A photograph showing a laboratory setup for biomass valorization. In the foreground, there is a clear glass beaker containing a green liquid, with a scale on the side ranging from 0 to 500 ml. To the left of the beaker is a small pile of fresh green leafy vegetables, likely spinach. In the background, there is a white bowl containing more of the same green leafy material. To the right of the beaker, there is a clear glass petri dish containing a small amount of dark green powder. The entire setup is on a light-colored surface.

A Path Towards Biomass Valorization Assisted by Green Chemistry Tools

6th PATH Spring Workshop

September 30th 2021

Meliã Ria Hotel & Spa, Aveiro, Portugal

BOOK OF ABSTRACTS

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BOOK OF ABSTRACTS

Organizing Committee

Alejandro Cifuentes

Ana M. Ferreira

Ana Rita R. Teles

Elena Ibáñez

Helena Passos

João A.P. Coutinho

The 6th PATH Spring Workshop will focus on biomass valorization assisted by green chemistry tools, namely the application of alternative solvents and techniques to facilitate the extraction, separation, and final application of bioactive and value-added compounds.

Scientific Program

Morning

8h30		Check-in (online & in-person)
8h55	<i>João A.P. Coutinho</i>	Welcome
Session I (Chair João A.P. Coutinho)		
9h00	Ana C. Sousa	Invited Lecture - Waste valorization and circular economy
9h30		<i>Discussion</i>
9h40	<i>Ana M. Ferreira</i>	Enhanced extraction of bioactive compounds from biomass using Ionic liquids: hydrotropic vs micelle-based effects
9h55		<i>Discussion</i>
10h00	<i>Isabela Sales</i>	Efficient extraction and improved anti-malaria activity of artemisinin using ionic liquids and salts hydrotropes
10h05	<i>José D.S. Martínez</i>	Blood-brain barrier permeability study of potential neuroprotective compounds recovered from plants and agrifood by-products.
10h10	<i>Jordana Benfica</i>	Efficient extraction of levodopa from biomass using aqueous solutions of eutectic solvents and organic acids (Online)
10h15		<i>Discussion</i>
10h25	<i>Felipe S. Bragagnolo</i>	Extraction of bioactive compounds using NADES and alternative extraction processes
10h40		<i>Discussion</i>
10h45	<i>João A.P. Coutinho</i>	A molecular perspective-guided solvent screening using COSMO-RS
10h55		<i>Discussion</i>
11h00		Coffe Break
Session II (Chair Elena Ibáñez)		
11h25	<i>Maria I.L. Neves</i>	A synergic combination with natural deep eutectic solvents and low-frequency and high-power ultrasound to extract genipin from <i>Genipa americana</i> L.
11h30	<i>Rodrigo T. Neto</i>	Impact of eutectic solvents utilization in the microwave assisted extraction of Proanthocyanidins from grape pomace
11h35	<i>Bárbara Socas-Rodríguez</i>	Application of natural deep eutectic solvents for the evaluation of pesticides occurrence in citrus and olive valorized by-products (Online)
11h40		<i>Discussion</i>
11h50	<i>Inês S. Cardoso</i>	Towards the valorization of orange peels: selective and integrated extraction of rutin and naringin using aqueous solutions of ionic liquids
12h05		<i>Discussion</i>
12h10	<i>Ana I. Valente</i>	Improved extraction of RuBisCO from spinach leaves using aqueous solutions of biobased ionic liquids
12h15	<i>Marguerita E. Rosa</i>	Integrated extraction and separation of beet leaves pigments using thermoreversible aqueous biphasic systems
12h20	<i>Inês L. D. Rocha</i>	Separation and purification of phenolic compounds present in lignin's oxidative depolymerization liquor using ARIZONA liquid biphasic systems and Centrifugal Partition Chromatography (CPC)
12h25	<i>Zully J. Suárez-Montenegro</i>	Enhancement of the neuroprotective potential of olive leaf extracts through a selective fractionation strategy (Online)
12h30		<i>Discussion</i>
12h40		Lunch

Afternoon

Session III (Chair Sónia P.M. Ventura)		
14h10	<i>Elena Ibáñez</i>	Invited Lecture - Biorefineries and circular economy
14h40		<i>Discussion</i>
14h50	<i>Alejandro Cifuentes</i>	Searching for natural bioactive compounds: global workflow in the Foodomics Lab
14h55	<i>Gerardo Álvarez-Rivera</i>	Metabolomics profiling strategies based on HRMS to identify bioactive compounds from agri-food by-products
15h00	<i>Alberto Valdés</i>	Lipidomics study of the neuroprotective potential of olive leaves by-products (Online)
15h05		<i>Discussion</i>
15h15	<i>Bruna P. Soares</i>	Alkanediols as efficient hydrotropes to enhance the aqueous solubility of syringic acid and their application for ultrasound-assisted extraction of phenolic compounds from juçara fruit
15h30		<i>Discussion</i>
15h35	<i>Sandra S. Silva</i>	Enhanced extraction of phenolic compounds from Kiwi waste using biobased solvents
15h40	<i>Chongting Guo</i>	Optimization of pressurized liquid extraction and bioactive potential evaluation of thinned peaches polyphenols from different drying treatments
15h45	<i>Norelhouda Abderrezag</i>	Bioprospecting through compressed fluids: Neuroprotective compounds from <i>Ammodaucus leucotrichus</i>
15h50		<i>Discussion</i>
16h00	<i>Eduarda S. Morais</i>	Dual-function (solvent and catalyst) eutectic-based systems to improve furfural production
16h15		<i>Discussion</i>
16h20	Coffe Break	
Session IV (Chair Gerardo Alvarez)		
16h50	<i>Sónia P.M. Ventura</i>	Blue biorefinery using ionic liquids
17h05		<i>Discussion</i>
17h10	<i>Meena Bisht</i>	Extraction and purification of Collagen type I using aqueous solutions of deep eutectic solvents from Atlantic Codfish (<i>Gadus morhua</i>) (Online)
17h15	<i>Mariam Kholany</i>	Recovery of bacterioruberin from marine archaea using eutectic solvents
17h20	<i>Bárbara M.C. Vaz</i>	A simple approach to recover chlorophylls from AmberLite™ HPR900 OH using tensioactive ionic liquids
17h25		<i>Discussion</i>
17h35	Closing Session	

Waste valorization and circular economy

Ana Catarina Sousa

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The generation and management of waste is extensively associated with the history of humankind. Throughout centuries, humans developed strategies to deal with the different types of waste generated. However, the increasing amount of wastes produced and released into the environment since the mid XX century generated unprecedented environmental and human health challenges. According to the United Nations Environment Programme (UNEP), waste management is a basic human need being also regarded as a basic human right.¹ As a way to mitigate the environmental, societal and health burden of waste generation while boosting resource efficiency, there is an urgent need to take action. In this line, several strategies for waste management have been adopted, including waste reduction/avoidance or waste valorization.² For that purpose, the implementation of effective waste prevention and management actions along with a transition from a “take-make-use-dispose” to a circular economy, in which waste is considered a valuable resource, is critical.² In this presentation, the waste generation and composition will be described and the evolution of waste management systems discussed in light of the major challenges and innovations in the field.

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Acknowledgments

This work was developed within the scope of the project CICECO-Aveiro Institute of Materials, UIDB/50011/2020 & UIDP/50011/2020, financed by national funds through the Portuguese Foundation for Science and Technology/MCTES and the Comprehensive Health Research Centre (CHRC), University of Évora, through the project UIDP/04923/2020.

Enhanced extraction of bioactive compounds from biomass using Ionic liquids: hydrotropic vs micelle-based effects

Ana M. Ferreira, Emanuelle L. P. de Faria, João A. P. Coutinho, Armando J. D. Silvestre, Mara G. Freire

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The use of bioactive compounds in different commercial sectors, such as pharmaceutical, nutraceutical, and cosmetic industries, has faced a significant increase in the last years, due to their beneficial biological activities, such as anticancer, antimicrobial, antioxidant, analgesic effects, among others). To overcome the concerns associated with the commonly used volatile organic solvents for their extraction from biomass, this work is focused on the potential of aqueous solutions of ionic liquids (ILs) as alternative solvents. More specifically, various aqueous solutions of ILs, with hydrotropic or surfactant characteristics, were evaluated to extract triterpenic acids (from apple peel) and syringic acid (from Rocha pear peel). The use of both classes of ILs allowed to understand which ILs are the best candidates for solubilizing/extracting the different types of bioactive compounds. The first study shows that surface active ILs are promising solvents to enhance the extraction of triterpenic acids (highly hydrophobic compounds) from biomass.¹ On the other hand, the second study shows the relevance of the hydrotropic concept as the main factor behind the improved performance of ILs aqueous solutions in the extraction of syringic acid (moderately hydrophobic compound).² In a nutshell, surface active ILs enhance the solubility of highly hydrophobic biocompounds, thus their extraction, while moderately hydrophobic biocompounds have a higher solubility in hydrotrope ILs. The developed studies demonstrate the potential of IL aqueous solutions as alternative solvents to extract different types of bioactive compounds from waste food, avoiding the use of the commonly applied volatile organic solvents, while valorizing food waste and contributing to a circular economy.

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Efficient extraction and improved anti-malaria activity of artemisinin using ionic liquids and salts hydrotropes

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The search for a cure for malaria led researchers to find artemisinin in the plant *Artemisia annua L.*, in the early 1970s. This drug has a high therapeutic effect against the disease that affected 229 million people in 2019.¹ Given its relevance, extraction methods for ART have been developed in the past years, yet resorting to volatile organic solvents, such as hexane and petroleum ether.^{2,3} Thus, it is essential to develop more benign and cost-effective extraction processes, while assuring the bioactivity of artemisinin.

In this work, aqueous solutions of ionic liquids (ILs) with hydrotropic character and the respective sodium salts were investigated as alternative solvents to extract artemisinin from *Artemisia annua L.* The solubility of artemisinin in aqueous solutions of ILs and salts at different concentrations at 30°C was first ascertained to identify promising extraction solvents. The results obtained show that both classes of compounds (ILs and salts) could enhance the solubility of artemisinin in aqueous media, increasing the solubility from 2.5- to 750-fold when compared to its solubility value in pure water. After identifying the most promising IL and salt, a response surface methodology was applied to optimize the extraction operating conditions (extraction time, concentration of hydrotrope, and temperature), in which extraction yields of artemisinin up to 0.68 and 0.64 wt.% were obtained with hydrotrope aqueous solutions of sodium and cholinium salicylate, respectively. Finally, the antimalarial activity of the extracts rich in artemisinin was confirmed against *P. falciparum*, demonstrating their potential in the treatment of malaria.

References

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Acknowledgments

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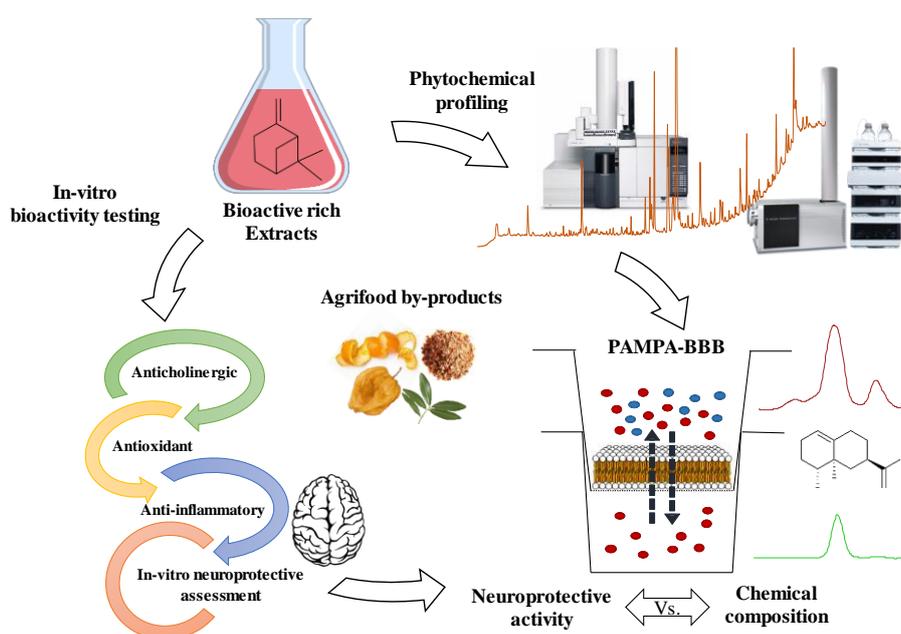
from CAPES and CNPq/Brazil (CAPES: Proc. 88881.189075/2018-01 and 88887.494428/2020-00.
CNPq: Grant 303089/2019-9 and Proc.438036/2018-2).

Blood-brain barrier permeability study of potential neuroprotective compounds recovered from plants and agrifood by-products

José David Sánchez Martínez, Gerardo Alvarez-Rivera, Rocio Gallego, Zully Jimena Suárez-Montenegro, Alberto Valdés, Elena Ibáñez, Alejandro Cifuentes

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Neurodegenerative disorders are a group of progressive neurological diseases characterized by cognitive impairment, loss memory and anxiety, among others symptoms.¹ Alzheimer's disease (AD) is consider the main neurodegenerative disorder affecting 50 million people in 2018, and this number of cases can be increased to 152 million by 2050.² AD neuropathology mechanism still unclear, however, several findings placed amyloid-beta ($A\beta$) plaques aggregation, neurofibrillary tangles (NFT), oxidative stress, neuroinflammation and cholinergic hypothesis as principal hallmarks of AD.³ In fact, nowadays there is no effective cure for AD and the palliative treatment basis on acetylcholinesterase (AChE) and butyrylcholinesterase (BChE) inhibitors.^{1,4} For this reason, during decades several researchers consolidate their efforts in elucidate neuroprotective properties of bioactive compounds from plants extracts.^{5,6} Agricultural and food industry generate several tons of wastes that can be considered as a renewable and low cost source of neuroprotective compounds.⁹ The cross through the blood brain-barrier (BBB) is one of the main drawback of bioactive compound to achieve brain tissue to establish their neuroprotection capacities. Parallel artificial membrane permeability assay for the blood–brain barrier (PAMPA-BBB) was firstly used by Di et al. in 2003,¹⁰ represents a low cost high-throughput non-cell-based and reproducible permeability test ideal for novel drugs or therapeutic bioactive compounds.¹¹



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Acknowledgments

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Efficient Extraction of Levodopa from Biomass Using Aqueous Solutions of Eutectic Solvents and Organic Acids

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Levodopa is an amino acid commonly used in the treatment of Parkinson's disease found in several plants, such as *Mucuna pruriens*.¹ Aiming at finding more sustainable solvents and develop efficient extraction processes, in this work, aqueous solutions carboxylic acids (acetic (AA), propionic(PA), citric(CA), glycolic(GA), and lactic acid(LA)) and (poly)alcohols (ethanol (EtOH), ethylene glycol(EG) and glycerol(G)) and aqueous solutions of eutectic solvents (ES) combined with cholinium chloride ([Ch]) were studied for the extraction of levodopa from *Mucuna pruriens* seeds. An initial screening with aqueous solutions of these compounds was conducted at 50°C, with an extraction time of 90min at a solid/liquid (biomass/solvent) ratio of 1:10. Based on these results, the ES [Ch]Cl:LA (1:2) and CA aqueous solutions were identified as the best solvents and experimental conditions such as the temperature, solid–liquid (solvent/biomass) ratio, and concentration in aqueous solutions were optimized by a response surface methodology, with the aim of maximizing the levodopa extraction yield. The recovery of levodopa from the [Ch]Cl:LA (1:2) and CA aqueous solution was achieved using an ion exchange column, allowing the recovery of approximately 87% for [Ch]Cl:LA and 84% for CA aqueous solution of levodopa. The solvent was shown to be reusable in three successive extraction cycles, with no significant losses in the extraction efficiency of levodopa. The results here obtained show that eutectic mixture [Ch]Cl:LA (1:2) (9.9 ± 1.0 wt%) and CA aqueous solutions (9.2 ± 0.1 wt%) can lead to the effective extraction of levodopa from seeds of *Mucuna pruriens*, serving as basis for the development of more effective and environmentally friendly processes to recover natural products with therapeutic properties.

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Extraction of bioactive compounds using NADES and alternative extraction processes

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Natural Deep Eutectic Solvents (NADES) were first mentioned in 2011 years ago by Choi et al.¹ In this past decade, many works have applied the potential of NADES in many areas and purposes. Extraction, cryoprotection, enzyme reactions, CO₂ capture, chromatography separation, and other applications have been developed using NADES.² Different natural compounds, such as amino acids, choline, sugar, organic acids, and prenol lipids, in addition to different molar ratios of such substances, modify NADES properties and increase their range of uses. Quoted applications emerge since NADES were discovered as the “third liquid phase in organisms”, supporting physiological processes of drought resistance, germination, enzymatic reactions, and storage.¹ The latter effect presents an interesting observation to the extraction of bioactive compounds since NADES can store high contents of specialized metabolites in plants. Perform extractions using NADES with alternative techniques, as Pressurized Liquid Extraction (PLE), becomes a potentially sustainable and powerful approach to generate bioactive extracts. In summary, since PLE is an efficient technique and uses less solvent, and most NADES are considered nontoxic and environmentally friendly, a greener process could be feasible by combining such approach.

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Acknowledgments

The authors would like to thank the São Paulo Research Foundation - FAPESP (2020/09500-0, 2018/21128-9, and 2017/06216-6) and the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil - CAPES (Finance Code 001).

A molecular perspective-guided solvent screening using COSMO-RS

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Trial and error remains the most common method for selecting the appropriate solvent for extracting natural products, particularly when dealing with novel and poorly studied solvents, such as deep eutectic solvents (DES). Conductor-like Screening Model for Real Solvents (COSMO-RS) is a quantum chemistry-based thermodynamic model that can be used as a screening tool to choose solvents for the extraction of biocompounds from natural sources.¹ For any compound, its solubility in a solvent is inversely proportional to its activity coefficient (γ) in the system. Thus, COSMO-RS was used to predict the activity coefficient of some target molecule at infinite dilution (γ_∞). Through a *Matlab* script, the ability of 1372 DES in solubilizing two antioxidant compounds from Rosemary (*Rosmarinus officinalis* L.) was evaluated. The results were depicted as contour plots, where the color represents $\ln \gamma_\infty$, Figure 1. Therefore, COSMO-RS allows a quick and qualitative *in silico* evaluation of the solubility of bioactive molecules in a large number of solvents, reducing the number of experimental at the laboratory.

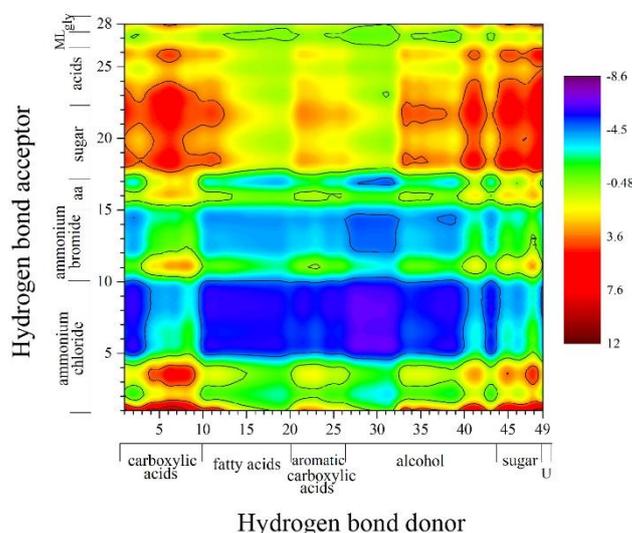


Figure 1. Predicted $\ln \gamma_\infty^{\text{solute}}$ in DES using COSMO-RS.

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A synergic combination with natural deep eutectic solvents and low-frequency and high-power ultrasound to extract genipin from *Genipa americana* L.

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Jenipap (*Genipa americana* L.) is a natural source of genipin, an iridoid used as a natural cross-linking reagent to produce hydrogels for drug delivery.¹ Genipin also presents anticancer, anti-oxidative, and anti-inflammatory activities.^{2,3} To recover this compound, a betaine:lactic acid-based deep eutectic solvent (BL-DES) was used as alternative green extraction solvent, testing different ratio of betaine:lactic acid (n:n) (1:2; 1:3; 1:4), and different solvent/feed ratio (S/F) (w/w) (29; 19; 14; 9). The extraction was performed using the high intensity ultrasound (HIUS) assisted extraction. For each experiment, 30 grams of a system, constituted by BL-DES and MiliQ water in a ratio of 1:1 (w/w), and crushed jenipap was prepared. The extraction was performed with a 12,6-mm probe diameter at 19 kHz. The nominal power used was 315 W during 3 min, in triplicate for each treatment. The effect of BL-DES formulation and S/F in genipin recovery was evaluated by high-performance liquid chromatography with a diode-array detector (HPLC-DAD). The best results were obtained using a molar ratio of 1:3 for the proposed DES and a S/F of 19. The work reported suggest the suitability of the betaine-based DES combined with HIUS assisted-extraction as an efficient and sustainable genipin extraction process.

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Acknowledgments

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Impact of Eutectic Solvents Utilization in the Microwave Assisted Extraction of Proanthocyanidins from grape pomace

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Proanthocyanidins (PACs), or condensed tannins, are natural polymers composed of catechin units and its derivatives. These components have been increasingly used in the production of leather, wood agglomerates and wine, and therefore, their global demand has also increased. The extraction of PACs despite being an important and limiting aspect of their industrial application is still largely unexplored and is currently based in the extraction with hot sulfite solution from dedicated crops such as quebracho. This process has low specificity and yield which limits the industrial application of this type of extracts.

Consequently, the development of novel extraction methodologies and the use of more sustainable biomass sources (e.g., agroforestry by-products rich in this type of components such as grape pomace (GP)) is of great importance.

In this context, Eutectic Solvents (ES) have been proposed as good alternatives for conventional solvents due to their low price, easiness of preparation, biocompatibility, and ability of being custom made to a specific application. Additionally, Microwave Assisted Extraction (MAE) is frequently combined with ESs and was used here in the extraction of PACs from GP to improve not only extraction yield but also the mean degree of polymerization (mDP). The combination of choline chloride with lactic acid was shown to be the most effective combination for PACs extraction yield (135 mgPAC/gGP) and, despite the occurrence of some depolymerization, also enabled to achieve the highest mDP (7.13). Additionally, the combination with MAE enabled the process to be completed in 3.56 min, resulting in a considerable reduced extraction time.

Acknowledgments

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Application of natural deep eutectic solvents for the evaluation of pesticides occurrence in citrus and olive valorized by-products

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Development of food industry has brought about economic benefits worldwide, but it has also led to negative effects on the environment due to the increment of food waste. Such aspect involves the necessity of establishing effective strategies to reduce the impact of the organic residues generated. Valorization of food by-products, based on circular economy, has been suggested as an interesting and effective strategy to address this problem. However, as occur with the rest of food products, the commercialization of valorized by-products requires a strict control to ensure consumers' safety since diverse hazardous compounds used during their growth and production, such as pesticides, could appear in the final products.¹

The development of suitable methodologies which allow the reliable evaluation of food by-products' safety is essential in this area. In addition, it is also of great importance that such procedures comply with the principles of sustainability. Within this framework, the use of novel extraction materials that offer low toxicity and high biodegradability has become one of the most important action lines in the field. Among them, the study of natural deep eutectic solvents (NaDESs) has sharply increased because they are constituted of primary or secondary metabolites and other compounds usually present in cells which provide them great biocompatibility and almost absence of toxicity.²

In this work a novel and sustainable methodology based on the application of betaine-based NaDESs as extraction solvents was proposed in combination with gas chromatographic separation and mass spectrometry detection for the assessment of pesticides occurrence in citrus and olive valorized by-products. The procedure was thoroughly optimized by the evaluation of the type of NADES used, the extraction volume and the percentage of water added during the extraction. Afterwards the methodology was successfully validated obtaining good extraction efficiencies, with recovery values in the range 70-120 %, and good limits of quantification, lower than 67 µg/kg. Finally, the developed method was applied to the evaluation of citrus and olive by-products produced in Spain.

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Towards the valorization of orange peels: selective and integrated extraction of rutin and naringin using aqueous solutions of ionic liquids

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Nowadays, there is an increasing food demand as a consequence of worldwide population growth. As such, industries are pushing the limits of their food supply chains, generating large amounts of untreated waste, which indeed represent a source of high-value compounds. Due to the socioeconomic awareness, the research community is contributing towards the development of sustainable extraction processes to obtain high value bioactive compounds (HVBCs) from food waste within the circular economy model. Aiming to overcome current bioeconomy challenges, the development of new integrated processes is a priority. This work intends to valorize orange peels, a waste from the food industry, through the isolation of high-value phenolic compounds using novel biobased ionic liquids derived from glycine-betaine, able to increase the solubility of rutin and naringin by several orders of magnitude. Experimental design was applied to optimize extraction conditions, resulting in the efficient and selective extraction of rutin and naringin, allowing the solvent reuse. The developed process is a competitive alternative to conventional processes comprising volatile solvents.

Acknowledgments

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Improved extraction of RuBisCO from spinach leaves using aqueous solutions of biobased ionic liquids

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The world population growth is motivating the search for new sources of proteins. Vegetable biomass is a promising source since it allows the use of agricultural crop residues, being a continuous source of proteins.¹ RuBisCO (Ribulose-1,5-bisphosphate carboxylase/oxygenase) is the most abundant protein on the planet and naturally founded in plants. RuBisCO can be applied in the most diverse areas, such as pharmaceutical, cosmetic, and feed industries.² However, the current methods applied in the extraction and purification of this protein are not efficient (they do not allow purities and extraction efficiencies close to 100%).³ Therefore, the development of an efficient and selective RuBisCOs' extraction method is required. In this work, aqueous solutions of biocompatible ionic liquids (ILs) were applied for RuBisCO extraction from spinach leaves. Response surface methodology was applied to optimize RuBisCO extraction conditions. Under optimum conditions, extraction yields of 10.92 and 10.57 mg of RuBisCO/ g of biomass were obtained with the ILs cholinium acetate ([Ch][Ac]) and cholinium chloride ([Ch]Cl), respectively. When compared with a conventional solvent (NH₄OH), the extraction yield results were better for the IL solutions. Moreover, Circular Dichroism spectroscopy results show that the secondary structure of the RuBisCO is better preserved in IL solutions when compared to the commonly used extraction solvent. In conclusion, cholinium-based ILs demonstrate to be a promising and viable alternative to conventional solvents. The next step is the development of an integrated process to separate and purify the protein after the extraction, such as the development of an aqueous biphasic system or apply an ultrafiltration step to recover and reuse the IL.

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Integrated extraction and separation of beet leaves pigments using thermoreversible aqueous biphasic systems

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Biocompounds extracted and recovered from natural sources are relevant to several industries.¹ For instance, pigments such as betalains (red pigments) and chlorophylls (green pigments) extracted from food waste are of high value for pharmaceutical and cosmetics industries.² To purify the previously referred biomolecules, thermoreversible aqueous biphasic systems (ABS) can be used, while taking advantage of their monophasic regime to carry out the extraction and the biphasic one for the biomolecules separation.³ In this work, thermoreversible ABS composed of cholinium-based ionic liquids (ILs) and PPG 400 (poly(propylene)glycol 400 g.mol⁻¹) were investigated for the extraction and purification of betalains and chlorophylls pigments directly from red beets' stems and leaves. The investigated ABS were characterized at 25°C, 35°C, and 45°C. The best extraction conditions were carried out in the monophasic regime at 20°C, 70 min and using a solid/liquid ratio of 0.12, according to a design of experiments approach. The separation of both pigments was achieved in one step by increasing the temperature up to 35°C. As a result, chlorophylls migrate to the PPG-rich phase, whereas betalains preferentially migrate towards the IL-rich phase (Figure 1). Finally, both pigments have been recovered from the respective ABS phases recurring to affinity resins with high percentages of recovery: 94% and 98% for betalains and chlorophylls, respectively. Thus, the developed process reveals itself as an innovative process fitting the circular economy context.

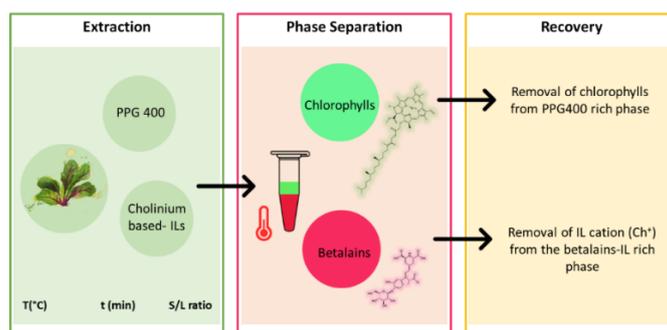


Figure 2. Schematic representation of the overall process developed.

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Separation and purification of phenolic compounds present in lignin's oxidative depolymerization liquor using ARIZONA liquid biphasic systems and Centrifugal Partition Chromatography (CPC)

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Lignin is one of the main structural components of lignocellulosic biomass, accounting for 15% to 30% of its dried weight. It is available at large-scale from the pulping liquor produced by the pulp and paper industry, representing a potential renewable resource. Nowadays, pulp and paper mills largely burn this liquor in an integrated process to produce electric and thermal energy. Although this combustion is still a valuable contribution to reduce fossil fuel consumption, lignin offers perspectives for higher added-valued applications and could be converted into several profitable commodities or fine chemicals. In this framework, oxidative processes for lignin conversion to phenolic compounds are widely recognized. However, their high price, as well as the inherent difficulty of separating the resulting structurally similar phenolic compounds, creates a significant drawback while simultaneously highlighting the need for more effective purification methods and downstream processes. As such, in this work we propose to determine an efficient process to separate a synthetic, representative mixture of lignin's oxidative depolymerization liquor, composed of five aromatic compounds: vanillin, vanillic acid, syringaldehyde, acetovanillone, and p-hydroxybenzaldehyde. Arizona biphasic systems were employed to achieve this goal. These highly tunable liquid biphasic systems composed by various proportions of heptane, ethyl acetate, methanol and water were shown to be highly effective in the fractionation of the established representative phenolic mixture and particularly suitable for a later application in Centrifugal Partition Chromatography (CPC). Furthermore, a modification of these systems was also performed by substituting heptane for limonene and methanol for ethanol, significantly increasing the sustainability of the process while preserving the separation efficiency previously achieved.

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Enhancement of the neuroprotective potential of olive leaf extracts through a selective fractionation strategy

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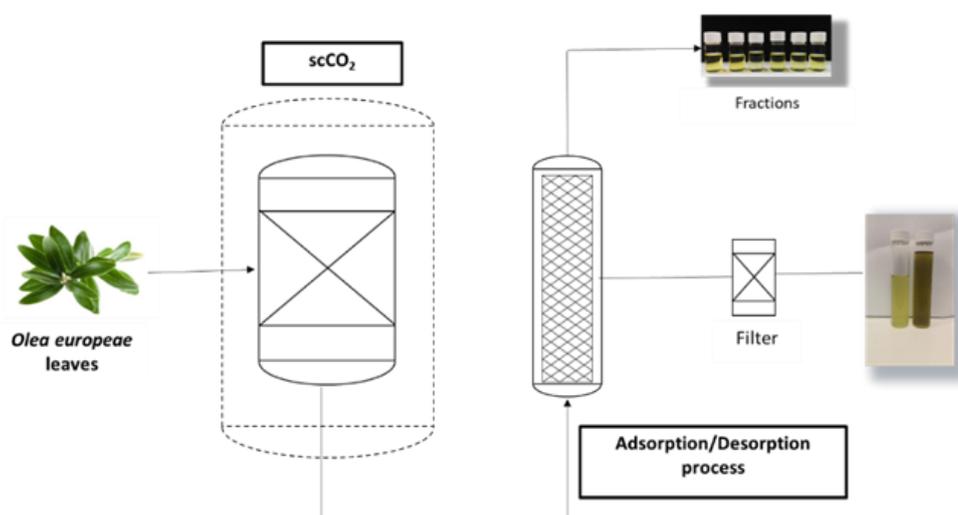
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Alzheimer disease (AD) is a disorder-related neurodegeneration, accounting for approximately two thirds of all cases of dementia and mainly attributed to factors associated to elderly people.¹ This painful illness has multi-pathological mediators that promote the loss of psychomotor functions, reasoning, memory, language and cognitive impairment². So far, there is no effective cure, hence, a current strategy is to target different factors, by the study of new bioactive metabolites from natural sources.

Olive (*Olea europaea* L.) leaves represents 25 percent of the total biomass generated in the olive oil industry³. It is a natural source of phenolic compounds with recognized bioactivity^{4,5} but it also has a wide terpenes group with high potential.

This work was oriented towards the improvement of the neuroprotective potential of olive leaves extracts as a therapeutic approach in delaying the detrimental effects of AD through the selective enrichment of terpenoids based on a dynamic adsorption-assisted supercritical fluid extraction (SFE). Thus, a battery of *in vitro* bioactivity assays was tested to screen for neuroprotective potential of these extracts. A multivariate analysis tool suggests the existence of latent variables that allow explaining the *in-vitro* neuroprotective potential of OLE on the basis of their terpenoids enrichment being sea sand and silica adsorbates the fractions with most representative neuroprotective potential due to the presence of pentacyclic triterpenes-enriched fraction such uvaol, erithrodyol and amyriins group.



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Biorefineries and Circular Economy

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At present, humanity is facing great challenges; among them, the attempt to reach (by 2030) the Sustainable Development Goals (SDGs).¹ Among them, zero hunger is one of the most important. This complex challenge demands new approaches on agricultural development for food security and nutrition, allowing an easier access to safe and nutritious food for sustaining life and promoting good health. Moreover, this challenge is of even more important with the continuous growth of the world's population and the pressure exerted on the environment. Among the different strategies to achieve these goals, in our laboratory we have been working on the revalorization of food wastes and by-products to generate new high added-value products using sustainable processes. Important concepts arise such as the rational use of resources and the implementation of biorefineries for biomass conversion in high added-value products, fuels, electricity, moving towards a circular economy model.² This can undoubtedly contribute to the wellbeing, the economy and the environment.

On the other hand, to develop sustainable processes is necessary to account for the 12 Green Chemistry Principles considering that is possible to design and improve products, materials and processes and systems, trying to preserve the environment.³ Specially important are the extraction processes complying with these rules while improving selectivity, speed, efficiency, production and costs. Some of these processes are based on the use of compressed fluids such as Supercritical Fluid Extraction (SFE), Pressurized Liquid Extraction (PLE), Subcritical Water Extraction (SWE, PHWE), Gas-expanded Liquids (GXLs) and the integration and intensification of processes.

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Searching for Natural Bioactive Compounds: Global Workflow in the Foodomics Lab

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One of the main topics in our lab during 2021,¹⁻¹³ has been the search of new food compounds with neuroprotective activity following a Foodomics evaluation. This work has included: a) the development of new green extraction processes to obtain bioactive compounds from different natural sources (algae, microalgae, food by-products, plants, etc); b) the determination of the neuroprotective effect of the new extracts against different in vitro and in vivo models of Alzheimer; c) the development of advanced analytical approaches for the chemical characterization of the bioactive extracts; d) the identification of metabolites differentially expressed in neuroblastoma cells using non-targeted whole-lipidomics approach based on LC-MS.

These strategies represent a good example of the important challenges that still have to be addressed by Foodomics in order to scientifically link Food & Health at molecular level and will allow us to discuss in this work some of the current and future challenges in this area of research.

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Metabolomics profiling strategies based on HRMS to identify bioactive compounds from agri-food by-products

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The hyphenation of new HRMS instruments (working in the MS or MS/MS mode) with high throughput chromatographic techniques has become a powerful strategy for the sensitive screening analysis and rapid identification of secondary metabolites in complex natural matrices. In this regard, HRMS-based techniques, coupled via liquid chromatography (LC-HRMS) or gas chromatography (GC-HRMS), are the most popular options for comprehensive phytochemical profiling and metabolomic purposes in natural products research. These hyphenated approaches significantly increase the speed of analysis, improving selectivity, resolution and efficiency, and provide improved structural determination capabilities compared to other methods.¹

The huge amount of datasets generated by HRMS measurements pose a great challenge due to the need of processing thousands of MS features to extract useful information. In this regard, several data-mining strategies have emerged to facilitate the post-acquisition data processing, according to the proposed workflow.² The implementation of these filtering strategies, along with MS search against well-established MS databases (e.g. NIST, Fiehn Lib, Wiley, MassBank, METLIN, HMDB) is a powerful tool to discover new drugs and natural medicines.

Following this approach, selected metabolomic profiling studies are presented in this work as remarkable case studies, providing an insight on how to get the highest throughput from the HRMS data. Thus, untargeted and semi-targeted MS and MS/MS data-mining strategies were successfully implemented to characterize highly valuable secondary metabolites from tropical fruits (e.g., withanolides, sucrose esters and procyanidins), genetically modified *Kalanchoe daigremontiana* hairy roots (e.g., bufadienolides) and from highly productive cyanobacteria grown in selected media (e.g., phytosterols), as promising sources of health promoting compounds with demonstrated anti-cancer, anti-inflammatory and anti-cholinergic properties under in-vitro bioactivity testing.

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Lipidomics study of the neuroprotective potential of olive leaves by-products

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Worldwide, around 50 million people have dementia with nearly 10 million new cases every year. Alzheimer's Disease (AD) is the most common form of dementia and it may contribute to 60–70 % of cases affecting about 24 million people. However, only a few drugs are approved for the therapeutic treatment of AD (with unpleasant side effects), and many studies have suggested that diet and/or food components can prevent the onset of AD. Phenolic derivatives and terpenoids from olive leaves have been deeply investigated, and our group has demonstrated that different olive leaves extracts have neuroprotective potential based on *in-vitro* experiments. In the present work, the neuroprotective and anti-inflammatory potential of an olive leaves fraction enriched in triterpenoid compounds has been confirmed in a neuronal cell culture model. In addition, a comprehensive lipidomics study of the response of SH-SY5Y neuroblastoma cell line to this fraction was carried out using advanced analytical methodologies, namely, charged-surface hybrid chromatography-quadrupole-time of flight mass spectrometry (CSH-Q-TOF MS/MS). This technology allowed the annotation of more than 250 intracellular lipids, and among them, a number of phosphatidylcholines and phosphatidylethanolamines were significantly increased while several triacylglycerols were found decreased, suggesting triterpenoids from olive leaves as good neuroprotective candidates.

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Alkanediols as efficient hydrotropes to enhance the aqueous solubility of syringic acid and their application for ultrasound-assisted extraction of phenolic compounds from juçara fruit

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Following the Green Chemistry principles, water is the most sustainable solvent. Unfortunately, many compounds presenting relevant properties and bioactivities are poorly soluble in water. Rather than using different solvents to process these compounds, hydrotropes can be used to overcome this limitation. Alkanediols are a class of di-alcohols widely used in the cosmetic, food, and pharmaceutical industries that can provide tunable solvent properties and act as hydrotropes to enhance the solubility in water of hydrophobic compounds. Despite that, alkanediols have been so far poorly explored as extractive solvents. In this work, a series of ten alkanediols were studied to increase the aqueous solubility of syringic acid. To better understand the hydrotropy effect of alkanediols, the experimental solubility data were fitted using the cooperative hydrotropy model and predicted by COSMO-RS modeling. Finally, three alkanediols were selected as extraction biosolvents to recover phenolic compounds from Juçara fruit by Ultrasound-Assisted Extraction (UAE) and compared to the hydroethanolic extract. From the solubility curves, enhancements up to 60-fold were found. The hydrophobic part of the chain is the main contributor to increase the aqueous solubility of syringic acid, in line with the recently proposed molecular mechanism of hydrotropy.¹ The differences from the 1,2-ndiol and 1,n-ndiol series were found and correlated to the aggregation mechanism around the solute. The quality of the extracts from UAE was evaluated in terms of monomeric anthocyanin concentration (MAC) and total phenolic compounds (TPC), while the antioxidant activity was found by FRAP and ABTS assays. Alkanediols aqueous solutions demonstrated to be as efficient as hydroethanolic ones since alkanediols juçara extracts achieved similar or even higher MAC, TPC, FRAP, and ABTS responses. Furthermore, low alkanediols concentrations (4-16% w/w) are sufficient to generate extracts with high antioxidant capacity.

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Enhanced extraction of phenolic compounds from Kiwi waste using biobased solvents

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Kiwifruit is rich in bioactive compounds such as phenolic compounds which exhibit biological activity with potential health benefits. However, around 30% of the total kiwi produced is wasted due to commercial pre-requisites.² Thus, and considering the concept of resource valorization based on sustainable chemistry, the wasted Kiwi represents the loss of a great source of high value compounds. Furthermore, the extraction of phenolic compounds from food waste is poorly investigated as a potential source, and when this is carried-out, volatile organic solvents are still the preferred choice for their extraction.³ In this work, aiming to develop a green approach for the extraction of the phenolic compounds from Kiwi peels, a combination of alternative techniques and renewable biobased solvents was investigated. Mixtures of biobased solvents with ethanol and water demonstrate higher levels of phenolics and antioxidant activity comparing to conventional solvents, with gamma-valerolactone (GVL) mixtures being the most efficient. GVL solution composition was optimized, and the mixture composed of 70 wt % GVL + 30 wt % ethanol was identified as the solvent with the best performance. The response surface methodology was used to optimize the extraction conditions (temperature/amplitude, extraction time and solid/liquid ratio), of conventional solid-liquid extraction (SLE), ultrasound-assisted extraction (UAE) and microwave-assisted extraction (MAE). From the three extraction techniques, MAE was the most efficient, as it yielded an extract with the highest content of phenolic compounds (Total phenolic content, TPC: 29.7 mg gallic acid equivalent/g dry weight) and the highest antioxidant activity (Ferric reducing antioxidant power, FRAP: 87.2 mg Trolox/g dry weight, [2,2'-azinobis-(3-ethylbenzothiazoline-6-sulfonate)], ABTS: 131.1 mg Trolox/g dry weight), with the shortest extraction time (6.0 min). The results obtained prove that biobased solvents are superior solvents for the extraction of phenolic compounds from Kiwi peels and combined with alternative extraction techniques can lead to the development of more sustainable and efficient extraction processes.

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Optimization of pressurized liquid extraction and bioactive potential evaluation of thinned peaches polyphenols from different drying treatments

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Thinned peaches are the redundant young fruits that are thinned out at 4~5 weeks after the blooming period, which have abundant bioactive compounds such as polyphenols. In this study, pressurized liquid extraction (PLE) has been adopted to extract thinned peaches polyphenols. The extraction yield, total phenolic content (TPC), acetylcholinesterase (AChE) inhibitory activity, lipoxygenase (LOX) inhibitory activity and reactive oxygen species (ROS) scavenging capacity were used as response variables to optimize the extraction condition of temperature (50, 115, 180 and 200°C) and solvent composition (H₂O, 50% ethanol in H₂O and ethanol). The optimum PLE conditions were 180°C and 50% ethanol in H₂O for achieving an extraction yield of 79.06%, 100.24 mg GAE/g of TPC, AChE IC₅₀ of 231.08 µg/mL, LOX IC₅₀ of 65.04 µg/mL and ROS IC₅₀ of 3.87 µg/mL. Furthermore, the polyphenols in thinned peaches after freezing drying (FD), natural drying (ND) and hot air drying at 50°C (HAD50), 70°C (HAD70) and 90°C (HAD90), respectively, were extracted using the optimum PLE conditions. In addition to the previous *in vitro* experiments, total flavonoid content (TFC), anti-butyrylcholinesterase activity (BChE) and reactive nitrogen species (RNS) scavenging capacity were conducted to study the effects of the different drying treatments on the bioactive potential of thinned peaches polyphenols. The FD thinned peaches extracts had the highest TPC, TFC and bioactive activities, the HAD90, HAD70, HAD50 and ND extracts decreased sequentially, except for the LOX experiment (FD > HAD90 > ND > HAD70 > HAD50). In general, FD could retain the polyphenols and biological activities in thinned peaches to the greatest extent.

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Bioprospecting through compressed fluids: Neuroprotective compounds from *Ammodaucus leucotrichus*

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The bioprospecting of the African plants is almost nonexistent comparing with those in the others continents, especially, the endemic plants of arid and semiarid zones. Furthermore, the ability of plant adaptation to the extreme conditions of climate in desert led to synthesize new molecules, which possessing a wide range of interesting biological activities. As case study, *Ammodaucus leucotrichus* (AL) was selected; it is a spontaneous aromatic plant belonging to Apiaceae family of Saharan countries of North Africa extending to tropical Africa. It have been traditionally applied in herbal Medicine [1]. On the other hand, the research for new source of bioactive molecules that can have a positive effect on the Alzheimer's disease (AD) has arisen in recent years. Around the world, 45 million people are suffering from AD symptoms. Previous research demonstrated that plants belonging to the Lamiaceae and Apiaceae families are capable of exerting effects on the nervous system [2]. Nowadays, The principal drugs used for treatment of AD are the Acetylcholinesterase (AChE) inhibitors [3]. The development of green extraction processes based on the use of compressed fluids is explored in present work. Among them, Supercritical Carbon Dioxide, Pressurized Liquid and Subcritical Water Extractions, have proven to be more efficient and sustainable than conventional solvents [4]. In this work, two green extraction techniques have been tested to obtain bioactive molecules with high AChE inhibitory activity from AL. Supercritical CO₂ extraction with ethanol and Pressurized Liquid Extraction (PLE) using water and ethanol were tested. The main factors influenced both techniques were optimized. An additively tests to enhance the bioactivity using different combinations of both methods, in optimum conditions, was also performed.

The best results (lower AChE IC₅₀) are in agreement with the highest values of total phenolic content and total carbohydrate. The lower AChE IC₅₀ value obtained was 55.59 µg/mL using PLE (subcritical water at 180°C), while 332.33 µg/mL using SFE (55°C, 100bar using 15% of ethanol). In our case, in term of total recovery, total phenolic content, total carbohydrate and neuroprotective activity, subcritical water at 180°C is the appropriate conditions for AL comparing to the SFE or to the different combinations used. Whereas UHPLC-q-TOF-MS/MS techniques was selected to determinate chemical profile of extracts. The results reinforce the demand for further research on the other biological activities of AL, as well, as support to continue bioprospecting endemic plants of arid and semiarid zones.

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Dual-function (solvent and catalyst) eutectic-based systems to improve furfural production

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Furfural has been identified as a key platform chemical derived from biomass. This chemical is almost exclusively obtained by the acidic-catalysed conversion of pentosane-based sugars under harsh temperature and acidic conditions. Therefore, continuous research has been pursued to optimize this process and to turn it more cost-efficient and sustainable,¹ and for which Deep eutectic solvents (DES) can be considered. DES are a type of neoteric solvents that can be easily prepared by simply mixing, at appropriate ratio, at least two compounds and where a significant deviation from the ideal liquid-phase behavior occurs.² DES can be designed to have good solvation features and acidic characteristics, thus being able to act both as solvents and catalysts in furfural production.

In this work, we report the use of acidic based deep eutectic solvents for the hydrolysis of xylans into xylose and further dehydration into furfural. The effects of the DES molar ratio, temperature, solid-liquid ratio and amount of water were investigated, and the process was optimized for maximum furfural production. The DES [Ch]Cl:Malic acid was used in microwave-assisted reactions as both solvent and catalyst for furfural production. The process developed allowed an outstanding 75.0% of furfural yield in 2.5 minutes of reaction time under microwave heating. The recovery and reusability of the solvents was evaluated, being possible to reuse the DES in at least three cycles.³ The obtained results highlight the potential of greener and recyclable solvents based on deep eutectic mixtures for the creation of integrated platforms for the valorization of hemicellulose fractions in biomass.

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Blue biorefinery using ionic liquids

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Due to their relevance in our nowadays life and application in several industries, separation processes are highly studied. With the goal of developing more cost-efficient processes, ionic liquids (ILs) have been applied in different fields in which large-scale separation is mandatory, namely in the extraction of various products from biomass and residues.

Macroalgae, microalgae and cyanobacteria are part of the biomass matrices practically unexplored, although recognized as rich raw materials since they are composed of a large range of bioactive compounds with commercial interest. Included in the range of commercially interesting bioactive compounds are the pigments, which can be divided in three main categories, phycobiliproteins, chlorophylls and carotenoids/xanthophyll.¹ Over the years, all of which have been applied in different industries, including cosmetics, human food, and energy.

Despite the high economical value of some of the ingredients accumulated in cells, its commercialization has still not reached its maximum, due to the high costs of the downstream processes being applied up to date.² These are normally related with the processes' complexity, using large amounts of organic solvents or those using more sophisticated equipment and specialized human resources, compromising the compounds' sustainable and profitable commercialization. In here, some processes of extraction and purification of pigments from algae based on the use of ILs will be presented.

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Extraction and purification of Collagen type I using aqueous solutions of Deep Eutectic Solvents from Atlantic Codfish (*Gadus morhua*)

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Marine fish industries discard huge amounts of fish wastes every year, which in turn impose problems of environmental pollution and loss of economic value. About 75% of total weight of fish is discarded in the form of skins, bones, fins, heads, guts, and scales, which contain high levels of collagen type I. Generally, major sources for commercial collagens are the skin and bone of pigs and cows, however, these sources are chiefly associated with the risk of transference of zoonotic diseases or religious issues. The traditional protocols applied on the extraction of collagen are outdated, mainly in what concerns the present demands to develop more sustainable processes. This work explores the use of sustainable solvents, such as deep eutectic solvents, to develop a more efficient, cost-effective and biocompatible process to extract collagen from wastes of the fish industry. The extraction of collagen from the skin of Atlantic cod (*Gadus morhua*) using aqueous solutions of various deep eutectic solvents (DES) was studied, and after selection of the best solvent, the aqueous solution of urea (U) and lactic acid (LA) at a molar ratio of 1:2 - U:LA (1:2). The collagen extracted, was shown to be of type I. The results demonstrated an improvement on the yield and quality of the extracted collagen when DES are applied instead the conventional approach using acetic acid. After optimization of the process conditions, a maximum of 6% of yield of extraction was obtained for the aqueous solution of U:LA (1:2) at 0.75 M. The present work demonstrates the potential use of waste codfish skins and aqueous solution of DES to obtain high quality collagen as an effort to convince industries to valorize their own residues under the guidelines of a circular economy.

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Recovery of bacterioruberin from marine archaea using eutectic solvents

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In the biorefinery context, both bulk and specialty chemicals can be produced and extracted from biomass, resulting in a potentially more sustainable alternative to petroleum-derived products. An example of this is Haloarchaea, an interesting class of extremophile microorganisms, also present in the Aveiro salt pans. Amongst these, *Haloferax mediterranei* is a highly promising candidate to produce bacterioruberin owing to its rapid growth and ability to consume a variety of carbon sources. Bacterioruberin is an uncommon C50 carotenoid with great biotechnological interest. This type of carotenoid exhibits a higher antioxidant capacity than the C40 carotenoids such as β -carotene, which can be explained due to the higher number of pairs of conjugated double bonds. This makes this carotenoid remarkably interesting for the food, pharmaceutical and biomedical industries. In this work, the valorization of *Haloferax mediterranei* ATCC 33500 was pursued through the recovery of bacterioruberin and its separation and purification from the remaining cell components. To formulate food and cosmetic grade compatible extracts, pigment recovery was accomplished through the use of bio-derived eutectic solvents. Operational conditions such as the solid-liquid ratio, concentration of eutectic mixture, hydrogen bond-donor and hydrogen-bond acceptor ratio and the time of extraction were accessed. These eutectic mixtures arise as more sustainable and appropriate solvents compared to the typical solvents used in chemical processes because of their simple preparation, extraction efficiency and integration of the extraction and separation in a “one-pot” method by using water as counter-solvent.

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A simple approach to recover chlorophylls from AmberLite™ HPR900 OH using tensioactive ionic liquids

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A multiproduct biorefinery is a concept to be aware in order to achieve the highest value of the compounds present in the same biomass. Natural pigments, such as chlorophylls and carotenoids, besides having a high market demand due to their biological interest, their extraction is usually performed under the same operational conditions thus complicating their separation. In this way, purification procedures need to be developed to obtain multiple compounds and to reach a complete valorization of the biomass. The solid-phase extraction can be used as a technique to separate chlorophylls and carotenoids from a liquid matrix by chlorophylls adsorption into the resin. However, in past works the recovery of chlorophylls was never achieved. Therefore, the novelty of this work remains with the elution of chlorophylls as a second valuable compound, where between the several solvents tested, the highest performance was obtained by aqueous solutions of tensioactive ionic liquids. Then, a central composite rotatable design was performed in order to optimize the process including three variables, namely solid-liquid ratio, time of contact, and concentration of ionic liquid in water, reaching a 97.0 ± 0.9 % elution of the initial adsorbed chlorophylls. In addition, after five cycles of resin reuse no loss of the process efficiency was detected. Finally, a procedure for the chlorophylls polishing was developed and a scale-up in a continuous mode assessed, envisioning the process implementation in the industry.

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