ligands **L1-L3**) and B3-carboranyl derivatives (ligands **L4** and **L5**), in order to compare the different effects of these electronegative groups. In addition, an organic analogue (ligand **L6**) was also prepared for comparison purposes[1].

By obtaining their complexes with dichloride palladium(II), it was possible to study the coordination capacity of these ligands. Thus, the comparison of these structures with organic analogues and with carboranyl ligands derived from *dpmm*[2] diphosphine indicates that the length of the spacer between the donor atoms is an important variable for the coordination mode of the ligand, since the coordination force of the nitrogen atom is reduced by the effect of the carborane cage.

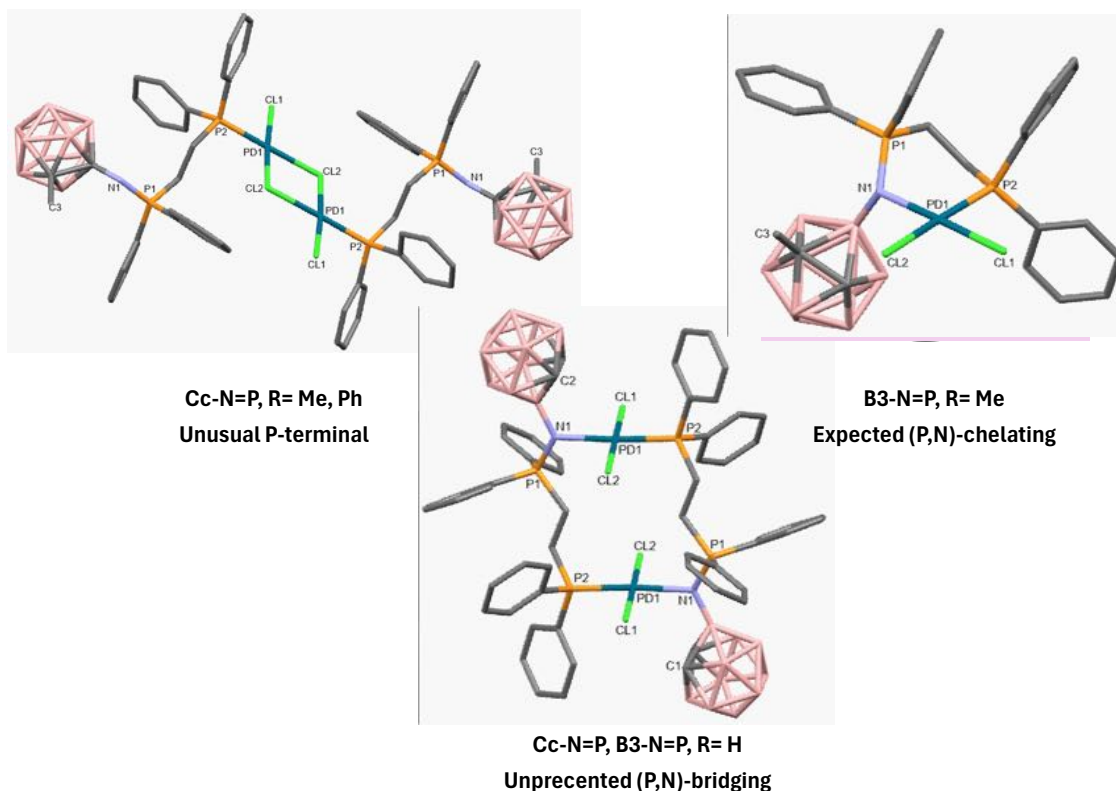


Fig.1. Coordination modes of carboranyl phosphine-iminophosphoranes (3-atom-spacer).

Agradecimientos

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Estudo comparativo das arquitecturas helicoidais dinucleares formadas pola coordinación de ligandos *biscarbamato* con ións metálicos

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As arquitecturas metalosupramoleculares helicoidais, denominadas *helicatos*, adquiriron unha gran relevancia grazas á súa capacidade para interaccionar co ADN. Este feito indica a utilidade destes compostos no recoñecemento celular [1] e outórgalles un notable potencial como candidatos a novos fármacos anticanceríxenos[2] Ademais, os *helicatos* son empregados en ámbitos tan diversos como a catálise e a Química dos Materiais, entre outros.[3-5].

Existen numerosos complexos helicoidais descritos na bibliografía, porén os baseados en ligandos carbamato son moi escasos. Nesta comunicación preséntase un estudo comparativo do comportamento coordinativo de dous ligandos *biscarbamato* con diferente espazador. O obxectivo é analizar como as modificacións no entorno coordinativo de ditos ligandos inflúen na arquitectura helicoidal final obtida.

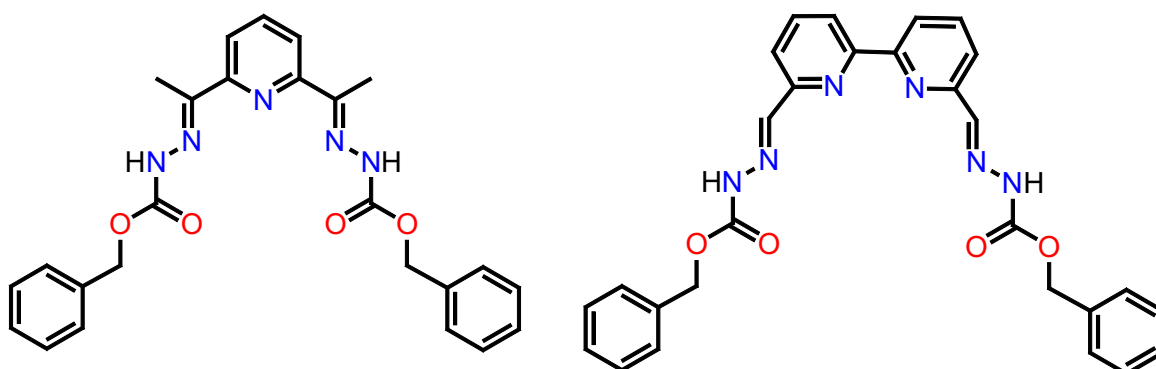


Fig.1. Estrutura dos ligandos *biscarbamatos* H₂L (esquerda) e H₂L¹ (dereita) empregados neste traballo.

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Biological evaluation of Cu(I) phosphino-azine mesocates

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Mesocates are a type of achiral metallocupramolecular architecture formed by the spontaneous interaction of, at least, two metal ions and two organic ligands without crossing each other. [1] Even though this kind of supramolecular architecture have not been extensively studied, could be relevant due to their properties such as magnetic, luminescent, or biological [2]. Both the metal ion nature and the design of the ligands are relevant for the obtention of a particular metallocupramolecular architecture. [3] Particularly, copper complexes are well known for their ability to induce oxidative stress, causing DNA damage. This property joined to copper's versatile coordination chemistry, makes copper complexes attractive as potential antitumoral agents.

In this communication, we have designed a phosphinoazine ligand L [N₂P₂] donor as the precursor of two copper(I) mesocates (Figure 1). The compounds were fully characterized by different characterization techniques such as mass spectrometry, elemental analysis, IR spectroscopy, ¹H and ³¹P NMR and X Ray diffraction. Furthermore, the biological activity of both complexes was evaluated.

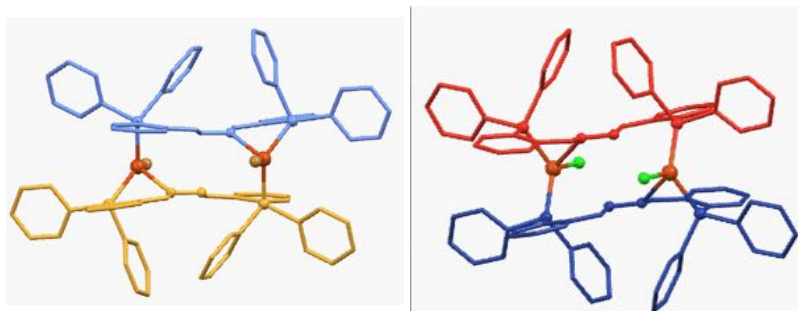


Fig.1. Crystal structure of copper(I) mesocates derived from phosphinoazine ligand

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A mono-silanolate Dy- N_6 macrocyclic complex with SMM behavior

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Molecular magnetism is an increasingly attractive field, primarily focused on enhancing the properties of single-molecule magnets (SMMs). These magnets exhibit quantum nature, making them highly appealing for technological applications as high-density and ultra-fast information storage [1].

Lanthanoid-based SMMs have gained significant attention, especially following the introduction of the Rinehart and Long electrostatic model, which established that the geometry surrounding lanthanoid ions plays a crucial role in determining the anisotropy of the system [2]. For oblate ions, such as Dy³⁺, maximum anisotropy is achieved when the ligands favor axial geometry.

Despite recent advances, several challenges remain. One key challenge is the development of air-stable SMMs with high blocking temperatures (T_B). While significant milestones have been reached, such as the $T_B = 100$ K reported in a dysprosium amide-alkene complex [3], the practical utility of these organometallic magnets is hindered by their instability in air. This limitation highlights the need for further exploration of more stable alternatives. Recent efforts have thus focused on the synthesis of coordination compounds as SMMs with improved properties and enhanced air stability. One strategy to ensure axiality in these complexes is the use of N_6 macrocyclic ligands in the equatorial plane, combined with monodentate anionic oxygen donors in the apical positions [4].

With these considerations in mind, we present the synthesis, structural, and magnetic characterization of the dysprosium complex $\{[Dy(L^{N6prop})(OAc)(OSiPh_3)](BPh_4)\} \cdot 0.5CH_2Cl_2$ (**1**·0.5CH₂Cl₂), shown in Fig. 1. Magnetic alternating current (ac) studies reveal that it is a SMM.

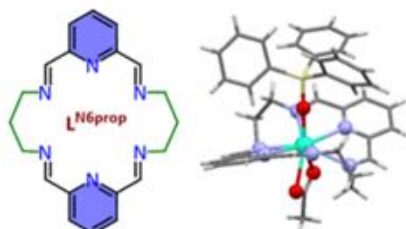


Fig.1. (Left) Schematic representation of the L^{N6prop} macrocyclic ligand; (Right) Balls and sticks diagram of the $[Dy(L^{N6prop})(OAc)(OSiPh_3)]^+$ cation in **1**·0.5CH₂Cl₂.

Acknowledgements

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Hansen solubility parameters of *p*-coumaric acid and *trans*-resveratrol for solubility prediction in binary solvent mixtures

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The experimental determination of solubility is a key step in the rational selection of solvents for extraction and purification processes. Hansen solubility parameters (HSPs) are a powerful tool for predicting solute–solvent affinity [1]; however, their experimental values are not usually available for solid compounds. This work presents the determination of the HSPs of *p*-coumaric acid and *trans*-resveratrol —two polyphenols with antioxidant properties of pharmacological, cosmetic, and food interest [2]— using the semi-empirical method proposed by Hernández & Díaz de los Ríos (2020) [3].

For this purpose, the solubility of both polyphenols was experimentally determined in 21 pure solvents, encompassing different functional groups. The data were used to classify solvents as “good” or “bad”, allowing the calculation of the HSPs through an iterative procedure. Additionally, the solubility of both compounds was determined in several binary solvent mixtures composed of methanol, ethanol, ethyl acetate, and methyl ethyl ketone (MEK). It was observed that *p*-coumaric acid and *trans*-resveratrol exhibited solubility maxima at a specific solvent composition in those binary solvent mixtures composed of solvents with different functional groups. Calculated HSPs were then used to assess whether this synergistic behavior could be predicted theoretically. Eventually, a clear correspondence was found between experimental solubility maxima and minimum distance in the Hansen space.

These results confirm that HSPs are a reliable tool not only for identifying suitable extraction solvents but also for predicting synergistic effects in binary mixtures, which yield solubility enhancement of the studied polyphenols. These insights aim to support more efficient and sustainable solvent selection in extraction processes involving bioactive compounds.

Acknowledgements

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Simulation-based assessment of membrane requirements to improve CO₂ hydrogenation to methanol

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The European Commission has emphasized the urgent need to expand the deployment of renewable energy to reach carbon neutrality by 2050 and reduce carbon dioxide emissions. Within this framework, direct CO₂ hydrogenation has emerged as a promising pathway for converting CO₂ into valuable chemicals and energy carriers such as methanol. Therefore, renewable methanol, derived from captured CO₂ and green hydrogen, holds significant potential as a key driver for global decarbonization and energy transition [1]. Nevertheless, the direct CO₂ hydrogenation route faces thermodynamic and kinetic limitations (thermodynamic conversion < 30 %) and accelerate catalyst deactivation due to water formation. To overcome these challenges, membrane reactors have been proposed as a promising approach; employing a water-permeable membrane enables continuous water removal from the reaction medium, shifting the equilibrium towards product formation while enhancing catalyst lifetime. To achieve high performance in terms of CO₂ conversion and methanol selectivity, membranes with high permeability to water and permselectivity are required [2].

In this work, phenomenological simulations were conducted to determine the key target membrane characteristics – water permeance/permeability and water-over-hydrogen permselectivity – under relevant operating conditions, namely, temperature, pressure, space velocity, and feed H₂/CO₂ molar ratio. The simulations indicated that for typical operating and design conditions, permeance to water should be > 10⁻⁸ mol m⁻² s⁻¹ Pa⁻¹, while steam/other species ideal selectivity should be > 20. These targets were then compared with reported separation performances of membranes used for water/gas and/or water/methanol separations, including polymeric and inorganic types (zeolitic, ceramic, and carbon-based), compiled from the literature. The comparison highlights both the advances achieved and the remaining critical gaps, pointing out where current membranes meet the required targets and where further innovation is needed. Overall, this work provides guidance for membrane material selection and operating strategies, offering insights into the practical potential and limitations of current membranes in membrane reactor technology for CO₂ hydrogenation to methanol.

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Photoactivity of azobenzene derivatives in deep eutectic solvents: correlating phase diagrams and UV-Vis spectroscopy

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Metals play a crucial role in modern society, but the depletion of high-grade mineral reserves, combined with the rapid increase in electronic waste generation, highlights the urgent need for sustainable routes for metal ion separation and recovery [1][2]. Among the alternatives, deep eutectic solvents (DES) stand out for their simple preparation, lower environmental impact, and the dynamic nature of their molecular interactions, which can be exploited in selective separation processes.

In this study, systems of azobenzene and 4-methoxyazobenzene were investigated, exploiting their trans–cis photoisomerization as a tool to probe molecular organization and the cohesive energy of the system [3]. Solid–liquid equilibrium properties were initially predicted using COSMO-RS and then experimentally validated. UV-Vis spectroscopy confirmed the trans → cis conversion in diluted samples of azobenzene and 4-methoxyazobenzene in acetonitrile (15 μ M), highlighting the potential of these photoactive molecules as structural modulators in DES.

The phase diagrams revealed deviations from ideal behavior and well-defined eutectic points. Combined with the observed photochemical response, these results suggest that external stimuli, such as light, can tune the supramolecular organization of DES in real time. Thus, conformational switching between isomers may be exploited in steps such as stripping, enabling controlled metal ion release and paving the way for smart extraction systems with greater selectivity and efficiency.

Future work will include metal extraction assays, the study of the photoresponse of established DES formulations at the same azobenzene concentrations, and the investigation of the effect of dilution on molecular interactions using FTIR spectroscopy, to provide deeper insight into the role of concentration in light-induced behavior.

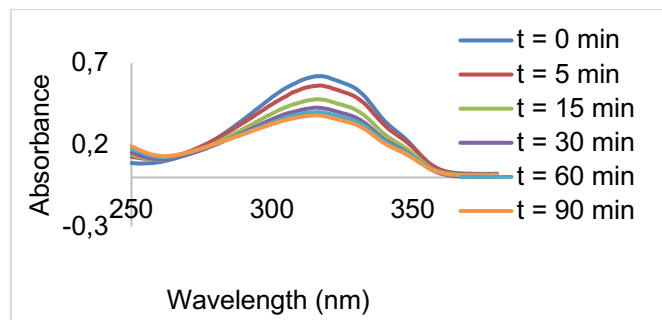


Fig. 1 - UV-Vis spectra at different irradiation times. Variation of azobenzene absorbance in diluted solution under UV irradiation (0–90 min).

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Impact of isomers and conformers on the phase behavior of deep eutectic solvents: an experimental and theoretical approach

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Deep eutectic solvents (DESs) are emerging as sustainable alternatives to conventional organic diluents in metal extraction processes. A key factor for the application of a given DES as the organic phase in solvent extraction (SX) is its liquidus window under ambient conditions, which is determined by the solid–liquid equilibrium (SLE). Whilst the binary intermolecular interactions determining DES formation are relatively well studied, there is less information on how isomerism and nature of conformers for a given DES component can impact its SLE and any resulting SX application. Because comprehensive experimental determination of SLEs for all potential systems is both time-consuming and expensive, theoretical approaches provide an attractive alternative.

In this study, we combined experimental and computational approaches to investigate DESs formed by bipyridines with thymol and carvacrol. Bipyridines are well-known ligands for transition metals^{1,2}, however, their use has traditionally relied on dissolution in organic solvents. When bipyridines are incorporated into DES, they can function simultaneously as solvent and extractant, although several fundamental aspects must be addressed before practical implementation. SLE at different bipyridine molar ratios were determined experimentally by Differential Scanning Calorimetry (DSC), while COSMO-RS was used to predict phase behavior. For this, conformers of all bipyridine isomers were generated with COSMOconf, optimized in TURBOMOLE®, and introduced into COSMOtherm® to estimate the SLE based on the melting enthalpies and temperatures of the pure components. The results highlight the significant influence of both the isomer type and the conformational distribution on the phase diagram. It is recommended to employ the COSMO-RS ensemble for more reliable SLE predictions. The position of the –OH group in phenol molecules significantly influences non-ideality; in this system, carvacrol exhibits stronger deviations from ideality, resulting in a deeper eutectic depression. As a next step, the performance of these DESs will be investigated in the extraction of transition metals.

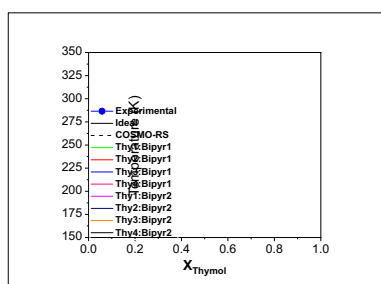


Fig.1. SLE of the thymol–2,2′-bipyridine system, showing experimental data, the ideal curve, and COSMO-RS predictions for individual conformers as well as the Boltzmann-weighted average.

Acknowledgements

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Exploring PEG 400 as an alternative solvent for monoterpene fractionation

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Terpenes and terpenoids are a large and diverse class of natural compounds widely distributed in plants. They play essential ecological roles and are highly valued in the pharmaceutical, cosmetic, and food industries, driving demand for efficient and sustainable separation processes to obtain them in high purity. Conventional separation methods, such as distillation and solvent extraction, are commonly applied but present significant drawbacks, including high energy requirements, the risk of thermal degradation of thermolabile compounds, and reliance on volatile organic solvents, which raise both environmental and safety concerns. In this context, polyglycols such as polyethylene glycol (PEG) have emerged as attractive alternatives due to their low volatility, biodegradability, and cost-effectiveness [1]. This work investigates the use of PEG 400 as an alternative to traditional solvents for the processing of essential oils.

To evaluate its separation potential, the infinite dilution activity coefficients of 20 terpenes in PEG 400 were determined via inverse gas chromatography in the temperature range of (343.15–433.15) K and modelled using the COnductor-like Screening MOdel for Real Solvents (COSMO-RS). The selectivity, capacity, and solvent performance index were used as key parameters to assess the potential of PEG as an entrainer for some challenging terpene separations.

Based on COSMO-RS predictions and experimental separation factors, specific component pairs were chosen for liquid-liquid equilibrium (LLE) measurements. Thus, the LLE of PEG400 with the limonene/linalool and carvacrol/*p*-cymene pairs was measured at 298.2 K. As expected, these studies reveal that PEG 400 has a higher affinity for terpenoids compared to terpenes, which translates into favourable selectivities. PEG 400 resulted in higher solute distribution ratios in the carvacrol/*p*-cymene system, which is typically advantageous for separation. However, its high affinity for both compounds, carvacrol and *p*-cymene, reduces its efficiency. Consequently, further studies should be performed to design the optimal mass separation agent, including the possibility of adding cosolvents to PEG 400.

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Análisis tecno-económico de un diseño de biorrefinería integrada a partir de subproducto de pieles de patata

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La creciente demanda de energía y productos químicos, junto con el agotamiento de los recursos fósiles, ha impulsado la búsqueda de alternativas sostenibles. En este contexto, la biomasa se consolida como un recurso estratégico. Entre los residuos agroindustriales, las pieles de patata (*Solanum tuberosum* L.), ampliamente disponibles y ricas en almidón, celulosa y hemicelulosa, constituyen una materia prima idónea para procesos de biorrefinería [1].

La valorización de este residuo es esencial tanto para mitigar impactos ambientales como para reducir pérdidas económicas en la industria procesadora de patata, dado que se destina a usos de bajo valor (forraje o fertilizantes). Investigaciones recientes han explorado aplicaciones como la producción de biogás o la síntesis de compuestos de valor añadido, entre ellos ácido succínico (AS) y bioetanol [2].

El bio-AS es un químico de plataforma producido mediante fermentación microbiana de azúcares derivados de biomasa vegetal, con aplicaciones como plastificantes, antibióticos o productos químicos industriales de alto valor, como el 1,4-butanodiol [3]. Por otro lado, el bioetanol es un biocombustible biodegradable de alto octanaje, con menores emisiones de CO₂ que los combustibles fósiles y reduce el riesgo de formación de ozono. Su producción a partir de materias primas renovables ricas en carbohidratos, como las pieles de patata, garantiza un balance energético neto positivo sin comprometer el suministro de alimentos ni los recursos humanos.

Este estudio evalúa dos estrategias de valorización de las pieles de patata: (i) la producción únicamente de AS mediante fermentación microbiana (Escenario 1), y (ii) un sistema acoplado que integra la producción de bioetanol, en el que las emisiones de CO₂ de la fermentación del etanol se reutilizan para la síntesis de AS (Escenario 2).

Agradecimientos

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Characterisation of the residence time distribution in an immobilised structured photoreactor for green ammonia synthesis

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In this work, the behaviour of a unitary element of an immobilised mesostructured photoreactor was analysed using Computational Fluid Dynamics (CFD) tools and compared with experimental results. The main purpose of the experimental task was to validate the numerical model, previously developed to predict the hydrodynamics of the system and quantitatively assess its performance in terms of the residence time distribution (RTD). For a newly designed photoreactor, RTD analysis is essential to understand how fluid motion and mixing patterns work and how they influence light utilisation, mass transfer and overall reactor performance.

Simulations using the laminar flow model were performed to track the transient transport of a tracer throughout the cell and compute the RTD function, $E(t)$. To validate the model, step injections of rhodamine (tracer) were performed for the same range of Reynolds numbers (25-500). The monitoring of outlet concentration was done through UV-VIS spectroscopy, which allowed the tracing of the experimental breakthrough curves and further comparison to the numerical model. Despite showing significant agreement for $Re < 100$, the discrepancy between the two RTD curves tended to increase for higher flow rates. Because the experimental response is inevitably influenced by the external dynamics of the pump and tubing system, a deconvolution procedure was applied to remove these experimental artefacts and isolate the intrinsic RTD of the reactor, thus ensuring a fair comparison with CFD simulations. Despite filtering and data regularisation, preliminary results of Figure 1 still show numerical noise due to high-frequency oscillations. However, the main features of the RTD, such as peak position and tail behaviour, are still reproduced in the deconvoluted curve, which is crucial in estimating the mean residence time of the fluid elements.

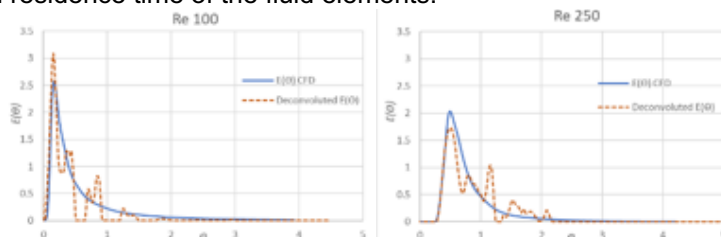


Fig.1. $E(t)$ curves from CFD simulations and from direct deconvolution methods.

To confirm these results, a blind deconvolution approach will be followed: the parameters of a known RTD model are adjusted until the convolution of the modelled curve with the experimental input reproduces the measured output response. [1] Besides serving as a check for the direct deconvolution, a model-based approach will provide a smoother and less noisy estimate of the RTD, making it easier to apply to other operating conditions or reactor configurations.

Agradecimentos

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Effect of crosslinking and dispersing agents on the barrier properties of coatings made from aqueous dispersion of renewable elastomers

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This work presents the development of sustainable water-based food contact barrier coatings for paper. These coatings are formulated using ACTEGA Artística's Mix Aqueous Dispersions (MADs) technology which enables the preparation of high-solids dispersions based on blends of renewable elastomers and copolymers [1].

The dispersions were applied onto paper substrates to form a barrier coating in which high resistances to water and grease are demanded. Formulations contained up to 95% non-fossil components, including natural elastomers, a crosslinking agent (acid ZnO), a dispersing agent (protein), environmentally friendly additives (resin), and fillers. In this way, these formulations perfectly match the current trend of plastic replacement, either by social awareness or by regulations, like the European Union's Single-Use Plastics Directive (SUPD) [2].

In this study the barrier properties of the coatings were specifically investigated at different:

- natural protein-based dispersing agent concentration (3.4-10.3% w/w)
- crosslinking agent formulated as the combined action of zinc oxide (ZnO, 0.5–2.5% w/w) and a weak acid (0.5–7% w/w).

Water resistance was measured using the Cobb 1800 test [2], which involves exposing the coated paper surface to water for 30 min to determine its absorption capacity (g/m²). Grease resistance was evaluated by disposing of olive oil drops on the coated surface and after certain times (0.5-24 h) are removed monitoring both staining and oil drops pass through over time on the coated paper.

The results indicated that high concentration of the dispersing agent decreased the water resistance (Cobb test values: from 40 up to 54 g/m²). On the other hand, grease resistance was improved noticeably (achieved value: higher than 24 h) due to the increased hydrophilicity and viscosity of the dispersions.

The presence of both components of the crosslinking agent was essential to enhance both barrier resistances (Cobb test achieved value: 24 g/m²; grease resistance value: > 24 h). Although ZnO appeared to have the most significant influence on barrier properties, a threshold concentration (> 1 % w/w) was required to achieve effective crosslinking.

These results confirm that the use of dispersing and crosslinking agents and their concentration significantly influence barrier performance. Furthermore, optimal dispersant levels improve surface and dispersion qualities but may compromise water resistance while effective crosslinking depends on reaching a critical ZnO concentration.

Acknowledgments

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Estabilidad física de oleogeles de aceite de colza con HPMC almacenados a temperatura ambiente

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Actualmente, en la industria alimentaria, se aborda la búsqueda de alternativas más saludables a las grasas sólidas, cuya ingesta se asocia a colesterol LDL elevado y enfermedades cardiovasculares [1,2]. En este trabajo, se estudió la estabilidad de aceite de colza gelificado empleando como organogelador hidroxipropilmetilcelulosa (HPMC, 80-120 cP) a partir de una emulsión aceite en agua (50% p/p) mediante el método “*emulsion-template*” con una concentración de HPMC de 2% p/p. El método consiste en la formación de una emulsión que posteriormente se deshidrata y homogeneiza para la formación del oleogel final.

Los oleogeles obtenidos fueron caracterizados físicamente mediante análisis reológico (barrido de deformación, frecuencia y rampas de temperatura), perfil de textura (TPA) y retención de aceite (OBC). Estos ensayos se realizaron en los oleogeles recién producidos y después de 30 días en los que se mantuvieron a temperatura ambiente (20°C) para evaluar la estabilidad física del producto.

Se observa (Figura) que el comportamiento viscoelástico de los oleogeles se mantiene con el tiempo, aunque los módulos elástico (G') y viscoso (G'') aumentan significativamente (a 1 Hz, aproximadamente 70%), indicando una mayor rigidez y compactación de la muestra. El TPA confirma este efecto (Tabla) con un incremento significativo en la dureza, acompañado de una disminución en adhesividad, cohesividad y elasticidad. Finalmente, la retención de aceite (Tabla) mejora de manera significativa con el tiempo debido a la mayor estructuración y rigidez de la matriz [3].

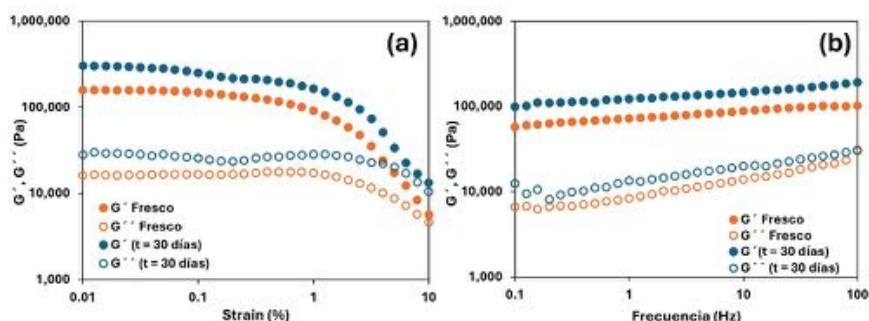


Figura. Análisis reológico de los oleogeles frescos y después de 30 días. (a) barrido de deformación, (b) barrido de frecuencia

Tabla 1. Parámetros del perfil de textura y capacidad de retención de aceite de las muestras.

Días	Dureza (kPa)	Adhesividad (N·s)	Cohesividad (-)	Elasticidad (-)	OBC (%)
0	14.44 ± 1.16	0.73 ± 0.28	0.30 ± 0.03	0.54 ± 0.18	95.98 ± 1.61
30	19.24 ± 1.71	0.54 ± 0.17	0.24 ± 0.04	0.32 ± 0.08	99.23 ± 0.50

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Are machine learning models ready for natural product retrosynthesis?

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Natural products have been used as a foundational source for the development of new therapeutic agents due to their structural diversity and bioactivity [1]. The primary challenge in harnessing natural products for therapeutic applications is their synthetic accessibility, as their molecular complexity limits synthetic efficiency [2]. Machine learning approaches offer promising solutions for retrosynthetic analysis by predicting viable synthetic routes and optimizing reaction pathways for these structurally complex compounds [3]. Furthermore, the identified synthetic routes can be strategically adapted and optimized to access molecules inspired by natural scaffolds.

In this work, we aimed to explore machine learning methodologies to support the design of synthetic pathways to natural products. To fulfil this aim, a curated dataset of 61 natural products with reported total synthesis was assembled. For each natural product, molecular descriptors and physicochemical properties were computed and analyzed to characterize the molecular diversity contained within the dataset. We evaluated the performance of three state-of-the-art retrosynthetic planning tools—NeuralSym, AiZynthFinder, and IBM RXN—in generating plausible synthetic pathways for the identified natural products. AiZynthFinder outperformed the other tools (~40% success rate), while IBM RXN showed moderate performance (~25%), and NeuralSym consistently failed to generate complete pathways for complex natural products.

Their predictions were benchmarked against the published synthetic routes to assess consistency, reliability, and the influence of molecular complexity on algorithmic performance. ML models generated chemically plausible disconnections, occasionally matching published routes or proposing alternative strategies. However, not all AI-generated pathways were chemically feasible, indicating that expert oversight remains essential for retrosynthetic planning.

These results provide valuable insights into the potential of ML-assisted approaches for the design and synthesis of therapeutically relevant compounds, highlighting both current capabilities and limitations in the computational retrosynthesis of complex natural scaffolds.

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Synthesis of aminochalcone derivatives with potential antimitotic activity in multidrug-resistant cancers

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Cancer remains one of the leading causes of mortality worldwide, and the search for new small molecules capable of overcoming therapeutic limitations such as resistance and toxicity is ongoing. In particular, the overexpression of P-glycoprotein (P-gp) in cancer cells is a major factor underlying the reduced efficacy of many anticancer drugs, as it actively expels chemotherapeutics and contributes to multidrug resistance [1]. To address these challenges, chalcones and their derivatives have emerged as attractive candidates due to their simple structures, synthetic versatility, and reported anticancer properties [2]. The anticancer activity of many chalcones is often linked to their ability to bind at the colchicine binding site (CBS), thereby inhibiting tubulin polymerization and disrupting microtubule dynamics. The 3,4,5-trimethoxyphenyl group has often been highlighted for tubulin binding, with derivatives shown to inhibit colchicine interaction at the CBS [3]. Within this context, we identified 2',4'-dihydroxy-3,4,5-trimethoxychalcone (**C345**) as a promising antimitotic agent capable of suppressing human cancer cell growth. Based on this, a molecular hybridization strategy was designed to obtain analogues with both antimitotic and P-gp inhibitory activities. Our approach aimed to generate analogs that integrate the antimitotic activity of **C345** with P-gp inhibitory potential achieved through the incorporation of an amine moiety, a structural feature known to enhance P-gp inhibition. Herein, the total synthesis of a group of aminochalcones is described. These derivatives were prepared via a bimolecular nucleophilic substitution (S_N2) reaction and Claisen–Schmidt condensations. The purity of the new synthesized chalcones was determined by high-performance liquid chromatography (HPLC), and nuclear magnetic resonance (NMR) techniques and high-resolution mass spectrometry (HRMS) allowed their characterization. Assessment of their antiproliferative properties in human cancer cell lines is currently in progress.

Acknowledgments

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Nova metodologia oxidativa em síntese peptídica para diversificação estrutural e funcional da melanostatina

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Os péptidos *N*-hidroxilados têm ganho destaque devido ao seu comportamento conformacional único e às suas propriedades físico-químicas distintas, que influenciam a sua atividade biológica [1]. Apesar disso, a introdução de *N*-hidroxiprolina em cadeias peptídicas continua a ser um desafio sintético, exigindo normalmente várias etapas de proteção e desproteção, as quais reduzem a eficiência e a sustentabilidade do processo. Neste trabalho [2] apresentamos uma nova abordagem para a *N*-hidroxilação da prolina em síntese peptídica, baseada numa oxidação *in situ* mediada por uma reação de eliminação de Cope. A aplicação desta metodologia foi demonstrada na preparação de um derivado *N*-hidroxilado do neuropéptido melanostatina (L-Pro-L-Leu-Gly-NH₂).

A nossa abordagem explora a dupla função do grupo *N*-(cianoetilo), que atua como um grupo protetor durante a síntese peptídica e, subsequentemente, como grupo de saída na etapa oxidativa. Neste trabalho, realizou-se uma reação de acoplamento peptídico entre a *N*-(cianoetil)-L-prolina e o dipéptido previamente sintetizado H-L-Leu-Gly-OMe, originando um intermediário que, após tratamento com o ácido *m*-cloroperbenzóico, sofre uma reação de eliminação de Cope, conduzindo à *N*-hidroxilação no resíduo da prolina (Figura 1).

Este processo oxidativo numa única etapa resultou no tripéptido pretendido com um rendimento de 93%, sem necessidade de etapas adicionais de desproteção. Para além disso, verificou-se que a introdução do grupo *N*-(cianoetilo) numa etapa inicial da síntese conduz a um rendimento superior (78%) comparativamente a derivatizações em fases mais tardias do processo sintético (41%). Em suma, este trabalho apresenta uma estratégia sintética inovadora para a modificação seletiva de péptidos contendo resíduos de prolina, exemplificada pela síntese da *N*-hidroxi-melanostatina. Esta metodologia, baseada numa reação de eliminação de Cope, configura-se como uma ferramenta promissora em síntese peptídica para o desenvolvimento de péptidos bioativos *N*-hidroxilados.

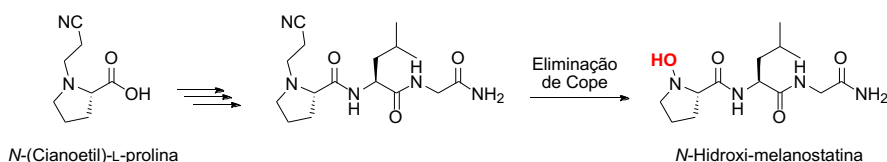


Fig. 1. Síntese da *N*-hidroxi-melanostatina via reação de eliminação de Cope.

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Structural modifications of abietic acid and a computational protocol to predict their compatibility in different environments

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Abietic acid is one of the main constituents of rosin, a natural product resulting from the exudation of pine trees and other conifers. Rosin, and therefore abietic acid, is a raw material that is used industrially in the manufacture of adhesives, paints, varnishes, chewing gum, detergents, among others. Thus, the synthesis of abietic acid derivatives is of paramount importance, both academically and industrially [1]. In this context, this study involved the synthesis of several abietic acid derivatives and the development of a computational protocol to predict how the structural modifications of abietic acid confer compatibility in different environments (Figure 1). We started to identify the most stable conformers of abietic acid derivatives by carrying out a conformational search at the molecular mechanics level of theory using the MMFF94 force field [2] implemented in the MarvinSketch program [3]. From the generated conformers, the lowest energy structures within a defined threshold were further optimized at the Density Functional Theory (DFT) level. DFT-based calculations were then employed to estimate the compatibility of the compounds with solvents of different polarities, namely pentyl ethanoate ($\epsilon = 4.7297$) and n-octane ($\epsilon = 1.9406$). The free energy of solvation was then calculated. Based on the obtained results, a Relative Solubility (RS) parameter was defined as the normalized difference between the free energy of solvation in pentyl ethanoate and n-octane. For a series of synthesized derivatives, the computed RS values are given in Figure 1, which show some correlation with the RF values from experimental TLC results.

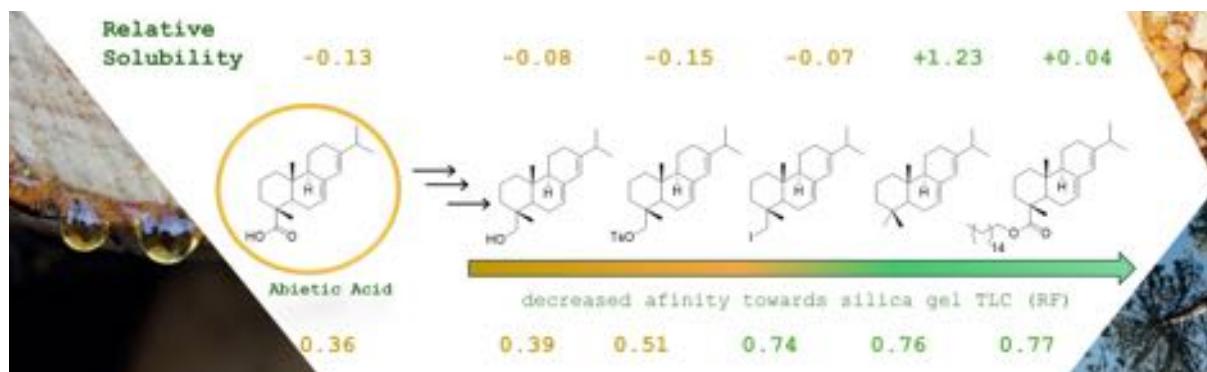


Fig.1. Structural modifications of abietic acid.

Acknowledgments

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Rational synthesis and characterization of a novel pH-responsive pyranoflavylium-starch conjugate from potato sidestreams

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The sustainable valorization of agricultural sidestreams offers viable path towards developing functional biopolymers with advanced properties. In this work, it is reported the rational synthesis and characterization of a novel pH-responsive pyranoflavylium-starch conjugate using starch from potato processing sidestreams. Using a targeted conjugation strategy, pyranoflavylium moieties were covalently bound to modified starch, generating materials with pH-responsiveness features. After proper synthesis and characterization, the Starch-Pyranoflavylium conjugation was evaluated by Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy and the resulting material exhibited predictable and reversible colorimetric responses across physiologically relevant pH ranges, indicating potential utility as a natural pH sensor. Furthermore, the conjugate retained favorable starch-based properties such as biocompatibility and processability. The developed material will be subsequently incorporated into a biomembrane and submitted to pH-responsive color change performance tests using buffer solutions at different pH values using and biogenic amines-enriched model solutions. This work introduces a new class of starch-based smart materials and demonstrates a sustainable approach to upcycle potato sidestreams into high-value functional biopolymers with potential applications in food packaging biosensing and preservation [1,2].

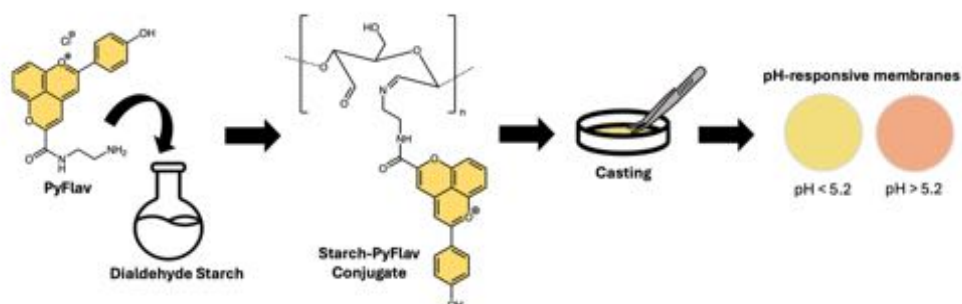


Fig.1. Scheme of the starch-pyflav assembly and evaluation.

Acknowledgments

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Nadifloxacin: enantioseparation and structural modification for further environmental studies

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Fluoroquinolones (FQs) are widely used antibiotics and important emerging pollutants due to their persistence, bioaccumulation, and incomplete removal in wastewater treatment [1]. Despite occurring at low concentrations (ng/L to µg/L), they may have significant consequences due to the potential for accumulation and biomagnification through the food chain, as well as their role in the spread of bacterial antibiotic resistance [2]. Nadifloxacin (NAD) is a chiral FQ marketed as a racemate for acne treatment, with S-NAD being significantly more potent than R-NAD [3]. The prodrug of S-NAD (L- alanine derivative- S-ALANAD) has been developed to improve oral bioavailability against infections caused by gram-positive bacteria, including methicillin-resistant *Staphylococcus aureus* [4]. Despite its relevance, data on enantioselective environmental monitoring and ecotoxicity of NAD are missing.

This work aimed at the enantioseparation of NAD by liquid chromatography under normal-phase in a semipreparative scale, using the CHIRALPAK AD-H DAICEL column (10 mm I.D. x 250 mm L, 5 µm) as well as the synthesis of the prodrug S-ALANAD and its stereoisomers.

The enantiomers of NAD were achieved in high enantiomeric purity, higher than 98%. The stereoisomers of NAD were synthesised through a coupling reaction with (RS)-NAD and the pure enantiomeric forms of Boc-L-alanine and Boc-D-alanine followed by a deprotection reaction to achieve the pairs of diastereomers: S-LANAD/R-LANAD and S-DANAD/S-DANAD. All compounds were characterized by IR and NMR. The CHIRALPAK® AD-H also enabled the separation of both pairs of diastereomers. The enantioseparation of NAD and the synthesis of its derivatives provided access to enantiomerically pure standards for further enantioselective environmental monitoring and ecotoxicological studies.

Understanding the distinct behaviour of each stereoisomer under ecotoxicological conditions bridges synthetic chemistry with environmental science, contributing to sustainable pharmaceutical development and to strategies for mitigating environmental pollution.

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Chemosensors for enantioselective fluorescent detection of amino acids

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The presence of D-amino acids (D-AAs) has been associated with “non-natural” molecules, creating the doubt of the possible presence of this homochirality in organisms [1-3]. Nevertheless, D-AAs have been detected in various biological matrices, including brain tissue, kidneys, liver, blood, urine, cerebrospinal fluid, teeth, and eye lens. Their presence has been associated with several diseases, including neurodegenerative disorders. The D/L AAs enantiomeric ratio has been investigated as a biomarker for early diagnosis, while D-AAs have been pointed for potential applications in targeted therapies, for example therapies modulating D-serine or D-aspartate to fine-tune NMDA receptor activity in Alzheimer's or schizophrenia [2, 3]. Given that, this work describes the synthesis of rhodamine derivatives designed to be linked with a chiral unit, aiming at the development of chiral fluorescent probes for the detection and quantification of D-AA and D/L AA enantiomeric ratios. The synthetic strategy was based on the coupling reactions of rhodamine B with an adequate chiral moiety and an aldehyde derivative for further enantioselective Schiff base formation with amino acids. Another method is also being tested, through the formation of a maleimide using rhodamine 6G as the starting material. The coupling reactions are ongoing, considering different chiral units. NMR and IR spectroscopy confirmed all the synthesized structures. The chiral rhodamine derivatives will be analysed for chemo and enantioselective fluorescent identification of amino acids, and it will be further analysed in a variety of biological matrices.

Acknowledgments

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Green-coloured imidazole-based azo dyes: halochromism and antimicrobial activity

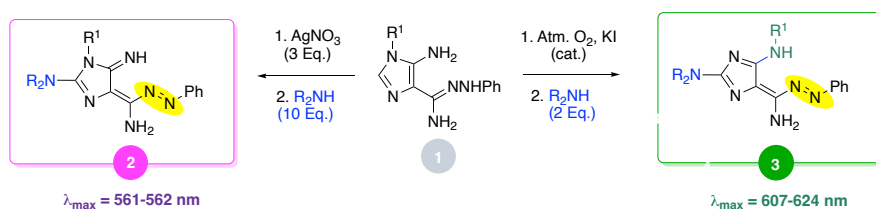
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Halochromic molecules have received significant attention for smart materials development due to their capacity to exhibit a visual colour change when exposed to pH variations stimuli [1]. Aminoazo dyes have been commonly used as pH indicators due to the significant spectral differences between their acid and base forms, but they typically exhibit yellow-red colours [2]. Unusual spectral properties and improved bioactivity have been also achieved by incorporating heterocyclic moieties into azo dyes [3]. As the imidazole scaffold interacts with a wide range of biomolecules, it is one of the most important synthons in medicinal chemistry [4]. Although this scaffold is highly relevant in the design of new imidazole-based antifungal agents, reports on antimicrobial imidazole-based azo dyes remain scarce.

In a previous work, a novel route was developed to obtain unique 2-aminoimidazole azo dyes **2** exhibiting vibrant colours that change from magenta to deep blue as the pH decreases, and potent antimicrobial properties against infectious yeasts (**Scheme 1**) [5]. Here we present a new domino reaction yielding a novel class of green-coloured 2-aminoimidazole azo-dyes (**3**) when an environmentally benign oxidant was used. These new dyes also presented solvatochromism, halochromism with colours changing from green to purple, and potent fungicidal activity against *C. neoformans* [6].



Scheme 1. Synthesis of the halochromic & antimicrobial 2-aminoimidazole azo dyes **2** and **3** from amidrazones **1**.

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Annihilator performance in TTA-UC: donor-acceptor substituted dithienopyrrole derivatives

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Triplet–triplet annihilation upconversion (TTA-UC) has emerged as a promising strategy to convert low-energy photons into higher-energy emission through energy transfer processes. This mechanism relies on a sensitizer-annihilator pair: the sensitizer harvests low-energy photons and undergoes intersystem crossing to its triplet excited state, followed by triplet-triplet energy transfer (TTET) to populate the triplet state of the annihilator. Subsequent triplet-triplet annihilation between two annihilators generates a higher-energy singlet state, which upon radiative decay yields upconverted emission. Such systems hold relevance for applications in photovoltaics, bioimaging and photocatalysis [1-2].

Recent advances have highlighted the challenges of designing efficient annihilators that combine favourable triplet energy levels with good solubility, stability and strong emission properties. Molecular engineering through the introduction of functionalized heterocycles and alkylation, has emerged as powerful strategy to address these limitations, enabling improvements in solubility, intermolecular interactions and TTET efficiency [3-4]. Inspired by these developments, we report herein the synthesis and characterization of novel dithienopyrrole (DTP) derivatives specifically tailored for use in TTA-UC as annihilators. Employing platinum(II) octaethylporphyrin (PtOEP) as sensitizer, we prepared toluene solutions containing the sensitizer-annihilator pair and thoroughly characterized its photophysical properties. Efficient upconversion emission was observed, confirming the role of DTP derivatives as active annihilators in TTA-UC.

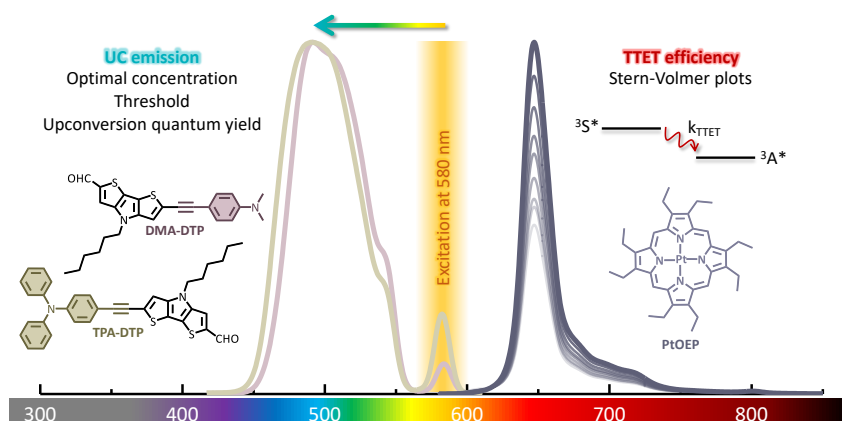


Fig.1. Upconverted emission of the studied TTA-UC pairs and phosphorescence of PtOEP upon additions of different concentrations of annihilator.

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Photodynamic inactivation of *Staphylococcus aureus* and *Escherichia coli* through light-activated boronic acid-based porphyrin derivatives

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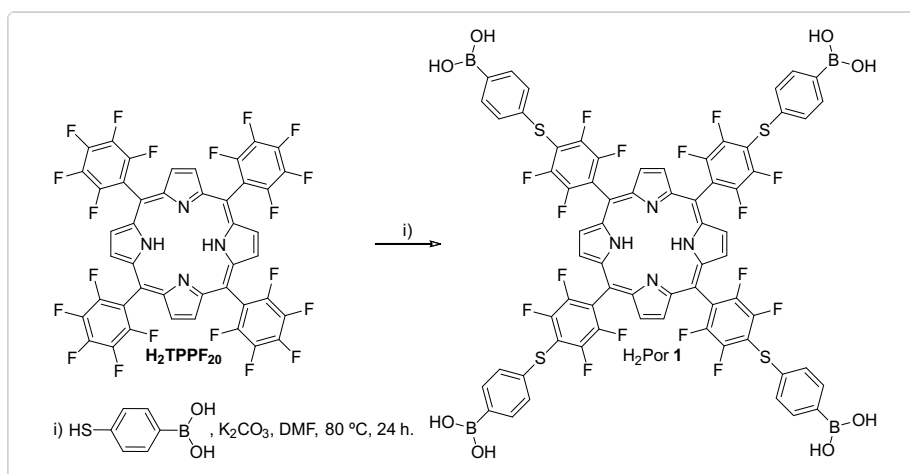
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Photodynamic inactivation (PDI) is a promising strategy against resistant pathogens [1]. Porphyrin (Por) derivatives generate reactive oxygen species (ROS) and act as efficient PDI agents, while boronic acid groups enhance antimicrobial activity through reversible binding to bacterial surface glycans [2]. Here, we report a novel free-base porphyrins substituted with 4-mercaptophenylboronic acid units (H₂Por **1**) – Scheme 1. The antimicrobial activity of H₂Por **1** was evaluated without and with KI addition against *Staphylococcus aureus* and *Escherichia coli* at 5.0 and 10.0 μM under white light (0–360 J cm⁻²). The results obtained demonstrate that H₂Por **1**, especially with KI [3], is a potent antimicrobial PS at low concentration and light dose, highlighting its potential for clinical application.



Scheme 1. Synthesis of H₂Por **1**.

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Tyrosine dehydropeptides as bioinspired candidates for hemorrhage control

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Uncontrolled hemorrhage remains a major clinical challenge, and bioadhesives have emerged as promising alternatives to conventional hemostats and sutures [1]. Inspired by natural adhesive proteins from mussels, which rely on residues such as L-DOPA and tyrosine to form strong interfacial interactions, peptide-based systems offer a versatile platform for wound sealing and tissue repair [2,3]. However, peptide hydrogel bioadhesives often suffer from non-uniform cross-linking and low mechanical adhesion [4,5]. In this work, we designed and synthesized new tyrosine dehydropeptides as bioadhesive candidates. These molecules were evaluated for their ability to undergo hydrogelation and to form cohesive networks with improved interfacial adhesion. Mechanical performance was assessed using single lap-shear tests, while structural and morphological features were characterized to elucidate the relationship between sequence design and adhesive behavior. Preliminary results demonstrate that tyrosine dehydropeptides form stable hydrogels with strong adhesive properties, supporting their potential as next-generation bioadhesives for hemorrhage control. (Figure 1).

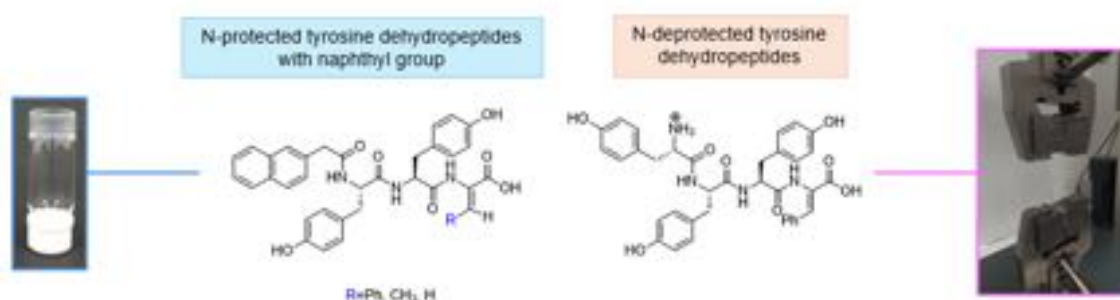


Fig.1. New bioadhesives based on tyrosine dehydropeptides.

This study highlights dehydropeptides as a new class of minimalistic yet powerful bioinspired building blocks, with strong potential to become the next generation of clinically useful peptide-based adhesives.

Acknowledgements

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Functional NaCMC solid polymer electrolytes doped with LiTFSI and glycerol for sensor-integrated solid-state batteries

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The transition to renewable energy systems and high-energy-density applications requires safer and more efficient storage technologies [1–3]. In this context, solid polymer electrolytes (SPEs) have emerged as promising alternatives to conventional liquid electrolytes, offering enhanced stability, compatibility with alkali metal anodes, and potential for integration with sensors for *in situ* and *in operando* monitoring in battery systems [4–7].

Sodium carboxymethyl cellulose (NaCMC)-based membranes doped with lithium bis(trifluoromethanesulfonyl)imide (LiTFSI) and plasticised with glycerol were synthesised, varying dopant (10–200 wt% LiTFSI) and plasticizer mass ratios (NaCMC:glycerol = 1:0–1:2) to optimise performance. Membranes were characterized by ultraviolet-visible spectroscopy, attenuated total reflectance Fourier transform infrared spectroscopy, electrochemical impedance spectroscopy, X-ray diffraction, scanning electron microscopy, thermogravimetric analysis, tensile testing, and contact angle measurements. The highest ionic conductivity (4.40×10^{-4} S/cm at 25 °C) was obtained for 150% LiTFSI with a 1:2 NaCMC:glycerol ratio, while the 1:1 formulation with 50% LiTFSI showed the best balance between conductivity (2.62×10^{-5} S/cm at 25 °C) and mechanical strength ($F_{\max} = 3.0 \pm 1.4$ N; $\epsilon_b = 58 \pm 15\%$). All membranes were thermally stable up to 160 °C. Preliminary loop-antenna tests confirmed the feasibility of integrating SPEs into embedded sensors in solid-state batteries.

These results advance the development of natural polymer-based functional materials for safer, more sustainable, and intelligent energy storage systems.

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Acid-induced delamination as a strategy for recycling PE/PET films

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Across the European Union, approximately 3 million tonnes of multilayer plastic packaging (MPP) are generated annually, accounting for ca. 17 % of all plastic packaging [1]. This volume highlights the pivotal role of MPP in modern supply chains, where barrier and protection performance are crucial for extending product shelf life. To achieve these functions, MPP structures typically consist of layers from different classes of materials, including polyolefins (polyethylene, PE; polypropylene, PP), polyesters (PET), paper, and aluminium foil, often bonded using adhesives and complemented by functional coatings. However, this heterogeneous architecture is poorly compatible with established sorting and reprocessing schemes, thus incineration and landfill remain the prevailing end-of-life routes [2].

To address these challenges, alternative recycling routes such as pyrolysis, gasification, depolymerisation and dissolution–precipitation have been explored. Within this context, delamination emerges as a promising pathway for laminated packaging, as it directly targets adhesive or interfacial layers. It may occur physically, through the dissolution of the adhesive or an internal layer, or chemically, via degradation, enabling the separation and recovery of valuable materials (e.g., PE, PET, aluminium) without compromising their chemical integrity [3].

In this study, acid-induced delamination of PE/PET films was investigated using selected inorganic and organic acids under controlled concentration, temperature, agitation and time. Concentration and temperature were varied to assess their influence on delamination efficiency. Delamination yields ranging from 88 % to 98 % were obtained for both acid types, with organic acids demonstrating excellent performance even at lower concentrations, underscoring their potential for more sustainable applications. Fourier Transform Infrared (FTIR) analyses confirmed the preservation of the chemical integrity of the recovered layers. Future work will focus on evaluating the mechanical properties and processability of the recovered polymers, optimisation of operational parameters, and exploring the reuse of organic acids. Together, these steps aim to advance the scalability of delamination processes, contributing to circular economy strategies, and reducing the reliance on fossil-based resources.

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Non-isocyanate polyurethanes (NIPUs) from tannic acid and sucrose

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Polyurethanes are among the most widely used types of polymers worldwide, and their market has not only been growing in recent years but is also expected to continue expanding in the coming ones [1,2]. This growth is driven by the increasing demand for their applications, such as rigid and flexible foams, coatings, and adhesives [1,2].

Currently, the production of polyurethanes is achieved through the reaction between polyols and isocyanates, which pose serious threat to the environment [3,4]. While polyols are mainly derived from fossil fuels, isocyanates are extremely toxic and polluting, as are their precursors, aniline and phosgene. Hence, a greener synthesis pathway is urgently necessary. The production of Non-Isocyanate Polyurethanes (NIPUs) has emerged as a promising alternative. One of the most promising synthesis routes involves the reaction between poly(cyclic carbonate)s and polyamines [3].

In this work, a reaction mechanism for the most promising route to obtain NIPUs is proposed, supported by Fourier Transform Infrared and Nuclear Magnetic Resonance analyses. Two different raw materials were tested as NIPUs precursors: tannic acid and sucrose.

Acknowledgments

This work was developed within the scope of the project CICECO-Aveiro Institute of Materials, UIDB/50011/2020, UIDP/50011/2020 & LA/P/0006/2020, financed by national funds through the FCT/MCTES (PIDDAC); and : LEPABE, UIDB/00511/2020 and UIDP/00511/2020, ALICE, LA/P/0045/2020; and CIMO, UIDB/00690/2020 and UIDP/00690/2020; and SusTEC, LA/P/0007/2020. Thanks are also due to FCT/MCTES for the financial support to CERES (UIDB/00102/2020). This work was carried out as part of Agenda “H2Driven Green Agenda”, nr. C644923817-00000037, investment project nr. 50, financed by the Recovery and Resilience Plan (PRR) and by European Union - NextGeneration EU.

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Inducción de propiedades barrera en films de PVA mediante la adición de lignina

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El desperdicio alimentario constituye un desafío económico, social y ambiental de gran magnitud. En este contexto, el desarrollo de envases capaces de prolongar la vida útil de los alimentos representa un reto clave para la ciencia y la tecnología de materiales. El alcohol polivinílico (PVA) es un polímero atractivo por su transparencia, flexibilidad y carácter biodegradable; sin embargo, su limitada capacidad de protección frente a la radiación UV y los procesos de oxidación restringe su aplicación directa.

La lignina kraft (KL), un subproducto abundante de la industria papelera, presenta propiedades antioxidantes y de bloqueo UV que la convierten en un aditivo natural de gran interés para mejorar el desempeño del PVA (Fig. 1a) [1,2]. En este trabajo se incorporó KL, en forma directa y nanoparticulada (KL NP) (Fig. 1b), en películas de PVA (Fig. 1c). Los análisis FTIR y TG confirmaron interacciones entre PVA y lignina, más evidentes en las formulaciones con KL NP. Las pruebas ópticas mostraron excelente capacidad de bloqueo UV, mientras que los ensayos TBARS en carne de cerdo (Fig. 1d) demostraron que los recubrimientos redujeron la oxidación lipídica respecto al control sin recubrir, con un efecto ligeramente superior en las películas con KL NP. En conjunto, los resultados muestran que la lignina kraft es un aditivo natural prometedor para películas de PVA, con propiedades funcionales que potencian su aplicación en envases activos y sostenibles.

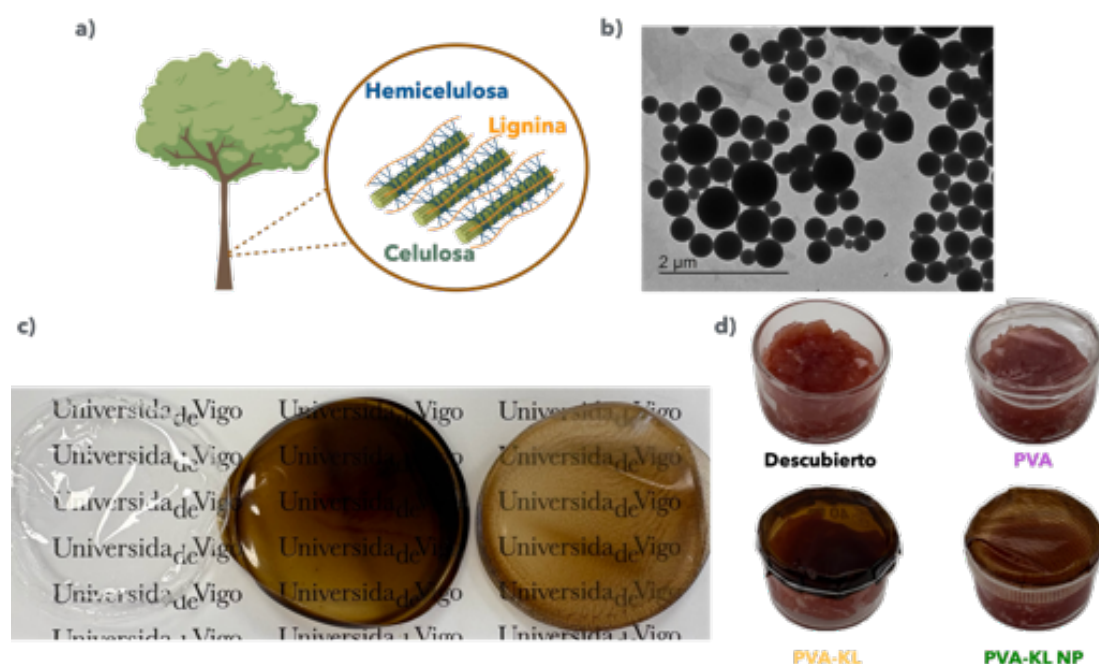


Fig.1. (a) Esquema de la pared celular vegetal con distribución de celulosa, hemicelulosa y lignina; (b) Imagen TEM de nanopartículas de lignina; (c) Películas de PVA, PVA-KL y PVA-KL NP; (d) Muestras almacenadas para el ensayo TBARS.

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Discovery of potent bicyclic allosteric modulators of the dopamine D₂ receptors for application in Parkinson's therapy

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Melanostatin (MIF-1) is a naturally occurring neuropeptide with the sequence Pro-Leu-Gly-NH₂, known for its ability to enhance dopamine signaling by acting as a positive allosteric modulator (PAM) of the dopamine D₂ receptor (D₂R) [1,2]. Its potent PAM activity and D₂-selectivity make it a promising candidate for treating dopamine-related neurological disorders, including Parkinson's disease [2,3].

Previous findings from our research group have shown that substitution of the L-proline (Pro) residue in MIF-1 with L-pipecolic acid (Pip) produces potent PAM derivatives with favorable toxicological profiles [3]. In this work, we report the design and synthesis of a new series of MIF-1 derivatives by merging Pro and Pip scaffolds into a rigid bicyclic framework, (1*R*,3*S*,4*S*)-2-azanorborene-3-carboxylic acid. Using this bicyclic structural motif, we synthesized a library of 16 bridged MIF-1 derivatives and characterized their PAM activity through pharmacological functional assays at human D₂R.

Among the tested compounds, four promoted up to a 6.6-fold improvement in dopamine potency at subnanomolar concentration (0.01 nM), indicating potent PAM activity. Additionally, these compounds were found to display safety profiles and improved pharmacokinetic properties compared to the parent neuropeptide.

These results support the potential of bridged MIF-1 derivatives as a new class of potent and D₂-selective modulators with therapeutic potential for application in Parkinson's disease.

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Design and synthesis of novel compounds targeting the serotonin 2A receptor

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The serotonin 2A receptor (5-HT_{2A}R) is a G protein-coupled receptor involved in numerous physiological processes. It is also associated with psychiatric disorders such as schizophrenia, depression, anxiety, bipolar disorder, and addiction, representing an important pharmacological target [1–3]. For instance, 5-HT_{2A}R antagonism by the antipsychotic clozapine drew attention to the potential role of serotonergic mechanisms in mediating antipsychotic effects [4]. Despite significant advances in medicine, improving the understanding and treatment of psychiatric disorders remains an urgent priority. In our research group, clozapine inspired the design and development of novel molecules with potential antipsychotic activity. One of these compounds, isolated as a mixture of two structural isomers, showed high affinity for the 5-HT_{2A}R and potent antagonist activity. Initial biological tests suggested that only one of the isomers was responsible for this high activity [5].

A synthetic methodology for the selective formation of each isomer was established and implemented. Starting from 4- or 5-chloro-2-nitroaniline, two main challenges were encountered: the reduction of the nitro group and the formation of an amidine moiety. An *in silico* approach involving protein–ligand docking was developed and optimized to predict the binding conformation of the isomers and their relative binding affinities. It also provided further insights into the binding determinants of this promising receptor. A computational procedure was applied to design a library of molecules and optimize their affinity for the target. Pharmacological properties and synthetic feasibility were also considered during these *in silico* studies. Novel molecules were rationally designed with potential affinity for the 5-HT_{2A}R, representing promising candidates to advance therapeutic strategies and deepen our understanding of psychiatric disorders.

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Discovery and optimization of benzimidazole-pyrazole chemical tools targeting RIPK1

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Necroptosis, a regulated form of necrosis, is the major mechanism of cellular death upon extracellular inflammatory signalling and is crucially dependent on the activity of the receptor-interacting serine/threonine-protein kinase 1 (RIPK1) and its downstream mediators: RIPK3 kinase and the pseudokinase MLKL. Consequently, RIPK1 has emerged as a promising therapeutic target for the treatment of a wide range of human neurodegenerative, autoimmune, and inflammatory diseases [1,2].

While research into RIPK1 modulators has progressed significantly, only a limited number of different chemotypes have advanced to clinical trials. To address the scarcity of chemotypes targeting necroptosis and RIPK1, iMed.Ulisboa recently developed a phenotypic high-throughput screening strategy to identify novel necroptosis inhibitors from a library of more than 250,000 compounds (AstraZeneca) [3]. This led to the discovery of several new compounds active against necroptosis, including a new family of **benzimidazole-pyrazole**, which we further explored to uncover their mode of action. In this communication, we report the optimisation of these benzimidazole-pyrazole hits, using computational and synthetic chemistry approaches, generating new chemical probes with nanomolar potency against RIPK1 (Figure 1).

By expanding the toolbox of RIPK1/necroptosis small-molecule probes, we will contribute to a better understanding of necroptosis at a molecular level, providing a promising therapeutic alternative for pathologies with necroptosis pathway alterations.



Fig. 1: **A)** Probing the chemical space around **benzimidazole-pyrazole** core structure (SAR studies); **B)** biological evaluation of the new analogues; and **C)** bioorthogonal “turn-on” probes for live-cell imaging of **benzimidazole-pyrazole** inhibitors.

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Core-shell polycationic dendrimers: from molecular design to cancer treatment

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Exploiting the structural and chemical alterations of cancer cell plasma membranes represents a promising avenue for the development of next-generation nanotherapeutics. Cancer cells display a distinct membrane composition, often characterized by the externalization of anionic phospholipids and other negatively charged components. These features provide unique molecular signatures that can be selectively targeted by cationic nanostructures, enabling improved therapeutic specificity and efficacy.

To harness these properties, the design and synthesis of new nanoscale systems capable of acting as therapeutic agents or as vehicles for targeted drug delivery have gained increasing relevance in nanomedicine. Cationic anticancer peptides (ACPs) serve as an excellent biological model, as their preferential interaction with the negatively charged membranes of cancer cells leads to membrane disruption and cell death. Inspired by this mechanism, synthetic mimics of anticancer peptides (SMACPs) have been developed to combine the biological efficacy of ACPs with the structural precision and stability of nanoscale materials [1].

In this framework, dendrimer-based SMACPs offer a powerful and versatile approach. We report the design of core-shell polycationic PURE dendrimers, engineered to mimic the amphiphilic and cationic nature of ACPs [2]. These dendrimers interact preferentially with cancer cell membranes through electrostatic interactions, promoting selective cytotoxicity while minimizing off-target effects. The modularity of the dendrimer architecture allows fine control over surface charge, hydrophobicity, and molecular weight, making these systems highly tunable platforms for targeted nanomedicine. Overall, this work highlights the potential of core-shell dendrimers as synthetic, peptide-inspired nanotherapeutics for precision cancer treatment.

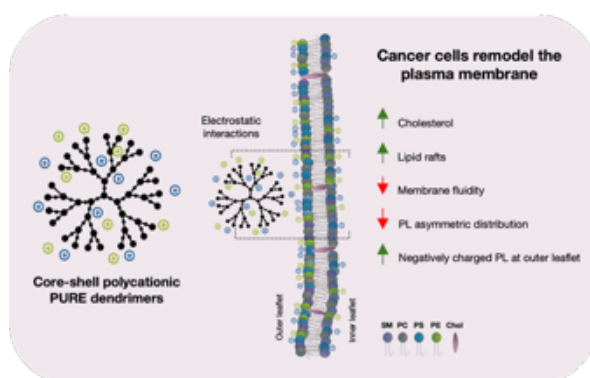


Fig.1. Targeting cancer cell membranes with core-shell polycationic PURE dendrimers.

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Chemical strategies for cancer therapy: targeted degradation of mutant p53 via PROTACs

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Cancers carrying mutant p53 remain particularly challenging to treat, as these mutations drive enhanced cell proliferation and metastasis. These mutations occur preferentially in the DNA-binding domain of p53, leading to changes in the protein structure of this protein that inactivate its wild-type functions [1]. Moreover, mutated forms are in several cases associated with overexpression because mutant p53 is less susceptible to degradation than wild-type p53. In particular, R273H and R280K mutant p53 are two DNA-contact mutations frequently found in different types of cancers, such as colorectal and breast cancers [2].

One of the most promising therapeutic approaches in medicinal chemistry is the use of Proteolysis-targeting chimeras (PROTACs) to induce selective degradation of overexpressed or disease-associated proteins. In contrast to conventional small-molecule inhibitors, PROTACs provide distinct advantages, including a catalytic mechanism of action and the potential to overcome drug resistance [3].

Building on previous work from our research group, that led to the discovery of enantiopure tryptophanol-derived isoindolinones able to reactivate R273H and R280K mutant forms of p53, we envisioned the development of PROTACs for the selective degradation of these p53 mutants [4]. In this communication, we present our latest results on the design and synthesis of tryptophanol-derived isoindolinone-based PROTACs aimed at generating novel degraders of mutant p53 R273H and R280K. The synthesis of the compounds will follow two different synthetic approaches: amide bond and click chemistry (Figure 1).

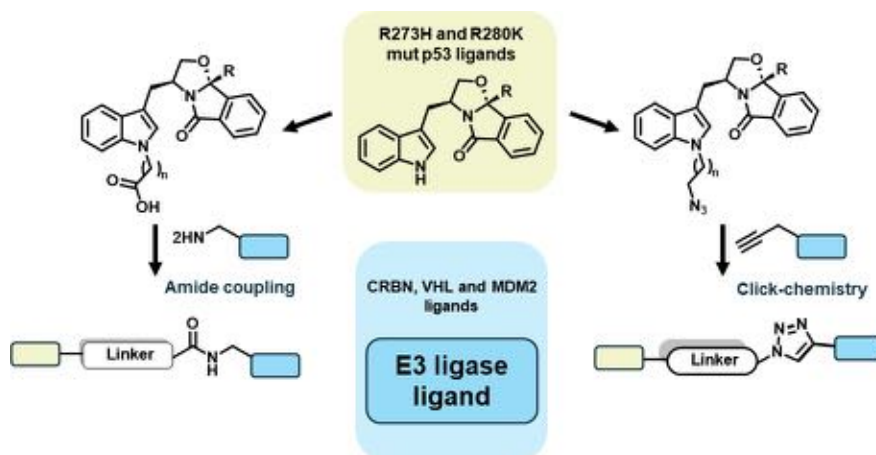


Fig.1. PROTACs designed to selectively target the R273H and R280K mutant forms of p53.

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Synthesis and antibacterial evaluation of methyl 3-amino-6-(hetero)arylthieno[3,2-*b*]pyridine-2-carboxylates and their encapsulation in hyaluronic acid hydrogel

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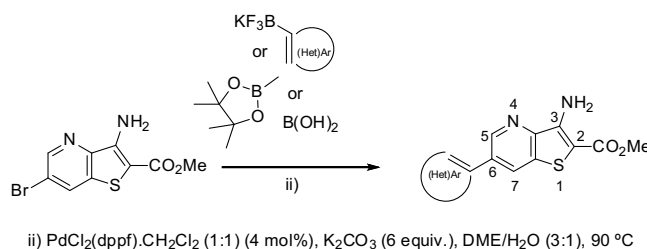
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For several years, our research group has focused on the functionalization of the thieno[3,2-*b*]pyridine scaffold through Pd-catalyzed C–C (Suzuki–Miyaura) couplings either in the pyridine or thiophene ring. The resulting compounds were evaluated for their antitumor activity against several human tumor cell lines. Some derivatives have demonstrated promising efficacy with no observed toxicity [1,2]. In this work, we report the synthesis of twelve 6-(hetero)arylthieno[3,2-*b*]pyridines by Suzuki–Miyaura coupling of the methyl 3-amino-6-bromothieno[2,3-*b*]pyridine-2-carboxylate with aryl or (hetero)aryl boronated compounds (Scheme 1).



Scheme 1. Synthesis of 6-(hetero)arylthieno[3,2-*b*]pyridines by Suzuki–Miyaura cross-coupling.

Bacterial infections are currently a significant cause of morbidity and mortality worldwide, and the prevalent treatments are failing to respond to the growing problem of antimicrobial resistance (AMR). The development of new effective antimicrobial agents constitutes a priority. For this purpose, the compounds prepared were evaluated for their antibacterial activity against Methicillin-resistant *Staphylococcus aureus* (MRSA) [3]. The three most promising compounds, were subsequently encapsulated within a hyaluronic acid (HA)-based hydrogel to enhance delivery and biocompatibility [4]. The compounds-loaded hydrogel systems showed significant activity against MRSA and a positive effect on the proliferation of mammalian L929 fibroblast cells. These systems have potential for long-term treatment and controlled antibiotic delivery.

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Design and synthesis of novel potential inhibitors of p52 dimerization

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Cancer represents a substantial impact on the global healthcare system and remains the second leading cause of mortality, according to the World Health Organization. Due to the profound impact of this disease, both in terms of the cost in human lives and the pressure it imposes on healthcare resources, there is an urgent need for increased research efforts to mitigate the consequences of cancer care [1]. The activation of nuclear factor-kappa B (NF- B) signaling has emerged as a crucial molecular link between inflammation and cancer [2]. The non-canonical NF- B pathway involves the processing of p100 into p52 [3], and the overexpression of non-canonical NF- B subunit p52 has been reported in cancers of epithelial origin [4]. Therefore, targeting p52 represents a promising therapeutic approach in cancer treatment. The purpose of this study was the rational design and the synthesis of novel potential p52 inhibitors, focusing on blocking the formation of p52:p52, p52:RelA and p52:RelB dimers through structure-based drug design. A virtual screening of 48 billion compounds from Enamine's REAL Space was performed using motif matchers from several chemical families of interest (e.g., acridine, acridone, chromone, quinolone, triazole, xanthone, indazole, pyrazole, quinoline, pyridine and steroid derivatives). This screening resulted in 796245 hits, which were evaluated against chain B of the p52:p52 dimer (PDB: 1A3Q) through AutoDock Vina. Chain B of the p52:p52 dimer was selected since it corresponds to a monomer that aligns with the monomeric subunits of both the p52:RelB dimer (PDB: 3DO7) and the p52:RelA dimer (predicted by AlphaFold), including residues located within the dimerization domain, thereby supporting its suitability as a docking model. Based on the docking score analysis, compound families such as indazole, pyrazole, pyridine, quinoline, quinolone, triazole and chromone were excluded. Following this selection, suitable synthetic strategies were sketched for the promising scaffolds, namely xanthenes, acridones, acridines and steroid derivatives. Herein, we present the synthesis of the selected xanthone family, as well as the preliminary results for the steroid derivatives.

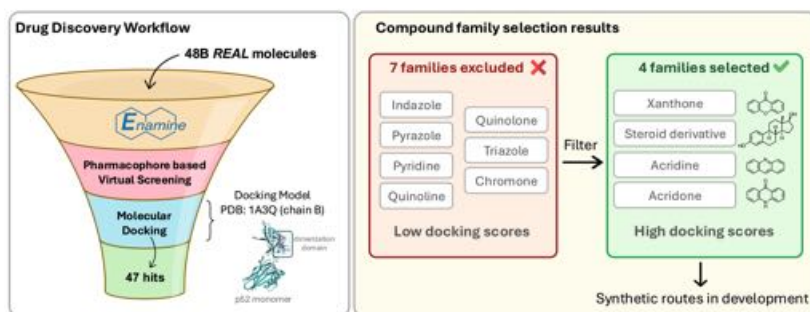


Fig.1. Schematic representation of the design strategy and selected scaffolds proposed for synthesis.

Acknowledgements

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Spectroscopic insights into polyamide 6 microplastics after simulated gastrointestinal digestion

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Microplastics (MPs) are increasingly recognized as ubiquitous pollutants. Emerging evidence indicates that MPs can undergo structural and chemical transformations in environmental, chemical and biological context [1]. Multiple reports have documented MPs in various foodstuffs [2], and that ingestion represents a major entry route into the human body [3], being therefore critical to understand their fate within the gastrointestinal (GI) tract. Here, fluctuations in pH, enzymatic activity, bile salts, and microbial interactions may drive to transformations, potentially altering how MPs interact with tissues and contribute to toxicological outcomes [3]. Considering it, this study applies a standardized *in vitro* digestion model [4] to polyamide 6 (PA6), also known as Nylon 6, a food-packaging polymer seldom studied in this context [5], and combines it with surface-sensitive Fourier-Transform Infrared Spectroscopy (FTIR) to evaluate GI-induced transformations. Spectral analysis revealed that while the characteristic amide I (~1360 cm⁻¹) and II (~1535 cm⁻¹) band of PA6 remained identifiable, shifts in peak intensity and slight band broadening occurred across digestion phases. After the oral stage, minor surface alterations were detected, with more pronounced modifications being observed after the gastric exposure (notably in amide-related regions, suggesting protein/enzyme interactions). Interestingly, an additional peak at ~2070 cm⁻¹ was observed in the oral and gastric phases, which may correspond to by-products formed during early digestion. Following intestinal digestion, additional spectral changes suggested interactions with bile salts and enzymatic hydrolysis, indicating partial surface reorganization rather than extensive bulk polymer degradation. These results demonstrate, for the first time, that PA6 MPs undergo subtle, but detectable, surface chemistry alterations under GI-like conditions, challenging the assumption of inertness and offering new spectral markers to probe microplastic–host interactions. Further analyses and biological assays are required to clarify their toxicological relevance.

Acknowledgments

This work received financial support from Fundação para a Ciência e a Tecnologia (FCT)/ Ministério da Ciência, Tecnologia, e Ensino Superior (MCTES) (UIDB/50006/2020, LA/P/0008/2020 and UIDP/50006/2020) through national funds. Mariana Lamas thanks FCT/MCTES and ESF for her PhD grant (2024.05133.BD). M. Oliveira thanks the TENURE – FCT Tenure Program – 1st Edition (Reference PRR No. 02/C06-i06/2024), with financial support from FCT/MCTES through national funds. F. Rodrigues was supported by FCT, I.P under the TENURE program [2023.14718.TENURE.016].

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Development of a grape stem polyphenol-based mouthwash for periodontal health: from byproduct valorization to therapeutic application

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Periodontal diseases continue to represent a major global health challenge, while the wine industry generates significant amounts of byproducts, particularly grape stems. In this study, we explore the translation of grape stem polyphenols into a sustainable, bioactive mouthwash aimed at preventing and managing periodontal diseases. Grape stem extracts, rich in polyphenols, exhibit potent antibacterial and anti-biofilm activities against key periodontal pathogens, including *Porphyromonas gingivalis*, *Aggregatibacter actinomycetemcomitans*, and *Fusobacterium nucleatum*, effectively inhibiting bacterial adhesion and biofilm formation. Additionally, these polyphenols display notable anti-inflammatory properties by downregulating pro-inflammatory cytokines, and they exert strong antioxidant effects, scavenging reactive oxygen species and supporting cellular antioxidant defenses to mitigate oxidative stress in periodontal tissues.

Beyond antimicrobial and anti-inflammatory effects, grape stem polyphenols modulate osteoclastogenesis and promote osteogenic differentiation, potentially preserving alveolar bone integrity, while also contributing to a favorable modulation of the oral microbiome. Leveraging grape stems in this context not only provides therapeutic benefits but also aligns with circular economy principles, transforming a low-value winemaking byproduct into a high-value ingredient for oral healthcare, reducing environmental impact and creating new economic opportunities.

Currently, these findings are being translated into the development of a functional mouthwash formulation. Preliminary formulation studies focus on maximizing the bioactivity and stability of the polyphenols within the product while maintaining safety and sensory acceptability for end-users. This work demonstrates a practical path from byproduct valorization to the creation of an eco-friendly, multi-targeted oral healthcare solution. Future steps will involve clinical testing to evaluate efficacy, safety, and consumer acceptability, ultimately positioning grape stem polyphenols as a sustainable and effective intervention for periodontal health.

Acknowledgments

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Novel 3-indolyl-thiazoloisoindolinones as potential p53 activators

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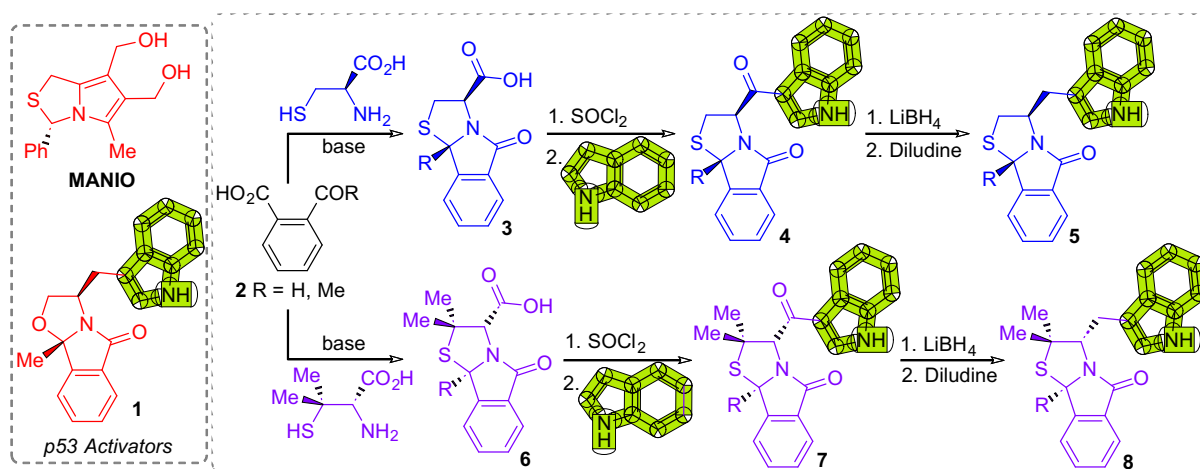
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Cancer is a leading cause of death worldwide, accounting for nearly 10 million deaths in 2020 [1]. A critical event in cancer is the disruption of the tumor suppressor p53 protein pathway [2]. Therefore, restoring the p53 pathway, namely by activating p53 is a valuable strategy in the fight against cancer. Our interest in this research topic led us to the development of a new and promising p53-activating anticancer agent, **MANIO** [3]. Other molecules, such as the tryptophan-derived oxazoloisoindolinone **1**, have also been described as p53 activators [4]. This led us to design new molecules that share some structural features of the latter compounds, but which are derived from thiazoloisoindolinones (Scheme 1). Tricyclic isoindoles **3** have been previously synthesized in our research group and obtained in a one-pot procedure involving the reaction of *L*-cysteine with 2-substituted benzoic acids **2** [5]. The Friedel–Crafts acylation of indole using compounds **3** as the acylation agent after activation, produced the compounds **4**. Reduction with LiBH₄ gave two diastereoisomeric alcohols, which were efficiently separated and their structure unambiguously established by X-ray crystallography. Further reduction with diludine yielded target compounds **5**. Similarly, compounds **8** could be produced starting with *D*-penicillamine (Scheme 1). Details of this study will be disclosed.



Scheme 1. Synthetic strategy to novel indolyl-functionalized thiazoloisoindolinones.

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Innovative microfluidic paper-based analytical device for the determination of zinc(II) in children's urine samples

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Zinc(II) is an important metal ion, present in the human body, that plays vital roles in biological processes, specifically in child growth, development, and improvement of cognitive behaviour. Its deficiency can cause serious health risks, which makes urinary zinc(II) analysis very relevant, as it is considered a biomarker of children's psychological development and can even indicate urinary tract diseases. Zinc supplementation has been applied as a preventive therapeutic measure, resulting in a positive effect on child development, which makes supplementation monitoring critical to ensure child growth worldwide.

Paper-based sensors have gained relevance over the years in providing reliable, rapid, inexpensive, and user-friendly analytical analysis. When combining this tool with fluids of non-invasive collection, namely urine, it enables an effective monitoring of health-related issues frequently, especially in children, who often oppose taking blood.

As such, in this work, an innovative microfluidic paper-based analytical device (μ PAD) was developed, based on the colorimetric reaction between zinc(II) and dithizone, in an alkaline medium. The sensor quantifies zinc(II) within a dynamic range of 0.050–0.750 mg/L, where the reaction occurs for 5 minutes and the given signal remains stable for 30 minutes, with a LOD (limit of detection) of 0.010 mg/L and a LOQ (limit of quantification) of 0.034 mg/L. Before loading the sample in the μ PAD and after its assembly with layered paper discs impregnated with the reagents, it can be used for up to one week, if stored in air or vacuum conditions and at room temperature. The accuracy assessment of this method was carried out by comparing and validating the results of different urine samples (directly used or diluted 1:2) in the μ PAD and in the AAS (atomic absorption spectrometry).

The developed μ PAD offers a point-of-care analysis of urinary zinc(II) levels in children, an important tool for practical clinical diagnosis. It can also be employed for testing zinc(II) levels in food supplements.

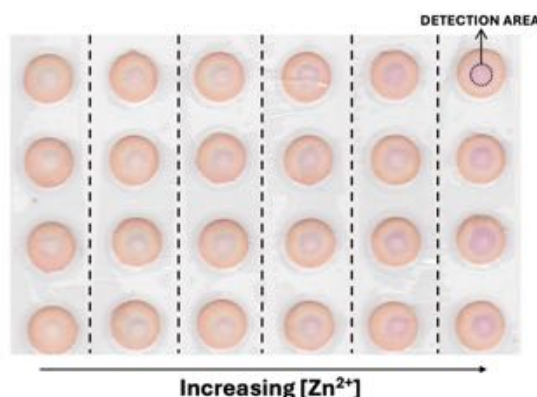


Fig.1. Scanned image of the developed device showing the pink coloured complex intensity increase with the increase in zinc(II) concentration after 5 min reaction time and highlighting the detection area.

Acknowledgments

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Photoactive glycoconjugates: a sweeter approach to cancer imaging and therapy

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Despite significant scientific and technological advancements in diagnostic and treatment, cancer continues to be one of the leading causes of death worldwide [1]. Therefore, the development of new diagnostic and therapeutic agents, ideally more specific and effective, remains a top priority [2]. Diagnostic methodologies include fluorescence imaging (FI), allowing for the visualization and monitoring of tissue accumulation and treatment progression, while therapeutic capabilities, such as photodynamic therapy (PDT), allow for a more effective and selective treatment [3]. However, some common agents (e.g. BODIPY, Porphyrins) exhibit hydrophobic cores, resulting in poor water solubility and limited specificity toward tumor tissues [4]. This limitation can be mitigated through bioconjugation with different “bio” motifs, such as carbohydrates, improving water solubility and recognition of overexpressed lectins and other carbohydrate-binding proteins on cancer cell membranes, improving targeting efficiency [5]. To further enhance and fine-tune these properties, glycodendritic architectures offer a versatile and sophisticated approach for constructing structurally diverse bioconjugates [5], [6]. In this communication, we will present the design and synthesis of novel Glycodendritic Bioconjugates incorporating dendritic units with different carbohydrate moieties (Fig. 1).

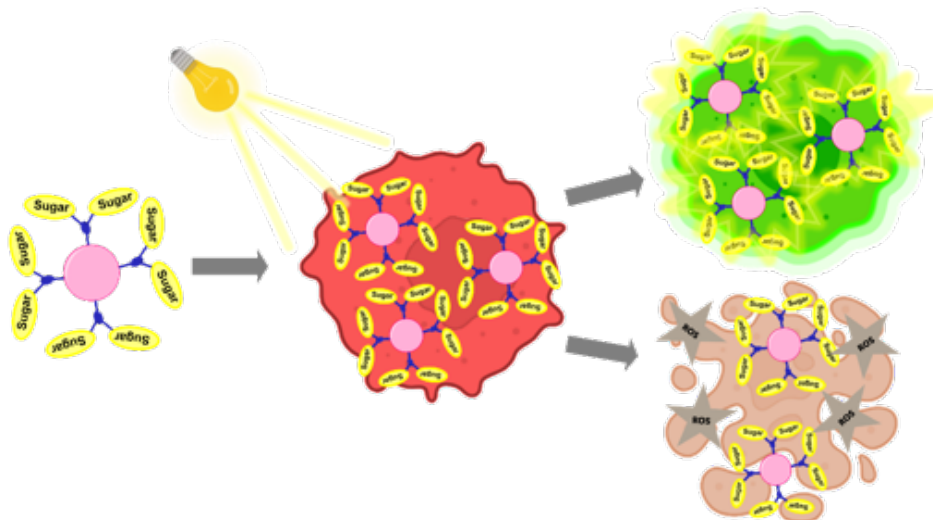


Fig.1. Photoactive Glyco-conjugates and their applications in FI and PDT.

Acknowledgements

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Nanoparticles-assisted proteomic analysis of circulating proteins to predict therapeutic response in HER2-positive breast cancer

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Despite the increasing use of neoadjuvant chemotherapy (NAC) in HER2-positive breast cancer (BC) patients, the clinical problem of predicting individual treatment response remains unanswered. Furthermore, the use of ineffective chemotherapeutic regimens should be avoided [1]. Conversely, serum biomarkers are increasingly being investigated for their potential to predict therapeutic response and to guide the development of personalized treatment regimens [2].

This study aimed to identify protein networks and biomarkers capable of predicting the response to NAC in patients with HER2-positive BC by conducting a comprehensive large-scale LC-MS/MS-based qualitative and quantitative proteomic analysis of serum samples from responders and non-responders. Serum samples were collected prior to NAC and processed using three different approaches, both with and without nanoparticles. Qualitative analyses revealed distinct proteomic profiles between responders and non-responders, particularly involving proteins associated with the complement and coagulation cascades as well as apolipoproteins. Notably, three proteins as afamin (AFM), alpha-1-antitrypsin (SERPINA1), and apolipoprotein D (APOD) were correlated with resistance to NAC. These findings demonstrate that serum biomarker profiles can discriminate between responders and non-responders and, if further validated, could be of significant clinical value in guiding the design of individualized treatment regimens for patients with HER2-positive breast cancer in the neoadjuvant setting.

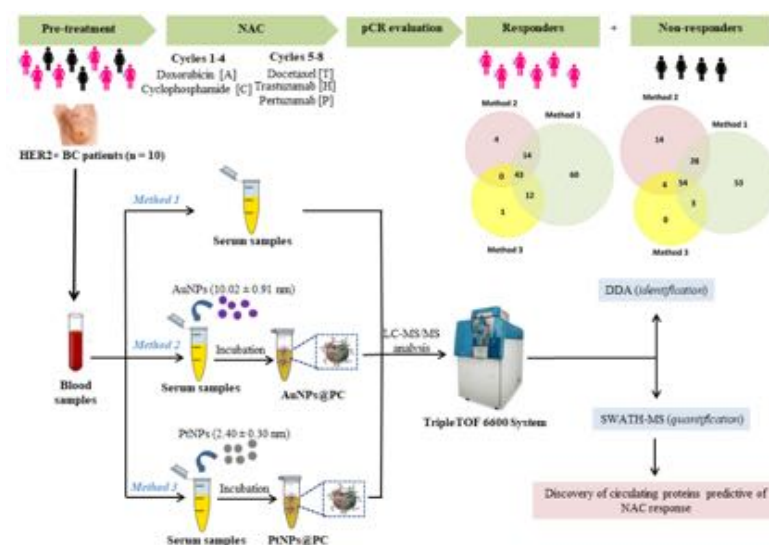


Fig.1. A schematic diagram of experimental workflow.

Acknowledgments

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Novel donor-acceptor-donor probes for amyloid species: design, synthesis, and evaluation

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Early detection is essential for effective management of Alzheimer's disease (AD) [1]. Several near-infrared fluorescent (NIRF) probes have been developed to target tau and A β aggregates, including curcumin derivatives, carbazoles, DANIRs, BODIPYs, and cyanines [2]. These probes typically employ donor-acceptor (D-A), or donor-acceptor-donor (D-A-D) frameworks that emit within the NIR-I region (700-900 nm) [2,3]. Chromone-based scaffolds, in particular, show strong affinity for A β species and have enabled the design of aggregation-induced emission (AIE) active probes [4].

Here, we report the design, synthesis, and full NMR characterization of a new series of chromone-derived D-A-D probes for amyloid detection. Starting from 2-methylchromones, three sets of derivatives were obtained by varying: (i) east-side π -bridge length (0-2 conjugated double bonds), (ii) west-side donor groups (aliphatic vs. aromatic amines), and (iii) west-side π -bridge extension with electron-donating styrenes.

The native fluorescence of these compounds was first assessed in solvents of different polarity. Selected candidates with red-shifted emission were then evaluated for their fluorescence responses to A β species (monomers, oligomers, and fibrils). We disclose the photophysical profiles of these probes, correlating structural features with emission properties and amyloid-binding behavior. (Fig.1).

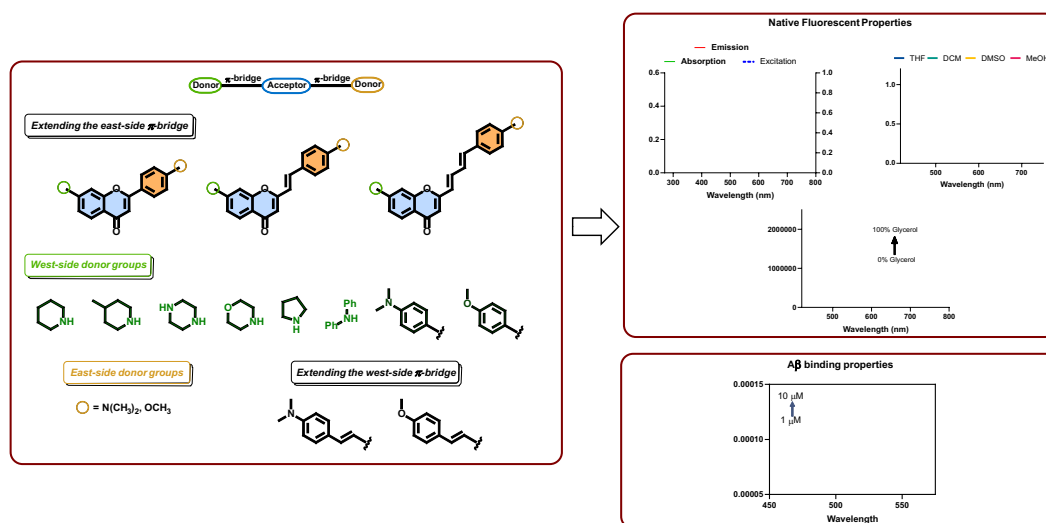


Fig.1. New amyloid targeting chromone-type probes with D-A-D architectures.

Acknowledgments

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Multifunctional theranostics for Alzheimer's Disease: bridging early detection and treatment

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Alzheimer's disease (AD) is a multifactorial neurodegenerative condition primarily driven by the aggregation of β -amyloid (A β), which contributes to neuronal injury, cognitive deterioration, and behavioral impairments [1]. Despite decades of intensive research and major financial investment, therapeutic options capable of effectively preventing or reversing A β aggregation remain scarce. Since 2003, only a limited number of drugs have been approved, most notably the monoclonal antibodies Aducanumab, Lecanemab, and Donanemab. Although these represent significant progress in AD pharmacotherapy, their elevated costs raise serious concerns regarding global accessibility, particularly within resource-limited healthcare systems [2,3]. In addition, reliable identification of A β deposits is still largely restricted to post-mortem examinations, posing considerable challenges for early diagnosis and real-time disease monitoring [4]. To address these unmet needs, we have recently introduced a novel molecular scaffold with the ability to interact with and disrupt A β fibrillation. Building upon this framework, we modified the compounds by integrating amine groups to boost their intrinsic fluorescence, thus generating multifunctional candidates with both diagnostic and therapeutic potential (**Fig.1**). This strategy opens new avenues for simultaneous early detection and intervention in AD.

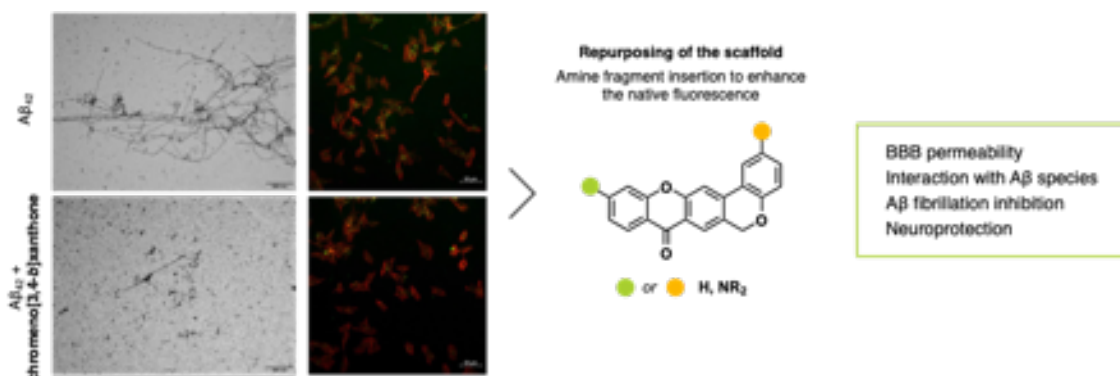


Fig.1. Repurposing of the chromeno[3,4-*b*]xanthone scaffold.

Acknowledgments

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Design and evaluation of novel DprE1-targeting agents against *Mycobacterium tuberculosis*

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Tuberculosis (TB) remains one of the leading causes of death from infectious diseases worldwide. A worrying concern is the growing prevalence of multidrug-resistant (MDR) and extensively drug-resistant (XDR) strains, underscoring the urgent need for new therapeutic strategies[1]. One of the most promising drug targets is decaprenylphosphoryl- β -D-ribose 2'-epimerase (DprE1), an essential oxidoreductase in the synthesis of DPA, the sole donor of arabinose residues for the mycobacterial cell wall[2]. Some DprE1 inhibitors, such as nitrobenzamides and benzothiazinones, are believed to act as suicide inhibitors by covalently binding to Cys387 after FAD-mediated activation, thereby blocking substrate access to the active site, and have demonstrated potent antitubercular activity[2].

Our group has been exploring nitrobenzamide-based scaffolds, with a particular focus on introducing structural flexibility—an underexplored but potentially crucial factor for the inhibition efficacy [3,4]. Initial efforts led to the identification of a new family of amides structurally related to the dinitrobenzamides (DNB)[3]. The synthesis began with 3,5-dinitrobenzoic acid as the nitroaromatic core, which was coupled with linear or cyclic amines to generate the corresponding amides[3], followed by the introduction of terminal aromatic groups through ether, ester, or amide bonds[4]. Synthetic strategies relied on nucleophilic addition/elimination, S_N2 , and Mitsunobu reactions. Several derivatives showed relevant activity (MIC ranging from 35 to 150 nM), supporting the importance of the introduction of flexibility in these structures. In parallel, computational studies were also performed to try to identify structure–activity relationships, also comparing our compounds to known DprE1 inhibitors to validate the mechanism of action and to find structural features for further development. This combined synthetic and computational strategy strengthens the understanding of the key molecular elements for activity, providing a foundation for the rational design of the next generation of inhibitors.

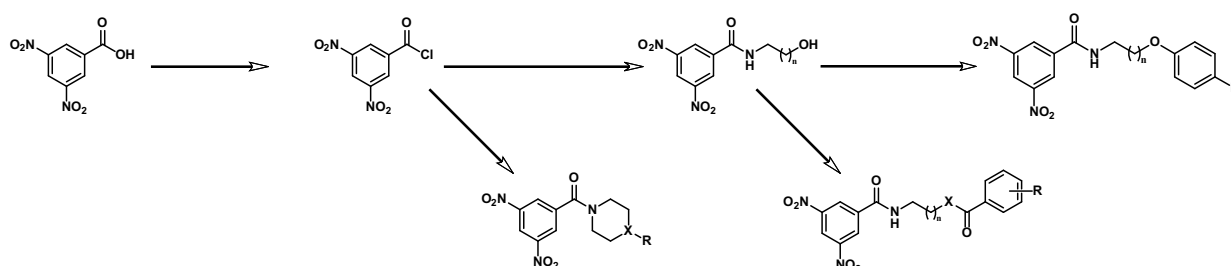


Fig.1. Overview of the synthesis of substituted amide derivatives starting from 3,5-dinitrobenzoic acid.

Acknowledgements

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Assessment of solubility and stability of rifampicin using betaine-based deep eutectic solvents

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Tuberculosis, caused by *Mycobacterium tuberculosis*, remains a widespread disease with significant global public health relevance [1]. Treatment failures, particularly in cases of meningoencephalic tuberculosis affecting children under 10 years, are frequently attributed to challenges such as the poor water solubility, limited blood-brain barrier permeability, and suboptimal palatability of key active pharmaceutical ingredients (APIs) such as Rifampicin. These limitations hinder the effective development and administration of pharmaceutical therapies [2].

Deep eutectic solvents (DES) have emerged as versatile excipients with substantial potential to enhance API solubility - a fundamental factor for improving drug bioavailability and therapeutic efficacy [3]. The highly tunable molecular interactions and physicochemical properties of DES not only increase solubility but also contribute to the chemical and physical stability of drugs, leading to more robust formulations with extended shelf life and reduced degradation. Of particular relevance for paediatric applications, DES can be tailored to improve palatability and enable customization of formulations, addressing the acceptability challenges that frequently limit treatment adherence among children. Recent research highlights that DES-based systems can be engineered to mask undesirable tastes and facilitate appropriate dosing for each age group, offering major advances in paediatric and tailored medicine [3-5].

This work investigates the influence of selected DES on the solubility and stability of Rifampicin in solution. Among the betaine-based DES tested, betaine:1,3-propanediol (1:2) DES containing 30 wt% H₂O showed the most promising enhancement of rifampicin solubility, alongside with improved molecular stability. Further investigation was addressed by searching for the solvent composition which led to the highest rifampicin solubility and stability. Further exploration focused on optimizing the solvent composition to maximize rifampicin's solubility and stability. Ongoing efforts aim to enhance palatability and develop DES-based formulations suitable for additive manufacturing using pharmaceutical 3D printing, ultimately enabling customized therapies tailored to distinct patient populations.

Acknowledgements

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Exploiting respiratory poisoning as a strategy to develop novel antitubercular agents

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Tuberculosis (TB) remains a global epidemic and the deadliest infectious disease worldwide, with 1.25 million deaths in 2023, and 2 billion estimated to be infected with *Mycobacterium tuberculosis* (Mtb), its etiological agent [1]. Global TB control is limited by Mtb's ability to shut down its metabolism to a latent state, with high natural resistance to most antibiotics, including traditional anti-TB agents. This limited efficiency imposes prolonged therapeutical regimens with cumulative adverse effects, that result in low patient compliance, and the emergence and spread of drug-resistant TB, associated to higher lethality and lower treatment success [2].

As Mtb requires a sustained supply of ATP, compounds targeting the energetic metabolism through multiple modes of action (MoA) have been considered an effective strategy to overcome its metabolic adaptability. Currently, the nitroimidazoles, delamanid and pretomanid, are the only drug class with such MoA, targeting replicant Mtb through mycolic acid synthesis inhibition and latent Mtb through respiratory poisoning, caused by the *in vivo* reduction of a nitro moiety, releasing nitric oxide and free radical species that can react with bacterial biomolecules [3].

In a screening performed at iMed against Mtb, a hit nitrofuranylamide (**Fig.1.**) emerged with sub-micromolar MIC value, low cytotoxicity, and a good pharmacokinetic (chemical, plasmatic and metabolic stability) and physicochemical profile. Here, we report the synthesis of a compound library of derivatives containing nitro 5-membered heterocycles. In tandem with traditional synthetic routes, multicomponent reactions were exploited to rapidly expand the chemical library, with high efficiency and selectivity, by reducing the necessary synthetic steps. The biological activity was tested against Mtb H37Rv and drug-resistant strains, and cytotoxicity was assessed against HEK293 cell lines. Compounds aqueous solubility was determined, and an early *in vitro* pharmacokinetic profile was evaluated by HPLC [4] for selected compounds.

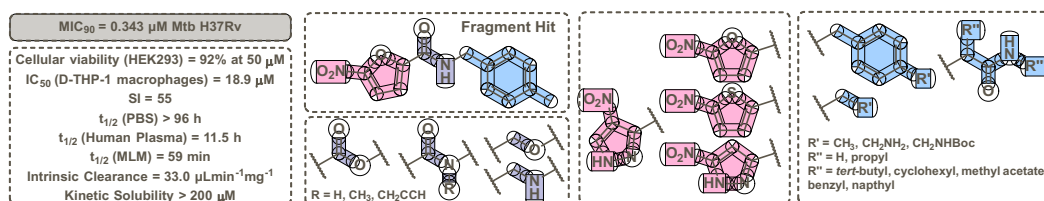


Fig.1. Early pharmacokinetic and biological profile of nitrofuranylamide hit and library expansion.

Acknowledgments

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Parabens' exposure modulates inflammatory mediators in HepG2 cells and human neutrophils

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Parabens, a family of alkyl esters of 4-hydroxybenzoic acid, are widely used as preservatives in cosmetics, pharmaceuticals, and food products. Despite their extensive use, parabens have raised concerns due to reported endocrine-disrupting properties and potential adverse health effects. Humans are mainly exposed to parabens through dermal absorption, ingestion and, to a lesser extent, inhalation [1]. The liver, owing to its central role in xenobiotic uptake, metabolism, and detoxification, is particularly vulnerable to oxidative stress and inflammation arising from these processes, which may ultimately compromise hepatic function. In parallel, neutrophils are key mediators of inflammation, and their activation by xenobiotics can intensify tissue injury through the release of reactive species (RS).

To address this interplay between metabolism and immune response, the present study investigated the effects of commonly used parabens in two complementary human cell models: HepG2 cells, representing hepatic metabolism and oxidative stress, and primary human neutrophils, as key mediators of inflammation. HepG2 cells and primary human neutrophils were exposed to methylparaben, ethylparaben, propylparaben, and butylparaben. In HepG2 cells, viability was assessed using the MTT assay, while RS generation was quantified with the fluorescent probe dichloro-dihydro-fluorescein diacetate (DCFH-DA). In neutrophils, cell death was evaluated by annexin V/propidium iodide staining, and RS production was measured using dihydrorhodamine 123 (DHR 123). Furthermore, expression of key inflammatory mediators in HepG2 cells was analyzed by western blotting, and specific enzymatic inhibitors were applied to elucidate the mechanisms underlying RS generation in neutrophils.

Our results showed that paraben's exposure did not induce cytotoxic effects in either HepG2 cells or human neutrophils. Similarly, no significant changes in RS production were detected in HepG2 cells. However, paraben's exposure altered the expression of inflammatory mediators in HepG2 cells. In neutrophils, parabens stimulated RS generation via activation of phospholipase C and protein kinase C, which in turn promoted the downstream activation of nicotinamide adenine dinucleotide phosphate (NADPH) oxidase and myeloperoxidase.

These findings reveal that, while parabens do not compromise cell viability or hepatic oxidative balance under the tested conditions, they can modulate inflammatory signalling in hepatocytes and trigger RS generation in neutrophils through defined enzymatic pathways, underscoring their potential to disturb immune-metabolic homeostasis and warranting further investigation into their long-term health risks.

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Development of a scaffold-Free 3D osteosarcoma model uncovers selective drug responses

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Osteosarcoma (OS) is the most aggressive primary bone tumor, with high prevalence among children and adolescents. Despite advances in chemotherapy, therapeutic options remain limited, and progress is further hampered by the lack of physiologically relevant preclinical models [1]. Conventional two-dimensional (2D) systems are widely employed; however, they fail to adequately recapitulate tumor architecture and biological responses. In contrast, scaffold-free three-dimensional (3D) culture systems (i.e., spheroids) better mimic in vivo conditions, reproducing tumor-like organization with an inner necrotic core, a quiescent middle zone, and a proliferative outer layer, thereby improving the accuracy of drug screening [2]. Flavonoids, a class of polyphenolic compounds, are being extensively investigated for their anticancer properties. They are known to induce cell cycle arrest, promote apoptosis, and inhibit cancer cell migration and invasion, underscoring their therapeutic potential and supporting their evaluation as adjuvants in OS therapy [3].

In this work, we developed a scaffold-free 3D OS model using U2OS cells cultured in agarose-coated plates for drug screening with the flavonoid myricetin and the standard chemotherapeutic agent doxorubicin. U2OS spheroids successfully formed within 72 h after seeding at a density of 10×10^3 cells/mL. Drug screening revealed dose-dependent inhibitory effects of both myricetin and doxorubicin on U2OS spheroids and human mesenchymal stem cell-derived spheroids; however, only doxorubicin demonstrated selectivity for U2OS spheroids. These findings validate the scaffold-free 3D OS model as a reliable platform for drug screening and highlight its relevance for the preclinical evaluation of novel therapeutic candidates for OS.

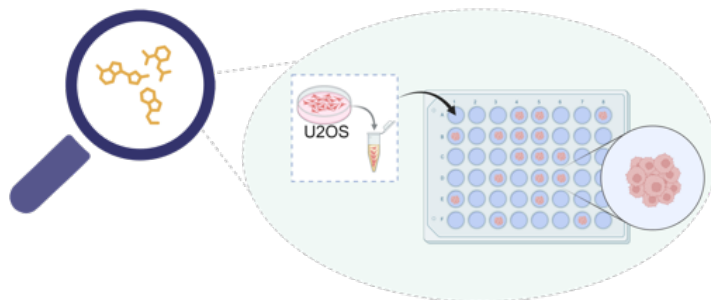


Fig.1. Drug screening analysis on the scaffold-free 3D osteosarcoma model.

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Bypassing antiviral resistance: SARS-CoV-2 M^{pro}-targeting PROTACs as a novel antiviral strategy

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The development of antiviral drugs that directly target viral components, i.e., direct-acting antivirals (DAAs), has been the gold standard of antiviral drug development, although they present a major challenge – the emergence of drug-resistant variants. The rapid evolution of viral genomes hinders the progress made by traditional occupancy-based inhibitors that depend on a high binding affinity to their target, which underscores the need for innovative antiviral therapies (Fig.1A) [1].

The COVID-19 pandemic caused by SARS-CoV-2 is a stark reminder of how rapidly emerging viruses can escalate into global health threats. Importantly, COVID-19 is not an isolated event but part of a recurring pattern of coronavirus-driven outbreaks. The SARS-CoV-2 main protease (M^{pro}) represents an attractive target for DAA development, as it is essential for viral replication and highly conserved among coronaviruses. While approved M^{pro} inhibitors have improved clinical outcomes, drug-resistant variants have already been reported [2]. To address this ongoing risk, it is crucial to develop antiviral strategies that not only combat COVID-19 but can also be repurposed to target novel coronaviruses in the future.

PROteolysis-TARgeting Chimeras (PROTACs) offer a novel mechanism of action (MoA) by inducing targeted protein degradation through the hijacking of the cellular Ubiquitin-proteasome system (Fig.1B). Mechanistically, PROTAC-mediated protein degradation involves a transient binding event to form a ternary complex combining the protein of interest (POI), the PROTAC and a E3 ligase that assists or directly catalyzes the transfer of ubiquitin to the POI, which leads to its degradation. Unlike classical inhibitors, PROTACs do not require high binding affinity to their target, making them a promising strategy to overcome antiviral resistance. Despite its therapeutic potential, the applicability of PROTAC technology in the field of antivirals and the possibility of overcoming viral resistance, and in particular SARS-CoV-2, remains largely unexploited [3].

To address this unmet need, a first-generation of fragment-based M^{pro}-targeting PROTACs were synthesized. For the M^{pro} ligand, a fragment was chosen by analyzing X-ray crystallographic data from SARS-CoV-2 M^{pro} binding fragments. The analysis of the crystal structure of the complex M^{pro}-POI ligand provided a structural basis to rationally choose a linker attachment site. Several combinations of E3 ligase ligands and linkers were synthesized and conjugated to the POI ligand. Currently, M^{pro} degradation assay in a HEK293T cellular system with a stable expression of M^{pro} is undergoing to assess the degradation capabilities of the synthesized PROTACs. This work aims to establish proof-of-concept for fragment-based antiviral PROTACs and pave the way for a new generation of antivirals.

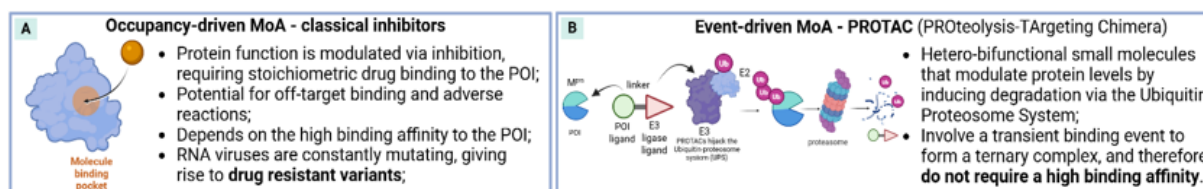


Fig.1. (A) Classical inhibitors MoA; (B) PROTACs MoA.

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Insights into the development of anticancer agents targeting the spindle assembly checkpoint

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Chromosomal instability (CIN) is a common trait of cancer characterized by the continuous gain and loss of chromosomes during mitosis. However, excessive levels of CIN can suppress tumor growth, providing a possible therapeutic strategy [1]. The Spindle Assembly Checkpoint (SAC), a cell surveillance system required for the fidelity of chromosome segregation, has been one of the prime targets to explore this concept. Severe SAC inactivation results in levels of aneuploidy beyond the adaptation capacity of cancer cells, hence culminating in cell death [2]. We previously identified one Aurora B inhibitor that killed almost 100 % of cancer cells in culture, with little toxicity to normal cells. With the aim of improving its therapeutic window, several analogs of the Aurora B inhibitor (INLs) were synthesized. An in-house library of heterocyclic compounds (MACs) was also screened to potentially identify new hit compounds. INLs were synthesized through a nucleophilic aromatic substitution, and their structural elucidation was performed by nuclear magnetic resonance. Using an automated screening instrumentation, INLs and MACs were tested for their ability to induce mitotic slippage in cells blocked in mitosis by nocodazole. The most promising compounds were then tested for their toxic effect in healthy and cancer cell lines and for their ability to inhibit Aurora B by immunostaining. Finally, biomimetic chromatographic studies were performed to evaluate the drug-likeness of the most promising compound, namely its lipophilicity, interaction with biological membranes, and human serum albumin (HSA) binding.

MACs were unable to induce mitotic slippage. Eight INLs were successfully obtained, and all of them were able to induce mitotic slippage of cancer cells. Two INLs (INL4 and INL7) were then tested for their ability to inhibit Aurora B. These compounds not only decreased the levels of Aurora B at the centromeres but also reduced the enzyme's activation status. In cell viability studies at different time points, INLs were capable of killing cancer cells, but only decreased adenosine triphosphate (ATP) levels of non-cancer cells by less than 50%. Results obtained from biomimetic chromatographic studies suggest that the most promising compound (INL7) presents moderate to high lipophilicity and high affinity to HSA. Collectively, the data obtained provides new insights into the discovery of new anticancer agents targeting SAC.

Acknowledgments

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Donor–acceptor benzothiadiazole derivatives as lysosome-targeted photosensitizers for photodynamic therapy

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Photodynamic therapy (PDT) is a minimally invasive treatment that relies on light-activated photosensitizers to generate reactive oxygen species (ROS), thereby inducing selective cell death [1]. The therapeutic efficiency of PDT critically depends on the development of photosensitizers with high photostability, efficient cellular uptake, and robust ROS production [2]. Beyond their therapeutic function, photosensitizers with intrinsic fluorescence can also be strategically employed for bioimaging, enabling real-time evaluation of their biodistribution, selective accumulation within target tissues, and treatment response. This dual functionality provides the basis for theranostic agents, which integrate diagnostic and therapeutic modalities into a single platform [3-4]. Among the promising candidates in this field, benzothiadiazoles (BTDs) are particularly noteworthy due to their exceptional photostability, strong absorption capacity within the visible spectrum, and biocompatibility, making them ideal scaffolds for imaging applications [5-6].

In this work, we present a series of donor–acceptor (D–A) heterocyclic derivatives based on BTD, functionalized with triphenylamine (TPA) as the electron-donating group and formyl or cationic heterocyclic salts as electron-accepting moieties. This molecular design strategy enhances solubility, promotes efficient ROS generation under visible-light irradiation, and improves cellular internalization. Cellular assays using 4T1 cancer cells revealed preferential lysosomal localization, and, upon light activation, the derivatives produced both type I and type II ROS, effectively inducing cancer cell death. Overall, these results highlight the potential of TPA–BTD heterocyclic derivatives as theranostic agents, functioning both as fluorescent markers for labelling lysosomes within cells and as photosensitizers for anticancer photodynamic therapy.

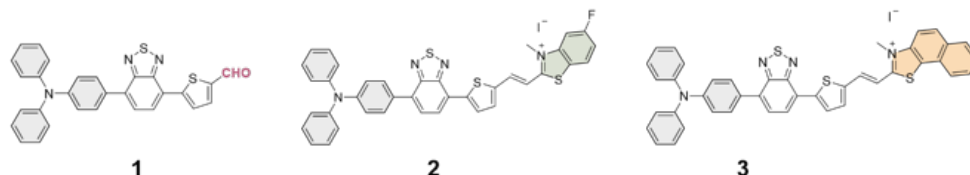


Fig.1. Chemical structure of the triphenylamine-benzothiadiazole derivatives under study.

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SAR Study of new pyrimidine-based compounds as novel anticancer agents for osteosarcoma therapy

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Osteosarcoma is the most common and aggressive primary bone neoplasm, predominantly affecting children and young people. This type of cancer is characterized by heterogeneity, chemoresistance and a high rate of metastasis, with 3770 new cases and 2190 deaths estimated for 2025. [1-4]

The treatment of osteosarcoma has remained unchanged and is mainly based on conservative surgery and adjunctive chemotherapy. For this reason, limited progress has been made in improving survival in patients with osteosarcoma over the past four decades. The 5-year survival rate is approximately 69%, for non-metastatic osteosarcomas, whereas in cases of metastases, this value drops considerably, becoming less than 20%. [1,2,4,5]

Considering the poor prognosis observed in metastatic or recurrent osteosarcoma [2,4,5] due to the aforementioned factors, it is necessary to identify new therapeutic strategies for treating osteosarcoma. In this case, our aim was to explore pyrimidine-based compounds as a novel therapeutic group for osteosarcoma.

Previously, in the research group, a set of pyrimidine-based compounds was synthesized and *in vitro* tested for their activity against osteosarcoma cell lines (143B) and toxicity against healthy lung fibroblast cells (MRC-5), using colorimetric assays for cell viability (resazurin) and growth (sulforhodamine B). The tested compounds showed promising activity, so in the present work, a new set of pyrimidine-based compounds was designed, synthesized and tested against the same cell models, aiming to study their structure-activity relationship (SAR). All the results and a first pyrimidine-based pharmacophore model for osteosarcoma therapy will be presented.

Acknowledgements

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Discovery of potent modulators of the dopamine D₂ receptors with enhanced biochemical stability for Parkinson's therapy

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The gold standard of Parkinson's therapy focuses on dopamine potentiation in the central nervous system through the administration of levodopa [1]. However, long-term therapy with this drug is associated with severe adverse effects that can worsen the motor symptoms of the disease [1]. Melanostatin, an endogenous neuropeptide, is a positive allosteric modulator (PAM) of the dopamine D₂ receptors, being therefore clinically relevant in the context of Parkinson's disease and other dopamine-related disorders [2]. Nevertheless, this neuropeptide fails to exhibit adequate pharmacokinetic properties due to its biochemical nature, hampering its clinical use [2].

This project fosters overcoming the pharmacokinetic limitations of melanostatin by designing and synthesizing azapeptide derivatives through the rational replacement of the proline residue with an azaproline scaffold. This subtle yet effective approach was applied to assemble a library of 24 melanostatin derivatives. Functional pharmacological assays at the dopamine D₂ receptors demonstrated that melanostatin azapeptides display potent PAM activity, reducing the EC₅₀ of dopamine by up to 8.8 times, outperforming the parent neuropeptide at 1 nM.

In addition, melanostatin azapeptides exhibited no cytotoxicity in differentiated SH-SY5Y cells at 100 µM, indicating a favourable therapeutic window, and did not interact with human ERG channels. Preliminary pharmacokinetic studies revealed a 4.7-fold improvement of microsomal stability. Altogether, these results endorse the potential of melanostatin azapeptides as promising candidates for the development of next-generation of therapeutic agents for Parkinson's disease.

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Antibacterial activity of CW49 peptide and analogues for wound healing applications

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Diabetes mellitus (DM) is a chronic metabolic disease, which is among the top ten causes of death worldwide and was, as a result, considered a global epidemic in 2021 by the World Health Organization [1]. DM patients have a 25% lifetime risk of developing diabetic foot ulcers, severe chronic wounds which are significantly susceptible to infections, that can become systemic and lead to limb amputation [2]. The undecapeptide CW49 has demonstrated effects of inhibition of some inflammatory factors such as IL-6 and TNF- α , and of upregulation of some angiogenic proteins, which are advantageous properties for diabetic wounds healing [3]. However, no antimicrobial features have been identified, which, for a multiple wound healing approach, would be crucial since the healing progression is also suppressed by the presence of pathogens [4].

Here, we report the study of the wound-healing peptide CW49 secondary structure through circular dichroism analysis. Its antibacterial activity against gram-positive and gram-negative bacteria was evaluated through minimum inhibitory and bactericidal concentration tests. Furthermore, the improved antibacterial features of some CW49 peptide analogues, with the aim of enhancing the peptide potential for future wound healing applications, will be presented.

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Enhanced antiplasmodial activity of aminoquinoline-derived bile salts

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Malaria remains a major global health challenge, primarily affecting children under five in African regions. The emergence of drug-resistant *Plasmodium falciparum* strains has intensified the need for effective, low-cost therapeutic strategies. In this context, chloroquine and primaquine were reformulated by acid-base pairing with bile acids, yielding amphiphilic drug-derived bile salts that behaved like surface-active ionic liquids (SAIL). These SAIL demonstrated enhanced *in vitro* antiplasmodial activity across multiple parasite developmental stages, including hepatic, blood, and gametocyte forms^[1].

The most active SAIL, namely, chloroquinium cholate (iCQP1) and chloroquinium glycocholate (iCQP1g), were encapsulated in Pluronic F127 polymeric micelles, forming mixed micellar systems with favourable physicochemical characteristics. Differential scanning calorimetry, surface tension analysis, and dynamic light scattering revealed synergistic interactions between the bile salts and F127, resulting in reduced micellization temperature, lower critical micelle concentration, and smaller micelle size. *In vitro* assays against blood-stage *P. falciparum* showed significantly improved efficacy of the micelle-loaded salts, with IC₅₀ values markedly lower than those of the free SAIL^[2].

These findings underscore the potential of both drug-derived SAIL and Pluronic F127 micelles as nanocarriers, offering a promising avenue for repurposing classical antimalarials through biocompatible nanotechnology platforms.

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Bio-based photosensitive polymers and composite films for bacterial photodynamic inactivation

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The global spread of multidrug-resistant (MDR) bacteria poses a major threat to public health, accounting for over 1.3 million deaths annually.[1] Contaminated surfaces in healthcare settings and medical devices are key contributors to infection transmission. Hence, there is an urgent demand for innovative materials and alternative disinfection technologies that are efficient, safe and sustainable. In this context, the development of photosensitive polymeric materials for antimicrobial photodynamic inactivation (aPDI) have emerged as effective solutions against pathogenic microorganisms.[2] In this study, we describe the development of two families of bio-based, light-responsive polymers and composite films for application in bacterial aPDI (**Figure 1**).

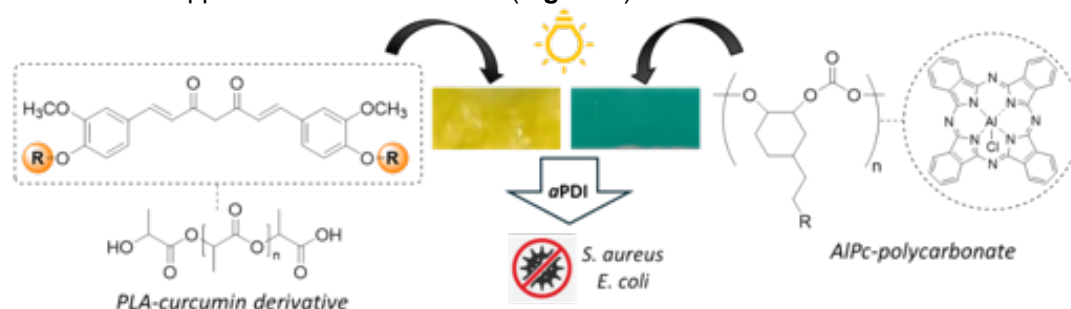


Fig.1. Photoinactivation of bacteria using bio-based photosensitive materials.

The first strategy involves the incorporation of curcumin-derived amphiphilic compounds into biodegradable poly(lactic acid) (PLA) matrices. The second approach entails a one-step catalytic polymerization between CO₂ and epoxides using aluminum phthalocyanines, simultaneously as catalysts and photosensitizers, followed by blending with cellulose derivatives.[3,4] Biocomposite films were obtained by solvent casting and tested against *Staphylococcus aureus* and *Escherichia coli* under appropriate wavelength light irradiation, achieving reductions up to 7 log CFU. The influence of photosensitizer structure and bio-based matrix composition on the photodynamic activity will be presented and discussed.

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Sustainable light-activated PLA-porphyrin fFilms: paradigm shift in infection control

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Multidrug-resistant (MDR) pathogens are responsible for approximately 1.3 million deaths each year, with contaminated surfaces acting as major reservoirs for their transmission [1,2]. At the same time, the growing burden of plastic waste, largely from disposable products, underscores the urgent need for sustainable alternatives, especially in hospital facilities, where the majority of the materials are single-use plastics [3]. Light-based antimicrobial approaches, namely photosensitive surfaces, are gaining attention due to their ability to inactivate pathogens without generating harmful residues using visible light [4].

Herein, we introduce biodegradable polymeric films composed of a polylactic acid (PLA) matrix incorporating newly synthesized imidazolium–porphyrin photosensitizers (**Fig. 1**) [5]. The materials were systematically evaluated for their physicochemical, thermal, and mechanical features, confirming their suitability for single- or repeated-use applications. Upon exposure to blue light (415 nm) doses between 16–24 J/cm², the PLA–porphyrin films achieved complete eradication (>7-log reduction) of both drug-sensitive and multidrug-resistant strains of *Staphylococcus aureus* and *Escherichia coli*.

Biodegradable and with great photoactivity, these films represent a paradigm shift for hospital environments: moving away traditional insufficient disinfectants and disposable plastics towards reusable, eco-friendly and light-activated materials. Overall, our material serves as a proof of concept for the potential of light-responsive biopolymeric surfaces in next-generation infection control.

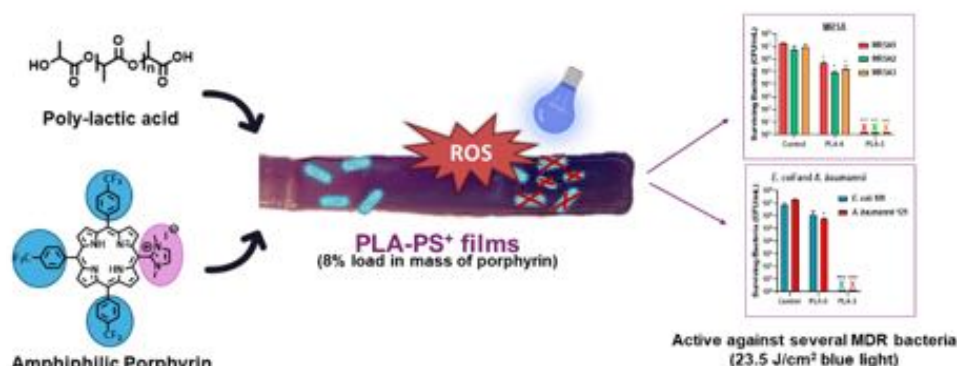


Fig.1. Schematic representation of PLA-porphyrin films and their photoactivity against MDR bacteria.

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Antimicrobial features of a series of Polymyxin B analogues against Gram-negative and Gram-positive bacteria

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Multidrug-resistant (MDR) Gram-negative bacteria have increased dramatically in recent years, which led the World Health Organization (WHO) to flag these pathogens, for which the development of new antibiotics is urgent. The ESKAPE group includes both Gram-negative bacteria (*Klebsiella pneumoniae*, *Acinetobacter baumannii*, *Pseudomonas aeruginosa* and *Enterobacter* species) and Gram-positive bacteria (*Enterococcus faecium* and *Staphylococcus aureus*) that have become resistant to several classes of antibiotics. In addition to being multi-resistant, these bacteria have been shown to be increasingly virulent, being a public health problem worldwide. [1,2] Thus, the available antibiotics no longer seem to address current needs and polymyxin B (PMB) has been reintroduced in the treatment of infections by MDR Gram-negative bacteria, as last resort drug. In this way, the development of PMB analogues aims to increase its effectiveness and reduce side effects, namely, neuro- and nephrotoxicity. [3]

In this communication, we present the evaluation of a series of PMB analogues, obtained through modification of the amino acids at positions 1 and/or 3 or position 6 by structurally related unnatural amino acids. The antimicrobial activity of the six analogues was evaluated against Gram-negative (*P. aeruginosa*) and Gram-positive (*S. aureus*) strains, by determining the minimum inhibitory concentration (MIC) and minimum bactericidal concentration (MBC).

Three PMB derivatives demonstrated promising activity against *P. aeruginosa*. All six derivatives exhibited strong activity against *S. aureus*, comparable to or better than native PMB, with emphasis on the analogues obtained by modification at position 6.

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From diagnosis to treatment: the impact of light in biomedical science

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Light-based technologies are transforming biomedical research and clinical practice by offering non-invasive solutions for diagnostics and therapy. One key application is photodynamic inactivation (PDI) of microorganisms, where light-activated compounds, such as phthalocyanines (Pcs), porphyrins (Pors), BODIPYs, and chlorins (Chls), generate reactive oxygen species (ROS) to eliminate several microbials (e.g., bacteria, fungi, and bacteriophages) [1–5]. Recent advancements in optical imaging use the unique photophysical properties of these chromophores, for high-resolution visualization of biological processes. For instance, porphyrin-based photosensitizers are effective in fluorescence-guided surgery and tumor imaging, improving surgical precision. Additionally, bioorthogonal click chemistry combined with light-responsive probes enhances tumor imaging and therapy by allowing precise labeling and tracking. [6]. Also, fluorescence-based sensors and light-activated probes offer a means for the rapid and sensitive detection of disease-associated nucleic acids, such as microRNAs, thereby opening new avenues for early diagnosis [7]. The research presented here will highlight the transformative potential of light in biomedical science in the quest for enhanced diagnostics and therapeutics.



Fig.1. The biomedical application of light with dyes.

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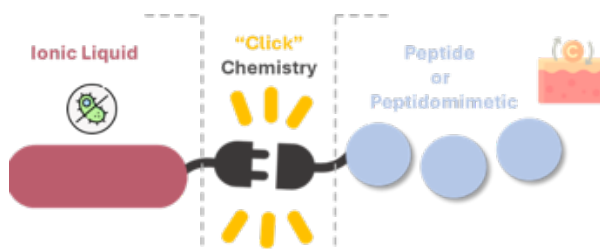
Shorter peptides, smarter wound healing treatments? Redesigning peptides through mimetics

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The development of advanced materials for topical wound healing requires new molecular entities that combine chemical versatility with biological efficacy. [1] Peptides are attractive candidates due to their structural tunability and inherent bioactivity, however, their therapeutic application is often limited by size, stability, and production costs. Our team has previously reported peptide–ionic liquid conjugates for the topical treatment of diabetic foot ulcers, which demonstrated *in vivo* activity in diabetic wound models. [2-3] In the present work, we describe their bactericidal potency in direct comparison with a conventional antibiotic, along with progress obtained so far in a new minimalist design strategy. This approach involves the synthesis of shorter peptide sequences and peptidomimetic analogues incorporating the ionic liquid motif. By systematically varying peptide length and evaluating the effect of removing the ionic liquid component, we establish a structure-activity relationship for the antibacterial performance of this new class of compounds. This strategy not only streamlines synthetic efforts but also highlights the potential of peptidomimetic–ionic liquid hybrids as chemically innovative and therapeutically relevant platforms for topical wound treatment.



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Adsorption systems for diclofenac removal using biomass-derived carbon materials with laccase-functionalized biochar

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The persistent presence of pharmaceuticals in aquatic environments, particularly the anti-inflammatory diclofenac (DCF), represents increasing risks to ecosystems and human health. Conventional wastewater treatment plants (WWTP) are not designed to remove such microcontaminants, resulting in their frequent detection in surface waters and effluents[1]. Adsorption has emerged as promising and cost-effective technology, especially when applying sustainable adsorbent such as biochar (BC) derived from residual biomass. Moreover, functionalization with oxidative enzymes like laccase (LAC) offers the potential to couple adsorption with enzymatic degradation, enhancing removal efficiency and contributing to delay adsorbent saturation[2].

This work aims to produce BC from spent brewery grains using microwave-assisted pyrolysis. The resulting materials were characterized and their performance for DCF removal from water was evaluated, in both fixed-bed column and continuous stirred tank systems. Additionally, the potential of LAC immobilization on BC (BC-LAC), carried out through physical adsorption, was explored as a synergistic strategy to enhance DCF degradation. The adsorptive removal of DCF was investigated in three successive stages, in the semi-continuous systems under evaluation, with performance evaluated through breakthrough curves. First, the effect of adsorbent dose (0.5, 1.0 and 1.5 g L⁻¹ for the tank and 1.0, 1.5 and 2.0 g for the column) was assessed, using buffered ultrapure water (pH 7.0). Subsequently, the influence of flow rate (1.2 and 4 L day⁻¹) was examined, under the same conditions. Finally, the performance of BC and BC-LAC was tested using effluent (i.e., wastewater collected after secondary treatment from an urban WWTP in Aveiro South, Portugal), under optimized operational conditions. For BC-LAC, the feed solution was constantly bubbled with compressed air to create ideal conditions for the enzymatic activity. The DCF concentration at the outlet was monitored by HPLC with UV detection, and experimental data from the breakthrough curves were fitted to Thomas, Yan and Yoon-Nelson models.

Results demonstrated that BC exhibited promising adsorption capacity, with breakthrough curves well fitted by the Yan model. While BC-LAC showed low efficiency in fixed-bed column, a slight initial improvement was observed in the stirred tank, attributed to favourable hydrodynamic conditions and oxygen availability. Overall, the study highlights BC as an effective and low-cost adsorbent for DCF removal. However, the application of BC-LAC still requires further optimization of immobilization methods and operational parameters to achieve consistent performance. Notably, few studies have explored BC obtained from industrial residues for pharmaceutical removal from water, in semi-continuous systems. Therefore, this work fills this gap and provides valuable insights into the applicability of this material under realistic conditions. While also highlighting the added value of enzyme-functionalized BC, opening new perspectives for more sustainable and efficient treatment.

Acknowledgments

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One-pot strategy for the assembly of *cis*-4-hydroxy-L-prolyl amides

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cis-4-Hydroxy-L-proline (*cis*-Hyp) is a rare yet highly valuable isomer of 4-hydroxy-L-proline, found in natural products such as phallotoxins from *Amanita* mushrooms [1]. Due to its unique stereochemistry, *cis*-Hyp has emerged as a privileged scaffold for both pharmaceutical and cosmeceutical applications [1]. However, its high cost and the inefficiency of existing synthetic strategies have hindered its application in drug development.

In this work, we disclose a sustainable, scalable, and highly efficient telescoped one-pot methodology for the preparation of *cis*-Hyp amides from readily available *trans*-4-hydroxy-L-proline (*trans*-Hyp), without the need for peptide coupling reagents or capping steps (Figure 1) [1]. These transformations feature stereoinversion of *trans*-Hyp via an intramolecular Mitsunobu reaction, followed by lactone aminolysis as a privileged strategy for amide bond construction employing a wide range of nucleophiles, including primary, secondary, and aromatic amines, amino alcohols, amino acid derivatives, and dipeptides (76-97% overall yield, 25 examples), all achieved without isolation of intermediates and with complete diastereoselectivity [1,2]. This methodology is readily scalable (up to 91% yield) and can be extended to a three-step telescoped process, incorporating an additional O-silylation reaction, affording an 83% overall yield [1,2]. Compared to traditional disconnected approaches, this strategy offers operational simplicity, superior atom economy, and reduced waste generation [1,2]. The synthetic utility of the method is showcased by the formal synthesis of a bioactive compound with anti-amnesic properties [1,2]. This streamlined and sustainable protocol significantly expands access to *cis*-Hyp-containing peptides, paving the way for their broader use in peptide research.

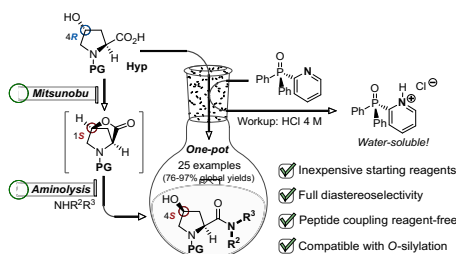


Fig.1. A telescoped one-pot assembly of *cis*-Hyp from readily available *trans*-Hyp.

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Extraction of cannabinoids from THC-Rich cannabis using supercritical CO₂

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Cannabis contains over 750 compounds, including cannabinoids, terpenes, stilbenoids and alkaloids [1]. Cannabinoids, in particular tetrahydrocannabinol (THC), cannabidiol (CBD), cannabigerol (CBG), and cannabigerolic acid (CBGA) have been recognized for their potential therapeutic activity in several applications, such as in the treatment of chronic pain, anxiety, epilepsy, and neurodegenerative disorders. The most conventional method for cannabinoids extraction from biomass is maceration, which consists of a solid–liquid extraction process usually done at ambient or low temperature and with a long contact time. The most common solvent is ethanol, mainly when the final product is for medical applications due to its low toxicity, polar nature and low boiling point. However, this method presents some drawbacks such as the co-extraction of chlorophylls and waxes, which confer unpleasant organoleptic properties to the extracts and hamper their use in the food and medical industries.

Supercritical fluid extraction with CO₂ (SCF) has gained attention over the years, due to its high selectivity, low environmental impact, production of solvent-free and wax-free extracts, and its adaptability to be used at both small-scale and large industrial-scale [2,3]. However, the majority of research on SFE of cannabis has focused predominantly on hemp, a term commonly used to describe the varieties of *Cannabis sativa* L. that contain typically low concentrations of tetrahydrocannabinol (THC), the main psychoactive cannabinoid, within the legally regulated limits [4].

The present work aims to evaluate the SFE of cannabinoids from THC-rich cannabis inflorescences, and subsequent optimization of the operating conditions to maximize selectivity towards the main cannabinoids of interest. A series of SFE assays were conducted in a 0.5 L extractor at several pressures and temperatures. The identification and quantification of cannabinoids in the extracts were performed by HPLC using a UV-Vis detector. The results were compared to Soxhlet extractions (benchmark) using three solvents with different polarities: ethanol, dichloromethane, and hexane (non-polar, thus more comparable to carbon dioxide). Overall, these results contribute to a deeper understanding of the advantages and limitation of SFE, highlighting its potential as a more efficient and selective alternative when compared to conventional methods.

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From ecological threats to electrochemical solutions: invasive plants as unique resources for sustainable electrocatalysts

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The current global energy crisis, reflected in the depletion of fossil fuels and growth of environmental pollution has stimulated the development of novel renewable energy storage and conversion technologies. Electrocatalysis plays a central role enabling several sustainable processes for future technologies. Oxygen reduction and evolution reactions (ORR and OER) are two of the crucial energy-related processes in fuel cell and electrolyser systems [1]. For this reason, regarding the real implementation of these technologies, efficient electrocatalysis of these processes is required, stimulating the quest for new, non-expensive, and highly active electrocatalysts over the last years.

Future alternatives may include biochar, a porous carbon-rich solid material produced by the thermochemical degradation of biomass. These materials have attracted increasing interest due to their tunable physical-chemical properties, renewable feedstock, and low production cost [2].

Invasive plants like water hyacinths (*Pontederia crassipes*) and water milfoil (*Myriophyllum*) are two of the most dreadful aquatic invasive plants in Portugal, but also a valuable source of biomass. Their absorbent capacities depleting the water bodies of oxygen and nutrients may pose an advantage for the preparation of biochar.

Herein, we prepared different nitrogen-doped supported metal electrocatalysts using melamine as nitrogen precursor [3]. Then, wet impregnation was used for metal functionalization (Co, Fe). Activation under chemical/physical experimental conditions resulted in high surface areas (some $A_{BET} > 1000 \text{ m}^2/\text{g}$). The successful preparation of electrocatalysts was confirmed by XPS, XRD, Raman and SEM/EDS. The ORR and OER electrocatalytic properties were evaluated in an alkaline medium and the results showed good performance towards the reactions envisaged with some of the electrocatalysts matching the state-of-the-art Pt/C performance.

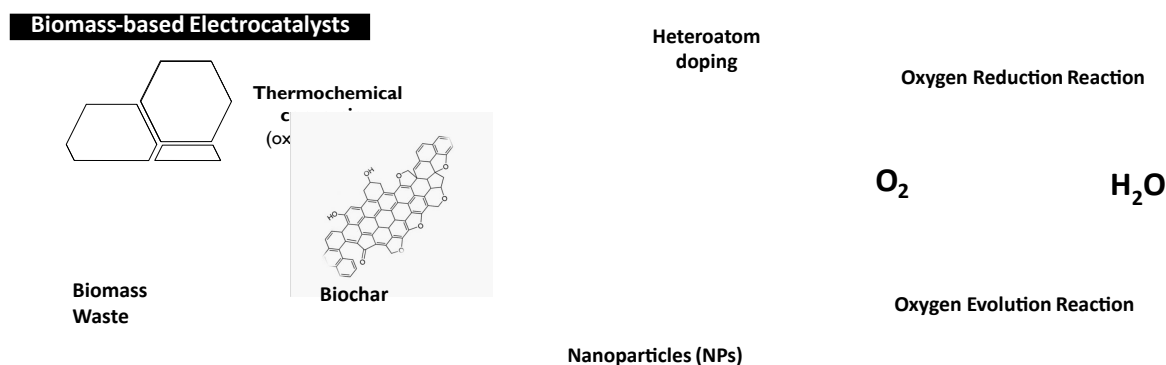


Fig.1. Schematic representation of biomass-based electrocatalysts preparation and application.

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Sertraline removal by green-based activated carbons

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Sertraline, an antidepressant, has been detected in Portuguese wastewater treatment plant influents at ng L^{-1} level concentrations [1]. This pharmaceutical compound, commonly prescribed for the treatment of depressive disorders, is characterized by its persistence and limited removal efficiency in conventional treatment processes, constituting a potential environmental hazard as these trace concentrations adversely affect aquatic organisms like bivalves such as *Dreissena rostriformis* [2].

The application of activated carbons is a promising technique for removing sertraline and other emerging pollutants. This study investigates sertraline removal (initial concentration of 3 ppm) using activated carbon derived from olive pits, a lignin-rich and underexploited biomass. The residual sertraline was quantified by HPLC using a mobile phase of 60% acetonitrile and 40% water with 0.01% TFA. A preliminary study compared two carbons produced via chemical activation (ZnCl_2 and H_3PO_4) followed by carbonization at 500°C for 1.5 hours, and a char produced by simple carbonization at 800°C for 1 hour without chemical agents. The material produced by simple carbonization was the most effective, achieving 89% sertraline removal within 3 hours.

The influence of pH on adsorption was tested at pH levels 3, 5, 7, 9 and 11, with removal efficiency increasing from 67% to 95%, respectively. The results suggest that non-electrostatic mechanisms, such as π - π stacking and hydrophobic interactions, are dominant [3]. These interactions are enhanced as sertraline ($\text{pK}_a=9.16$) becomes more neutral at higher pH values, proving more significant than the electrostatic attraction present when the carbon surface is anionic ($\text{pH}_{\text{PZC}}\approx 7$).

Kinetic assays performed at pH 7 and 11 showed no statistically significant difference, according to an ANOVA test. This is an advantage for wastewater treatment plants, which could apply the process at the pH commonly found in residual waters. The pseudo-second order model best described the adsorption kinetics at pH 7, and the Elovich model at pH 11, these systems are usually used as an indicative of chemisorption processes [3]. Table 1 presents the model applied to each experimental data, the equation of the kinetic models and the values adopted for the constants after the fitting process. The equation symbols have the respective meanings: q_t the amount of adsorbate adsorbed by the adsorbent (mg/g); q_e the amount of adsorbate adsorbed by the adsorbent at equilibrium (mg/g); k_2 the pseudo-second order rate constant ($\text{g}/(\text{mg}\cdot\text{min})$); t the contact time (min); β the desorption constant (mg/g); α initial adsorption rate ($\text{mg}/(\text{g}\cdot\text{min})$) [3].

Table 1: Kinetic non-linear fittings for experimental data at pH 7 and 11

Model	Equation	pH	Constants	R ²
Elovich	$q_t = \frac{1}{\beta} \ln(1 + \alpha\beta t)$	11	$\alpha = 9.7619 \pm 3.86$ $\beta = 1.1688 \pm 0.0839$	0.89
Pseudo-second order	$q_t = \frac{q_e^2 k_2 t}{q_2 q_e t + 1}$	7	$q_e = 6.67 \pm 0.25$ $k_2 = 0.01929 \pm 0.0046$	0.97

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Cork extracts as bioactive ingredients: antioxidant and enzyme-inhibitory potential for anti-aging and cosmetic application

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The bioactive potential of cork extracts has garnered increasing attention due to their remarkable antioxidant, anti-inflammatory, and enzyme-inhibitory properties. This study aimed to evaluate the therapeutic and cosmetic potential of cork-based formulations by analyzing the effects of different concentrations and combinations of cork extracts on anti-aging and skin-protective properties. Various formulation strategies were explored to maximize the bioactivity of cork extracts, particularly their ability to counteract oxidative stress, inflammation, and enzymatic degradation associated with skin aging.

To this end, six colorimetric assays were conducted to assess antioxidant capacity, complemented by enzymatic assays evaluating anti-elastase, anti-tyrosinase, and anti-hyaluronidase activities. The results demonstrated that the cork extracts possess significant enzyme-inhibitory activity, alongside strong antioxidant properties, revealing their potential to modulate key mechanisms involved in skin aging and pigmentation. A concentration-dependent effect was observed, with higher extract levels correlating with enhanced bioactivity. Additionally, different extract combinations influenced overall efficacy, highlighting the importance of formulation strategies in optimizing the bioactive potential of cork.

These findings confirm that cork extracts can serve as potent bioactive ingredients in cosmetic formulations, supporting cellular protection, skin regeneration, and anti-aging effects. Their ability to inhibit elastase, tyrosinase, and hyaluronidase underscores their promise for the development of innovative cosmetic products targeting oxidative stress, wrinkles, and hyperpigmentation. Further research is warranted to optimize extraction methods, formulation techniques, and bioavailability to fully harness the therapeutic and cosmetic potential of cork extracts.

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Organic acid profiling and antioxidant potential of underexploited ornamental species

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The substitution of synthetic treatments and pesticides for natural alternatives is an urgent demand in both the agri-food and pharmaceutical sectors. Underexploited ornamental and aromatic plants constitute promising sources of bioactive compounds, although their chemical and functional potential remains poorly documented. Exploring their composition provides crucial evidence for their valorization as sustainable functional agents to act as natural biocidal agents.

Therefore, in the present work, hydroethanolic extracts of *Ceanothus thyrsiflorus* "Repens", *Diosma* spp, *Juniperus* "Hughes", *Rosmarinus prostatus*, and *Grevillea* spp. were obtained by maceration (ethanol:water, 80:20, v:v). Organic acids were quantified by ultra-fast liquid chromatography coupled to a diode array detector (UFLC-DAD), while antioxidant capacity was assessed using DPPH radical scavenging, ferric reducing antioxidant power (FRAP), and total phenolic content (TPC). All the analyses were performed in triplicate, and data were statistically evaluated by one-way ANOVA with Tukey's HSD test ($p < 0.05$).

Significant compositional variability was found among species. *J. "Hughes"* displayed the highest oxalic acid concentration (2.10 ± 0.0003 mg/100 g dw) and strong antioxidant activity (DPPH: 0.256 ± 0.003 µg/mL; FRAP: 0.089 ± 0.002 µg/mL). *Ceanothus* contained the highest total phenolic content (29.0 ± 0.2 mg/g), while *Grevillea* was rich in citric acid (5.38 ± 0.04 mg/100 g dw) and phenolics (25.96 ± 0.08 mg/g). *Diosma* distinguished itself by its ascorbic acid concentration (0.3255 ± 0.0005 mg/100 g dw). Conversely, *R. prostatus* showed the lowest levels across most organic acids and antioxidant assays.

The marked diversity of organic acids and antioxidant properties highlights the biotechnological relevance of these species. Within the scope of the SUBSUS project, these results provide a solid scientific basis for developing natural plant-based biocides for pest and disease control.

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Sustainable Chemistry in textiles: solvent-based removal of dyes from wastewaters and cotton

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Rising global populations combined with the effects of climate change are intensifying the scarcity of water and other essential resources, including textiles. Traditionally, the textile industry has operated under a linear model: extracting raw materials from nature, processing them into products, and distributing these goods to consumers. This system, however, generates significant amounts of waste. Because current recycling processes remain relatively inefficient, much of this waste ends up in landfills, creating serious environmental problems. For this reason, recent efforts have increasingly focused on building a circular, zero-waste economy within the textile sector.

Two main research directions can be identified. The first involves wastewater treatment, where scientists are developing and testing new solvents that can remove contaminants—particularly dyes—from water, allowing it to be reused in production. Among these, Deep Eutectic Solvents (DESs) have shown great promise due to their ability to effectively decolorize textile wastewater while remaining environmentally friendly, since they are derived from sustainable raw materials. The second focus is on fiber recycling, with cotton being especially important. Countries such as the United States and the United Kingdom produce around 15.1 and 1.7 million tons of cotton waste respectively. Although their recycling rates are relatively higher than many others, they still only manage to recycle about 20% on average. This highlights the pressing need for new methods to improve cotton recycling, particularly sustainable techniques capable of removing dyes from cotton fibers.

Determination of new DES-DES biphasic system for metal separation

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A deep eutectic solvent (DES) is a mixture of two or more components who exhibit a significant melting point depression relative to that predicted by thermodynamic ideality, resulting in a large liquidus range. DES are composed of hydrogen bond acceptors (HBA) such as choline chloride, and hydrogen bond donors (HBD) such as organic acids or polyols. DES incorporating HBD with acidic groups along with a halide HBA significantly extended the potential for selective leaching of critical metals from various waste streams due to the change in coordination environment relative to aqueous media. However, the recovery of metals from DES is not straightforward and remains a significant hurdle to their implementation. For metals difficult to electrodeposit, this often requires the addition of a co-solvent to disturb the DES metal-ligand complex, most often water, followed by precipitation and distillation of the co-solvent prior to reuse, rendering DES reuse a difficult and expensive proposition.

The work described herein proposes a novel methodology for inducing the formation of a biphasic DES-DES system by addition of a third species. This allows for the recovery of metal cations from the leaching DES into a second phase without significantly altering the original DES phase composition and contributing towards greener metal separation processes. Malonic acid – trioctylphosphine oxide [1] and malonic acid – choline chloride [2] binary systems were chosen to illustrate the methodology due to their reported binary phase diagram as well as metal extraction and separation capabilities.[1,3] The DES-DES biphasic equilibrium is characterized through experimental and computational techniques and the partition coefficient is measured for a representative set of metals. This work presents a new alternative for the regeneration of hydrophilic DES, thereby expanding the applicability of this interesting class of solvent.

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Sustainable recovery of technology-critical elements using algae microspheres

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The European Union's (EU) reliance on other countries for critical raw materials, such as Rare Earth Elements (REEs), creates a strategic weakness. These resources power a wide range of industries, from telecommunications and sustainable industry, to even the aerospace sector. To achieve self-sufficiency and in a sustainable way, the EU is promoting the recycling of electronic waste (e-waste) to obtain REEs [1]. However, methods for extracting and recovering REEs from e-waste are frequently inefficient, characterized by low selectivity, high costs, and complex processes [2, 3]. This work proposes a novel approach based on alginate-encapsulated algal powder to extract these valuable materials from complex solutions (e.g. e-waste leachates).

Alginate microspheres containing marine algal biomass were produced using a droplet microfluidic system with ionic gelation, enabling biomass immobilization, enhanced mechanical stability, and easy recovery from solutions. The selectivity and sorption performance of the algal microspheres were evaluated in multi-element solutions containing yttrium (Y), europium (Eu), and mercury (Hg). The effects of initial metal concentration, pH, and salinity on sorption efficiency were analyzed using the Response Surface Methodology (RSM) in conjunction with a Box-Behnken Design.

The biomass of *Fucus* sp. outperformed that of *Ulva* sp. and *Gracilaria* sp. in terms of selectivity for Y and Eu: after 24 h, 77.6 % of Eu and 59.2 % of Y had been recovered by *Fucus* sp., while only 27.9 % of Hg was removed. Comparatively, the microspheres containing *Fucus* sp. removed over 90 % of both REEs, while extracting only 21.6 % of Hg from solution.

Reduced model equations developed showed a strong fit with the experimental data ($R^2 \geq 0.886$ and adjusted $R^2 \geq 0.738$) and Analysis of Variance (95% confidence level) indicated that salinity (p -value ≤ 0.002) was the most influential factor on the removal efficiency, whereas the initial molar concentration and pH showed no statistically significant effect. The model effectively pinpointed the optimal conditions for maximizing REE recovery and minimizing Hg uptake, and these predictions were subsequently experimentally confirmed. Under “optimal” conditions, encapsulated *Fucus* sp. recovered 93.7 % of Eu (predicted: 96.0 %), 87.2 % of Y (predicted: 85.8 %) and “only” 14.9 % of Hg (predicted: 19.3 %).

Further investigation will assess the material's reusability and its performance in real electronic waste leachates—both critical factors for scaling the process to industrial application.

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Shrimp shells as a multifunctional platform: from green extraction for cosmetics to catalyst production

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The valorisation of seafood processing waste is a cornerstone of the transition toward a circular bioeconomy. Shrimp shells (SS), commonly discarded as by-products, are rich in high-value biopolymers such as chitosan and a powerful antioxidant such as astaxanthin [1]. In the scope of Shrimp4NanoCosmetic project this work explores the versatility of this marine biomass in developing integrated solutions for two high-impact sectors: circular cosmetics and energy production. The Shrimp4NanoCosmetic project intends to develop a biorefinery process to recover value-added products from SS, including chitin/chitosan and astaxanthin to incorporate in new cosmetic products in collaboration with Mesosystem's company (<https://mesosystem.com/>).

Using environmentally friendly extraction methods — including mechanochemistry, ultrasound-assisted processes, and bio-based deep eutectic solvents — astaxanthin and chitosan were obtained, suitable for incorporation into bioactive cosmetic formulations. In parallel, the residual carbonized fraction of the shells was transformed into heteroatom-doped biochars and metal-supported catalysts, showing promising activity in energy-related reactions (oxygen reduction and evolution) and in furfural hydrogenation toward value-added bio-based chemicals [2]. Mechanochemistry, ultrasound assistance, organic acids (citric and maleic acid), biobased deep eutectic solvents (ChCl:levulinic acid) and vegetable oils were selected as alternatives to conventional chemical treatment. Variations of the ratio of shrimp shells to solvent, extraction time and temperature variables were study. For astaxanthin extraction the highest concentration (42.915 ± 0.022 µg/ml) was obtained with a 1:10 ratio, extraction time of 30 min, and a temperature of 60 °C. High efficiency was also obtained using ball-milling and citric acid in the recovering of chitin/chitosan. Chitin/chitosan were characterized by FTIR and SEM and compared with commercial pure chitosan.

Cobalt N-doped biochar (electro)catalysts were synthesized from shrimp shells via a solventless ball milling approach, followed by functionalization with cobalt through wet impregnation. The successful preparation of the functionalized catalysts was verified by XPS, XRD, Raman and SEM/EDS. The catalysts exhibited good electrocatalytic performance in OER, with maximum current densities of 25.65 mA cm⁻² and overpotential value of 0.52 V. In catalytic transfer hydrogenation with furfural with formic acid (80 µl), conversion reached 82.4 %, with 95.6 % selectivity towards furfuryl alcohol.

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From olive oil by-products to value: integrating phenolic content, fatty acid and nutritional composition of olive cakes for sustainable valorization

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The olive oil industry, a pillar of Mediterranean agriculture, generates large volumes of by-products that simultaneously pose an environmental challenge and represent a reservoir of high-value bioactive compounds [1]. This study integrates biochemical and nutritional data from olive cakes (OC) originating in northern Portugal, specifically in the Trás-os-Montes and Alto Douro region, obtained through different oil extraction systems: pressing (COC), two-phase centrifugation (TPOC, pitted and dehydrated), and exhausted (EOC) to assess their compositional variability and potential for sustainable valorisation.

Phenolic content revealed high variability among samples. EOC exhibited the highest concentrations of total phenols, ortho-diphenols, and flavonoids (19.61, 21.82, and 20.12 mg CAT/g, respectively), corresponding to the greatest antioxidant capacity in FRAP, DPPH, and ABTS assays (129.98, 78.00, and 56.65 μ mol Trolox/g, respectively). In contrast, COC showed the lowest values. The total tannin content was highest in the dehydrated and pitted TPOC (250.31 and 240.89 mg CAT/100 g, respectively), whereas COC had the lowest level (88.17 mg CAT/100 g). Conversely, condensed tannins showed even greater variability, ranging from 20.56 mg CAT/100 g in COC to 328.17 mg CAT/100 g in EOC. Twenty-two compounds were identified by HPLC–PDA–MS, with luteolin and verbascoside being predominant.

Chemical and nutritional analyses revealed that dehydrated TPOC presented the highest dry matter content (96.8%), whereas COC retained the largest lipid fraction (up to 14.5%). Neutral detergent fibre (NDF) exceeded 65% in most samples, with marked lignification (ADL > 23%). EOC exhibited lower crude fat ($\approx$ 1.5%) but higher crude protein (up to 7.3%). The fatty acid (FA) profile showed distinct patterns among the OCs. COC presented the highest proportion of monounsaturated FA (MUFA, 76.36 g/100 g), reflecting its residual oil content, whereas EOC displayed higher levels of saturated (SFA, 16.56 g/100 g) and polyunsaturated FA (PUFA, 14.14 g/100 g). Dehydrated TPOC exhibited an intermediate composition, balancing MUFA and PUFA fractions. Phosphorus and phytic acid contents (g/100 g) showed mean values of 0.12 ± 0.02 and 0.44 ± 0.00 , respectively, with the lowest levels recorded in EOC (0.07 ± 0.01 and 0.26 ± 0.04).

Integrating phenolic, antioxidant, nutritional, and antinutritional results highlights the potential of OC as a multifunctional raw material for agro-food, nutraceutical, and cosmetic applications. This characterisation reinforces the principles of sustainable chemistry, transforming agricultural residues into a renewable, high-value resource and converting OC waste into a driver of circular innovation [2].

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Shrimp shells powder as an efficient biosorbent for rare earth elements removal from wind turbine waste

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The global shift towards renewable energy technologies has significantly increased the demand for critical raw materials, particularly rare earth elements (REEs) like Nd, Pr, Dy, and Gd, which are key components of NdFeB magnets used in wind turbines and electric vehicles [1]. However, the supply of REEs is limited and vulnerable to geopolitical and economic disruptions, which makes their sustainable recovery more important than ever. Approximately 15–30 % of the raw materials are lost in the manufacturing of NdFeB magnets through machining and shaping waste [2]. At the end of their useful life, these magnets become waste due to loss of magnetic properties, which further contributes to material loss and environmental concerns [3]. To support circular economy initiatives and reduce dependency on primary resources, it is vital to recover WEEs from waste so that they can be reused in the production of new magnets. Within the technologies being developed for this purpose, biosorption has emerged as a promising, eco-friendly, and cost-effective strategy. Biosorbents derived from shrimp shells (waste-product of the seafood industry - 45–48 % of the shrimp is discarded as a waste [4]) have shown high capability to remove metals due to their abundant functional groups. Shrimp shell (SS) sorbents offer several advantages as low-cost, and high abundance. Utilizing such bio-waste could not only provide an efficient pathway for REEs recovery but also support waste valorization and add value to seafood industry byproducts.

This study investigates the use of shrimp shells as a low-cost alternative for the selective recovery of REEs from multi-element solutions simulating a wind turbine magnet dissolution.

A synthetic multi-element solution was prepared to simulate the composition of the nacelle component, containing B, Ni, Co, Nd, Dy, Pr, and Gd at a concentration of 100 µM. Both untreated shrimp shells (USS) and pyrolyzed shrimp shells (PSS) heated at 400, 500 and 600 °C) were investigated as biosorbents for the removal of REEs from the solution. Among the tested materials, USS demonstrated superior selectivity for REEs uptake, achieving 99 % removal of REEs while limiting the removal of competing metals, Co and Ni, to just 28 and 27 %, respectively. In contrast, PSS exhibited lower REEs removal efficiencies. Statistical analysis using response surface methodology identified initial REEs concentration and sorbent dosage as the most influential parameters for sorption efficiency, whereas pH and salinity had negligible effects. Kinetic studies further revealed that REEs sorption was rapid, reaching equilibrium within 6 hours, with a maximum sorption capacity of 80.4 mg/g. These findings highlight the potential of untreated shrimp shells as an effective, low-cost, and sustainable biosorbent for the selective recovery of REEs from complex, multi-metal solutions.

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Seaweed-based remediation of acid mine drainage: a dual benefit approach

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The growing importance of critical raw materials (CRMs) in driving the green and digital transitions is leading to a surge in global demand. Simultaneously, geopolitical instability and intensified resource competition have heightened concerns about secure and sustainable CRMs supply chains [1]. Mining practices are still the main source of CRMs and have caused serious environmental problems around the world. Recovering CRMs from alternative sources offers a promising solution, that contributes both to the security of resources and to the management of waste. Acid mine drainage (AMD) is a widespread and persistent environmental problem characterised by low pH and high metal(loid) concentrations [2]. However, AMD also represents a potential secondary source of valuable CRMs [3]. Two factors support its valorisation: the long-term persistence of AMD, creating a quasi-renewable metal reservoir, and the need to comply with costly environmental regulations. Integrating metal recovery into AMD treatment could reduce remediation expenses and support ecosystem restoration [4]. Among the potentially low-cost sorbent materials, seaweed is considered a greener, cost-effective and environmentally sound alternative with a high potential for recovering CRMs demonstrated in synthetic aqueous solutions.

This study investigates the potential of seaweed (*Gracilaria* sp. and *Ulva* sp.) to remove, concentrate, and recover CRMs from real/untreated AMD, while simultaneously improving water quality and contributing to Sustainable Development Goals No 6, 12, and 14.

AMD samples were collected from a closed Portuguese mine and characterised using Inductively Coupled Plasma Mass Spectrometry, selecting the elements of economic interest (rare earth elements (REEs), Co and Ni). Initial experiments conducted under the native conditions of AMD water (pH 2.5, salinity 2.6) resulted in seaweed mortality, highlighting the need for pH optimization. Upon adjusting the pH to 5 - the highest value at which REEs remain soluble - and testing various seaweed dosages (3, 6, 9, and 12 g/L), no mortality was observed. Under these conditions, REEs removal efficiencies ranged from 26 to 49 %, while Co and Ni were negligently removed. Subsequent experiments showed that increasing pH to 8 significantly increased Co and Ni biosorption, with seaweed biomass accumulating up to 409-fold more Co and 41-fold more Ni than the baseline concentrations.

Based on these findings, a two-stage extraction strategy is proposed: removal of REEs at pH 5, followed by removal of Co and Ni at pH 8. This approach allows efficient, cost-effective and selective recovery of CRMs, while simultaneously contributing to the treatment of AMD and supporting their valorisation for industrial reuse. This efficient, greener, and sustainable technology based on living seaweed can be easily incorporated into the current passive treatment systems, to value AMD-containing CRMs.

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How the molecular structure of hydrophobic eutectic solvents influences Lanthanides separation

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Lanthanides (Ln) are one of the most critical elements to modern society, for economic and political reasons, however only around 1% of are recycled from wastes [1]. Hydrometallurgical process consists of three steps: leaching, solvent extraction and stripping. Solvent extraction (SX) is defined as the transfer of a solute between two (immiscible) phases with different polarities, in contact with each other [2], and is the primary industrial technique for the separation of Ln. SX depends on the coordination of Ln with the extractant, which are amphiphilic molecules presenting both polar and apolar groups to enable both Ln complexation and solubility in the organic phase respectively. The similar chemistry of Ln, specially their identical stable cation state (Ln^{3+}), makes Ln separation technologically and environmentally difficult due to the lack of selectivity during SX [3], [4]. The limitation of SX selectivity based on complexation alone places renewed focus on the manipulation of secondary “soft” interaction arising from solvent restructuring to tune the selectivity[5].

In this context hydrophobic (deep) eutectic solvents (HES) were first proposed as a substitute for organic solvents in SX. Unlike most pure solvents, HES are liquid mixtures, resulting in significant melting point depressions due to strong intermolecular interactions. HESs are typically obtained by combining hydrogen bond donors (HBDs) with acceptors (HBAs) [6]. Despite their potential, HES in SX remain underutilized because they require further understanding. Research has focused on the characterization of new mixtures, neglecting that the nature of the binary interactions in the pure HES will be affected under SX conditions due to the co-extraction of polar solutes (namely water, acids, and metal ions) and subsequent rearrangement of the hydrogen bond network.

This work aims to understand the role of polar-apolar segregation in non-ionic HES, the influence of water and acid on the latter, and their relation to Ln ion partition in SX systems. To this end, we compare the extraction efficiency of different HES consisting of TOPO ($\chi=0.3$ or 0.5) and HBDs of varying polarity (acetic, hexanoic and decanoic acid). Luminescence analysis of the extracted complexes in the various HES rules out speciation as the cause of the different extraction behavior observed by changing decanoic acid to acetic acid. Instead, the greater Ln distribution in the acetic acid system are shown to depend on the increased partition of other polar solutes between phases namely water and acid, demonstrating that co-extraction of polar solutes has a direct effect on Ln partition.

Acknowledgements

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Eugenol azo dyes as eco-friendly alternatives for cotton dyeing

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The textile industry is widely recognized as one of the most polluting sectors, largely due to the extensive utilization of synthetic dyes and the environmental consequences of dyeing effluents, which often contain hazardous chemicals, heavy metals, and high levels of organic load [1]. These effluents have the potential to cause severe water contamination, ecosystem disruption, and health hazards for communities in proximity to textile manufacturing facilities [2,3]. This underscores the pressing need for more sustainable and eco-friendly dyeing methodologies.

In this context, there has been an increasing focus on alternative strategies, including the use of bio-inspired dyes and fiber modifications. Eugenol (4-allyl-2-methoxyphenol), a natural compound present in essential oils such as clove oil, contains phenolic and allylic groups, making it an ideal precursor for azo dye synthesis [4] and a renewable, environmentally friendly alternative to conventional synthetic dye precursors.

In the present work, cotton, the most widely used natural fiber, was selected as the textile substrate and eugenol-based azo dyes were tested both in cotton raw state and after pretreatments to enhance dye uptake and fiber-dye interactions [5]. Subsequently, an evaluation of the dyed fabrics was conducted for their colorimetric properties using the CIEL*a*b* system, as well as for washing and light fastness.

The results demonstrate the potential of eugenol-based azo dyes as a sustainable alternative for textile coloration, while highlighting the need for further optimization to improve their performance on cotton fabrics.

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Selective cobalt extraction from lithium-ion batteries: process optimization with saponification

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Lithium-ion batteries (LIBs) are essential as portable energy storage sources. Their increasing use amplifies the demand for efficient recycling technologies.[1] The recovery of valuable metals, such as nickel (Ni), cobalt (Co), manganese (Mn), and lithium (Li), is critical for both economic and ecological reasons. However, the diversity of battery composition makes total recovery difficult through a single process[2]. Due to the high metal concentrations in LIBs, they constitute a relevant secondary source. The combination of mechanical methods with hydrometallurgical techniques has proven to be an effective solution for their recovery.[3].

This study aimed to develop a process for optimizing the extraction of Co from inorganic leachates (obtained by treating lithium nickel manganese cobalt oxide cathodes with hydrochloric acid) using the solvent extraction method with the extracting agent Cyanex 272 in dodecane. Metal concentrations were quantified using Total Reflectance X-ray Fluorescence (TXRF) spectroscopy or Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) technique.

The study started with a synthetic solution containing Co and Ni. Various aqueous-to-organic phase ratios were tested to determine the optimal conditions for Co extraction. The results demonstrate successful Co recovery from used LIBs, with the best phase ratio identified for selective Co extraction. In addition, the study of the effect of pH indicated that the ideal range for extracting the metals involved revealed an ideal range for metal extraction between 4.5 and 6. To better simulate the diverse composition of LIBs and their impact on the process under study, the contamination of the synthetic Co and Ni solution with aluminium (Al), iron (Fe), and copper (Cu) was also tested, providing further insight into the process optimization.

Acknowledgments

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Aplicación de aditivos quirais para a mellora da eficiencia da Reacción de Evolución do Osíxeno (OER) en medio alcalino

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A electrólise da auga estase a converter nunha nova alternativa para producir H₂ sostible. Sen embargo, a reacción de evolución do osíxeno (OER) segue a ser un paso limitante que impide unha produción de hidróxeno mais eficiente.[1] Por outra parte, demostrouse que os compostos quirais poden influír nesta reacción, reducindo o sobrepotencial que debe aplicarse grazas ao efecto de selectividade de espín inducido por quiralidade (CISS).[2]

Por esta razón, unha das principais motivacións do noso grupo de investigación é obter compostos quirais estables e robustos para mellorar a OER. É por iso que escollemos os espirobifluorenos (SBFs), compostos que presentan un eixo de quiralidade axial e posúen un esqueleto ríxido e unha alta estabilidade conformacional,[3] o que os converte en candidatos prometedores para o seu uso como aditivos na OER.

Neste estudo empregáronse dietinilespirobifluoreno (DES) quirais (P₁/M₁)-DES₁ e os respectivos oligómeros macrocíclicos (P₂/M₂)-DES₂ e (P₃/M₃)-DES₃ para investigar o seu potencial en electrocatálise para a OER alcalina (Figura 1). Os electrodos de Níquel foron funcionalizados cos SBFs quirais mediante “drop-casting” e empregados para OER.

Os resultados amosan que os electrodos funcionalizados con (P₂)-DES₂ e (P₃)-DES₃ incrementan a actividade catalítica en comparación co feltro de Ni só, mentres aqueles funcionalizados cos respectivos enantiómeros dos oligómeros ((M₂)-DES₂ e (M₃)-DES₃) e o (P₁/M₁)-DES₁ empeoran a OER. É notable que ambos os dous macrociclos, (P₂)-DES₂ e (P₃)-DES₃, presentan un ECD e un *g*-factor significativamente maiores en comparación co monómero, correlacionando o *g*-factor dos aditivos quirais coa eficiencia da reacción. Por outra banda, creemos que a diferenza entre os enantiómeros (*P*) e (*M*) dos oligómeros ten relación co ángulo formado entre os seus respectivos momentos eléctricos-magnéticos de transición (ETDM e MTDM respectivamente), sendo o ángulo (Θ) <90° para o (*P*) e Θ >90° para o (*M*), o que creemos que pode ser unha nova contribución estereo-electrónica que denominamos efecto do ángulo suplementario (SAE).[4]

Prevedemos que os nosos resultados abran novas vías cara unha produción eficiente de H₂ verde, empregando SBFs quirais con *g*-factors mellorados, un obxectivo no que estamos a traballar actualmente.

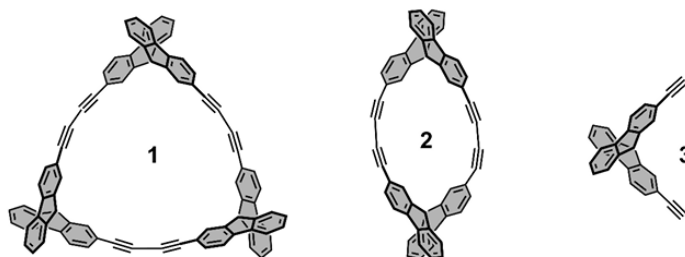


Fig.1. Estruturas homoquirais do (P₃)-DES₃, (P₂)-DES₂ e (P₁)-DES₁

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Sustainable hydrochromic materials for soil humidity monitoring

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Humidity monitoring by using sensors is essential in agriculture, food processing, environmental control, and healthcare. Recent progress in sensor technology has emphasized the need for multifunctional and sustainable materials compatible with the Internet of Things (IoT) [1].

In this context, cellulose acetate (CA), a cellulose derivative, is particularly suitable for sensors due to its biocompatibility, water insolubility, mechanical flexibility, hydrophobicity, and compliance with green chemistry principles [2]. Its combination with ionic liquids (ILs), commonly defined as salts composed of cations and anions, opens potential applications in various fields, including sensing, due to their versatility and tailorable functional response.

This work reports the development of a sustainable hydrochromic humidity-sensing material. The IL bis(1-butyl-3-methylimidazolium) tetrachlorocobaltate (II) ([Bmim]₂[CoCl₄]) was incorporated into a CA matrix. The resulting CA/IL composite exhibited a blue color at room temperature, which became transparent upon immersion in water. Increasing IL content promoted pore formation and enhanced surface wettability, thereby improving the sensitivity of the electrical response. The optimised CA/[Bmim]₂[CoCl₄]₂₀ composition showed reliable performance within the 50–90% relative humidity range.

These results demonstrate the potential of CA/[Bmim]₂[CoCl₄]₂₀ for soil moisture monitoring within the framework of Agriculture 4.0, offering a sustainable approach to addressing global challenges such as water scarcity and climate change. This study also highlights the importance of natural-based smart materials that combine strong electrical responsiveness with visual readout while meeting both environmental and technological demands.

Acknowledgments

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Valorização de resíduos de biomassa para a remoção de estrogénios em matrizes aquosas

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Os estrogénios são substâncias consideradas disruptores endócrinos, ou seja, têm a capacidade de desregular o sistema endócrino dos seres vivos. Estes compostos podem ocorrer naturalmente, como a estrona (E1) e o estradiol (E2), ou na forma sintetizada, como o etinilestradiol (EE2) [1]. Os efeitos nocivos destes fármacos manifestam-se mesmo em baixas concentrações ($\mu\text{g/L}$ ou ng/L), sendo considerados como micropoluentes [1]. Por outro lado, estas substâncias são consideradas poluentes emergentes devido à sua elevada persistência, sendo encontradas em concentrações sucessivamente crescentes no ambiente [2].

Neste trabalho, pó de cortiça *in natura* (CiN) e carbonizado a 550°C por 1 h (CC) foram utilizados como materiais adsorventes para estudos de cinética e de equilíbrio de adsorção. Os resultados experimentais dos ensaios cinéticos obtidos com os dois materiais adsorventes, assim como a sua modelação, são apresentados na Figura 1.

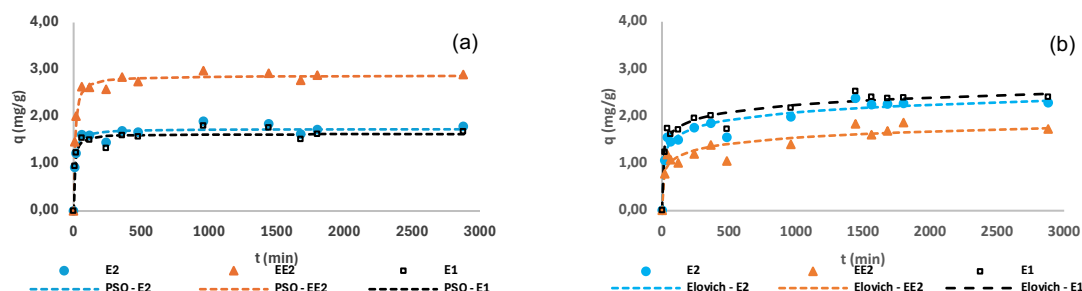


Fig. 1. Estudo cinético para (a) cortiça *in natura*, CiN e (b) carbonizada, CC.

O tempo de equilíbrio para CiN foi 6 h, enquanto para CC foi 1 dia. A cinética de adsorção seguiu o modelo de pseudo-segunda ordem (PSO) para CiN e de Elovich para CC. Como não houve um aumento significativo da capacidade de adsorção com a carbonização da cortiça, apenas o material CiN foi estudado nos ensaios de equilíbrio posteriores. As isotérmicas a 25°C são apresentadas na Figura 2.

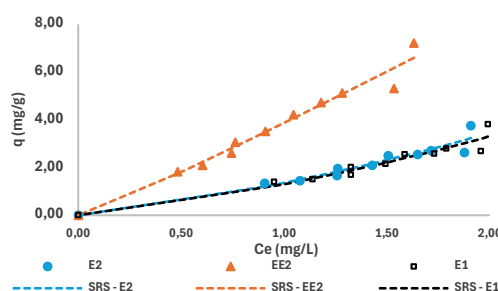


Fig. 2. Isotérmicas de equilíbrio de adsorção para CiN a $T=25^\circ\text{C}$.

A adsorção foi bem descrita pelo modelo multicomponente Sheindorf-Rebuhn-Sheintuch. Obtiveram-se remoções de 66% e capacidades de adsorção de $14,8 \text{ mg/g}$ quando se utiliza 1 g/L de adsorvente.

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Valorizing carbohydrate-rich wastewater and spent frying oil through acidogenic fermentation by mixed microbial cultures

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Frying waste generated from food processing and household kitchens had a noticeable increase over the years, raising environmental concerns when improperly disposed. Spent frying oil (SFO), a lipid-rich waste, gained attention due to its difficult treatment. In addition, the cleaning of SFO transport tanks generates wastewaters (TTWW), a largely overlooked effluent of growing concerns. Both streams contain toxic and recalcitrant compounds that require effective valorization strategies [1]. The aim of this work is to explore the possibility of using SFO and TTWW as substrates for acidogenic fermentation (AF) to produce short-chain organic acids (SCOAs). SFO consisted predominantly of triacylglycerides (97.9%), mainly esterified oleic (49.1%), linoleic (26.6%) and linolenic (14.5%) acids, with a high chemical oxygen demand (COD) of 1032.14 g COD/L. TTWW exhibited a carbohydrate-rich composition (67%), with glucose as the main constituent, and a COD of 90.04 g COD/L. TTWW also had a content of 7.58% volatile solids (VSS) and 9.34% total solids (TSS), with a total nitrogen concentration of 1.990 g/L, of which 0.229 g/L were characterized as nitrates. Sludges collected in two different treatment plants were tested as inocula, two aerobic (AES and AESER) and two anaerobic (ANS and ANSER). Experiments were conducted with varying food-to-microorganism (F/M) ratios of 1:1, 1:2, 2:1 and 3:1 g COD/g VSS. Additional assays were performed with pH control at 5.5 and 10.5, using buffer solutions, and ANS as inoculum, at a F/M ratio of 1:2, to assess its effect on SCOAs production and variability. All assays led to significant SCOAs production. TTWW-based fermentations achieved the highest acidification degrees (46-209%), generating diverse SCOAs mixtures of acetic (27-54%), propionic (18-42%), butyric (18-34%) and valeric acids (7-10%). SFO-based experiments mainly produced acetic and propionic acids. pH-controlled experiments broadened the SCOAs spectrum, with increases in valeric and iso-butyric acids, though overall yields remained comparable to uncontrolled trials. Future work will focus on scaling up AF in bioreactor to maximize SCOAs production and integrate it in a three-step process for the synthesis of polyhydroxyalkanoates (PHAs). PHAs are biodegradable biopolymers with similar physicochemical properties to those of fossil-fuel based plastics. Overall, this work demonstrates the potential of biologically valorizing of recalcitrant waste streams, generating added-value products. Such an approach provides an environmentally sustainable valorization route, reduces waste, and aligns with circular economy principles.

Acknowledgments

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Optimizing protein extraction and recovery from *Hermetia illucens*

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The urgent need to identify alternative protein sources for improved food sustainability, as well as the substantial economic and environmental costs of conventional protein sources, has cast a light on insects as a promising alternative [1]. Insects provide significant amounts of energy, protein, essential amino acids and micronutrients, while requiring less feed, water, and land for the production of 1 Kg of edible protein compared to livestock such as poultry or cattle [2]. Accordingly, this work aimed to develop a more efficient and sustainable technology for the extraction and purification of proteins from insect flour, specifically from *Hermetia illucens* (Black soldier fly, BSF), with the ultimate goal of incorporating them into several innovative food products.

Conventional solvents already in usage for protein extraction, such as sodium hydroxide (NaOH) [3], were initially applied for the extraction and recovery of proteins from different BSF flours (defatted and non-defatted samples). Several operational parameters were optimized, assisted by response surface methodology (RSM), including extraction time, temperature, solvent concentration and solid-to-liquid ratio. Protein content and profile of the extracts were assessed by the Bicinchoninic acid assay (BCA) and SDS-PAGE, respectively. Based on the results obtained, it is possible to demonstrate the development of a highly efficient and more sustainable extraction process, surpassing previously reported findings [3,4]. Ongoing work continues to optimize operational conditions to maximize the efficiency of protein recovery.

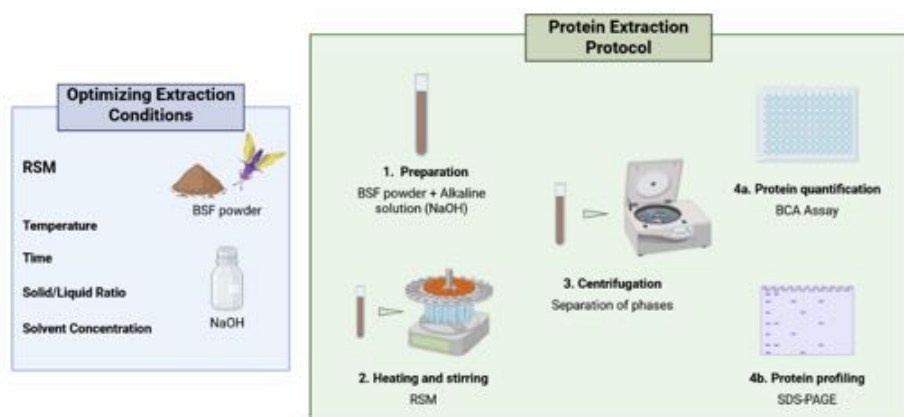


Fig.1. Graphical representation of the protein extraction process.

Acknowledgments

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Hydrometallurgical recycling of lithium-ion batteries - Achieving metal separation through extraction with deep eutectic solvents

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The rapid expansion of electric mobility and energy storage technologies has created an urgent need for efficient recycling strategies for spent lithium-ion batteries (LIBs). These batteries contain considerable concentrations of valuable metals such as lithium (Li), cobalt (Co), manganese (Mn), nickel (Ni) and aluminum (Al) [1]. However, the presence of metals such as copper (Cu) and iron (Fe), often considered contaminants in the recycling process, can diminish the reliability of current recycling processes. As such, the development of efficient recycling techniques can reduce the reliance on primary mining and mitigate one of the EU's largest concerns regarding the management of critical raw materials: the overreliance on importation. While effective, conventional hydrometallurgical approaches require high consumption of concentrated acids and reducing agents, multiple separation and precipitation stages, and the use of harmful organic solvents. These limitations highlight the necessity for more sustainable alternatives.

In this work, we explore the application of deep eutectic solvents (DES) as tunable alternative media for the extraction and separation of critical metals from LIB leachates. More specifically, with combinations of trioctylphosphine oxide (TOPO) with dibutylthiourea (DBT) as the organic phase, selective extraction of Cu and Fe, common contaminant metal ions present in LIB leachates, was achieved when the aqueous phase was comprised of HCl 2 M (Figure 1). Due to a strong deviation from the ideal melting point, the DES became liquid at room temperature, meaning that the extraction could be performed without additional heating. Each of the components of the organic phase also demonstrated specific metal targeting, with TOPO being responsible for the removal of Fe, and DBT removing mostly Cu. While optimization is ongoing regarding viscosity control, stripping efficiency, and reusability, these findings show that the selected DES is a promising candidate for an efficient hydrometallurgical approach to the recycling of LIBs at room temperature. By combining reduced environmental impact with selective recovery of critical raw materials, DES-based processes could play a key role in enabling a circular economy for used lithium-ion batteries.

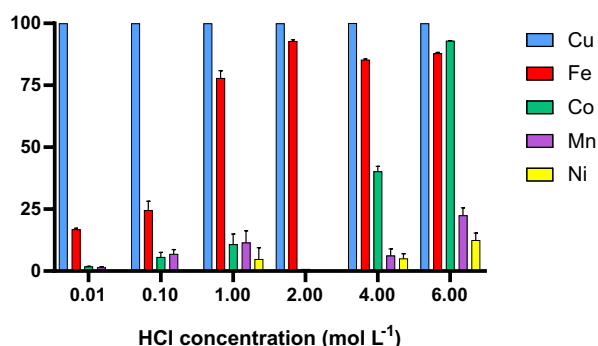


Fig.1. Efficiency of extraction for several metals (initial equimolar concentration of 0.01 M) from an aqueous phase (HCl 0.01 – 6 M) by a DES composed of TOPO and DBT ($X_{\text{TOPO}} = 0.5$).

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Effect of ball-milling conditions on the phenolic composition and antioxidant capacity of avocado seed flour

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The valorization of agro-industrial by-products, such as the avocado (*Persea americana* Mill.) seed, is an important approach for developing sustainable and high-value functional ingredients, in line with the principles of the circular bioeconomy. In this work, the effect of different ball-milling conditions on the physical characteristics, phenolic composition, and antioxidant capacity of avocado seed flours was investigated. The samples were milled under controlled variations of rotational speed and powder-to-ball ratio, followed by chemical characterization through HPLC-ESI-MS to identify individual phenolic compounds. Complementary spectrophotometric assays were used to quantify total phenolics, ortho-diphenols, total flavonoids, and antioxidant activity (ABTS, DPPH, and FRAP). The results showed that milling intensity had a clear impact on the stability of phenolic compounds. Moderate milling conditions promoted the preservation of highly polymerized procyanidins and glycosylated flavonols, which were associated with stronger antioxidant activity. Conversely, more intense milling led to structural breakdown and oxidation of phenolics, resulting in a pronounced loss of bioactivity. Samples obtained under milder conditions (Control and Seed 5) exhibited total phenolic contents close to 40 mg GAE/g and the highest antioxidant values, whereas the most severe treatments reduced these levels by more than 60%. Chromatographic profiles confirmed the predominance of oligomeric procyanidins (dimers, trimers, and tetramers) in moderately milled samples, while highly milled flours showed fragmented and oxidized derivatives. Overall, the findings highlight milling as a decisive technological step influencing both the preservation and functionality of phenolic compounds. Adjusting operational parameters, such as speed and ball-to-powder ratio, can therefore enhance the recovery of bioactive molecules from avocado seeds, promoting their use as a sustainable source of natural antioxidants for food, nutraceutical, and cosmetic formulations.

Note: The results presented here are part of a broader study currently submitted to an international high-impact journal focusing on the technological valorization of avocado by-products.

Suzuki coupling in pyrrolo[4,3,2-*de*]quinolinone derivatives: synthesis and computational studies

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Suzuki coupling is a well-known reaction for which various experimental conditions are described in the literature [1,2]. This diversity often makes it challenging to identify the most suitable ones for specific compounds, such as complex heterocyclic compounds. A striking example is pyrrolo[4,3,2-*de*]quinolinones (Figure 1), whose electronic features can influence their reactivity. In this work, we attempted to rationalize the conditions that optimize the yield of the synthesis of pyrroloquinolinone (PQ) derivatives by Suzuki coupling, using a multidisciplinary strategy combining synthesis and computational chemistry. The PQ core with the halogen in position 6 was synthesized, following the methodology reported by Yang *et al.* [3], in three steps, and an overall maximum yield of 26% (chlorine) and 49% (bromine). Density Functional Theory (DFT) calculations were then employed to investigate the underlying factors governing the mechanism of the Suzuki coupling on this scaffold, focusing on the effect of solvent (1,4-dioxane, dimethoxyethane, and methanol), the position of the coupling (5 vs 6), and the halogen (Cl vs Br) (Figure 1). The computed energy profiles revealed that the solvent polarity impacts the relative energies of key intermediates and transition states. Indeed, in the oxidative addition step, methanol was predicted to lower reaction barriers and favor product formation. However, in the subsequent reductive elimination step, dimethoxyethane induced a decrease in the energy barrier of 4 kcal/mol. Regarding the halogen effect, we observed that regardless of the position, changing bromide to chlorine facilitates the reaction, as expected. On the other hand, changing the position of the halogen affects both the oxidative addition and the reductive elimination steps: the halogen in position 5 is favored in the latter, and unfavorable in the former. These *in silico* predictions were validated through small-scale reactions with the core, with the halogen in position 6. Then, monitored by HPLC-MS, which partially confirmed product formation under the simulated conditions. However, experimental trials also indicated that methanol may induce product degradation. Additionally, when the boronic acid was changed, predicted conditions did not work as expected. After a Bayesian analysis [4] of the features of the reactions, we concluded that the temperature of the reaction was the most relevant aspect (excluding the boronic acid). Therefore, the combination of solvent/temperature were adapted to obtain the final compounds with yields varying between 22-84%.

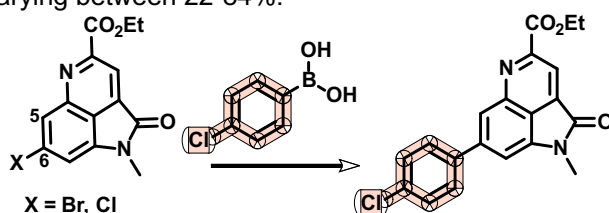


Fig.1. General scheme for the Suzuki Coupling of pyrroloquinolinones. Coupling in position 6 is shown, though position 5 was also explored *in silico*.

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Enhancing artemisinin extraction with natural compounds and evaluation of the antimalarial activity

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With 263 million cases reported in 2023 across 83 endemic countries, malaria remains a significant global public health challenge. The African continent accounts for 95% of the 597,000 deaths, 76% of which occur in children under five years of age. Although recent advances include new vector control strategies, insecticide-treated bed nets, and the introduction of complementary vaccines, oral drugs remain essential for treatment. Among these, artemisinin-based combination therapies (ACTs) stand out due to their high efficacy and fast action [1]. Nevertheless, the emergence of parasite resistance, coupled with the inherently low aqueous solubility of artemisinin, compromise its bioavailability, limiting oral absorption and requiring higher therapeutic dosages.

These limitations, combined with malaria's persistent global prevalence, have driven an increased demand for artemisinin. Its industrial extraction is commonly carried out with hazardous solvents such as hexane, petroleum ether, or halogenated compounds, which pose risks to both the environment and human health. As an alternative, researchers have been investigating greener solvents, including ionic liquids, biosolvents, and natural *deep* eutectic solvents [2]. However, the wide range of available options makes experimental screening unfeasible. Therefore, computational methods such as the COnductor-like Screening MOdel for Realistic Solvents (COSMO-RS) are often used to predict molecular interactions and identify the most suitable candidates [3].

In this study, COSMO-RS was employed to investigate the interactions between various solvent families and antimalarial compounds, including artemisinin, quinine, quinidine, tetracycline, artemether, dapson, and pyrimethamine. The best-performing natural excipients, namely terpenes, were then employed for artemisinin extraction from *Artemisia annua* L. In addition, the antimalarial activity of the reconstituted extracts was assessed against *Plasmodium falciparum*. The results showed that terpene-based extractions outperformed conventional solvents such as dichloromethane. Furthermore, terpene extracts exhibited IC₅₀ values comparable to those of the standard artemisinin, highlighting their potential as sustainable candidates for the development of novel antimalarial formulations.

Acknowledgements

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Deployment of an energy harvester prototype in an urban environment location

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This work belongs to the Symposium Energy Production and Storage, promoted by the Working Group on Sustainable Energy of the CRUSOE network.

Wind excited energy harvesters are devices that transform the kinetic energy in the wind into oscillatory mechanical energy, which is transformed into electrical energy through electromagnetic or piezoelectric effects. Multiple prototypes have been studied over the years; however, their industrial penetration has been so far very limited. One of the main barriers faced by this technology is the uncertainty associated with its performance in real applications. Most of the research conducted so far has focused on optimizing the performance of a certain prototype based on wind tunnel test and, in some cases, CFD (Computational Fluid Dynamics) applications [1, 2].

A prototype of wind-excited piezoelectric energy harvester has been deployed on the roof of the Area Científica building of the University of A Coruña. The device has been exposed to wind conditions representative of an urban environment. The wind characteristics were sampled continuously during one month by a 3D sonic anemometer, recording the power output of the harvester as well. The prototype demonstrated its ability to generate electricity over extended periods of time. Although there was a general correlation between wind velocity magnitude and power output, the variability in the natural wind prevented the identification of a clear linkage between these two variables.

Aiming at providing a more comprehensive description of the intimate relationship between wind and power output, several Machine Learning (ML) models were trained to improve the understanding of the energy harvester's performance. It was found that local flow features at the location of the harvesters played a decisive role in the power output. This signals the importance of considering the local flow characteristics for the assessment of the power generation of energy harvesters deployed in urban environment.

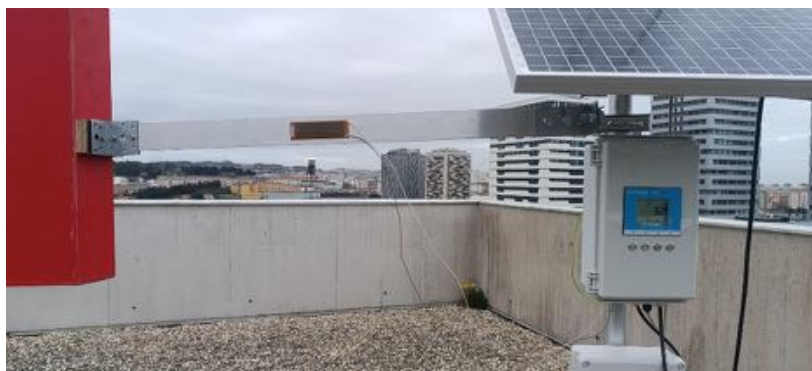


Fig.1. Prototype of the wind-excited piezoelectric energy harvester deployed at the Campus de Elviña of the University of A Coruña.

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Empowering the internet of things through sustainable energy harvesting: the inanoEnergy approach

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inanoEnergy is a technology company focused on developing advanced energy harvesting solutions for self-powered IoT systems. By converting ambient energy sources into electrical power through micro- and nanogenerators—such as thermoelectric and piezoelectric devices—the company enables autonomous and maintenance-free sensor networks for a wide range of industrial and environmental applications.

In the IoT domain, inanoEnergy's technologies empower distributed sensors and smart devices to operate reliably in harsh or remote environments without the need for batteries or wired connections. Their applications span several sectors, including:

- Oil & Gas: downhole power generation for deep-well sensors operating under extreme conditions.
- Water management: continuous monitoring of water distribution networks using locally powered sensors.
- Waste management: connected sensors in containers for optimized logistics and collection routes.
- Blue energy: harvesting ocean wave energy to power buoys and coastal monitoring systems.
- Smart textiles and footwear: integration of energy-generating materials for wearable electronics.

By merging sustainable energy harvesting with IoT architectures, inanoEnergy is paving the way for energy-autonomous smart networks, ensuring continuous monitoring and communication even in inaccessible areas. This synergy between nanotechnology and IoT represents a key step toward scalable, self-sufficient, and intelligent sensing ecosystems.

Acknowledgments

This work acknowledges the contribution and technological developments carried out by inanoEnergy within several innovation projects focused on sustainable energy harvesting and self-powered IoT systems. The Energy4Oil project, part of ATTRACT Phase 1, received funding from the EU's Horizon 2020 Research and Innovation Programme under Grant Agreement No. 777222. The PiPe 4.0 project was funded through ATTRACT Phase 2 of the EU Horizon 2020 programme under Grant Agreement No. 101004462 and represents a collaboration including Pietro Fiorentini S.p.A. and inanoEnergy for smart gas monitoring. The I.nano.WEC project was supported by Portugal's Fundo Azul (Blue Fund, via the Direção-Geral de Política do Mar) under project reference FA_02_2017_002, aiming to develop wave-energy converters using nanogenerators. Finally, the PORTOS (Ports Towards Energy Self-Sufficiency) project was co-financed by the Interreg Atlantic Area Programme through the European Regional Development Fund, under grant number EAPA-784/2018 led by FEUP with inanoEnergy as a partner to promote renewable energy implementation in Atlantic ports.

Triboelectric nanogenerators for energy harvesting in remote and harsh environments

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The world faces the challenge of finding new sources of renewable and clean energy to reduce carbon emissions to counteract global warming and the current energy crisis. Triboelectric nanogenerators (TENGs) are an emerging technology capable of converting mechanical and thermal energy into electricity across a wide range of environments [1,2]. Their simplicity, high output, scalability, and adaptability make them particularly suitable for powering autonomous sensors, wearable devices, and remote monitoring systems, where conventional energy sources are limited or impractical.

This work demonstrates the broad potential of TENGs by exploring their performance in diverse and challenging energy harvesting scenarios. In fluid flow environments, a rotary TENG was developed to harvest energy from water, achieving voltages exceeding 100 V and generating sufficient power to feed sensors, charge capacitors, and light up LEDs, demonstrating its suitability for autonomous monitoring in water networks [3]. For marine applications, rolling-sphere TENG architectures were integrated into floating structures, where laboratory and large-scale wave basin experiments demonstrated their ability to harvest energy from realistic sea states [4]. Results highlight the dependence of electrical performance on hydrodynamic response and buoy geometry, with optimized configurations reaching voltages up to 70 V and proving their viability for powering navigational and environmental sensors in remote waters.

Having demonstrated efficient energy harvesting in water environments, TENGs were next evaluated under extreme thermal and chemical conditions to assess their robustness. Their performance was studied in methane and crude oil at pressures up to 830 bar and temperatures up to 120 °C [5], where devices maintained energy generation, confirming their suitability for oil and gas applications. Additionally, a hybrid triboelectric/thermomagnetic device was developed, capable of converting low-grade heat into mechanical oscillations and subsequently into electricity [6]. This system achieved power outputs more than 35× higher than conventional thermomagnetic generators, further expanding the range of accessible energy sources.

Overall, the results highlight the suitability of triboelectric nanogenerators for energy harvesting in remote and hostile environments, paving the way for their integration into next-generation Internet of Things technologies and opening new avenues for autonomous and renewable energy systems.

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Piloto virtual GIS de la cadena de valor de la eólica offshore de Galicia y Norte de Portugal

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Siendo la energía eólica offshore una de las fuentes de energía renovable marcadas como objetivo 2030 en materia de lucha contra el cambio climático por ambos países, España y Portugal, y debido al tipo de batimetría existente en la plataforma oceánica, las soluciones más probables para el aprovechamiento energético serán las de tipo flotante que aún están en fase de investigación, desarrollo o explotación precomercial como en [1] o en [2] para asegurar la viabilidad técnica, económica y medioambiental [3].

Dentro del proyecto POCTEP Interreg AOWINDE donde se busca dar un apoyo a la cadena de valor de la industria de la eólica offshore asentada en la región de Galicia y Norte de Portugal se ha realizado un análisis de la cadena de valor de la eólica marina para conocer la importancia que esta industria tiene en la eurorregión.

Se ha elaborado una herramienta tipo GIS para analizar las entidades de la cadena de valor y observar los puntos fuertes y débiles en la eurorregión, estimar las características que pueden tener los proyectos industriales en las áreas legalmente asignada para la eólica marina, áreas POEM y SPOEM, de España y Portugal respectivamente, las inversiones económicas, carga de trabajo y flujo de material que pueden suponer para cada país. En la Figura 1 se muestra un mapa a modo de ejemplo de un posible caso de aplicación de esta herramienta.

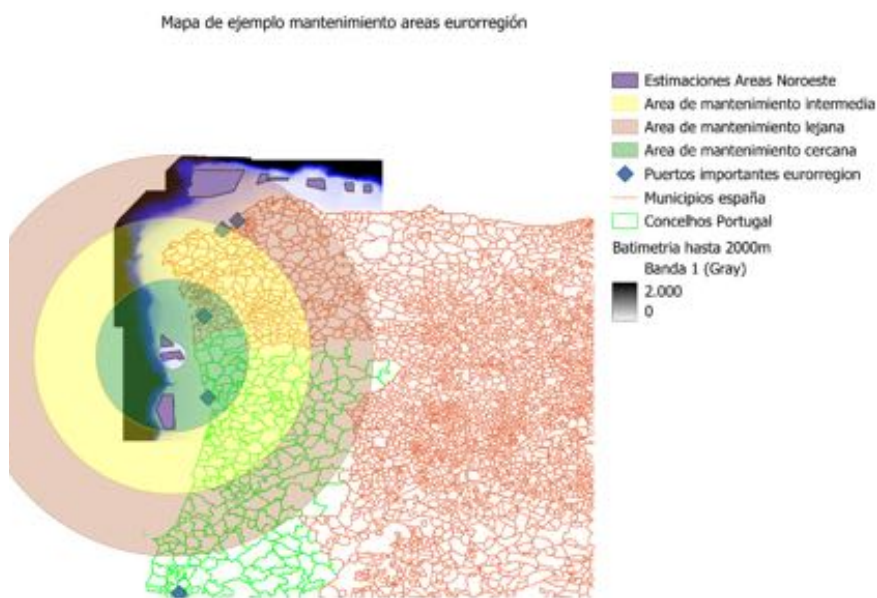


Figura 1, mapa de ejemplo

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Inverted flexible perovskite solar modules for sustainable urban integration

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Transitioning to renewable energy is essential for mitigating climate change, and solar power offers a carbon-free and geographically versatile solution. However, conventional crystalline silicon cells remain limited by high production costs, environmental impact, and reduced ability to harvest diffuse light [1]. Perovskite solar cells (PSCs) have emerged as a disruptive alternative due to their low fabrication costs, high power conversion efficiencies, scalability, and enhanced performance under diffuse illumination. Their tunable transparency and color, flexibility and lightweight nature make them particularly well-suited for integration into urban environments, such as buildings and street furniture [1-2].

Scaling PSCs into larger devices involves developing perovskite solar modules (PSMs). These are fabricated by subdividing large-area devices into narrow subcells interconnected in series via laser scribing steps (P1, P2, and P3) designed to minimize electrical losses. Specifically, the P1 scribe defines the bottom electrode, while P2 and P3 are scribed before and after the deposition of the top electrode, respectively [3].

This work presents a prototype integrating inverted flexible PSMs designed to meet the electrical requirements of a bus stop equipped with an advertising display. The proposed system demonstrates a sustainable and aesthetically compatible solution for decentralized energy generation in urban spaces. Each PSM comprises three interconnected subcells, with an active area of 5 cm² per module. The fabricated PSMs exhibited an open-circuit potential of 3 V and a power output of 10.33 mW·cm⁻² under 1 sun (100 mW·cm⁻², AM 1.5 G, room temperature). Multiple modules were assembled within the prototype to satisfy the energy demands of the installation. Moreover, due to the sensitive features of the flexible substrates, a careful optimization of the laser parameters for each scribe in PSMs was performed to prevent the damage induced by laser heat and radiation dispersion of the films along the scribed lines.[4] Overall, this work demonstrates the viability of flexible inverted PSMs as scalable, design-integrated power sources for next-generation smart cities.

Acknowledgments

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Flexible thermally-chargeable supercapacitors with a flavylum derivate solid-gel electrolyte: thermoionic & energy storage performance evaluation

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The development of wearable electronics and flexible devices demands compact, lightweight, and sustainable energy storage systems capable of autonomous operation. Thermally-chargeable supercapacitors (TCSCs) offer a promising solution by combining thermal energy harvesting with electrochemical energy storage. However, they face challenges such as low energy conversion efficiency and limited energy density [1]. 4'-Hydroxy-7-diethylaminoflavylum (7N), a natural flavylum derivative known for its photoluminescence, pH sensitivity, and chemical stability [2], is of interest as an electrolyte additive for its structure and photophysical properties that may enhance ion transport.

In this work, planar TCSCs were fabricated using knitted cotton coated with commercial carbon nanotubes (CNTs) as the electrodes, and poly(vinyl alcohol) (PVA)/H₃PO₄ solid-gel electrolyte doped with different loadings of 7N (0–3.5 wt% relative to PVA). The electrochemical performance was evaluated by electrochemical impedance spectroscopy, cyclic voltammetry, and galvanostatic charge-discharge measurements. Additionally, the thermoelectric performance of each device was assessed under different temperature gradients. The planar device containing 1.5 wt% 7N within the electrolyte exhibited the highest capacitance of 170 $\mu\text{F cm}^{-2}$, representing a 113% improvement over the reference device with undoped electrolyte. The device based on the electrolyte with 2.5 wt% 7N (PVA_7N_2.5) achieved maximum thermoelectric voltages of 14 mV and 39 mV at temperature gradients of 8 K and 35 K, respectively. This device also displayed the highest Soret coefficient of 1.05 mV K^{-1} , corresponding to a 20.7% increase relative to the reference device (Fig.1). This work presents an innovative approach for smart, energy-autonomous textiles, combining energy harvesting, storage, and fluorescence response (under UV light) for next-generation wearable devices.

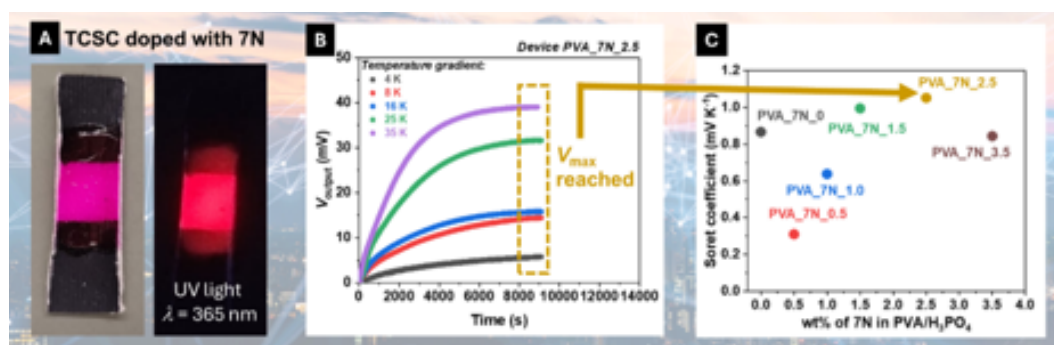


Fig.1. (A) TCSC using solid-gel electrolyte doped with 7N under visible and UV light - $\lambda = 365 \text{ nm}$; (B) Output voltage measured over time at different temperature gradients for PVA_7N_2.5; (C) Soret coefficients vs. loading of 7N in the electrolyte (right).

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New carbon/mixed-valence metal oxide nanohybrids for thermal energy harvesting and electrochemical energy storage

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In recent years, there has been a growing demand for new alternatives to fossil fuels through technologies capable of capturing and storing energy. These technologies are interconnected with the domain of the Internet of Things (IoT), particularly through wearable systems that integrate energy harvesting and storage components into textiles. Such devices allow efficient energy management and support the connectivity and autonomous operation of smart clothing. Thermally-chargeable supercapacitors (TCSCs), which convert thermal energy into electrochemical energy and simultaneously store it, represent a promising strategy for self-powered wearable electronics [1-3].

Herein, new multifunctional energy harvesting/storage textile devices were fabricated using oxidized multiwalled carbon nanotubes (CNT-ox) hybridized with bi- (Bi_2WO_6 – BW) and pentametallic bismuth oxides (Bi:W:V:Mo:Fe – HEM) as electrode nanomaterials and a solid-gel polymer electrolyte of poly(vinyl alcohol)/ H_3PO_4 (Fig. 1). The bismuth oxide-based nanomaterials were grown *in situ* onto the CNT-ox surface through an optimized hydrothermal route and comprehensively characterized by X-ray diffraction, Raman spectroscopy, scanning electron microscopy-energy dispersive X-ray spectroscopy, transmission electron microscopy, chemical analyses and X-ray photoelectron spectroscopy.

Textile electrodes were prepared by blade coating of an ink of CNT-ox or hybrid onto cotton fabric and then assembled into TCSCs with asymmetric multilayer configuration. Both devices demonstrated a hybrid energy storage mechanism combining faradaic and non-faradaic processes. The BW@CNT-ox/CNT-ox device exhibited the highest power density ($630.1 \mu\text{W cm}^{-2}$), while HEM@CNT-ox achieved the highest energy density ($2.34 \mu\text{W h cm}^{-2}$) and excellent long-term stability, retaining 99% of its capacitance after 5000 CV cycles at 25 mV s^{-1} . Finally, the HEM@CNTox//CNT-ox was evaluated through thermionic tests by applying four temperature gradients, resulting in output potentials of 1.67, and 7.55 mV for temperature gradients of 6.50, and 9.75 K, respectively, yielding $\Delta V/\Delta T$ of 0.257, and 0.774 mV K^{-1} , respectively.

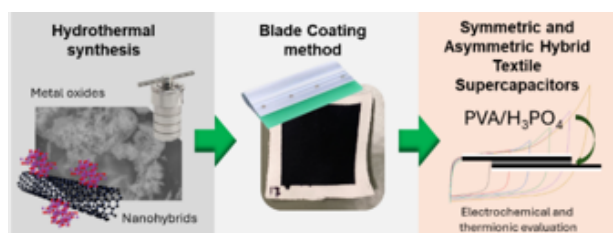


Fig.1. Process of fabrication and performance evaluation of a hybrid textile supercapacitor.

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Boosting CO₂ hydrogenation to methanol efficiency through carbon–metal oxide synergy

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Over the past century, growing industrialization and globalization have driven up carbon dioxide (CO₂) emissions, causing serious climate-related impacts on Earth [1]. Carbon Capture, Storage, and Utilization (CCSU) technologies represent an effective strategy to mitigate CO₂ emissions while enabling the conversion of CO₂ into valuable products such as methanol [2]. Among these, CO₂ hydrogenation to methanol is particularly attractive, as methanol can also serve as a clean and versatile alternative fuel. Nevertheless, the most efficient catalyst for this reaction is still actively sought [3].

Bimetallic catalysts comprising Copper (Cu) and Zinc (Zn) nanoparticles supported on pristine Activated Carbon (AC) and Carbon Nanotubes (CNT), and on composites containing varying proportions of AC or CNT, and Aluminium Oxide (Al₂O₃) were developed. The catalysts were prepared by incipient wetness impregnation, and the composites were synthesized by a ball milling treatment. The catalytic experiments were conducted at 250 °C and 30 bar ($GHSV = 24\,000\text{ cm}^3\text{ g}^{-1}\text{ h}^{-1}$) using a Microactivity XS-15 reactor from PID Eng & Tech. The gaseous products obtained were analysed with an Inficon Micro GC, and the liquid products were examined using a DANI GC 1000. The catalysts and supports were extensively characterized by various techniques.

The CNT-supported catalyst demonstrated superior performance compared to CuZn/AC, achieving a CO₂ conversion of 23% and a methanol selectivity of 33%, whereas CuZn/AC showed only 3% of CO₂ conversion and no methanol formation. Despite the CuZn/AC catalyst exhibiting the highest specific surface area of all catalysts developed, its catalytic activity improved only when combined with Al₂O₃, more specifically for the CuZn/AC–Al₂O₃ (50:50) and CuZn/AC–Al₂O₃ (70:30) catalysts. This enhancement is likely due to increased mesoporosity, which facilitates mass transport, and to a possible synergistic effect between AC and Al₂O₃. Regarding the catalysts supported on composites containing CNT, CuZn/CNT–Al₂O₃ (90:10) exhibited a catalytic performance comparable to that of CuZn/CNT, with slightly lower CO₂ conversion but higher methanol selectivity. It also outperformed the CuZn/CNT–Al₂O₃ (50:50) and CuZn/CNT–Al₂O₃ (70:30) catalysts. These improvements were attributed to stronger metal–support interactions, higher Cu and Zn loadings, and a particle size similar to that of CuZn/CNT.

These preliminary results demonstrate the potential of integrating carbon materials with metal oxides in composite supports to achieve efficient CO₂ hydrogenation to methanol.

Acknowledgements

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COSMO + AI for screening of solvents: case study of methanol synthesis

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The conversion of carbon dioxide (CO₂) into methanol offers a promising strategy for sustainable energy storage and chemical production. However, the process is thermodynamically limited by equilibrium constraints. One solution is the use of ionic liquids (ILs), which can selectively extract methanol and water *in situ*, thereby shifting the equilibrium and enhancing overall conversion. Given the vast chemical diversity of ILs, efficient screening methods are essential.

Here, we integrate ILBERT, a BERT-based chemical language model for predicting twelve properties of ILs, with COSMO-RS thermodynamic calculations to identify ILs with optimal characteristics for high-temperature CO₂ hydrogenation. We screened 19.5 million ILs—generated from combinations of diverse cation and anion families—using ILBERT to predict decomposition temperature (T_d), melting point (T_m), and viscosity (η). From this dataset, 20,000 top candidates were selected based on predicted infinite dilution activity coefficients (γ^∞) for methanol and water. These were further refined using COSMO-RS activity coefficient predictions across 298–513 K. For ranking, weighted scores were assigned: $\gamma^\infty(\text{MeOH}) = 5$, $\gamma^\infty(\text{H}_2\text{O}) = 4$, $T_d = 2$, $T_m = 1$, and $\eta = 1$.

Analysis of the screened cation–anion combinations revealed that **phosphonium-based cations were most prevalent among the top-ranked ILs**, primarily due to their affinity with methanol/ water and high thermal stability. **Imidazolium and ammonium cations** also showed good performance across these criteria. Among the anions, **fluorinated sulfonates, phosphates, and sulfonyl imides dominated**.

This combined AI–thermodynamic framework accelerates the discovery of functional ILs for efficient CO₂-to-methanol conversion, enabling high-temperature operation and continuous product removal.

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Designing Fe–N–C single-atom catalysts using carbon black for efficient oxygen reduction reaction

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The oxygen reduction reaction (ORR) is one of the hottest reactions in sustainable energy technologies. This reaction is crucial for both fuel cells and metal-air batteries, which are essential for enabling the transition to a carbon-neutral energy future. Nonetheless, the ORR presents sluggish kinetics that significantly hinder device efficiency, posing a critical challenge in developing highly active and durable electrocatalysts. Moreover, platinum and its alloys are the most commonly used ORR catalysts to overcome these limitations, but they are very scarce and expensive, thereby increasing the overall device cost. Therefore, large research efforts have been devoted to the development of noble metal-free ORR electrocatalysts, including those from our group [1–4]. In this sense, the combination of iron and nitrogen on carbon supports surges as an optimal candidate due to their adjustable active sites and high electrocatalytic activity towards the reaction. Carbon materials are considered excellent candidates for the replacement of Pt, as they generally present a more competitive price and exhibit high versatility. In this context, this work focuses on using different carbon blacks (CBs) as support for ORR catalysts. They bring several advantages due to their high electrical conductivity, which ensures efficient electron transport, and their high surface area and tuneable porosity, which allow a uniform dispersion of active sites and facilitates the mass transport during the ORR [3, 5]. Additionally, the surface defects in the CBs provide anchoring points for coordinating iron and nitrogen species, promoting the formation of highly stable single-atom Fe-N_x active centres [3]. Moreover, their chemical robustness under electrochemical conditions ensures long-term durability, while their low cost and scalability make them highly attractive for practical fuel cell and metal–air battery applications. These combined properties make CBs a promising carbon support to develop next-generation, non-precious metal ORR catalysts. In the present study, selected CBs were modified by adapting a previously reported methodology, which has been shown effective to incorporate nitrogen-coordinated single-atom iron into different carbon frameworks by using only broadly available and low-cost precursors [3, 4]. The successful incorporation of iron was confirmed by inductively coupled plasma–optical emission spectrometry (ICP–OES), which revealed an iron content of approximately 4 wt.% in all catalysts.

The electrochemical performance of the synthesised electrocatalysts was evaluated using linear sweep voltammetry and chronoamperometry using a three-electrode cell with a rotating ring-disc electrode and a rotating disc electrode, respectively.

CBs modification led to an increase of 600–800% in the current density observed at 0.4 V vs. the reversible hydrogen electrode (RHE) and an increase in the onset potential of 300–400 mV in comparison to the bare CBs. Furthermore, the successful incorporation of single-atom iron was suggested by a shift in the reaction mechanism towards the four-electron ORR pathway with negligible by-product formation, as opposed to the indirect two-electron pathway observed on the bare CBs.

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Carbon-based nanomaterial in solid biopolymer electrolytes: effect of graphene oxide on methylcellulose films

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The global demand for advanced energy storage fosters the development of more efficient and environmentally friendly devices in modern society. The conventional batteries with liquid electrolytes have potential hazards with risk of explosion, which for instance, could hinder their application in electric vehicles. Thus, developing safer electrolytes is a priority and solid polymer electrolytes can be a promising substitution [1,2]. Solid biopolymer electrolytes (SBEs) have stood out in this context, because of the need for safer and environmentally friendly devices. Nevertheless, their ionic conductivity and mechanical strength still need further improvements [3]. Various fillers are used to improve the ionic conductivity, electrochemical stability, and mechanical property, including lithium and sodium salts. Meanwhile, graphene and its derivatives, such as graphene oxide (GO), has caught attention especially due to its large specific surface area. It is also a hydrophilic material that exhibits great quantity of oxygen functional groups on its surface, which might contribute to ionic dissociation [1,2].

This work investigates different SBE films based on methylcellulose (MC) and GO, comparing their chemical and thermal properties, as well as the ionic conductivity in each sample at different temperatures. The films were prepared using the solvent casting technique. Firstly, the GO dispersion synthesized by the modified Hummers method [4] was diluted in Milli-Q water. By means of comparison, three systems were tested: 1) only GO in water, 2) by adding polyethylene glycol to GO dispersion, 3) by adding Sodium bis(fluorosulfonyl)imide (NaFSI) in dimethyl carbonate:ethylene carbonate (DMC:EC) to the aqueous GO dispersion. MC was added to each system, and the samples were placed in an incubator at 25 °C until forming a thin and flexible film [2].

The nanocomposite films based on MC and GO were characterized. The physical-chemical and thermal properties of the samples were analysed through FTIR and DSC techniques, along with the ionic conductivities' values, which was obtained through electrochemical impedance spectroscopy analysis. The films with the presence of GO show influence on the thermal, physicochemical properties, and ionic conductivity value compared to the control sample with pure MC, or even to the MC/NaFSI film [3]. For instance, Jia *et al.* [1] observed that the ionic conductivity of the composite film containing only 1 wt% GO reached a conductivity value one order of magnitude higher than the bare polyacrylonitrile matrix at 30 °C. In general, the investigated systems can be promising alternatives for applications as SBEs in energy storage devices with improved properties due to the presence of a small amount of GO.

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Para um futuro mais verde: células flexíveis com materiais sustentáveis

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O desenvolvimento de células flexíveis e ambientalmente mais sustentáveis, através da substituição de componentes convencionais por materiais alternativos de base biológica e reciclada, representa um avanço estratégico, ao reduzir o impacto ambiental, promover a economia circular e diminuir a dependência de matérias-primas críticas. Estas soluções sustentáveis oferecem maior segurança e desempenho (como o aumento da densidade de energia), permitindo novas aplicações em mobilidade elétrica e eletrónica flexível, alinhando-se com os Objetivos de Desenvolvimento Sustentável (ODS) e reforçando a competitividade no setor das energias limpas.

Neste contexto, no projeto “NGS – New Generation Storage” estão a ser desenvolvidos materiais alternativos para os diferentes componentes das células, desde novos ânodos, cátodos, separadores e eletrólitos, baseados em biopolímeros, como a celulose, compostos de carbono e materiais reciclados. Estes desenvolvimentos oferecem uma alternativa mais ecológica em comparação com os componentes convencionais, demonstrando propriedades mecânicas, térmicas e eletroquímicas promissoras, comparáveis às dos materiais comerciais utilizados em baterias de ião-lítio. Numa fase mais avançada, os componentes desenvolvidos serão integrados e testados numa linha piloto, permitindo a avaliação do seu desempenho em ambiente pré-industrial.

De um modo geral, as baterias desenvolvidas no âmbito deste projeto apresentam um elevado potencial num largo espectro de aplicações, desde a mobilidade elétrica até à eletrónica flexível, promovendo um ciclo de vida de baterias mais eficiente e sustentável, e contribuindo assim para a transição para uma economia circular e para a redução da pegada ecológica do setor.



Fig.1. Bateria de ião-lítio desenvolvida na linha piloto do CeNTI.

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Cathodes manufacturing strategies for improved efficiency, security and sustainability in lithium-ion batteries

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In recent years, driven by the advancements of Industry 4.0, the Internet of Things (IoT), and the integration of Artificial Intelligence (AI), there has been a growing demand for modern, compact electronic devices, sensors and actuators capable of capturing environmental data and enhancing system performance. These technologies require reliable energy sources, highlighting the critical need for high-performance and sustainable energy storage solutions. Lithium-ion batteries (LIBs), when combined with additive manufacturing technologies, have emerged as a promising alternative to conventional LIB manufacturing methods. This approach offers advantages such as improved material efficiency, scalable production and reduced costs. However, achieving a balance between optimal performance and environmental sustainability remains a significant challenge particularly due to the solvents employed during the manufacturing process and particles proportion [1].

Building on the previously discussed context, this work investigates the influence of different solvent and cathode components ratio for the fabrication of printed cathodes, focusing on both sustainability and performance. Studies evaluate the effects of alternative solvents as Dimethyl sulfoxide (DMSO) and N,N-Dimethylformamide (DMF), in comparison to the conventional 1-Methyl-2-pyrrolidone (NMP), within the framework of the Direct Ink Writing (DIW) printing technique. Also, the different proportions of active material, conductive additive and polymer binder was evaluated in ranges of LiFePO₄ active material between 80-93 %. The cathode samples was analysed with particular attention to morphology, adhesion, electrochemical stability and overall performance. The goal is to identify the most viable compromise between high efficiency and reduced environmental impact. The cathode surfaces exhibited consistent morphology with no visible cracks, demonstrating the feasibility of using the DIW technique for cathode fabrication. The low impedance observed across all samples confirms the suitability of each solvent for battery manufacturing. Notably, the sample prepared with DMSO achieved the highest discharge capacity, reaching 145 mAh·g⁻¹ at a C/8 rate. Furthermore, at C/8-rate, discharge capacity value of 150.2 mAh·g⁻¹ for cathode with 85% (C85) of active material was obtained. Showing that C85 sample present higher discharge capacity due to the excellent uptake capacity, significant electric conductivity and lower porosity, compared to other samples. Comparing the different lithium-ion diffusion coefficient, C85 cathode exhibit the highest value with $6.7 \times 10^{-14} \text{ cm}^2 \cdot \text{s}^{-1}$, demonstrating that this cathode present higher properties compared to the other samples.

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Next generation solid polymer electrolytes based on ternary components for high-performance room-temperature solid-state lithium-ion batteries

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The environmental issues that modern society faces are a topic of increasing concern. In this regard, there is a global trend to increase the proportion of renewable energy, which contributes to reducing the dependency on fossil fuels and their detrimental consequences on the environment. Because renewable energy sources are unpredictable, they must be combined with effective storage technologies to guarantee a sufficient energy supply. For a number of applications, such as smartphones and electric vehicles, lithium-ion batteries are the most practical choice for energy storage devices [1]. Nonetheless, this technology still presents challenges, primarily because the electrolyte composition contains highly reactive and easily combustible liquid components. Solid polymer electrolytes are the best way to address these problems, as they increase safety while lowering their negative effects on the environment [2]. Using a polymer matrix and two combination fillers, and ionic liquid and another filler that may be ceramic particles, zeolites, or metal organic framework (MOF), this work demonstrates the production of solid polymer electrolytes based on a three-component approach. This study shows the advantages of the three-component method, which allows to obtain solid polymer electrolyte's with improved ionic conductivity and thermal/mechanical stability. When compared to conventional electrolytes, this ternary composite approach allows improved cycling stability and battery capacity. Excellent cycle capacity has been achieved, with values above 150 mAh.g⁻¹ at the C/10 rate at room temperature.

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Advancing safety and performance in next-generation smart batteries

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Rechargeable batteries are essential in the transition towards clean energy, storing renewable energy and powering electric vehicles and electronic devices [1]. Lithium-ion batteries (LIBs) are widely recognised as one of the most established electrochemical energy storage systems [2]. They currently dominate the market in a variety of applications, mainly due to their high energy density and relatively fast charge and discharge rates. However, there are safety concerns, particularly regarding the risk of thermal runaway (TR), which can cause fires or explosions, and limit their practical application [3].

This work explores two approaches to enhance the safety of LIBs by preventing TR, focusing on the thermal shutdown effect (TS), **Fig.1** [3,4]. Different percentages of thermally expansive microspheres were incorporated into polymer binders as poly(vinylidene fluoride) copolymers and applied to the cathode and separator components. The morphological, chemical, physical and electrochemical properties of the developed materials were evaluated. It was demonstrated reversible battery cyclability and a discharge capacity of around 150 mAh.g⁻¹ at a C/8 rate, with a TS effect at approximately 100 °C. In summary, this work contributed to the development of safer cathodes and separators for next-generation high-power battery systems. The work also offers the advantage of easy integration into existing manufacturing processes and current battery technologies at a lower cost.

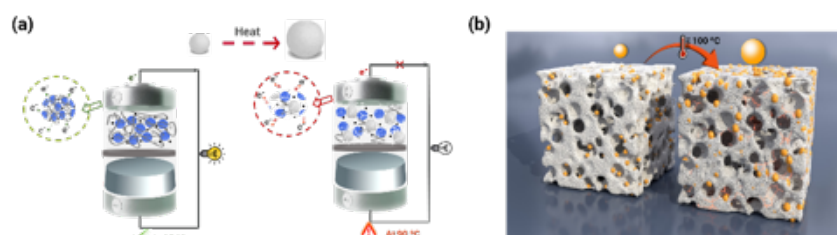


Fig.1. Schematic illustration of both approaches: **(a)** cathode [3] and **(b)** separator [4] incorporating thermally expansive microspheres, along with their respective TS mechanisms.

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Solid-state Na-metal batteries with improved performance via facile liquid electrolyte wetting at NaSICON interface

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Solid-state sodium (Na) metal batteries are prominent devices in the race towards ecological, low-cost, and high energy density energy storage solutions, particularly as complementary alternatives to lithium-based systems [1]. At present, their development is mainly held back by significant challenges at the electrode/electrolyte interfaces, originating from poor contact at the cathode side, and poor sodium wettability and high propensity for dendrite nucleation at the anode side, both contributing to high interfacial resistance and premature performance degradation [2,3]. In this work, a novel solid-state Na-metal battery architecture is reported, integrating a NASICON-type $\text{Na}_3\text{Zr}_2\text{Si}_2\text{PO}_{12}$ (NZSP) solid-state ceramic electrolyte, a carbon-coated $\text{Na}_3\text{V}_2(\text{PO}_4)_3$ (NVP/C) cathode, as well as a electrolyte surface modifier consisting of a sodium salt (NaClO_4 , NaPF_6) together with ethylene carbonate (EC) and propylene carbonate (PC) organic solvents, aiming to simultaneously address both interfaces.

Comprehensive electrochemical characterization of the electrolyte + wetting agent composite was performed via galvanostatic charge/discharge cycling and electrochemical impedance spectroscopy (EIS) in Na/Na symmetric cells, with area-specific resistance (ASR) and critical current density being significantly improved by the presence of the surface modifiers compared to the pristine solid electrolyte, indicating improved interfacial contact. The electrochemical performance of the full NVP/NZSP + wetting agent/Na system is then evaluated for multiple wetting agent compositions, with a remarkable specific discharge capacity of 107.1 mAh/g (91.1% of theoretical capacity) being obtained at a current density of 0.1 C, as well as stable cycling for 100 cycles at 0.2 C. Additional post-mortem morphological and material characterization via SEM and EDX mapping provides key insight into degradation mechanisms in Na-metal solid-state batteries and possible mitigation strategies.

Our findings provide key insights into solid-state interface design, with the proposed interfacial contact modification strategy highlighting a facile and scalable route to unlocking the full potential of Na-metal batteries by simultaneously addressing the Na metal and cathode interfaces with a functionalised NaSICON-type solid-state electrolyte.

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Biphasic aqueous electrolytes combining TFSI and salting-out agents for stable and selective membraneless redox systems

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Membrane-free aqueous redox flow batteries (RFBs) based on biphasic aqueous electrolytes represent a promising approach to overcome the cost and durability limitations of ion-exchange membranes. In this work, we developed and characterized a series of salt-induced aqueous biphasic systems (ABS) formed by combinations of LiTFSI or NaTFSI with LiCl or $(\text{NH}_4)_2\text{SO}_4$, aiming to establish stable, selective, and immiscible liquid phases suitable for membraneless electrochemical applications. The systems exhibit a well-defined TFSI-rich lower phase and a hydrophilic upper phase, whose polarity contrast enables efficient confinement of redox-active species.

Partition experiments demonstrated exceptional phase selectivity for all tested redox mediators. Potassium ferrocyanide was completely retained in the hydrophilic top phase, while methyl viologen and its ethyl and benzyl derivatives showed strong affinity for the TFSI-rich phase. In all cases, the partition coefficients (*K*) were close to or above 100, confirming excellent thermodynamic separation and minimal crossover potential between phases.

Electrochemical characterization by cyclic voltammetry (CV) revealed highly reversible redox processes for both ferrocyanide and viologen species, exhibiting narrow peak separations and stable current profiles. Repetitive cycling over 100 consecutive scans demonstrated remarkable electrochemical stability, with negligible signal decay or potential drift. From the formal potentials determined in each phase, the theoretical open-circuit voltages (OCV) of the combined redox pairs were estimated to exceed 1.0 V, confirming their favorable energetic alignment and electrochemical compatibility.

These findings establish a direct link between electrolyte composition, ion-specific solvation, and redox-species partitioning, highlighting the versatility of LiTFSI, NaTFSI, LiCl, and $(\text{NH}_4)_2\text{SO}_4$ based ABS for energy applications. As a next step, the performance of these optimized systems could be further validated through static membraneless cell tests, providing deeper insight into interfacial transport and redox coupling phenomena.

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RESUMOS COMUNICAÇÕES POSTER



Eutectic Extraction of Bioactive Compounds from Propolis

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Natural Deep Eutectic Solvents (NADES) represent a promising green alternative to conventional organic solvents for extracting bioactive compounds from natural matrices [1]. This study evaluated three NADES formulations: Proline:Citric Acid (1:2), Betaine:Glycerol:Citric Acid (1:2:1), and Citric Acid:Monopropylene Glycol (1:4) for extracting antioxidant compounds from propolis collected in Montesinho Natural Park, Portugal [2]. Ultrasound-assisted extraction was optimized using L-Optimal experimental design, varying extraction time, temperature, water content, and ultrasound amplitude.

Among the tested eutectic systems, Citric Acid:Monopropylene Glycol (1:4) demonstrated superior performance, achieving maximum antioxidant activities of 31 mg AA/mL of extract (ABTS) and 31 mg AA/mL of extract (DPPH). This was the closest performance to conventional ethanol extraction, with activities only 6.6% and 6.8% lower than the best ethanol extract done in the same conditions (ET99: 33 mg AA/mL of extract (ABTS), 33 mg AA/mL of extract (DPPH)), eliminating toxic solvent residues. The Proline:Citric Acid (1:2) system achieved moderate performance with 13 mg AA/mL (ABTS) and 13 mg AA/mL (DPPH), representing 59% and 60% of the ethanol extraction efficiency, respectively. The Betaine:Glycerol:Citric Acid (1:2:1) system showed the lowest performance with 12 mg AA/mL (ABTS) and 7 mg AA/mL (DPPH), achieving 37% and 23% of conventional extraction efficiency.

Despite lower absolute values compared to conventional methods, all eutectic systems demonstrate significant advantages, such as solvent biocompatibility, elimination of toxic residues, short extraction times, and direct incorporation potential into formulations without solvent removal steps. The Citric Acid:Monopropylene Glycol system particularly showed promise for industrial applications, maintaining near-conventional efficiency while providing environmental sustainability. Statistical modeling confirmed robust process optimization with high predictive capability ($R^2 > 0.96$).

These results establish eutectics as viable green alternatives for propolis bioactive compound extraction. The study demonstrates that while they may not match the absolute extraction efficiency of conventional solvents, their environmental and safety advantages, combined with acceptable bioactivity recovery, make them attractive alternatives for sustainable natural product processing.

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Lupin-powered brownies: nutritional quality and consumer acceptance

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Global interest in gluten-free foods rose significantly over the past years, placing the category among the top ten current food trends [1]. To formulate these products, legume flours can stand in for wheat. In this context, lupin (*Lupinus albus* L.), noted for its high nutritional quality, is a compelling wheat-flour alternative for applications such as baked goods. Thus, this study evaluated the nutritional and sensory properties of a brownie formulated with lupin flour. Nutritional composition was determined using the official AOAC 2023 methods [2]. Consumer acceptance was assessed with an affective test involving 140 untrained participants, who rated the samples on a nine-point hedonic scale (1- disliked extremely; 9 – liked extremely). The attributes evaluated were overall impression, appearance, aroma, flavour, and texture. A purchase-intention test was also applied simultaneously. A wheat-flour brownie served as the control in all analyses. The lupin brownie showed moisture, ashes and lipid contents comparable to the control, but a higher protein content (14.61 ± 0.21 g/100 g vs. 9.80 ± 0.62 g/100 g, respectively). In the sensory evaluation, mean scores for the lupin brownie ranged from 6.90 ± 1.78 (texture) to 7.47 ± 1.27 (overall impression), while the control was slightly higher, ranging from 7.44 ± 1.64 (texture) to 7.87 ± 1.10 (appearance). In the purchase-intention test, the options “certainly would buy” and “likely would buy” were selected most frequently for both samples. Overall, the lupin-based formulation demonstrated improvements in nutritional quality and was generally well accepted by consumers, representing a promising option to expand gluten-free bakery choices for individuals requiring reduced levels or absence of gluten in their diets.

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Avaliação da citotoxicidade e efeitos metabólicos de um extrato de semente de *Rubus fruticosus* Linnaeus em células Caco-2

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As sementes de *Rubus fruticosus* Linnaeus (amora-preta) são reconhecidas como fontes promissoras de compostos fenólicos e outras moléculas bioativas com potenciais aplicações na alimentação e na saúde [1]. Para investigar a sua segurança e possíveis efeitos funcionais, avaliamos o potencial citotóxico de um extrato de semente de *R. fruticosus* em células de adenocarcinoma colorretal humano (Caco-2), utilizando três ensaios de viabilidade complementares: MTT (metabolismo mitocondrial), Sulforodamina B (SRB, proteína celular total) e Vermelho Neutro (NR, integridade lisossomal). As células foram expostas durante 24 horas a quatro concentrações do extrato (2,5–0,3125 mg/mL).

No ensaio de MTT, a maior concentração (2,5 mg/mL) aumentou significativamente a atividade metabólica celular para aproximadamente 160% em relação ao controle não tratado ($p < 0,01$), enquanto as concentrações mais baixas permaneceram próximas de 80–90%, indicando ausência de citotoxicidade e uma possível estimulação metabólica na dose mais alta. O ensaio de SRB mostrou um ligeiro aumento no conteúdo de proteína total a 2,5 mg/mL (~111% do controle) e pequenas reduções nas concentrações intermediárias, com diferenças significativas apenas em 1,25 e 0,625 mg/mL ($p < 0,05$), sugerindo que não houve perda relevante de biomassa celular. De forma semelhante, os resultados do ensaio de NR revelaram atividade lisossomal preservada em todas as concentrações, com a dose mais alta atingindo ~213% do controle ($p < 0,05$), o que é consistente com um aumento da captação do corante em vez de toxicidade.

Foi evidente que o extrato de semente de *R. fruticosus* não é tóxico para as células Caco-2 dentro da faixa testada e pode até promover uma leve estimulação das funções metabólicas e lisossomais na concentração mais elevada. Estes resultados reforçam o potencial das sementes de amora-preta como ingrediente funcional, contribuindo para a valorização de subprodutos agroindustriais e os princípios da economia circular.

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Therapeutic Potential of Hardy Kiwi against Cutaneous Melanoma

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Melanoma is one of the most common cancers worldwide, with more than 1.5 million new cases reported in 2022 [1]. When located in the basal layer of the epidermis, melanoma is classified as cutaneous melanoma (CM). CM is widely recognized as the most aggressive skin malignancy, presenting a high metastatic potential and notable therapeutic resistance, and being accountable for ~80% of skin cancer-related mortality [1-3]. As CM cells originate from epidermal melanocytes, they share multiple developmental processes with healthy cells [2-4]. Alterations in signaling pathways within pigment-producing melanocytes, as well as mutations in specific genes, can lead to uncontrolled cell proliferation, disruption of cell cycle regulation, increased resistance to apoptosis, and enhanced invasion [2, 3]. For instance, mutations in the NRAS and BRAF genes activate the MAPK and PI3K/AKT pathways, driving uncontrolled proliferation [2]. Therefore, understanding these molecular mechanisms, particularly the signaling pathways underlying CM progression, is crucial for developing effective therapies.

Treating CM poses considerable challenges due to its complex biology and capacity to develop resistance to conventional treatments [4, 5]. Current therapeutic options often include surgical excision, radiation, chemotherapy, immunotherapies, and targeted molecular therapies, with growing emphasis on the latter, which target key molecules, proteins, and regulators of the pathways involved in CM development and progression [2, 4, 5]. Topical therapies, such as imiquimod, have also emerged as promising alternatives, becoming standard of care for early-stage melanoma or melanoma in situ by offering less invasive treatment options [4, 5]. However, undesirable side effects remain a limitation.

Consequently, recent research has increasingly focused on natural compounds and their analogues, which already account for approximately one-third of the FDA-approved anticancer drugs [5]. Among natural sources, *Actinidia arguta* fruit (hardy kiwi) has drawn significant attention due to its rich content of phenolic acids, vitamins, and other bioactive compounds [6, 7], and the exhibition of remarkable antioxidant, anti-inflammatory, and anti-cancer properties [7]. Compounds from *A. arguta* have demonstrated the ability to exert anti-melanoma effects, either alone or in synergy with conventional therapies, through mechanisms such as neutralizing free radical-induced oxidative damage, inducing apoptosis in malignant melanocytes, inhibiting proliferation, and preventing metastasis [8].

Our work aims to explore the potential benefits of bioactive compounds from *A. arguta* and its by-products on CM. By highlighting their role as complementary or alternative therapeutic agents, this study encourages the development of new melanoma treatment strategies that are both more effective and less toxic than conventional approaches, offering renewed hope in the fight against this deadly disease.

Acknowledgements

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Precise Quantification and Purity Determination of Minicircle DNA (mcDNA) using a HPLC-DAD Chromatographic Method

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In an aging society, the rising incidence of cancer is expected to escalate and consists one of the most relevant societal challenges of our era, with accentuated consequences due to a different access to healthcare between lower and higher income countries. To overcome this issue, biopharmaceuticals, including nucleic acids, have emerged as novel effective therapeutic agents which, due to recent advances in gene therapy, offer high therapeutic effectiveness, specificity and fewer side-effects [1]. Non-viral vectors, such as minicircle DNA (mcDNA), are receiving significant attention mostly because they can facilitate a safer and more efficient transfection of gene products when compared with traditional plasmid DNA (pDNA) [2]. However, its structural similarity to parental plasmid (PP), the presence of related isoforms, and host-derived impurities, present significant challenges in its detection and precise quantification.

To overcome this issue, this study investigated a novel anion-exchange chromatographic method for mcDNA quantification was developed and validated. High Performance Liquid Chromatography (HPLC) effectively enabled the separation of mcDNA from the reminiscent species, including PP, isoforms, and RNA, present within the clarified *Escherichia coli* (*E. coli*) strain ZYCY10P3S2T lysate.

The developed method demonstrated excellent linearity ($R^2 = 0.9994$) over the 1–100 $\mu\text{g/mL}$ range, with accuracy and precision aligned meeting with the regulatory criteria for concentrations $\geq 10 \mu\text{g/mL}$ [3]. The lower limit of quantification (LLOQ) and limit of detection (LOD) were also investigated and established at 10 $\mu\text{g/mL}$ and 1 $\mu\text{g/mL}$, respectively, therefore ensuring a suitable and reliable level of precision for mcDNA quantification. Reproducibility was confirmed through intra-day and inter-day precision tests, with coefficients of variation below 8%. Finally, the method was applied to a complex lysate containing mcDNA, PP, miniplasmid (mP), and RNA, confirming its robustness and applicability to typical purification process samples.

In conclusion, this work presents, for the first time, a selective, accurate, and effective chromatographic strategy for mcDNA quantification, which ultimately foreseeing to be included as valuable tool for routine quality control in mcDNA-based biopharmaceutical production.

Acknowledgements

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Nanocatalysts for the epoxidation of biobased olefins

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The chemical valorisation of olefins via epoxidation routes holds considerable industrial importance, since epoxides are versatile intermediates employed in the manufacture of a wide range of chemicals and materials. While homogeneous molybdenum-based catalytic technologies reached industrial implementation for olefin epoxidation, the same does not apply for heterogeneous molybdenum-based ones, which continue of great interest, albeit challenging.

In this work, selective and stable epoxidation solid nanocatalysts were developed for the epoxidation of bulky biomass derived olefins such as, unsaturated fatty acid methyl esters (FAMES), using *tert*-butylhydroperoxide (TBHP) as oxidant, under mild reaction conditions. The nanocatalysts consisted of oxides of molybdenum and M = Ta, Nb or W, prepared via solvothermal methodology (Fig. 1) [1]. The influence of the material synthesis conditions on the material properties was investigated to meet superior catalytic performances. The catalysts Mo(75D)M-0.3 with M = Ta, Nb and W led to 84-95 % and 97-100 % methyl oleate and methyl linoleate conversion, respectively, giving the corresponding epoxides in yields greater than 80 % at 24 h and 70 °C [1].

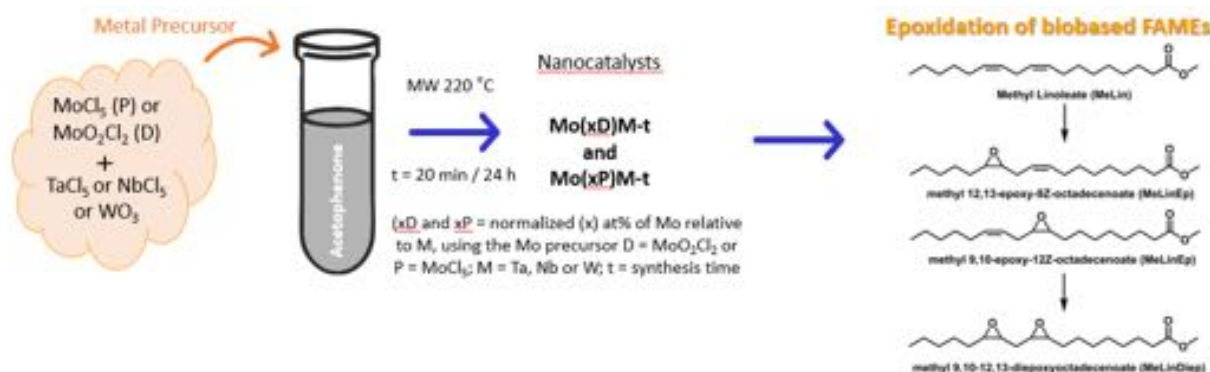


Fig.1. Solvothermal synthesis of the catalytic materials for the epoxidation of biobased FAMES.

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Valorisation of α -Angelica Lactone over Zirconia Nanocrystals

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Climate change and the need to reduce greenhouse gas emissions demand a transition towards renewable energy and carbon sources. Lignocellulosic biomass, an abundant and renewable source, represents a promising alternative to several fossil feedstocks. Among its derivatives, α -angelica lactone (AnL) emerges as a versatile chemical, obtained from hemicelluloses via acid-catalysed reactions. Its selective upgrading to levulinate esters (LEs) and γ -valerolactone (GVL) provides routes to useful bio-based products. LEs find promising applications as fuel additives, solvents, plasticisers, and intermediates for pharmaceuticals and polymers, while GVL stands out as a promising green fuel additive due to favourable properties, such as relatively low vapour pressure and reduced emissions. Maximising the sustainability of such processes requires selective, non-toxic, and recyclable catalysts, operating under moderate reaction conditions and avoiding hazardous reagents. In this work, we present the synthesis of ZrO_2 nanocrystals via a solvothermal route using zirconium tetrachloride and acetophenone, and their application as multifunctional catalysts for the one-pot conversion of AnL to GVL [1]. Importantly, 2-butanol was employed as a hydrogen donor, eliminating the need for external H_2 and associated safety concerns. Among the prepared catalysts, small cubic ZrO_2 nanoparticles (2.3 nm, c100-1) exhibited the most favourable balance of activity, selectivity, and recyclability, achieving a 71 % GVL yield at 180 °C. These results demonstrated how tailored zirconia nanocatalysts may promote biomass valorisation, supporting the transition towards renewable carbon platforms.



Fig.1. Integrated catalytic conversion of biomass derived α -angelica lactone (AnL).

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Nanoparticles of Metal Oxides Embedded in Silica for Batch and Continuous-Flow Catalysis

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Although there are technological advancements of epoxidation processes (especially for light olefins), which have importantly contributed to the epoxides market expansion (several end-user products), challenges remain in developing adequate heterogeneous catalysts for converting bulkier olefins, using simple catalyst synthesis procedures, and, on the other hand, studying the catalytic performances under different operation modes. In this work, heterogeneous epoxidation catalysts were prepared via simple one-pot procedures, with the necessary versatility to tune the material properties for batch and continuous flow operation (Fig. 1) [1]. Specifically, heterogeneous epoxidation Mo catalysts consisting of silica-embedded transition-metal oxide nanoparticles (Si/Mo(x) with $x = \text{Mo}$ loading, and Si/MoM with $M = \text{Ta}$, Nb or W) were developed. The material properties, and thus the catalytic performances depended on the types and concentrations of the constituent metals, as well as the operating conditions such as the reaction temperature and operation mode (i.e., batch or continuous flow). They promoted the epoxidation of cis-cyclooctene with *tert*-butyl hydroperoxide as oxidant, at 70 °C. Epoxide yields in the range of 90%–100% were reached within 4 h, at 70 °C, under batch operation. Under continuous flow, steady state conditions (fixed bed reactor), catalyst Si/MoNb led to ca. 44% epoxide yield, at 70 °C. At 90 °C, Si/MoNb exhibited multifunctionality leading to 51% cyclooctanone yield. Si/MoNb is, to the best of our knowledge, the first Mo heterogeneous catalyst reported for one-pot olefins-to-cycloalkanones processes (with hydroperoxide oxidants), opening the way to scientific research of new catalytic applications of Mo catalysts for integrated continuous flow processes.

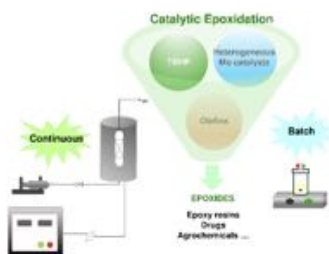


Fig.1. Olefin epoxidation under batch or continuous flow operation mode.

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Enhanced Photocatalytic Hydrogen Evolution over TiO₂/RuO₂ Heterojunctions with Optimized Ruthenium Loading

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With the increasing concern regarding the depletion of fossil fuels, the exploration of renewable energy sources has become essential [1]. Hydrogen (H₂) has emerged as a clean and sustainable energy carrier. In this study, a TiO₂/RuO₂ photocatalytic heterojunction was synthesized and its application in hydrogen production was investigated. Ruthenium was highly dispersed on the TiO₂ surface through a wet impregnation method, with varying ruthenium contents (0.5, 1.0, 2.5, 5.0, and 7.5 wt%). The H₂ evolution experiments were carried out in a borosilicate reactor. The photocatalyst mass was fixed at 50 mg and dispersed in 100 mL of a sacrificial agent aqueous solution (0.75 M TEOA). Before the photocatalytic tests, the reactor was degassed in the dark for 20 min using a nitrogen flow (50 mL min⁻¹) [1]. After confirming the complete removal of dissolved oxygen, the nitrogen flow was maintained at 7.5 mLmin⁻¹. The temperature was maintained at 20 °C by circulating water through the photochemical reactor using a thermostatic bath. All photocatalytic reactions were conducted under LED irradiation (λ = 370 nm) using four visible-LED lamps positioned 4 cm from the cylindrical glass reactor wall. The suspensions were irradiated for 2 h, and the evolved H₂ was quantified using an online gas chromatograph (Inficon Micro GC 3000, Agilent) equipped with a 5 Å molsieve column and a micro-TCD detector, with argon as the carrier gas [1]. The Photoluminescence Spectroscopy (PL) analysis revealed that the heterojunction structure formed between RuO₂ and TiO₂ significantly enhanced the separation efficiency of photogenerated charge carriers compared to the individual oxides. In addition, PL spectra exhibited broad emission bands between 350 and 550 nm, suggesting different electronic transitions. The optical and electronic properties of the photocatalysts were further evaluated by UV–Vis DRS. The results showed that the introduction of ruthenium induced a redshift, which can be attributed to the creation of additional energy levels. Moreover, an increase in ruthenium content resulted in a progressive redshift. The heterojunction effectively promoted rapid charge migration under illumination, thereby markedly enhancing photocatalytic activity. Under light irradiation, the H₂ evolution rate of TiO₂/RuO₂ (0.5%) reached ~300 μ mol after 2 h of reaction. Increasing the ruthenium content from 0.5% to 1.0% led to a considerable enhancement in photocatalytic performance, with TiO₂/RuO₂ (1.0%) achieving a maximum H₂ yield of 500 μ mol. However, further increasing the ruthenium content from 1.0% to 7.5% resulted in decreased photocatalytic H₂ production, which may be attributed to ruthenium segregation on the TiO₂ surface. This demonstrates the existence of optimal ruthenium loading for efficient modification of TiO₂/RuO₂ photocatalytic heterojunctions. An optimized TiO₂/RuO₂ heterojunction can substantially increase photoactivity by improving charge separation, providing efficient (and selective) surface reaction sites, and doing so with a realistic material-cost trade-off, making TiO₂-based water-splitting systems far more practical and better characterized for photocatalytic H₂ production scale-up.

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Synergistic Photocatalytic Performance of Graphitic Carbon Nitride over Mg-Al Layered Double Hydroxide Composites for the Degradation of Complex Pharmaceutical Matrices under Solar Light

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Traditional wastewater treatment systems are often ineffective in removing pharmaceutical contaminants in aquatic environments, especially in complex mixtures. Therefore, heterogeneous photocatalysis using visible-light-active semiconductors has emerged as a promising strategy for environmental remediation. In this context, the work aims to synthesize g-C₃N₄@MgAl LDH (layered double hydroxide) composites, to synergistically combine the properties of each component for the photocatalytic degradation of a mixture of six pharmaceuticals – venlafaxine, citalopram, carbamazepine, tramadol, ketoprofen and diclofenac – under visible light. The materials were synthesized via an electrostatic self-assembly route in aqueous solutions [1]. The UV–vis diffuse reflectance technique was employed to investigate the optical characteristics of the prepared materials. MgAl-LDH exhibited a pronounced absorption band in the 200–265 nm region, along with a weaker absorption in the 265–420 nm range, which is attributed to the ligand-to-metal charge transfer within the hydrotalcite layers [2]. For g-C₃N₄, the absorption edge appeared around 455 nm, confirming its ability to harvest visible light. Upon increasing the loading of MgAl-LDH, the light absorption intensity of the X%LDHGCN (X = 10, 30, and 70 LDH) composites gradually enhanced, and their absorption edges showed a slight redshift compared with pristine g-C₃N₄. Nevertheless, the 70%LDHGCN composite exhibited a blue-shifted edge, resembling the optical profile of MgAl-LDH, and this phenomenon is still under investigation. The band gap energies of the synthesized composites were determined through Kubelka–Munk function fitting combined with Tauc plot analysis. The obtained band gap values for the 10%LDHGCN, 30%LDHGCN, 70%LDHGCN composites were 2,73 eV, 2,69 eV, and 2,70 eV, respectively. These values were lower than those of pure g-C₃N₄ (2,91 eV) and MgAl-LDH (4,8 eV), indicating enhanced activation under visible light. The variation in band gap energy may be associated with the formation of an intimate interfacial structure between MgAl-LDH and g-C₃N₄ [1], where interfacial electron transfer occurs, leading to rearrangements in the energy band structure. This effect could account for the band gap narrowing observed in the composites. The chemical bonds and functional groups of MgAl-LDH, g-C₃N₄ and X%LDHGCN composites were investigated using FT-IR spectra, with g-C₃N₄ bands within 1650-1200cm⁻¹ corresponding to the stretching vibration of C-N heterocycles [1]. As for MgAl-LDH, peaks at 3500 cm⁻¹ and 1650 cm⁻¹ represent the stretching and bending vibrations of -OH, respectively [1]. As the content of MgAl-LDH in the composites increases, the characteristic bands of g-C₃N₄ weakened, strongly indicating a successful synthesis of the composites.

Acknowledgements

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Nature-based catalysts for CO₂ cycloaddition reactions to epoxides

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Catalytic transformations of CO₂ have emerged as a promising strategy for mitigating greenhouse gas emissions while generating valuable chemicals. Among the various routes, the cycloaddition of CO₂ to epoxides to produce cyclic carbonates stands out, due to its excellent atom economy and the broad utility of cyclic carbonates as green solvents, electrolytes in lithium-ion batteries and as versatile intermediates for polymer and fine chemical synthesis [1]. In this context, the design of efficient, selective, and reusable catalysts derived from natural sources is gaining increasing attention, as these materials combine sustainability, low cost, abundance, structural tunability, and catalytic potential [2]. In this work, we report the development of two families of naturally derived catalysts and their application in CO₂ cycloaddition reactions to epoxides (**Fig. 1**).

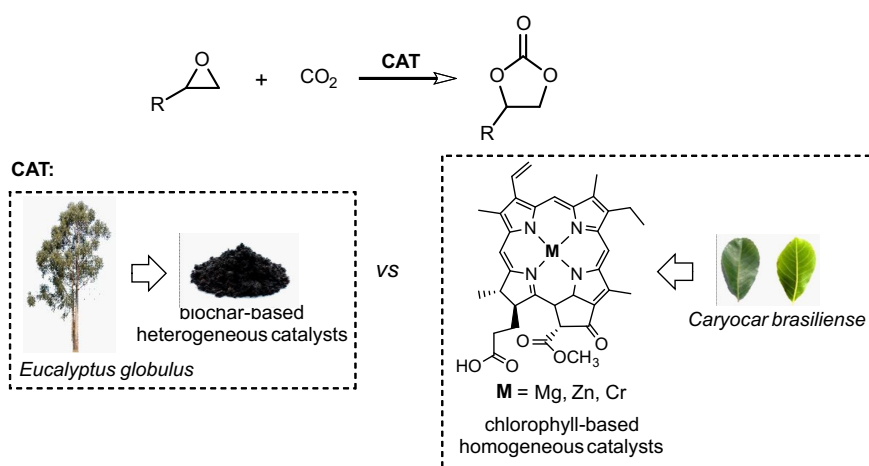


Fig.1. Nature-based catalysts for CO₂ cycloaddition to epoxides.

The first family consists of renewable carbon materials obtained via pyrolysis of *Eucalyptus globulus* wood, subsequently functionalized using physical vapor deposition techniques to produce N-doped and metal-doped biochar (e.g., Al, Cu, Cr) as heterogeneous catalysts [3]. Another approach focuses on the extraction of chlorophyll from *Caryocar brasiliense* (pequi) leaves and its subsequent complexation with different metal centers (Zn, Cr) to yield bioinspired homogeneous catalytic systems. Their performance in catalytic CO₂ cycloaddition reactions to epoxides is evaluated. The influence of catalyst type, functionalization, metal center, substrate structure and reaction parameters (temperature, CO₂ pressure and co-catalyst addition) on the catalytic activity, selectivity toward cyclic carbonates and recyclability is presented and discussed.

Acknowledgements

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[Pd(NH₃)₄]²⁺NaY as Heterogeneous Catalyst for Sonogashira Coupling of Thiophene and Pyridine Halides with Arylacetylenes

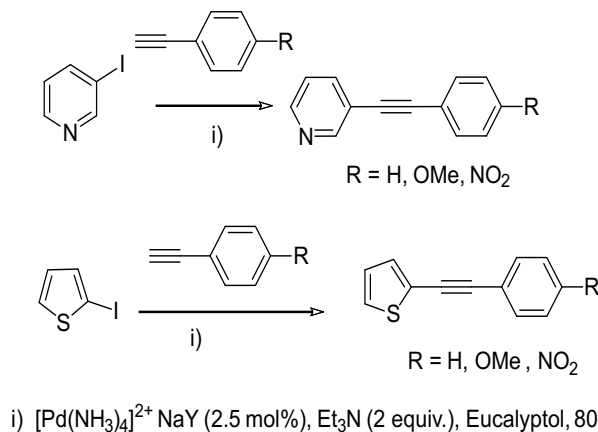
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In recent years, there has been increasing interest in developing greener and more sustainable strategies for organic synthesis, particularly through Cu-free, Pd-catalyzed heterogeneous systems for Sonogashira couplings [1]. These reactions are powerful tools in organic chemistry for constructing C-C bonds and play a crucial role in the synthesis of compounds with applications in pharmaceuticals, materials science, and agrochemicals [2]. In this work, we report the preparation of a heterogeneous palladium catalyst, [Pd(NH₃)₄]²⁺NaY, synthesized using a NaY zeolite framework and a PdCl₂/NH₃ solution. The catalyst was characterized by SEM/EDX (Scanning Electron Microscopy/Energy Dispersive X-ray Spectroscopy) and XPS (X-ray Photoelectron Spectroscopy), which confirmed the structural integrity of the zeolite and the successful, uniform incorporation of Pd. This heterogeneous catalyst was then applied in Cu-free Sonogashira couplings of heteroaryl halides with arylacetylenes under mild conditions, employing eucalyptol (1,8-cineole) as a green solvent at 80 °C. Eucalyptol, a renewable and biodegradable compound derived from natural sources, provides a safer and more sustainable alternative to conventional solvents [3]. The reactions proceeded in moderate to high yields using either 3-iodopyridine or 2-iodothiophene with phenylacetylene or arylacetylenes bearing electron-donating (OMe) or electron-withdrawing (NO₂) substituents on the phenyl ring (**Scheme 1**).



Scheme 1. Heterogeneous catalysis in Sonogashira reactions using eucalyptol.

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Hydrogen production from the photo-assisted ammonia decomposition using metal-loaded graphitic carbon nitride

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Energy is a cornerstone of modern society, sustaining industrial activity, transportation, communication, healthcare, and virtually every aspect of daily life. However, the increasing reliance on fossil fuels such as coal, oil, and natural gas has raised significant concerns. The combination of environmental degradation and economic challenges has accelerated the global transition toward sustainable energy systems, with renewables playing a central role in future energy alternatives. Alternative methods have been and are being studied to take over fossil fuels as the next generation of energy vectors. One promising alternative is hydrogen (H_2), since it can be derived from renewable resources, its utilization does not directly produce carbon emissions and has a very high mass energy density [1]. On the other hand, storage complications and low volumetric density mean that an H_2 carrier is desirable for H_2 to be viable as an energy vector in a centralised production perspective. A substance suited for this purpose is ammonia (NH_3), due to its high H_2 mass content (17.8%), well-established distribution networks [2] and carbon emission-free H_2 recovery. However, this same H_2 recovery process is difficult to achieve, since the decomposition of NH_3 is not a well-established technology and occurs at very high temperatures [3]. In the present work, H_2 is obtained by the photocatalytic decomposition of NH_3 in solution, using an optical semiconductor, thermally exfoliated graphitic carbon nitride (GCN-T), loaded with metal nanoparticles (Au, Cu, Fe, Ni, Pd, Pt and Ru) acting as cocatalysts. The primary objectives are the development of a process capable of achieving NH_3 decomposition under mild conditions and with the capability of using sunlight as a light source. The produced photocatalysts were prepared by the incipient wetness impregnation of a metal precursor solution over GCN-T under sonication, which produced metal-loaded GCN-T (M/GCN-T, M: Au, Cu, Fe, Ni, Pd, Pt and Ru). The photocatalytic activity was assessed using a batch reactor irradiated by a light-emitting diode system ($\lambda_{max} = 420$ nm), with an ammonium hydroxide (NH_4OH) solution with a nominal initial NH_3 concentration of 0.5 M and a catalyst loading of 1 g/L. Pd/GCN-T exhibited the highest H_2 production of 987 $\mu\text{mol/g}_{cat}$ after 6 h of irradiation. Reproducibility and reusability of Pd/GCN-T were studied, and the material was revealed to yield reproducible results and withstand reutilization for at least 3 consecutive cycles with little activity loss. This research seeks to maximize H_2 yield under mild conditions while minimizing by-products, positioning ammonia as a useful H_2 carrier for sustainable energy applications such as dedicated fuel cells and large-scale storage, as in decarbonization of the shipping industry.

Acknowledgments

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Photocatalytic oxidation of anisyl alcohol into *p*-anisaldehyde using a carbon nitride modified perovskite

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Shifting from hazardous and high-energy consumption chemical routes to light-driven catalytic paths is a necessary step towards the chemical industry's green transition. The development of more efficient and selective semiconductors is an integral part of photocatalysis research and, for years, graphitic carbon nitride (GCN) has been proved a stable and cost-effective metal-free photocatalyst with a large range of applications, from H₂ production and CO₂ reduction to wastewater treatment and organic synthesis. Yet, its low specific surface area and quick charge carrier recombination limits its activity [1]. More recently, halide perovskites, such as Cs₃Bi₂Br₉ (CBB), have shown to be very promising materials with a broad light absorption range and applications in solar cells and photodetectors, but their use as photocatalysts in organic synthesis remains scarcely explored [2]. Therefore, in this work, xGCN/CBB hybrid materials, where x represents the GCN weight percentage, were produced, characterised and used for the visible-light-induced oxidation of anisyl alcohol (AA) into *p*-anisaldehyde (*p*-AAD) to explore the potential synergistic behaviour between both semiconductors. GCN was produced by thermal polymerisation of dicyandiamide, while CBB was synthesised by stoichiometrically mixing the CsBr and BiBr₃ precursors in N,N-dimethylformamide. The composites containing varying quantities of GCN (50 to 98 wt%) were prepared by adding the desired amount of GCN one hour after starting the CBB synthesis. The morphological and optical properties of the materials were characterised by different techniques, namely scanning electron microscopy, X-ray diffraction, Fourier transform infrared spectroscopy, ultraviolet-visible spectroscopy and solid-state photoluminescence spectroscopy. The photocatalytic performance of the materials was assessed through 1-hour long experiments, with a catalyst load of 1 g L⁻¹ and an initial AA solution concentration of 5 mM, under ambient pressure and temperature conditions, using visible light-emitting diode irradiation. The conversion of AA and production of *p*-AAD were quantified using high-performance liquid chromatography with samples taken periodically from the reaction medium. The most promising photocatalyst was immobilised on the surface of a polyethylene terephthalate structure and its reusability was assessed through three consecutive 2-hour reaction cycles. Hybrid materials with lower amounts of GCN exhibited larger and more abundant CBB crystals while showing enhanced optical properties. The addition of CBB to GCN, even in small quantities, revealed a remarkable increase in AA conversion and, among the synthesised composites, the 70GCN/CBB catalyst demonstrated the best compromise between photocatalytic activity and stability. When immobilised, the 70GCN/CBB was able to keep its performance over three consecutive cycles, during a total of 6 hours of reaction, allowing a more practical and efficient use of these materials in future industrial applications with lower environmental impact than traditional organic synthesis processes. The visible-light oxidation of AA to *p*-AAD is important because it provides a green, selective, and industrially relevant pathway to a high-value aldehyde, while also serving as a benchmark reaction in photocatalysis research, linking fundamental studies to practical applications in fragrance, pharmaceuticals, and sustainable chemistry.

Acknowledgements

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Fotocatálisis activada con luz visible mediada por nuevos compuestos de cobre(I) funcionalizados con ácidos carboxílicos

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La fotocatálisis ha alcanzado una enorme importancia en Química Orgánica. Los fotocatalizadores (PC) hacen posible, ya sea mediante mecanismos de transferencia de energía (EnT) o de electrones (SET), acceder a rutas de síntesis termodinámicamente desfavorables, reducir tiempos de reacción y llevarlas a cabo en condiciones más suaves, incluso empleando luz visible en lugar de UV de alta energía.

Atendiendo a la búsqueda de fotocatalizadores que utilicen metales sostenibles, los complejos de cobre(I) con ligandos de tipo bipyridina (N[^]N) destacan por sus excepcionales propiedades fotofísicas. Por un lado, la configuración de capa cerrada del Cu(I) (d¹⁰) impide las desactivaciones d-d, de naturaleza no radiante. Por otro lado, la coordinación de los ligandos N[^]N aumenta la absorción en el visible y posibilita la formación de estados excitados de transferencia de carga metal-ligando (MLCT). De este modo, en la literatura existe un buen número de ejemplos de compuestos homolépticos [Cu(N[^]N)₂]⁺ y heterolépticos [Cu(N[^]N)(P[^]P)]⁺, siendo P[^]P una disfosfina rígida.[1,2]

En esta comunicación se presentan cuatro nuevos compuestos de Cu(I) que cumplen el arquetipo comentado (**Fig. 1**). Como ligandos bipyridínicos, se han seleccionado el ácido 2,2'-biquinolina-4,4'-dicarboxílico [BIQ(CO₂H)₂] y el ácido 4-(6,9-dimetil-1*H*-imidazo[4,5-*f*][1,10]fenantrolin-2-il)benzoico (L1-CO₂H) (**Fig. 1**). Para los complejos heterolépticos, se ha empleado la difosfina XantPhos. Se determinó que la actividad fotocatalítica en isomerizaciones de alquenos (EnT) y deshalogenaciones (SET reductora) se ve favorecida por el ligando L1-CO₂H frente a BIQ(CO₂H)₂; y por la presencia de XantPhos frente a los complejos homolépticos. Finalmente, el estudio de las propiedades emisivas sugiere la presencia de fluorescencia retardada activada térmicamente (TADF) para alguno de los compuestos.

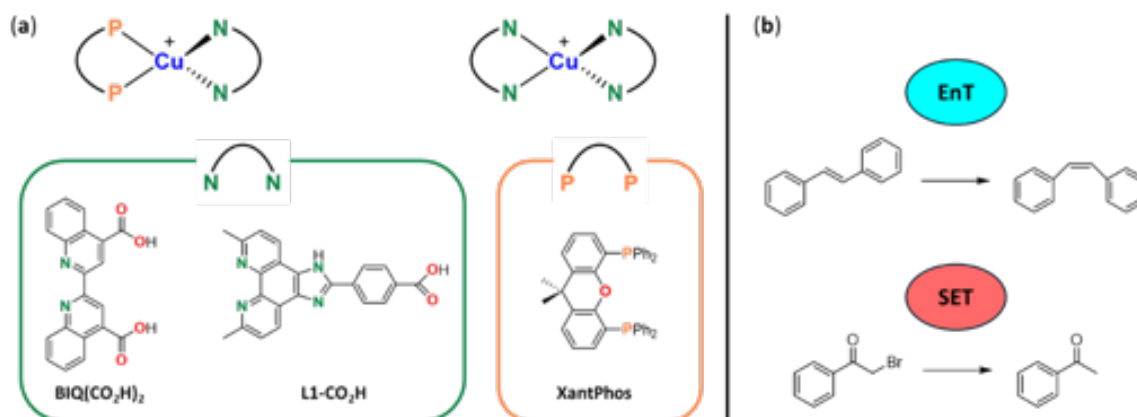


Fig.1. Compuestos de cobre(I) utilizados (a) y reacciones fotocatalíticas evaluadas (b).

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Charge transfer mechanisms in hybrid catalysts for sustainable ammonia production

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Ammonia (NH₃) is a key chemical for fertilizer production and a promising carbon-free energy carrier. The increasing demand for NH₃ has led to significant research in artificial nitrogen (N₂) fixation. Although nitrogen is abundant in the atmosphere, fixing it is a complex and energy-intensive process due to the high activation energy barrier of 941 kJ/mol required to break the N≡N bond. By improving the development of more effective catalysts for N₂ reduction, we can greatly enhance sustainable NH₃ synthesis and make significant progress in this vital area [1]. This study introduces a novel series of Ti-MOF(NH₂-MIL-125)-based catalysts for visible light-assisted photocatalytic N₂ fixation, using varying ratios of graphitic carbon nitride, g-C₃N₄ (GCN) through physical and chemical mixing methods (Fig. 1). In different ratios, the photocatalytic NH₃ production of the 25% GCN/MOF material (810 μmol.g⁻¹.h⁻¹) is three times higher than that of its individual catalysts. When exposed to visible light, electrons in GCN are excited and migrate to defect sites in Ti-MOF, creating an electron depletion layer in GCN and an accumulation layer in MOF. This results in a positively charged GCN interface and a negatively charged MOF, generating an internal electric field from GCN to MOF. This field accelerates the transfer of photogenerated electrons from MOF to GCN, reducing N₂ molecules and significantly enhancing the efficiency of photocatalytic N₂ fixation. The Physical mixing method produces better results than the chemical mixing. This synthesis approach can introduce useful defects, like linker vacancies in MOFs or surface irregularities in GCN, which enhance N₂ adsorption and activation. Furthermore, physical mixing avoids harsh chemical processing that could degrade MOF or GCN structures, preserving their intrinsic properties [2]. The enhanced NH₃ yield arises from defect-mediated charge separation and interfacial redox reactivity that accelerates electron transfer, highlighting the crucial role of mechanism elucidation in guiding photocatalyst design.

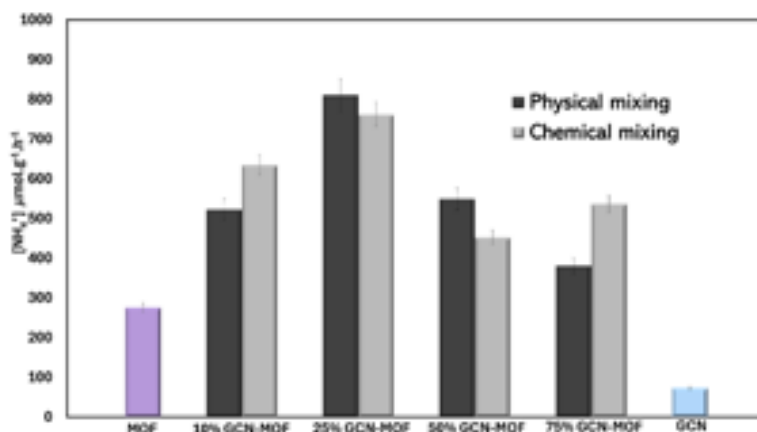


Fig.1: Photocatalytic ammonia generation using different catalysts

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Enhanced catalytic ozonation of oxalic acid using carbon nanotubes supported on glass frits

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The increasing presence of emerging contaminants, such as pharmaceuticals, in water resources is not fully mitigated by conventional water treatment processes. Catalytic ozonation is effective but commonly relies on suspended metal catalysts, raising separation and environmental concerns. Glass frits offer an inert, stable, and porous support, while carbon nanotubes (CNTs) are well-known metal-free catalytic alternatives, making their combination a promise for catalytic ozonation. This work investigates for the first time carbon nanotubes (CNTs) immobilized on glass frits as catalysts for oxalic acid (OXL) degradation, a common ozone-resistant byproduct of organic pollutants' ozonation [1]. To immobilize CNTs, first these were dispersed in ultrapure water using the surfactant Triton X-100 and an ultrasonic probe. Then, the glass frits were immersed in this solution using the dip coating technique, followed by calcination at 550 °C under N₂ to remove the surfactant and enhance CNTs adhesion. The material's catalytic performance was evaluated in recirculating batch ozonation experiments, using a gas stream of 150 Ncm³/min of O₃ containing 50 g/m³ of this oxidant. O₃ was continuously bubbled in a solution of [OXL]₀ = 10 mg/L, and adsorption and stability tests were conducted prior to the ozonation cycles. Results indicate that it was possible to impregnate 7.3 mg of CNTs, but a mass loss of 12% (6.4 mg CNTs) and 42% (3.7 mg CNTs) was observed after the stability and adsorption tests, respectively. After this, the material was tested for 5 consecutive cycles to infer about its reusability (Fig. 1a). Between ozonation cycles, there was no mass loss until the 5th cycle (18.7%) (Fig. 1b). Despite this loss, the material performed well in the catalytic ozonation process, achieving OXL degradation of up to 90% in 3 hours of reaction, with a decline in efficiency in the 4th cycle, and partial recovery in the 5th (~80% degradation) (Fig. 1c). One hypothesis for this behavior is that O₃ progressively regenerates the CNTs throughout the cycles, enhancing their porosity and, consequently, their efficiency. The adsorption test showed a maximum OXL removal of 6%, indicating that the main contribution to pollutant removal was indeed due to the reaction with ozone. Finally, the immobilization of carbon nanotubes on glass frits proved to be an efficient and sustainable strategy for catalytic ozonation, combining high catalytic activity with easy catalyst reuse.

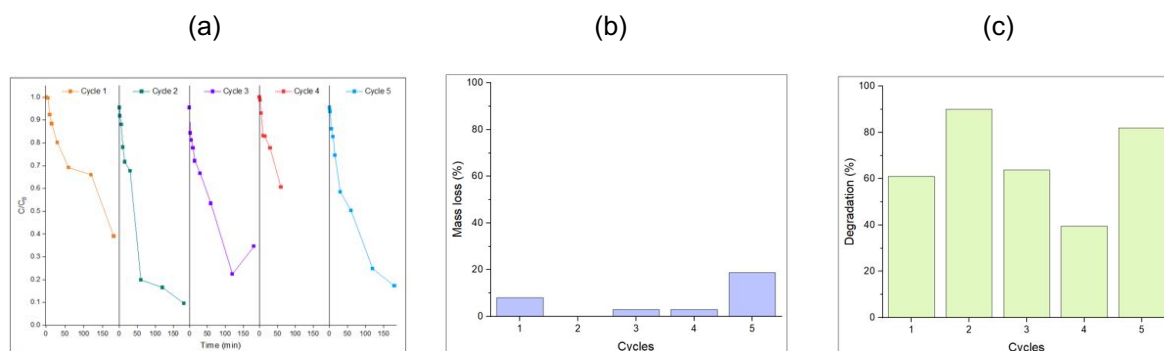


Fig.1. Mass loss of CNTs (a) and removal efficiency of oxalic acid (b).

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Desafios dos Museus de Química

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Partindo-se da premissa de que a química, como sendo uma ciência central, necessita de uma compreensão pública, então, tal implica ter uma representação significativa em museus de ciência e em centros de ciência.

No entanto, tem-se vindo a constatar que os museus de ciência e centros de ciência são frequentemente deficientes em química, comparando com a física e a biologia [1] [2]); e ainda, que os museus de química são pouco visitados pelo público, comparando com os outros museus de ciência.

Por um lado, as razões para tais factos poderão estar relacionadas com as dificuldades em mostrar os princípios da química, que poderão ser razões práticas e económicas. De facto, a construção e a manutenção de um espaço seguro e com sucesso requer bastante tempo e dinheiro [1] [2] . Por outro lado, existe na população, em geral, uma perceção negativa face à química [1], principalmente aquela com menor literacia científica. Acresce, ainda, que se tem verificado que alguns dos museus mais centrados na química têm sido museus históricos ou centros de conservação. A imagem da química que emerge dos museus de química poderá ser resumida, *grossa modo*, como estática e afastada da sociedade real [1].

Então, quais serão, atualmente, alguns dos desafios dos museus de química, no sentido de se mudar o paradigma existente até há pouco tempo?

Decerto, através dos museus poder-se-á conseguir um maior contacto com a sociedade e mostrar o papel e o impacto da química no dia a dia, em ambas as vertentes, a positiva e a negativa [1].

Têm sido apontados alguns aspetos a desenvolver nos centros de ciência, museus e exposições, como por exemplo, o investimento em atividades dedicadas aos estudantes, como laboratórios didáticos, demonstrações, visitas guiadas, experiências interativas, projetos educativos e *online*, e a inclusão de recursos multimédia, como bases de dados, visitas virtuais ou explicações interativas [1], assim como, a oferta de atividades de exploração, de descoberta e experiências novas [3].

Poderão, ainda, incluir-se os estudantes mais velhos nas atividades dos museus, seja através da catalogação, preparação de atividades ou como guias [1].

Os museus e centros de ciência deverão estimular e desenvolver a literacia científica do público [2], oferecer oportunidades para estudar os princípios da química e, ainda, mostrar a beleza dos princípios da química e a utilidade das suas aplicações [1].



Fig.1. Tabela Periódica com os espectros atómicos dos elementos químicos, Museu CosmoCaixa, Barcelona.

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O Arduino na Química dos ensinos básico e secundário: uma revisão sistemática da literatura (até 2025)

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A integração de tecnologias no ensino da Química, especialmente em contextos laboratoriais, tem sido identificada como um fator crucial para fomentar aprendizagens significativos e aproximar os alunos de práticas científicas autênticas [1] [2] [3]. O Arduino destaca-se neste cenário devido à sua versatilidade e baixo custo, permitindo democratizar o acesso à instrumentação científica e possibilitando experiências laboratoriais inovadoras, desde o ensino básico e secundário [4] [5]. Revisões de literatura recentes sublinham o crescimento acelerado do uso do Arduino na educação científica, com contribuições em variados domínios – analítica, ambiental e química física –, mas ressaltam a necessidade de uma sistematização clara da sua aplicação no ensino pré-universitário [3] [6].

Este estudo apresenta uma revisão sistemática da literatura guiada pela questão: “De que modo o Arduino tem vindo a ser utilizado no ensino da Química nos níveis de ensino básico e secundário?”. A revisão foi conduzida na base de dados SCOPUS, considerando artigos até 2025, nos idiomas inglês, espanhol e português, com critérios definidos segundo o protocolo PRISMA [7]. Após exclusão dos trabalhos direcionados ao ensino superior, foram selecionados 68 artigos, que passaram por uma triagem e leitura integral. Após esta etapa, apenas 12 artigos se focaram nos níveis de ensino básico ou secundário, ou na formação de professores, tendo sido realizada a codificação temática (área de aplicação, uso do Arduino, estratégias pedagógicas e resultados reportados) [5] [6].

A análise revela que a maioria dos estudos envolve a química analítica (ex.: espectrofotometria, titulação, ...) e instrumentação científica com sensores de baixo custo, seguidos por química ambiental (monitorização de água e gases), eletroquímica e química física (ex.: calorimetria). Destacam-se experiências de aquisição (automática) de dados e prototipagem (embora em menor extensão), com enfoques pedagógicos em aprendizagem por projetos, questionamento e integração em atividades prático-laboratoriais. Os resultados relatados apontam para ganhos de motivação, envolvimento dos alunos, aprendizagem conceptual e técnica, além de contributos para acessibilidade e inclusão no ensino da Química. Contudo, apontam-se desafios, como a falta de formação dos professores, limitações de recursos, dificuldades associadas à curva de aprendizagem técnica e à programação do Arduino, bem como risco de foco excessivo no dispositivo. Existe escassez de estudos robustos sobre ganhos efetivos de aprendizagem, apontando para a necessidade de investigações empíricas que avaliem resultados em contextos reais e integração curricular consistente.

Neste contexto, o Arduino destaca-se como ferramenta promissora para democratizar a experimentação e promover aprendizagens ativas em Química, devendo as limitações práticas e pedagógicas ser consideradas nas investigações futuras [4] [8].

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From Wildfire Hazard to Biorefinery Potential: Student Infographics on *Ulex europaeus*

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Infographics are increasingly used in education to promote active and critical learning by combining images, graphics and text to communicate complex information clearly and effectively [1]. Such tools can be used for agricultural and environmental engineering students, to focus and apply in local environmental challenges. A relevant example of study is *Ulex europaeus* (gorse), an invasive species that poses significant environmental risk in Galicia and northern Portugal due to its high flammability and biomass accumulation, which increases the likelihood of wildfires. In August 2025, Galicia experienced one of its most severe wildfire seasons, with over 120000 hectares burned, highlighting the urgent need for effective forest management practices. These include traditional methods like brush clearing, with help reduce fuel loads and mitigate fire risks. Simultaneously, gorse represents a biomass resource with multiple potential uses, offering an opportunity to integrate environmental management with sustainable resource utilization [2,3].

To address this issue, students used gorse as a “case of study” to explore sustainable solutions from the perspective of biorefinery concepts. They reviewed scientific literature, identified potential uses on how different parts of the plant can be utilized to produce bioproducts (including bioactive compounds, biofuels, biochemicals and materials), and evaluated practical strategies for biomass recovery and utilisation. Using infographics, students synthesized and presented their findings visually to their peers, promoting discussion and feedback. The most comprehensive and well-structured projects were selected for further consideration, highlighting the students’ ability to integrate scientific evidence with sustainability considerations and local environmental relevance. This approach fostered critical thinking, interdisciplinary problem-solving, and effective scientific communication, while promoting peer learning and collaborative assessment.

Overall, this case of study illustrates how combining infographic-based learning with biorefinery approaches provides a comprehensive framework for addressing local environmental issues. Students not only deepened their understanding of the ecological potential of invasive species, but therefore developed essential skills for future careers in agricultural and environmental sectors, with the added experience of presenting and defending their proposals to their peers. This process reinforced the connection between theory, practice, and sustainability-oriented innovation, while rewarding comprehensive and creative solutions.

Acknowledgements

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From chemistry to photonics: A multidisciplinary practice for nanoscience students

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In this contribution, we present a multidisciplinary hands-on practice for nanoscience students that brings together important concepts of chemistry and physics, while addressing the nanoscopic origins of the beautiful play of color exhibited by opals in nature. The practice consists in fabricating an artificial opal in the laboratory and analyzing how its structure, composed of stacked silica nanoparticles (NPs), produces its characteristic iridescent color.

It is ideal to start the practice revisiting the concept of color and its microscopic origins. Most students correctly describe color as the consequence of wavelength-selective absorption of light but fail to identify why some of them are iridescent, i.e., observation angle-dependent. During this discussion, opals are introduced as an example of natural photonic crystals, materials composed by a periodic structure that efficiently reflect light because of the spatial periodicity of their refractive index. Hence, students are invited to fabricate their own opals by synthesizing [1] and further stacking silica NPs [2]. In this stage, it is possible to let the students tune the synthesis parameters to obtain NPs of different size and use different characterization techniques included in the curricula of the nanoscience course to determine the size distribution of the NPs (DLS, SEM or AFM) or their composition (PXRD).

After the NPs synthesis, opal thin films can be prepared by evaporating at room temperature the solvent containing the NPs, a process that lasts about 2-3 days. These films present an iridescent color that the students must relate with the diameter of the NPs they are made of. This can be done qualitatively (by naked-eye identification of the reflection wavelength) or quantitatively, using a reflection spectrometer setup that the students can easily build by themselves, working specific competences associated to the photonics curriculum. From optical quantitative measurements the diameter of the NPs can be extracted from a least-square fitting of the data, yielding values remarkably close to those obtained by other characterization techniques.

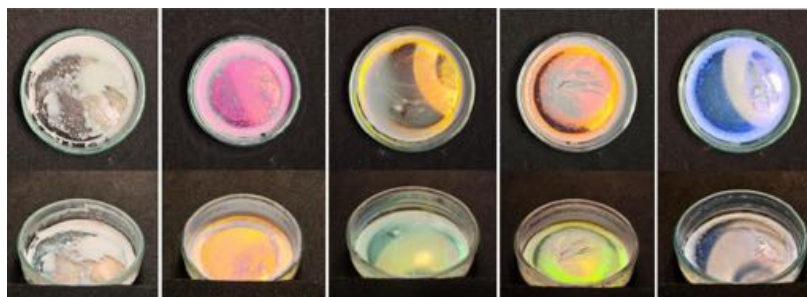


Fig. 1. Artificial opals fabricated during the practice under two different observation angles, 0° (top) and 30° (bottom). The diameter of the nanoparticles composing the opals increases from left to right, the same as the reflected wavelength.

Acknowledgements: Financial support was acquired from the Program of Consolidation and Structuring of Units of Competitive Investigation of Xunta de Galicia (ED431C-2021/56 and ED431C-2025/57). Terán-Baamonde (ED481D-2025-014) and Rodríguez-Fernández (ED481B-2024-101) acknowledge Xunta de Galicia for their postdoctoral grants and the financial support associated to their research lines.

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Nanocarrier Encapsulation of Quaternary Phosphonium Salts

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Quaternary phosphonium salts (QPS) exhibit broad-spectrum and potent antibacterial and antifungal activity, making them promising candidates as antimicrobial agents. Their bioactivity is strongly influenced by the alkyl chain length, which can be optimized to enhance efficacy [1]. Compounds with intermediate aliphatic chain lengths (C14–C16) demonstrate particularly high antimicrobial activity against microorganisms such as *Staphylococcus aureus*, *Escherichia coli*, and *Candida albicans*. However, this increased potency is often accompanied by higher cytotoxicity in human embryonic kidney (HEK293) and hepatocellular carcinoma (HepG2) cell lines [2]. Encapsulating QPS within nanocarrier-based drug delivery systems offers a potential strategy to mitigate these drawbacks by reducing toxicity, improving stability, enabling targeted delivery, and allowing controlled release [3].

In this study, two delivery platforms—nanostructured lipid carriers (NLCs) and liposomes were developed for the encapsulation of QPS with varying alkyl chain lengths (C8, C10, C12, C14, and C16). Following successful synthesis, the QPS-loaded nanosystems were characterized for their physicochemical properties, morphology, and time-dependent stability using dynamic light scattering (DLS). Encapsulation efficiency was determined by high-performance liquid chromatography (HPLC). Finally, their cytotoxicity was evaluated in human hepatocellular carcinoma (HepG2) and normal human dermal fibroblast (NHDF) cell lines by measuring their metabolic activity and cell mass. For NLCs, both NLC-C14 and NLC-C16 produced monodisperse populations with sizes below 200 nm. A similar outcome was observed for liposomes encapsulating C-12, C-14, and C16-QPS. Beyond yielding nanoparticles of an appropriate size for tissue delivery, the nanostructures incorporating C-16-QPS also demonstrated enhanced cell viability in HepG2 and NHDF cells compared with QPs alone. In liposomes, this effect was particularly pronounced in NHDF cells. Overall, these results indicate that an intermediate chain length not only enhances antimicrobial activity but also improves the stability and biocompatibility of the nanostructures.

Acknowledgements

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Algae-mediated green synthesis of gold nanoparticles as a potential strategy against melanoma

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Melanoma is one of the most aggressive and lethal forms of skin cancer, characterized by high resistance to conventional therapies and a rising global incidence, highlighting the urgent need for novel therapeutic approaches [1]. Gold nanoparticles (AuNPs) have emerged as promising tools in oncology due to their unique physicochemical properties, including high biocompatibility, stability, nanoscale dimensions, large surface area, and functionalization potential, which enable targeted antitumor applications [2].

In this study, AuNPs were synthesized from different previously obtained fractions of *Ulva* spp. [3] through multiple experiments in which concentration, temperature, and reaction time were varied. The nanoparticles were characterized in terms of morphology, crystalline structure, surface potential, and chemical composition using HRTEM, STEM, XRD, EDS, FTIR, and zeta potential. Their antioxidant potential was also evaluated through reducing capacity, total phenolic content, and radical scavenging activity.

In addition to characterization, the cytotoxic activity of the AuNPs was assessed in human melanoma cells with different features (A375 and SK-MEL-23) and normal human fibroblasts (BJ-5ta). Cell viability was assessed with the MTT assay after incubation with different concentrations of AuNPs and the corresponding extracts. *Ulva* fractions-derived AuNPs did not exhibit toxicity toward fibroblasts but some induced selective cytotoxicity towards melanoma cells, whereas the isolated extracts did not affect cell viability.

These results indicate that *Ulva* fractions-derived AuNPs may display tumor cell selectivity, representing a promising strategy for targeted therapies against melanoma. Future studies will focus on elucidating the molecular mechanisms underlying this selectivity.

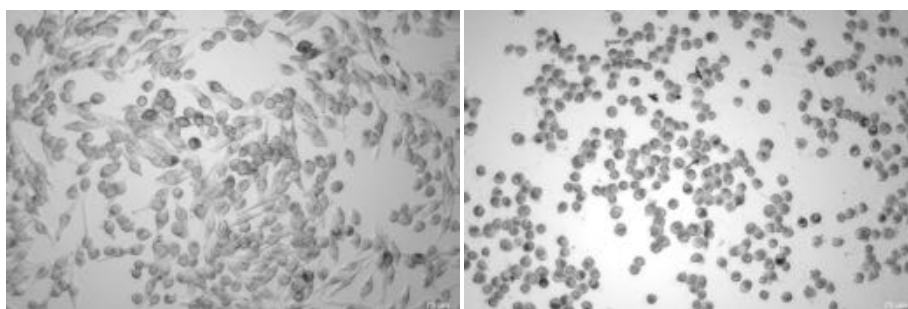


Fig. 1. Malignant melanoma cells (SK-MEL-23) in untreated conditions (left) and after 24 h exposure to AuNPs (125 µM; right).

Acknowledgements

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Ingeniería de nanomateriales funcionales: avances en síntesis y propiedades emergentes

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La síntesis de nanomateriales funcionales constituye un área estratégica dentro de la nanotecnología, debido a su potencial para generar materiales con propiedades emergentes imposibles de lograr en la escala convencional. Entre los métodos más relevantes destacan el proceso sol-gel, la síntesis hidrotermal y las técnicas de autoensamblaje molecular [1]. El control de parámetros como la temperatura, el pH, la relación de precursores y el tiempo de reacción permite obtener nanoestructuras con elevada homogeneidad, morfología definida y propiedades ajustables.

El proceso sol-gel, en particular, ha mostrado gran versatilidad en la obtención de óxidos metálicos con propiedades ópticas, catalíticas y magnéticas [2]. El confinamiento cuántico y la elevada relación superficie/volumen de estos nanomateriales se traducen en un comportamiento distinto al observado en materiales masivos. Dichas propiedades han abierto oportunidades en aplicaciones avanzadas como sensores químicos de alta sensibilidad, materiales para almacenamiento de energía, recubrimientos funcionales y aplicaciones biomédicas.

Además, el desarrollo de nanomateriales funcionales plantea un fuerte compromiso con la sostenibilidad. En los últimos años, se han incorporado métodos de síntesis verdes que reducen la toxicidad de los precursores y minimizan el consumo energético [3]. Estas aproximaciones consolidan un marco de innovación responsable, en línea con los objetivos de la química verde y la ingeniería de materiales orientada a un futuro sostenible.

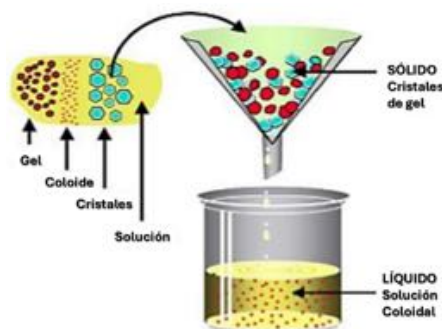


Fig.1. Esquema ilustrativo del proceso sol-gel mostrando la separación entre fases

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Functionalization of textiles with metallic nanoparticles obtained from *Undaria pinnatifida*

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Undaria pinnatifida (UP) is a brown macroalga native to Asia, currently listed among the world's 100 most invasive species [1]. In previous work, we demonstrated its potential for the sustainable synthesis of gold and silver nanoparticles (Au@UP and Ag@UP, respectively), yielding spherical polycrystalline nanoparticles with mean sizes of 6.8 ± 1.0 nm and 14.1 ± 2.8 nm. These nanoparticles exhibited promising antimicrobial activity, with Ag@UP showing potent antibacterial effects and both exhibiting strong antifungal activity even at low concentrations [2]. In this study, Au@UP and Ag@UP were incorporated into cotton and polyester-based materials to assess their bioactivity in functional textiles (Figure 1). Their binding, mainly due to the presence of hydroxyl groups [3], was confirmed through SEM and FTIR analysis. Antioxidant and antimicrobial evaluations revealed that nanoparticle immobilization on textiles reduced their antioxidant potential. Nevertheless, Ag@UP functionalized textiles retained strong antimicrobial activity, achieving > 90% bacterial reduction against *Staphylococcus aureus* and *Pseudomonas aeruginosa*. Biocompatibility assays using reference line L929 revealed the absence of cytotoxicity (> 80% cell viability). These findings highlight the safe and sustainable potential of UP-mediated nanoparticle-functionalized textiles, particularly with Ag@UP, as effective tools to tackle bacterial infections.

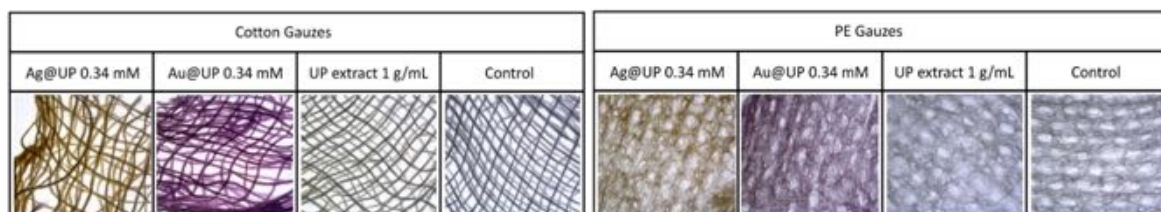


Fig.1. Cotton and PE gauzes functionalized with Ag@UP, Au@UP and UP extract, at indicated concentrations, and ultrapure water, as control.

Acknowledgements

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***Salvia Officinalis* L. extracts encapsulated in lipid-polymer hybrid nanoparticles for neurodegenerative diseases**

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Sage species have been used in ancient medicinal and culinary practices, given they present remarkable medicinal properties in preventing various diseases, including neurodegenerative diseases [1]. However, natural compounds tend to present poor bioavailability and low blood-brain barrier penetration. Nanomedicine presents itself as an interesting solution to overcome these limitations. Hence, lipid-polymer hybrid nanoparticles were developed to encapsulate *Salvia officinalis* L. extracts [2,3].

The extracts presented both antioxidant and iron-chelation activities as well as lipid peroxidation inhibition, among others. The lipid-polymer hybrid nanoparticles presented hydrodynamic size smaller than 200 nm and zeta potential values lower than -30 mV. Moreover, the nanoformulations did not present cytotoxic effects in dopaminergic neuroblastoma cells (SH-SY5Y) and caused a depletion on the ROS production of the cells, demonstrating an antioxidant profile. This work highlighted the importance of nanoparticles as a tool for improving natural compounds physicochemical properties and their translation for medicine.

Acknowledgements

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Otimização e caracterização da imobilização de nanopartículas de ouro em elétrodos modificados com MPTS

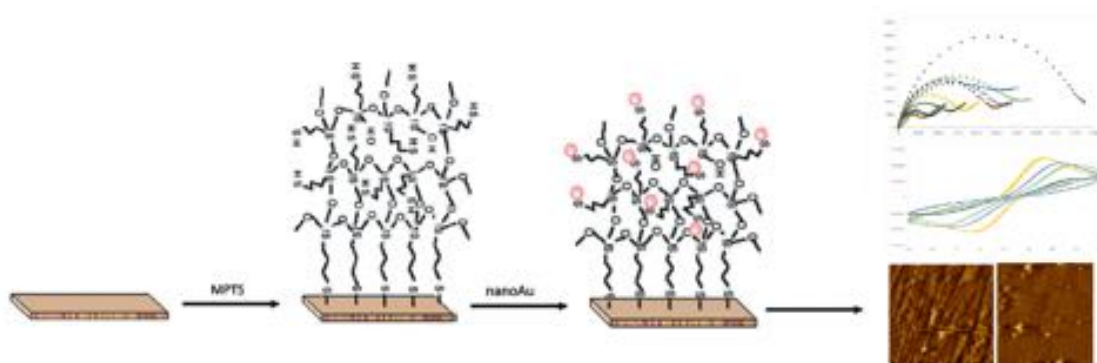
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A modificação de superfícies por meio de monocamadas automontadas (SAMs) tornou-se uma abordagem amplamente utilizada para conferir diferentes propriedades físicas e químicas, especialmente úteis na imobilização de biomoléculas. As SAMs são compostas por moléculas lineares que formam arranjos altamente organizados em grandes áreas, sendo as mais comuns formadas com alcanotióis ou organossilanos imobilizados na superfície de metais nobres, óxidos metálicos ou materiais semicondutores [1]. Organossilanos são moléculas ambifuncionais com a estrutura geral $(R'O)3Si-R-X$. Onde a porção alcoxisilano da molécula é capaz de se ligar a uma variedade de superfícies minerais ou metálicas por meio de reações complexas de hidrólise/condensação, onde ligações $Si-O-metal$ são formadas [2]. O 3-mercaptopropiltrimetoxissilano (MPTS) é um organossilano que apresenta grande versatilidade quando comparado a outros silanos, uma vez que a sua reatividade pode ser explorada para a reação de formação de ligação com superfícies de óxidos metálicos, enquanto o grupo tiol terminal pode ligar-se ao ouro e à prata. [3].

Alguns estudos avaliam as condições ideais dos processos de hidrólise e condensação e suas influências no processo de adsorção de silanos a superfícies [4]. O presente trabalho pretende avaliar e caracterizar através das técnicas de voltametria cíclica (CV), impedância eletroquímica (FRA) e microscopia de força atômica (AFM) a deposição de nanopartículas de ouro em superfícies de elétrodos à base de ouro e carbono vítreo modificados com MPTS, a fim de encontrar as melhores condições para trabalhos subsequentes sobre imobilização.



Esquema 1. Representação adaptada da deposição de nanopartículas de ouro em elétrodos modificados com MPTS, para posterior avaliação e caracterização por CV, FRA e AFM. [5]

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Optimization of superparamagnetic Fe₃O₄ nanoparticles for magnetic hyperthermia applications in aqueous media

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The conversion of electromagnetic energy into heat driven by superparamagnetic (SPM) magnetite (Fe₃O₄) nanoparticles is a promising strategy with applications ranging from biomedicine to energy systems [1]. Achieving high heating efficiency requires precise control over particle size, morphology and surface chemistry [2].

In this work, Fe₃O₄ nanoparticles were synthesized by thermal decomposition [2], optimizing surfactant combinations and synthesis reflux time to simultaneously improve size control and colloidal stability in aqueous media. Structural and magnetic characterizations confirmed the formation of monodisperse, highly crystalline nanoparticles with high saturation magnetization [3].

Magnetic hyperthermia experiments yielded ILP values above 3 nHm²/kg, highlighting the heat efficiency gained through synthesis optimization. Particle sizes of 14–16 nm were identified as optimal to maximize the superparamagnetic response [3], guiding the rational design of efficient magnetic nanofluids for energy store systems.

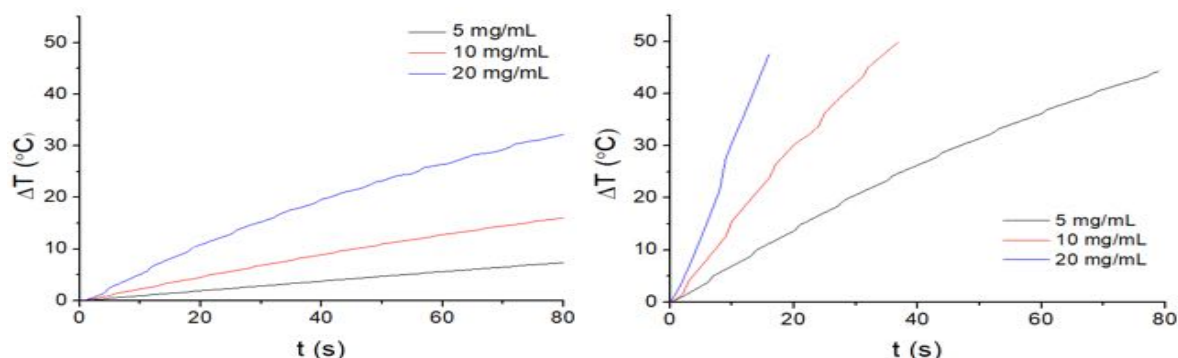


Fig.1. Temperature increase (from R.T.) of SPM Fe₃O₄ nanoparticles before optimization (left) and after optimization (right).

Table 1. Structural, magnetic and hyperthermia properties of Fe₃O₄ nanoparticles before and after optimization.

Sample condition	Size (nm)	Morphology	Ms (emu/g _{mag})	ILP (nH m ² kg ⁻¹)
Before optimization	12-14	Spherical-cubical	96	0.442-0.592
After optimization	14-16	Fully spherical	106	3.14-3.41

Acknowledgments

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From nature to batteries: solid electrolytes produced by blending biopolymers boosted with sodium bis(fluorosulfonyl)imide and graphene oxide

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Recently, solid polymer electrolytes (SPEs) have attracted significant interest as key components for safe and high-performance batteries. The use of natural polymers, such as agar (A) and methylcellulose (MC), offers environmental and biocompatibility advantages over synthetic polymers while maintaining good mechanical and chemical properties. [1] MC is a water-soluble cellulose derivative, widely used in biocompatible materials, while agar, a polysaccharide from red algae, is valued for its gelling ability and biological compatibility [1-2]. The incorporation of sodium salts, such as sodium bis(fluorosulfonyl)imide (NaFSI) and graphene oxide (GO) can enhance the ionic conductivity, electrochemical stability, and improve the mechanical and thermal properties of the materials, making these systems promising for solid-state devices. [3-4]

In this study, films were prepared using a mixture of A and MC as the polymer matrix, to which NaFSI in dimethyl carbonate:ethylene carbonate (DMC:EC) and GO were added. MC was dissolved in ultrapure water at room temperature under magnetic stirring, while agar was dissolved in ultrapure water at 90°C. Once the agar solution reached 60°C, it was mixed with MC to prevent both its precipitation, and premature agar gelation. Subsequently, NaFSI and/or GO were added to each system, under stirring, following the desired formulation. Each sample was poured onto Petri dishes and incubated until uniform films were formed. Figure 1 (a) shows the control sample, containing only the mixed polymers (1:1 wt./wt.). As seen in the photo, the polymers are mixable, producing a clear transparent film. Figure 1 (b) shows the A+MC+NaFSI sample. The transparent film obtained is expected to have an increased ionic conductivity. [3] Figure 1 (c) shows the A+MC+NaFSI+GO sample, and due to the presence of GO, the sample's color is darker. GO addition is expected to increase the amorphous content of the film while enhancing its thermal stability. Characterizations techniques were performed to evaluate the physical-chemical characteristics and ionic conductivity of the films.

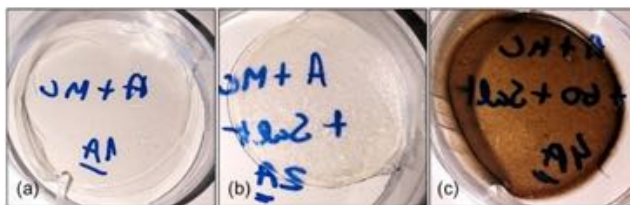


Fig.1. Photographs of the A+MC sample (a), A+MC+NaFSI sample (b), and A+MC+NaFSI+GO (c)

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Development of a nanoparticle formulation with anti-inflammatory and skin lipid replenishment effects

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Skin diseases are estimated to be the fourth most common cause of all human diseases, affecting almost a third of the world's population, but their impact is often underestimated. The stratum corneum (SC), the outermost layer of the skin, provides the first and most elaborate barrier to skin permeation of allergens and aggressive agents, but also of cosmetic and pharmaceutical compounds. The barrier function of the SC depends mainly on the complex structure and organization of the intercellular lipid matrix, which consists of a combination of ceramides, fatty acids, and cholesterol, forming a multilamellar structure [1, 2].

Olive pomace is a solid byproduct of olive oil processing, consisting mostly of the skin, flesh, and seed remains of the olive after processing, and contains mainly carbohydrate, lipids, phenols, and inorganic compounds [3]. These polyphenols are reported to have various beneficial effects, namely their anti-inflammatory activity, which makes a polyphenol extract prepared from olive pomace a natural material that can be used to provide anti-inflammatory properties to nanoparticles [4 – 6].

In this project, we aimed to develop Solid Lipid Nanoparticles (SLN) loaded with olive pomace extract due to its skin inflammation treatment potential. Concomitantly, they were designed for skin lipid replenishment, using also biomimetic lipids, reinforcing the desired cosmetic and pharmaceutical applications.

The optimized formulation has a mean diameter of 230nm, polydispersity index of 0.18 and a zeta potential of -33 mV. Such characteristics contribute to its good storage stability at room temperature. Regarding the incorporation percentage and incorporation capacity they revealed to be 25.5% and 4.9 %. These outcomes indicate that the production of the nanoparticle formulation was successfully achieved highlighting its skin lipid replenishment potential.

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Exploring the conjugation of tetrapyrrolic macrocycles with carbon nanomaterials: New Applications and Challenges

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Photodynamic therapy (PDT) has been considered as a non-invasive treatment with less side effects when compared to conventional cancer treatments. In this therapeutic approach, the activation of molecules by visible or near infrared light irradiation at an appropriate wavelength in the presence of dioxygen is fundamental to generate cytotoxic reactive oxygen species (ROS), responsible by the oxidation of cellular constituents in the target tissue provoking the cell death. Ideally, the dyes, known as photosensitisers (PS), should be compounds with insignificant dark toxicity and some degree of hydrophilicity [1].

Among the high number of PS that have been studied in vitro and in vivo a special attention has been dedicated to porphyrins, corroles and phthalocyanines due to their high singlet oxygen yield, low toxicity and unique optical and electronic properties. However, the poor water solubility of tetrapyrrolic macrocycles can lead to formation of aggregates compromising their use in PDT.

To prevent aggregation, several studies have been focused on the incorporation of PS into nanomaterials such as carbon nanomaterials. The development of conjugates based on corroles/porphyrins /phthalocyanines and carbon nanomaterials for cancer therapy is a recent topic of research with promising results found for graphene quantum dots (GQDs) [2].

GQDs possess excellent optical and electronic properties coupled with high photostability, aqueous solubility and biocompatibility. The presence of carboxyl and hydroxyl groups on their surface and edges enable covalent attachment, electrostatic interactions and hydrogen bonding with other suitable moieties. In the present study GQDs have been conjugated covalently and non-covalently with corroles, porphyrins and phthalocyanines. The optical and structural characterization of the resultant hybrid materials is discussed [3]. Preliminary studies on cellular uptake and distribution, using confocal and multiphoton microscopy, are presented.

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Enhancing food allergen detection: the impact of biorecognition platforms in ELISA and electrochemical immunosensors

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The enzyme-linked immunosorbent assay (ELISA) is a widely recognized and reliable immunoassay for the quantitative detection of food allergens [1]. The aim of this study was to examine batch-to-batch consistency, optimize detection parameters, and assess the robustness and sensitivity of each biorecognition platform to ensure reliable detection and quantification of the Gly m TI allergen in model foods. Antibody batch performance was evaluated against an electrochemical immunosensor used as a functional substrate for antigen immobilization [2].

To investigate this, four batches of anti-Gly m TI IgG (Ab1, Ab2, Ab3 and Ab4) were evaluated, with pairs produced either within the same year or five years apart. Antibodies from the same year but from different batches showed consistent behavior in terms of sensitivity, dynamic range, and dilution ratio. In contrast, their performance differed significantly from that of antibodies produced five years earlier. Specifically, Ab1 and Ab3 (from 2019) outperformed Ab2 and Ab4 (from 2024), offering an extended dynamic range of 14 orders of magnitude and detecting as little as 0.1 mg/kg of the target protein in model foods (biscuits, hams and sausages), irrespective of processing conditions. Moreover, these earlier batches required concentrations 40 times lower to achieve effective Gly m TI detection. The reduced binding affinity and overall effectiveness of Ab2 and Ab4 limited ELISA development, as the resulting sensitivity fell well short of expectations, a finding further supported by the weaker performance of immunosensors using the same antibodies.

This study's outcomes underscore the versatility and robustness of the indirect ELISA for immunodiagnostic applications while also highlighting the potential of electrochemical immunosensors for achieving greater reproducibility, particularly in the presence of antibody lot variability. To our knowledge, this represents the first study comparing multiple antibody batches across both ELISA and biosensing platforms. These findings also suggest opportunities for future development of multiplex allergen detection and high-throughput screening.

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Development of a flow methodology for creatinine quantification in sports supplements

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Creatine is among the most widely used and thoroughly studied sports supplements for performance enhancement. Given its widespread use, the accurate and reliable determination of creatine and its degradation product, creatinine is critical for quality assurance and regulatory compliance [1]. Conventional analytical methods, namely chromatography, require complex and laborious sample preparation, and are costly, which makes them impractical for routine high-throughput testing.

In this context, this project aims to develop and validate a simple, cost-effective analytical method that supports efficient quality control in industrial settings. The method to be developed will be based on a flow analysis technique, which offers important advantageous features such as high sample throughput, low reagent consumption, and strong automation potential [2].

A key innovation lies in the integration of a novel chemical detection reaction for creatinine, a key marker for product stability and quality. The detection will be based upon the spectrophotometric quantification of the colored product formed involving NED and nitrite. The parameters for the best performance of the chosen reaction will be optimized, including the reagent and sample volumes, the flow-rate, the reagents concentration, among others. In the end, a detailed interference study is carried out to provide a robust alternative to interference-prone standard methods. Moreover, the proposed method will be a greener alternative to the commonly used Jaffé reaction, which, despite its simplicity, suffers from poor selectivity and relies on reagents such as picric acid that raise environmental and safety concerns [3].

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Impact of Physical Pretreatments on the Reduction of Antinutritional Factors in Lentil Flours

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Lentils (*Lens culinaris* spp.) are one of the oldest cultivated leguminous worldwide. Although lentils are recognized as nutritious leguminous, which makes them a suitable option in vegetarian diets as an alternative to meat products, their composition and nutritional characteristics vary between different varieties. In addition, the overall composition and health benefits of lentils can be influenced by the presence of other components known as antinutritional factors (ANFs), which can exert effects contrary to optimal nutrition. The presence of ANFs can be modulated through various processing methods, some of which have been associated with improving the nutritional value of legumes by partial or total suppression of ANFs, as well as increasing certain nutrients, protein and fiber digestibility, and mineral bioavailability.

Thus, the aim of this work is to investigate the effect of different physical pretreatment techniques, namely soaking, cooking, germination and microwave roasting, on specific ANFs (phytates, condensed tannins and trypsin inhibitors) in lentils. The results showed a significant influence ($p < 0.05$) on the total concentration of ANFs, with germination and cooking standing out as the most effective in reducing their presence in the overall lentil flours. Phytic acid was completely eliminated during germination, due to its degradation by the phytase enzyme activated during this process, while condensed tannins and trypsin inhibitors were more affected by cooking, where the catalytic enzymes and the thermolabile nature of the compounds are mainly responsible for their greater reductions.

The results obtained showed that all processing methods exerted some influence on the ANFs analyzed, allowing better inferences about the behavior of lentil flours in the formulation of new food products through different methods that can meet the current nutritional and health demands of today's society.

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Development and Validation of an HPLC-DAD Method for the Simultaneous Quantification of Reduced and Oxidized Glutathione

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Oxidative stress is defined as a state of imbalance between the generation and elimination/degradation of reactive oxygen species (ROS) [1], [2], resulting in an elevated intracellular level of these species [3]. At moderate concentrations ROS play important roles, acting as second messengers and regulating intracellular signal transduction pathways [2]. However, in the case of imbalances, an excess of ROS can cause lipid, protein and DNA damage [3], leading to metabolic dysregulation [2]. One of the mechanisms our organism has to prevent oxidative stress is the GSH-GSSG system, which involves a molecule called glutathione, in its reduced (GSH) and oxidised (GSSG) form [4]. Ideally, this system functions as a cycle, with GSH reducing ROS and forming GSSG, which is then converted back into GSH by glutathione reductase [4], [5]. One of the most frequent ways to evaluate the cellular redox balance is through the GSH/GSSG ratio [2], [5], [6]. However, many conventional techniques for glutathione quantification lack specificity or sensitivity, making HPLC a more robust and accurate alternative for simultaneous determination of GSH and GSSG. With this in mind, the aim of this work was to develop and validate a method for the accurate quantification of glutathione, with potential applications in different biological systems, particularly in HT29 and Caco-2 cell lines [7], [8].

In order to achieve optimal chromatographic separation of GSH and GSSG, several parameters were systematically optimized. Different solvents and acidic modifiers were tested, including acetonitrile (ACN), methanol, H₂O, H₃PO₄, trifluoroacetic acid (TFA) and HCl. Variations in organic content and gradient profiles were also explored to improve peak separation and stability. In addition, flow rate, injection volume, and column temperature were adjusted to enhance sensitivity and reproducibility. The best results were obtained with an isocratic mobile phase consisting of water with 1% ACN and 0,1% TFA, combined with a flow rate of 1 mL/min, 10 µL injection volume, and oven temperature of 30 °C. The construction of calibration lines showed good R² values (R²_{GSH} = 0,9998 and R²_{GSSG} = 0,9998) in the 0,7 – 10 µM range, and additional validation protocols confirmed a LLOQ of 0,7 µM for both forms of glutathione. Further protocols are being implemented to fully validate the method across the forementioned range, in hopes that it may serve as a simple but effective method to quantify intracellular glutathione. Its specificity and ability to simultaneously detect GSH and GSSG represent clear advantages over other techniques, making it a valuable tool for studies of redox balance in biological systems.

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Compost as a Sustainable Adsorbent for Heavy Metal Removal from Water

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Water contamination by heavy metals is a critical environmental issue due to their persistence, non-biodegradability, and potential to accumulate in aquatic organisms. Among these contaminants, nickel, copper, and zinc are of particular concern, as they are widely released into aquatic media through industrial activities, mining, and agricultural runoff. Their presence in water bodies can compromise ecosystem health and pose risks to human well-being. The limitations of conventional treatment methods highlight the need for sustainable, cost-effective, and environmentally friendly strategies for the removal of these metals [1-3].

Activated carbon adsorption has been widely employed for this purpose, however, its production cost limits large-scale application [4]. To enable broader implementation, the use of lower-cost and more readily available adsorbents is highly desirable. Compost, obtained from organic wastes, has been investigated as a promising alternative for this purpose [5, 6]. In addition to fulfilling the desired performance criteria, it also adds value to organic wastes, thereby contributing to the reduction of the amount of material sent to landfill.

In this study, the use of compost to remove nickel, copper and zinc from water by adsorption was evaluated. Experiments were conducted to assess how different operational conditions affect adsorption performance. The effect of contact time, initial metal concentration, compost dose, and solution pH on adsorption efficiency was studied. In addition, adsorption experiments were carried out both individually and in competition for nickel, copper, and zinc. The metal concentrations in solution were determined by photometry or by inductively coupled plasma mass spectrometry. Adsorption capacities of 18.9, 25.3, and 35.5 mg g⁻¹ and removal efficiencies of 95.6, 95.1, and 95.5% were observed for nickel, copper, and zinc, respectively, in single-metal experiments. The results show that compost has strong potential to be used as an effective and sustainable adsorbent for removal of nickel, copper and zinc from water.

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Total phenolic content assessment with a developed microfluidic paper-based device method

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In this work, the design of a microfluidic paper-based analytical device (μ PAD) for the determination of total phenolic content (TPC) by the Fast Blue BB (FBBB) method [1] is described. The choice of this method was due to its feature of not reacting with non-phenolic compounds, unlike other methods usually employed for the determination of TPC. In addition, as far as we know there are not μ PADs developed for TPC determination employing the FBBB method.

Paper devices are cheaper than traditional spectrophotometric methods, more accessible, and portable. Small amounts of reagents are used, and they are easily disposable. For the device construction, a vertical flow 3D arrangement was chosen and filter paper discs aligned within perforated laminating pouches was the procedure used to establish hydrophilic and hydrophobic areas [3]. This procedure is more environmentally friendly than other techniques such as wax printing, one of the most used procedures. In fact, currently wax printing has limited application as is no longer easily accessible as commercial wax printers have been discontinued due to their reported harmful and toxic consumables [4].

For the optimization of the TPC determination μ PAD, several variables associated with the design of the device were studied, namely filter paper porosity, and paper disc size. Afterwards, different standards were tested: gallic, caffeic and trans-ferulic acids, catechin and epicatechin. This testing intended the application to different samples such as wine and herbal infusions. The studies were carried out in Gallic Acid Equivalents (GAE) for tea/infusion samples and in Catechin equivalents (CaE) for wine samples. The developed μ PADs (Fig 1.) achieved a linear range of 5-200 mg GAE/L and 25-125 CaE/L with a limit of detection of 4.20 mg GAE/L and 5.29 mg CaE/L, and a limit of quantification of 14 mg GAE/L and 17.6 mg CaE/L. Precision and accuracy below 11% were obtained and stability studies under different conditions were also performed.

The designed device was stable for 7 days when stored at room temperature in vacuum atmosphere and the color reaction stability depended on the standard, up to 30 min for GAE and up to 60 min for CaE. The transfer of the traditional spectrophotometry to μ PADs demonstrates that this last one is a more sustainable technique as AGREE scores improve from 0.58 to 0.75.

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Gold detection on dry Ti-Au thin films electrodes for biopotential monitoring

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The ageing of the population has been progressively increasing, accompanied by a growing demand for medical care and the need for effective remote rehabilitation solutions. To prevent the overload of healthcare services, it is crucial to develop more efficient and accurate monitoring systems, as well as advanced rehabilitation platforms capable of delivering therapy directly to individuals without requiring travel [1]. A promising approach involves the development of smart wearables that integrate sensing devices capable of continuously monitoring physiological parameters. In particular, electrodes for biopotential acquisition, such as those used in electromyography, are central to the diagnosis of neuromuscular conditions and the optimisation of functional electrical stimulation therapies.

Traditionally, these signals are collected using wet Ag/AgCl electrodes, which, despite their high signal quality, present limitations such as short durability, dependence on conductive gels, and the possibility of skin irritation [2]. Biocompatible dry electrodes overcome some of these drawbacks, offering improved durability, recyclability, and easier integration into portable devices. However, issues related to contact impedance, motion artefacts, and stability in sweat still drive the development of new material solutions [3]. In this context, Ti-Au films were deposited onto polymeric resin substrates previously printed via stereolithography using a commercial resin. The combination of Ti with Au aims to enhance electrode durability and electrical performance in physiological environments [4]. To assess their stability, the Ti-Au electrodes were immersed in an artificial sweat solution (ISO standard 3160-2) for different durations (4 h, 24 h, 48 h, 4 days, 7 days, and 10 days) simulating a corrosive environment.

The aim of this study was to investigate the degradation behaviour of these dry electrodes in a corrosive environment simulating human sweat. After each degradation interval, the artificial sweat solution was analysed to quantify the concentration of Au released from the electrodes, thereby assessing their stability in this corrosive medium. Gold was first characterised by cyclic voltammetry, evaluating the effect of scan rate and pH (1–10), prior to the optimisation of deposition parameters in anodic stripping voltammetry. Subsequently, an anodic stripping voltammetry method was developed and optimised for the quantification of Au³⁺, with definition of optimal conditions, calibration curve construction, and assessment of analytical parameters. In parallel, other methodologies, UV-Vis absorption spectrophotometry and inductively coupled plasma atomic emission spectroscopy, were employed to characterise and validate the results obtained via the electroanalytical method, also involving calibration curve construction and performance evaluation.

This study demonstrates that the Ti-Au thin film combination represents a promising choice, as unlike the degradation observed in previous studies with Ti-Cu and Ti-Ag electrodes [3], no measurable release of gold was detected, indicating the high longevity of these electrodes. These findings are of great relevance in the current context of healthcare and remote rehabilitation, contributing to the advancement of research into more durable dry electrodes suitable for prolonged use in direct contact with the skin.

Acknowledgements

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Alkyl caffeates: electrochemical characterization and correlation with antioxidant protection

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Oil-in-water emulsions are widely used not only in the food industry, but also in the cosmetic, pharmaceutical and medical industries [1]. However, these applications face a serious problem due to the low oxidative stability of emulsified systems. To retard or inhibit the oxidative process and increase the health aspect and nutritional value of foods, addition of an antioxidant agent, such as a phenolic acid, is a very appealing strategy. However, phenolic acids suffer from a lack of bioavailability due to their high hydrophilicity. Esterification of these compounds with alkyl chains has proven to efficiently increase their lipophilicity and even antioxidant activity [2].

In this work, a series of previously synthesized alkyl caffeates [3], with chains varying from C1 to C16 were tested using cyclic voltammetry, in order to establish the effect of increasing lipophilicity on potential antioxidant activity [4]. The voltammetric measurements showed a strong correlation with the radical scavenging capacity previously determined using the DPPH radical assay [3]. Furthermore, it was observed that increasing the alkyl chain length alters the oxidation mechanism, shifting from a diffusion-controlled to an adsorption-controlled process (Figure 1).

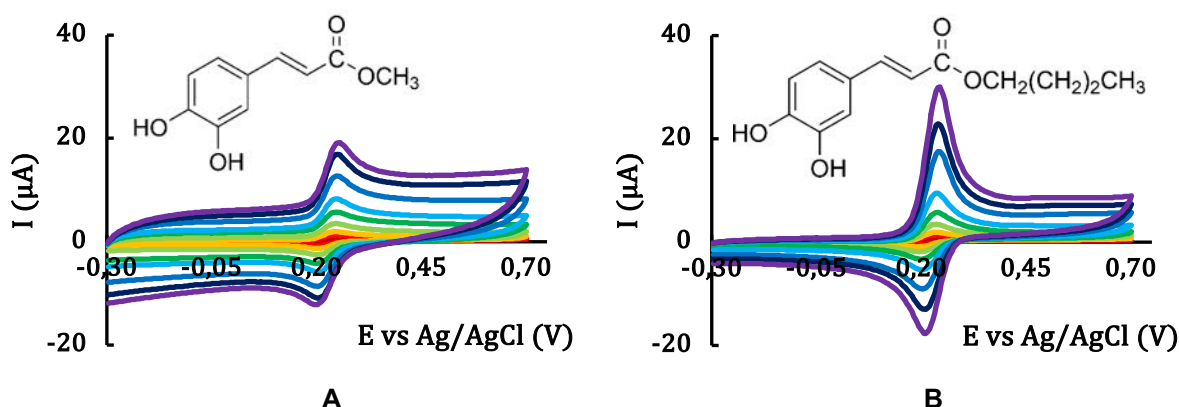


Fig.1. Effect of the scan rate on the shape of cyclic voltammograms recorded at a GCE for 0.1 mM solutions of: (A) CA1 – diffusion-controlled process; (B) CA4 – adsorption-controlled process. Scan rates used: 5, 10, 20, 50, 100, 200, 400, 600, and 800 $\text{mV}\cdot\text{s}^{-1}$.

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Validation of Methods for the Determination of Alcohol Content in Artisanal Spirits: Alcoholmeter and Refractometer

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The determination of alcohol content is a fundamental parameter for quality control and legal compliance in alcoholic beverages, being particularly relevant in artisanal products, where greater variations may occur due to the diversity of raw materials and production processes [1].

This study aimed to validate methods for determining the alcohol content in artisanal spirits using a Gay-Lussac alcoholmeter and a refractometer, two low-cost and easily implemented analytical techniques. Calibration curves for both instruments were constructed using hydroalcoholic solutions, with temperature corrections applied to alcoholmeter readings and an empirical correction factor determined for the refractometer. The evaluated performance parameters included quantification limit, linearity, repeatability, intermediate precision, trueness, selectivity, and measurement uncertainty [2]. Method equivalence was demonstrated through the analysis of 22 samples of artisanal spirits (Figure 1), and the advantages of each approach were also discussed. The results show that the use of accessible equipment allows for the adequate assessment of alcohol content in samples containing between 19 and 64 % (v/v) ethanol, representing a viable alternative for contexts with limited laboratory resources.

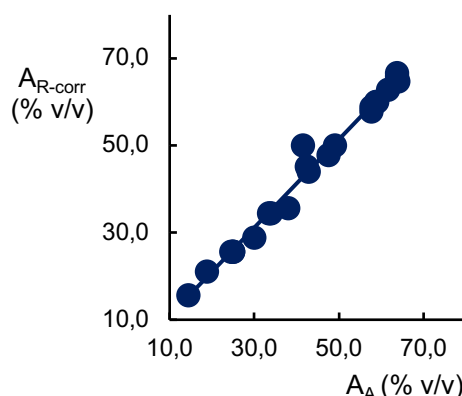


Fig.1. Comparison of alcohol content in artisanal spirits measured by refractometer (AR-corr) and Gay-Lussac alcoholmeter (A_A).

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Evaluation of analytical strategies for in vitro gastrointestinal bioaccessibility/bioavailability studies of metallic species (free and desorbed from polymeric microparticles)

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The presence of microplastics (MPs) in the environment has become a subject of significant concern due to their environmental persistence and their potential to act as vectors of metallic additives. These additives may be released during gastrointestinal digestion, which could pose toxicological risks by disrupting cellular metabolism. [1]

In this study, a range of analytical approaches were assessed to evaluate the bioaccessibility and bioavailability of these metals, both as free species and as released from polymeric matrices. In vitro digestion assays were performed using different static models (UBM and INFOGEST protocols) [2,3], complemented with diffusion systems based on dialysis membranes and natural animal intestines to reflect the physiological complexity of mammalian barriers (Figure 1a), as well as Caco-2 intestinal cell cultures. The quantification of bioaccessible and bioavailable metal fractions was conducted by ICP-MS, following the implementation of appropriate treatment steps on these diverse fractions.

The findings suggest that extended digestion times in UBM result in higher bioaccessibility and bioavailability. Natural intestinal tissues have been shown different (and probably) more realistic data compared to polymeric membranes, which is a probe of the physiological complexity of mammalian barriers, such as the greater surface contact between the real intestinal structure and the digestion medium. The metal bioaccessibility/bioavailability exhibit divergent trends, as evidenced by the findings of the present study. In the context of Caco-2 assays, microwave-assisted digestion has been demonstrated to surpass conventional lysis protocols in terms of its efficacy for the determination of intracellular metal content. Furthermore, the cellular-level intestinal absorption of metals was determined at different times, including the evaluation of cell morphology or apoptosis processes by optical microscopy (Figure 2a).

Overall, this study highlights the importance of combining complementary methodologies to achieve a realistic evaluation of the toxic behavior of metallic species and underscores the need to develop standardized approaches that can support risk assessment and regulatory frameworks.

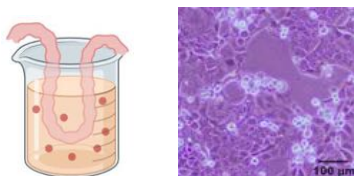


Fig.1. (a) Representation of bioavailable assay using natural intestines, and (b) Optical microscopy picture of Caco-2 cells after incubation with bioaccessible fractions.

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Mercury enrichment approaches based on paper-based analytical devices and nanomaterials prior to direct mercury analyzer

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Mercury (Hg) is a highly toxic metal that supposes a global threat to human health and the environment, due to its persistency and accumulation through the food chain. In the literature, several methods have been published focused on its determination and preconcentration especially in drinking waters [1,2]. Maximum allowed levels of Hg in drinking water was 1 µg/L according to the European Commission. Lately, nanomaterials are advantageous for sensing and enrichment of pollutants [2]. Also, cellulose-based materials have been used in multitude of applications in the analytical process due to its abundance, biodegradability, easiness of chemical modification and excellent sorption properties [3].

In recent years, in our group we developed different methodologies for trapping Hg based on the use of paper-based analytical devices (PADs) and nanoparticles (NPs): i) PADs modified with Ag NPs [4], ii) magnetite NPs modified with Pd (Fe₃O₄/Pd) [5] and iii) formation of core-shell magnetic NPs (Fe₃O₄@HgS) [6]. Firstly, PADs modified with Ag NPs were used as filters for trapping Hg(II) through amalgamation without the need for a previous reduction of Hg(II) to Hg(0) [4]. The synthesis was based on chemical reduction of Ag⁺ to Ag⁰ from AgNO₃ by NaBH₄ by directly placing the filters sequentially inside these solutions. Thus, Ag NPs in the range of 30-60 nm were measured by scanning electron microscopy. Then, 100 mL of standards or sample solution at pH 4 were filter through a 1 cm diameter filter paper through a Dr. Hirsch filter, allowed obtaining an enrichment factor of 3500 and a limit of detection of 0.2 ng/L Hg. Secondly, an in situ ultrasound-assisted co-precipitation method was applied to prepare magnetic nanocomposites (Fe₃O₄/Pd) for Hg(II) preconcentration [5]. The method was applied to 18 mL of standard or sample solution by adding FeCl₂ and FeCl₃ and 2.6 mL of NH₃ solution and adding Pd, for 5 min in the cup-horn sonoreactor, where finally, 50 µL of the enriched magnetite nanocomposite were formed. For a 5 µg/L Hg(II) concentration, a Hg/Pd ratio between 1:5 and 1:100 showed the highest increase in sensitivity. The particle size was determined by transmission electron microscopy in the range 7-10 nm. The limit of detection obtained was 3.2 ng/L Hg(II) and the preconcentration factor achieved was 100. Thirdly, magnetite previously synthesized were used for the extraction of Hg(II) at pH 9 in presence of L-cysteine, which in ultrasound energy produced the formation of core-shell NPs and trapping of Hg as Fe₃O₄@HgS. The preconcentration factor achieved was 91 and the detection limit of 40 ng/L using 20 mL of sample volume and 1 mL of magnetite nanoparticles. In the last two applications, it is remarkable the easy magnetic separation of the Hg enriched-NPs from the aqueous phase, thus avoiding additional steps of filtration or centrifugation. In the three applications, a direct mercury analyzer (DMA) was selected as the analytical technique for specific Hg quantification, since it allows direct analysis of the filter paper from PADs-Ag-Hg and Hg-enriched magnetite (Fe₃O₄/Pd-Hg and Fe₃O₄@HgS) without requiring analyte elution or digestion from the sorbent phase, thus, simplifying the procedure.

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Plasma and whole blood manganese concentrations in chronic haemodialysis patients vs healthy individuals

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Chronic kidney disease (CKD) is an irreversible condition characterised by a gradual decline in renal function. End-stage kidney disease (ESKD), the most advanced stage of CKD requires kidney replacement therapy (KRT). Haemodialysis (HD) is the most common modality of KRT [1], and previous studies have shown that HD patients are at an increased risk of trace element imbalances [2]. Manganese (Mn) has been the focus of investigation in this context. However, evidence remains inconsistent: some studies report increased serum/plasma or whole blood concentrations [3], while others describe decreased levels [4] or no significant differences compared to healthy individuals [5].

This study compared the manganese concentration in both whole blood and plasma samples collected from patients undergoing chronic regular HD therapy and healthy individuals (controls). Blood samples were collected by conventional venipuncture into trace element-free whole blood (K₂EDTA) collection tubes. After careful homogenization, an aliquot was transferred to a metal-free Eppendorf tube, and the remaining blood was centrifuged to obtain plasma. Samples were analysed by inductively coupled-plasma mass spectrometry (ICP-MS) using a properly validated procedure and adequate analytical quality control.

A total of 154 HD patients and 86 healthy individuals were included in this study. The median (P25–P75) plasma Mn concentration was 0.471 (0.380–0.564) µg/L in HD patients and 0.525 (0.418–0.706) µg/L in healthy controls, with a statistically significant difference between the two groups ($p < 0.001$, Mann-Whitney U test). In contrast, whole blood Mn concentrations did not differ significantly between groups ($p = 0.725$, Mann-Whitney U test). The median (P25–P75) whole blood Mn concentration was 7.92 (6.53–10.9) µg/L in HD patients and 7.82 (6.37–10.6) µg/L in healthy controls.

These findings suggest that observations on trace element imbalances in CKD patients undergoing chronic regular HD therapy may differ depending on the analysed specimen (serum, plasma, whole blood). Further studies evaluating trace element levels in different blood fractions are necessary to elucidate the underlying mechanisms and clarify the clinical significance of these imbalances.

Acknowledgements

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Validation of a near-infrared spectroscopy method for rapid evaluation of wine quality

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Wine is a complex matrix due to its diverse chemical composition and the multitude of factors influencing its quality, flavor and typology. During both production and post-fermentation stages it is important to control wine quality with fast, reliable and non-destructive analytical methods. The OenoFoss2[®] is a near-infrared (NIR) spectroscopy-based instrument designed for the rapid analysis of key oenological parameters, including alcoholic strength, total sugars, pH, total acidity, volatile acidity and density, all in a single analysis.

This study aimed to evaluate the performance and applicability of the OenoFoss2[®] for routine analysis across a broad spectrum of wine types, including white, red, rosé, sparkling and liqueur wines. For that, replicate analyses were performed on certified reference materials representing these wine matrices, and the results obtained for each parameter were compared with the certified reference values. To assess the accuracy of the results, z-scores were calculated, with values between -2,00 and 2,00 considered acceptable (**Table 1**).

Upon acceptance of these results, method validation was carried out through the evaluation of key performance parameters, including precision under repeatability and intermediate precision conditions, trueness, working range and uncertainty. After performing the necessary adjustments, commercial wine samples were analyzed using both internally accredited traditional methods and the OenoFoss2[®].

It was concluded that the OenoFoss2[®] was able to produce data fitting the acceptance criteria, with consistent and reproducible results and analysis times under two minutes per sample. Its ease of use, minimal sample preparation, and environmental benefits highlight its suitability for on-site applications in wineries. The results indicate that the OenoFoss2[®] holds significant promise as a versatile tool for rapid quality assessment across diverse wine profiles, supporting more efficient and data-driven decision-making in winemaking processes.

Table 1. Z-scores obtained for each parameter across the different certified reference wine matrices.

Parameter	Z-score					
	Rosé	Pineau	Red	Sparkling	White Port	White wine
Alcoholic strength	0,20	0,99	-0,70	0,06	0,35	-0,03
Total acidity	0,15	0,16	-0,42	0,22	0,40	1,30
Volatile acidity	0,78	-0,22	-0,72	0,33	-0,56	0,67
Density at 20°C	0,41	-0,14	0,50	0,19	-0,03	0,21
pH	-0,40	0,57	0,80	-0,29	0,74	-1,40
Total sugars	^a	0,09	^a	-0,75	0,37	0,44

^a Result obtained by the laboratory is below the limit of quantification.

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Sequential injection chromatographic system for the expeditious molecular weight screening in hydrolysates

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According to the regulations of the European Union, the use of additives in animal nutrition must be rigorously assessed to ensure safety, efficacy, and quality. These guidelines emphasize the importance of reliable and efficient analytical tools for characterizing bioactive compounds in feed formulation [1].

Currently, the evaluation of proteins is predominantly performed using high-performance liquid chromatography (HPLC). This is a well-established technique known for its robustness, but on the other hand, it is associated with high operational costs, lengthy analysis times, and complex instrumentation. In response to these limitations, this work presents the development of a sequential injection analysis (SIA) system incorporating a laboratory-made chromatographic column, specifically designed to determine compounds across a range of molecular weights, with particular focus on species below 10 kDa (**Figure 1**).

Integrating the column into the SIA circuit enables selective separation and quantification of low-molecular-weight compounds, significantly enhancing analytical throughput and overall process efficiency. The sequential injection chromatography offers distinct advantages, including improved precision, automation, and reduced reagent consumption [2]. This method approach provides a cost-effective, miniaturized, and automated solution with strong potential for real-time control in industrial production environments. This is particularly relevant in the animal feed manufacturing sector, where accurate monitoring of peptides, amino acids, and other bioactive molecules under 10 kDa is essential to ensure nutritional quality, regulatory compliance, and optimized formulation strategies.

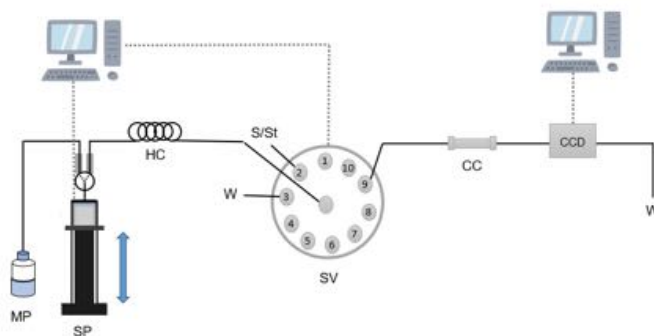


Fig. 1. Sequential injection analysis manifold for the molecular weight screening in hydrolysates; MP, Mobile phase; SP, Syringe pump 5 mL; HC, Holding coil 3 m; S/St, Sample/Standard; SV, Selection valve; CC, Chromatographic column; CCD, UV-Vis detector; W, waste.

Acknowledgments

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Integrated volatilome and chemical profiling for storage stability assessment of plant-based beverages using microextraction and GC-FID/MS

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Plant-based beverages are increasingly relevant as sustainable, functional alternatives to dairy, yet their stability remains a critical parameter influencing quality, safety, and consumer acceptance [1].

In this work, the storage stability of 14 commercial almond-, rice-, coconut-, soy-, and oat-based beverages was assessed combining targeted and untargeted analyses. Eight carbonyls, including malondialdehyde, acrolein and glyoxal, were quantified by a previously developed ultrasound-assisted dispersive liquid–liquid microextraction (US-DLLME) coupled to GC-MS [2]. Volatilome profiling was performed by HS-SPME-GC-MS, while 37 fatty acids were determined as their corresponding methyl esters by GC-FID [3]. A Matrix extension study of the developed methods was performed for accurate quantification. For the stability study, a representative sample of each beverage type was stored under light protection and inert atmosphere (N₂) at three temperatures (room temperature, 4 °C, and 60 °C) for one month. Periodic monitoring revealed significant variations in carbonyl levels, volatilome patterns, and fatty acid profiles depending on beverage type and storage conditions.

These findings provide new insights into the chemical mechanisms underlying quality loss in plant-based beverages and highlight the usefulness of advanced microextraction and GC-based strategies for assessing storage stability.

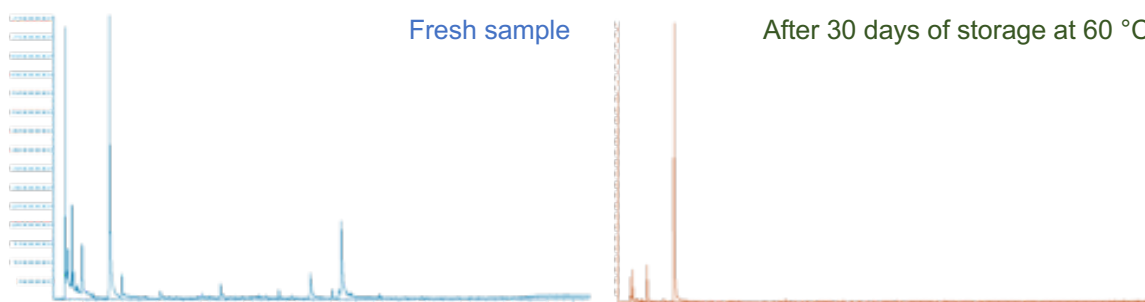


Fig.1. Volatilome profile of coconut-based beverage analyzed by SPME-GC-MS.

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Prospecting natural compounds from ornamental plants to develop innovative biobased antimicrobial formulations

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Sustainability challenges in agriculture and food production are accelerating the search for natural bioactive compounds capable of replacing synthetic agents. Plants rich in organic acids and phenolics are potential candidates to act as biocidal agents, representing an opportunity since many species remain underexplored. Their systematic characterization is fundamental to support their industrial and agronomic valorization.

In this work, hydroethanolic extracts of *Laurus nobilis*, *Hedera "Green Ripple"*, *Photinia* spp., *Gardenia jasminoides*, and *Solanum jasminoides*, were produced by maceration (ethanol:water, 80:20, v:v). Organic acids were quantified by UFLC-DAD, and antioxidant capacity was assessed by DPPH, FRAP, and TPC assays. Triplicate analyses were performed, and differences among extracts were assessed using one-way ANOVA with Tukey's test ($p < 0.05$).

Distinct chemical and functional profiles were observed. *S. jasmonoides* showed the highest malic (19.8 ± 0.7 mg/100 g dw) and oxalic acid contents (8.48 ± 0.02 mg/100 g dw), accompanied by the strongest antioxidant activity (DPPH: 0.4381 ± 0.0093 µg/mL; FRAP: 0.12 ± 0.005 µg/mL). *Gardenia* presented elevated succinic acid (6.59 ± 0.09 mg/100 g dw), while *L. nobilis* was the only species with quantifiable ascorbic acid (0.307 ± 0.006 mg/100 g dw). In terms of total phenolics, *L. nobilis* (16.0 ± 0.1 mg/g) and *Photinia* (12.63 ± 0.06 mg/g) showed relevant levels, whereas *H. "Green Ripple"* displayed the lowest phenolic content (5.03 ± 0.06 mg/g) and weaker antioxidant capacity.

The obtained extracts demonstrated high chemical diversity and significant antioxidant capacity, supporting their exploitation as natural biocides. In line with the SUBSUS project, these results provide insights on the use of these species in developing sustainable biocidal solutions for agricultural pest and disease management.

Acknowledgments

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Study of the Biological Activity of *Fucus vesiculosus*: Aqueous Extractions and Potential Food Application

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Marine macroalgae are increasingly recognized as sources of natural bioactive compounds with potential to replace synthetic preservatives [1,2]. This study aimed to evaluate the chemical composition and biological properties of aqueous extracts from *Fucus vesiculosus*, focusing on their applicability as natural food preservatives. For that, cold and hot water extracts were prepared and analysed for total phenolic and phlorotannin contents using the Folin-Ciocalteu and DMBA assays, respectively, and carbohydrates by the Dubois method, including quantification of key monosaccharides. Antioxidant activity was assessed using ABTS^{•+} scavenging and FRAP assays, while antimicrobial activity was determined through minimum inhibitory (MIC) and bactericidal (MBC) concentrations against methicillin-resistant *Staphylococcus aureus* (MRSA), a major foodborne pathogen of concern for the food industry.

The hot water extract provided the highest yield (43.2%) and higher total carbohydrates content, with notable levels of fucose (445 mg/g), suggesting that fucoidan extraction was enhanced by thermal solubilization. Both extracts exhibited comparable antioxidant activity, with IC₅₀ values around 300 µg/mL, consistent with the phenolic and phlorotannin contents that were also found identical between samples (approximately 2.3 g GAE/100 g and 0.4 g PGE/100 g of extract, respectively). Antimicrobial analysis revealed that both extracts inhibited MRSA growth at 20 mg/mL, with bactericidal activity expected at concentrations above range tested (MBC values likely approaching 80 mg/mL). Overall, these results highlight *F. vesiculosus* as a sustainable source of bioactive compounds with relevant antioxidant and antibacterial activities, supporting its potential application in the food industry as a natural food preservative.

Acknowledgements

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Scaffolds de proteína de soro do leite para encapsulação de vitamina A

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A vitamina A é um micronutriente essencial, cujo défice na dieta está associado a vários riscos de saúde. No entanto, o seu uso é limitado pela sua fraca solubilidade aquosa e elevada sensibilidade à luz, temperatura, oxigénio e outros fatores ambientais [1]. Com o objetivo de melhorar a estabilidade de tais compostos bioativos, utilizam-se métodos de encapsulação. Apesar do elevado número de agentes de encapsulação atualmente conhecidos, são necessários novos materiais para a encapsulação de compostos altamente sensíveis, como é caso de vitaminas e polifenóis [2].

A β -lactoglobulina (β -LG) é a principal proteína do soro do leite, um subproduto da produção de queijo. Fruto das suas propriedades estruturais, a β -LG liga-se naturalmente a um grande número de compostos bioativos de interesse. A β -LG é um biopolímero natural com grande potencial como agente encapsulante [3].

Neste trabalho foram desenvolvidos novos *scaffolds* de proteína de soro de leite para a encapsulação de compostos altamente sensíveis. A vitamina A (retinol), escolhida como composto modelo, foi encapsulada em β -LG, obtendo-se complexos β -LG:retinol, cuja formação foi confirmada por espectroscopia de fluorescência. A estrutura cristalina do complexo β -LG:retinol foi obtida por cristalografia de raios-X, o que permitiu verificar a ligação e a estabilização do ligando. A integridade molecular da vitamina foi verificada por espectrometria de massa. Confirmou-se assim a eficácia do método de encapsulação.

Demonstrou-se que a proteína de soro de leite é um agente encapsulante eficaz na proteção da vitamina A, o que sugere a sua aplicação também na proteção de outros compostos bioativos de alto valor.

Agradecimentos

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Tracing *Bacopa monnieri* in plant food supplements: application of model extract formulations with real-time PCR

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Despite their benefits, the popularity of plant food supplements (PFS) and lack of effective regulation increase the risk of contamination and adulteration. Substitution of high-value plants with lower cost species, whether accidental or intentional, poses significant risks [1]. Therefore, accurate identification of plant content in PFS is crucial for quality control and food safety. *Bacopa monnieri*, known as Brahmi, is an herbaceous plant used in Ayurvedic medicine and plant food supplements (PFS) owing to its nootropic properties. Recently, we reported the first real-time PCR approach with EvaGreen dye, targeting the chloroplast *ycf1* gene to detect *B. monnieri* in herbal products [2]. This study proposes a novel real-time PCR calibration system using simulated plant extracts as calibrants to be applied to analyse PFS. For this purpose, dried and ground *B. monnieri* leaves were extracted with water:ethanol (1:1) and used to prepare reference mixtures (75% - 0.1% w/w in cellulose) and blind mixtures (50%–0.5%) for method validation. DNA of dried reference bacopa extracts and PFS samples containing *B. monnieri* were extracted using NucleoSpin Plant kit, applying five pre-treatments to increase DNA yield of PFS. A real-time PCR calibration model using reference extracts of *B. monnieri* in cellulose was proposed, exhibiting single melt peak (78.42 ± 0.10 °C) that confirmed fragment specificity and acceptable performance parameters of slope (-3.661), PCR efficiency (87.6%) and R^2 (0.996). The limits of detection (LOD) and quantification (LOQ) were 1%. The calibration model was successfully validated with blind mixtures and applied to analyse twelve commercial PFS products. Of those declaring bacopa extract, only three contained quantifiable *B. monnieri* but at levels far below the label claims. The remaining products showed amplification signals below the LOD, likely due to low level of bacopa extracts and/or DNA degradation and excipient interference. These results corroborate the need for pre-treatments to reduce interfering compounds. In conclusion, the developed method enables reliable detection and quantification of *B. monnieri* in complex PFS matrices, addressing a critical gap in quality control of herbal supplements.

Acknowledgements

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Impacto do cultivo intercalar na qualidade nutricional de favas e lentilhas em Marrocos ao longo de dois anos

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As leguminosas e as oleaginosas ocupam uma posição importante na dieta de muitos países, especialmente na região mediterrânica, devido ao seu valor nutricional, à sua capacidade de melhorar a fertilidade do solo e à sua contribuição para a sustentabilidade agrícola [1-3]. Culturas como a fava (*Vicia faba* L.) e a lentilha (*Lens culinaris* L.) são particularmente valorizadas por esses atributos. Sistemas de cultivo intercalar com árvores frutíferas, como amêndoas, têm sido propostos como uma estratégia ecológica para otimizar a eficiência do uso da terra e melhorar a qualidade das culturas [4]. Este estudo teve como objetivo avaliar o impacto do cultivo intercalar com amendoeiras na composição nutricional de favas e lentilhas cultivadas durante dois anos consecutivos de produção em diferentes regiões de Marrocos. As amostras foram coletadas de parcelas intercalares e de controle em Azilal e Ain Taoujdate, em ensaios estabelecidos pela Universidade Sultan Moulay Slimane e pelo Instituto Nacional de Pesquisa Agronômica (INRA). A composição nutricional - incluindo proteínas, gorduras, cinzas, carboidratos, umidade e energia - foi determinada de acordo com os Métodos Oficiais da AOAC. As análises das lentilhas revelaram padrões consistentes ao longo dos dois anos: as parcelas em consórcio apresentaram menor teor de proteínas e maior teor de carboidratos em comparação com os controles, enquanto a umidade aumentou e a gordura bruta diminuiu no segundo ano. Nas favas, foram observadas diferenças regionais claras. Em Azilal, os níveis de proteína aumentaram significativamente no segundo ano (até 24 g/100g pf), acompanhados por uma redução no teor de carboidratos e valores energéticos mais elevados. Em contrapartida, em Ain Taoujdate, a proteína permaneceu estável ou ligeiramente mais baixa, enquanto os níveis de carboidratos foram comparativamente mais elevados. No geral, o estudo mostra que o cultivo intercalar com amendoeiras afeta a qualidade nutricional das leguminosas, com respostas fortemente influenciadas pelas condições locais, destacando a necessidade de um manejo específico para cada região para melhorar a sustentabilidade e o valor nutricional.

Agradecimentos

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BEE4CHEESE project: alternative green extraction technologies of propolis for preservation of artisanal cheeses

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Honey, bee pollen and propolis are some of the products that result directly from beekeeping and whose commercial value is globally recognised. As honey is the most representative product in the Portuguese market, there is a need for a paradigm shift that will reinforce the dynamism and socio-economic value of other hive products by creating innovative products. Portugal has a long tradition of producing artisan cheeses that stand out for their cultural importance and unmistakable flavour, making them a national and international benchmark.

The BEE4CHEESE project meets the need to preserve traditions by using natural products such as bee pollen and propolis to enhance and preserve artisanal cheeses from inland Portugal. We rely on artificial intelligence as a tool to understand the effects of heterogeneity and key characteristics of hive products that enhance the production of genuine cheeses, without losing flavour, texture and identity. One of the project's objectives is to explore alternative green extraction technologies, including ultrasound-assisted extraction as a promising green technology to obtain bioactive-rich natural extracts in a fast, efficient, and environmentally responsible way [1]. In this study, propolis samples were extracted with different solvents, including the more conventional ethanol, glycerol and using green natural deep eutectic solvents (NADES), designed from biodegradable, food-grade components. Quantification of phenolics (TPC) and flavonoids (TFC) revealed that NADES extracts, especially lactic acid/1,2-propanediol and menthol/1,2-propanediol reached values up to ~167 mg GAE/g and ~65 mg QE/g of raw propolis, comparable or superior to extracts obtained with conventional solvents [2].

In this project, we were also able to scale-up the ultrasound-extraction of propolis up to 500 g of raw propolis, using the conventional solvent, achieving yields above 80%. The results highlight ultrasound-assisted extraction with NADES as a sustainable strategy to valorise regional propolis from the Bragança region, generating high-value food and cosmetic ingredients.

Acknowledgments

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Bioactive Compounds from Lettuce Waste and Losses: Valorization Approaches

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Population growth and the increasing demand for balanced diets represent a challenge for agriculture in providing sustainable and nutritious foods. The intensification of agriculture generates significant waste and losses (WL) [1,2]. Lettuce (*Lactuca sativa* L.), a commonly consumed vegetable, suffers notable losses due to management practices and adverse transport and storage conditions [3]. This work aims to determine phenolic compounds and antioxidant activity in WL of different varieties of *L. sativa*, to valorize and repurpose these matrices. Samples from varieties *crispa* (curly leaf, lollo rossa), *capitata* (iceberg), *longifolia* (little gems, romaine), and *crispum* (frisée) were lyophilized, then extracted by decoction and ethanol/water maceration. Phenolic compounds were identified using HPLC-DAD-ESI/MSn; antioxidant activity was assessed through DPPH (Free radical scavenging effect), Reducing Power, TBARS (Inhibition of lipid peroxidation) assays, and anti-bacterial activity was tested using food-borne bacterial strains. In both extracts tested, lollo rossa variety contained the highest levels of phenolic compounds, with hydroethanolic showing the greatest concentrations (29.77 mg/g extract). Quercetin malonyl hexoside was the main phenolic compound (15.3 mg/g extract), while anthocyanins totalled 1.20 mg/g extract, with cyanidin-O-hexoside (0.497 mg/g extract) and cyanidin-3-O-(6"-malonyl)glucoside (0.706 mg/g extract) predominating. In addition, the decoction extract of this variety showed the best results in terms of antioxidant activity in the following assays: TBARS (0.08 mg/mL), DPPH (0.104 mg/mL), and Reducing Power (0.2 mg/mL). Hydroethanolic extracts from curly-leafed, iceberg, little gems, and romaine varieties displayed notable antibacterial activity against *Yersinia enterocolitica* (MIC value 0.007 mg/mL). Overall, the biochemical characterization of *L. sativa* WL highlights their potential as a source of valuable bioactive compounds and supports a sustainable approach to managing agricultural residues.

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Extração assistida por calor: uma via simples e sustentável para valorização de subprodutos de frutas

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O presente estudo teve como objetivo otimizar a extração assistida por calor (HAE), visando maximizar a recuperação de compostos fenólicos com capacidade antioxidante a partir de três subprodutos frutícolas: anona (*Annona cherimola* Mill.), ameixa (*Prunus domestica* L.) e carambola (*Averrhoa carambola* L.), a fim de determinar as condições operacionais mais eficientes para esta técnica. Para tal, um delineamento composto central circunscrito com cinco níveis foi aplicado no planeamento experimental, considerando como variáveis independentes o tempo (t, 5–60 min), a temperatura (T, 30–90 °C) e a percentagem de etanol como solvente (S, 0–100%). Como variáveis de resposta foram avaliadas o rendimento, a atividade antioxidante (DPPH) e o teor de compostos fenólicos totais (HPLC-ESI-QqQ-MS/MS). Os resultados apresentaram diferenças entre as variáveis de resposta e os subprodutos avaliados. A atividade antioxidante constituiu a exceção, apresentando comportamento semelhante entre as amostras, com melhores resultados obtidos em condições de menor tempo (5 min), baixa temperatura (30 °C) e elevada concentração de etanol (100%). Por outro lado, tempo e temperaturas mais elevados favoreceram a máxima extração de compostos fenólicos totais. A variável rendimento apresentou maior diferença entre as matrizes. Para a anona, as condições de 0% etanol, 90 °C e 32,5 min resultaram num rendimento de 68,7±18,6%. Na ameixa, a combinação de 90 °C, 12,8 min e 65,6% de etanol levou ao rendimento máximo de 92,8±8,4%. Já na carambola, o melhor resultado (69,7±10,3%) foi obtido a 30 °C, 79,6% de etanol e 43,5 min. Considerando o objetivo deste estudo, foram definidas condições globais que maximizassem simultaneamente a atividade antioxidante e o teor de compostos fenólicos. Nesse contexto, a carambola destacou-se, alcançando melhor equilíbrio em condições menos severas (5 min, 30 °C e 100% etanol), com rendimento de 41,3±3,2%, atividade antioxidante expressa em EC₅₀: 1,6±0,6 mg de matéria seca/mL e 21,7±2,3 mg de compostos fenólicos totais/g de extrato. Estes resultados confirmam que a resposta ao processo de extração é influenciada pela matriz, mesmo quando se aplica uma técnica convencional. Além disso, demonstram que a HAE constitui um método de referência fundamental para a validação de técnicas emergentes, além de representar uma abordagem prática e sustentável para a valorização de subprodutos frutícolas. A obtenção de extratos com capacidade antioxidante abre perspectivas promissoras para aplicação em produtos funcionais ou aditivos naturais nas indústrias alimentar e farmacêutica, contribuindo para a redução do desperdício e alinhando-se com os princípios de inovação e sustentabilidade da Agenda 2030.

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A Impressão Digital Bioquímica do Carapau: Autenticação de Origem via Perfil de Aminoácidos e *Machine Learning*

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A fraude no pescado, incluindo falsificação de origem, ameaça a sustentabilidade e a confiança dos consumidores [1]. Embora métodos espectroscópicos/elementares estejam consolidados [2–4], o perfil de aminoácidos (AAs) permanece subexplorado, oferecendo dupla utilidade (autenticação geográfica e avaliação nutricional). Neste estudo, um inquérito a 102 consumidores revelou alta perceção de fraude (68%) e baixa confiança na sua deteção (56%), reforçando a necessidade de ferramentas científicas robustas.

Foram analisados 150 espécimes de *Trachurus trachurus* (n=30/local) de cinco portos (Sesimbra, Peniche, Matosinhos, Olhão, Sines). O músculo dorsal foi submetido à extração de gordura com n-hexano e à hidrólise com ácido metanossulfónico 4 M suplementado com triptamina, sob purga de N₂, preservando AAs lábeis como o triptofano. Para a análise de AAs, os hidrolisados foram derivatizados com OPA/FMOC e analisados por HPLC com deteção por fluorescência. O teor de gordura (TG) foi determinado gravimetricamente e a proteína bruta pelo método Kjeldahl. Para autenticação da origem, 17 AAs foram avaliados por Análise de Componentes Principais (PCA) e modelados por Análise Discriminante Linear (LDA) e *Random Forest* (RF), com validação cruzada e conjunto independente.

Observou-se um gradiente regional significativo no TG (13,5% em Peniche–18,2% em Sines, base seca; ANOVA: $F(4,145)=24,26$, $p<0,001$; ANCOVA ajustada a comprimento/peso: $F(4,143)=11,06$, $p<0,001$). No entanto, por refletir sobretudo triacilgliceróis e variações fisiológicas/sazonais, o TG não se mostrou um marcador de origem adequado, embora indique valor nutricional. A proteína bruta variou entre 76,2–80,2%. A PCA dos AAs revelou assinaturas distintivas por local (83,1% da variância). A LDA superou a RF em precisão ($82,7\pm4,4\%$ vs. $76,0\pm7,8\%$), e o modelo final alcançou 66,7% de precisão na validação independente, com erros concentrados entre locais geograficamente próximos. O coeficiente absoluto da LDA refletiu a importância relativa de cada AA na separação geográfica, sendo os mais informativos: ácido glutâmico (Glu, 7,65), ácido aspártico (Asp, 6,08), tirosina (Tyr, 6,00), triptofano (Trp, 5,49) e isoleucina (Ile, 4,65).

Estes resultados confirmam que o perfil de AAs constitui uma assinatura bioquímica robusta e biologicamente significativa para autenticação geográfica, oferecendo uma ferramenta promissora para combater a fraude e valorizar produtos da pesca sustentável. A eficácia da LDA reforça ainda o valor desta abordagem, alinhando-a com os princípios de transparência e Inteligência Artificial responsável exigidos para aplicações no setor alimentar [5].

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Deep Eutectic Solvents-DES coupled with microwave heating as a green strategy for phenolic extraction from *Fucus vesiculosus*

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The exploitation of third-generation aquatic biomass, particularly algae, has emerged as a promising alternative to fossil-based raw materials. Among them, *Fucus vesiculosus* (Fv) stands out for its potential in the food and nutraceutical sectors, since it contains relevant bioactive molecules such as phenolics, proteins, carotenoids, and polysaccharides [1].

Conventional extraction is the most common approach for isolating phenolic compounds and antioxidants. Nevertheless, this strategy relies on organic solvents, which are associated with several drawbacks, including high volatility, limited efficiency, toxicity, elevated costs, and environmental concerns. To overcome these limitations, deep eutectic solvents (DES) have been introduced as greener and more sustainable alternatives. These solvents combine advantageous characteristics such as low vapor pressure, reduced toxicity, biodegradability, low melting point, and economic feasibility [2]. Moreover, their versatile physicochemical nature enables them to solubilize diverse compounds, which explains their increasing use in extracting bioactives from different biomass sources. At the same time, the development of more efficient and cost-effective processing methods is needed. Microwave (MW) irradiation has attracted growing interest as an intensification technology, since it can reduce energy demand and provide rapid, selective, and homogeneous heating of both biomass and solvent [3].

In this study, the extraction of phenolic compounds from Fv was optimized, focusing on total phenolic content (TPC), total flavonoid content (TFC), and antioxidant activity (DPPH, ABTS, FRAP, TAC). The influence of three variables—water proportion in DES (10–50%), extraction temperature (100–150 °C), and extraction time (0–40 min)—was investigated using response surface methodology (RSM), which confirmed the reliability of the model. Major phenolic compounds were subsequently identified and quantified by HPLC-ESI. These results highlight how combining novel solvents such as DES with a more energy-efficient heating source like microwaves provides an attractive alternative for valorizing the bioactive fraction of this brown macroalga.

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Simultaneous Determination of Pesticides and PAHs in Stingless Bee Honey from Northeastern Brazil Using GC-MS/MS

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In Northeastern Brazil, the Caatinga biome, a tropical dry forest, predominates almost exclusively. This unique ecosystem is characterized by two sharply contrasting seasons: a short rainy period and a prolonged drought [1]. During the dry season, both intentional and accidental fires are common and the combustion of organic matter under these conditions can lead to the formation of Polycyclic Aromatic Hydrocarbons (PAHs) [2]. Once released into the atmosphere, PAHs can disperse over long distances and bioaccumulate in soil, vegetation, and water bodies [3]. In parallel, the widespread use of agricultural pesticides in surrounding areas poses an additional threat to environmental and food safety. These chemical residues can be transported through air, water, and plant surfaces, eventually contaminating bee foraging resources. Both PAHs and pesticides are of particular concern due to toxicological effects, including mutagenic, carcinogenic and endocrine-disrupting properties [4,5]. This study aimed investigates the occurrence of these contaminants in honey produced by *Melipona subnitida* a stingless bee species native to the Caatinga, in order to assess potential risks to pollinator health and honey quality.

A total of 48 honey samples from *Melipona subnitida* colonies were collected during harvest seasons from 2023 to 2025 across the Caatinga biome in Northeastern Brazil. To determine the presence of environmental contaminants, 20 PAHs and 13 pesticide residues were extracted using a modified QuEChERS protocol combined with dispersive solid-phase extraction (d-SPE). The cleanup step employed primary-secondary amine (PSA) sorbent to minimize matrix interferences. For analyte enrichment, a low-density solvent-based dispersive liquid-liquid microextraction (LDS-DLLME) was performed using acetonitrile as the disperser solvent and toluene as the extractant. The final extracts were subsequently analyzed by gas chromatography coupled with tandem mass spectrometry (GC-MS/MS) under optimized conditions, enabling sensitive and simultaneous detection of both PAHs and pesticide residues in complex honey matrices.

The analytical data obtained in this study contribute to ongoing research on environmental contamination and its ecological and toxicological implications. The results are intended to support conservation strategies for native stingless bee species in the Caatinga biome and provide a scientific foundation for the development of public health policies.

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Sustainable processing of *Himanthalia elongata* seaweed through deep eutectic solvents by microwave-assisted technology

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The continuous growth of the global population and the rising demand for sustainable resources are driving the search for alternative sources of nutrients and bioactive compounds. In this context, marine macroalgae have emerged as promising candidates due to their rapid growth rates, low cultivation requirements, and richness in bioactive metabolites with applications in the food, nutraceutical, cosmetic, and pharmaceutical sectors. Among them, the edible brown seaweed *Himanthalia elongata* (He) stands out as a valuable biomass for the development of functional ingredients.

In this work, dried He was chemically characterized using standardized NREL protocols. Target compounds were extracted with deep eutectic solvents (DES), which represent a safe, efficient, and environmentally friendly alternative to conventional organic solvents. The extraction strategy combined different DES formulations (HBA: choline chloride or betaine; HBD: acetic acid, urea, lactic acid, or glycerol) with microwave-assisted extraction (MAE), enabling the simultaneous recovery of bioactive polysaccharides and phenolic compounds. Conventional solvents such as 70% ethanol, 0.1 M NaOH, and water were used for comparison.

The results demonstrate that water and NaOH yielded the highest recoveries of oligosaccharides, whereas nearly all tested DES (except for Choline chloride:glycerol, 1:2) enhanced the extraction of phenolic compounds compared to conventional solvents. The most effective DES for phenolic compounds extraction were choline chloride:acetic acid (1:2) and choline chloride:lactic acid (1:3), both with 30% of water. The antioxidant activity tests (DPPH, ABTS and FRAP) further demonstrated that extracts obtained with Choline chloride:urea (1:2) and Betaine:urea (1:2) exhibited superior antioxidant capacity.

By maximizing extraction yields, preserving the structural integrity of biomolecules, and enhancing the bioactivity of the recovered fractions, this approach provides a sustainable pathway for the comprehensive valorization of He. Overall, the proposed methodology contributes to the advancement of green extraction processes and supports the exploitation of edible seaweeds as renewable sources of functional ingredients for food and health-related applications.

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Selección de disolventes con COSMO-RS para la extracción de crisina de flor de retama: análisis HPLC-MS y capacidad antioxidante

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La búsqueda de métodos sostenibles y eficientes para la extracción de compuestos bioactivos naturales está cobrando cada vez mayor relevancia en el marco de la química verde y las ciencias de los alimentos y farmacéuticas. En este estudio se aplicó el modelo COSMO-RS (Conductor-like Screening Model for Real Solvents) para predecir los disolventes más adecuados para la extracción de crisina a partir de flores de retama (*Cytisus striatus*), validando dichas predicciones mediante análisis cromatográfico (HPLC-ESI-MS) y ensayos de capacidad antioxidante (TPC, DPPH, FRAP).

El cribado computacional identificó a los ésteres y alcoholes como las familias de disolventes más idóneas para la solubilización de crisina, mientras que los hidrocarburos y disolventes apróticos polares resultaron problemáticos. Los resultados experimentales confirmaron estas predicciones: el metanol y el etanol proporcionaron los mayores rendimientos de extracción de crisina (27 422 µg/L y 24 920 µg/L, respectivamente), seguidos de la acetona (18 452 µg/L) y el acetato de etilo (13 348 µg/L). En contraste, el agua (585 µg/L) y el hexano (158 µg/L) presentaron una eficiencia marcadamente inferior.

Además de la crisina, se cuantificaron otros flavonoides y ácidos fenólicos. Los extractos de metanol y etanol destacaron por su alto contenido en luteolina (3 072 y 3 971 µg/L), rutina (2 756,00 y 2 792,50 µg/L) y kaempferol (1 004 y 1 439 µg/L). Por su parte, el acetato de etilo y la acetona mostraron perfiles intermedios. Aunque el agua fue poco eficiente en la extracción de crisina, permitió recuperar cantidades elevadas de ácido p-cumárico (7 408 µg/L) y ácido ferúlico (1 962 µg/L), lo que evidencia la selectividad de los disolventes hacia distintas clases de compuestos fenólicos. El hexano, en cambio, presentó una capacidad extractiva prácticamente nula para la mayoría de compuestos.

Las mediciones de actividad antioxidante se correlacionaron, en cierta medida, con el contenido fenólico, excepto para el agua que puede contener otros compuestos (por ejemplo, proteínas y oligosacáridos) que tienen mayor afinidad con solventes como el agua. Los extractos obtenidos con etanol, metanol y acetona mostraron las mayores actividades de captación de radicales, en consonancia con su mayor riqueza en flavonoides. La integración de las predicciones de COSMO-RS con los análisis experimentales confirma que los disolventes con propiedades favorables de polaridad y capacidad de enlace de hidrógeno no solo mejoran la recuperación de crisina, sino también el potencial antioxidante global de los extractos.

En conjunto, este trabajo demuestra la utilidad de COSMO-RS como herramienta para guiar la selección de disolventes en la extracción de productos naturales, reduciendo el ensayo y error experimental y promoviendo procesos más verdes y eficientes. La validación experimental refuerza la viabilidad de esta estrategia para optimizar la valorización de recursos vegetales infrutilizados como la retama.

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Bioactive compounds from marine organisms – a bridge between Chemistry and Biotechnology

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The valorization of natural products of marine origin has gained increasing prominence in the scientific community due to applications in diverse socio-economic sectors (e.g.: food industry, health, cosmetics), the blue bioeconomy holding enormous potential for exploration. Within the BEAP-MAR project, whose ultimate objective is to value marine organisms not traditionally exploited from a commercial viewpoint, namely sponges, fungi and algae, we focus our attention on microalgae. These organisms are a rich source of macromolecules (lipids, carbohydrates, proteins) as well as of low molecular weight molecules bearing relevant structural features and associated functional groups (e.g. phenolic groups), such as pigments and carotenoids, that provide potential for various applications [1].

Although several studies report the bioactive potential of microalgae, namely diatoms [2,3], few studies effectively identify and characterize the bioactive molecules and even fewer provide insights on the molecular mechanisms responsible for their effects, so there remains a gap of knowledge demanding for further research to be developed in this field. Taking advantage of our location, we focus on microalgae found in the southernmost area of the Atlantic region, due to the differentiating characteristics they present, influenced by their proximity to the Mediterranean area. Among them, we focus our study on underexplored diatoms of the genus *Nitzschia*. Preliminary studies unraveled anti-bacterial [4,5], antioxidant [5] and some anti-cancer [4] properties in some species of this genus.

In this study, several parameters were optimized to enhance cell lysis and metabolite extraction from *Nitzschia thermalis*. After obtaining sample fractions in different solvents, each fraction was analyzed by UV-Vis spectroscopy. Also, the metabolic profile was characterized using chromatographic techniques coupled with mass spectrometry, namely LC-MS and GC-MS, evidencing the extraction of distinct compounds in each fraction. The results revealed a profile rich in potentially bioactive compounds, and bioactivity studies are currently underway.

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Hidrólisis acelerada por ultrasonidos para la caracterización y cuantificación de aminoácidos en proteínas vegetales por HPLC-UV

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La caracterización nutricional de proteínas en matrices vegetales requiere métodos de hidrólisis eficaces que permitan una liberación completa de aminoácidos, minimizando al mismo tiempo la degradación de compuestos sensibles [1,2]. En este trabajo se presenta un método innovador de hidrólisis acelerada por ultrasonidos aplicado a proteínas de bebidas vegetales, comparado con técnicas convencionales. El uso de ultrasonidos facilita la ruptura de enlaces peptídicos bajo condiciones controladas, reduciendo significativamente los tiempos de reacción y el consumo de reactivos agresivos [3].

Tras la hidrólisis, los aminoácidos totales liberados se cuantificaron mediante cromatografía líquida de alta resolución, obteniéndose perfiles completos de composición que permiten evaluar tanto la calidad proteica como el valor nutricional de las muestras. Los resultados muestran que la metodología ultrasónica asegura una recuperación reproducible y eficiente de aminoácidos, con ventajas claras frente a la hidrólisis ácida tradicional en términos de rapidez, sostenibilidad y preservación de analitos.

Este trabajo ofrece una herramienta versátil y escalable para el análisis de proteínas en nuevas formulaciones de alimentos basados en plantas y alimentos funcionales, contribuyendo a un mejor conocimiento de su potencial nutricional y a la optimización de procesos en el desarrollo de alimentos sostenibles.

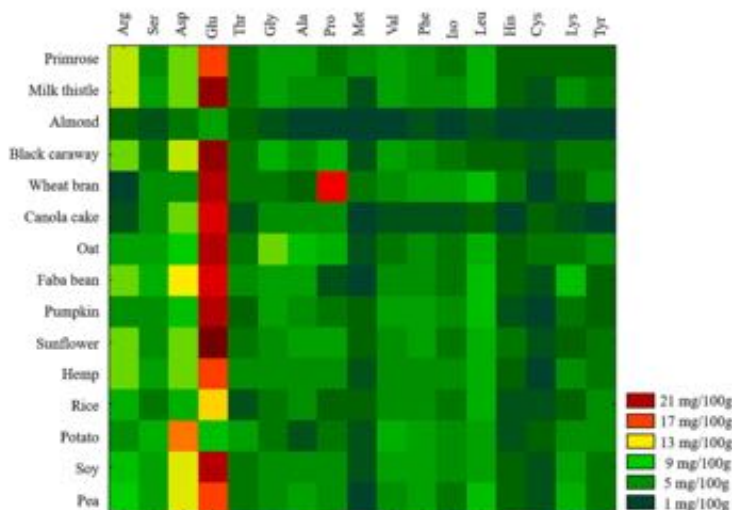


Fig.1. Mapa de calor de aminoácidos totales en aislados proteicos vegetales.

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Exploring the Effect of *Cinnamomum burmannii* on Blood Coagulation

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Cinnamomum burmannii is a widely used medicinal plant with various therapeutic properties. Recent research has focused on identifying natural alternatives for vascular diseases, particularly anticoagulants, to mitigate the risks associated with synthetic drugs, such as increased bleeding risk, drug interactions, and resistance development over time. This study evaluates the coagulation potential of *C. burmannii* extracts in vitro, assessing their effect on blood clotting in both animal and human models to determine if this plant has this capacity for both. We specifically analyse whether the entire aerial part of the plant (leaves, stems, and cork) contributes to coagulation activity.

Preliminary phytochemical screening revealed the presence of phenols, tannins, terpenoids, and proteins, suggesting a potential role in coagulation modulation. Further phytochemical analysis quantified a total phenolic content of 467.5 ± 6.4 mg GAE/g and a flavonoid content of 8.32 ± 0.18 mg QE/g, specifically in the cork sample. Additionally, essential oil extraction yielded 4.67% from the cork and 0.5% from the leaves and the stems.

These bioactive compounds and essential oils are known for their potential role in modulating coagulation processes. Further analysis will determine whether these components are responsible for the coagulation effect observed.

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Rainwater as a Proxy for Atmospheric Metal Pollution in Estarreja, Portugal

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Estarreja, home to the largest chemical complex on the northern coast of Portugal, has long been recognized as an area of environmental concern due to intensive industrial and agricultural activity. Among the pollutants released, potentially toxic metals are particularly critical because of their persistence, bioaccumulative potential, and toxicity to both humans and ecosystems. Previous studies have reported contamination of soils, sediments, and groundwater in the region [1, 2]. Rainwater plays a central role in the atmospheric deposition of these elements, acting as a pathway for their transfer from emissions to terrestrial and aquatic systems. Therefore, monitoring the metal composition of rainwater provides valuable insight into the air quality of Estarreja and the broader environmental impacts of its industrial activities.

This study assessed the influence of the Estarreja industrial complex on the metal composition of rainwater between March 2022 and September 2022. Six precipitation events were sampled simultaneously at four sites, three within the industrial area and one background site, yielding 24 rainwater samples. Metal concentrations in the samples were quantified, and spatial-temporal variations were analysed considering also the influence of the air mass trajectories. The findings highlight rainwater as a key mechanism for the removal of atmospheric metals in Estarreja, underscoring its role as both an environmental pathway and a monitoring tool.

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Assessment of Renewable Energy Performance Across Spain

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Energy access has become increasingly recognized not just as a technical challenge, but as a cornerstone of long-term social, economic, and environmental stability. Within the framework of the European Union's evolving energy strategy, Spain has emerged as a significant actor in the renewable transition. However, as ambitions grow, so do the complexities associated with ensuring that energy systems are not only cleaner, but more resilient, equitable, and coherent [1, 2].

This research develops a national-level assessment of the sustainability of Spain's renewable energy system, with a focus on the interplay between economic viability, environmental responsibility, and social inclusiveness. A structured evaluation approach was used to integrate expert insights and prioritize sustainability factors across these dimensions. Rather than treating energy infrastructure components in isolation, the analysis considers how they function together—revealing systemic gaps that may weaken overall performance or limit long-term effectiveness.

By grounding the assessment in both expert-based weighting and cross-sectoral indicators, the study offers a more contextualized understanding of where Spain's renewable energy system aligns with European policy targets—and where strategic recalibration may be needed [3]. Although specific evaluation outcomes are withheld in this summary, the framework provides a valuable tool for comparative analysis and policy foresight.

The intention is not to prescribe a singular path forward, but to support more integrated decision-making—helping policymakers, energy planners, and investors see beyond short-term metrics toward a more resilient and inclusive energy future. In doing so, the study contributes to the broader European dialogue on how national systems can balance ambition with grounded, systems-oriented planning.

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Application of an electrochemical sensor based on molecularly imprinted polymers for the monitoring of water treatments

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The development of electrochemical sensors has become increasingly important for the detection of emerging contaminants in water. These sensors offer multiple advantages over traditional analytical techniques, such as high-performance liquid chromatography (HPLC) or spectrophotometry, given that they are more cost-effective, portable, fast and require less reagent consumption, making them ideal for in-situ applications [1]. Among electrochemical sensors, those based on molecularly imprinted polymers (MIPs) stand out. These devices use polymers specifically designed to recognize target molecules, thanks to cavities formed during the molecular imprinting process that replicate the shape and chemical properties of the analyte. This technology allows for high selectivity and sensitivity in the detection of specific contaminants [2]. However, MIP sensors developed to date have generally been limited to the detection of pollutants in water sampling, without exploring their potential in monitoring water treatments.

In this work, a MIP electrochemical sensor was developed for the detection of carbamazepine (CBZ), a widely used pharmaceutical that was included in the Directive (EU) 2024/3019 on Urban Wastewater Treatment. The sensor was fabricated on disposable screen-printed carbon electrodes, using 3,4-ethylenedioxythiophene as the functional monomer and CBZ as the template in the electropolymerization process to obtain the MIP layer. The obtained sensor yielded good analytical performance in the concentration range from 1 to 50 μM , showing great accuracy for the detection of carbamazepine in different water matrices. Once the suitability of the sensor had been assessed, it was used to monitor an adsorption treatment.

The adsorption capacity of different biochar obtained from agroforestry residues (orange tree waste and basil residues) was evaluated for the removal of CBZ from water. Results showed that the biochar derived from orange tree residues achieved more than 60% removal within only 2 h, which is remarkable considering the inherent difficulty of CBZ adsorption and the added value of using a sustainable adsorbent material under a circular economy approach. The adsorption treatment performed with that biochar was monitored using the developed MIP sensor, HPLC and spectrophotometry, obtaining results that differ in less than 5%. Thus, the implementation of the MIP electrochemical sensor proved to be a fast and reliable alternative for the detection of CBZ in aqueous samples, highlighting its potential for real-world environmental monitoring.

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Elimination of carbamazepine from water using biosorbents derived from agri-food waste

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The presence of emerging contaminants in wastewater and freshwater bodies represents a significant environmental challenge. Despite advances in wastewater treatment technologies, many of these facilities are not designed to efficiently remove these compounds, resulting in their continuous release into the environment. Consequently, the Directive (EU) 2024/3019 on Urban Wastewater Treatment [1] enforces the implementation of a quaternary treatment to ensure the removal of 12 target micropollutants, among which is carbamazepine, a widely prescribed anticonvulsant drug [2].

In this context, this research focused on the use of biosorbents derived from agri-food waste as a sustainable solution for drug adsorption. In particular, the following precursors were tested: nutshell, pistachio shell, hazelnut shell, almond shell and banana peel. The biosorbents were obtained through pyrolysis (tubular reactor, 400 °C, 2 h, under a N₂ atmosphere) and hydrothermal carbonization (hydrothermal reactor, 220 °C, 20 h, with the addition of deionized water), generating biochar and hydrochar, respectively. All the obtained biosorbents were characterized in terms of their physicochemical properties. The performance of the materials (without modification and after being modified with 1 M NaOH or H₃PO₄) was assessed in terms of their carbamazepine uptake capacity. The ones reporting higher removals (see Fig. 1) were selected and tested in adsorption, electrochemical and a combination of both treatments. Preliminary results indicate that, for the nutshell hydrochar modified with NaOH, adsorption was the most effective treatment, with no significant improvement observed upon application of an electric field. Conversely, for the almond shell hydrochar modified with NaOH, an increase in the removal from 69% to 77% was attained when adsorption and electrochemical treatments were applied together. Considering the results obtained, the synthesized biosorbents have demonstrated to be a cost-effective and viable solution for the treatment of polluted water.

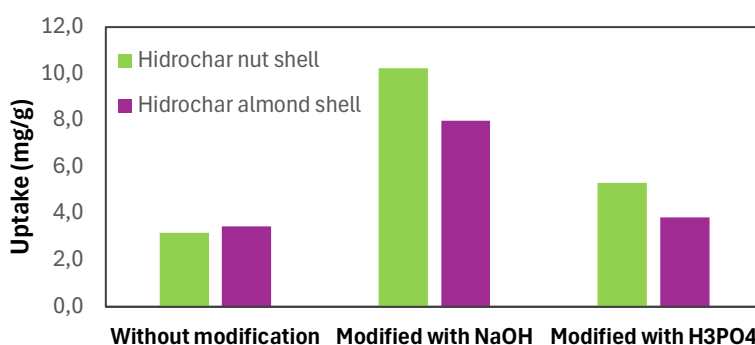


Fig.1. Adsorption capacity of the two biosorbents that showed higher removals of carbamazepine.

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Critical Analysis of sustainability Indicators and Policy Integration: In the framework of energy and environment

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Achieving sustainable development requires coherent environmental and energy policies at both international and national levels. Global frameworks such as the UN Sustainable Development Goals (SDGs) embody this vision, but countries differ widely in their ability to deliver on these commitments [1][2]. Assessing the real impact of these policies requires a selected set of sustainability indicators that reflect both environmental and energy outcomes and the quality of implementation [3].

This study aims to analyse existing sustainability indicators to assess the effectiveness of policies implemented to promote sustainable development. The research is focused on chemical indicators related to environmental and energy sectors. This approach seeks to offer a critical analysis of these indicators and their link with current environmental policies

Many of these indicators have already been established to evaluate sustainability, such as the study of gas emissions (CO₂, SO₂, NO_x), air quality, and the participation of renewable energies [4]. However, indicators including the water footprint of energy or the energy circularity index have not been given as much importance and could play a significant role in advancing sustainable development [5]. Indicators should offer accurate and meaningful insights into the sustainability of the evaluated system, maintaining a well-defined connection to the factual data that supports them [6]. Always taking into account that sustainability must be assessed from a point of view that integrates social, economic and environmental factors [1].

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Renewable Energy and Environmental Impacts in the European Union: A Panel Data Analysis

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The European Union has intensified its efforts to reduce environmental degradation by accelerating the adoption of renewable energy, particularly through updated climate and energy strategies in recent years. Over the past decade, the share of renewables in electricity generation has grown significantly, driven by rapid expansion in solar, wind, and hydroelectric capacity [1]. However, despite this progress, the extent to which these developments are effectively reducing CO₂ emissions and delivering measurable environmental improvements remains an open question [2].

This study applies panel regression techniques to examine how changes in renewable energy deployment influence environmental quality across European Union member states. By observing variations in quantitative sustainability indicators over a ten-year period, the analysis identifies statistical associations between shifts in energy profiles and environmental performance. The model is designed to isolate within-country effects over time, enabling evaluation of how incremental increases in renewable energy adoption correspond with reductions—or stagnation—in environmental impact. This framework captures both the strength and direction of these relationships, offering a quantitative lens on the dynamics of national energy transitions [3].

While numerous studies have explored the relationship between renewable energy and environmental outcomes, there remains limited consensus on how national differences within the European Union shape these effects. The diversity of energy systems, governance structures, and economic conditions complicates the task of drawing generalized conclusions. By adopting a panel regression approach across multiple years, this study contributes to a more nuanced understanding of how renewable energy transitions unfold across diverse national contexts. Building on recent research that highlights variability in environmental performance among EU states, the study seeks to clarify the role of structural and policy-related factors in moderating environmental impact [2][4].

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Industrial by-products for biogas production

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Industrialization has relied on fossil fuels, driving development but also causing severe environmental damage [1]. In response, interest in renewable energies like biogas is growing. The European Union and international organizations are promoting its use to move toward climate neutrality and universal access to clean energy. Biogas is a renewable energy source that can be produced from anaerobic digestion of waste, landfills, wastewater sludge, animal manure, and energy crops [2]. Although it requires technical processes and faces challenges such as resource variability, it is a sustainable alternative compatible with existing infrastructure. Biogas uses organic waste to produce clean energy, and two billion tonnes of municipal solid waste are produced worldwide each year, 34-53% of which is biodegradable [3].

The objective of this study is to review the main sources of waste used for biogas production and to analyse research on organic substrates currently used in its generation. This study presents a classification and analysis of waste suitable for biogas production, employing an innovative approach that integrates an industry-specific perspective. Different types of by-products and wastes that can be used as substrates for biogas production are classified either individually or in co-digestion with other residues, taking into account the International Standard Industrial Classification (ISIC). By aligning waste types with industrial sectors, the methodology enables a more comprehensive and practical assessment of biogas applications across diverse industries.

The studies analysed reveal a clear trend in the production of biogas primarily through the use of agricultural residues, food waste and animal manure. A significant part of the reviewed works focuses on evaluating physical, chemical or biological pretreatment techniques to improve substrate degradability, reflecting an increasing interest in maximizing process efficiency. Codigestion also emerges as an active and diverse area of research. In addition, a considerable number of studies focus on microbial dynamics and the use of additives to accelerate anaerobic digestion. Although most investigations are still carried out at laboratory scale, some involve high-rate reactors and pilot plants, reflecting a gradual progression toward real-world application.

This approach offers a sustainable solution for waste management, emphasizing the importance of identifying and utilizing diverse waste streams as valuable substrates for biogas production. In doing so, it supports the advancement of circular economy practices and sustainable waste management strategies. Future research should integrate new waste categories and their influence on process efficiency, as well as assess sustainable upgrading technologies for the conversion of biogas to biomethane.

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Study of antioxidant activity in aqueous leaf extracts by a simple spectroscopic method based on silver nanoparticles bio-synthesis

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Some phenolic compounds found in various plants, fruits, vegetables, and legumes can act as natural antioxidants. These compounds are synthesized in response to ecological and physiological stress factors, such as pathogen attacks or exposure to ultraviolet (UV) radiation, and play a crucial role in plant defense mechanisms [1]. In particular, phenolic compounds have been identified in species of the genus *Eucalyptus*. The analysis of these compounds in aqueous extracts of *Eucalyptus* leaves is of special interest, both for understanding their potential phytotoxic effects and for evaluating their applicability as natural herbicides.

Certain phenolic acids and ortho-trihydroxy phenols are known to reduce silver precursors, leading to the formation of silver nanoparticles (AgNPs) [2]. Consequently, the antioxidant activity associated with these phenolic compounds can be assessed by monitoring the surface plasmon resonance (SPR) band characteristic of AgNPs.

In this study, we present a simple UV–visible spectroscopic method for evaluating the antioxidant activity in aqueous leaf extracts of *Eucalyptus globulus* Labill. Four phenolic compounds with varying numbers of hydroxyl groups, gallic acid, chlorogenic acid, *p*-coumaric acid, and ellagic acid, were tested using a synthesis protocol previously optimized for pyrogallol. Under basic aqueous conditions, all compounds except *p*-coumaric acid produced a SPR band within 1 minute. In contrast, *p*-coumaric acid required 24 hours to induce the formation of AgNPs.

Depending on the specific phenolic compound used, the SPR maximum was observed within the 400–500 nm range, likely reflecting differences in the size of the AgNPs formed. The synthesis was also successfully carried out in ethanolic medium. When applied to *Eucalyptus* leaf extracts, the method revealed behavior distinct from that of the pure standards. Therefore, the standard addition method was employed for quantification.

Figures of merit were determined for chlorogenic acid, gallic acid, and ellagic acid. The limits of detection (LOD) ranged from 11 μ M to 23 μ M, while the limits of quantification (LOQ) ranged from 36 μ M to 77 μ M. The antioxidant capacity of the leaf extracts can thus be expressed in terms of the equivalent concentration of added polyphenol. The presence of phenolic compounds in the extracts was further confirmed by high-performance liquid chromatography (HPLC).

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Environmental impact of energy: Strategies for Climate Change Mitigation

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Climate change has become one of the most environmental issues worldwide, mainly caused by the continuous use of non-renewable energy sources such as coal, oil, and natural gas. These energy systems emit large amounts of greenhouse gases, increasing global warming and environmental degradation [1]. In contrast, renewable energy sources like solar, wind, and hydropower present a sustainable alternative capable of reducing emissions and limiting the harmful effects of climate change [2].

This study aims to evaluate the environmental impacts of renewable and non-renewable energy systems in relation to climate change. The research involved a comparative analysis based on data from international climate and energy reports, focusing on carbon emissions, air quality, and ecosystem effects [3]. The development also considered the technological, economic, and social aspects that influence the global transition from fossil fuels to renewable energy sources [4].

The results demonstrate that renewable energy significantly reduces greenhouse gas emissions and contributes to cleaner and more sustainable ecosystems. However, these technologies are not entirely harmless and can also generate environmental impacts, such as land use changes, material extraction, and waste management issues, which must be properly addressed. In addition, some challenges remain, including energy storage capacity and electrical stability, infrastructure needs and initial investment costs [3,4]. In conclusion, the findings emphasize the necessity of accelerating the global shift toward renewable energy as a key strategy to mitigate climate change, protect the environment, and promote sustainable development.

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Environmental Screening for Organochlorine Pesticides on the Coastal Beaches of Northern Mozambique

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Organochlorine pesticides (OCPs) are persistent organic pollutants of global concern due to their environmental persistence, bioaccumulation, and adverse health effects. Mozambique faces particular challenges, as concentrations of micropollutants in surface waters often exceed those reported in European rivers. In this context, the present study aimed to evaluate OCP contamination in coastal sediments from Pemba Bay and Nacala-Porto, two key urbanized areas of northern Mozambique.

Sediment samples were collected from eight beaches and analyzed using the QuEChERS extraction method, followed by gas chromatography with electron capture detection (GC-ECD). This approach, widely recognized as rapid, cost-effective, and reliable, enabled the detection and quantification of OCP residues.

The results confirmed the presence of OCPs across all sites. HCH- α was the predominant compound, with concentrations ranging from 0.014 to 0.054 $\mu\text{g/g}$, followed by HCH- β (0.027 $\mu\text{g/g}$) and HCH- γ (0.062 $\mu\text{g/g}$). These findings provide the first baseline assessment of OCP contamination in the coastal sediments of northern Mozambique.

This study highlights the need for continued monitoring and environmental management strategies to mitigate the potential ecological and human health risks associated with pesticide contamination in coastal ecosystems.

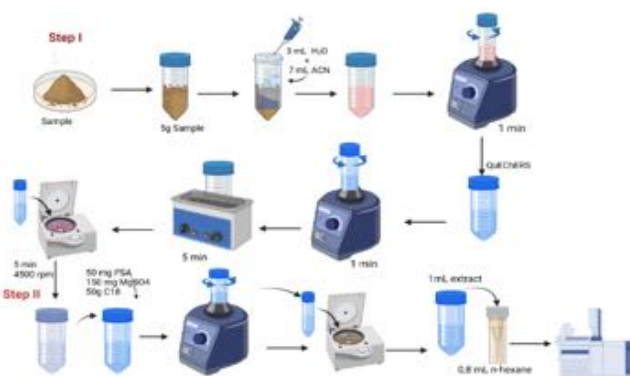


Fig.1: Experimental set-up for the extraction method (SPE).

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NOMME: Ferramenta para o reconto, clasificación e estimación de masa de microplásticos identificados mediante Espectroscopia Infravermella de Transflectancia

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A espectroscopia infravermella de transflectancia baseada no uso dun láser sintonizable de cascada cuántica (QCL-IR) consolidouse como unha técnica de referencia para a identificación de microplásticos (MPs). Actualmente, o equipo LDIR 8700 de Agilent ven de se converter nun referente para implementar estudos de monitorización. Nembargantes, a información que aporta por defecto límitase ao reconto de partículas, identificación dos polímeros presentes, e unha pequena listaxe de parámetros xeométricos das partículas; nomeadamente, diámetro, ancho, longo, solidez, circularidade, e relación de aspecto.

Para facilitar o traballo dos investigadores desenvolveuse NOMME (Number Of Microplastics and Mass Estimation), unha aplicación en Excel que procesa automaticamente os ficheiros exportados polo equipo LDIR. NOMME clasifica, nun primeiro paso, os MPs identificados en partículas e fibras; organízalos en rangos de tamaño predefinidos e segundo tipo de polímero; mostra o número de MPs en cada categoría (considerando polímero e rango de tamaño), e fai unha estimación da súa masa. Esta última baséase nun modelo híbrido que combina diferentes aproximacións xeométricas, corrixidas pola chamada “solidez” das partículas.

Deste xeito, NOMME ofrece unha visión integrada que combina identificación, clasificación, rangos de tamaño e estimación da masa. Parámetros importantes para os estudos ambientais e toxicolóxicos.

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¿Interfiere la conservación de muestras biológicas en el análisis de microplásticos?

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La durabilidad de los plásticos, junto con el aumento exponencial de su producción desde mediados del siglo XX, y la deficiente gestión de sus residuos, ha generado un grave problema ambiental global. Su presencia en los ecosistemas hace que los microplásticos (MPs) resulten potencialmente biodisponibles para una amplia variedad de organismos, por lo que son objeto de estudio prioritario.

El análisis de MPs en muestras de biota tiene como finalidad identificar y cuantificar partículas plásticas ingeridas o incorporadas por distintos organismos. Este proceso comprende toma de muestra, conservación de la misma, digestión del material biológico, separación de las partículas de la matriz biológica, y finalmente su identificación y caracterización mediante, preferentemente, la espectroscopía en la región infrarroja media.

El proceso de análisis presenta varias dificultades, entre ellas la conservación de las muestras. Es común que deban almacenarse entre el momento de su recolección y el procesado final. Las opciones más frecuentes son la congelación (que puede romper tejidos) y el formol (formalina); esta última muy habitual en el campo de la Biología porque estabiliza los tejidos e impide su degradación.

El formol (formalina) para conservación de muestras biológicas consiste en una solución acuosa de formaldehído (aproximadamente, al 4 %) usado como estabilizador de la materia orgánica y fijador químico. No obstante, no es bien conocido si su presencia puede interferir en la detección y caracterización de polímeros, dado que puede generar residuos o reacciones químicas que dificulten la separación y posterior identificación de microplásticos.

En este trabajo se pretende evaluar si la conservación en formol de muestras biológicas de cnidarios y moluscos interfiere en el proceso de análisis de microplásticos mediante comparación con la técnica de conservación por congelación. Para ello, se digieren con el mismo protocolo muestras de medusas, unas conservadas con formol y otras congeladas en agua de mar. Se pretende identificar posibles problemas durante el procedimiento de trabajo y conocer si el uso de formol es compatible con estudios de microplásticos que requieren digestión previa de la biota.

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Simultaneous *Enterococcus faecalis* and Tetracycline treatment by UV-C/Photocatalysis in Aquaculture Matrices

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The increasing demand for sustainable food production has accelerated the implementation of recirculating aquaculture systems (RAS), which minimize water consumption through intensive recirculation [1]. However, this strategy also leads to the accumulation of micropollutants and the proliferation of microorganisms, posing risks to fish health and system performance. Advanced Oxidation Processes (AOPs) have emerged as effective solutions for simultaneous contaminant degradation and pathogen inactivation, relying on the generation of highly reactive species [2]. Among them, photocatalysis stands out as a promising heterogeneous AOP, offering the potential for integrated treatment of both chemical and biological pollutants in RAS, thereby supporting safe and sustainable aquaculture practices. In this work, the simultaneous treatment of tetracycline (TC) and *Enterococcus faecalis* in aquaculture matrices will be assessed.

A UV-C batch system (PhotolabLED265-0.1er/cb, Apria Systems, Ltd.), operating at 0.5–2.5 W/m² irradiance, was used to treat simulated aquaculture water spiked with *E. faecalis* (10⁶ CFU/mL; ATCC 29212, Scharlab), and TC (25 mg/L, Sigma). The photocatalytic performance of commercial Ga₂O₃ (Sigma-Aldrich) and a synthetic material based on lanthanum and gallium (Cat) was assessed in the concentration of 0.1–1.0 g/L. Peracetic acid (C₂H₄O₃, Panreac) served as an oxidant for the intensification of the process.

After assessment of the UV-C dosage (2.5 W/m², 120 minutes) for the degradation of TC, a study of the photocatalytic performance of the materials is carried out. Cat/UV-C system achieves a 33% removal of the antibiotic in 120 minutes with an optimal catalyst concentration of 0.5 g/L. None of the studied materials present intrinsic antibiotic treatment properties in the dark phase. Application of PAA supposes an increase of the antibiotic degradation yield, with the Cat/UV-C/PAA system achieving >99% TC removal after 60 minutes of treatment with an oxidant dose of 0.5 mM. Bacterial inactivation below the detection limit (10 CFU/mL) is ensured for both the Cat/UV-C and Cat/UV-C/PAA system in less than 15 minutes. A scavenging study is carried out, showing that ¹O₂ and HO[•] are the main reactive oxygen species participating in the photocatalytic process.

Lanthanum and gallium-based materials/UV-C photocatalysis, especially when intensified with the application of PAA, is a promising technology for the advanced treatment of aquaculture matrices. This technology allows the quantitative elimination of antibiotic pollutants, while ensuring matrix disinfection, offering a solution to the challenge of water quality assurance in aquaculture systems. Future studies, aiming at the system scale-up, and water parameters (such as pH) influence on the treatment process, are to be considered.

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Assessment and mitigation of biocides in urban waters - Porto city as a case study

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The urban landscape presents a multitude of challenges, among them the often-ignored menace of biocides infiltrating urban waters. These substances, ubiquitous in urban settings, serve diverse roles including disinfection, wood and paint preservation, and pest control across homes, public spaces, medical facilities, and industrial sites [1]. Yet, their insidious presence extends beyond intended applications, seeping into urban water systems through runoff, surface leaching, and improper disposal practices. Once in the environment, they accumulate in sediments and aquatic organisms, disrupting fragile ecosystems [2].

Biocides in urban waters not only imperil the environment but also pose risks to human health, through drinking water, recreational activities, or consumption of contaminated seafood. Within this context, the PortoBiocide project (ea516bd1-c5a0-40a0-968b-68740cd7258f) addresses this issue with four main goals: i) Characterize the presence of biocides in Porto's waters, identifying zones of emission, transformation, and accumulation; ii) Develop and implement a catalytic reactor for photo-oxidation of biocides to be applied in the treatment of water effluents; iii) Develop a new natural deep eutectic solvent (NADES) with biocidal activity; and iv) Characterize the ecotoxicological profile of the new NADES.

This innovative project brings together the efforts of four renowned research organizations—REQUIMTE, ICBAS, ÁGUAS E ENERGIA DO MUNICÍPIO DO PORTO, and ISAVE—with expertise in chemistry, engineering, and ecotoxicology. Overall, PortoBiocide outcomes are expected to characterize and reduce the impacts of biocides on human health and the environment.

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Optimized method for the quantification of airborne microplastics in high traffic environments

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Road traffic, a major source of air pollution, generates not only exhaust-related emissions but also non-exhaust emissions, including microplastics (MPs), which have recently attracted growing global concern. These airborne MPs, originating primarily from tire and brake wear as well as the re-suspension of road dust, represent potential environmental and human health risks due to their physical and chemical properties and likelihood of inhalation [1].

The method proposed by Prata et al. [2] was adapted and optimized to quantify particles suspected to be microplastics in air samples from high-traffic locations. Organic matter was first removed by treating samples with 30 mL of 15% H₂O₂ in glass beakers covered with aluminum foil, which were then placed in a laminar flow cabinet at room temperature for eight days. The digested samples underwent density separation using a NaI solution (1.26 g/cm³); after vortexing and settling for 90 minutes, only the upper fraction was filtered onto pretreated glass microfiber filters. To facilitate identification, Nile Red dye (0.01 mg/mL in ethanol) was applied, and the filters were subsequently rinsed, dried, and stored in the dark. The filters were then analyzed under a stereomicroscope with fluorescence illumination, photographed, and assessed using ImageJ and/or FastStone Image Viewer, with suspected microplastic particles counted manually. For counting, the following criteria was adopted: all dark, opaque, black particles were classified as suspected micro-rubbers, while fluorescent particles were classified as microplastics.

Air samples were collected inside the Marquês Tunnel, in Lisbon, between October 22 and 30, 2024, using a high-volume sampler, equipped with a PM₁₀ size selective inlet and operated at a flow rate of 216.7 L/min. Fifteen samples were collected on quartz fiber filters and analyzed along with two laboratory blanks.

The total concentration of particles suspected to be microplastics in the tunnel air ranged from 520.1 to 4216 particles per m³, exceeding the levels commonly reported in outdoor urban environments [3, 4]. Such high concentrations are most likely attributable to the confined nature of the tunnel, which restricts ventilation and consequently limits the dispersion of airborne pollutants. Suspected micro-rubbers accounted for more than 90% of the suspected microplastics identified in these samples.

The optimized method provides a reliable approach for detecting and quantifying airborne microplastics in urban traffic environments.

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Valorisation strategies for an oily sludge coming from a petrol refinery: processes simulation and environmental life cycle assessment

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Oily sludge wastes (OSWs) are generated in refinery treatment plants during the petroleum refining processes. The hazardousness associated with the disposal of these OSWs, makes necessary the development of innovative technologies to handle them adequately. In this work, we study the treatment of a real oily refinery sludge and its valorisation through its transformation into hydrochar (HC), activated carbon (AC) and energy, considering the treatment of the residual aqueous phase. For this purpose, different strategies (Fig. 1a) were considered to treat the sludge by hydrothermal carbonisation (HTC), pyrolysis of the hydrochar and catalytic wet air oxidation (CWAO) of the aqueous phase. Following this strategy, HTC, pyrolysis and CWAO have been performed at lab-scale obtaining several samples, which were thoroughly characterised. Afterwards, the different strategies were simulated by ASPEN Plus v12.1 and, then, the environmental life cycle assessment (LCA) performed using SimaPro and ecoinvent database. As observed in Fig. 1b-d, the strategy B (HC production) and D (AC production) show the lowest impacts in terms of climate change and acidification categories, while strategy A (incineration) and strategy D are the most favourable options for eutrophication freshwater.

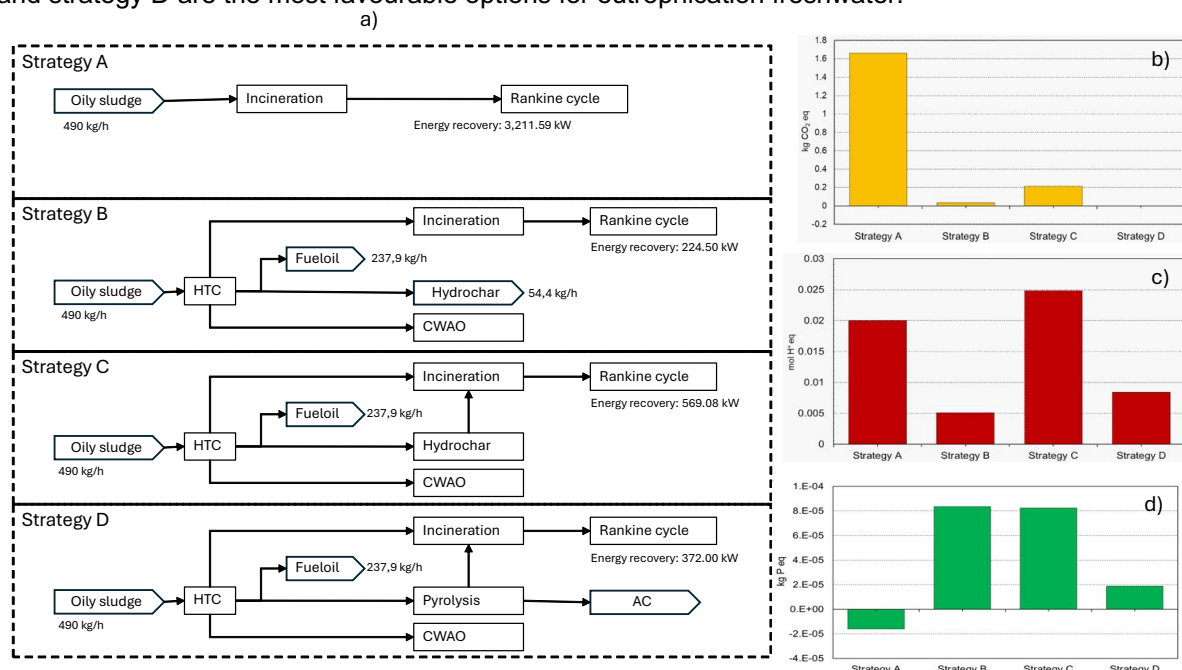


Fig.1. Valorisation of oily petrochemical sludge through (a) four strategies and the results obtained for different impact categories through LCA in terms of b) climate change, c) acidification, and d) eutrophication freshwater.

Acknowledgements

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Valorización de Residuos Agroforestales

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Los residuos lignocelulósicos representan una de las principales fuentes de materia prima renovable con potencial para ser valorizada dentro de la bioeconomía circular. Entre ellos destacan tanto los residuos forestales generados en las operaciones de tala de *Eucalyptus globulus* como los subproductos agrícolas como la paja de cebada. Estos materiales pueden convertirse en una alternativa sostenible para la obtención de compuestos de valor añadido.

La composición de estas biomasas se basa principalmente en la celulosa, hemicelulosa y lignina, una matriz estructural compleja que impide un acceso directo y eficiente a los azúcares fermentables. Por ello, resulta necesario aplicar procesos de pretratamiento que permitan romper dicha estructura y separar de manera selectiva sus componentes principales. Entre las estrategias más destacadas se encuentran la autohidrólisis acuosa y el pretratamiento organosolv con mezclas hidroalcohólicas. Ambos métodos, además de ser considerados tecnologías respetuosas con el medio ambiente, facilitan la solubilización parcial de hemicelulosas y lignina, obteniéndose una fracción sólida enriquecida en celulosa con una accesibilidad significativamente mejorada.

Los productos derivados de estos pretratamientos pueden destinarse a diferentes aplicaciones de interés. Los azúcares liberados tras el fraccionamiento pueden emplearse en procesos fermentativos orientados a la producción de biocombustibles como el etanol o el biohidrógeno.

De este modo, tanto los residuos forestales de *Eucalyptus globulus* como la paja de cebada constituyen ejemplos representativos de recursos lignocelulósicos cuyo aprovechamiento puede contribuir al desarrollo de tecnologías sostenibles de conversión energética y a la consolidación de una **economía circular** basada en el uso eficiente de materiales renovables.

Cyclodextrins as carriers for drug delivery: methods for the thermodynamic analysis of inclusion complex formation

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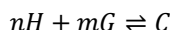
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Cyclodextrins (CDs) are cyclic oligosaccharides composed of D-(+)-glucopyranose units linked by α -1,4 bonds, obtained through the enzymatic degradation of starch. They exhibit a hollow truncated cone-shaped structure, with a lipophilic inner cavity and a hydrophilic outer surface. Owing to this dual nature, they can encapsulate non-polar organic molecules while maintaining good aqueous solubility. This property is particularly valuable in the pharmaceutical field, as it enhances both the stability and solubility of drugs with poor water solubility. Among the native cyclodextrins (α , β , and γ), the β -CD has limited aqueous solubility; therefore, chemically modified derivatives bearing different functional groups (e.g., hydroxypropyl or sulfobutyl ether) have been developed, significantly improving their solubility in water.

For several years, our research has focused on the synthesis of drugs and prodrugs that are incorporated into native and modified cyclodextrins in order to increase their solubility and bioavailability through the formation of an inclusion complex. This association is governed by the a simple binding equilibrium model^{1,2}



where H corresponds to cyclodextrin, G to the guest, and n and m are the stoichiometric coefficients. The association constant, expressed in terms of concentrations, is defined as:

$$K = \frac{[C]}{[H]^n[G]^m}$$

The aim of this study was to develop a methodology for the thermodynamic analysis of inclusion complex formation using UV-Vis spectroscopy and spectrofluorimetry, taking as a reference the complex formed between 2-anthracenecarboxylic acid (2AC) and β -cyclodextrin (β -CD), with the goal of applying it to the study of other similar inclusion complexes based on CDs.

The Job's method was applied to determine the stoichiometry, the Benesi-Hildebrand method to calculate the association constant (K), and the van't Hoff equation to obtain the thermodynamic parameters. The results were compared with those reported by Wei *et al.*³ for the same system, in which circular dichroism was used as the analytical technique. The low solubility of 2AC required the preparation of its potassium salt (K2AC) and a change of solvent.

The formation of the K2AC/ β -CD complex was confirmed, with an association constant on the order of 10^3 M^{-1} . UV-Vis spectrophotometry enabled estimation of K but did not allow accurate determination of the stoichiometry due to its relatively low sensitivity. Spectrofluorimetry proved to be more effective, allowing not only a more precise determination of K but also the calculation of thermodynamic parameters, which indicated a spontaneous and exothermic process.

Acknowledgements

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Pharmaceutical profiling of leflunomide: exploring polymorphic forms and cocrystals

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One of the major challenges in the development of active pharmaceutical ingredients (APIs) is their poor aqueous solubility, as it often results in reduced oral bioavailability and limited therapeutic efficacy. This issue is particularly relevant for drugs classified as classes II and IV of the Biopharmaceutical Classification System (BCS).[1] Among the most promising approaches developed to improve these properties are crystal engineering techniques, including finding new polymorphs and cocrystallization. Polymorphism refers to the ability of a compound to exist in more than one solid form and has been observed in approximately 80% of APIs. On the other hand, pharmaceutical cocrystals are multicomponent crystalline solids comprising a defined stoichiometric ratio of an API and a pharmaceutically acceptable coformer, both solid at room temperature. Both polymorphism and cocrystallization can lead to solid forms with different physical and chemical properties such as solubility, stability, dissolution rate and bioavailability, while preserving the therapeutic effect.[2-4]

In this work, leflunomide, Fig. 1(a), a poorly water-soluble, disease-modifying antirheumatic drug (DMARD), belonging to class II of BCS, was investigated with the aim of discovering new polymorphs and pharmaceutical cocrystals. For polymorph screening, the drug was crystallized from solutions in various solvents, by solvent evaporation under different conditions. Additionally, cocrystallization experiments were conducted using different coformers by mechanochemistry. The solid forms obtained through both solvent crystallization and mechanochemistry were characterized by differential scanning calorimetry, hot stage microscopy, Fourier transform infrared spectroscopy and X-ray powder diffraction. Two new polymorphic forms of leflunomide were obtained from crystallization in ethanol and in a 1:1 (V/V) ethanol/water mixture. Furthermore, a novel cocrystal of leflunomide with 2,4-dihydroxybenzoic acid (Fig. 1b), in a 1:1 molar ratio, was successfully synthesized. The material obtained by mechanochemistry was recrystallized by solvent evaporation from an ethyl acetate solution, which allowed the growth of single crystals suitable for structure resolution by single crystal X-ray diffraction.

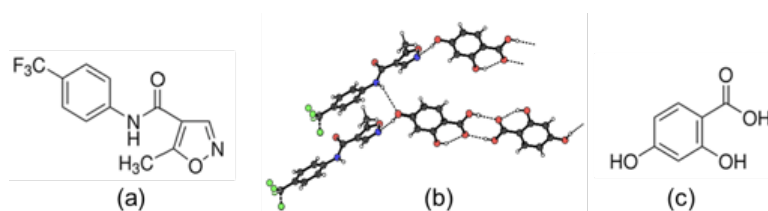


Fig. 1. Molecular structures: (a) leflunomide; (b) Partial view of the hydrogen-bonding interactions in the cocrystal; (c) 2,4-dihydroxybenzoic acid.

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Produtos derivados da auga mineral medicinal para usos cosméticos

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A auga mineral medicinal é un recurso natural presente no noso planeta e que se utilizou para diversos fins desde a antigüidade. A auga mineral medicinal brota en todos os continentes e, debido aos procesos hidroxeolóxicos, cárgase con diversos elementos durante o seu tempo dentro da Terra. A auga de mar [1] tamén é unha auga mineral medicinal natural cuxa composición abrangue a maioría dos elementos químicos.

As augas mineral medicinais posúen numerosas propiedades farmacolóxicas [2], debido á súa composición química e propiedades físicas [3]. Moitas das propiedades da auga mineral medicinal son beneficiosas para tratamentos dermatolóxicos [4], principalmente debido aos seus efectos hidratantes e calmantes. Numerosos artigos na literatura demostran os efectos cosméticos da auga mineiro-medicinal [5,6]. Isto levou a un aumento considerable no uso da auga mineiro-medicinal nos balnearios con fins cosméticos.

As características da auga mineiro-medicinal fan que agora sexa un ingrediente importante en numerosos produtos cosméticos [3], incluíndo cremas, champús, xabóns, etc. Un destes produtos derivados da auga mineiro-medicinal son os peloides termais [7], que se utilizaron con fins cosméticos en varios balnearios termais de todo o mundo, como se demostrou no recente Congreso Iberoamericano de Peloides celebrado no Balneario de Arnedillo en maio de 2025 [8].

Os produtos de auga mineral conteñen elementos de alto valor cosmético, como selenio, calcio, magnesio, zinc e outros oligoelementos [9], que lles confiren un alto valor engadido.

Este traballo presenta os produtos de auga mineral que se usan actualmente en todo o mundo.

Agradecimentos

Agradecemos o apoio financeiro prestado polo proxecto GRC-ED431C 2024/25 Z424 da Xunta de Galicia. Este proxecto está cofinanciado con fondos FEDER.

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Projeto AQUAPRED para monitorização de águas mineromedicinais utilizando inteligência artificial

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O AQUAPRED é um projeto multiterritorial, multidisciplinar e interdisciplinar que envolve física, medicina, farmácia, química, biologia e ciência da computação. Concentra-se na monitorização e controlo de águas mineromedicinais em spas e na previsão de contaminantes com base na digitalização de dados em tempo real dos parâmetros fundamentais da água mineromedicinais.

O projeto determinará os parâmetros fundamentais dos spas termais, bem como as suas taxas de flutuação na água termal dentro de uma instalação termal. Estudará também a sua influência na segurança terapêutica e na qualidade da água, bem como possíveis modelos para a sua relação.

O projeto estabelecerá um modelo do hidrobioma das águas termais e associá-lo-á às suas propriedades terapêuticas. Sistemas piloto para recolha de dados em tempo real serão desenvolvidos e instalados em spas na área SUDOE. Os parâmetros serão monitorizados através de dispositivos concebidos especificamente para este fim, para posterior digitalização e análise. Será desenvolvido um sistema inteligente que, com base nestes dados, permitirá o controlo dos sistemas de tratamento de água, e será desenvolvido um modelo de aprendizagem automático para prever o potencial aparecimento de contaminantes na água. Com base nos dados recolhidos, serão também aplicados modelos de eficiência energética para otimizar o consumo e reduzir a procura.

Agradecimentos

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Lanthanoid-based luminescent anion sensing using a $\text{Eu}^{3+}/\text{Tb}^{3+}$ heterometallic complex

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Selective anion contamination in water has become a pressing environmental and public-health issue, as naturally occurring species can reach harmful levels due to human activity.[1] Against this backdrop, lanthanoid-based optical sensors have emerged as promising tools thanks to their unique photophysical signatures, which enable fast readout and high spatial resolution.[2]

Here we focus on chemical anion sensors in aqueous media, where changes in luminescence provide the analytical signal that correlates the analyte-sensor interaction. Ratiometric designs using heteronuclear Eu/Tb complexes are particularly attractive: if the bands associated with each metal center respond differently to a given anion, the Eu/Tb intensity ratio affords quantitative analysis and mitigates the non-linearity of absolute intensity with concentration. In our group, we have described metal complexes with macrocyclic ligands that efficiently sensitize lanthanoids. [3-4]

With these considerations in mind, we present the synthesis and structural/optical characterization of the heterometallic complex $\{\text{Eu}_{0.9}\text{Tb}_{0.1}(\text{LN6, en, r})(\text{OAc})_2\} \cdot 0.5\text{CHCl}_3$ ($\mathbf{1} \cdot 0.5\text{CHCl}_3$). As a ratiometric anion sensor, $\mathbf{1} \cdot 0.5\text{CHCl}_3$ displays distinct luminescence responses toward F^- , HSO_4^- , and H_2PO_4^- when each anion is tested individually. In binary and ternary mixtures containing these anions, $\mathbf{1} \cdot 0.5\text{CHCl}_3$ acts as a selective detector for HSO_4^- , except in the specific $\text{HSO}_4^-/\text{H}_2\text{PO}_4^-$ combination, where the selectivity is compromised. Overall, these results position $\mathbf{1} \cdot 0.5\text{CHCl}_3$ as an effective ratiometric platform for anion sensing in water.

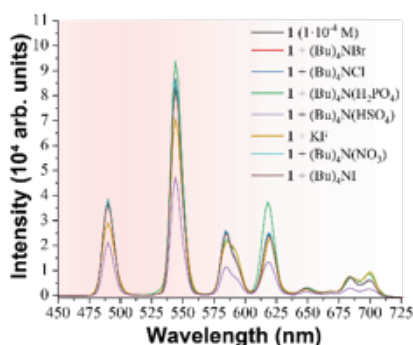


Fig.1. Emission spectra of $\mathbf{1} \cdot 0.5\text{CHCl}_3$ and their variation in the presence of a series of anions.

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Catechol oxidase activity of synthetic dinuclear copper(II) complexes

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Catechol oxidases are copper enzymes that use molecular oxygen to catalyze the oxidation of ortho-diphenols into ortho-quinones. Recently, synthetic models of this enzyme have attracted interest due to their potential application in areas such as biosensing, phenol degradation, and redox-modulating therapies [1-3].

In this work, we report the catechol oxidase activity of nine dinuclear copper complexes (Figure 1 left) prepared from a set of pentadentate $[N_2S_2O]$ bithiosemicarbazone and bishydrazone ligands H_3L^1 - H_3L^9 , by using an electrochemical methodology.

Kinetic studies of the catalytic oxidation by these synthetic complexes of 3,5-di-tert-butylcatechol to the corresponding quinone, followed by UV-Vis spectroscopy, led to the determination of the observed rate constant value (K_{obs}) at different substrate concentrations. Plot of the K_{obs} over initial substrate concentration gave the Michaelis constant (K_M , see Figure 1 right) for each of the catalysis, thus enabling the discussion of the activities of the different complexes based on the structure of these models, which had previously been resolved by X-ray crystallography.

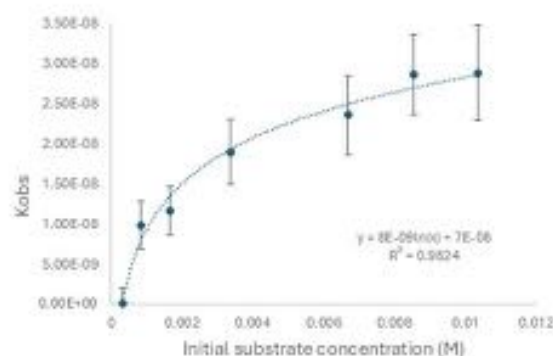
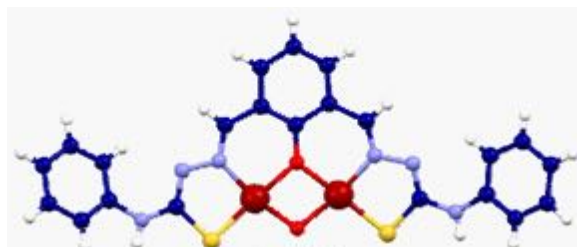


Fig.1. Left: dinuclear copper(II) complex $[Cu_2(L^3)(OH)]$; right: plot of the k_{obs} over initial substrate concentration, a logarithmic fit was applied to the obtained curve, and from the equation of the curve in the form of $y = a \ln(x) + b$, the value of a was determined and recorded as K_M .

Acknowledgments

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Synthesis and study of semicarbazone ligands derived from carborane

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Recently, carboranes have gained importance in the field of biological applications, as their properties allow the increase of metabolic stability of drugs, the development of new biological probes, and antitumour treatment through BNCT [1-3].

This work studies the synthesis, characterisation and coordination ability of two new semicarbazone ligands functionalised with carborane groups, one with the *meta*-carborane isomer and the other with the *ortho*-carborane isomer. This type of ligand has not been studied previously, despite its potential interest in pharmacological applications.

To study the coordination ability of the ligands, their complexes with transition metals were synthesised: palladium, iron, copper and cobalt. The complexes [PdL₂] and [CuL₂] were obtained with the *meta* isomer, characterised by IR spectroscopy. It was not possible to synthesise the [FeL₂] complex with the *meta* isomer using electrochemical methods, nor the [CoL₂] complexes with either of the two isomers.

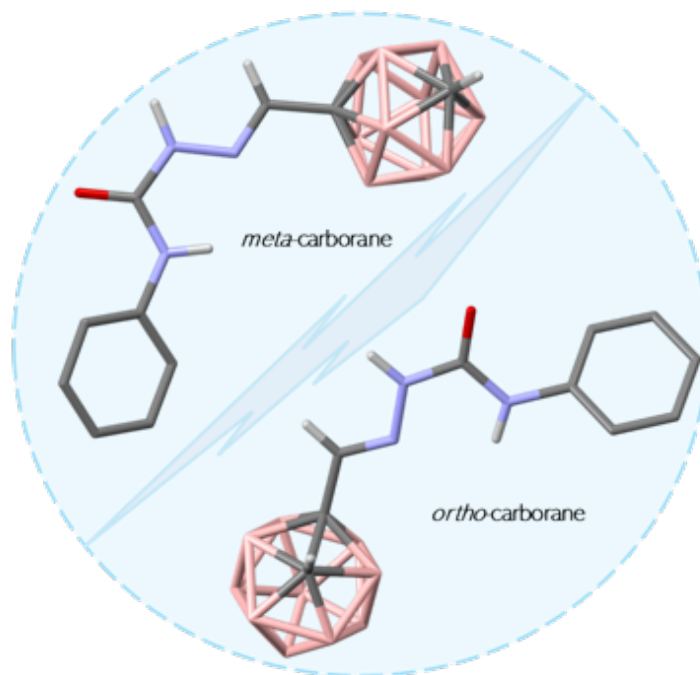


Fig.1. Ligands studied in this work.

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Modificación dun ligando *biscarbamato* para o estudo da hidrólise dos seus complexos derivados

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Os carbamatos son un tipo de compostos orgánicos amplamente estudados [1]. Os seus complexos metálicos presentan propiedades únicas que os fan útiles en catálise, no deseño de materiais, e tamén mostran potencial en aplicacións biomédicas, como fármacos anticancerixenos, debido á súa capacidade de interactuar con biomoléculas e modular procesos celulares [2,3].

O grupo de investigación SUPRABIOIN estudou o comportamento coordinativo dun ligando *biscarbamato* cun espazador piridina que posúe ramas 4-metoxibencilo [4]. Durante o estudo observouse que o ligando experimentaba un proceso de hidrólise, liberando CO₂ e o correspondente alcol, e orixinando complexos do tipo hidrazona. Co obxectivo de analizar qué factores están implicados neste proceso de hidrólise, nesta comunicación realizouse unha modificación no esqueleto do ligando. Así, levouse a cabo a substitución do grupo aromático 4-metoxibencilo por un grupo alifático etilo (ver Fig. 1), e estudouse a súa coordinación fronte a metais de transición e post-transicionais.

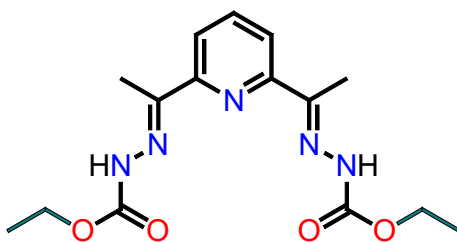


Fig.1. Representación do ligando *biscarbamato* H₂L¹, funcionalizado cun grupo terminal etilo

Agradecementos

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Obtaining a Ag(I) coordination polymer derived from an azine ligand

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Nowadays, alternative therapeutic strategies are being studied due to the increasing of bacteria resistance to antibiotics. In this context, interest in silver complexes has grown because of their potential antimicrobial properties which could serve as an alternative treatment to conventional antibiotics.[1] Coordination chemistry offers a way to design different types of metallosupramolecular architectures such as coordination polymers. These types of polymers represent a class of metal complexes with promising applications in fields such as Material Science [2] and Pharmacology. [3] Moreover, the design of the organic ligands plays a crucial role in the obtained architecture and its properties. For instance, Schiff bases are well known ligands not only for their coordination versatility but also for their inherent biological activities. [4] In the present work, we have designed a silver coordination complex derived from a phosphinoazine ligand (Fig. 1) with potential antimicrobial activity. Both the ligand and the polymeric complex were characterized by common characterization techniques in solid state and solution.

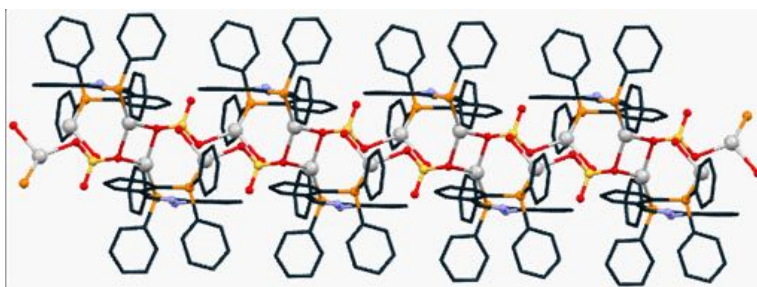


Fig.1. Crystal structure of the silver coordination polymer

Acknowledgments

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A new manganosalen synthetic model with remarkable antioxidant activity in different enzyme-like activity assays

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Manganosalen complexes are a class of synthetic catalysts inspired by natural antioxidant enzymes such as catalases or glutathione peroxidases [1]. The role of these enzymes is to neutralize reactive oxygen species, which are strong oxidants that cause oxidative stress, in order to maintain biological balance and avoid cellular damage. The synthetic antioxidants like manganosalen have advantages comparing them to natural antioxidants because of their characteristics, such as their low molecular weight and their low production costs [1-2]. Therefore, they are an alternative to natural antioxidants therapy.

In this communication, we report the synthesis of a new metal manganese complex **1** (Fig. 1) using a base Schiff ligand obtained by condensation of 1,2-diaminopropane and 2-hydroxy-3-methoxybenzaldehyde. The complex was characterized by conductivity measurements, IR and UV-Vis spectroscopies, X-ray crystallography and mass spectrometry among other analysis. Its redox potential was tested by cyclic voltammetry (Fig. 1).

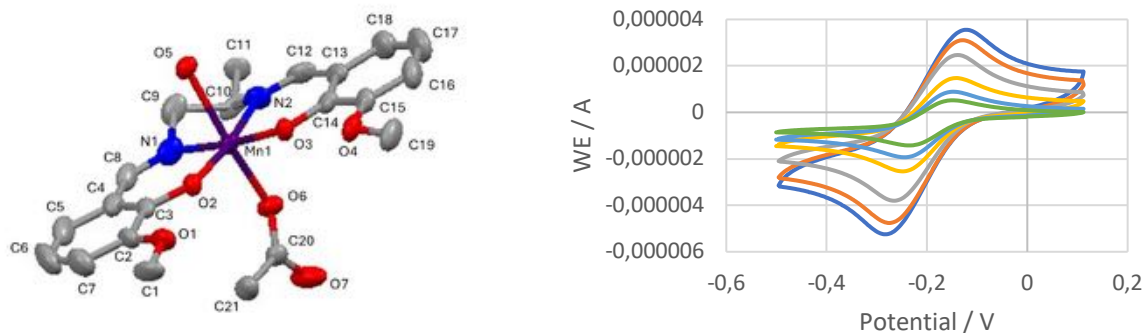


Fig. 1. Left: crystal structure of the synthetic manganosalen complex **1**; Right: Cyclic voltammograms for **1** at different scan rates (0.005-0.1 V s⁻¹).

The catalytic antioxidant activity of this synthetic model was determined by peroxidase, catalase and catechol oxidase assays. The results showed the high rate of reversibility for the Mn(II)/Mn(III) process and remarkable activities, which confirm the antioxidant mimetic role of this synthetic model.

Acknowledgments

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Influence of the anion on ionic cyclometalated Pt(II) complexes

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The development of novel luminescent materials based on transition metal complexes has long put special emphasis on cyclometalated Pt(II) derivatives, which continue to play a central role in the topic [1]. Their rich spectroscopic and photophysical properties enable applications in diverse fields such as biomarkers and optoelectronic devices [2]. These properties can be tuned by modifying the electronic nature of the cyclometalated and the auxiliary ligands [3].

In this work, we report mononuclear cationic cyclometalated-diphosphine Pt(II) complexes of the type $[\text{Pt}(\text{C}^{\wedge}\text{N})(\text{P}^{\wedge}\text{P})]^+$, where $\text{C}^{\wedge}\text{N}$ = 1-phenylisoquinolate (piq) or 2-phenylbenzothiazolate (pbt), and $\text{P}^{\wedge}\text{P}$ = 1,2-bis(diphenylphosphine)benzene (dppbz). The compounds have been fully characterized by mass spectrometry, NMR and X-ray diffraction. Their optical properties have also been studied, and the origin of their bright yellow ($\text{C}^{\wedge}\text{N}$ = pbt) and orange-red ($\text{C}^{\wedge}\text{N}$ = piq) emissions has been rationalized on the basis of the ligand characteristics, using also theoretical calculations.

For some applications, cationic species are of special interest, but the effect of the anion is usually ignored. For this reason, we are investigating the effect of the counterion to change the behavior of the ionic Pt(II) complexes. The widely employed PF_6^- anion is compared with the unexplored weakly coordinating $[\text{Al}(\text{OC}(\text{CF}_3)_3)_4]^-$ in two representative systems, $[\text{Pt}(\text{pbt})(\text{dppbz})]^+$ and $[\text{Pt}(\text{piq})(\text{dppbz})]^+$, in order to explore how the nature of the anion can influence the optical properties, solubility and stability.

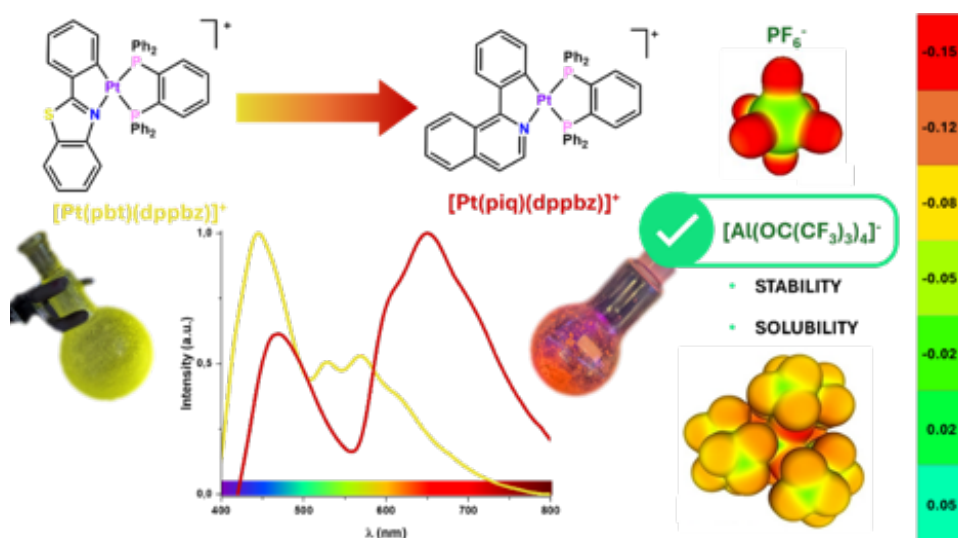


Fig.1. Emission of the complexes $[\text{Pt}(\text{C}^{\wedge}\text{N})(\text{dppbz})]\text{PF}_6$ in CH_3CN and comparison of the effect of the studied anions.

Acknowledgments

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4,4'-di-*tert*-Butylbiphenyl Pt(II) and Au(III) analogs complexes incorporating cyanide and/or isocyanide auxiliary ligands: synthesis and optical properties

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Complexes of d⁸ Pt(II) and Au(III) ions have both demonstrated interesting photophysical properties that make them suitable for use in light-emitting devices. However, they display notable differences in their redox, chemical and catalytical properties, as illustrated in the fact that whereas the first Pt(II) carbonyl complex was synthesized in 1868, the related first Au(III) carbonyl complex was not obtained until 2015.

To achieve luminescent complexes, featuring intense emissions^[1], conventional strategies to favour ³MLCT or mixed ³IL/³MLCT transitions, suppressing non-radiative decay pathways, are usually sought. These strategies involve the use of rigid chromophoric ligands^{[2], [3]}, such as neutral (N[^]N, N[^]N[^]N) or anionic cyclometalated or dimetalated (C[^]N, C[^]C[^]N, C[^]N[^]C, etc) groups. Moreover, to reduce non-radiative decay, generally, auxiliary ligands that induce strong crystal field splitting, such as acetylides, isocyanides, or carbenes, should be introduced.

With these requirements in mind, we found it of interest to study biaryl bimetalated (C[^]C) systems with the 4,4'-di-*tert*-butyl-1,1'-biphenyl ligand, which, in combination with other strong-field coligands, can enhance luminescence^[4]. As coligands, cyanides and/or isocyanides will be employed, which, unlike carbonyls, form stable complexes with Pt(II) and Au(III), what has allowed us to comparatively study the photophysical properties of analogous complexes with both ions.

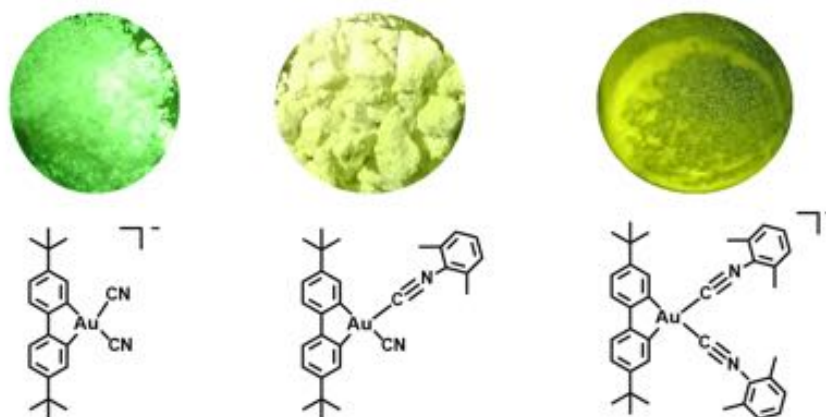


Fig.1. Emissions of different Au(III) complexes.

Acknowledgments

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Preparation and molecular structure analysis of dinuclear palladacycles with tertiary phosphine ligands

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Palladacycles have been known for the past decades and the cyclometallation reaction has been profusely investigated in view of the rich chemistry it renders; complexes with a great variety of ligands and metal centers may be found in the literature [1-3]. In particular, multiple donor ligands are quite interesting in view of the properties they add to the ensuing compounds. It is worth mentioning here the potentially bidentate ligands such as the Schiff bases that give dinuclear species with an assortment of bridging ligands [4].

Reaction of the acetate dinuclear complexes with dpmm ($\text{Ph}_2\text{PCH}_2\text{PPh}_2$) followed by treatment with ammonium hexafluorophosphate gave new doubly mix-bridged palladacycles as 1:1 electrolytes. Treatment of the halide-bridged species with *trans*-dppe (*trans*- $\text{Ph}_2\text{PCH}_2=\text{CH}_2\text{PPh}_2$) afforded the corresponding dinuclear molecular complexes.

The crystal and molecular structures of $[\text{Pd}\{2,4-(\text{MeO})_2\text{C}_6\text{H}_2\text{C}(\text{H})=\text{N}(3,4-(\text{Cl})_2\text{C}_6\text{H}_2)\}(\mu\text{-OAc})]_2$ (**1**) $\{[\text{Pd}\{2,4-(\text{MeO})_2\text{C}_6\text{H}_2\text{C}(\text{H})=\text{N}(3,4-(\text{Cl})_2\text{C}_6\text{H}_2)\}_2(\mu\text{-OAc})(\mu\text{-Ph}_2\text{PCH}_2\text{PPh}_2)]\text{[PF}_6\text{]}\}$ (**2**) and $[\text{Pd}\{2,4-(\text{MeO})_2\text{C}_6\text{H}_2\text{C}(\text{H})=\text{N}(3,4-(\text{Cl})_2\text{C}_6\text{H}_2)\}(\text{Br})_2(\mu\text{-trans-Ph}_2\text{PCH}_2=\text{CH}_2\text{PPh}_2)]$ (**3**) (Fig. 1.) are described. The crystals were triclinic, space group *P*-1, with $a = 9.5409(13)$, $b = 14.8059(18)$, $c = 19.082(4)$ Å, $R_1 = 0.0561$, $wR_2 = 0.1328$, for 17756 independent reflections with $I > 2.0\sigma(I)$; monoclinic, space group *c*2/*c*, with $a = 21.057(2)$, $b = 20.817(2)$, $c = 32.852(3)$ Å, $R_1 = 0.0634$, $wR_2 = 0.1682$, for 17676 independent reflections with $I > 2.0\sigma(I)$; and triclinic, space group *P*-1, with $a = 9.125(5)$, $b = 12.412(5)$, $c = 16.598(5)$ Å, $R_1 = 0.0338$, $wR_2 = 0.0902$, for 18238 independent reflections with $I > 2.0\sigma(I)$, respectively.

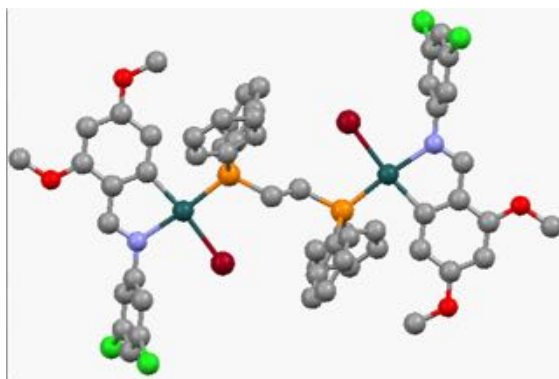


Fig.1. Molecular structure of compound **3**.
Dark blue: Pd; blue: N; red: O; dark red: Br; Green: Cl; orange: P.

Acknowledgements

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Energy management based on smart grid technologies: integration of renewable energy and energy storage systems

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Energy powers our homes, transportation, organizations, and even our personal activities. The global issues of atmospheric pollution, climate change, water pollution, thermal pollution, and solid waste disposal are expected to remain closely associated with the production and consumption of energy. Limiting global warming and reducing its effects, such as rising sea levels, extreme weather, and ecosystem disruptions, is going to require cutting back on greenhouse gas emissions [1].

Renewable Energy Sources (RESs), such as solar energy, emit few or no greenhouse gases, in contrast to fossil fuels; however, the fluctuating output power of RESs limits their large integration to the grid. In this context, Energy Storage Systems (ESSs) such as batteries stand as a promising solution to ensure balancing between generation and consumption and grid stability [2]. In line with the technology development, Smart Grids (SG) appeared, aiming to ensure the effective use of energy systems [3]. The smart grid concept is expected to become increasingly utilized in electric power and storage systems.

This work highlights the role of ESSs, such as batteries, including fresh batteries and Second-Life Batteries (SLBs) in the electrical power systems and their contributions to reducing energy cost, reducing CO₂ emissions, and maintaining battery lifecycle. Moreover, it highlights how ESSs operate in the SG, providing efficient energy management.

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Recovery and Separation of Valuable Metals of Spent Lithium-Ion Batteries

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Lithium-ion batteries (LIBs) are one of the most important electrochemical energy storage devices [1]. Given the content of LIBs in metals and their increasing production rate, it is vital to develop efficient, ecological, and economical worthwhile recycling processes [1,2]. This need becomes even more pressing when considering that the metal concentrations in LIBs often exceed those found in natural mineral reserves [3]. Nevertheless, at the end of their lifecycle, only a limited number of LIBs undergo recycling treatments [4].

Among the various recycling techniques, hydrometallurgy—which includes leaching, separation, and refining—is a viable metallurgical option. When compared to pyrometallurgy, hydrometallurgy offers better recovery rates, higher purity, and lower greenhouse gas emissions, thus holding greater ecological potential [1].

In this work, a new hydrometallurgical process for the treatment of waste lithium-ion batteries (based on lithium nickel manganese cobalt oxide cathodes) is presented (schematic representation in figure 1). The developed process integrates various approaches, ranging from inorganic leaching using an alternative mineral acid to more complex separation methods as solvent extraction and precipitation processes.

The quantification of the metals involved in the different processes was performed using techniques such as ICP-OES (Inductively Coupled Plasma Optical Emission Spectrometry) and XRF (X-Ray Fluorescence). Overall, the results demonstrated that the developed integrated process can produce LiCl solutions with ppm levels of other cationic impurities.



Fig.1. Schematic representation of the hydrometallurgical process for the treatment of waste lithium-ion batteries.

Acknowledgments

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Purification of bioactive molecules using a Simulated Moving Bed (SMB) system: from assembly to preliminary experiments

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The separation of isomers and enantiomers is challenging due to their similar physicochemical properties. Conventional methods often present drawbacks such as low selectivity, limited productivity, and high solvent consumption. Alternatives including adsorption processes (e.g., elution chromatography) and membrane technologies have been explored but are frequently hindered by poor efficiency and scalability. In this context, the Simulated Moving Bed (SMB) process emerges as an advanced chromatographic technique, enabling continuous operation with higher productivity and reduced solvent usage compared with traditional batch separations [1,2].

An SMB unit consists of chromatographic columns connected in a closed loop, with two inlets and two outlets whose positions are periodically switched. This generates an apparent countercurrent movement between solid and liquid phases, enhancing mass transfer efficiency and allowing continuous application of chromatographic principles.

This work reports the design, assembly, and validation of a four-zone SMB unit for the separation of bioactive compounds. The setup employs a one Select-Trapping (ST) valve per column (1ST1C) system [3], integrated with four pumps, a mass flow controller, and relief valves at the pump outlets. Different pump configurations were tested to minimize dead volumes, pressure drops, and flow instabilities. To validate the unit, the enantioseparation of nadolol racemates was performed using methanol with 0.1% diethylamine as the mobile phase and a chiral stationary phase characterized by experimentally determined linear-plus-Langmuir isotherm model. Operating conditions were defined using the Triangle Theory, and performance was evaluated in terms of purity, productivity, and solvent consumption [4]. The results confirm the reliability of the developed unit and demonstrate its potential for efficient and scalable chiral separations.

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Selective fractionation of bergamot oil components using the ionic liquid 1-ethyl-3-methylimidazolium acetate

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Citrus bergamia, commercially known as bergamot, is widely used for its essential oil, which provides a refreshing scent in perfumes and cosmetics, as well as a flavouring in food and confections, most famously in Earl Grey tea. Bergamot essential oil is mainly composed of the terpene limonene (LIM), and the terpenoids linalool (LOL) and linalyl acetate (LA). However, the terpene-rich fractions are prone to instability, which can negatively affect the quality and shelf life of the oil. Deterpenation processes are therefore important for improving oil stability and quality, as well as enabling recovery of valuable components from citrus industry waste streams [1]. The current separation methods used for terpenes fractionation have, however, some drawbacks, namely the high toxicity, flammability and/or volatility of the organic solvents used (usually hexane). To overcome this limitation, researchers have been investigating alternative solvents for deterpenation processes, among which neoteric ionic liquids have attracted increasing attention [2,3].

In this work, the ionic liquid 1-ethyl-3-methylimidazolium acetate ([C₂mim][OAc]) was evaluated for the deterpenation of bergamot essential oil using liquid-liquid extraction. The oil was modelled as a mixture of LIM, LOL and LA, and the liquid-liquid equilibrium with the ionic liquid was measured at 298.2 K. Based on the experimental data, relevant parameters (distribution ratios and selectivities) were determined to assess the extraction performance of [C₂mim][OAc]. Overall, the results show that [C₂mim][OAc] exhibits strong potential for extracting LOL. However, the low affinity observed between the ionic liquid and the other main components, LIM and LA, indicates that achieving the integral deterpenation of the essential oil remains a significant challenge. Nevertheless, this study provides valuable insights and experimental data that may contribute to the development of more sustainable strategies for the fractional extraction of the major terpenoids in bergamot essential oil.

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Estudo dun deseño de biorrefinería integrada con peles de pataca: Análise de ciclo de vida (ACV)

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Na actualidade, arredor do 80% da enerxía mundial procede de combustibles fósiles, ambientalmente insostibles e principais responsables das emisións de CO₂. A Axenda 2030 e os Obxectivos de Desenvolvemento Sostible (ODS) promoven a transición cara sistemas renovables mediante alternativas sostibles como os biocombustibles, entre os que destaca o bioetanol pola súa contribución á descarbonización e á economía circular. Paralelamente, o aproveitamento da biomasa residual agroindustrial permite reducir refugалlos e obter bioproductos de alto valor engadido [1].

Na produción de bioetanol, preto dun terzo do carbono da biomasa libérase como CO₂, limitando o rendemento teórico. Este subproduto pode ser valorizado para a biosíntese de ácido succínico (AS), incrementando a eficiencia global. A súa reutilización resulta estratéxica, xa que, aínda que o CO₂ non se considera contaminante, a súa acumulación contribúe significativamente ao quecemento global e á acidificación oceánica. Estes feitos evidencian a necesidade de integrar procesos innovadores que reduzan o impacto ambiental e aumenten a eficiencia global do sistema. A produción combinada de bioetanol e AS constitúe unha estratexia de valorización das emisións de carbono.

Para avaliar a súa viabilidade e beneficios ambientais, a análise del ciclo de vida (ACV) preséntase como ferramenta esencial, xa que ofrece unha visión holística das compensacións ambientais e facilita o deseño de rutas de valorización optimizadas [3]. Neste contexto, o obxectivo deste estudo é comparar dous escenarios de valorización ambiental das peles de pataca: (i) a produción exclusiva de AS mediante fermentación microbiana e (ii) un sistema integrado bioetanol-AS se baseado na reutilización do CO₂.

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Autohidrólise asistida por microondas como estratexia sostible para a produción de oligosacáridos e bioetanol a partir de madeira de *Acacia dealbata*

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A biomasa lignocelulósica constitúe unha das fontes renovables máis abundantes e accesibles, cun elevado potencial para a produción de biocombustibles e compostos de alto valor engadido [1]. *Acacia dealbata*, especie anxiosperma orixinaria de Australia e catalogada pola UE como invasora, presenta unha ampla distribución na península ibérica dende o século XX [2]. Na actualidade existe un interese crecente pola súa valorización como estratexia para mitigar parcialmente os custos asociados ao seu control [3].

A autohidrólise ou pretratamento hidrotérmico é unha tecnoloxía consolidada que emprega auga a altas presións e temperaturas para solubilizar de forma selectiva a fracción hemicelulósica da biomasa. En particular, a autohidrólise asistida por microondas (MAA) aparece como alternativa ao tratamento hidrotérmico convencional (CHT), xa que permite reducir os tempos de residencia e, en consecuencia, o consumo enerxético e a formación de subproductos. Os oligosacáridos obtidos presentan interese como prebióticos e como materia prima para producir biocombustibles ou compostos de aplicación alimentaria, mentres que o sólido residual pode ser valorizado mediante procesos fermentativos.

Neste estudo avalíase a aplicación de MAA sobre madeira de *A. dealbata* a severidades (S_0 , parámetro que integra tempo e temperatura) comprendidas entre 3,63 e 4,64, cunha consistencia de 6 g de sólido seco por cada 100 g de peso total. A concentración máxima de oligosacáridos no licor obtívose a $S_0 = 3,77$ (230 °C, 0,25 min), cun valor de 12,97 g/L, dos cales o 77% correspondían a xilooligosacáridos (9,97 g/L). A caracterización estrutural destes compostos realizouse mediante MALDI-TOF-MS e HPAEC-PAD.

Os sólidos recuperados foron sometidos a un proceso de presacarificación e sacarificación e fermentación simultáneas (PSSF), acadando arredor de 17 kg de bioetanol/100 kg de biomasa seca a $S_0 = 3,77$ –3,80, e en torno a 15 kg/100 kg a $S_0 = 4,44$ –4,49. En condicións equivalentes ($S_0 = 3,80$ e 4,44), o CHT resultou menos eficiente, confirmando que MAA proporciona maiores rendementos en bioetanol, menor xeración de subproductos de degradación (furfural, HMF), un maior contido de oligosacáridos en condicións severas e, adicionalmente, un consumo enerxético ata 2,75 veces inferior.

Os resultados amosan que MAA permite valorizar biomasa de *A. dealbata* mediante unha estratexia multiproducto con potencial aplicación no ámbito alimentario, en liña cos principios da economía circular e o desenvolvemento sostible.

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Green solvent and microwave-assisted strategies for efficient conversion of white grape pomace into 5-hydroxymethylfurfural

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The shift towards sustainable chemical production has intensified the search for innovative ways to transform agro-industrial residues into high-value products. Portugal and Spain are two of the world's largest wine producers [1], and consequently, this vast production generates a substantial amount of wine waste. White grape pomace (WGP), a sugar-rich by-product of the winemaking industry, is often discarded despite its potential as a renewable feedstock for bio-based chemicals. Harnessing such residues not only reduces waste but also supports the principles of green chemistry and circular economy. In this context, this study explores the conversion of WGP into 5-hydroxymethylfurfural (5-HMF), a versatile platform chemical with applications in biofuels, polymers, and fine chemicals [2].

The process combines deep eutectic solvents (DES) and microwave-assisted heating, leveraging green solvents and energy-efficient technology. Aqueous extraction at 55 °C effectively released soluble sugars (77.8 g/L of hexoses), which were subsequently transformed into 5-HMF using DES formulations based on betaine and organic acids. Among the tested systems, the betaine:malic acid mixture (1:2, 30% H₂O) allowed to achieve a yield of 30.9% higher than the 18.4% obtained with sulfuric acid as catalyst.

Using a centered full-factorial design, the reaction conditions (time and temperature) were optimized for 5-HMF production from WGP. The model predicted a maximum 5-HMF concentration of 9.60 g/L at 150 °C and 120 min, while the highest yield (30.39%) was achieved at 153 °C and 88 min.

These findings showcase a sustainable and scalable strategy for converting WGP into valuable chemicals, illustrating how green solvents, microwave technology, and experimental design can converge to advance eco-friendly biorefineries and circular economy initiatives.

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Desarrollo de un modelo matemático para predecir el POFF del biodiesel basado en el tipo de aceite usado en su producción

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La naturaleza del aceite empleado en la obtención del biodiesel, su composición en ácidos grasos y su origen determinan las propiedades del producto final. El biodiesel obtenido tiene un perfil específico de ésteres que depende del aceite utilizado para su producción que le confiere sus propiedades.

A nivel mundial, las materias primas lipídicas habituales para la producción de biodiesel son los aceites vegetales. La elección del aceite varía, convirtiéndose el lípido más abundante geográficamente en la materia más común para la producción de biodiesel; esto se debe a que en la elección de la materia prima no sólo se busca disminuir los costes, sino también garantizar un suministro suficiente de la materia prima para la producción. No existe una materia prima ideal para la producción de biodiesel que cumpla con todos los criterios de calidad y a su vez resulte una materia prima económicamente viable, la solución pasa por estudiar las mezclas óptimas de aceites que proporcionen un producto de calidad al menor coste posible [1]

En este trabajo se estudiaron, desde el laboratorio de una planta de producción, las propiedades del biodiesel, las mezclas de aceites a partir de las que es producido y la relación entre ambos [2], prestando especial interés al comportamiento en frío (POFF) [3]. El objetivo final: facilitar el diseño de proceso de producción del biodiesel en planta. Mediante la simulación en el laboratorio del proceso de transesterificación que se lleva a cabo en la planta de producción y el estudio de la correlación entre las características de la materia prima utilizada (aceites de soja, colza y palma) y las propiedades en frío del biodiesel obtenido como producto final [4] se ha obtenido con ayuda del programa estadístico Statgraphics Centurion, mediante el método clúster, un modelo matemático que permite predecir a partir de la composición de los ácidos grasos de los aceites utilizados en su obtención, el comportamiento en frío que presentará el biodiesel producido.

Se ha comprobado la fiabilidad del modelo propuesto.

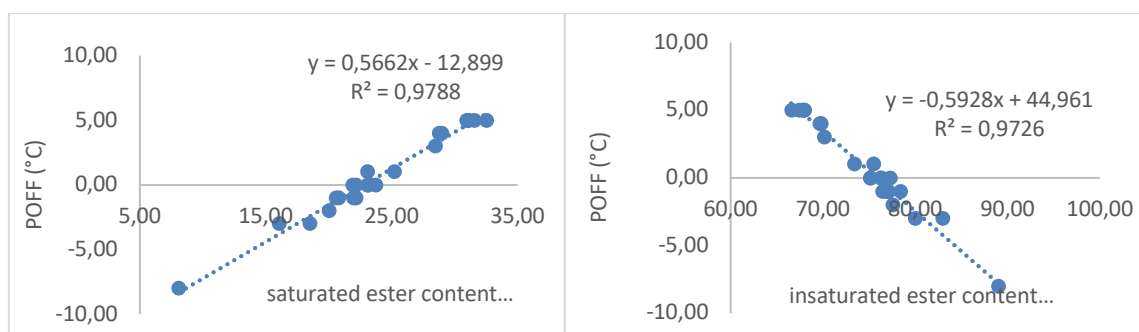


Fig.1. POFF / contenido en éster

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Predicción del número de cetano del gasóleo a partir del índice de cetano calculado y el contenido en mejorador de ignición usado en su formulación obtenido por FTIR

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El número de cetano, a través del que tenemos la capacidad de valorar la calidad de la ignición, es extremadamente importante en la formulación de combustible diésel; es una de las características del gasóleo relacionadas directamente con las prestaciones del motor. Normativamente, el número de cetano de un gasóleo se obtiene tras su análisis en un motor normalizado (ASTM D613/ISO 5165), pero desde siempre ha suscitado interés disponer de una metodología que no requiera la utilización de un motor de ensayo y permita predecir el número de cetano en función de las características propias del combustible [1].

En este trabajo se analizaron, a lo largo de 2024, 343 muestras de gasóleo comercial. A cada una de las muestras se le determinó, conforme a los requisitos establecidos en la EN 590 [2], el índice de cetano calculado (ASTM D4737/ISO 4264) y su contenido en mejorador de ignición diesel (DII) por FTIR. El objetivo: encontrar una correlación entre estas dos variables que permita predecir el número de cetano de forma que se ajuste al obtenido experimentalmente.

El estudio estadístico de los datos obtenidos permitió plantear una ecuación para el cálculo del número de cetano, con buena correlación, para valores de índice de cetano calculado de entre 47-55 y concentraciones del DDI de entre 0-500 ppm.

Se compararon los valores del número de cetano calculados mediante la ecuación propuesta con los valores obtenidos por FTIR obteniendo variaciones de entre el 0,0% y el 2,5 %.

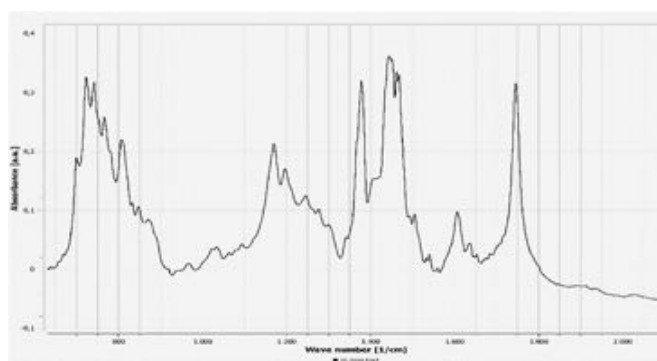


Fig.1. FTIR_GA

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Produción de compostos plataforma a partir de madeira de paulonia utilizando quecemento asistido por microondas e disolventes eutécticos profundos

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A valorización sostible da biomasa lignocelulósica constitúe unha das vías máis prometedoras para avanzar cara a unha bioeconomía circular, permitindo obter compostos de interese industrial a partir de recursos renovables [1]. Neste traballo avalíouse a produción de 5-hidroximetilfurfural (HMF) empregando madeira de *Paulownia elongata* × *fortunei* como materia prima, a través dunha estratexia baseada na aplicación combinada de disolventes eutécticos profundos (DES) e quecemento asistido por microondas.

O procedemento experimental desenvolveuse en varias etapas: (i) autohidrólise da madeira para solubilizar a fracción hemicelulósica, (ii) deslignificación con DES para obter un sólido enriquecido en celulosa, (iii) sacarificación enzimática para liberar glicosa, (iv) isomerización enzimática a frutosa, e (v) reacción de deshidratación asistida por microondas en presenza de DES como medio catalítico.

En total, avalíáronse cinco formulacións diferentes de DES, que foron empregadas na etapa final de deshidratación. As concentracións obtidas de HMF situáronse entre 6,7 e 11,03 g/L, dependendo da combinación de DES e das condicións de reacción aplicadas.

Estes resultados mostran o potencial do uso de DES como alternativa verde a catalizadores convencionais, ao tempo que evidencian a viabilidade da integración de microondas e DES en procesos de biorrefinaría de segunda xeración orientados á produción de compostos plataforma.

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Can data-driven AI optimize experimental conditions for biomass valorization into furfural?

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Artificial Intelligence (AI) is expected to transform the optimization of biorefinery processes by enabling the prediction of optimal reaction conditions from complex, multidimensional datasets. In particular, AI tools offer an approach to model solvent effects, reaction kinetics and yield responses in biomass valorization routes, helping to accelerate process development while minimizing experimental effort [1].

This study focuses on AI-guided optimization of furfural production, a key bio-based platform molecule, recognized for its role in the production of resins, solvents, plastics and fuel additives. Derived from the acid-catalyzed dehydration of pentoses in aqueous media, furfural is a pivotal intermediate in lignocellulosic biorefineries. Among the biomass constituents, hemicelluloses are especially rich in pentoses such as xylose, and represent a promising feedstock for furfural production. However, furfural production in acid-catalyzed aqueous systems is hampered by degradation pathways, in particular the formation of insoluble humins, which reduce furfural yields [2].

To mitigate these losses, organic solvents are commonly introduced in biphasic reaction systems, enabling the in situ extraction of furfural into the organic phase [2]. This separation protects furfural from rehydration or condensation reactions, resulting in improved yields.

In this work, hemicelluloses from gorse (*Ulex europaeus*) a fast-growing and invasive shrub, were used as a model substrate due to their high pentose content. A comparative evaluation of different systems constituted by mixtures of MIBK, GVL+ NaCl, 1-butanol, 1-pentanol or toluene is performed, assessing both, furfural yields and partitioning behavior [3].

Using experimental data and literature-derived datasets, AI-guided optimization enabled the identification of optimal reaction conditions tailored to each solvent systems, facilitating more efficient process development in biomass valorization [4].

This study underscores the importance of solvent selection and reaction conditions coupled with data-driven methodologies, for the sustainable high yield production of furfural from renewable resources.

Acknowledgements

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Efecto de la temperatura del aire en la eficiencia energética y exergética del secado durante el periodo antecrítico

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El secado es una operación crítica que representa hasta un 25% del consumo energético industrial, por lo que su optimización es clave [1,2]. Para ello se aplican balances de energía y exergía, siendo esta última la medida del trabajo útil disponible, usada para identificar irreversibilidades respecto a un estado de referencia, llamado “estado muerto” [2,3]. En este trabajo se analizó un proceso modelo de secado que transcurre exclusivamente en el periodo de velocidad constante (antecrítico), evaluando la eficiencia a distintas temperaturas y localizando las irreversibilidades del sistema.

El sistema analizado incluye como elementos básicos: un ventilador que impulsa el aire ambiente, un calentador eléctrico que eleva su temperatura y un secadero túnel donde ocurre la transferencia de agua del sólido al aire. Se evaluaron temperaturas de secado entre 50 y 100°C, modificando la velocidad de evaporación de agua con la temperatura y suponiéndola constante durante el secado. Se aplicaron balances de energía y exergía (ec. (1) y (2), respectivamente). A partir de estos, se puede obtener las eficiencias energéticas y exergéticas dadas por las ec. (3) y (4), respectivamente [1].

$$\dot{m}_{a,ent}h_{a,ent} + \dot{m}_{p,ent}h_{p,ent} + \dot{W}_{vent} + \dot{W}_{cal} + \dot{W}_{cin} = \dot{m}_{a,sal}h_{a,sal} + \dot{m}_{p,sal}h_{p,sal} + \dot{Q}_{vap} + \dot{Q}_{per} \quad (1)$$

$$\dot{Ex}_{a,ent} + \dot{Ex}_{p,ent} + \dot{W}_{vent} + \dot{W}_{cal} + \dot{W}_{cin} = \dot{Ex}_{a,sal} + \dot{Ex}_{p,sal} + \dot{Ex}_{vap} + \dot{Ex}_{per} + \dot{Ex}_{dest} \quad (2)$$

$$\eta_{en} = \frac{\dot{E}n_{vap}}{\dot{E}n_{ent}} \quad (3) \quad \eta_{ex} = \frac{\dot{Ex}_{sal}}{\dot{Ex}_{ent}} = 1 - \frac{\dot{Ex}_{dest}}{\dot{Ex}_{ent}} \quad (4)$$

Siendo $\dot{m}$ el flujo másico, h la entalpía específica, $\dot{Ex}$ el flujo exergético, $\dot{W}_{vent}$, $\dot{W}_{cal}$ y $\dot{W}_{cin}$ los trabajos del ventilador, calentador y cinta del secadero respectivamente. Los subíndices son a aire, p producto y o estado muerto ($T_o = 288,15$ K y $P_o = 101,3$ kPa). Además, ent y sal se refieren a entrada y salida; vap , $dest$, per son vaporización, destrucción y pérdida, respectivamente.

Las eficiencias energética y exergética aumentan con la temperatura de secado, Figura (a). Esto guarda relación con que la exergía destruida por kg de producto tiende a disminuir con la temperatura, generando un coste termo-económico inferior, Figura (b). Por último, destacar que el calentador es el equipo que presenta mayor irreversibilidad del proceso, siendo así, la prioridad en cuanto a la optimización del proceso.

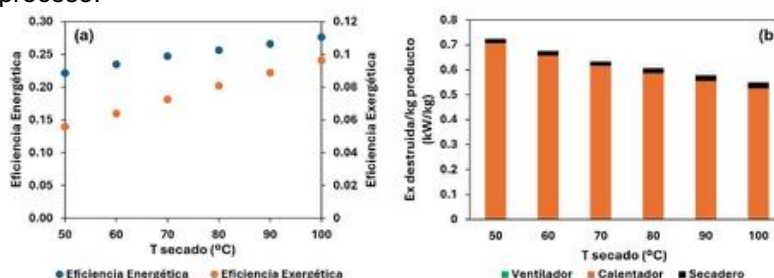


Figura. (a) Variación de la eficiencia energética y exergética global. (b) Desglose de la exergía destruida por kg de producto a cada temperatura.

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Chemical routes to HMF and levulinic acid from biomass: integrated enzymatic and acid-catalyzed mechanisms

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5-hydroxymethylfurfural (HMF) and levulinic acid (LA) are widely recognized as top-tier platform chemicals derived from biomass. Due to their versatility and wide application range, they are considered cornerstone compounds in lignocellulosic biorefineries. HMF is a key intermediate for the production of bio-based polymers and alternative fuels, while LA, obtained through the rehydration of HMF, is included in the US Department of Energy's list of "Top Value Added Chemicals from Biomass" with industrial uses as green solvents, biodegradable additives and specialty polymers [1].

Both "platform chemicals" can be produced from polysaccharides constituted by hexoses, such as cellulose, which is made up of glucose units. Under acid-catalyzed aqueous conditions, hexoses are dehydrated to HMF, which can be further rehydrated to form LA and formic acid. However, yields are typically below stoichiometric due to side reactions that lead to unwanted by-products such as humins. Among hexoses, fructose exhibits a more favourable kinetics for HMF and LA production, compared to glucose. Thus, glucose isomerization to fructose, using isomerase enzymes, is a strategic step to improve selectivity and yields [2].

In this study, birch wood was selected as lignocellulosic biomass, considering its high content of cellulose. Chips of birch were subjected to hydrothermal processing to solubilize hemicellulose and obtain a cellulose-rich solid. This solid was subjected to enzymatic hydrolysis using the commercial enzymatic complex Cellic CTec3, yielding glucose-rich solutions. Subsequently, glucose was isomerized to fructose and, under mild acidic conditions, converted to HMF and LA. The incorporation of the isomerization step allowed obtaining higher LA yields at lower temperatures and with shorter reaction times, offering a more selective alternative.

Acknowledgements

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Water and grease resistances of sustainable barrier coatings for paper using mix aqueous dispersion technology

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Plastics cause serious pollution because they persist in the environment for years contaminating all ecosystems. To avoid this growing problem, Single-Use Plastics Directive (SUPD) [1] from European Union actively promotes the replacement of plastic by sustainable and eco-friendly products. In response to increasing regulatory and social pressure to reduce single-use plastics, this work deals with the development of sustainable water-based barrier coatings for paper intended for food packaging.

New coatings are designed through the ACTEGA Artística Mix Aqueous Dispersions (MADs) technology (Figure) being possible to formulate high-solids water-based dispersions of elastomers and copolymers blends [2].

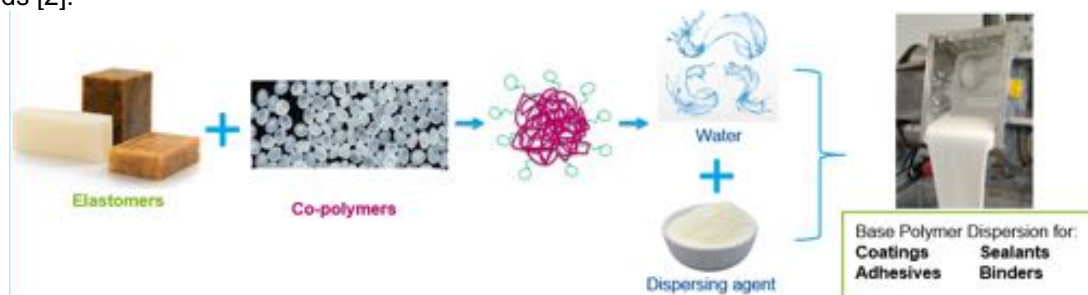


Figure. Mix Aqueous Dispersion Technology.

Dispersions were applied on paper to create an effective barrier coating resulting in sustainable packaging for food applications. Specifically, water and grease resistances were measured in coatings made up to 95% of non-fossil natural (liquid and solid) elastomers, renewable co-polymers such as PLA, additives and fillers.

Water resistance testing, by Cobb 1800 [3] and contact angle, showed a minimum dependence of the drying temperature (from 70 to 100°C) and coalescent concentration (from 0 to 12% w/w). Best results were obtained with formulations of liquid elastomer, Cobb 1800: 3 g/m² and contact angle up to 100°.

Grease resistance was evaluated by monitoring over time both staining and the passage through oil drops on the coated paper. In that case, coatings formulated with solid elastomers led to the best performance and can be improved by adding biowaxes and/or fermented co-polymers.

Therefore, sustainable coatings powered by MAD technology represent a sustainable alternative for the replacement of plastics in food packaging. Its versatility allows the design of tailored, eco-friendly coatings that meet both functional and regulatory requirements.

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Híbrido: motores combinados para máxima eficiencia

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Un motor híbrido es un sistema de propulsión que combina dos o más fuentes de energía diferentes para impulsar un vehículo, generalmente un motor de combustión interna y uno o más motores eléctricos. Este tipo de sistema se diseñó con el objetivo de aprovechar las ventajas de cada tecnología para maximizar la eficiencia energética y reducir las emisiones contaminantes.

En un motor híbrido, el motor de combustión interna puede funcionar con diferentes tipos de combustibles, como gasolina, diésel, etanol o gas natural. Por otro lado, el motor eléctrico se alimenta de una batería recargable que puede ser cargada mediante el frenado regenerativo o conectándola a una fuente de energía externa, como una toma de corriente eléctrica.

La operación de un motor híbrido puede variar según su diseño específico, pero generalmente se pueden distinguir varios modos de funcionamiento:

1. Modo de funcionamiento solo con el motor de combustión interna: El vehículo se impulsa únicamente utilizando el motor de combustión interna, mientras que el motor eléctrico permanece inactivo o en un estado de apoyo mínimo
2. Modo de funcionamiento solo con el motor eléctrico: El vehículo se impulsa únicamente utilizando el motor eléctrico, mientras que el motor de combustión interna permanece apagado.
3. Modo de funcionamiento combinado: Ambos motores, tanto el de combustión interna como el eléctrico, trabajan juntos para proporcionar potencia y propulsar el vehículo. Este modo suele utilizarse en situaciones de mayor demanda de potencia, como durante aceleraciones intensas o al circular a velocidades más altas.

La gestión inteligente de la energía es una característica clave en los sistemas híbridos, ya que el control electrónico decide de manera dinámica cuándo y cómo utilizar cada fuente de energía para maximizar la eficiencia y minimizar las emisiones. Esto puede incluir la selección automática del modo de funcionamiento más adecuado según las condiciones de conducción, la carga de la batería y las preferencias del conductor.

Mientras que los motores de combustión interna dependen de combustibles fósiles y producen emisiones contaminantes, los motores eléctricos son más limpios y eficientes al utilizar electricidad como fuente de energía. Los motores híbridos buscan combinar lo mejor de ambos mundos, ofreciendo una alternativa que reduce las emisiones y mejora la eficiencia sin sacrificar la conveniencia y el alcance de los vehículos de combustión interna.

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MEA-solvent regeneration improvement using acidified activated carbon during stripping

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The use of chemical solvents for the separation of acid gases - particularly those based on amines - is widely employed in industrial processes when the CO₂ or H₂S removal is required. However, this type of operation presents several drawbacks that implicate continuous improvements in order to remain competitive and aligned with emerging circular economy goals. Specifically, the regeneration step of the CO₂-rich solvent entails various disadvantages, among which the high energy requirement stands out. Several strategies have been evaluated to reduce this energy demand, including alternative stripping configurations, the use of different solvents or alternative separation technologies. One of the main contributors to this high energy consumption is the difficulty in reversing the chemical reaction between the amine (typically monoethanolamine, MEA) and CO₂ (which occurs in the absorber), often resulting in relatively low regeneration efficiencies.

This research work proposes enhancing the regeneration efficiency of the chemical solvent through the introduction of an activated carbon (purchased from Merck) that has been surface-functionalized via nitric acid treatment (1M HNO₃ during 3h at boiling temperature and under stirring). After saturation of the solvent with absorbed CO₂, the solvent was brought into contact with the acid-treated carbon at different temperatures. Afterwards, CO₂ absorption capacity was evaluated in a new absorption cycle in the presence of this modified activated carbon (MAC).

Figure 1 presents the experimental results of CO₂ absorption capacity after thermal treatment (at various temperatures) with and without the acidified activated carbon. A significant improvement in the absorption capacity was observed in the presence of the modified carbon at all tested temperatures. Specifically, enhancements of 29%, 25%, and 17% were achieved when the treatment was performed at 50, 70, and 90 °C, respectively. Additionally, the effect of the concentration of acid-treated carbon in the solvent was also investigated. The best results were obtained at a concentration of 10 g·L⁻¹. The conclusion that higher carbon concentrations (higher than 10 g·L⁻¹) lead to reduced performance could be attributed to a higher adsorption of amine molecules on the carbon surface, thereby reducing their availability in the solvent phase.

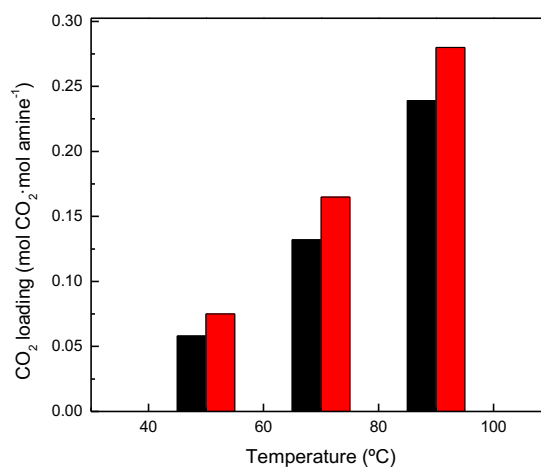


Fig. 1. Effect of temperature and MAC presence upon the carbon dioxide loading after regeneration. (■) without MAC, (■) with MAC (10 g·L⁻¹).

CO₂ adsorption in biomass-based activated carbons modified with nitric acid

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The use of activated carbons as adsorbents or catalyst supports has always been of significant importance in research within these fields. The high importance of this type of materials produced from biomass waste resources is related with their positive impact on the circular economy of the processes in which they are applied. One of the common drawbacks of this type of adsorbents is the difficulty in controlling their porosity, as well as in understanding the role of each pore type. This work aims to advance the understanding of the behavior of such adsorbents in CO₂ adsorption, for their subsequent application in selective separation processes of this gas when present in multicomponent gas streams.

The adsorbents were produced through an initial carbonization process at 600 °C under an inert nitrogen atmosphere of almond shell and olive stones wastes. Subsequently, activated materials were fabricated at 850 °C using two carbon:activating agent ratios (1:2 and 1:4 w/w), with KOH employed as the activating agent. This carbon was modified with 1M HNO₃ during 3h at 80°C under stirring. The produced materials were characterized in terms of both their porous structure and surface chemistry. Studies were conducted to obtain CO₂ and N₂ adsorption isotherms, with the objective of analyzing the behavior of the material in the presence of these gases. To reach this aim, alongside the experimental adsorption isotherms of each gas, the selectivity towards CO₂ adsorption was estimated using the Ideal Adsorbed Solution Theory (IAST), which takes into account the influence of N₂ molecules present in the gas phase on the CO₂ adsorption capacity. Figure 1 shows the CO₂ adsorption selectivity for the different materials using CO₂/N₂ mixtures of 15%/85% and 50%/50%. It can be observed that, in all cases, the most efficient separation (in terms of selectivity) is achieved with non-activated carbons, due to the high pore volume contributed by pores with diameters below 0.5 nm. Although the selectivity achieved by these non-activated materials is very high, it is also important to consider the adsorption capacity, which is clearly higher for the activated carbons (reaching twice the value of non-activated carbons) due to the significantly larger specific surface area. A low influence of biomass precursor was observed.

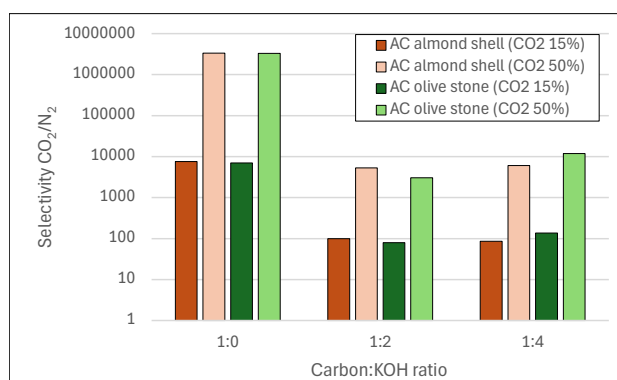


Fig.1. CO₂ absorption selectivity using the different biomass based activated carbons. T = 0 °C.

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3D Printed modular permeation cells for ultrafiltration

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Membrane separation processes, particularly ultrafiltration, have become increasingly relevant in fields such as wastewater treatment, drinking water purification, and the pharmaceutical, biotechnology, food, cosmetic, biofuel, and biomedical industries. Their advantages include low energy consumption, straightforward scale-up and, easy integration with other operation units [1, 2].

Despite this versatility, and given the diversity of applications, optimization of ultrafiltration systems for specific applications especially when dealing with limited sample volumes or complex fractionation tasks, remains a significant challenge. Traditional systems are often pre-built, limiting process flexibility and the ability to scale down for both research and industrial needs.

This work addresses these limitations by introducing a novel modular permeation cell designed using additive manufacturing. The 3D-printed ultrafiltration system provides high flexibility in experimental configuration, allowing researchers to adapt easily to different sample types and volumes. This is particularly important when concentrating bioactive compounds in small sample volumes, where large-scale ultrafiltration systems are not feasible. Moreover, applications involving compound fractionation may require multiple permeation cells equipped with membranes of distinct properties. Our presentation will demonstrate how additive manufacturing became a versatile approach to overcoming these challenges [3,4].

Acknowledgements

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Prolyl dehydropeptides: potential inhibitors of dipeptidyl peptidase 4?

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Dipeptidyl Peptidases (DPP) are Proline-Specific Peptidases that belong to the Serine Protease class S9. The enzyme DPP4 is bound to the cell membrane, as an ectoenzyme, in a variety of tissues (*e.g.*, gastrointestinal tract, pancreas, kidneys) or occurs as an extracellular soluble enzyme in some body fluids, *e.g.*, serum, saliva and seminal fluid. Changes in the level (overexpression) of DPP4 was associated to a variety of cancers (thyroid, ovarian, lung, skin, prostate), central nervous system tumors, lung metastasis, type 2 diabetes and celiac disease.

The enzyme DPP4 removes dipeptide units **Xbb-Pro** (Xbb - any a.a. except Pro) from the *N*-terminus of small peptides Xbb-Pro↓Xbb-(Xaa)_n (*n* = 2-12) (Xaa - canonical a.a.). DPP4 is involved in the proteolytic deactivation of incretin hormones, glucagon-like peptide 1, gastric inhibitory polypeptide and neuropeptides. Inhibitors of DPP4 were developed as drugs (Gliptins) for treatment of type 2 diabetes. DPP4-activatable (turn-on) fluorescence probes were developed as potential diagnostic tools for esophageal squamous cell carcinoma tumors. DPP4 expression in human serum, urine, thyroid cells and in mouse thyroid tumor models was reported using an IR probe.

In this poster we report the design, synthesis and preliminary characterization of molecular architectures containing dehydroamino acids and Pro as potential DPP4 inhibitors (Figure 1).

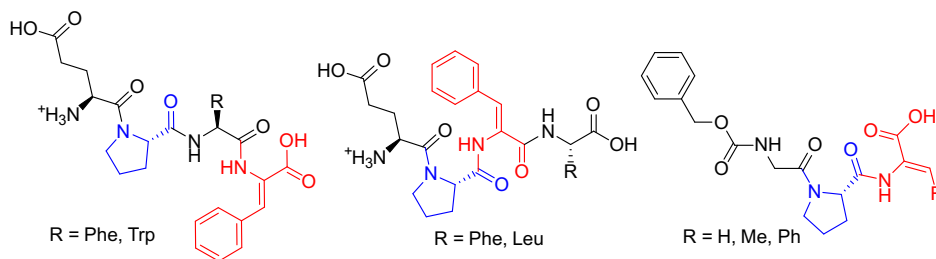


Fig. 1. Molecular architectures designed as potential DPP4 inhibitors.

The prolyl dehydropeptides were characterised by ¹H and ¹³C NMR spectroscopy and mass spectrometry. The biocompatibility of the library was evaluated using the MTT assay against the HaCaT keratinocyte and Caco-2 intestinal epithelial cell lines. The inhibitory properties of the prolyl dehydropeptides against DPP4 (ectoenzyme) expressed by the Caco-2 cell line was evaluated using a fluorogenic assay.

Proteolytic resistance allied to conformational constraints imposed by the dehydroamino acids makes the prolyl dehydropeptides interesting leads for development of DPP4 inhibitors.

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1,1-Disubstituted Ureas by Oxidation of Sulfonylurea Herbicides

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Sulfonylurea herbicides represent one of the most essential classes of agrochemicals due to their high efficiency and selectivity, low toxicity, and low application rates (10 – 50 g / ha) [1]. Understanding their degradation processes and the metabolites formed is essential to assess their environmental and biological impact [2].

In this work, we present a preliminary study on a new synthetic methodology for preparing a representative metabolite derived from sulfonylurea herbicides, a 1,1-disubstituted urea formed with the loss of the sulfonyl moiety. Flazasulfuron, a sulfonylurea herbicide widely used in the olive oil crop to control a wide range of grasses [3], was previously converted to rearranged urea, albeit with the formation of various side products, after long reaction times, and being limited to a single substrate [1]. We pretend to develop a general and straightforward protocol to obtain the 1,1-disubstituted ureas from their corresponding sulfonylureas.

The strategy involves the reaction of sulfonylureas with mild and environmentally friendly oxidants, resulting in the formation of 1,1-disubstituted ureas (Figure 1). Flazasulfuron was used as a substrate model under mild conditions, and LC-MS and ¹H NMR were used to analyze the reaction crude and obtain the reaction profile.

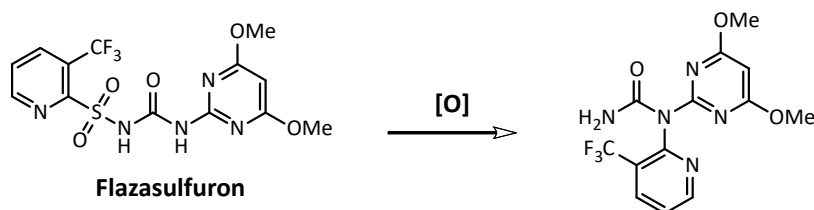


Fig.1. Synthesis of a 1,1-disubstituted urea from Flazasulfuron.

Acknowledgements

This work received support from PT national funds (FCT/MCTES, Fundação para a Ciência e Tecnologia and Ministério da Ciência, Tecnologia e Ensino Superior) through the projects UIDB/50006/2020, UIDP/50006/2020, and a PhD scholarship 2024.04089.BDANA.

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Synthesis of 5-aminopyrrolo[2,3-*d*]imidazoles: A new and efficient method

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Pyrrole is an important scaffold present in co-factors and other important natural products such as vitamin B12, bile pigments, chlorophyll, cytochrome, myoglobin and hemoglobin. In addition, several pyrrole-containing secondary metabolites isolated from natural sources have exhibited potential biological activity. Moreover, synthetic pyrroles have found diverse therapeutic applications like fungicides, antibiotics, anti-inflammatory drugs, cholesterol-reducing drugs, as well as anti-tubercular, antiviral, and antitumor agents [1]. On the other hand, the development of the imidazole-based compounds is in rapid expansion, not only because this heterocycle is found in several natural compounds (histamine, histidine, biotin, alkaloids, etc.), but as it is also part of multiple classes of approved drugs [2]. Thus, combining pyrrole and imidazole scaffolds offers additional binding potential to a variety of enzymes, proteins, and receptors.

As part of a research program aiming to develop new 2-aminopurine derivatives from imidazole precursors and cyanamide, a series of key intermediates (1) were obtained in very good yield. A comprehensive study on the reactivity of these intermediates has been previously accomplished, and it was found that they not only suffer an easy nucleophilic substitution of the cyano group, but they also undergo fast intramolecular cyclization reactions. In fact, a series of highly fluorescent 2-amino-6-cyanopurines were obtained by reacting compounds 1 in acid [3]. When NaOH was used, a different intramolecular cyclization reaction of 1 occurred, giving novel 5-aminopyrrolo[2,3-*d*]imidazoles in low-moderate yields [4]. Recently, the reactivity of compounds 1 in the presence of a diversity of bases was studied and a new and efficient method was developed to obtain 5-aminopyrrolo[2,3-*d*]imidazoles. The detailed experimental data will be presented and discussed.

Acknowledgements

The authors thank the Foundation for Science and Technology (FCT) for financial support through the Chemistry Center of the University of Minho (UID/00686) and the Physics Center of the Universities of Minho and Porto (UID/04650).

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Solid-phase synthesis and structure elucidation of linear tetrapeptides precursors of marine cyclic peptide analogues

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Over the years, the use and development of peptide-based drugs have been growing [1]. Peptides can be obtained from natural sources, both terrestrial and marine, with chemical synthesis being also a remarkable source. Marine cyclic peptides are very promising for the development of new therapeutic agents due to their unique structures, chemical properties, and interesting biological activities, such as antimicrobial [2]. Among them, unnarmicins A and C exhibited strong *in vitro* and *in vivo* antimicrobial activity with efficacy against fungal infections, notably those caused by multidrug-resistant [3].

This work describes the synthetic route of a series of linear tetrapeptides, analogues of the peptide chains of marine cyclic peptides unnarmicins A and C. The strategy used was solid-phase peptide synthesis (SPPS), following an orthogonal strategy, using 2-chlorotritylchloride (2-CTC) resin as solid support and 9-fluorenylmethyloxycarbonyl (Fmoc) as protecting group (Figure 1). Repeated cycles of coupling, deprotection, and washing were carried out until the peptide sequence was fully assembled, affording the desired peptides with good yields (57% - 87%). The structure of the synthesized tetrapeptides was established by IR, NMR and MS spectral analysis.

The synthesized linear peptides will be further explored as bioactive compounds and as precursors for new marine cyclic peptide analogues.

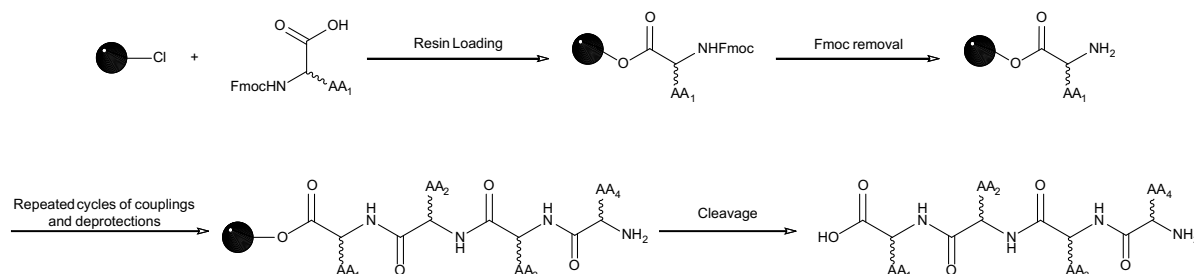


Fig.1. General scheme of solid-phase peptide synthesis of tetrapeptides.

Acknowledgments

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Continuous flow synthesis of di-imidazolyl porphyrin

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Tetrapyrrolic macrocyclic compounds are planar aromatic molecules with remarkable physicochemical properties, such as the ability to form stable metal complexes and to act as photosensitizers, particularly in photodynamic therapy (PDT). Several clinically relevant examples, including Foscan [1], and Redaporfin [2], highlight their biomedical potential. However, traditional synthetic routes, such as the Lindsey method [3], suffer from major limitations, namely the need for very dilute conditions that result in excessive solvent consumption (e.g., 20 L of DCM for 1 g of porphyrin) [4]. Continuous flow chemistry offers a promising alternative for large-scale synthesis, enabling safer operations, more efficient mass and heat transfer, improved yields and reproducibility [5].

In this communication, we present our results regarding the transposition of the synthesis of 5,15-*bis*-(1-methyl-1H-imidazol-2-yl)porphyrin following the Lindsey's method, from batch to continuous flow processing, optimizing the reaction conditions for both cyclization and oxidation synthetic steps regarding temperature and residence time. Under optimal conditions, we were able to obtain up to 22% yield for the desired 5-15- imidazolyl porphyrin, corresponding to a productivity of 0,238 mmol/day or 0,112 g/day using a 11.5 mL sequential reactor setup (Fig. 1). This result represents a significant improvement in reaction time and solvent consumption relative to the standard Lindsey procedure. Optimization procedures and isolation process will be presented and discussed.

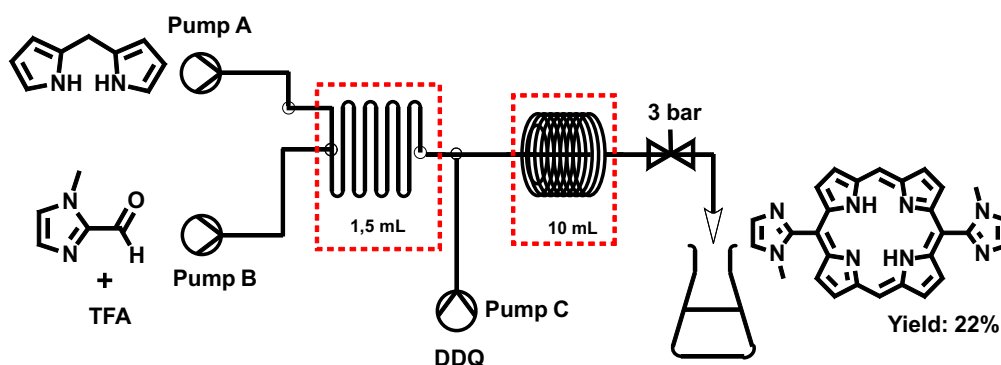


Fig.1. Continuous flow setup for the synthesis of 5,15-*bis*-(1-methyl-1H-imidazol-2-yl)porphyrin.

Acknowledgements:

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Synthesis of Chitosan Derivatives: From Classical to Innovative Strategies

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Chitosan, a linear polysaccharide obtained from the deacetylation of chitin, possesses reactive amino and hydroxyl groups that allow chemical modifications, serving as a platform for the development of advanced materials. Among the available approaches, regioselective and quantitative functionalization stands out for enabling the production of well-characterized derivatives [1].

Schiff base reactions, which rely on the condensation of aldehydes or ketones with primary amines to yield imines, represent a straightforward and efficient synthetic approach due to their versatility and mild conditions [2]. Moreover, *click* chemistry is an efficient, selective, and high-yielding method for the modular synthesis of complex polymers under mild conditions, paving the way for new compounds with promising applications [3].

The aim of this work is to obtain promising chitosan derivatives using both synthetic strategies. The synthesis of chitosan derivatives was performed via Schiff base reactions or *click* chemistry, two synthetic strategies that provide distinct structural and functional possibilities. Two chitosan–xanthone conjugates were obtained through multistep synthetic procedures employing Schiff base reactions. Through *click* chemistry, another chitosan derivative was obtained involving four reactions steps: protection of chitosan amino groups, substitution of OH by an electron-withdrawing group and then by an azide group, and finally the coupling reaction with a suitable functionalized xanthone. Additionally, the total synthesis of some xanthone derivatives were also carried out, via benzophenone intermediate, and through diverse synthetic strategies. All structures were confirmed by spectroscopic methods.

The synthesis, purification, and structural analysis were successfully achieved. It was found that both strategies yielded promising results in the synthesis of the target compounds.

Acknowledgements

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Screening of Catechol-rich Polyphenols for Dynamic Complexation with a Boronic Acid-Chalcone Receptor

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Boronate–catechol interactions have emerged as a versatile tool in molecular recognition, sensing, and materials design for drug delivery, yet their application potential towards complex natural polyphenolic-based systems remains underexplored.

In this work, we screened the binding behavior between a synthesized boronic acid–containing chalcone receptor and commercially available catechol-rich polyphenols such as Quercetin, Catechins, Protocatechuic Acid, among others.

Through systematic spectroscopic studies (UV–Vis, fluorescence, and Boron Nuclear Magnetic Resonance), we demonstrate that the synthesized dye has the ability to undergo spectral and chemical shift variations upon complexation with catechol-containing polyphenols in a pH-regulated manner.

This work provides valuable insights into the binding affinity of structurally diverse catechol moieties within the polyphenol framework, offering a deeper understanding of boronate-catechol complexation. This study will allow the development of novel colorimetric and fluorometric sensors for polyphenolic compounds towards food and environmental applications [1,2].

Acknowledgments

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Synthesis of imidazole-based hydrazonoyl cyanides as new photoswitches: mechanistic studies

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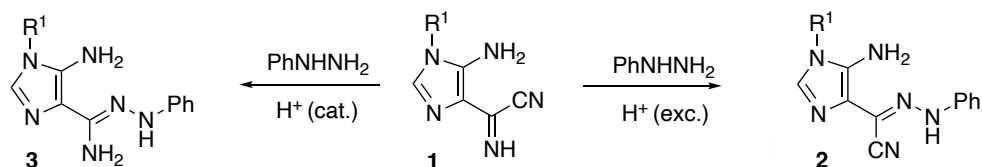
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Molecular photoswitches play a significant role in various areas of modern science and technology and their huge potential in the development of smart materials ranges from nanotechnology to pharmacology [1]. Imidazole nucleus is an attractive scaffold in the heterocyclic synthesis and drug discovery, because it is a key component in many bioactive molecules [2,3]. Aryl hydrazonoyl cyanides are important starting reagents for the preparation of a wide variety of heterocyclic compounds that are uniquely substituted [4]. They also exhibit many biological activities namely as antimicrobial, anticancer, anti-inflammatory and analgesic. However, limited methods can be found in the literature for the synthesis of the highly functionalized hydrazonoyl cyanides.

In our research group, studies on the condensation of 4-cyanoformimidoyl 5-aminoimidazole precursors (**1**) with aryl hydrazines in acidic medium showed that this reaction generated imidazole-based hydrazonoyl cyanides (**2**) and 5-aminoimidazole 4-carboxamidrazones (**3**) (Scheme 1). A series of new 4-hydrazonoyl cyanides **2** incorporating diverse aromatic hydrazonoyl moieties was obtained, but the products were only isolated in moderate yields. The photoinduced structural changes have been studied and the obtained results revealed that these hydrazonoyl cyanides are photoswitchable compounds. Given this interesting photoswitching behavior, the synthetic method needs optimization to increase the reactions yield. In this sense, the mechanisms underlying the selective preparation of these two significant classes of imidazole derivatives are being studied using experimental and theoretical assays. The experimental and *in silico* data obtained in these studies will be presented and discussed.



Scheme 1. Competitive synthetic pathways to obtain imidazole-based hydrazonoyl cyanides (**2**) and 5-aminoimidazole 4-carboxamidrazones (**3**).

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Derivados do ácido cinâmico modulam o comportamento ansiolítico semelhante em zebrafish adulto (*Danio rerio*).

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O uso contínuo de ansiolíticos pode estar associado a desafios, como dependência [1] e potenciais efeitos colaterais relacionados com a perda de memória [2]. Os produtos naturais constituem uma importante fonte de novos fármacos para o tratamento de diferentes patologias, incluindo a depressão [3]. Dentro dessa perspectiva, o ácido cinâmico é um polifenol derivado do ácido fenólico e um dos metabólitos secundários vegetais mais abundantes. Este estudo investigou os efeitos ansiolíticos do ácido cinâmico em zebrafish (*Danio rerio*) adulto. O composto foi administrado nas doses de 4, 20 e 40 mg/kg e não apresentou toxicidade após 96 horas de monitoramento. Em experimentos comportamentais que avaliam a locomoção, foi observado que a dose de 40 mg/kg do ácido cinâmico, apesar de comprometerem as funções motoras dos animais, demonstraram respostas melhores que o fármaco de referência. O composto apresentou comportamento ansiolítico realizado em teste claro e escuro, constatado através do aumento da permanência na zona clara do aquário, onde a menor dose (4mg/kg) apresentou o melhor efeito ansiolítico se comparada as demais doses. No estudo do mecanismo de ação desta molécula, observa-se um potencial bloqueado por pelo pizotifeno: 5-HT_{1A}/5-HT_{2A/2C}, isso sugere que o efeito ansiolítico pode envolver o sistema serotoninérgico.

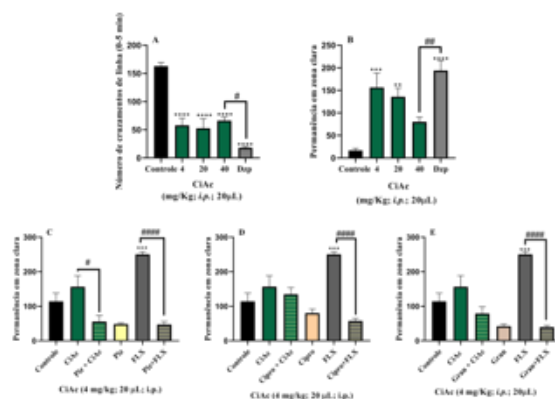


Fig.1. (A) Influência nos parâmetros de locomoção, (B) Efeito ansiolítico do ácido cinâmico, (C) Efeito do pizotifeno, (D) ciproheptadina e do (E) granisetron no mecanismo de ação ansiolítica.

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Synthesis and Photophysical Properties of Push-Pull Tetraphenylethylene–Dithienopyrrole Derivatives

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Push-pull heterocyclic molecules exhibit unique photophysical behaviour resulting from their distinct donor-acceptor architecture, which facilitates efficient intermolecular charge transfer upon photoexcitation. This process leads to pronounced bathochromic shifts in both absorption and emission bands. The ability to systematically modulate electronic properties by fine-tuning donor, acceptor and π -spacer moieties empowers molecular design and targeted applications. Such tunability directly influences key properties including optical transition energies, dipole moments and charge transfer processes, making heterocyclic push-pull molecules attractive for advanced optoelectronic devices, nonlinear optical materials and energy conversion technologies [1-2].

The incorporation of dithienopyrrole (DTP) heterocyclic system, as the electron donor in push-pull derivatives is particularly impactful, as its rigid tricyclic conjugation improves intramolecular charge transfer and boosts electronic delocalization [3-4]. Combining DTP with tetraphenylethylene donor groups and various electron-accepting moieties, including aldehyde, cyanoacetic acid, rhodanine, and thiobarbituric acid, results in molecules with tunable photophysical properties. To explore the concepts mentioned above, a comprehensive photophysical study of these newly synthesized derivatives was undertaken (Fig 1).

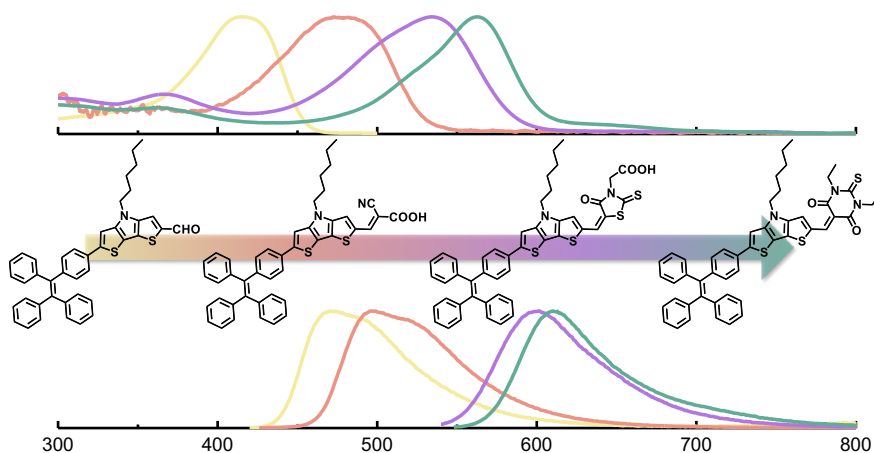


Fig.1. Absorption and emission spectra of the synthesized push-pull heterocyclic derivatives in THF solutions.

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Síntese e caracterização fotofísica de 2,4,5-triaminopirimidina-6-carboxilato para ensaios de viabilidade celular

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As sondas fluorescentes têm sido vastamente utilizadas para monitorização de viabilidade celular. De entre as sondas fluorescentes, destacam-se os análogos de nucleobases fluorescentes (ANFs) [1,2], que surgem como uma alternativa viável para a marcação de DNA. Entre os diferentes ANFs, salientam-se as pirimidinas [3], que são especialmente utilizadas devido ao seu tamanho reduzido e devido à sua ampla distribuição em produtos naturais e materiais funcionais. Contudo, os ANFs são raramente utilizados para ensaios de viabilidade celular, devido às suas propriedades fotofísicas (emissão na região do UV e desvios de Stokes pouco significativos). Para combater esta limitação, têm sido desenvolvidos métodos de síntese de novas pirimidinas [4], com propriedades fluorescentes que as tornem sensíveis a variações no ambiente celular [5].

Em trabalhos anteriores do grupo de investigação, foram obtidas 2,4,5-triamino-6-cianopirimidinas com excelentes propriedades fotofísicas, que se apresentaram muito promissoras como marcadores de viabilidade celular [3]. Recentemente, foi sintetizado um novo conjunto de pirimidinas análogas (2,4,5-triaminopirimidina-6-carboxilato) que apresentam propriedades fotofísicas melhoradas, uma vez que apresentam emissão de fluorescência na zona do verde (500 nm). Nesta comunicação, será apresentada a síntese das 2-amino-pirimidinas-6-carboxilato, bem como a análise das suas propriedades fotofísicas.

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2-Amino-purinas-6-carboxilato como análogos de nucleobases fluorescentes: síntese, caracterização e propriedades fotofísicas

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A fluorescência é, nos dias de hoje, uma técnica versátil, rápida e sensível para a monitorização de interações químicas entre biomoléculas [1]. Um dos grandes problemas na marcação fluorescente de DNA reside no tamanho das moléculas tradicionalmente utilizadas, o que reduz a seletividade de ligação ao alvo. Uma alternativa que tem vindo a ser explorada envolve o uso de análogos de nucleobases fluorescentes (ANFs). No entanto, os ANFs encontrados na literatura apresentam espectros de absorção e emissão na região do UV. Devido a esta limitação, existe uma crescente necessidade de novos ANFs com propriedades fotofísicas melhoradas. Por outro lado, para permitir a deteção de mudanças em ambientes celulares, estas moléculas devem apresentar uma emissão de fluorescência sensível a essas variações [2]. No entanto, modificações que tornam as nucleobases emissivas são difíceis de realizar e, por isso, a sua síntese permanece um desafio.

Em trabalhos anteriores do grupo de investigação, foram obtidas 2-amino-6-cianopurinas [3] e 2-amino-purinas-6-carbimidato através de métodos simples e eficientes. Estas duas novas classes de ANFs exibiram espectros de emissão na zona visível (com máximo de emissão entre 430 e 450 nm), rendimentos quânticos de fluorescência e desvios de Stokes elevados, e resultados promissores na bioimagem de células. Recentemente, foi sintetizada uma série de novos ANFs com a estrutura de 2-aminopurina-6-carboxilato e a sua caracterização fotofísica revelou propriedades melhoradas em relação às duas classes anteriores. Uma vez que estes compostos emitem na zona do azul-verde (450-500 nm), são muito promissores como sondas para ensaios de bioimagem.

Os resultados obtidos na síntese desta nova classe de ANFs, bem como as suas propriedades fotofísicas, serão apresentados neste trabalho.

Agradecimentos

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New 2,4-aminoimidazoles as potential optical amine sensors

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Chemical sensors are devices used to detect the presence of an analyte in a sample, as well as provide information about its concentration. Amine sensors, in particular, have applications in the food industry by helping to assess the safety and freshness of food, in biomedical fields as tools for both imaging and diagnosis, and in environmental monitoring by detecting air contamination [1]. Due to their colorimetric properties, organic dyes have been often used as optical sensors for the detection of biogenic amines found in food spoilage [2].

Previous work by the group reported a novel route for the synthesis of 2-aminoimidazole azo dyes (**3**) from 5-aminoimidazole-4-carboxamidrazones (**1**), presenting unique substituents pattern in the imidazole unit, vibrant colours and antimicrobial activity [3]. Recent studies on the reactivity of these azo dyes with various nucleophiles showed that they suffer fast addition of amines, resulting in the formation of new colourless 2,4-aminoimidazoles **4** (Fig. 1).

As such, this reaction is under investigation considering the potential use of the 2-aminoimidazole azo dyes **2** as optical amine sensors for the development of new smart materials. The results obtained in the synthesis and characterization of 2,4-aminoimidazoles **4** will be presented and discussed.

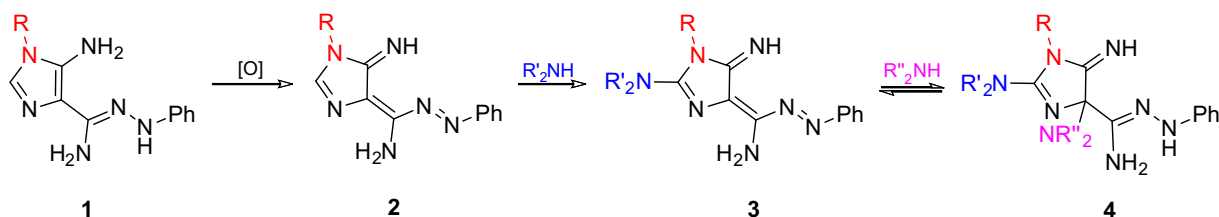


Fig. 1. Synthesis of new 2,4-aminoazoimidazoles **4** from amidrazones precursors **1**.

Acknowledgements

The authors acknowledge the Foundation for Science and Technology (FCT) for the financial support through the Chemical Research Centre of the University of Minho (UID/QUI/00686).

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Expanding on a new class of red-shifted and antimicrobial imidazole-based azo dyes

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Azo dyes are the most used colorants in industry due to their wide range of colours, luminosity, simplicity of manufacture and good dyeing performance. They have a myriad of applications in various fields, including photopharmacology, nonlinear optics and as chemosensors [1]. The addition of heterocyclic moieties to azo dyes can confer them enhanced colouring abilities, as well as improved pharmacological properties, resulting in a broader range of potential applications [2]. Particularly, the addition of the imidazole scaffold has shown to increase antimicrobial activity [3].

Previous work by the group resulted in eighteen compounds of a new class of green-coloured 2-aminoimidazole dyes, which proves a significant red-shift in their absorption spectrum [4]. These new dyes also presented solvatochromic and halochromic properties, changing colour from green to purple in acidic medium, as well as potent antimicrobial activity, mainly against *C. neoformans*.

The main synthetic route to obtain azo-dyes consists in coupling of diazonium salts with electron-rich nucleophile segments. However, the synthetic pathway to obtain 2-aminoimidazoles **3** relied on a domino reaction between secondary amines and previously reported 5-aminoimidazole-4-carboxamidrazones **1** (Fig. 1). This method warranted good to excellent yields, in part due to the use of a O₂/KI oxidant system, which allowed for regeneration of the oxidant and minimization of side reactions. Despite the significant number of compounds obtained, all of them presented aryl groups in the N-1 position.

Therefore, in this communication, new compounds obtained using the same environmentally friendly method and optimized reaction conditions will be presented. More specifically, compounds containing alkyl groups in the N-1 position.

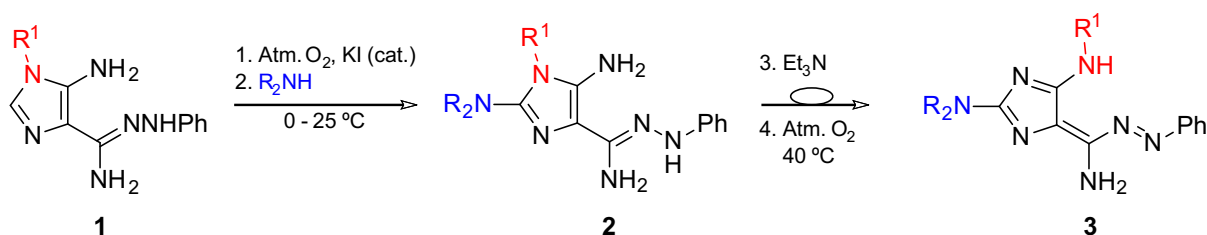


Fig. 1. Synthesis of halochromic & antimicrobial 2-aminoimidazole azo dyes **3** from amidrazones precursors **1** [4].

Acknowledgements

The authors acknowledge the Foundation for Science and Technology (FCT) for the financial support through the Chemical Research Centre of the University of Minho (UID/QUI/00686), and the project MEDCOR (PTDC/CTM-TEX/1213).

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Investigation of the Antinociceptive and Anti-inflammatory Pharmacological Potential of Hydrazones in Zebrafish

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Inflammation is an essential biological response against pathogens and tissue damage, characterized by signs such as pain, redness, heat, and edema. This process involves the activation of the nociceptive system and immune cells. In this context, conventional therapy is based on drugs capable of inhibiting the excessive production of inflammatory mediators or modulating the recruitment and activation of immune cells involved in the process [1]. Hydrazones are organic compounds that exhibit anti-inflammatory and analgesic properties, and to discover new drugs, animal models such as zebrafish (*Danio rerio*) have been used due to their genetic homology with humans [2]. Thus, this study was conducted to investigate the anti-inflammatory and antinociceptive effects of hydrazones in adult zebrafish models, as well as to explore their mechanism of action and toxicity [1,3]. The tested doses of hydrazone (HDZI 2OH, 4, 20, and 40 mg/kg) did not show toxic effects on zebrafish during 96 hours of analysis ($LD_{50} > 40$ mg/kg). The compound demonstrated promising pharmacological effects, preventing formalin-induced nociceptive behavior. It is noteworthy that the analgesic effect of HDZI 2OH was blocked by camphor, indicating the involvement of TRPA1 channel neuromodulation in zebrafish. Regarding anti-inflammatory activity, carrageenan (1.5%)-induced edema was reduced by the 4 and 40 mg/kg doses of HDZI 2OH after the fourth hour of analysis, showing an effect similar to ibuprofen. HDZI 2OH exhibited a safe profile, an antinociceptive effect mediated by TRPA1, and anti-inflammatory action comparable to ibuprofen, highlighting its relevance as a promising candidate for the development of new drugs. However, further specific studies are necessary to complement these findings.

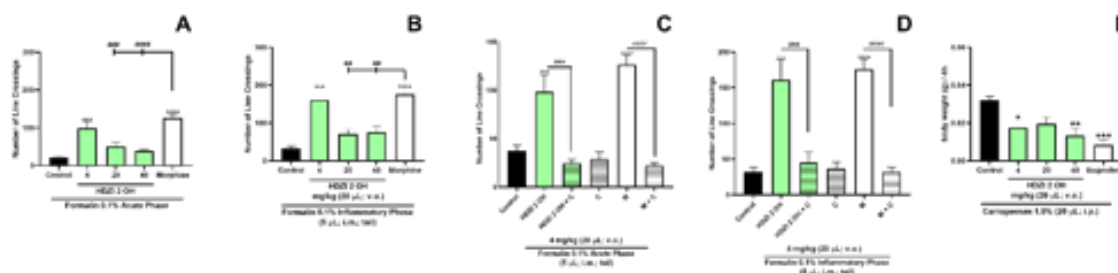


Fig.1. Results obtained in the study with hydrazones. Effect of HDZI 2OH in the acute phase (A) and inflammatory phase (B) of formalin-induced nociception. Effect of HDZI 2OH in the acute phase (C) and inflammatory phase (D) on the mechanism of action via the TRPA1 channel. (E) Effect of HDZI 2OH on carrageenan-induced inflammation.

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Síntesis y aplicación de nuevos *N*-sesquiterpen-aril derivados con actividad antioxidante

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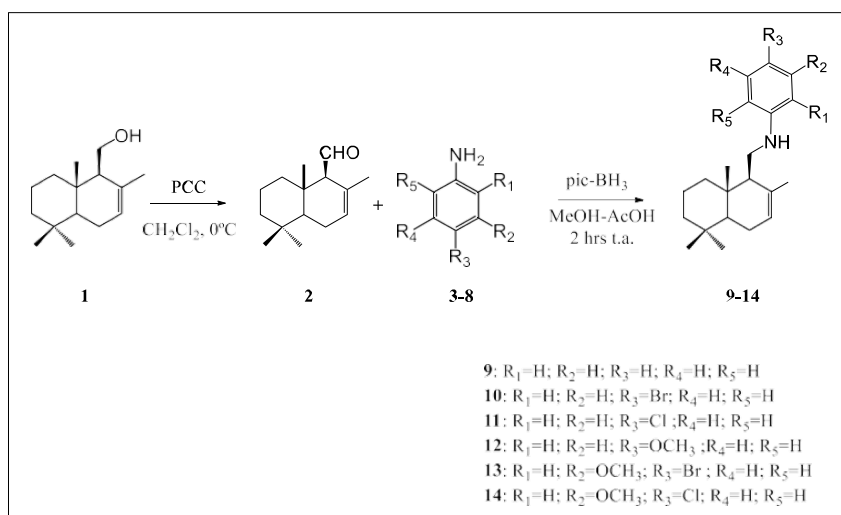
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Cuando hay un exceso de radicales libres y no suficientes antioxidantes para neutralizarlos en el cuerpo, puede ocurrir un estrés oxidativo, que está relacionado con el envejecimiento y con enfermedades como el cáncer [1]. Actualmente existen distintos productos antioxidantes naturales o sintéticos con distintas limitaciones como baja solubilidad y biodisponibilidad [2]. Debido a lo anterior, es de gran interés desarrollar nuevos compuestos que posean alta actividad antioxidante. Una alternativa a lo anterior fue sintetizar una serie de seis nuevos derivados utilizando reacciones de oxidación a partir de drimenol (**1**), compuesto natural que se extrae de la corteza del canelo. Posteriormente el producto obtenido (**2**) se sometió a una reacción de aminación reductiva con variadas anilinas (**3-8**) (Esquema 1), obteniendo los productos finales (**9-14**) con rendimientos entre 50-85%. Luego, mediante el método de ABTS se determinó la capacidad antioxidante total de cada derivado sintético, siendo el compuesto **12** el que obtuvo mejor resultado antioxidante superando los compuestos referentes BTH y ácido galico. A partir de los resultados presentados es posible afirmar que el compuesto **12** podría tener alta bioactividad frente a distintas patologías.



Esquema 1. Reacción de oxidación y aminación reductiva

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Synthesis and characterization of “push-pull” D-A- π -A benzothiadiazoles for potential chemosensing and bioimaging applications

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In recent years, “push-pull” donor- π -acceptor benzothiadiazole molecules have recently attracted significant interest due to their versatile optical and electronic properties. These benzothiadiazole derivatives have been widely used in different applications, including organic solar cells, light-emitting diodes (LEDs), optical chemosensing and bioimaging due to their easy functionalization, remarkable photostability, and biocompatibility [1-5].

In the context of drug and probe design, tailoring the side-chain length is a crucial strategy to modulate cellular uptake and membrane permeability. In many cases, dyes with short side chains struggle to penetrate living cells, whereas those with longer chains tend to become trapped within the membrane. Thus, achieving an optimal side-chain length is crucial to enhance cellular permeability and, consequently, the bioimaging potential of the dye [6,7].

Here we present the synthesis, structural and photophysical characterisation of two benzothiadiazole derivatives (**1** and **2**) with a D-A- π -A structure (Figure 1). Compound **2** presents a longer side-chain which could be beneficial for future cellular uptake studies. These compounds were characterized by NMR techniques (proton and carbon), FTIR and mass spectroscopy. Photophysical characterization was performed in DMSO for both compounds. The results obtained highlighted their potential applications as bioimaging probes and/or optical chemosensors.

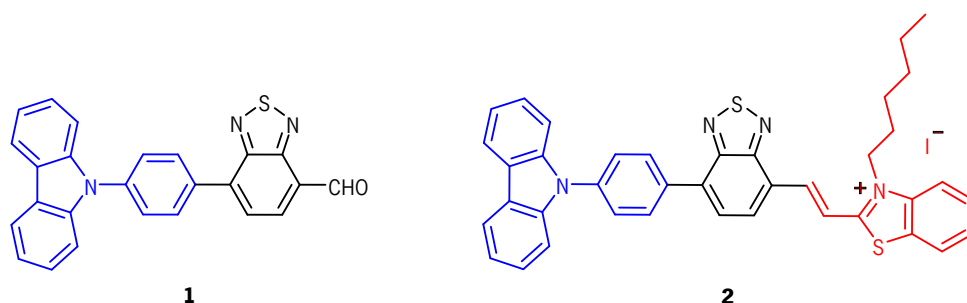


Fig.1. Structures of benzothiadiazole derivatives **1** and **2**.

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Síntese e avaliação preliminar da atividade antibacteriana de novas sulfonamidas

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A introdução de agentes antibacterianos na prática clínica teve um grande impacto na prestação de cuidados médicos, aumentando a esperança e a qualidade de vida. A eficácia dos antibióticos é, no entanto, afetada pela capacidade que as bactérias têm de se oporem à sua ação. Este fenómeno, genericamente denominado de resistência aos antimicrobianos (RAM), é atualmente um dos maiores e emergentes problemas de saúde pública.

Uma estratégia para mitigar o impacto da RAM envolve a descoberta de novos antibióticos através da modificação química das classes existentes, com o objetivo de dotar o fármaco com a capacidade de evasão aos mecanismos de resistência bacteriana, por exemplo, através de novos modos de ação.

As sulfonamidas foram dos primeiros antimicrobianos a serem utilizados na prática clínica (Figura 1). e são uma classe de fármacos bacteriostáticos, de amplo espectro de atividade, eficazes em bactérias Gram-positivas e Gram-negativas. No entanto, a sua utilidade terapêutica encontra-se atualmente limitada devido não só ao desenvolvimento de estirpes resistentes, mas também devido a questões de toxicidade inerentes a este grupo de compostos.

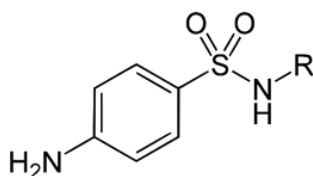


Fig.1. Estrutura geral das sulfonamidas.

Este trabalho tem como base a revitalização da atividade antibacteriana das sulfonamidas através da conjugação destas com sideróforos ou seus miméticos, promovendo a inclusão dos conjugados sulfonamida-sideróforo no citoplasma da bactéria, através de recetores específicos de sideróforos [2]. Os resultados obtidos serão apresentados nesta comunicação.

Agradecimentos

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Avaliação da atividade antibacteriana de novos derivados da ciprofloxacina

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Os sistemas de saúde dependem fortemente de antibióticos para tratar infeções bacterianas, mas o aumento da resistência antimicrobiana coloca a sua eficácia em perigo. Apesar de nas últimas décadas não terem sido introduzidas no mercado novas classes de antibióticos, têm sido propostas e desenvolvidas novas estratégias e abordagens inovadoras, que representam um sinal de esperança no combate à resistência aos antimicrobianos.

Estas estratégias têm incluído, entre outras, a pesquisa de novos alvos terapêuticos, a adoção do conceito *multi-target*, a aplicação de bacteriófagos e a inibição dos mecanismos de resistência microbiana. Neste último caso, uma das possibilidades envolve a utilização da estratégia frequentemente designada de “Cavalo de Tróia” em que são usados compostos (sideróforos) para facilitar a entrada do antibiótico na célula bacteriana e assim contornar um dos mecanismos de resistência a antibióticos mais frequentes, nomeadamente a permeabilidade membranar reduzida [1].

Este mecanismo de resistência é amplamente associado à perda de eficácia das fluoroquinolonas, uma classe de antibióticos bactericidas de largo espectro amplamente utilizados na prática clínica. O alvo terapêutico destes fármacos é nas bactérias gram-negativas a DNA girase, enquanto nas gram-positivas, é a topoisomerase IV, ambas localizadas no citoplasma bacteriano. Para exercer a sua ação, as fluoroquinolonas precisam atravessar a membrana bacteriana, e uma permeabilidade reduzida destes fármacos reduz e ou inativa a eficácia destes antibióticos [2].

Este trabalho utiliza a ciprofloxacina (Figura 1), uma fluoroquinolona de segunda geração, como ponto de partida para a obtenção de novos conjugados ciprofloxacina-sideróforo, com o objetivo principal de avaliar a sua atividade antibacteriana contribuindo para o desenvolvimento de novos agentes antimicrobianos mais eficazes face à crescente resistência antimicrobiana. Os resultados obtidos serão apresentados nesta comunicação.

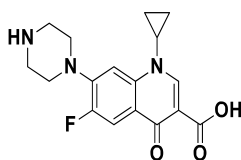


Fig.1. Estrutura química da ciprofloxacina.

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Isoflavone FPR Antagonists: Synthesis, Structure, and Spectroscopic Characterisation

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Isoflavones are naturally occurring compounds mainly found in soybeans, soy foods and vegetables, well-known for their ability to act as phytoestrogens [1,2]. Nevertheless, other health benefits have been attributed to this class of compounds, namely, their beneficial role in cancer, such as hormone-dependent cancers, in osteoporosis, cardiovascular disease and inflammation, and several mechanisms have since been proposed to explain these activities [3]. Interestingly, a series of synthetic isoflavones was recently reported as potent and competitive antagonists of formyl peptide receptors (FPRs), having an important role in the regulation of inflammatory reactions implicated in disease pathogenesis [4]. Thus, the isoflavone backbone represents a promising scaffold for the development of novel FPR antagonists.

As a result of their biological activity, there is considerable interest in developing synthetic procedures to obtain isoflavones and their derivatives for structure-activity relationship studies. Surprisingly, and to the best of our knowledge, the synthetic work and full characterisation of the synthetic isoflavones described as FPR antagonists have not yet been reported (Figure 1).

Accordingly, the work described herein comprises the synthesis of two series of 2-trifluoromethyl isoflavones, including those described as FPR antagonists, and their complete characterisation by 1D and 2D NMR techniques and high-resolution mass spectroscopy.

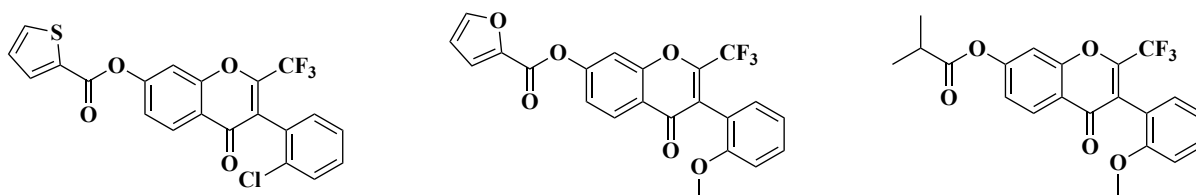


Fig. 1: Synthetic isoflavones described as FPR antagonists.

Acknowledgments

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Design, Synthesis, and Cytotoxic Profile of Melanostatin Derivatives Targeting Parkinson's Disease

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In Parkinson's disease (PD), the progressive degeneration of dopaminergic neurons in the substantia nigra leads to a severe dopamine deficit, resulting in hallmark motor symptoms such as tremors, rigidity, and bradykinesia [1,2]. While levodopa administration remains the gold standard treatment for PD, its long-term use often leads to motor disabilities, including dyskinesias and fluctuations in symptom control, underscoring the need for alternative therapeutic strategies [1,2]. Melanostatin (MIF-1), an endogenous neuropeptide with the structure Pro-Leu-Gly-NH₂, has demonstrated modulatory effects on dopaminergic neurotransmission through positive allosteric modulation of D₂ receptors [3,4]. This mechanism may enhance dopaminergic signaling in early stages of PD and potentiate the response to levodopa, potentially allowing for dose reduction [3,4]. Despite its therapeutic potential, the peptide nature of MIF-1 limits its pharmacokinetic profile, prompting the development of non-peptidic analogs with improved metabolic stability and oral bioavailability [3,4]. To address these limitations, pyridine-based scaffolds, recognized for their structural versatility and favorable central nervous system penetration [5,6] were employed as prolyl surrogates to synthesize twelve novel analogs exhibiting enhanced metabolic stability and drug-like properties. Preliminary cytotoxicity evaluation in differentiated SH-SY5Y cells revealed that analogs containing methyl ester groups exhibited marked cytotoxicity, whereas amide counterparts displayed favorable safety profiles for neuronal applications. This structure-cytotoxicity correlation provides a valuable framework for further development of pyridine-based MIF-1 derivatives with optimized toxicological properties, enabling subsequent pharmacological evaluation of their PAM activity and advancing the search for safer, more effective treatments for PD.

Acknowledgments:

FCT is also acknowledged for supporting the project UID/50006 - Laboratório Associado para a Química Verde - Tecnologias e Processos Limpos. I.E.S.-D. thanks FCT for funding through the Individual Call to Scientific Employment Stimulus with reference 2020.02311.CEECIND/CP1596/CT0004 (DOI: 10.54499/2020.02311.CEECIND/CP1596/CT0004). B.L.P.-L., X.C.C., and H.F.C.-A. thank FCT for the Ph.D. grants 2022.14060.BD (DOI: 10.54499/2022.14060.BD), 2024.02245.BD, and UI/BD/154888/2023 (DOI: 10.54499/UI/BD/154888/2023), respectively. N.V. thank FCT for funding through projects IF/00092/2014/CP1255/CT0004, PRR-09/C06-834107/2024.P11721, 2024.18026.PEX, and the Chair in Onco-Innovation at the FMUP. X.G.-M. thanks Xunta de Galicia for financial funding with reference GPC2020/GI1597. X.G.-M. thanks Consellería de Cultura, Educación e Ordenación Universitaria of the Galician Government (grant: ED431B 2023/25) and Ministerio de Ciencia e Innovación – Agencia Estatal de Investigación (PID2021-124010OB-I00) for the financial support. This work received financial support from PT national funds (FCT/MECI, Fundação para a Ciência e a Tecnologia and Ministério da Educação, Ciência e Inovação) through project 2023.14440.PEX (DOI: 10.54499/2023.14440.PEX).

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Quinolinium-Substituted Porphyrins as Promising Antimicrobial Photosensitizers

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Characterized by remarkable chemical versatility and outstanding photophysical properties, porphyrins stand out as one of the most promising classes of photosensitizers for antimicrobial photodynamic therapy (aPDT) [1,2]. In this work, the potential of two cationic quinolinium-substituted porphyrins, the free-base form and the corresponding Zn(II) complex, was evaluated in the photoinactivation of bacterial strains under white light irradiation [3]. Methicillin-resistant *Staphylococcus aureus* (MRSA) and *Escherichia coli* were chosen as representative models of Gram-positive and Gram-negative bacteria, respectively.

The results to be presented here demonstrate the high efficiency of these cationic porphyrins in photoinactivating both bacterial strains, highlighting their potential for aPDT [3]. The addition of potassium iodide (KI) as a co-adjuvant led to the production of reactive iodine species (RIS), mainly I₂/I₃, which significantly improved the photodynamic effect (Fig. 1). Both porphyrins were shown to act via a Type II photochemical mechanism, confirming that their cytotoxic action depends mainly on ¹O₂ production. The synthetic strategy, photophysical properties, and biological evaluation of these quinolinium-substituted porphyrin photosensitizers will be presented and discussed.

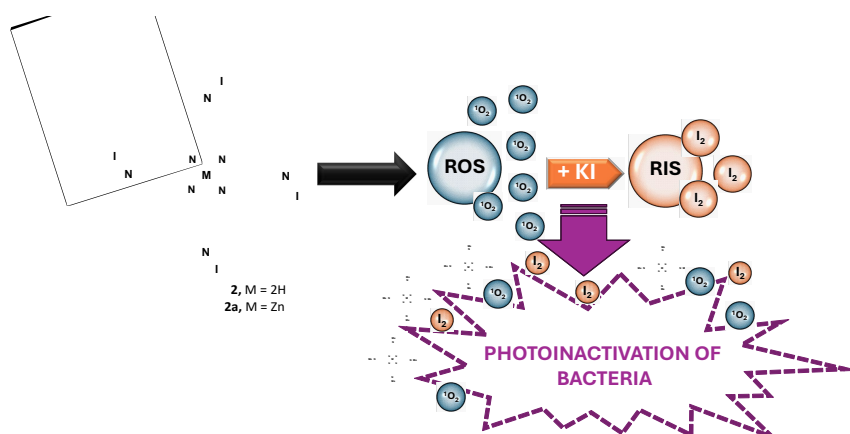


Fig.1. Schematic representation of the antimicrobial photodynamic effect triggered by both quinolinium-substituted porphyrins upon white light irradiation

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Targeting Cancer and Antimicrobial Resistance using Tryptophanol-derived Isoindolinones

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Growing evidence underscores the significant role of bacteria in cancer initiation and progression, although the underlying molecular mechanisms remain poorly understood. Certain cancer-associated bacterial species produce toxins that inactivate tumour-suppressor proteins such as p53, thereby promoting tumorigenesis through genomic instability, modulation of the tumour microenvironment, and evasion of immune surveillance [1,2]. Despite DNA damage and reactive oxygen species (ROS) being potent activators of p53, bacterial pathogens have developed strategies to suppress p53-mediated apoptosis. These observations highlight the importance of further exploring the interplay between bacteria and p53 regulation, which may open new avenues for cancer therapy [3]. At the same time, the global rise of antimicrobial resistance has markedly reduced the efficacy of last-resort antibiotics, including colistin, against Gram-negative pathogens. To tackle this challenge, indole-based compounds have attracted attention as promising scaffolds for the design of new antibacterial agents.³

Our group has previously identified enantiopure tryptophanol-derived isoindolinones as direct reactivators of both wild-type and mutant p53, with several derivatives demonstrating p53-dependent *in vivo* antitumor activity [4]. Within the framework of COST Action CA21145: European Network for Diagnosis and Treatment of Antibiotic-Resistant Bacterial Infections (EURESTOP), we further investigated the antimicrobial potential of this series of derivatives. Their activity was assessed against reference strains of *Acinetobacter baumannii* and *Escherichia coli*, both alone and in combination with colistin. Of the 30 synthesized compounds, 24 modulated the thermal stability of mutant p53 variants R273H and R280K in DSF assays. Among these, 11 exhibited significant antiproliferative activity in cancer cell lines harbouring the same mutations. Additionally, six compounds demonstrated enhanced activity in combination with colistin, indicating potential synergistic effects against reference strains of Gram-negative bacteria.

Acknowledgments

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Impact of Combat Activities on Oxidative Stress Levels of Firefighters

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The occupational activity as a firefighter involves exposure to complex mixtures of combustion products, including particulate matter, volatile organic compounds, and polycyclic aromatic hydrocarbons, which have been linked to respiratory, cardiovascular, and metabolic disorders [1, 2]. Repeated exposure to fire-generated pollutants, combined with physical exertion and heat stress, can induce oxidative stress, an imbalance between the formation of reactive oxygen species and antioxidant defences. This imbalance promotes lipid peroxidation, leading to cellular membrane damage and contribute to the development/aggravation of chronic diseases such as atherosclerosis and cancer [3]. Among oxidative stress biomarkers, urinary 8-iso-prostaglandin F₂α (8-iso) is a robust, non-invasive biomarker of lipid peroxidation. Measuring 8-iso levels at baseline and post-firefighting season can provide insights into the chronic oxidative stress at the end of wildfires season.

This study aims to assess urinary 8-iso levels in firefighters from the Alto Tâmega and Barroso region (Northern Portugal), who were actively engaged in firefighting activities during the 2024 wildfire season. The region registered 308 fire events with a total burned area of 8,813 hectares of land. Healthy firefighters from three fire departments in Alto Tâmega and Barroso agreed to voluntarily participate in the study, previously approved by the Ethics Committee of the University of Porto and conducted in accordance with the Declaration of Helsinki. Spot urine samples were collected before (baseline – June) and at the end fire season (post-season – October) at the fire stations. After collection, samples were stored at –20 °C until analysis. A group of 18 non-smoking participants was selected to conduct a preliminary assessment of urinary 8-iso levels at both time points. Enzyme-linked immunosorbent assays (ELISA) validated for use in urine samples were applied according to the manufacturer's instructions to quantify urinary 8-iso (competitive ELISA, item 516351, Cayman, Ann Arbor, MI, USA). All samples were analysed in duplicate at a 1:25 dilution. To minimize inter- and intra-individual variability related to hydration and urine concentration, biomarker levels were normalized to urinary creatinine (Jaff-colorimetric method). The median (P25 – P75) 8-iso concentrations in firefighters were 0.893 (0.703 - 1,154) and 1.035 (0.839 - 1.169) ng/mg creatinine at baseline and post-season, respectively. Urinary 8-iso levels presented interindividual variability between pre- and post-season assessments, with some firefighters presenting increases and others decreases in 8-iso concentrations, suggesting heterogeneous oxidative stress responses to firefighting activities. Additional studies are underway in a higher number of participants to better characterize 8-iso levels and its correlation with effect and exposure biomarkers.

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Lung Injury Assessment of Wildland Firefighters

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Work-related exposure as a firefighter encompasses several occupational risks that may contribute to or exacerbate the development of chronic diseases, including cardiovascular and respiratory disorders, as well as certain types of cancer [1]. Despite the use of adequate personal protective equipment and preventive measures, firefighters are exposed to numerous hazardous pollutants released during firefighting activities, which, once inhaled, can cause lung injury and trigger both local and systemic inflammation [2]. Although only a few studies have assessed urinary Clara Cell Secretory Protein 16 (CC16) in firefighters, this biomarker may serve as a useful non-invasive tool to evaluate acute lung epithelial injury before and after a wildfire season [3, 4]. This study aims to assess urinary levels of CC16 in firefighters from the Chaves region (Northern Portugal) both before and after the wildfire season.

Firefighters from three fire brigades in Chaves were enrolled in the study, which had been previously approved by the Ethics Committee of the University of Porto, in accordance with the Declaration of Helsinki. Pre-exposure spot urine samples of 20 healthy non-smoking subjects were collected in June 2024 (baseline) and October 2024 (post-season) at each fire station and stored at -20 °C until analysis. Enzyme-linked immunosorbent assays (ELISA) validated for use in urine samples were applied according to the manufacturer's instructions to quantify urinary CC16 (sandwich ELISA assay, item RD191022200, BioVendor R and D, Brno, Czech Republic). All assayed samples were analysed in duplicates in a dilution of 1:25. The inter- and intra-individual variability in hydration and urine dilution was reduced by normalizing biomarker concentrations with individual urinary creatinine, determined by Jaff-colorimetric method.

The median concentration of the lung injury biomarker was 12.24 (0.039–165.94) and 12.88 (0.082–198.57) ng/mg creatinine at the baseline and post-season, respectively. Urinary CC16 levels showed individual variability between pre- and post-season measurements, with some firefighters exhibiting increases (9.7 – 276.6%) and others decreases (5.3 – 72.1%), reflecting differences in acute lung epithelial response to combat activities. Further studies are ongoing to characterize the correlation between urinary CC16 levels and other effect biomarkers, as well as exposure biomarkers.

Acknowledgments

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Design, Synthesis, and Biological Evaluation of New Vitamin D Receptor Ligands for Application in Breast Cancer Therapy

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Vitamin D Receptor (VDR) is a nuclear receptor that mediates the biological effects of 1,25-dihydroxyvitamin D₃ (1,25D₃, calcitriol), including calcium homeostasis, cell differentiation, and proliferation [1, 2]. Although 1,25D₃ and other VDR agonists have shown therapeutic potential in cancer therapy, their clinical use is limited by severe hypercalcaemic effects at therapeutic doses [1, 2].

Here, a carbon-to-silicon bioisosteric strategy was explored for the first time in 1,25D₃ to improve VDR binding affinities, metabolic stability, and reduce hypercalcaemic effects.[3] To this end, six novel sila-derivatives of 1,25D₃, in which a silicon atom replaces the C25 side chain carbon, were designed, synthesised, and biologically evaluated (Figure 1). The derivatives were prepared via a Wittig-Horner strategy from the Inhoffen-Lythgoe diol. The crystal structures of the complexes of the VDR ligand-binding domain with the sila-derivatives revealed that the silicon-containing side chains establish additional stabilising interactions within the VDR ligand-binding domain, thereby reinforcing the active conformation of the receptor. Functional studies showed that sila-derivatives retain VDR binding and transcriptional activity comparable to 1,25D₃, while significantly reducing hypercalcaemic effects *in vivo*.

These derivatives also demonstrated anti-proliferative activity in breast cancer cell lines (MCF-7 and MDA-MB-231 cells) at 10⁻⁷ M and potentiated the efficacy of conventional chemotherapeutics. Collectively, these findings highlight carbon-to-silicon substitution at the C25 position as a promising strategy for developing non-calcaemic secosteroidal VDR ligands, opening new opportunities for vitamin D-based cancer therapies.

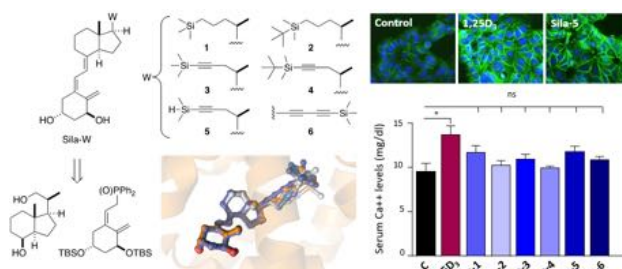


Figure 1. Sila-Derivatives of Vitamin D.

Acknowledgments

This work received financial support from PT national funds (FCT/MECI, Fundação para a Ciência e Tecnologia, and Ministério da Educação, Ciência e Inovação) through the project UID/50006 - Laboratório Associado para a Química Verde - Tecnologias e Processos Limpos. I.E.S.-D. thanks FCT for funding through the Individual Call to Scientific Employment Stimulus with reference 2020.02311.CEECIND/CP1596/CT0004 (DOI: 10.54499/2020.02311.CEECIND/CP1596/CT0004). M.B.I.-C. thanks REQUIMTE for the research grant with reference REQUIMTE-2025-42. This work received financial support from FEDER funds (COMPETE 2030), through project COMPETE2030-FEDER-00881500, with operation no. 15152.

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Descoberta de Potentes Moduladores Alostéricos dos Recetores de Dopamina D₂ por Conjugação da Melanostatina com Amantadina

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A melanostatina (Pro-Leu-Gly-NH₂; MIF-1) é um pequeno neuropéptido conhecido pela sua atividade como modulador alostérico positivo (MAP) dos recetores de dopamina D₂ (D₂R) [1]. O seu potencial terapêutico em doenças neurológicas, incluindo a doença de Parkinson, é limitado pela baixa absorção gastrointestinal e rápida degradação enzimática no sistema nervoso central [2]. Para superar estes desafios farmacocinéticos, este estudo explora a conjugação da MIF-1 com uma estrutura rígida e lipofílica, a Amantadina (Am), no seu resíduo C-terminal (Figura 1) [3].

Foram sintetizados dois novos derivados através de uma estratégia de alongamento de C-para-N em solução, com estruturas confirmadas por espectroscopia de ressonância magnética nuclear e espectrometria de massa de alta resolução. Ensaios de lipofilicidade através do método *shake-flask*, demonstraram um aumento significativo da lipofilicidade em comparação com a MIF-1. Estes compostos foram avaliados em D₂R humanos expressos em células de ovário de hamster chinês. Os conjugados aumentaram a potência da dopamina em 3,3 e 4,9 vezes (EC₅₀ = 26,39 ± 3,37 nM e 17,82 ± 4,24 nM) em relação à dopamina (EC₅₀ = 87,08 ± 24,87 nM) a uma concentração de 0,01 nM, apresentando uma maior potência neste ensaio do que o péptido parental. Nenhum dos compostos apresentou atividade agonista intrínseca na ausência de dopamina, confirmando o seu mecanismo alostérico.

Este trabalho demonstra que a substituição da amida primária C-terminal da melanostatina por uma amida secundária hidrofóbica permitiu a descoberta de dois novos MAP dos D₂R, com maior potência e propriedades farmacocinéticas melhoradas em comparação com a MIF-1, abrindo portas para o desenvolvimento de futuros moduladores com aplicações em distúrbios relacionados com a disfunção dopaminérgica.

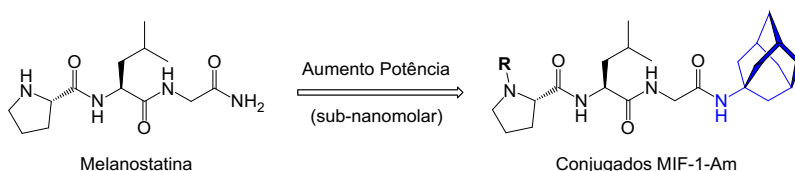


Fig. 1. Estrutura da melanostatina e dos conjugados sintetizados neste trabalho. R = H ou Boc.

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Synthesis of Porphyrin-Carboxamides: Efficient Photosensitizers Against Bacteria

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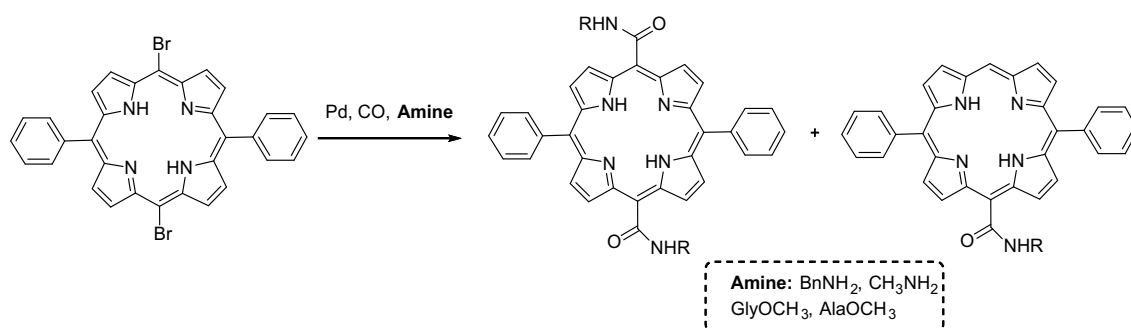
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The growth of multidrug-resistant bacteria represents one of the most critical challenges in current public health [1]. Consequently, nowadays the development of alternative therapeutic approaches capable of eradicating pathogenic microorganisms is crucial. Among these strategies, photodynamic inactivation (PDI) employing amphiphilic porphyrins has demonstrated significant potential [2].

In the present study, we report a novel approach for the functionalization of the *meso*-positions of 5,15-dibromo-10,20-diphenylporphyrin through a palladium-catalyzed aminocarbonylation reaction [3]. Furthermore, we present and discuss structure–activity relationship data regarding the efficacy of these porphyrins in the photoinactivation of both Gram-positive and Gram-negative bacteria.



Scheme 1 – Synthetic strategy for obtaining aminocarbonylated porphyrins.

Acknowledgments

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Exploring VAS2870 Structure for the Development of Homolog-Selective NOX4 Inhibitors

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NADPH oxidases (NOXs) are a family of seven enzymes responsible for producing reactive oxygen species (ROS) and have been considered potential therapeutic targets to treat oxidative stress-related disorders. However, NOX isoforms share a conserved catalytic core, making selective targeting a significant challenge. The currently available inhibitors face several limitations, such as assay-interfering effects, low potency, high cytotoxicity, and lack of selectivity(a). Among these, VAS2870 is a non-assay-interfering NOX inhibitor that covalently targets a cysteine residue within the dehydrogenase domain. Nevertheless, like other NOX inhibitors, it exhibits low selectivity, showing greater potency for NOX2 and NOX5, along with poor aqueous solubility, and cytotoxic effects(b). The main goal of this study was the design, synthesis, and biological evaluation of a small library of VAS2870-based compounds to identify a novel NOX inhibitor with enhanced potency, selectivity, and safety.

A small library of compounds, incorporating different cysteine-targeting warheads, was successfully synthesized and tested against full-length NOX1, NOX2, NOX4, and NOX5. Following the identification of a hit compound, a second series was synthesized and evaluated to further optimize the compound's properties and validate the use of the selected warhead. Ultimately, a potent, isoform-selective, non-interfering NOX4 inhibitor was successfully obtained. In summary, a series of novel VAS2870-based inhibitors with improved potency and selectivity were developed, and a compound emerged as a potent, NOX4-selective inhibitor. These results demonstrate the potential of maleimide-based covalent inhibitors as promising candidates for targeted NOX therapy.

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Bioaccessibility and antioxidant activity of Bacoside A from *Bacopa monnieri* (L.) Wettst

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Bacoside A, a saponin from *Bacopa monnieri* (L.) Wettst. to which most of the plant's beneficial effects are attributed, particularly nootropic and antioxidant properties, is a molecule with considerable potential for diverse therapeutic applications [1]. To fully exploit these benefits, however, it is essential to understand how this compound behaves during gastrointestinal digestion, since its efficacy largely depends on its bioaccessibility [2]. Given the high structural diversity of saponins, knowledge about their bioaccessibility cannot be generalized [3]. In the specific case of Bacoside A, no studies have yet directly assessed this parameter. This study aimed to evaluate the bioaccessibility of Bacoside A in a *B. monnieri* extract obtained by maceration following the INFOGEST 2.0 protocol. In addition, the antioxidant activity of the bioaccessible fraction was determined using thiobarbituric acid reactive substances (TBARS) and oxygen radical absorbance capacity (ORAC) assays and compared with the pre-digestion extract. *In vitro* digestion revealed that Bacoside A was bioaccessible and presented antioxidant activity for both tested methods. As the antioxidant activity of *B. monnieri* extract can be attributed to both its saponins and phenolic compounds, the results suggest that these bioactive constituents contribute to the antioxidant potential of the bioaccessible fraction [2]. These findings highlight the need for further research on Bacoside A bioaccessibility and the development of strategies to improve its gastrointestinal stability and absorption, thereby maximizing its health-promoting potential.

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Biomembranas funcionais baseadas em polissacarídeos e polifenóis para o tratamento da mucosite oral

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A mucosite oral (MO) é uma das condições mais debilitantes em pacientes com cancro da cabeça e do pescoço, quando submetidos a tratamentos de quimioterapia e radioterapia [1]. Estima-se que 40% dos pacientes oncológicos venham a desenvolver mucosite oral, sendo que esse número pode subir para 90% em pacientes com cancro nestas localizações específicas [2]. Esta condição trata-se de uma inflamação dolorosa, que muitas vezes se traduz numa pobre qualidade de vida, comprometimento de outros fatores como o estado nutricional e até interrupções no tratamento oncológico [1].

Devido à grande prevalência e gravidade desta condição, o desenvolvimento de novas estratégias para o tratamento e/ou prevenção da MO é crucial, de modo a se poder garantir uma melhor qualidade de vida a estes pacientes. O uso de biomembranas ou filmes funcionais baseados em polissacarídeos e polifenóis tem o potencial de ser uma das abordagens mais promissoras uma vez que a combinação destes compostos bioativos poderá conferir efeitos sinérgicos direcionados ao tratamento da MO nomeadamente propriedades promotoras da cicatrização, antioxidantes e antimicrobianas [1].

Assim sendo, o objetivo deste trabalho é o desenvolvimento de filmes/biomembranas de diferentes polissacarídeos com a incorporação de extratos polifenólicos de origem vegetal e, avaliar as suas características físico-químicas (e.g., morfologia, espessura, solubilidade, perfil de libertação de compostos) e propriedades biológicas, especialmente em culturas celulares de células epiteliais derivadas de língua humana, com vista à sua aplicação no tratamento da MO. Os resultados obtidos permitiram a identificação de polissacarídeos promotores do crescimento celular, propriedade biológica essencial na promoção da cicatrização celular.

Agradecimentos

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Pyrene-Benzothiadiazole Derivatives as Dual Type I and Type II Photosensitizers for Photodynamic Applications

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Photodynamic therapy (PDT) is a minimally invasive treatment that involves the activation of photosensitizers with visible light in the presence of molecular oxygen, resulting in the production of reactive oxygen species (ROS) that selectively induce cell death. ROS generation in PDT occurs through two main pathways, with type I relying on radical formation and type II involving the production of singlet oxygen [1-2]. The type I mechanism is particularly important in hypoxic tumor tissues and in microbial environments where oxygen availability is limited, thereby expanding the therapeutic potential of PDT to both cancer and infectious diseases [3].

The effectiveness of PDT critically depends on the design of suitable photosensitizers. Benzothiadiazoles (BTDs) have emerged as promising scaffolds due to their excellent thermal and photochemical stability and strong absorption in the visible spectrum. Functionalization of BTDs with cationic heterocyclic salts can significantly enhance water solubility, promote cellular uptake and ROS generation, and improve tumor selectivity [4-6].

In this work, we report novel pyrene-benzothiadiazole derivatives, which exhibit efficient internalization and biocompatibility in 4T1 cancer cells. Upon irradiation, these compounds effectively generate ROS through both type I and type II pathways, with type I ROS production being predominant, and induce significant cell death. This highlights their considerable potential as photosensitizers for anticancer photodynamic therapy. Considering the relevance of type I pathways in low-oxygen microbial environments, ongoing studies are currently evaluating their potential for antimicrobial photodynamic applications.

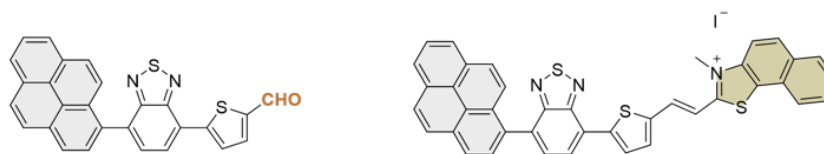


Fig.1. Chemical structure of the pyrene-benzothiadiazole derivatives under study.

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Synthesis of methyl 7-[(hetero)arylethynyl]thieno[2,3-*b*]pyrazine-6-carboxylates by Sonogashira coupling and anti-tumor evaluation

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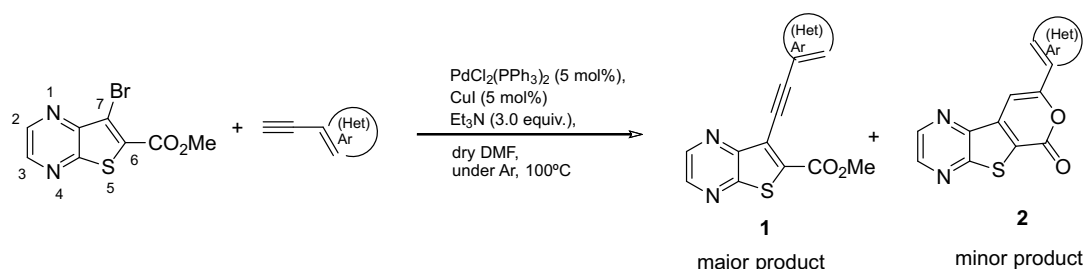
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In recent years the synthesis and biological activities of thieno[2,3-*b*]pyrazines have been predominantly reported in patents [1,2]. Since 2021 our research group has been focused on the functionalization of the thieno[2,3-*b*]pyrazine scaffold using C-N Buchwald-Hatwig coupling and C-C Sonogashira coupling. The resulting derivatives have been evaluated for antitumor activity against some human tumor cell lines and antiparasitic activity and some of them exhibited promising activity [3,4].

In this work, we report the synthesis of several 7-[(hetero)arylethynyl]thieno[2,3-*b*]pyrazines **1** by Sonogashira coupling of the methyl 7-bromothieno[2,3-*b*]pyrazine-6-carboxylate, also prepared by us, with various (hetero)arylacetylenes, using Pd and Cu catalysts and Et₃N as a base (Scheme 1). Additionally, tricyclic lactones **2** resulting from 6-*endo-dig* cyclization of compounds **1** were isolated as a very minor product, in some cases. The products were separated by column chromatography and all the new compounds were fully characterized by ¹H, ¹³C RMN and HRMS.



Scheme 1. Synthesis of 7-[(hetero)arylethynyl]thieno[2,3-*b*]pyrazines by Sonogashira cross-coupling.

The antitumor potential of the synthesized compounds was evaluated against several tumor cell lines (MCF-7, Caco-2, AGS, HeLa, and NCI-H460) using the sulforhodamine B assay. Among them, only the Sonogashira coupling product of 2-ethynylpyridine, methyl 7-(pyridin-2-ylethynyl)thieno[2,3-*b*]pyrazine-6-carboxylate, exhibited potent cytotoxicity, with GI₅₀ values below 10 μM across all tested tumor cell lines. Notably, this compound showed low toxicity in non-tumor PLP2 cells (GI₅₀ = 58 μM), indicating a favorable selectivity profile at concentrations effective against cancer cells.

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Electrochemical genosensors to detect the Single Nucleotide Polymorphism (c.100C>T, rs1065852) in the *CYP2D6**10 gene

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The *CYP2D6* gene encodes a highly polymorphic enzyme responsible for the metabolism of various psychotropic drugs, like antidepressants, antipsychotics, analgesics and beta-blockers [1]. One frequent variation in the *CYP2D6* gene is the single nucleotide polymorphism (SNP) rs1065852 polymorphism. This SNP defines the *CYP2D6**10 allele and is linked to an intermediate metabolizer phenotype, which results in a reduced enzymatic activity and can lead to an increased risk of adverse reactions to the psychotropic drugs [1,2].

To address this issue, a disposable electrochemical genosensor for the rapid and specific detection of the rs1065852 SNP in patients was developed. By analyzing public databases, two specific DNA-target probes; one completely complementary to the *CYP2D6**10 wild-type gene (Target T) and another with the single-nucleotide genetic variation (Target C) were selected and designed. Their complementary sequences were cut into two smaller oligonucleotide sequences: a thiol-modified DNA-capture and a fluorescein-labeled DNA-signaling to enable the hybridization reaction. So, the sensor's design involved the immobilization of a self-assembled monolayer composed of mercaptohexanol (MCH) and the linear thiolated DNA-capture probe onto screen-printed gold electrodes (SPGE), followed by the sandwich hybridization of a DNA-target sequence with the DNA-signaling probe. Detection of the electrochemical signal was obtained by chronoamperometry.

The developed genosensor successfully discriminated between the two target probes, with the fully complementary DNA-target sequence (Target T) generating an electrochemical signal 56.81% higher than the partially complementary SNP sequence (Target C). The genosensor also exhibited good sensitivity, selectivity and reproducibility. These results demonstrate the genosensor's potential as a rapid, precise and reliable tool for clinical applications.

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Synthesis by Buchwald-Hartwig Amination and anti-parasitic activity of di(hetero)arylamines in the thieno[2,3-*b*]pyridine series

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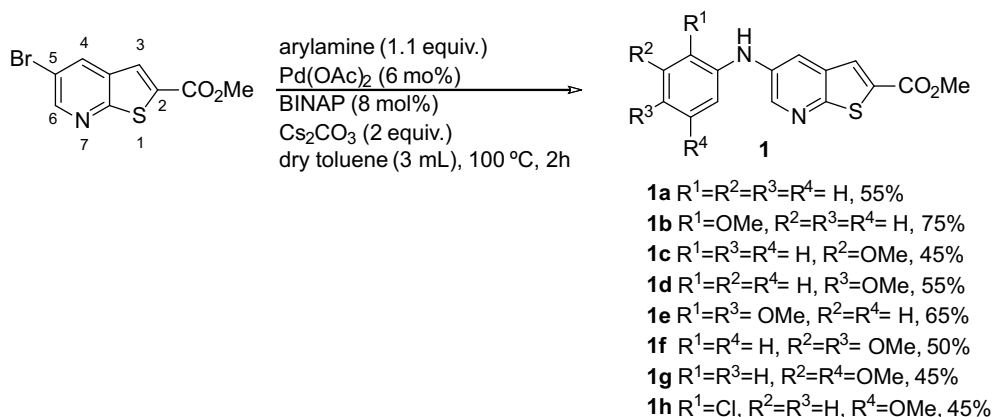
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The Buchwald-Hartwig amination, palladium-catalyzed C-N cross-coupling, has become a widely used method for synthesizing diarylamines from (hetero)aryl halides and anilines [1].

Thieno[2,3-*b*]pyridine derivatives are of particular interest owing to their wide-ranging biological properties, including anti-inflammatory, antiparasitic, antiviral, antifungal, and antidiabetic activities [2]. In this work, the synthesis of several methyl-5-(arylamino)thieno[2,3-*b*]pyridine-2-carboxylates **1a-1h** in good to high yields by Buchwald-Hartwig C-N coupling of methyl 5-bromothieno[2,3-*b*]pyridine-2-carboxylate, also prepared, with aniline, anisidines and dimethoxyanilines, is reported (**Scheme 1**). Compounds **1a-1h** were fully characterized by m.p., ¹H and ¹³C NMR and High-Resolution Mass Spectrometry (HRMS).



Scheme 1. Synthesis of methyl 5-(arylamino)thieno[2,3-*b*]pyridine-2-carboxylates **1a-1h**

The antiparasitic activity of the synthesized compounds was evaluated against *Trypanosoma brucei* (*T. brucei*) and *Leishmania infantum* (*L. infantum*) promastigotes, as well as intracellular amastigotes. The most promising results against *T. brucei* were obtained for compounds **1g** (IC₅₀ = 7.88 μM), **1f** (IC₅₀ = 9.66 μM), **1c** (IC₅₀ = 11.42 μM), and **1d** (IC₅₀ = 14.33 μM). The presence of a methoxy group in the *meta* position was found to be crucial for activity, with the *meta,meta*-diOMe derivative **1g** emerging as the most active compound. Among the series, only **1e**, the *orto,para*-diOMe derivative, displayed activity against *L. infantum* promastigotes, with an IC₅₀ of 11.21 μM. Cytotoxicity was assessed in PMA-differentiated THP-1 cells using the MTT assay. With the exception of compound **1h**, which bears a chloro substituent (CC₅₀ > 50 μM), none of the tested compounds exhibited significant toxicity (CC₅₀ > 100 μM). For compound **1g** the selectivity index (SI) is greater than 12.7, indicating a favourable safety and selectivity profile.

Acknowledgements

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Discovery of Potent Dopamine D₂ Receptor Modulators by Proline Homologation in Melanostatin Neuropeptide

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The gold standard for Parkinson's therapy focuses on dopamine potentiation in the central nervous system (CNS) through the administration of levodopa [1]. However, long-term treatment with this drug is known to cause serious adverse effects that can worsen the motor symptoms of the disease [1]. Melanostatin, an endogenous hypothalamic neuropeptide that acts as a positive allosteric modulator of D₂ receptors (D₂R), thereby exhibiting therapeutic potential in dopamine-related disorders of the CNS, such as depression and Parkinson's disease, among others [2]. Nevertheless, this neuropeptide fails to exhibit adequate pharmacokinetic properties, hampering its clinical use [2].

To overcome these limitations, a series of melanostatin homologous derivatives was synthesized using L-pipecolic acid as an L-proline surrogate (Figure 1) and pharmacologically evaluated by cAMP mobilization using mammalian cells transfected with human D₂R. In this series, two compounds exhibited significant PAM activity at 0.01 nM by promoting up to a 4-fold increase in dopamine potency. In addition, *in silico* conformational studies were performed to assess the preferential conformations of the bioactive compounds. These results endorse the potential application of melanostatin derivatives as a new generation of anti-Parkinson's agents with improved pharmacokinetics.

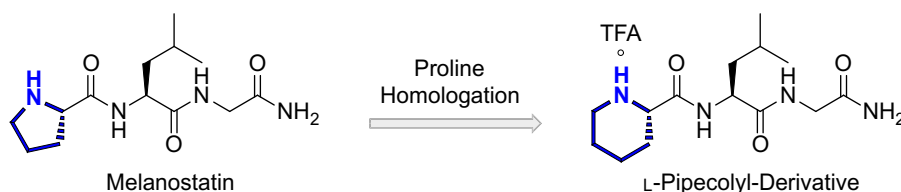


Fig.1. Proline homologation in melanostatin neuropeptide.

Acknowledgments

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Synthesis and characterization of the wound-healing peptide CW49

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Diabetes mellitus (DM) is a chronic metabolic disease with high prevalence, morbidity and mortality rates worldwide [1]. DM patients have a 25% lifetime risk of developing diabetic foot ulcers, severe chronic wounds which are significantly susceptible to infections. More than 50% of these wounds require hospitalization due to health complications, poor wound management and treatment ineffectiveness [2]. CW49, a little explored peptide, has demonstrated strong angiogenic activity and the ability of preventing excessive inflammation in diabetic wounds, which demonstrates its potential for wound healing applications [3,4].

Here, we report the synthesis of the undecapeptide CW49 by microwave-assisted solid phase peptide synthesis, on a Wang solid support using the Fmoc strategy. The peptide was purified using preparative high performance liquid chromatography, and the structure was confirmed by matrix-assisted laser desorption ionization time of flight mass spectrometry and two-dimensional nuclear magnetic resonance spectroscopy (NMR).

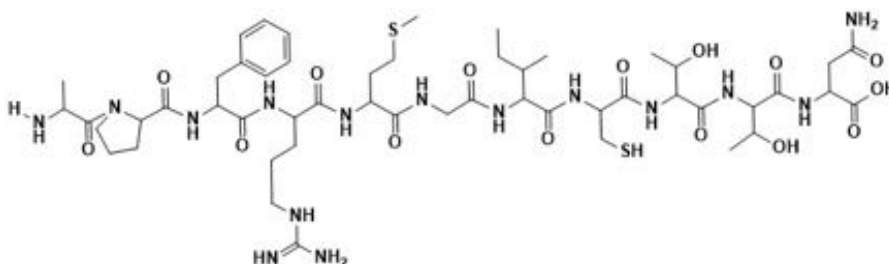


Fig. 1. CW49 structure

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Synthesis and characterization of Polymyxin B derivatives by conservative modification of selected amino acids

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Polymyxins are a class of antibiotics primarily used to treat infections caused by Gram-negative bacteria, of which Polymyxin B and Polymyxin E (also known as colistin) are currently in use in clinical settings, in spite of their side effects (neuro- and nephrotoxicity). Both are FDA-approved for treating serious infections caused by multidrug-resistant Gram-negative organisms, particularly *Enterobacteriaceae*, *Pseudomonas aeruginosa*, and *Acinetobacter baumannii*. Polymyxins are frequently administered a last-resort treatment (intravenous) when other antibiotics have failed, being especially valuable for managing infections of the urinary tract, central nervous system (including meningitis), and bloodstream [1].

Conservative modification in peptides involves replacing an amino acid by another that shares similar chemical properties, aiming to improve the peptide's stability, bioactivity, or pharmacokinetic properties while minimizing structural changes [2].

Having in mind the search for new antimicrobial compounds with better activity and lower toxicity than PMB (one of the reasons for its withdrawal from clinical practice in the past), we herein report the synthesis, through microwave-assisted solid-phase peptide synthesis, and characterization (by mass spectrometry and liquid chromatography) of six new PMB-based derivatives, obtained by replacing the amino acids of positions 1 and/or 3 (Dab) or 6 (Phe) by unnatural amino acids with similar structure and characteristics.

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Initial insights into the clinical safety evaluation of biosurfactant extracts derived from lactic acid bacteria through Patch testing

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Given the increasing interest in biosurfactants (BSs) for use in health and personal care products, it is crucial to assess their clinical safety through standardized methods to determine their potential for skin irritation and sensitization. For that, this study presents a clinical safety assay to verify the skin compatibility of the BSs derived from Lactic Acid Bacteria (LAB) in healthy adult volunteers involving an occlusive single patch test methodology. BSs from *Lactobacillus pentosus* were produced through fermentation at 31°C, pH 5.85, for 48 h, using two media: MRS broth and a cost-effective medium based on corn steep liquor (10 g/L), yeast extract (10 g/L), glucose (11 g/L), and xylose (18 g/L). Meanwhile, BS from *Lactobacillus paracasei* was fermented produced in a modified MRS broth with lactose (20 g/L) as the carbon source at 37°C, pH 7, for 72 h. On the other hand, ten volunteers, comprising both males and females aged between 18 and 65 years with a range of Fitzpatrick skin phototypes I to IV and both normal and sensitive skin, were selected according to strict inclusion and exclusion criteria. Each participant received a single application of an aqueous BS extract at the highest concentration assay that did not induce cytotoxicity in fibroblasts cells. The extract was diluted to 5% for enhanced safety and applied to the upper back under an occlusive patch for a duration of 48 h. The primary endpoint was the clinical evaluation of skin reactions, specifically erythema and oedema, at 30 min post-removal and again at 96 h. Results were assessed using the Mean Irritation Index (M.I.I.), a recognized dermatological scale. Throughout the study period, none of the ten volunteers exhibited any signs of irritation, erythema, or oedema following exposure to the test product; all individual and mean irritation indices were zero. Adherence to rigorous clinical standards and good clinical practices was maintained. The results unequivocally classified the BSs products as “non-irritating,” with “very good skin compatibility” under the tested conditions. The findings indicate that BSs from LAB is safe for topical use, at the concentration assayed, in healthy adults and did not provoke any adverse cutaneous reactions in this controlled setting. These results provide key support for the product’s future cosmetic or dermatological applications, consistent with EU standards for safety and efficacy testing of skin products. However, further research is necessary to evaluate the effect of higher concentrations and the synergistic effects of these extracts with active principles and other adjuvants.

Acknowledgments

This research is part of the Framework Collaboration Agreement between Universidade de Vigo (Spain) and Universidade do Minho (Portugal) and by the Xunta de Galicia under the project ED431F 2023/17. Also, X. Vecino acknowledges the grant RYC2021-030966-I funded by the MCIN/AEI/10.13039/501100011033 and by the European Union “NextGenerationEU/PRTR”.

HRIPT Initial Clinical Study on the Irritation and Sensitizing Potential of Biosurfactants from Lactic Acid Bacteria

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It is essential to ensure the safety of biosurfactants (BSs) in biomedical, cosmetic, and industrial applications, especially given their growing use as sustainable alternatives to synthetic surfactants. For that, the clinical evaluation of the irritation and sensitization potential of the BSs from lactic acid bacteria (LAB) was conducted according to the Marzulli-Maibach Human Repeated Insult Patch Test (HRIPT) protocol. BSs from *Lactobacillus pentosus* were produced by fermentation at 31°C and pH 5.85 for 48 h using two media: MRS broth and a cost-effective medium composed of corn steep liquor (10 g/L), yeast extract (10 g/L), glucose (11 g/L), and xylose (18 g/L). In parallel, BS from *Lactobacillus paracasei* was obtained through fermentation in a modified MRS broth containing lactose (20 g/L) as the carbon source, incubated at 37°C and pH 7 for 72 h. The study was carried out by a total of 62 volunteers (men and women aged 18 to 63 years) with Fitzpatrick skin phototypes I-IV, including both normal and sensitive skin types, were enrolled after meeting strict inclusion and exclusion criteria. Over a three-week induction phase, the BS products used at the highest concentration that did not induce cytotoxicity in fibroblasts, followed by a 5% dilution, was applied three times per week to a defined area on the back under semi occlusive patches. Following a two-week rest period, a challenge application was performed to assess potential sensitization. The primary endpoints were clinical evaluations of irritation - using the Mean Irritation Index (M.I.I.) - and sensitization, based on the International Contact Dermatitis Research Group (ICDRG) scale. Subjects were regularly monitored for signs of erythema, oedema, dryness, and vesicle formation. Results showed that BSs from LAB exhibited no irritation throughout the induction phase, with a Mean Irritation Index of 0.06-0.07, falling well within the “non-irritating” category. Similarly, no signs of sensitization were observed during the challenge phase, with no active allergic reactions reported among the 57 evaluable participants. The study was conducted in full compliance with clinical protocols and Good Clinical Practice (GCP) standards. In conclusion, under the tested conditions, the BSs extracts were demonstrated to be both non-irritating and non-sensitizing when they are topically applied to healthy adult skin. These findings provide a promising first insight and scientific support for the safe incorporation of BSs from LAB in future cosmetic or dermatological formulations. Nevertheless, additional assays should be conducted to assess the safety of biosurfactant concentrations that are closer to the critical micellar concentration and their interactions with other ingredients.

Acknowledgments

This research is part of the Framework Collaboration Agreement between Universidade de Vigo (Spain) and Universidade do Minho (Portugal) and by the Xunta de Galicia under the project ED431F 2023/17. Also, X. Vecino acknowledges the grant RYC2021-030966-I funded by the MCIN/AEI/10.13039/501100011033 and by the European Union “NextGenerationEU/PRTR”.

Invisible Threats: Microplastics in Human Systems

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Microplastics (MPs) are no longer confined to oceans and terrestrial ecosystems [1], being detectable in food, drinking water [2], and even within the human body. Their presence in blood, lungs, and placental samples [3] highlights an unsettling reality: humans are constantly exposed to these particles, yet the implications for health remain largely uncertain. While research has rapidly expanded in recent years, the biological pathways followed by MPs and their potential role in disease processes are only beginning to be understood. In this work, we reviewed the available literature up to May 2025 to assess how MPs are being identified in human samples and what this may reveal about their interaction with biological systems. Briefly, sixty-three studies were analyzed, showing consistent evidence of MPs penetrating and persisting in human tissues. Intriguingly, some findings suggest variations in MP burden between healthy and diseased tissues, raising questions about their contribution to pathological states. Moreover, this review also explored the methodological advances driving this field forward. Techniques such as micro-Raman spectroscopy, FTIR, and chromatographic analyses have improved the precision by which polymer type, morphology, and particle size can be characterized. These innovations not only strengthen confidence in the reported findings but also highlight the pressing need for standardized approaches that would allow results from different studies to be meaningfully compared. Taken together, the current body of evidence paints MPs as emerging markers of environmental exposure with potential relevance to human health. However, key gaps remain: the mechanisms of accumulation, transport, and interaction with tissues require further elucidation. Addressing these questions is crucial for assessing risks and guiding public health strategies in an increasingly plastic-saturated world.

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Non-invasive glucose determination in both human and canine saliva with a paper-based microfluidic device

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Monitoring glucose levels is the most common method for managing diabetes, a chronic metabolic disorder characterized by elevated blood glucose that can lead to severe complications. According to the International Diabetes Federation (IDF), in 2021, 10% of adults were living with diabetes, a figure projected to rise to 12.5% (1 in 8) by 2045 [1]. With the aging global population, elderly individuals face daily challenges in glucose monitoring. While current devices primarily rely on invasive blood sampling and electronic sensors, including implanted systems, these technologies can be difficult for older users.

Diabetes also significantly affects companion animals, particularly dogs and cats. The disease is estimated to affect about 0.3–1.3% of dogs in countries including the UK, USA, Sweden, and Italy, with prevalence increasing sharply over recent years [2].

To address accessibility and ease of use, we propose a simple, accurate, and user-friendly paper-based microfluidic device for glucose quantification in saliva (Fig. 1), eliminating the need for electronic readers. Microfluidic paper-based devices (μ PADs) are increasingly recognized as promising point-of-care tools due to their low cost, rapid response, and ease of use. Importantly, the developed μ PAD enables non-invasive glucose monitoring in both human and canine saliva, broadening its application to veterinary medicine as well as human healthcare. This dual-use approach highlights the potential of saliva as an alternative diagnostic fluid and offers a practical alternative to conventional blood-based invasive methods.



Fig. 1. Schematic representation of the assembly and determination procedure for the quantification of glucose with the developed device.

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Biocompatible Ru-MOF/PVA hydrogels for supporting and delivering drugs to osteoarthritic cartilage

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Metal-organic frameworks (MOFs) are emerging as promising biomaterials due to their tunable chemistry, high porosity, and potential for controlled drug release [1,2]. Here, we report the successful incorporation of Ru-HKUST-1, a ruthenium-substituted derivative of MOF-199, into poly(vinyl alcohol) (PVA) hydrogels.

Ru-HKUST-1 enhanced water uptake and porosity of hydrogels without compromising mechanical stability. A controlled release of ruthenium under sliding conditions suggested a potential mechanism for mechanosensitive and stimulus-responsive drug delivery.

Cytocompatibility tests with L929 fibroblasts and human chondrocytes up to 0.03 wt% Ru-HKUST-1 reveal no cytotoxic effects. Thus, Ru-HKUST-1/PVA hydrogels emerge as biocompatible, multifunctional materials that combine mechanical support and controlled drug release, promising for osteoarthritis therapy.

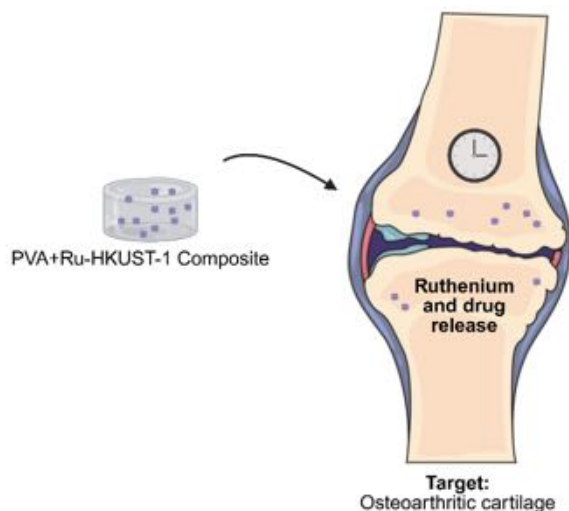


Fig.1. Application of a PVA/nano Ru-HKUST-1 composite for osteoarthritic cartilage treatment.

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Edible Mushrooms as Natural Sources: Antioxidant and Antimicrobial Insights against Wound Multidrug-Resistant

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Pseudomonas aeruginosa is one of the most relevant nosocomial pathogens, responsible for nearly 10% of hospital-acquired infections and listed among the WHO critical priority bacteria. Its high prevalence of multidrug resistance results in severe outcomes and highlights the urgent need for innovative therapeutic solutions. Within this context, the valorization of natural matrices is a promising strategy. Edible mushrooms are a rich source of phenolic metabolites, molecules whose chemical diversity is associated with both antioxidant and antimicrobial properties, making them potential leads for the development of new therapeutic agents [1].

In this study, aqueous and hydromethanolic extracts of *Pleurotus ostreatus*, *Pleurotus citrinopileatus* and *Agaricus bisporus* were characterized for their total phenolic content (TPC), ortho-diphenols, radical scavenging capacity (ABTS assay), and antimicrobial activity against clinical multidrug-resistant isolates of *P. aeruginosa*. Among the tested samples, the aqueous extract of *P. citrinopileatus* exhibited the highest TPC (28 mg GAE·g⁻¹ dw) and ortho-diphenols (9 mg GAE·g⁻¹ dw), coupled with strong antioxidant capacity (85% ABTS inhibition) and the most pronounced antimicrobial efficacy (inhibition halos up to 14 mm). In contrast, *A. bisporus* extracts showed markedly lower bioactivity [2].

These findings demonstrate a clear relation between phenolic composition, antioxidant performance, and antimicrobial capacity, underlining the relevance of chemical profiling in guiding biological applications. The study emphasizes the potential of mushroom-derived phenolics as sustainable resources for the design of innovative therapeutic approaches against multidrug-resistant pathogens, reinforcing the contribution of chemistry to human health and to the search for solutions to global challenges.

Keywords: phenolic compounds; antioxidant activity; edible mushrooms; multidrug-resistant; *Pseudomonas aeruginosa*

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***Pleurotus citrinopileatus*: A Natural Modulator of Gut–Brain–Immune Communication**

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Mushrooms are increasingly recognized as valuable sources of bioactive compounds capable of shaping immune responses and influencing host physiology. Among them, *Pleurotus citrinopileatus* has emerged as a particularly promising species, drawing attention not only for its nutritional value but also for its broad spectrum of biological activities and potential health benefits [1].

In this context, the present study examined whether aqueous extracts of *P. citrinopileatus* could inhibit acetylcholinesterase (AChE), the enzyme responsible for degrading acetylcholine. Beyond its role in classical neurotransmission, acetylcholine is a key mediator of the gut–brain axis, coordinating bidirectional communication between the enteric and central nervous systems. Its availability influences intestinal motility, barrier integrity, and local immune responses, while also impacting cognitive and emotional functions. Preserving acetylcholine levels is therefore essential to sustain gut–brain signaling and support immune homeostasis [2, 3].

To explore this potential, the extract was first analyzed for its phenolic content using the Folin–Ciocalteu method, which revealed substantial levels consistent with the antioxidant performance observed in the ABTS•+ scavenging assay. Building on these findings, enzyme inhibition assays showed that *P. citrinopileatus* displayed measurable AChE inhibitory activity, suggesting a role in sustaining acetylcholine levels and protecting against cholinergic dysfunction.

These results indicate that aqueous extracts of *P. citrinopileatus* combine antioxidant potential with AChE inhibition, offering a dual mechanism of action that may support neurotransmitter regulation within the gut–brain–immune axis. This duality underscores the relevance of *P. citrinopileatus* as a natural nutraceutical candidate with promising applications in neuroprotection, immune modulation, and the preservation of gut–brain axis equilibrium.

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Stable and bioactive supramolecular nanoparticles generated from short α,α -disubstituted peptides

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Self-assembly of amphiphiles has become an attractive tool to generate hierarchical structures, from the molecular to the microscale. Peptide amphiphiles have been explored in the biomedical field as they can often generate nanoscale supramolecular assemblies, i.e., tubes, spheres and fibrils, that, upon crosslinking using the ionic strength of the physiological environment generate bioactive hydrogels for regenerative medicine.

In particular, the peptide amphiphile Fluorenylmethoxycarbonyl-diphenylalanine (Fmoc-FF) is one of the most commonly used building blocks, known for its ability to self-assemble into a diverse set of supramolecular assemblies, e.g., spheres, tapes, and fibres [1]. However, Fmoc-FF presents long term toxicity due to the cleavage of the Fmoc group and its release into the cellular environment. Strategies to mitigate this drawback include the design of different hydrophobic peptide sequences that do not require the presence of the Fmoc moiety to self-assemble, e.g., FFD [2]. However, these alternative sequences reduce the stability of the hydrogels. Herein we address these limitations by designing new peptide systems take advantage of the increased hydrophobic character of the non-natural aminoacids α,α -dialkylglycines, such as dibenzylglycine (Dbng). These unnatural amino acids were obtained by a synthetic strategy based on the Ugi reaction [3] and then solid-phase peptide synthesis was used to create the short peptides Dbng-Phe-Asp, Phe-Dbng and Dbng-Phe. These peptides were solubilized in alkaline water (using NaOH) and self-assembly was promoted by neutralization of the peptide solutions (using HCl). Furthermore, the generated nanoassemblies were crosslinked upon contact with cell culture medium triggering hydrogelation. The formation of the nanoassemblies was confirmed using SEM, CD and AFM. Of note, all the synthesised peptides generated supramolecular structures, however, only Phe-Dbng was able to form hydrogels within the tested range of concentrations.

Acknowledgments

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Sensor eletroquímico à base de filmes de hidrogel para rastreio de doenças cardiovasculares

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As doenças cardiovasculares (DCV) reúnem um conjunto de condições que afetam o coração e os vasos sanguíneos, como a doença arterial coronária, acidente vascular cerebral, hipertensão e enfarte agudo do miocárdio. Essas enfermidades comprometem seriamente a saúde dos pacientes, resultando em baixa qualidade de vida ou mesmo a sua morte. Segundo a OMS, em 2021, as DCV foram responsáveis por 23% das mortes no mundo [1]. Neste contexto, o diagnóstico precoce, particularmente em point-of-care, é fundamental para prevenção, tratamento eficaz e redução de complicações.

A mioglobina, uma hemoproteína de ~18 kDa presente no coração e nos músculos esqueléticos, destaca-se como um importante biomarcador cardíaco devido à sua rápida libertação após lesão cardíaca, sendo fundamental no diagnóstico precoce de enfarte agudo do miocárdio (EAM). Os níveis aumentam dentro de 1-3 horas após o início dos sintomas, atingem o pico 4-12 horas depois e normalizam após 24 horas [2]. Nos últimos anos, diversos biossensores eletroquímicos de alta sensibilidade e de rápida resposta foram desenvolvidos para detetar este biomarcador [3,4]. No entanto, muitos destes ensaios estão restritos a ambientes clínicos centralizados e/ou requerem bioreceptores caros e de baixa estabilidade a longo prazo, como os anticorpos, havendo ainda dilemas éticos associados à sua produção. Por outro lado, os polímeros molecularmente impressos (MIPs) são considerados atualmente uma alternativa promissora aos anticorpos utilizados nesses dispositivos, especialmente os filmes impressos de hidrogéis com elevada afinidade para biomarcadores proteicos. Os MIPs de hidrogéis são materiais responsivos a estímulos externos, como formação de ligação com analito, temperatura e pH, capazes de expandir ou contrair sua estrutura sem danificar a configuração das suas cavidades de ligação conferindo boa seletividade ao sensor e facilitando a remoção da molécula molde [5].

Assim, o objetivo deste trabalho é desenvolver um dispositivo eletroquímico inovador, miniaturizado e portátil, similar a um glucómetro, para a deteção seletiva e quantificação da mioglobina em soro humano, permitindo diagnóstico e monitorização precisos de doenças cardiovasculares em ponto de cuidado de forma minimamente invasiva recorrendo a eletropolimerização, em meio aquoso, para a produção de filmes finos de hidrogel nanoestruturado em superfícies condutoras de forma simples, rápida e com possibilidade de controle da espessura do filme, uma abordagem relativamente inexplorada até ao momento.

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Serum protein corona formed on gold nanoparticles highlights coagulation pathway alterations in HER2-positive breast cancer patients

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Breast cancer (BC) is a molecularly heterogeneous disease comprising five principal intrinsic subtypes: luminal A (LA), luminal B HER2-negative (LB-), luminal B HER2-positive (LB+), HER2-positive (HER2+), and triple-negative breast cancer (TNBC) [1]. Therapeutic strategies are primarily guided by the identification of these molecular subtypes; however, treatment failure still occurs in a subset of patients, underscoring the need for deeper insights into the genetic and molecular characteristics that define each subtype [2].

In the present work, we employed gold nanoparticles (AuNPs, 12.96 ± 0.72 nm) as a scavenging platform in combination with Sequential Window Acquisition of All Theoretical Mass Spectra (SWATH-MS) to quantitatively characterize serum proteome alterations across the distinct breast cancer subtypes. Differentially regulated proteins specific to each subtype were further analyzed using the bioinformatics tools STRING and PANTHER to delineate key molecular functions, biological processes, cellular origins, protein classes, and pathways affected by the inherent proteomic heterogeneity of BC. Notably, our analysis revealed a distinct profile of blood coagulation proteins in the serum of patients with HER2-overexpressing breast cancer.

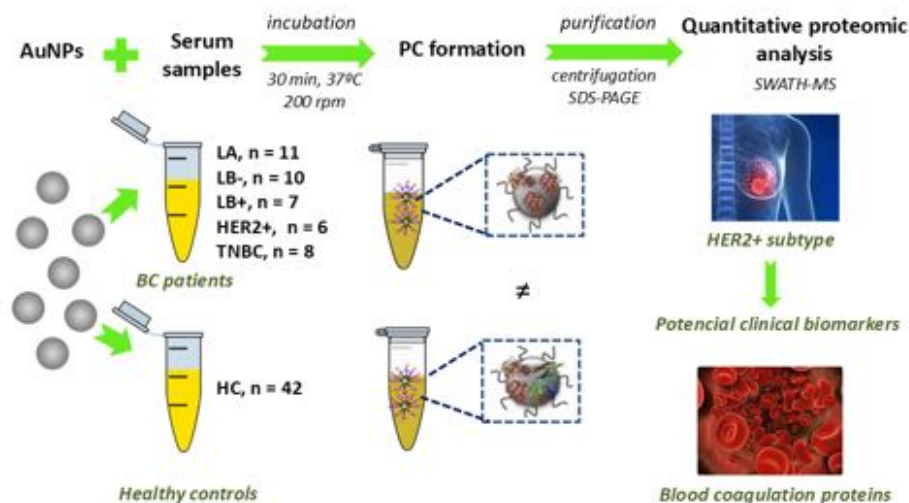


Fig.1. Simplified representation of the experimental procedure.

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Síntese de 9-((haloalcoxi)fenil)-6-cianopurinas

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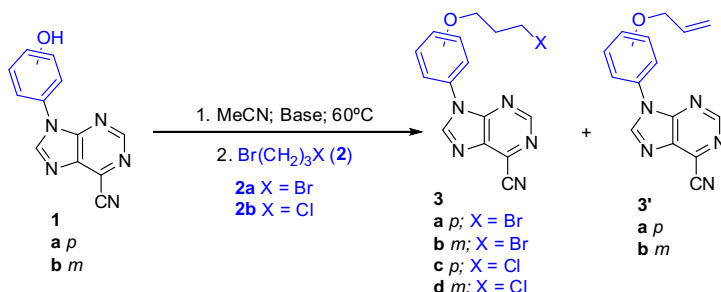
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A leishmaniose é uma doença parasitária que se encontra entre as principais doenças tropicais a nível global, com milhões de pessoas infetadas, centenas de milhares de novos casos, e dezenas de milhares de óbitos reportados, por ano [1-4]. Esta doença é especialmente nefasta e endémica em países subdesenvolvidos ou em desenvolvimento, com fracas condições sanitárias e malnutrição disseminada, bem como o é para hospedeiros imunocomprometidos [2-4].

A procura de novos fármacos antiparasitários é de extrema relevância, pois os disponíveis no mercado são caros, pouco eficazes e consideravelmente tóxicos, para além de serem difíceis de administrar [5-7]. A comunidade científica tem vindo a investir na procura de novos fármacos para esta doença, sendo que na literatura surgem os heterociclos de nitrogénio, em particular purinas, como potenciais núcleos para desenvolvimento de novos fármacos antiparasitários [7].

Anteriormente no grupo de investigação foi efetuada a alquilação de 9-hidroxifenil-6-cianopurinas com dibromoetano, obtendo-se os correspondentes produtos puros. Contudo, quando se aplicaram as mesmas condições reacionais usando o dibromopropano **2a** verificou-se a formação de um subproduto indesejado, o alqueno **3'a**. Neste trabalho foram estudadas as condições para gerar seletivamente 9-((haloalcoxi)fenil)-6-cianopurinas a partir de 9-(hidroxifenil)-6-cianopurinas e dihaloalcenos. Como dihaloalcenos foram usados o dibromopropano e o 3-bromo-cloropropano e como purinas foram usados os derivados contendo as unidades 3-hidroxi ou 4-hidroxifenil em N9 do núcleo de purina. Os produtos desejados **3** foram obtidos puros por reação de 9-hidroxifenil-6-cianopurinas com 1-bromo-3-cloropropano **2b** (Esquema). Todos os resultados serão apresentados.



Esquema

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Sustainable cosmetic formulations: anti-aging and depigmenting cream enriched with winemaking by-products

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Valorization of winemaking residues in the last decades has been increasingly pursued, in line with the principles of sustainable development and the circular economy. This approach is gaining relevance, as the incorporation of such by-products can be directed toward therapeutic and cosmetic applications. Its sustainable nature enhances consumer appeal and positions these strategies as highly promising within the cosmetic and pharmaceutical industries. Moreover, in cosmetics, the attractiveness of these formulations is further reinforced by their potential to counteract age-related skin barrier degradation and hyperpigmentation.

The main objective of this work was to evaluate the phenolic composition and antioxidant capacity of grape stems from a white cultivar, *Vitis vinífera* L. cv. 'Rabigato'. Phenolic quantification included the determination of total phenols, ortho-diphenols, and flavonoids, while antioxidant capacity was assessed through ABTS, FRAP and DPPH assays. Results revealed a diverse phenolic profile and significant antioxidant activity in the stem extract. Furthermore, bioactive compound identification was performed using HPLC - MS, where quercetin 3,7-di-O-glucoside and viniferin were identified as major constituents, representing two key classes of phenolic compounds.

In a subsequent step, and with a view to the valorization of these residues in cosmetic formulations, the extracts were incorporated at different concentrations (0, 0.33, 0.99, and 1.64%) into a cream base. Phenolic content and antioxidant capacity of the resulting formulations were evaluated using the same methodologies applied to the original stem extracts. In addition, their anti-aging and depigmenting potential was assessed through elastase and tyrosinase inhibition assays, respectively. The results showed that inhibitory activity emerged only from the concentration of 0.99%, which proved to be the most effective, whereas higher extract incorporation (1.64%) led to reduced activity, possibly due to saturation effects or pro-oxidant phenomena. These findings highlight the potential of grape stem extracts as functional ingredients in cosmetic formulations, while also emphasizing the importance of dose optimization to maximize bioactivity.

Synthesis and *in vitro* evaluation of activatable peptide probes for MMP-9

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Fluorescence-based optical imaging is widely applied in biochemical assays due to its high sensitivity and rapid response [1]. In particular, activatable fluorescent probes significantly enhance the signal-to-background ratio, providing reliable platforms for tracking protease activity. A promising design strategy relies on energy transfer between a donor (fluorescent dye) and an acceptor (fluorophore or chromophore) located at the termini of a peptide substrate. Following enzymatic cleavage, these probes undergo a transition from the quenched (“off”) state to the fluorescent (“on”) state, generating a signal directly proportional to enzyme activity [2].

Here, we report the synthesis of peptide-based activatable FRET probes targeting matrix metalloproteinase-9 (MMP-9), a protease involved in tissue remodelling and inflammation [3]. The decapeptides were synthesized by microwave-assisted solid-phase peptide synthesis (MW-SPPS) and functionalized with a suitable near-infrared FRET pair (Figure 1).

The probes were evaluated *in vitro* using recombinant human MMP-2 and MMP-9 enzymes. Results demonstrated selective fluorescence activation by MMP-9, confirming the feasibility of this approach for monitoring protease activity with high specificity and sensitivity.

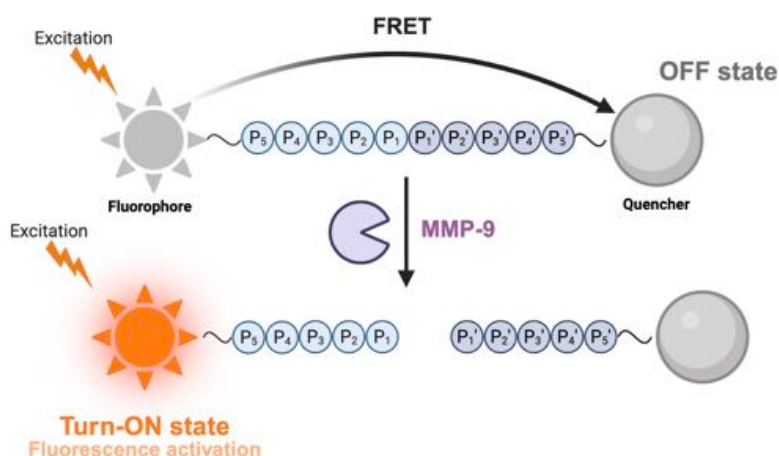


Fig.1. Schematic illustration of the MMP-9 activatable probe.

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Selective inhibitory activity of polyphenols against α -glucosidase: in vitro screening from yeast to human enzyme

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Type 2 diabetes *mellitus* has been an increasingly prevalent metabolic disorder, in which postprandial hyperglycaemia plays a critical role in disease progression. Maintaining near-normal glycaemia is therefore a primary therapeutic goal. One established strategy involves the inhibition of carbohydrate-hydrolysing enzymes, particularly α -glucosidase [1]. However, currently available inhibitors also target α -amylase, often causing undesirable gastrointestinal side effects. These drawbacks have driven the search for novel and selective α -glucosidase inhibitors with improved safety profiles [2,3].

Given the evidence supporting polyphenols as modulators of carbohydrate-digesting enzymes, a panel of five chalcones and thirteen related flavonoids was screened in vitro against α -glucosidase and α -amylase.

Enzyme activity was assessed spectrophotometrically by monitoring the hydrolysis of *p*-nitrophenyl- α -D-glucopyranoside (*p*NPG) to *p*-nitrophenol by α -glucosidase from *Saccharomyces cerevisiae*, and the hydrolysis of 2-chloro-*p*-nitrophenyl- α -D-maltotrioxide (CNP3) to 2-chloro-*p*-nitrophenol by porcine pancreatic α -amylase. Among the tested compounds, myricetin and genistein emerged as the most potent α -glucosidase inhibitors, with IC_{50} values of $14.5 \pm 0.6 \mu\text{M}$ and $17.7 \pm 0.7 \mu\text{M}$, respectively—far lower than that of the reference drug, acarbose ($267 \pm 12 \mu\text{M}$). Kinetic studies using Lineweaver–Burk plots indicated a mixed-type inhibition mechanism for both compounds. Furthermore, myricetin was found to be inactive against α -amylase, suggesting a more selective and potentially safer profile, whereas genistein displayed moderate inhibitory activity. However, the concentration required to achieve 50% inhibition was nearly 10 times higher than that required for α -glucosidase.

To examine translational relevance, myricetin and genistein were further tested against human α -glucosidase expressed in differentiated C2BBE1 cells using the same *p*NPG-based assay. However, neither compound showed significant inhibitory activity.

Although the inhibitory effects were not confirmed for the human enzyme, these findings underscore the potential of flavonoids as scaffolds for the rational design of next-generation selective α -glucosidase inhibitors.

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Potential 5-lipoxygenase inhibition by naturally inspired peptaibols

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Inflammation is a protective response to infection or injury, but if left untreated, it can lead to chronic tissue damage. Non-steroidal anti-inflammatory drugs (NSAIDs) and selective cyclooxygenase-2 inhibitors (COXIBs) are commonly used treatments; however, their long-term use is associated with gastrointestinal and cardiovascular side effects, respectively [1]. Moreover, complete inhibition of the COX1/COX2 pathways can redirect arachidonic acid toward the 5-lipoxygenase (5-LOX) pathway, potentially increasing leukotriene production, which are pro-inflammatory mediators involved in the pathogenesis of various chronic conditions. Therefore, directly targeting 5-LOX offers a promising alternative strategy.

Peptaibols, a distinctive family of protease-resistant peptides rich in hydrophobic residues, have been relatively poorly explored for anti-inflammatory activity. In this study, a library of over 30 natural and naturally inspired peptaibols was screened at 100 μ M for their ability to inhibit LOX in a cell-free assay. In general, natural peptaibols exhibited weak LOX inhibitory activity, reducing enzyme activity to ca. 70%. Among the semi-synthetic derivatives, those incorporating a Lys-Lys-Lys- β Ala motif at the *N*-terminus showed enhanced LOX inhibition. These modifications significantly improved not only the inhibitory activity but also the water solubility of the peptides. Remarkably, the acylation of the modified *N*-terminal motif with caprylic acid led to a further increase in LOX inhibition, with most derivatives achieving near-complete enzyme inhibition. For the most promising naturally inspired peptaibols, dose-response experiments were conducted to determine their IC_{50} values, using zileuton, the only FDA-approved 5-LOX inhibitor, as a benchmark.

In the model system employed, the optimized peptaibols displayed IC_{50} values that were ca. 5-fold lower than that of zileuton, suggesting that these molecules hold strong potential as leads for the development of novel anti-inflammatory agents.

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Biomedical applications of green carbon nanomaterials from chestnut waste

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Carbon nanomaterials (CNMs) represent an emerging class of nanostructures characterized by excellent biocompatibility, lower cytotoxicity, high photostability, and facile synthetic routes [1,2]. These properties feature their potential for diverse and advanced applications in pharmaceutical and biomedical fields [3-6].

In this study, CNMs were synthesized from agro-industrial residues derived from the chestnut processing industry, pointing to sustainable and value-added reuse of this biomass. The resulting nanomaterials were thoroughly characterized and evaluated for their biological activities, including antioxidants, anti-inflammatory, and cytotoxic effects.

The CNMs obtained were evaluated *in vitro*, showing a strong radical scavenging capacity and indicating potential anti-inflammatory activity. In addition, cytotoxicity assays were performed on both normal and tumor cell lines. Overall, these results highlight the promising potential of valorizing chestnut industry waste as precursors of functional CNMs with relevant biomedical research and applications.

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Exploring a sustainable method for the synthesis of cyclic imides

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Cyclic imides are a versatile class of compounds with applications ranging from industrial uses in polymer and dye synthesis to broad biological activities, including antineoplastic, anticonvulsant, sedative, hypnotic, fungicidal, bactericidal, and insecticidal properties [1]. Thalidomide is a classic cyclic imide drug with diverse medicinal applications, particularly in the treatment of leprosy and multiple myeloma. In 2010, thalidomide was found to bind to cereblon, a protein that forms part of an E3 ubiquitin ligase complex [2]. The E3 ligase cereblon and its ligands have become important tools in targeted protein degradation technologies such as PROTACs (PROteolysis TArgeting Chimeras) [3,4], an emerging area of medicinal chemistry. Considering the wide applicability of cyclic imides, the development of new sustainable methods for their synthesis is of great interest.

In this work, the synthesis of cyclic imides was investigated through reactions of primary amines with succinic, maleic, and phthalic anhydrides. Optimisation studies were performed to achieve sustainable conditions using solid-state and solventless reactions. Alternatively, the use of imidazole as a benign solvent with amphoteric properties was also investigated. Reactions of succinic anhydride with selected amines were studied by ¹H NMR to assess the influence of their structural and electronic properties on reactivity. In one of these NMR studies, a unique reactivity of 4-aminopyridine was observed, assigned to the catalytic role of the aromatic nitrogen atom.

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Sustainable Valorization of Winemaking By-products: Antioxidant and Enzyme-Inhibitory Activities of Douro Grape Stems

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Grape stems, commonly regarded as by-products of the winemaking process, represent an underutilized source of bioactive compounds with potential for high-value applications. This study investigated the phenolic composition and antioxidant capacity of grape stems from twelve red grape varieties cultivated in the Douro Demarcated Region, with the aim of exploring their suitability as natural ingredients for cosmetic formulations.

The samples were analyzed for total phenolic, *ortho*-diphenol, and flavonoid contents, and their antioxidant capacity was determined using ABTS, DPPH, and FRAP assays. Additionally, enzymatic inhibition assays were performed to assess the potential anti-aging and depigmenting activities of the extracts through the inhibition of elastase, tyrosinase, and hyaluronidase.

The results revealed that all grape stem extracts exhibited considerable levels of phenolic compounds and strong antioxidant activity, with notable differences among grape varieties. Several extracts demonstrated over 60% inhibition of elastase, tyrosinase, and hyaluronidase, suggesting promising anti-aging and skin-brightening potential.

Overall, the findings confirm that phenolic-rich extracts from grape stems possess significant antioxidant and enzyme-inhibitory properties, supporting their potential application as sustainable bioactive ingredients in natural cosmetic formulations. This work reinforces the importance of valorizing viticultural by-products, contributing to the advancement of circular economy practices and the development of environmentally responsible cosmetic products.

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Valorization of Grape Stems as a Source of Bioactive Compounds for Sustainable Cosmeceutical Applications

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The term cosmeceuticals were introduced in the 1980s to describe an emerging category of personal care products that combine cosmetic and pharmaceutical properties, providing therapeutic benefits such as improving the appearance and nourishment of the skin [1].

Today, growing concerns about the ecotoxicological risks associated with synthetic active ingredients have driven the cosmetic industry toward green cosmetics, which rely on bioactive compounds derived from plants or other natural sources [2].

Selecting bioactive ingredients from natural and renewable sources contributes to the development of value-added, sustainable products. This approach promotes the reuse and recycling of by-products, giving new life to waste materials and enhancing the market appeal of cosmetic formulations [3].

The wine industry generates substantial amounts of waste, both ecological and economic, as more than 30% of vinified grapes result in by-products such as grape pomace, seeds, skins, stalks, and stems. Like other plants, the grapevine (*Vitis vinifera*) produces a wide range of secondary metabolites — over 200 phenolic compounds have been identified — which are responsible for key organoleptic properties such as color, flavor, bitterness, and astringency, and play an important role in wine quality [4].

Phenolic compounds are known for their strong antioxidant, anti-inflammatory, and photoprotective properties, which make them promising candidates for use in cosmetic formulations aimed at promoting skin health [5].

The main objective of this study is to assess whether a wine industry by-product, namely grape stems, can serve as a potential source of bioactive ingredients for cosmetic formulations. To this end, colorimetric analyses were performed on three grape stem varieties (Tinta Roriz, Touriga Franca, and Sousão from the Douro Superior region) to determine their total phenolic, flavonoid, and ortho-diphenol contents, as well as their antioxidant capacity, using DPPH, ABTS, and FRAP assays.

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Synthesis and Characterisation of a Magnetic Nanocomposite Based on Exfoliated Graphite for Toxic Metal Removal

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Water plays a vital role in sustaining life on Earth, covering approximately 71 % of the planet's surface, though only about 1 % is directly available for human consumption, [1]. Moreover, demographic growth, extreme climatic events, and the limited efficiency of tertiary water treatments have intensified water stress in several regions worldwide. In this context, the treatment and reuse of wastewater and effluents containing toxic metals are essential to ensure sustainable water management. This need has motivated the development of efficient adsorbents for toxic metal removal, with adsorption standing out as a promising approach due to its high efficiency, operational simplicity, and relatively low cost [2].

Herein, we report the synthesis of a graphite-based magnetic nanocomposite designed for the removal of Pb(II) ions under conditions simulating industrial effluents. The nanocomposite was prepared via an electrostatic approach assisted by ultrasonic energy, using exfoliated graphite and magnetite nanoparticles as precursors. Exfoliated graphite, obtained by chemical exfoliation, provides a high surface area, chemical stability, and abundant active sites [3], favouring strong interactions with metal ions. The incorporation of iron oxide (Fe₃O₄) enabled facile recovery through magnetic separation.

The nanocomposite was characterized by Fourier-transform infrared spectroscopy, X-ray diffraction, zeta potential analysis, and electron microscopy, confirming the presence of both precursors and indicating promising adsorption behaviour. The effect of pH on adsorption efficiency was investigated, and the results showed that the nanocomposite outperformed its precursors, achieving superior Pb(II) removal, particularly under near-neutral pH conditions. These findings highlight the potential of graphite-based magnetic nanocomposites as efficient and cost-effective adsorbents for the remediation of lead-contaminated waters.

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Extração de hidrolisados proteicos de espinhas de bacalhau utilizando água subcrítica

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A produção mundial de pescado está estimada atualmente em cerca de 200 milhões tendo crescido nas últimas décadas a uma taxa média anual de 3%, quase o dobro do crescimento anual da população mundial para o mesmo período, e superior à taxa de crescimento de todos os outros alimentos de proteína animal (carne, laticínios, leite), de 2,1%. Cerca de 70% do pescado é processado antes da venda, produzindo uma quantidade significativa de resíduos [1]. Entretanto, apenas 20% destes subprodutos são aproveitados, principalmente para alimentação animal, sendo o restante depositado em aterros ou incinerado com óbvios danos ambientais, de saúde e económicos. No entanto, são uma fonte de substâncias valiosas como lípidos, proteínas, pigmentos, péptidos bioativos, aminoácidos e minerais [1,2]. Por meio de práticas sustentáveis, como a utilização de água subcrítica, é possível realizar a valoração deste subproduto por meio da extração destes componentes [3]. Neste contexto, o presente trabalho teve como objetivo avaliar diferentes condições de temperatura e pressão na extração de hidrolisados proteicos da espinha do bacalhau utilizando água subcrítica, e realizar a caracterização físico-química e bioativa dos mesmos. Foram avaliadas quatro diferentes temperaturas de extração (90; 140; 190 e 250 °C). Os extratos obtidos foram liofilizados e caracterizados em termos de rendimento, cinza, humidade, teor proteico, assim como atividade antioxidante pelos métodos de sequestro do radical DPPH e FRAP. A caracterização inicial dos resíduos utilizados demonstrou que o mesmo apresentava alto teor de humidade (70% b.h.), 11,21% (b.h.) de proteína (método de Kjeldahl), e 11,24% (b.h.) de cinza. O aumento da temperatura de extração resultou num aumento do rendimento até ao máximo obtido com 190 °C. A partir desta temperatura, pode ter havido degradação dos compostos, o que resultou em piores rendimentos (47% g/100 g de subproduto). Os rendimentos obtidos estiveram entre os 25% (g/100 g de subproduto) para a menor temperatura, e os 55% (g/100 g de subproduto) para 190 °C. Os extratos apresentaram alguma atividade antioxidante, pelos dois métodos testados, entretanto a extração realizada a 250 °C foi a que apresentou melhores resultados, 10,6±0,2 e 16,39±0,4 mg EAA/g extrato seco (Equivalente de Ácido Ascórbico) respetivamente pelos métodos DPPH e FRAP. Em relação ao extrato a 250 °C, os extratos obtidos na temperatura de 190 °C apresentaram apenas metade da atividade antioxidante pelo método DPPH e um terço pelo método FRAP. Desta forma, é possível realizar a extração de hidrolisados proteicos a partir das espinhas do bacalhau, utilizando água subcrítica. A bioatividade dos extratos obtidos demonstra o seu potencial para serem utilizados como ingredientes funcionais em alimentos e produtos nutracêuticos, assim como no desenvolvimento de embalagens ativas para conservação de alimentos.

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Can organic acids replace traditional leaching agents in e-waste recycling?

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The significant increase in the use of electronic devices containing valuable metals has led to the generation of huge quantities of electronic waste (e-waste), which are expected to reach 120 billion tonnes by 2050 [1]. This fact highlights the urgent need for sustainable e-waste management and metal recovery methods.

Although hydrometallurgy commonly uses strong leaching agents such as HCl, HNO₃, and H₂SO₄, these chemicals pose significant environmental impacts. Hence, organic acids like citric, acetic, and formic acids emerge as more sustainable alternatives [2,3].

This study investigates the use of sustainable (bio)organic acids to dissolve metal oxides (zinc and copper oxide), with a focus on optimizing metal recovery from e-waste by considering factors such as acid concentration, temperature, reaction time, and solid-to-liquid ratio.

According to preliminary results, more than 90 % of the metal oxides dissolve under optimum conditions. Atomic absorption spectroscopy and total reflection X-ray fluorescence spectrometry were used to quantify and compare the concentrations of dissolved metals.

These results confirm that organic acids can effectively replace inorganic acids, promoting sustainable metal recovery and supporting the circular economy.

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Sustainable Textile Coatings: Eugenol-Based Azo Dye Coloration

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Textile coating is an emerging and continuously developing technique in textile finishing, offering a versatile alternative to conventional dyeing methods [1]. Unlike traditional dyeing, which relies on the absorption of dyes into the fiber, coating allows for the deposition of functional molecules directly onto the fabric surface, enabling the introduction of specific properties while potentially reducing water consumption, chemical use, and environmental impact [2,3].

Azo dyes are among the most widely used colorants in the textile industry due to their wide range of colors and good stability; however, they are associated with toxicity and environmental concerns [4]. In this context, eugenol, a naturally occurring phenolic compound, was employed as a renewable precursor for the synthesis of azo dyes, thereby representing a more sustainable alternative [5]. The resulting eugenol-based azo dyes were deposited onto cotton knitted fabrics applied by knife-on-roller coating, that allows precise control of coating thickness and uniformity [6]. The coated textiles were evaluated for washing and light fastness to assess their performance under domestic and environmental conditions.

The results demonstrated good durability, indicating that eugenol-derived azo dyes can provide stable coloration when applied via coating. Furthermore, this study presents a sustainable alternative to conventional dyeing, combining potential environmental benefits with the flexibility of surface modification. Future studies will explore additional functionalities, such as antimicrobial activity, to further enhance the multifunctional properties of coated textiles.

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Refining aqueous extraction and purification methods for insect proteins

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The increasing global population has intensified the demand for sustainable protein sources [1]. Insects are promising candidates, with high protein content, nutrient richness, low environmental impact, and cost-effective production [2]. However, consumer acceptance of insects remains a challenge in Western countries. Therefore, the protein extraction from insects and their usage into new food products may improve consumer acceptance.

A Design of Experiments (DOE) was applied to optimize parameters and maximize protein extraction yields from two insect species: *Tenebrio molitor* and *Acheta domesticus*. Isoelectric precipitation was conducted, followed by freeze-drying. Protein contents were quantified using the BCA assay. Under the optimized parameters (temperature of extraction, extraction time, and aqueous solution concentration), yields of extraction over 89% for *T. molitor* and 85% for *A. domesticus* were achieved. Protein recovery ranged from 50–90% as influenced by the liquid-to-solid ratio. Thus, a versatile strategy is proposed, making it possible to obtain solid or liquid food formats through straightforward changes in volumes.

This work highlights the potential of integrating insect proteins into novel food products as a sustainable strategy for global protein demands. Ongoing scale-up tests aim to assess industrial feasibility.

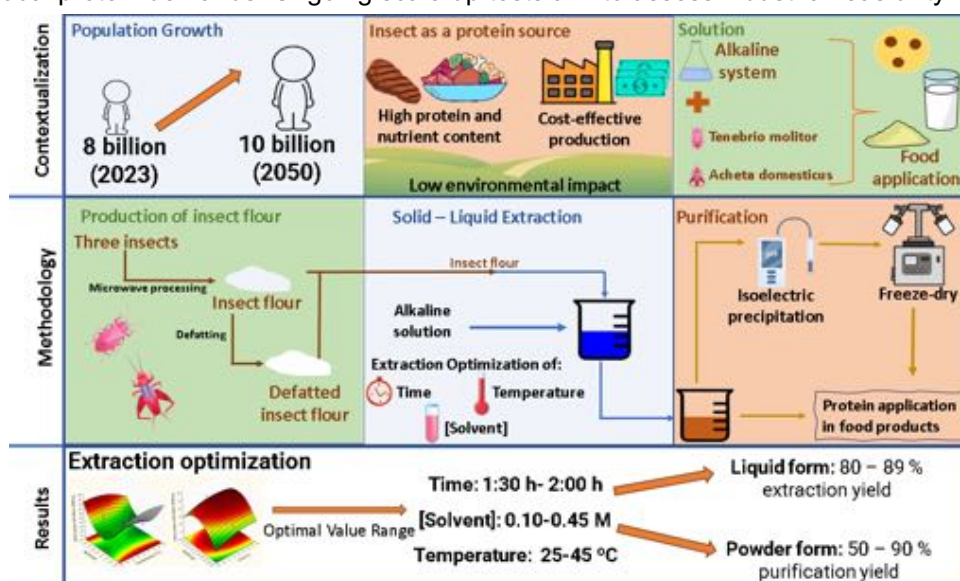


Fig.1. Graphical abstract.

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Luminescent Sodium Alginate Composites for Sustainable Sensor and Actuator Applications

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The growing demand for sustainable smart materials has stimulated interest in biopolymers capable of combining functionality with reduced environmental impact [1]. Sodium alginate (SA), a marine polysaccharide derivative of alginic acid comprised of 1,4- β -D-mannuronic (M) and α -L-guluronic (G) acids, provides a sustainable platform for the development of advanced functional materials [2]. To enhance its functionality, SA-based photoluminescent composites incorporating sodium tetra(2-thenoyltrifluoroacetate) europate (III) ($\text{Na}[\text{Eu}(\text{tta})_4]$) at 5–20 wt.% and the ionic liquid, IL, $[\text{Bmim}][\text{Eu}(\text{tta})_4]$ at 20 wt.% were developed by solvent casting. Structural analyses confirmed the integrity of the SA matrix, while thermal stability and electrical conductivity showed slight improvements. Mechanical tests revealed a plasticizing effect. The materials displayed strong photoluminescence after irradiated with UV light (Figure 1), suitable for anti-counterfeiting, and electromechanical bending actuators under voltage, demonstrating their potential for soft robotics applications. These results highlight europium–alginate composites as multifunctional, eco-friendly platforms bridging sustainability with emerging applications in security and robotics.

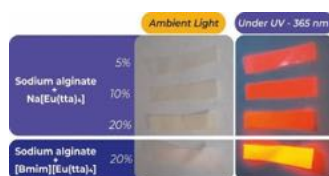


Figure 1. Scheme of SA-based composite films under ambient light (left) and under 365 nm-UV light (right) irradiation.

Acknowledgments

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Valorisation of juice pomace via enzymatic modification for infant food ingredients

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The juice industry faces a dual challenge: managing the environmental and economic costs of pomace disposal while overlooking its potential as a valuable resource. Pomace is a rich source of insoluble dietary fibre, phenolic compounds, and other nutrients, yet its incorporation into products is hindered by technological and organoleptic drawbacks [1]. Enzymatic modification emerges as a key asset in the strategy to transform this agro-industrial waste [2]. By specifically altering the structural properties of the fibre, this treatment can enhance its functionality, enabling its effective use in other products and providing a sustainable solution for the sector [3].

As such pear and apple pomace were subjected to an enzymatic treatment using a commercial enzyme solution Rapidase Fiber, which represents a commercial mixture of arabinofuranosidase, cellulase, and polygalacturonase. The phenolic content and profile were also determined using colorimetric methods and HPLC-DAD-ESI-MS/MS, as well as the antioxidant capacity using the ABTS (2,2'-Azino-Bis-(3-Ethylbenzothiazoline-6-Sulfonic Acid)) and DPPH (2,2-Diphenyl-1-Picrylhydrazyl) assays, and the reducing power (FRAP, Ferric Reducing Antioxidant Power). These assays were conducted on both the enzymatically untreated and treated pomace.

Thus, the main goal of this work is to formulate a fibre-enriched food product for infant nutrition, grounded in evidence from previous studies that underscore its potential benefits [4].

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Microwave-assisted autohydrolysis and membrane fractionation of *Castanea sativa* husks: targeted separation of phenolics and sugars for biorefinery applications

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Castanea sativa husks are an abundant lignocellulosic by-product in Southern Europe, mainly in Spain, Portugal, and Italy, which are currently underutilized, generating CO₂ emissions and resource loss [1,2]. Rich in lignin, cellulose, hemicelluloses, and bioactive polyphenols, they represent a promising resource for the extraction of useful compounds and microbial fermentation [3,4]. Green hydrothermal methods such as microwave-assisted autohydrolysis (MAA) enable the efficient and simultaneous release of sugars and phenolics under mild conditions and with low energy consumption [5]. This study analyzes the composition of these fractions after processing and membrane fractionation in order to identify those with the highest application potential.

Ground inner husks were treated using MAA at a solid–liquid ratio of 1:30 at 170 °C for 2.5 minutes. The extracts were filtered through membranes with cut-offs ranging from 100 to 1 kDa and analyzed using the HPLC DAD method. Phenolic acids (gallic acid, ellagic acids), hydrolysable and non-hydrolysable tannins were identified in the original extract, which showed 0.5825 g GAE/g dry extract and 0.4594 g/g dry extract (Trolox) antioxidant activity. The composition of the filtrated extract is shown in Table 1. After membrane filtration, the most active fraction was the 50–100 kDa range (0.9969 g GAE/g dry extract, 2.1365 g/g dry extract (Trolox)), which can be explained by the retention of tannins at this stage of membrane filtration and the corresponding visual characteristics of the permeate.

Tabela 1. Composition of the fractions after membrane filtration

Size of membrane, kDa	Permeate main compounds
100 kDa	Monosaccharides, oligosaccharides, phenolic acids (gallic, ellagic, coumaric)
50 kDa	Monosaccharides, oligosaccharides, phenolic acids (gallic, ellagic, coumaric), small tannins or fragments (monomeric/oligomeric tannins).
10 kDa	Monosaccharides, disaccharides, phenolic acids
1 kDa	Monosaccharides, disaccharides, phenolics

The membrane filtration system effectively concentrates antioxidant tannins in the 50–100 kDa fraction, while simultaneously allowing smaller molecules such as monosaccharides and disaccharides to pass through into lower MWCO permeates. This selective separation creates distinct fractions enriched either in sugars or in high-value phenolics and tannins, facilitating targeted downstream applications.

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Microparticle formulation from extracts obtained by sequential pressurized hot water extractions from *Laminaria ochroleuca*

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Brown algae contain important compounds such as fucoidan, florotanins and alginates, which have significant anti-inflammatory, antioxidant and anti-tumour properties. The extraction of bioactive compounds from these raw materials is a current topic of interest. In this context, pressurized hot water extraction (PHWE) is a sustainable and effective alternative, as it only uses water as a solvent and heat as an energy source [1].

This study sought an alternative that would allow the extraction of various types of compounds from *Laminaria ochroleuca*. Since, after an initial extraction using this method at 150 °C, an extract rich in fucoidan was found, based on its sulphate content, which is associated with biological activities such as antitumour activity [2]. In order to extract the phenolic compounds, a second extraction was carried out using the solid from the previous stage, observing a greater extraction of florotannins and antioxidant capacity at higher temperatures (220 °C).

For this reason, it was decided to use both extracts to produce microparticles using a Spray Dryer equipment [3], due to their potential applicability and their possibility of modifying the conditions to change the size of the final particle. To produce these microparticles, alginate, an abundant polymer in these edible brown algae, was added to the extracts separately and to mixture of both.

The microparticles were characterised by Scanning Electron Microscope (SEM) (Fig.1) and Fourier Transform Infrared Spectroscopy (FT-IR) to verify the morphology and the persistence of the functional groups in the final product.

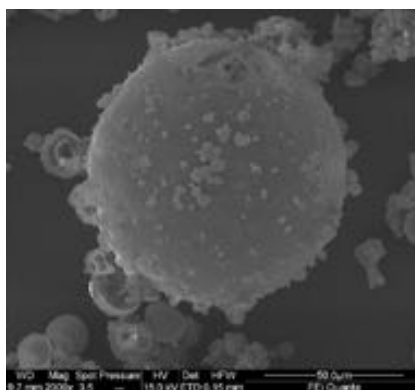


Fig.1. SEM image of a microparticle obtained with the first extract (150 °C).

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Podas de vid como fuente sostenible de lignina: extracción con disolventes eutécticos profundos (DES) y aplicación potencial en hidrogeles

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Según los datos de la Organización Internacional de la Viña y el Vino, en el año 2024 más de 7 millones de hectáreas de terreno en todo el mundo fueron destinadas al cultivo de uvas, con una producción superior a 220 millones de hectolitros de vino. Esto sitúa a la industria vitivinícola como una de las actividades económicas más relevantes a nivel global. Consecuentemente, millones de toneladas de residuos son generados anualmente en las bodegas, siendo los principales el bagazo de uva, las lías, los raspones y las podas de la vid. Se estima que por cada hectárea de viñedo se generan entre 2 y 4 toneladas de restos de poda de vid, los cuales suelen depositarse sobre el propio terreno como fertilizante o son quemados debido a su elevado poder calorífico. Sin embargo, dichas prácticas a menudo desencadenan problemas medioambientales y contribuyen a la infravaloración del potencial de este subproducto [1, 2].

Las podas de vid, al tratarse de material un lignocelulósico, están compuestas principalmente por celulosa, hemicelulosas y lignina. Esta última, es un polímero natural presente en las paredes celulares de las plantas leñosas, que ha despertado recientemente un gran interés como materia prima para el desarrollo de hidrogeles, debido a sus propiedades antioxidantes, antibacterianas y adhesivas [3].

El carácter recalcitrante de los materiales lignocelulósicos constituye uno de los principales problemas para su aprovechamiento. Resulta necesaria la aplicación de un tratamiento de deslignificación adecuado que permita el aislamiento eficiente y sostenible de la lignina sin comprometer su calidad de cara a futuras aplicaciones. En este contexto, los disolventes eutécticos profundos (DES) han atraído recientemente la atención de los investigadores debido a su capacidad para el aislamiento eficiente de la lignina de los materiales lignocelulósicos a la par que constituyen una alternativa verde a los disolventes orgánicos convencionales [4].

Este trabajo tuvo como objetivo la optimización de la deslignificación de las podas de vid para la recuperación de lignina con potencial aplicación en la elaboración de hidrogeles. Para ello, se estudiaron distintas formulaciones de mezclas eutécticas, así como dos estrategias de precipitación.

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Sustainable protein extraction from insects using deep eutectic solvents: a case study on *Tenebrio molitor*

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The increasing global demand for sustainable, high-quality protein sources has intensified the search for alternatives to conventional animal- and plant-based options. In this sense, insects have emerged as promising candidates for the extraction of high-value biomolecules, such as protein, as they offer nutritional, environmental, and economic advantages with potential industrial application [1]. Most studies still rely on extraction procedures with conventional solvents, which raise environmental and safety concerns. Deep eutectic solvents (DES) have attracted significant attention as sustainable alternatives, demonstrating strong performance in biomolecule extraction and offering biocompatible, low-impact solutions when properly designed [2].

The present work looks for biocompatible material-based DES for application in the extraction of proteins from *Tenebrio molitor* larvae powder. Among the various starting materials screened, two solvents, choline chloride:urea and choline chloride:levulinic acid, both at 1:2 molar ratio and 90% w/w water, were the most promising. Further optimization was conducted using response surface methodology, considering time, temperature, and solvent volume as key parameters to maximize protein yield. Also, to explore broader industrial applicability, the protein recovery was investigated.

Under optimal conditions, 57% of the extracted proteins were recovered with a purity of 73%. Ultimately, this study aims to serve as a stepping stone toward the development of sustainable and innovative industrial processes within the insect-based bioproducts sector.

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Carbon nanomaterials from chestnut lignocellulosic waste: high-pressure reactor and autoclave approaches

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Portugal is one of the largest chestnut producers in Europe, and the agro-industrial processing of chestnut generates substantial amounts of lignocellulosic waste, such as shells and husks. These carbon-rich by-products represent an underutilized biomass resource with a high potential for valorization [1,2].

Both pyrolysis and hydrothermal carbonization have been widely applied to transform biomass from various sources into valuable carbon-rich materials, aiming to enhance waste valorization, improve material properties, and promote eco-friendly resource utilization [3,4].

Recently, we reported the successful recycling of olive mill and tomato waste industries [5,6] and spent coffee grounds [7] as renewable and sustainable feedstocks for synthesizing carbon-based nanomaterials, demonstrating their applicability in sensing and biosensing platforms.

This work investigates the hydrothermal synthesis of carbon nanomaterials (CNMs) from the chestnut residues industry using a high-pressure reactor and autoclave system, aiming at scalable and sustainable conversion routes.

Several synthesis parameters (e.g., temperature, reaction time, and ratio of biomass vs. nitrogen source) were explored to enhance mass yield and control the morphological and optical properties of the CNMs.

The CNMs obtained were characterized by FTIR, UV-Vis, NMR, and fluorescence spectroscopy, confirming the presence of functional groups and tunable photoluminescent behavior. Morphological analysis was performed to correlate reaction parameters with particle size and distribution. The results demonstrate that chestnut industry waste can be effectively transformed into valuable CNMs, underscoring a promising approach for waste valorization, sustainable nanotechnology, and circular economy development in the Portuguese agri-food sector.

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Agro-waste-derived carbon dots: a green approach to biodiesel catalysis

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Biodiesel is a promising alternative to fossil fuels due to its renewable origin and lower pollutant emissions. However, large-scale production remains limited by the use of homogeneous catalysts, which are difficult to recover and require complex purification processes [1]. Heterogeneous catalysts represent a more sustainable and reusable solution, offering high selectivity and stability [2].

Carbon dots (CDs) are spherical carbon nanomaterials, usually with a diameter below 10 nm, characterized by their high surface area, excellent chemical stability, and ease of functionalization. They can be synthesized from low-cost precursors, including agro-industrial residues, through simple and scalable processes [3,4]. The presence of functional groups on their surface makes them particularly attractive as heterogeneous catalysts, allowing the anchoring of active species and enhancing reaction efficiency. This combination of physical and chemical properties makes CDs ideal candidates for transesterification reactions in biodiesel production [5].

In this work, CDs were synthesized from barley rootlets (CDs-BR), an agro-industrial residue from brewery industry, using ethylenediamine as a passivating agent via hydrothermal carbonization in a high-pressure reactor. CDs-BR were applied as heterogeneous catalysts in the transesterification of vegetable oil under conventional heating. Parameters such as catalyst loading, ethylenediamine concentration, as well as reaction time and temperature, were evaluated. Biodiesel samples were characterized by FTIR and ¹H NMR spectroscopy to confirm the formation of fatty acid methyl esters (FAMES).

The results obtained highlight the potential of CDs derived from barley rootlets as heterogeneous catalysts for more sustainable biodiesel production.

Acknowledgments

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Thymol-based azo dyes: synthesis and photophysical properties

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The development of azo compounds has attracted considerable attention owing to their multifunctional nature and broad spectrum of biological activities, including antimicrobial, anti-inflammatory, antiviral, and anticancer properties [1]. Furthermore, azo derivatives occupy a prominent position in the design of dyes, pigments, and advanced functional materials.

Essential oils (EOs) constitute complex and synergistic mixtures exhibiting remarkable antioxidant capacities, largely attributed to their phenolic constituents. These bioactive compounds are increasingly recognized as promising natural alternatives to synthetic antioxidants, particularly in the food and cosmetic industries [2].

Azo dyes based on thymol represent a promising class of bioinspired colorants combining the vivid chromophore properties of azo linkages with the biological and structural advantages of phenolic natural products. Thymol, a monoterpenoid phenol derived from *Thymus vulgaris*, provides a reactive aromatic nucleus that enables electrophilic substitution and diazo coupling, facilitating the synthesis of diverse azo derivatives. The introduction of the –N=N– functional group onto the thymol framework significantly alters its electronic structure, leading to intense coloration and tunable optical properties depending on pH and substituent effects. These dyes exhibit potential applications not only in textile and polymer industries but also in analytical sensing, antimicrobial coatings, and photochemical systems due to the inherent bioactivity and hydrophobic character of thymol [3-6]. Thymol-based azo dyes therefore represent an innovative bridge between natural product chemistry and functional dye design.

Considering these aspects, a series of azo dyes containing thymol were efficiently synthesized by coupling reactions of diazonium salts of different amines based on 3-aminobenzoic acid and thymol. The new thymol-based azo dyes were fully characterized by the usual analytical techniques. In addition, UV-vis absorption properties emphasizing on the solvent influence were also studied, and will be discussed.

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Rational design of new glucose-6-phosphate dehydrogenase inhibitors for cancer therapy

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Cancer remains the second leading cause of death worldwide, accounting for approximately 9.6 million deaths annually. Chemotherapy is the standard treatment for many cancers, but the emergence of drug resistance limits its effectiveness. The molecular basis of chemotherapy resistance is not fully understood, highlighting the urgent need for novel therapeutic strategies [1].

A hallmark of cancer is reprogrammed energy metabolism, where cancer cells preferentially metabolize glucose via glycolysis. In this context, glucose-6-phosphate dehydrogenase (G6PD), the rate-limiting enzyme of the pentose phosphate pathway (PPP), plays a central role. G6PD catalyses the conversion of glucose-6-phosphate into 6-phosphogluconolactone with concomitant NADPH production, thereby supporting biosynthesis and redox homeostasis [2]. Overexpression of G6PD has been correlated with cancer progression, including tumor growth, invasion, metastasis, and poor patient survival by equipping cancer cells with the metabolic flexibility required for survival and proliferation under hostile conditions [3].

As such, G6PD has proven to be a promising anticancer target. Notably, inhibition of G6PD not only suppresses tumor progression but also has demonstrated to help overcome chemotherapy resistance, offering a dual therapeutic advantage [4].

Recently, our group identified a series of novel small molecules with potent and selective G6PD inhibitory activity, alongside favourable in vitro ADMET properties. To accelerate their optimization, computational biochemistry provides powerful tools to understand ligand–target interactions at an atomic level. Approaches such as molecular docking can guide rational drug design, enabling structure-based optimization of target compounds against G6PD.

In this study, we employed molecular docking using the GOLD software with previously reported G6PD inhibitors to define the enzyme's active pocket and characterize key binding interactions [5,6]. The novel compounds developed by our group were then analysed within this framework to gain deeper insights into their ligand–target interactions at the molecular level. This approach provided a rational basis for the structure-guided design of new G6PD inhibitors with improved potency and selectivity, ultimately facilitating the development of more effective anticancer therapeutics.

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Green extraction strategies for *Ulva spp.*: effect of enzymatic and pressurised hydrolysis on protein and biocompounds recovery

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Marine macroalgae are increasingly recognised as valuable sources of bioactive compounds with applications in food, cosmetic, and pharmaceutical industries. Their composition rich in polysaccharides and proteins confers health-promoting properties. However, extraction efficiency strongly depends on the method employed, making it essential to compare conventional and advanced strategies to optimise recovery of targeted molecules from specific seaweed species such as *Ulva spp.*[1].

In this study, two food grade-compatible extraction approaches were evaluated for their potential to recover soluble matter and proteins from *Ulva spp.* powder: (i) enzyme-assisted extraction (EAE) using a carbohydrase blend, and (ii) solid–liquid extraction under high pressure and temperature (PHWE).

The enzymatic strategy is recognised for its mild and selective disruption of algal cell walls, enhancing solubilisation of carbohydrates and proteins while preserving bioactivity [2][3]. Conversely, PHWE enables rapid solubilisation of complex algal matrices, increasing extraction yield through thermal and pressure-assisted hydrolysis, although with potential effects on molecular integrity [4].

Results demonstrated that PHWE achieved a higher extraction yield of soluble compounds (41.3 %) compared with EAE (34.8 %). Regarding protein recovery, EAE yielded 8.45 mg/g (d.w.), whereas PHWE reached 34.7 mg/g (d.w.). These results highlight that although EAE may better preserve functionality, PHWE was markedly more efficient, recovering approximately 19% more soluble matter with fewer processing steps. Nevertheless, integrating EAE in sequential or combined workflows remains a promising strategy for developing seaweed-based functional ingredients and advancing sustainable biorefinery concepts.

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Effect of temperature on the recovery of phenolic compounds and antioxidant activity from *Melipona flavolineata* geopropolis using microwave-assisted extraction

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Geopropolis is a material produced by stingless bees of the *Melipona* genus from a mixture of plant resins, wax, salivary secretions, and soil. It is a natural resource of great importance for meliponiculture, especially in the Amazon region. Geopropolis extracts contain a variety of secondary metabolites, particularly phenolic compounds and flavonoids, which are associated with biological properties such as antioxidant activity [1]. The extraction of these constituents can be influenced by the method and operational variables employed, such as temperature, which can be a determining factor in extract quality. In this context, microwave-assisted extraction emerges as a promising technique due to its efficiency, selectivity, and lower environmental impact compared to conventional methods. The objective of this study was to evaluate the effect of temperature on the microwave extraction process of *Melipona flavolineata* geopropolis, focusing on extraction yield, phenolic compound content, total flavonoids and antioxidant activity.

The geopropolis sample was collected in the municipality of Belém (Pará/Brazil). The extraction was performed in a Monowave 450 reactor (Anton Paar, Austria), using a geopropolis and water ratio of 7.93:100 (w/w), in the range of 100 to 220 °C, for 5 min, under controlled stirring and power. The extracts obtained were evaluated for extraction yield, total phenolic compounds (TPC) [2], total flavonoids (TF) [3] content and antioxidant activity by TEAC [4], FRAP [5] and DPPH radical scavenging [6] assays. The temperature variation exerted a significant influence ($p < 0.05$) on the extraction yield (Fig. 1.), phenolic content (Fig. 2.) and antioxidant activity of geopropolis. The yield reached a maximum value of 5.2 at 200 °C, the phenolic content increased up to 2348.2 mg GAE/100 g at 200 °C and the flavonoid content was 463.3 mg QE/100 g at 200 °C. The reducing potential (FRAP) also increased significantly, but the TEAC and DPPH assays showed a progressive reduction at high temperatures, suggesting partial degradation of heat-sensitive compounds.

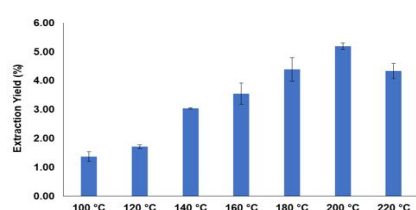


Fig. 1. Microwave-assisted extraction yield of *M. flavolineata* geopropolis.

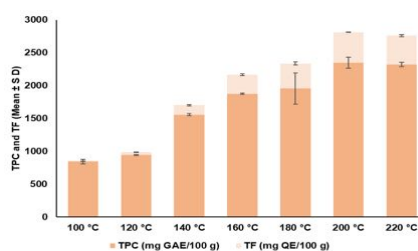


Fig. 2. Content of total phenolic compounds (TPC) and total flavonoids (TF) as a function of extraction temperature.

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Study of Laccase Activity in the Degradation of Poly(vinyl alcohol) Using Biobased Polyols

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Poly(vinyl alcohol) (PVA) is a water-soluble synthetic polymer widely used in polymers in liquid formulations (PLFs), including detergents, paints, and personal care products. Its high chemical stability makes it poorly biodegradable, leading to environmental accumulation and persistence. Microbial degradation of PVA has been reported but typically relies on pyrroloquinoline quinone (PQQ)-dependent dehydrogenases, which limits practical application. Cofactor-independent oxidoreductases such as laccase represent a more practical and sustainable alternative, although their catalytic efficiency toward unmodified PVA remains limited.

This study investigates the use of *Trametes versicolor* laccase for the oxidative depolymerization of PVA under mild conditions. Specifically, we assessed the effect of biobased polyols differing in carbon chain length and number of hydroxyl groups on laccase activity. Polyols with a higher number of hydroxyl groups, such as glycerol and xylitol, showed a stronger impact on enzymatic activity and were subsequently evaluated for their ability to support PVA depolymerization.

The work is still under development, with current efforts focused on expanding this strategy by testing redox mediators such as ABTS, syringaldehyde, and vanillin, and by applying a Design of Experiments (DoE) approach to optimize pH, mediator concentration, glycerol content, and reaction time. These findings highlight the potential of biosolvent-assisted enzymatic systems to improve PVA degradation, paving the way for scalable and sustainable strategies in polymer waste management.

Acknowledgements

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Designing Flavours and Fragrances by Biocatalysis: How Alcohol and Acid Choice Shapes Lipase Activity

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Lipases are key biocatalysts in the production of flavours and fragrances due to their ability to catalyse esterification and transesterification reactions under mild and eco-friendly conditions [1].

Enzymatic processes are particularly valuable for synthesising esters, the basis of many fruity and floral notes. Compared to chemical routes, lipase-catalysed methods are cleaner, generate fewer byproducts, and often qualify as “natural” under regulatory definitions, meeting consumer demand for sustainable and authentic products [2].

Among several ester-based compounds used for these applications, selecting the optimal lipase source for specific aroma synthesis remains a challenge. In the literature, several examples can be found; however, no reports directly compare the activity of different lipases with their reaction outcomes. Inspired by this lacuna, our team is dedicated to screening some of the most extensively studied lipases for industrial applications, specifically for the synthesis of a broad range of esters. In this way, the impact of alcohols and carboxylic acid length will be directly correlated with the enzyme's esterification activity.

Our findings revealed that the lipase source is indeed crucial, as its activity is directly influenced by the type of fragrance to be synthesised.

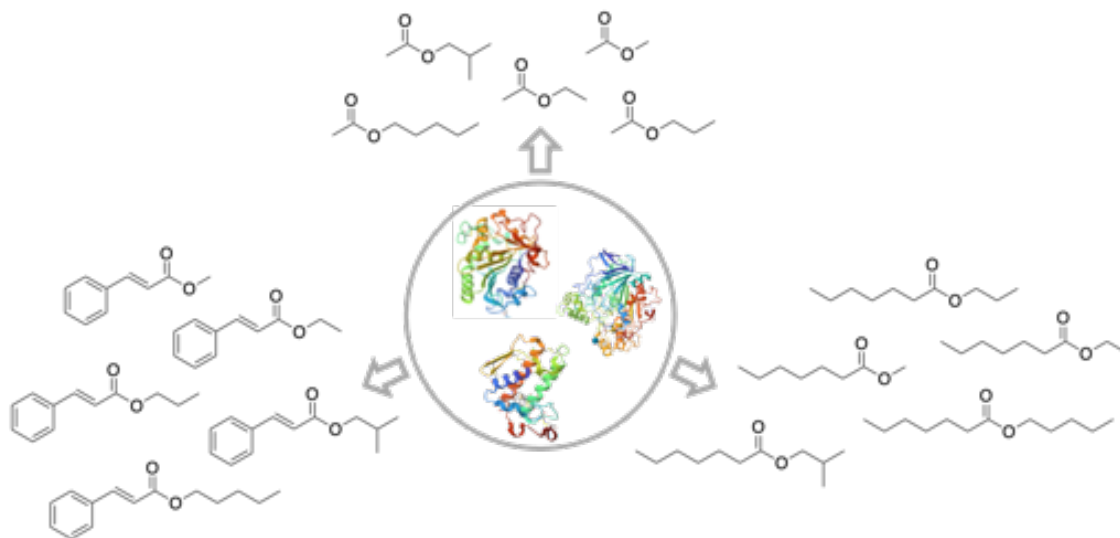


Fig. 1. The different esters that have been attempted to be synthesised by different lipases.

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Unlocking comprehensive valorization of hazelnut shells through microwave-assisted biphasic systems

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Agri-food residues represent an underexploited but highly promising biomass source for the development of sustainable value chains within a circular economy framework. Hazelnut shells (HS), which account for more than 50% of the total nut weight, stand out as a notable example. In 2022, global hazelnut production reached 1.22 billion tons [1], generating substantial amounts of HS that remain largely discarded or burned. Notably, HS are particularly rich in lignin (30-45 %), alongside hemicellulose (25-32 %) and cellulose (18-26 %) [2]. However, this high lignin content poses a significant challenge for efficient fractionation and further valorization of the main constituents, whose recovery requires effective fractionation strategies.

Traditional fractionation approaches typically focus on converting cellulose and hemicellulose into valuable products, neglecting lignin recovery, and leading to underutilization of biomass. Current strategies are moving towards an integrate use of biomass to minimize lignin recondensation, enabling the early stage of lignin while preserving cellulose and hemicellulose [3]. Building on this context, microwave (MW) heating offered a promising intensification tool, since rapid and uniform volumetric heating of polar molecules enhances solvent-biopolymer interactions and cell wall disruption. This results in shorter reaction times, reduced energy consumption and improved selectivity compared to conventional heating. In this context, biphasic water/1-butanol processing by MW-assisted heating offer a powerful single step approach for biomass fractionation. The limited miscibility of 1-butanol with water enables the separation into three distinct fractions: (i) an aqueous phase enriched in hemicellulose-derived products; (ii) an organic phase, rich in lignin; (iii) a cellulose-based solid [3].

The central contribution of this work lies in showing that biphasic microwave-assisted fractionation is not only technically feasible but therefore advantageous for agri-food residues such as HS. This strategy enabled the simultaneous generation of an organic stream containing high-purity lignin (up to 86 %), and aqueous phase containing up to 33 g/L of xylose and a solid constituted by 55 % of cellulose. Notably, this strategy also improves the structural properties of the processed solid, enhancing its suitability for subsequent enzymatic hydrolysis and fermentation to bioethanol, the most produced biofuel worldwide, with comparable energy content to gasoline with lower environmental impact [4]. These processes resulted in quantitative cellulose to glucose conversion at 96 h of enzymatic saccharification, and concentrations of 46-59 g/L, corresponding to yields of 95-98%. Overall, these results highlight that MW-assisted biphasic fractionation provides an integrated route for the valorization of HS, combining efficient lignin recovery, high sugar solution recovery and increased cellulose accessibility for subsequent bioprocessing.

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Phytochemical Analysis of *Camellia japonica* Husks and Leaves by GC-MS: Exploring bioactive compounds for future applications

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Camellia japonica is a tree from the Theaceae family, used in traditional medicine in Japan, China, and Korea for the treatment of diverse pathologies. The seed oil of this plant is already established as a medicinal component in the pharmaceutical and food industries [1], but its not the case of other tree parts. Therefore, further characterization of these parts is required to explore its potential applications.

In this study, an aqueous autohydrolysis extract of *Camellia japonica* husks obtained after non-isothermal treatment (140 °C) and two lipidic extracts of *Camellia* leaves were analysed using GC-ms to determine the volatile compounds. For this purpose, tetracosan (1 mg/ml) was used as a standard. *Camellia* leaves extracts were analysed using the Soxhlet and Folch method, two widely used methods, so as to see the specific differences in the extraction results of each method (Table 1). As to other raw materials - seed spices - both extractions produced comparable total lipid yields [2]. 25-hydroxycholesterol is a compound present in both extractions, which is known as an antiviral agent, at different concentrations. It is also effective in age-related disorders often linked to disrupted cholesterol metabolism, oxidative stress, inflammation, among others [3]. Other significantly important compounds that appear are ergosterol and lupeol, which, specially in the second case, present several biological activities, such as antiinflammatory or antibacterial [4]. Phenolic acids are significantly present in both plant parts.

Table 1. GC-ms analysis of the *Camellia* extracts

<i>Camellia japonica</i> husks aqueous extract		<i>Camellia japonica</i> leaves extracts			
		Soxhlet extraction		Folch extraction	
Compound	Relative content (%)	Compound	Relative content (%)	Compound	Relative content (%)
Glycerol	28.12	2-Naphtalenone	30.27	25-hydroxycholesterol	44.63
Quinic acid	19.41	Hexanodioic acid	17.16	Gallic acid	15.11
Malic acid	11.76	Ethylamine	16.66	Palmitic acid	14.35
Myoinositol	14.65	25-hydroxycholesterol	16.22	Methyl comate	8.12
Citric acid	5.57	Ergosterol	8.52	N-eicosane	7.88
Silanol	2.12	Octanodeoic acid	7.41	Neophitadien	6.27
Lactic acid	1.60	Lupeol	3.77	Oleic acid	3.63

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Removal of additives from plastic to enhance recycling

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Plastics are indispensable to modern society but pose severe environmental challenges due to their durability and resistance to natural degradation. Recycling offers a promising mitigation pathway, yet its effectiveness is often compromised by the presence of additives, particularly colorants, which reduce the quality of recycled materials. To address this limitation, this study explores an integrated recycling process based on solid-liquid extraction with mixtures of biobased solvents, designed to selectively remove colorants from common packaging polymers, including polyethylene (PE), polyethylene terephthalate (PET), and polypropylene (PP).

The process aims to extract colorants under mild conditions using mixtures of biobased solvents, while preserving the polymer matrix. Preliminary results indicate that such mixtures can be particularly effective; for example, one formulation enabled the simultaneous removal of blue and yellow colorants from post-consumer PET bottles. After additive removal, the polymers will be evaluated for their performance in both mechanical recycling and enzymatic depolymerization, directly assessing the benefits of colorant extraction on recycling efficiency.

This ongoing work highlights the potential of biobased solvent-based integrated processes to improve the quality and value of recycled polymers, paving the way for scalable and sustainable plastic waste management strategies.

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Propionate-based ionic liquids as solvents for challenging separation processes

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Complex separation processes are a central challenge in modern chemical engineering, as many valuable compounds are found in mixtures that are difficult to isolate efficiently. In the chemical industry, long-standing separation problems include the desulfurization and denitrification of fuels, the removal of aromatic hydrocarbons from aliphatics, and the separation of azeotropic mixtures. Conventional industrial processes designed to address these challenges typically require demanding operating conditions and often achieve only limited efficiency. Another example, which has existed for a long time but has only recently gained increased attention, involves terpenes and terpenoids. These compounds are widely abundant in nature and play an important role in the cosmetics, pharmaceutical, and food industries. However, they are usually present in complex multicomponent mixtures at low concentrations, making their separation and purification particularly challenging. The limitations of conventional separation methods have therefore encouraged the study of alternative solvents to perform these processes more effectively, particularly through liquid-liquid extraction. Among these, ionic liquids (ILs) have emerged as promising green alternatives to traditional organic solvents due to their negligible vapor pressure, and the possibility of tailoring their properties by combining different cations and anions [1,2].

In this work, the ILs 1-ethyl-3-methylimidazolium propionate ([C₂mim][Prop]) and choline propionate ([Ch][Prop]) were investigated as solvents for the separation of terpenes and fuels using both experimental methods and quantum chemical modelling with the COnductor-like Screening MOdel for Realistic Solvents (COSMO-RS) tool. Experimentally, inverse gas chromatography was applied to evaluate the potential of the ILs by determining the activity coefficients at infinite dilution, γ_{13}^{∞} , which were then used to calculate several separation factors, namely selectivities, S_{ij}^{∞} , capacities, k_j^{∞} , and solvent performance indexes, Q_{ij}^{∞} , at infinite dilution, for some important industrial mixtures. In general, [C₂mim][Prop] showed high S_{ij}^{∞} values for desulfurization and denitrification, suggesting potential for these separations. Regarding the fractionation of terpenes, high affinity was observed for alcohol terpenoids, moderate affinity for ketone terpenoids, and weak interaction with oxides. The best results were obtained for the separation of limonene/linalool, with high values for both, S_{ij}^{∞} and k_j^{∞} , indicating that [C₂mim][Prop] can act as solvents for the deterpenation of *Citrus* essential oils as well as for the valorisation of *Citrus* wastes.

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Comparison of microwave-assisted extraction strategies for Ulvan from *Ulva* sp.: combined microwave hydrodiffusion and gravity pretreatment vs. direct microwave-assisted extraction

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Over the past decade, scientific interest in compounds derived from marine resources has grown substantially, driven by their notable biological and physiological properties [1,2]. Ulvan is a high-value sulphated polysaccharide found in the cell walls of green seaweeds belonging to the Chlorophyceae class. It represents the primary structural polymer in *Ulva* species, where it can account for approximately 9–36 % of dry biomass [1]. It has been shown to exhibit a broad spectrum of biological activities, including antioxidant, anti-inflammatory, anticancer, antibacterial, and antiviral effects [3]. This study aims to explore the extraction of ulvan from the edible green seaweed *Ulva* sp. using various microwave-assisted techniques. It further investigates the molecular weight distribution of the extracted ulvan, the formulation of ulvan-based biofilms, and the assessment of their mechanical properties for potential biotechnological applications.

Two extraction strategies were implemented on *Ulva* sp. biomass. The first approach combined a pretreatment step using Microwave Hydrodiffusion and Gravity (MHG at 150 W for 25 min) with a subsequent Microwave-Assisted Extraction (MAE) using water as the solvent. In the second approach, ulvan was directly extracted via MAE, utilizing natural spring water as the extraction medium. MAE was conducted at 160 °C for 5 min with a 1:30 (w/w) solid–liquid ratio, 850 W power, 800 rpm stirring, and cooling to 50 °C. Ulvans were isolated from the soluble phase through ethanol precipitation, characterized for their molecular weight using HPSEC, and incorporated at 3% (w/w) with a 3:1 ulvan-to-glycerin ratio to formulate films, which were subsequently assessed for their viscoelastic behavior.

The combined MHG and MAE process yielded 26.8 % of ulvan, with molecular weight analysis revealing a prominent peak exceeding 786 kDa, comparable to that of commercial ulvans. In comparison, direct MAE using spring water resulted in a 24.86% ulvan yield. Molecular weight profiling indicated two distinct fractions with varying concentrations, spanning a range from approximately 783 kDa to 180 Da. The mechanical analysis revealed that both the elastic (G') and viscous (G'') moduli increased with rising frequency, indicating a predominantly elastic behavior of the matrix, as evidenced by G' consistently exceeding G'' .

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Valorisation of brown seaweeds through pressurized water extraction: influence of processing conditions on extract composition and bioactivity

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Brown seaweeds are gaining attention as a sustainable source of structurally diverse biopolymers with potential applications in biomedical, nutraceutical, and cosmetic sectors [1,2]. In this study, two species were investigated: *Rugulopteryx okamurae*, an invasive species proliferating along European coasts, and *Bifurcaria bifurcata*, native to the North Atlantic. The main objective was to evaluate how extraction parameters influence the yield and composition of fractions, with a focus on the recovery of functional biopolymers.

Pressurized hot water extraction (PHWE), a solvent-free and environmentally conscious method, was applied across a range of temperatures and algae conditions (fresh vs. dried). Following extraction, liquid and solid fractions were separated by filtration. Alginate was precipitated from the liquid phase using calcium chloride. The remaining liquid extract was analysed for protein and carbohydrate content, sulfate groups (as indicators of sulfated polysaccharides), phenolic compounds (phloroglucinol assay), and antioxidant activity (DPPH, TEAC, FRAP). The solid fraction, representing the insoluble residue, is currently under further compositional analysis.

The results demonstrated that both the extraction temperature and the physical condition of the algae significantly influenced the yield and biochemical composition of the extracts. The selective precipitation of alginate was successful under all tested conditions. The detection of sulfate groups in the alginate-free liquid fraction indicates the likely presence of sulfated polysaccharides. Furthermore, phenolic content and antioxidant activity varied notably with the extraction parameters, highlighting the sensitivity of these bioactive components to processing conditions.

These findings highlight the importance of controlled extraction strategies to tailor the composition and potential bioactivity of seaweed-derived extracts. The ability to influence extract characteristics through process conditions offers promising opportunities for the targeted development of functional materials. This work contributes to the broader goal of developing value-added applications from marine resources in alignment with sustainable and circular bioeconomy principles.

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Técnicas verdes de extracción de muestra para el análisis cromatográfico de compuestos carbonílicos en formulas nutricionales para bebés y adultos

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La reacción de Maillard y la peroxidación lipídica son procesos que tienen lugar en los alimentos sometidos a ultra alta temperatura (UHT) y son responsables de la generación de compuestos orgánicos volátiles como los carbonilos. Estos compuestos pueden causar estrés oxidativo a nivel celular relacionados por su relación con productos finales de glicación avanzada (AGE) y afectan a las propiedades organolépticas de los alimentos [1].

Las técnicas de microextracción minimizan el uso de disolventes y muestra, generan menos residuos, son rápidas, sostenibles y respetuosas con el medio ambiente. En este contexto se describen tres técnicas de microextracción de preparación de muestras verdes (microextracción en fase sólida, SPME; microextracción líquido-líquido dispersiva asistida por ultrasonidos, US-DLLME; microextracción por difusión de gas en modo inmersión, GDME) para el análisis cromatográfico (GC-MS y HPLC-UV) de carbonilos en formulas nutricionales para bebés y adultos (Fig. 1) [2-4].

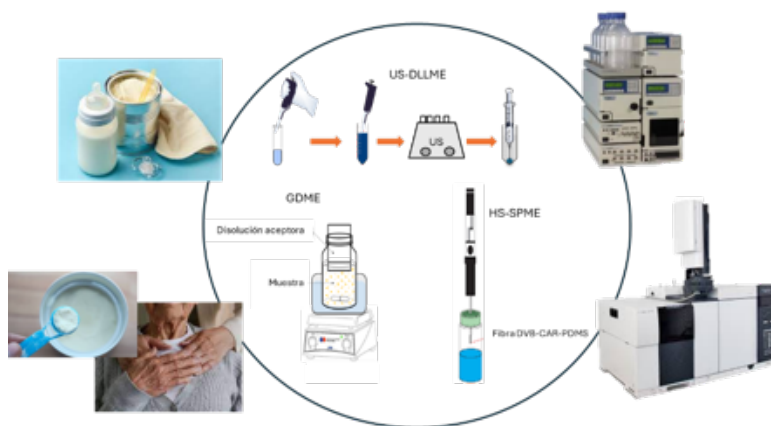


Fig.1. Técnicas verdes de microextracción utilizadas en el análisis cromatográfico de compuestos carbonílicos en formulas nutricionales para bebés y adultos

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Unraveling and comparing the effect of Li and Na-based salts on the ionic conductivity of methylcellulose solid biopolymer electrolytes

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A critical issue currently faced by researchers worldwide is the development of more efficient and environmentally friendly electrochemical devices. Solid biopolymer electrolytes (SBEs) have stood out in this context, due to the need for safer and more environmentally friendly devices. Among the cellulose-based biopolymers, methylcellulose (MC) can be considered for application as SBE because of its eco-friendliness, water solubility, and capacity for film formation [1]. However, some SBEs, including the cellulose-based ones, normally show low values of ionic conductivity [2]. To enhance ionic conductivity, several modifications of biopolymers have been proposed, including the addition of plasticizers and lithium (Li) salts. Nevertheless, despite lithium being widely applied in solid polymer electrolytes for energy storage, the limited reserves and the availability of lithium are concerning. Unlike Li, reserves of sodium (Na) mineral are more accessible at lower cost and uniformly distributed [3].

This work aims to study and compare the effect of Li and Na-based salts on the ionic conductivity of MC SBEs, since Na can be a viable alternative to the widely applied Li-based solid electrolytes. Lithium bis(fluorosulfonyl)imide (LiFSI) and Sodium bis(fluorosulfonyl)imide (NaFSI) were tested. The films were prepared using the solvent casting technique, where the salt was diluted in Milli-Q water, and mixture with the aqueous MC dispersion. Each sample was placed in an incubator (25 °C) until the film formation [3]. The films were characterized by SEM, XRD, FTIR, and DSC measurements. Electrochemical impedance spectroscopy analysis was performed, and the ionic conductivity (σ_i) values were estimated.

Table 1 shows the ionic conductivity values of the MC-based SBE films produced with different proportions of the salts LiFSI and NaFSI. As it can be observed, as the proportion of the respective salts increases in each sample, the ionic conductivity values also grow considerably. Regarding the samples with greater proportion of the salts, at room temperature, the ionic conductivity value is higher using Li salt ($1.43 \times 10^{-3} \text{ mS.cm}^{-1}$), compared to the sample with Na salt ($3.44 \times 10^{-4} \text{ mS.cm}^{-1}$). Even so, these values can be increased with the addition of plasticizers such as glycerol and polyethylene glycol. Overall, the rise in temperature significantly increased the σ_i values. In general, both investigated systems may represent promising alternatives for applications such as SBEs in energy storage devices. Further tests will be carried out to evaluate the effect of adding polyethylene glycol to the samples.

Table 1. Ionic conductivity values of the MC-based SBE films.

Sample	$\sigma_i \text{ (mS.cm}^{-1}) \text{ at } \sim 25^\circ\text{C}$	Highest $\sigma_i \text{ (mS.cm}^{-1})$	T highest $\sigma_i \text{ (}^\circ\text{C)}$
MC	Insulating	Insulating	-
MC 8:3 NaFSI	1.08×10^{-5}	7.46×10^{-5}	39.1
MC 4:3 NaFSI	3.44×10^{-4}	2.75×10^{-3}	46.0
MC 8:3 LiFSI	6.63×10^{-6}	3.98×10^{-5}	39.3
MC 4:3 LiFSI	1.43×10^{-3}	9.82×10^{-3}	49.8

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Increase of selectivity and chemical space of ABS with NaSCN additive: Case study of Membrane-Free Redox Flow Batteries

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The performance of membrane-free redox flow batteries (RFBs) relies critically on the selective partitioning of redox-active species between coexisting liquid phases. However, many ionic liquid (IL) + salt + water mixtures fail to form stable liquid–liquid equilibria (LLE) or display limited solute selectivity, restricting the accessible chemical space for biphasic electrolyte development. In this work, we investigate the ability of sodium thiocyanate (NaSCN) to both induce and modulate phase separation in ammonium sulfate ((NH₄)₂SO₄)-based aqueous biphasic systems (ABS) containing the ionic liquids [C₂mim]Cl, [C₄mim]Cl, [N₂₂₂₂]Cl, and [N₃₃₃₃]Cl.

Among the studied ILs, only [N₃₃₃₃]Cl naturally forms a biphasic system with ammonium sulfate. Remarkably, the addition of NaSCN in concentrations from 0 to 10 wt% progressively expanded the biphasic region, with 3.7 wt% NaSCN already sufficient to transform monophasic or solid–liquid systems into stable liquid–liquid equilibria. This behavior arises from anion exchange (SCN[−] ↔ Cl[−]) which alters ionic interactions and shifts the system composition across the binodal boundary.

The effect of NaSCN concentration on solute partitioning was examined using methyl viologen (MV), benzyl viologen (BV), TEMPO derivatives, and potassium ferrocyanide as molecular probes. The results indicate that NaSCN can significantly influence the phase selectivity and control the distribution of redox-active species, with improvements in partition efficiency observed in several of the investigated systems.

Overall, NaSCN acts as a dual-function additive, capable of inducing liquid–liquid equilibria and tuning the selectivity of ionic liquid–salt biphasic systems. These findings provide new insight into the molecular mechanisms governing ion migration and offer a flexible strategy to expand the library of selective aqueous biphasic electrolytes for membraneless redox flow battery applications.

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Tuning the Trade-Off between Viscosity and Selective Partitioning in Biphasic Electrolytes via Sodium Thiocyanate Addition

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Membrane-free redox flow batteries (RFBs) offer a cost-effective and durable alternative to conventional systems by removing ion-selective membranes. However, achieving efficient phase separation and selective confinement of redox-active species while maintaining suitable rheological properties remains a major challenge. In this work, we investigate the influence of sodium thiocyanate (NaSCN) on the phase behavior and partition of redox mediators in ammonium sulfate-based aqueous biphasic systems (ABS) containing various ionic liquids (ILs), specifically the quaternary ammonium salts [N₄₄₄₄]⁺Cl⁻, [N₄₄₄₄]⁺Br⁻, and the phosphonium salts [P₄₄₄₄]⁺Cl⁻ and [P₄₄₄₁₄]⁺Cl⁻.

Comprehensive analysis of binodal curves, ion partitioning, and thermophysical properties revealed that NaSCN strongly modifies the interionic interactions between IL and salt components. The introduction of thiocyanate triggers a selective anion exchange, where SCN⁻ partially or completely replaces Cl⁻/Br⁻, resulting in a pronounced redistribution of ionic species across the phases. The IL cations and SCN⁻ are driven to the upper, IL-rich phase, while hydrophilic ions such as sulfate and ammonium preferentially occupy the lower phase. This reorganization produces nearly complete segregation of the redox mediators, such as methyl viologen (MV) and 2,2,6,6-Tetramethylpiperidin-1-yl)oxyl (TEMPO), into opposite layers, significantly enhancing partition efficiency.

Simultaneously, NaSCN addition causes dehydration of the top phase, expelling water to the lower phase and increasing both the density contrast and viscosity of the system. The alkyl chain length of the IL cation further modulates these effects, controlling interfacial stability and mass transport.

Overall, the results unveil a key trade-off between viscosity and redox-species separation, which can be finely tuned through IL structure and NaSCN concentration. These insights provide essential design criteria for developing phase-stable, high-selectivity electrolytes suitable for next-generation membrane-free aqueous redox flow batteries.

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Deployment and Performance Analysis of Multi-Axis Piezoelectric Energy Harvesters in Urban Environments

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Wind-excited piezoelectric energy harvesters (PEHs) have emerged as promising devices for powering low-energy electronics and sensor networks using ambient wind-induced vibrations. While most previous studies have optimized PEH performance through wind tunnel experiments and CFD simulations [1], [2], their long-term behavior under realistic urban conditions remains insufficiently explored. Building upon our previous field study [3], which combined one month of in-situ measurements with machine learning (ML) models to predict the voltage output of a single harvester, the present work represents both the next stage and complementary expansion of our research.

The current measurement campaign involves a novel configuration combining three piezoelectric energy harvesters mounted on a single metallic bar, each separated by 120 °, to capture wind excitations from multiple directions simultaneously. The prototype is deployed on the roof of the Área Científica building at the University of A Coruña, being exposed to realistic urban wind conditions. To characterize the local wind field, two 3D ultrasonic anemometers are used for sampling the wind conditions at high frequency. The anemometers are connected via serial ports to a Raspberry Pi 4, so data can be recorded for the two anemometers simultaneously. In parallel, two Davis Vantage Vue anemometers are installed to record wind speed and direction. These devices provide the means for redundancy and cross-validation. As well as the wind properties, the power output provided by the set of wind-excited piezoelectric harvesters is continuously sampled. This arrangement enables high-resolution, synchronized monitoring of both aerodynamic and electrical signals in a realistic urban environment, where turbulence, flow interference, and variable wind angles play a crucial role in the energy conversion process. In this manner, the relationship between highly turbulent wind flow and the performance of the harvesters can be characterized.

The collected data will be used to analyze the influence of wind direction, turbulence, and local flow interactions on the voltage output of each harvester, generating a dataset that will serve to train ML models for predicting wind behavior and harvester performance under varying urban flow conditions. This work contributes to advancing the understanding of multi-axis PEH behavior in real environments and supports the development of data-driven design strategies for self-powered systems in urban applications.

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Advanced Smart Textiles Integrating Hybrid Carbon/Mo-V Sulfide Nanomaterials for Energy Storage

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The rapid expansion of the Internet of Things (IoT) market has stimulated the development of smart textiles with integrated energy storage capabilities [1]. Textile-based supercapacitors have emerged as promising power sources for low-consumption wearable devices namely in healthcare, sports, and military applications. Ideally, these systems should combine the high energy density of batteries with the long cycle life, high power density, and fast charging characteristics of conventional capacitors, thereby enhancing the overall efficiency of electrical energy storage [2].

In this work, textile-based flexible supercapacitors were fabricated on cotton substrates using hybrid nanomaterials composed of carbon nanotubes and metal sulfides as electrode components. The hybrids were synthesized via *in situ* functionalization of oxidized multiwalled carbon nanotubes (CNT-O) with Mo-V sulfides, employing optimized metal/sulfur molar ratios to favor the formation of high-purity sulfide phases. The structure, chemical composition, and morphology of the resulting hybrids were characterized by X-ray diffraction, Raman spectroscopy, X-ray photoelectron spectroscopy, chemical analysis, and scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy. Subsequently, CNT-O and the hybrid materials were deposited onto cotton fabrics, which were then assembled into asymmetric supercapacitor devices with a sandwich-type configuration using a solid-gel electrolyte. The asymmetric supercapacitor employing a hybrid of bimetallic Mo-V sulfide with CNT-O as the negative electrode and CNT-O as the positive electrode exhibited the best electrochemical performance, combining a high energy density of 1.11 W h kg^{-1} with a remarkable power density of 23.54 W kg^{-1} . These results demonstrate the potential of carbon/metal sulfide hybrid nanomaterials as efficient and flexible energy storage components for next-generation smart textiles.

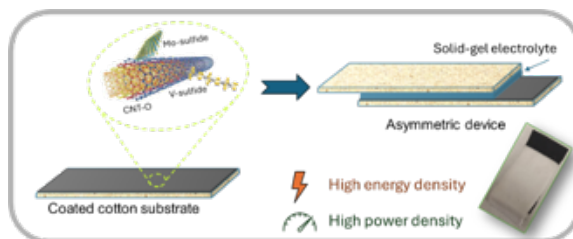


Fig.1. Schematic representation of the cotton substrate coated with the prepared nanohybrids and of the assembled asymmetric devices.

Acknowledgments: FCT/MECI (Fundação para a Ciência e a Tecnologia and Ministério da Educação, Ciência e Inovação) is acknowledged in the framework of the projects PTDC/CTM-TEX/4126/2021 (DOI: 10.54499/PTDC/CTM-TEX/4126/2021), UID/50006 – Laboratório Associado para a Química Verde - Tecnologias e Processos Limpos, LA/P/0008/2020 (DOI: 10.54499/LA/P/0008/2020), UIDB/04968/2020 (DOI: 10.54499/UIDB/04968/2020) and UIDP/04968/2020 (DOI: 10.54499/UIDP/04968/2020). MSN and JST acknowledge the work contracts through the ERC grant SelfEnergyDriver funded by the ERC.

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ThermoTwin-TCSCs: Dual-Side Coated Screen-Printed Thermally-Chargeable Textile Supercapacitors

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The growing demand for autonomous and flexible power systems in wearable electronics drives the need for lightweight, textile-integrated devices capable of both harvesting and storing energy. Thermally-chargeable supercapacitors (TCSCs) are promising, coupling ionic thermodiffusion with electrochemical energy storage to directly convert body or environmental heat into electrical energy [1]. Yet, conventional TCSC architectures rely on multilayer assemblies that reduce flexibility and scalability [2].

Herein, ThermoTwin-TCSCs were developed in a new single-substrate configuration where both the solid-gel electrolyte and the electrodes are directly deposited onto the textile (Fig. 1). The process involves dual-side coating of the electrolyte and screen-printing of conductive inks based on carbon nanotubes (CNT), graphene (GN) and their 1:1 mixture (CNT-GN), as well as their corresponding MnFe₂O₄-containing hybrids (HY), forming an entirely integrated and multifunctional energy textile device. Both symmetric and asymmetric architectures were fabricated, the latter integrating a carbon-based electrode with a hybrid ferrite counterpart to promote redox-assisted storage processes.

The electrochemical results revealed a dependence of the devices performance on electrode formulation. The hybrid asymmetric devices showed markedly higher energy densities than the corresponding symmetric all-carbon counterparts: GN//HY(GN) device exhibited 226% higher energy density than GC//GN (2.03 $\mu\text{Wh cm}^{-2}$ vs. 0.62 $\mu\text{Wh cm}^{-2}$), and the CNT-GN//HY(CNT-GN) device nearly doubled this output relative to CNT-GN//CNT-GC (3.31 $\mu\text{Wh cm}^{-2}$ vs. 1.62 $\mu\text{Wh cm}^{-2}$). This enhancement arises from the combination of two energy storage mechanisms: electrostatic charge accumulation at the electrode/electrolyte interfaces (non-faradaic) and faradaic redox reactions from the ferrite.

This work demonstrated a scalable route toward flexible dual-functional energy textiles through the ThermoTwin-TCSCs, where hybrid electrodes enhanced the energy density. Ongoing studies on thermal energy harvesting validated their self-charging capability, positioning these devices as a promising foundation for the next generation of self-powered smart fabrics.

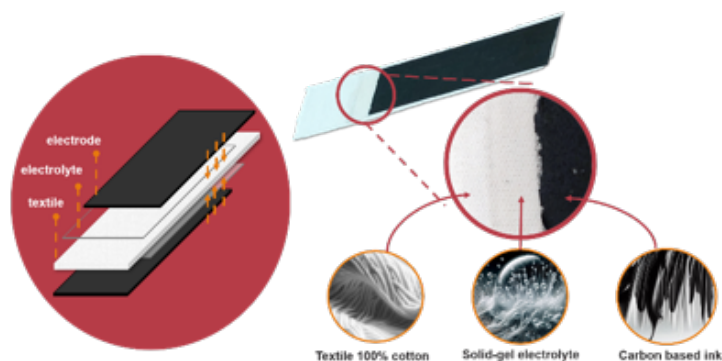


Fig. 1: a) Scheme of TCSC production method.

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Hybrid Integration of Water Evaporation-Induced Generators with Seawater Batteries for Enhanced Performance

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Seawater Batteries (SWBs) represent a promising solution for sustainable energy storage by utilizing the abundant sodium ions in seawater [1]. Central to SWB functionality is the NASICON (Na Super Ionic Conductor) separator, which facilitates sodium ion transfer between the anode and cathode. However, when submerged in water, the performance of NASICON can be diminished by humidity, reducing the overall efficiency of the SWB system [2]. To address this challenge, we propose the integration of Water Evaporation Induced Generators (WEIGs) with SWBs to harness renewable energy from water evaporation while mitigating performance losses in the SWB [3].

WEIGs, designed with porous membranes, can continuously generate up to 1 V through the natural process of water evaporation, sustaining this output over several months. By coupling WEIGs with SWBs, the system benefits from continuous supplementary energy generation that compensates for performance degradation due to environmental factors such as humidity. This hybrid setup not only supports stable operation but also enhances the overall energy harvesting and storage efficiency of the SWB.

In experimental setups, WEIG-SWB integration demonstrated a significant improvement in battery performance, maintaining stable voltage output even under prolonged submersion. The energy harvested by the WEIG balances the decline in SWB performance caused by NASICON's sensitivity to water exposure, thereby extending operational longevity and enhancing overall system efficiency. This integrated approach paves the way for more resilient and efficient SWB systems, with broad applications in marine and offshore renewable energy environments.

This work underscores the potential for WEIGs to elevate SWB performance, offering a robust solution to the challenges posed by humidity and water exposure in real-world applications, and advancing the development of sustainable energy technologies.

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From Structure to Capacitance: Probing Ethaline–Gold Interfaces via Molecular Dynamics

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Deep eutectic solvents (DES) are emerging as environmentally friendly alternatives to ionic liquids for electrochemical and nanotechnological applications [1–3]. In this work, molecular dynamics simulations were employed to investigate the interfacial structure and electrochemical response of the choline chloride : ethylene glycol (1:2) DES, known as ethaline, in contact with a monocrystalline Au(100) surface. The systems were examined across a temperature range of 313–343 K and under surface charge densities up to $\pm 24 \mu\text{C cm}^{-2}$. The analysis of density profiles, hydrogen bonding, and molecular orientations revealed that ethylene glycol dominates the innermost interfacial layer, followed by choline cations and chloride anions. Increasing surface polarization led to the formation of multilayer structures with alternation of ionic species, while both cations and hydrogen-bond donor molecules maintained a preferential parallel alignment to the electrode. The calculated differential capacitance curves exhibit a characteristic camel-shaped profile, consistent with experimental observations for choline chloride-based systems [4], indicating a complex interplay between molecular structuring and charge screening at the DES|Au interface. These findings provide molecular-level insights into the electric double layer behavior of deep eutectic solvents, relevant for optimizing their performance in electrochemical energy storage and surface modification technologies.

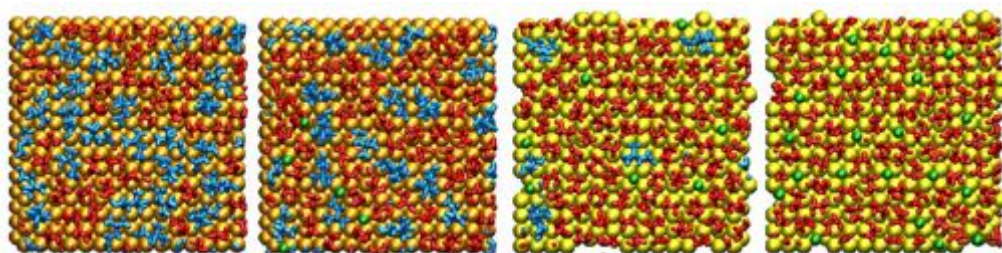


Fig.1. Snapshots of multilayer structures at electrode with cations, anions, and ethylene glycol molecules represented in blue, green, and red, respectively. Two left snapshots represent cathodes at various applied surface charges, while two right ones represent anodes at the same corresponding charges.

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Cork-Based Carbon Electrodes for Sustainable Supercapacitors

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Supercapacitors (SCs) are among the most promising energy storage devices to support the global energy transition. To minimize dependence on fossil fuels and reduce greenhouse gas emissions, these systems must be cost-effective and environmentally sustainable, ideally using renewable materials. Activated carbons (ACs) derived from local, renewable, abundant, and low-cost biomass offer a sustainable alternative to fossil-based electrode materials.

Cork stands out as an excellent bio-based resource—affordable, extremely abundant in Southern Europe, and readily available as a by-product of the cork industry (with Portugal being the world's largest producer). High surface area cork-derived ACs (ranging from 1000 to 2865 m² g⁻¹) can be obtained through conventional activation with KOH [1,2]. However, developing greener activation routes remains a key challenge, with increasing focus on sustainable chemical activation methods [3].

In this work, cork-derived ACs were synthesized using an eco-friendly, solvent-free physicochemical method employing K₂CO₃ as a benign activating agent and urea as a nitrogen source. The resulting materials exhibited specific surface areas of up to 1660 m² g⁻¹ and well-developed microporosity. Various synthesis parameters were explored, including mass ratios, heating ramps, final temperatures, durations, and gas flow rates. A comparison was also made with physical activation using CO₂.

The prepared ACs were evaluated as electrodes in symmetric two-electrode supercapacitor cells. Electrochemical characterization was conducted through cyclic voltammetry, galvanostatic charge–discharge tests, and electrochemical impedance spectroscopy. The activation method and synthesis temperature were found to significantly influence the materials' electrochemical performance.

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Safety and accident analysis in small modular reactors (SMRs) for controlled nuclear fission

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The current global situation places climate-driven by greenhouse gas emissions (GGE), population growth accompanied by a demand for improved quality of life across all áreas of human activity, and the need for reliable energy sources with low or zero carbon impact on the environment as three of the main challenges of this century at a global level. It must be noted that nearly 70% of GGE emissions are originated from the burning of fossil fuels for electricity and heat production. To achieve the goals established by the United Nations (UN) for NET-ZERO 050, as outlined in COPs and IPCCs from Rio de Janeiro in 1992, to Paris in 2015, and Dubai in 2023, there is a need for clean, reliable, firm and safe energy to complement the generation from renewable sources such as Wind and Solar photovoltaics, which are intermittent and Weather-dependent.

In this context, the use of Nuclear energy through Controlled fission emerges as a real option to fulfill this role, since it does not produce carbon emissions during its operational phase and has a low carbon impact throughout its entire lifecycle, and also contributing for energetic security for the electric power system. The Small Modular Nuclear Reactors (SMRs) [1,2] are emerging as one of the viable types for global deployment, due to six decades of the experience on Large-scale Nuclear Reactors existence in the world and has undergone innovations and improvements in the design, making them safer, cheaper, more efficient and producing less radioactive waste. The objective of this work is to present the initial neutronic/thermalhydraulic analysis study during normal operation of a similar Nuclear Reactor to SMR Integrated PWR South Korean SMART, fuelled with MOX of (TH,U)O₂ and (Th,Pu)O₂ cores. For neutronic analysis, the Monte Carlo N-Particle software MCNP6 was used, and for thermalhydraulic analysis the CFD code ANSYS CFX. The first objective in this study, is to evaluate SMART core hottest rod fuel and average rod fuel behavior at BOC Beginning-Of-Cycle), of three years (extended long cycle) using the codes, including the neutron flux distribution in the core, thermal power distribution (axial and radial), fuel temperature distribution in the fuel, clad and coolant, power peaking factors and DNBR (Departure from Nucleate Boiling Ratio) safety preliminary calculations. This ongoing work contributes to advancing the understanding of SMR safety and performance, supporting the future deployment of safe and sustainable Nuclear Technologies.

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M ^a João Rodrigues	Q07	155
M ^a Manuel Melo	QS11	174
M ^a Mercedes Fernández Feal	QIE17	339
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M ^a Pilar García-Santos	QF4	133
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M ^a Remedios Yáñez	QSU36	415
M ^a Teresa Oliveira	QIE7	146
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Mafalda Cunha	QA13	82
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Manuel Melle Franco	NN4	59
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Mara Costa	QS62	395
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María Rodríguez Padrón	QI1	134
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Miranildo Cabral da Silva	S27	442
Miriam Guimarães	QV9	429
Mohammed Benhamou	QAA37	301
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	S20	435
Nádia Santos	QS59	392
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Nerea Villarino	NN12	67
Nicola Armaroli	PL1	30
Nicolas Justus	BB2	37
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Nicolas Ruso	BB8	43
Nicole Ferreira	QSU13	208
Noelia González-Fernández	BB5	40
Noelia Vigo-Díaz	QS13 QS64	176 397
Nuna Ramos	Q09 Q021	157 359
Nuno Mateus	PL3	32
Nuno Santos	QF6	320
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Pablo Rodríguez	ED10 QIE22	259 344
Pablo Santiago Espiñeira	QAM17 QAM35	124 314
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Patrícia Santos	QAM23	302
Patrícia Varum	QP3	162
Patrícias Pais	NN16	262
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Paula Domínguez Carbón	QI4 QI10	137 326
Paula Oreiro-Martínez	QI6	139
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Pedro Almeida	QA12	81
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Stephanie Morais	QA6	75
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Thainara Ferreira	QSU14	209
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Tomás Barros	QA24	277
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Tomás Domingues	QS44	377
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Vanesa Romero	QA14	83
Vanessa Vieira	QS17	180
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Vasco Bonifácio	QS4	167
Verónica Bermudez	PL5	34
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Vitor Hugo Costa	QA1	70
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