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SCIENTIFIC PROGRAM

27 de junio	
Bloque 1. CO2 a compuestos de valor añadido	
9:00-11:00 Tema 1. CO2 a carbonatos cíclicos	<ul style="list-style-type: none">• R. Villa• Jorge Parra.• Francisco J. Ruiz➤ S. Alcalde
11:00-11:30 Pausa Café	
11:30-13:30 Tema 1. CO2 a carbonatos cíclicos	<ul style="list-style-type: none">➤ M. Cuesta.➤ M. Maireles➤ S. Akki➤ Juan Chiva➤ J. Sanchez
13:30-15:00 Pausa Comida	
15:00-16:30 Tema 2 CO ₂ a Fórmico	<ul style="list-style-type: none">• Álex Fernández• Víctor Candell• Jose Luis Serrano
16:30-16:40 Pausa	
16:40-18:00 Tema 2 CO ₂ a Fórmico	<ul style="list-style-type: none">➤ M. Cuesta.➤ S. Akki➤ J. Sanchez➤ F.Garcia-Cirujano





28 de junio	
9:00-11:00 Bloque 2. Reciclado espumas de poliuretano	<ul style="list-style-type: none">• Rocío Villa• Rebeca Salas.• Francisco Velasco.➤ M. Macia➤ M. Maireles
11:00-11:30 Pausa Café	
Bloque 3. Compuestos Bio-activos	
11:30-13:30 Tema 1. Cosmecéuticos	<ul style="list-style-type: none">• Juana M. Bernal• Susana Nieto.• Inmaculada Lozano e Isabel Fernández• Jose Francisco Costa➤ I. Muñoz
13:30-15:00 Pausa Comida	
15:00-17:30 Tema 2. ILs bioactivos	<ul style="list-style-type: none">➤ J.J. Andreu➤ G. Lauer



CONFERENCIAS



Advanced biocatalytic materials derived from ionic liquids.

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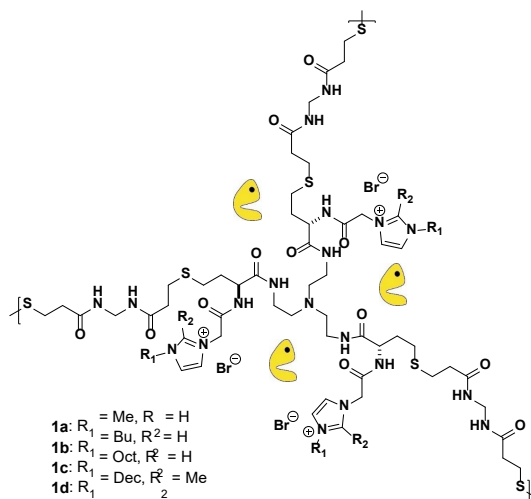
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Polymeric materials based on ionic liquids (ILs) have been highly effective for immobilizing and stabilizing enzyme systems.[1] The ability to finely adjust the structure of ILs allows for the creation of materials with varied molecular and macroscopic characteristics. This paper discusses the immobilization of alcohol dehydrogenase from *Rhodococcus ruber* (ADH-A) and its coenzymes using these sophisticated materials. The performance, stability, and recyclability of these biocatalysts were assessed for the enantioselective reduction of ketones. Results showed that the immobilized enzymes retained high activity and enantioselectivity and demonstrated remarkable stability in both aqueous and traditional organic solvents, even after several cycles of reuse.[2].



Scheme 1. Immobilization of ADH-A in the polymeric system.

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Continuous-flow scCO₂ method as a sustainable alternative to traditional solvents for CuACC reaction.

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The pharmaceutical industry is actively pursuing sustainable and cost-effective methods for synthesizing druggable molecules, with a focus on reducing solvent usage and adopting environmentally friendly alternatives.[1,2] Solvent minimization is a critical challenge, with organic solvents contributing significantly to process mass intensity and energy consumption, particularly during separation and purification steps. Continuous-flow processes are gaining traction due to reduced lead times, costs, and environmental footprints. However, they often require higher solvent usage to maintain homogeneity.

Supercritical carbon dioxide (scCO₂) presents a promising solution, offering advantages such as abundance, non-toxicity, and tunability of reaction conditions. Its use in continuous-flow processes has shown promise in accessing high-value compounds while reducing organic solvent consumption. Additionally, scCO₂ is established in supercritical fluid chromatography (SFC) for efficient purification of diverse compounds. Integrating synthesis and purification using scCO₂ as a primary carrier enables streamlined continuous processes with improved efficiency and reduced solvent usage. An example is the production of a diverse family of 1,2,3-triazoles, demonstrating enhanced overall efficiency and significantly decreased organic solvent consumption.

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Macrocyclic pseudopeptides as multifunctional catalyst for CO₂ valorisation)

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The growing emissions of carbon dioxide to the atmosphere have triggered the blossoming of technologies for its capture, activation, and conversion into added value chemicals [1]. Among the different carbon dioxide transformation technologies established to date, many efforts are being devoted for the development of efficient catalysts for the production of added-value cyclic carbonates [2]. Several metal-based systems have been proved as highly active in the catalytic conversion of CO₂ to CC, operating under mild conditions.[3] Furthermore, organocatalytic processes avoiding issues associated to metal contamination can be considered as greener catalytic approaches and, in the last decade, a variety of metal-free systems have been designed as sustainable alternatives for catalyzed CC formation.[4] Herein we report macrocyclic pseudopeptide systems presenting high activity in the cycloaddition of carbon dioxide to epoxides under mild conditions being govern by supramolecular interactions, [5, 6] furthermore their intrinsic chirality afforded remarkable results in terms of enantioselectivity. [5] The immobilization of the pseudopeptidic macrocyclic host and halide nucleophiles in a polymeric matrix providing a hydrophobic microenvironment afforded excellent catalytic systems with improved practical application. [7]

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AMPHIPHILIC DISULFIDE BASED NANOPARTICLES DERIVED FROM HOMOCYSTEINE THIOACTONE

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Keywords: polymeric nanoparticles • drug delivery • external stimuli • homocysteine thiolactone

The application of nanotechnology for drug delivery is widely expected to bring new hope in medical treatments.[1] In this regard, “smart” polymeric micelles have been developed as nanocarriers with specifically respond to certain triggers, as pH, temperature, radiation, Redox potential, ionic strength, and biological stimuli.[2]

In this project we have developed drug delivery systems based on polymeric micelles containing labile S-S cross-linking moieties with redox sensitivity. Here we present the synthesis and characterization of ionic liquid homocysteine thiolactone based polymeric nanoparticles. Furthermore, drug entrapment efficiency studies and in vitro release measurements under reductive conditions have been performed.

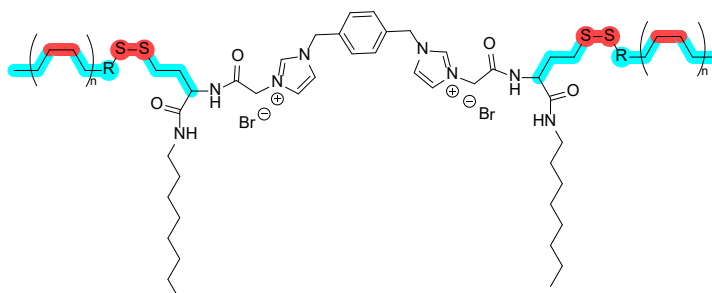


Figure1. General structure of the ionic polymeric nanoparticles based on homocysteine thiolactone.

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Acknowledgments

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"Desarrollo de Procesos Sostenibles para la obtención de Ingredientes Bioactivos Derivados del Ácido Cinámico para Aplicaciones Cosméticas"

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El ácido cinámico es ampliamente utilizado en la industria cosmética y farmacéutica debido a sus propiedades biológicas beneficiosas, como sus actividades antioxidantes, antiarrugas, antiinflamatorias y antimicrobianas. No obstante, este ingrediente presenta limitaciones en términos de penetración y estabilidad frente a la oxidación [1]. Por lo tanto, la industria cosmética está explorando la síntesis de derivados del ácido cinámico que mantengan sus propiedades antienvjecimiento, mejorando simultáneamente su estabilidad y capacidad de penetración. La biocatálisis se destaca como una herramienta eficiente, ofreciendo ventajas en actividad, enantio- y regio-selectividad, lo que contribuye a la sostenibilidad de los procesos químicos. Las lipasas, enzimas ampliamente utilizadas en la industria cosmética, son capaces de reconocer una variedad de sustratos y catalizar numerosas reacciones [2]. La síntesis enzimática mediante esterificación directa utilizando sistemas basados en Disolventes Eutécticos Profundos (DES, por sus siglas en inglés) ha demostrado ser un método excelente para obtener monoésteres acílicos de pantenilo [3, 4].

En este estudio, se extrapoló esta tecnología para la obtención de derivados del ácido cinámico, evaluando posteriormente su eficacia como potencial ingrediente cosmético antiedad. Los derivados esterificados de cinamatos de pantenilo obtenidos mediante estos nuevos procedimientos enzimáticos, demostraron su potencial acción como ingrediente cosmético en diferentes ensayos *in vivo*, donde se observaron que las formulaciones que incluían estos ingredientes mejoraron significativamente los niveles de hidratación, elasticidad, firmeza, contenido de melanina y topografía de la piel tras su aplicación vía tópica, y tras la evaluación del estado de la piel 2 horas después de su aplicación. Estos resultados subrayan el potencial de los derivados del ácido cinámico como ingredientes innovadores y eficaces en el desarrollo de productos cosméticos antiedad.

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Reducción de CO₂ por una Formiato Deshidrogenasa NAD⁺-dependiente con Regeneración del Cofactor

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La reducción de CO₂ a ácido fórmico catalizada por la enzima formiato deshidrogenasa (FDH) constituye una reacción fundamental de fijación de este gas como punto de partida para la obtención de compuestos de carbono de alto valor añadido [1]. La enzima más empleada para este cometido procede de la levadura *Candida boidinii* (CbFDH). Sin embargo, la FDH de la bacteria *Thiobacillus* sp. KNK65MA (*TsFDH*) presenta unas propiedades cinéticas más favorables para la obtención de ácido fórmico [2].

Este trabajo se ha centrado en el desarrollo y optimización de un método para la reducción de CO₂ a ácido fórmico mediante la acción catalítica de *TsFDH* (expresada y purificada en nuestro laboratorio) en la que interviene el cofactor nicotinamida adenina dinucleótido (NADH) como agente reductor. Con el objetivo de aumentar la producción de ácido fórmico se llevó a cabo la regeneración del cofactor mediante el acoplamiento con la reacción de oxidación de la glucosa, catalizada por la enzima glucosa deshidrogenasa (GDH) (Figura 1). De esta forma, se alcanzaron rendimientos de ácido fórmico de hasta 2,6 mM con 0,30 mg de *TsFDH* y 0,15 mg de GDH en disolución, NADH 1,4 mM y una presión de CO₂ de 10 bar, tras 8h de reacción a 30°C.

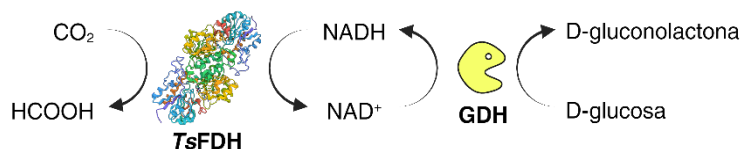


Figura 1. Reducción de CO₂, catalizada por *TsFDH*, acoplada a la oxidación de D-glucosa, catalizada por GDH.

Se inmovilizaron las enzimas sobre SILLP-MeIm-Cl [3] con un porcentaje de retención del 80%, obteniéndose una concentración de ácido fórmico de 0,260 mM con NADH 10 mM y 1 mg de enzima inmovilizada tras 4h de reacción. Se reutilizó el biocatalizador durante siete ciclos catalíticos hasta que la actividad residual cayó por debajo del 30%. Además, se utilizaron los MOFs MIL-101, UiO-67 y UiO-66-SO₃H. Mientras que en el primer caso no se consiguió retener proteína, en los otros dos soportes se alcanzó un porcentaje de retención del 40%, aunque no se detectó actividad para la reducción de CO₂ en ninguno de ellos.

Agradecimientos

Este trabajo ha sido realizado gracias a la subvención de la Fundación Séneca (21884/PI/22) y a MICINN/FEDER NextGeneration (PID2021-124695OB-C21).

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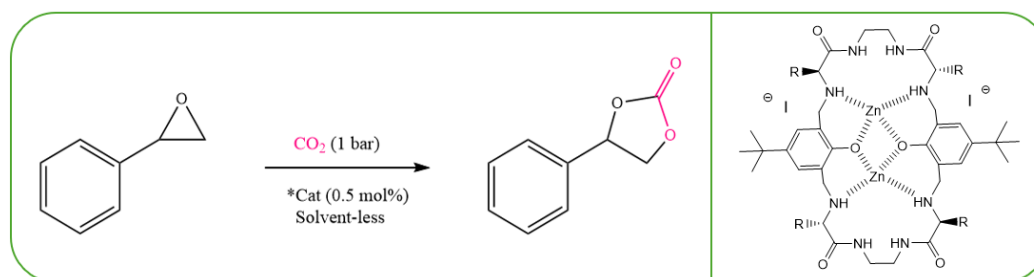
Bio-inspired synthesis of new pseudopeptide macrocycles for CO₂ conversion

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In nature, enzymes can accelerate the rate of chemical reactions with high efficiency, achieving additionally a high selectivity. Carbonic anhydrase, with an active center consisting of a Zn²⁺ cation coordinated with three imidazole rings, is one of the known enzymes that achieves a higher conversion rate (10⁶ s⁻¹) in the transformation of CO₂. [1] Based on this premise, this project focuses on the synthesis of pseudo-peptidic macrocycles to obtain the corresponding Zn²⁺ complexes capable of catalysing the cycloaddition reaction of CO₂ to epoxides. [2] In this case, the synthesis of the macrocycle has been carried out using tyrosine as the starting amino acid, as the alcohol group attached to the aromatic ring will allow us to derivatize it by introducing a vinyl group that will enable, through polymerization, the production of the corresponding supported catalyst.



Scheme 1. On the left, the catalyzed reaction of CO₂ cycloaddition to epoxides. On the right, structure of the macrocyclic catalyst (R group associated to the amino acid side chain).

We have obtained a synthetic methodology for the preparation of vinyl monomers from tyrosine-derived pseudopeptide macrocycles, suitable for subsequent polymerisation.

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Sustainable synthesis of hydroxytyrosol esters with antioxidant activity by biocatalysis in ionic liquids

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In the recent years, there has been a notorious interest for the incorporation of bioactive compounds from Nature into food, pharmaceutical and cosmetical formulations because of their benefits to human health. At the same time, the urgency for the transition of current methodologies towards sustainability means an additional challenge for the chemical industries and companies to face.

Hydroxytyrosol (HT) is a remarkable phenolic, bioactive compound found in virgin olive oil that shows antiinflammatory, antitumoral, antiangiogenic and cardioprotective properties, mostly due to its important antioxidant activity [1]. Due to these excellent properties, the European Food Safety Authority (EFSA) has approved claims for its content in food. Nevertheless, because of the hydrophilicity of HT, its applicability into hydrophobic mixtures, such as cosmetics, gets very limited. To solve this problem, HT's lipophilization is carried out, consisting of its covalent modification by means of (trans)esterification with another lipophobic molecule. These processes are commonly catalyzed by enzymes, avoiding intermediate protection and deprotection steps and their environmental impact. However, volatile organic solvents and excess of ethyl- and vinyl-fatty acid derivatives are used, contributing to waste production.

This project develops a lipophilization strategy for HT through direct biocatalytic esterification with fatty acids using ionic liquids, which are green non-volatile solvents, as the reaction medium [2]. Some reaction parameters have been optimized following the Green Chemistry principles, and a range of HT esters have been obtained with a high yield, demonstrating the strategy's viability at a larger production scale and the maintenance of their antioxidant activity. The sustainability of the developed strategy has also been confirmed using a variety of Green Metrics, which have proved its superiority compared to other procedures described in the bibliography.

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Transesterification of MFDC and cycloaddition by CO₂ using heterogeneous catalysis

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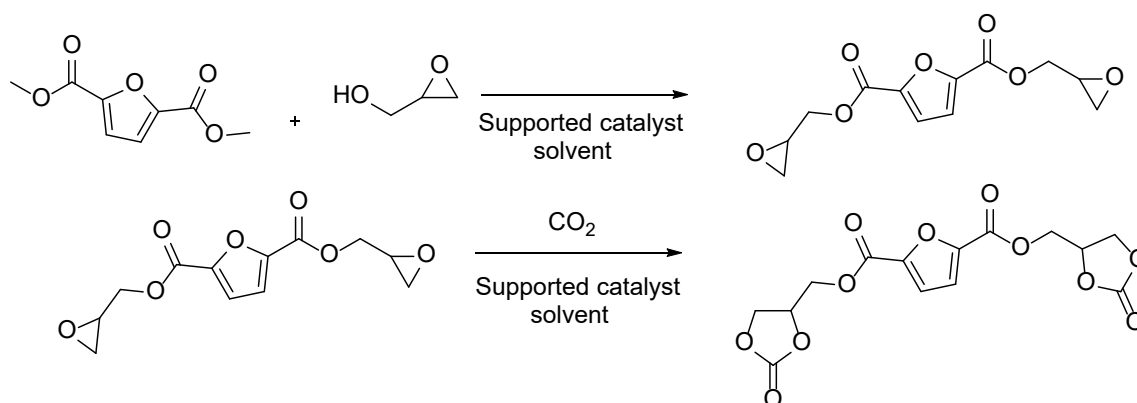
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Interest in cyclic carbonate compounds has been increasing in both industry and academia, as they represent a green alternative to various traditional compounds. [1] A notable example is the replacement of conventional toxic synthesis of polyurethanes with polyhydroxyurethanes (PHUs), which are formed through greener methods such as the ring-opening reactions of cyclic carbonates with alcohols and amines. [2] For these reasons, this study presents our efforts to create a heterogenous catalysis method to produce cyclic carbonates polymers started with Dimethyl Furan-2,5-dicarboxylate (MFDC).

The first step of the synthesis involves the transesterification of MFDC with glycidol to form bis(oxiran-2-ylmethyl) furan-2,5-dicarboxylate, also known as the diglycidyl ester of MFDC. Following the cycloaddition of carbon dioxide to the epoxide, bis((2-oxo-1,3-dioxolan-4-yl)methyl) furan-2,5-dicarboxylate is obtained. These two reactions are catalyzed by different ionic liquids and enzyme-ionic liquids (enzymes-ILs) supported on Merrifield resin. This product can be used to produce green polymers. [3]



Scheme 1. Synthesis method to obtain Bis(cyclic carbonate).

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Expression and Purification of a Metal-Independent Formate Dehydrogenase and its Application to CO₂ Fixation

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We have selected the codon for codifying the metal-independent (i.e., NAD⁺-dependent) Formate Dehydrogenase from *Thiobacillus sp* KNK65MA (*TsFDH*), cloned it into a commercial plasmid, transformed the resulting vector into *Escherichia coli*, and purified the expressed enzyme. The conditions for obtaining the protein were optimized and the final yield was 44 mg/L *E. coli* culture. *TsFDH* has been biophysically characterized and its activities for CO₂/HCOOH redox interconversion in both directions have been determined (Scheme 1).



Scheme 1. CO₂/HCOOH reduction/oxidation by NAD(H⁺) cofactor in the presence of *TsFDH*.

Optimization of the CO₂ fixation reaction (direct reaction, Scheme 1) was improved with the regeneration of the NADH cofactor by coupling it to the NAD⁺ reduction by glucose and the action of the glucose dehydrogenase enzyme. Altogether takes good formic acid production both in solution and in the immobilized enzyme onto SILLPs supports; indeed, up to 2.6 mM and 0,26 mM formic acid production, respectively. In the latter case, biocatalyst reutilization was partially successful with yields up to 30% after 7 cycles.

Acknowledgments

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Optimization of the scaling-up of a sustainable procedure for obtaining esters of panthenol with antioxidant activity

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A current trend in the cosmetic sector is the use of cosmeceuticals, cosmetic products with bioactive ingredients that offer therapeutic benefits, and the shift towards a more sustainable synthesis of cosmetic products.

A sustainable strategy has been developed to link panthenol with HCAs (trans-cinnamic, para-coumaric, caffeic, and hydrocaffeic acids) through esterification, thus obtaining new compounds that combine the bioactive properties of both precursors. This work presents the optimization and scaling of this strategy, based on a biocatalytic esterification using a hydrophobic ionic liquid (SLIL) as the reaction medium [1]. As a result of the synergy between the biocatalyst and the SLIL, high efficiency and selectivity in the synthesis of panthenol monoesters with different HCAs under mild reaction conditions (80 °C, 8-48h) have been achieved, demonstrating antioxidant activity [2]. Not only does this strategy allow for a clean synthesis, in the absence of volatile organic compounds, but it also avoids their use in the extraction stage, as well as excessive energy expenditure, since the SLIL spontaneously expels the reaction mixture, in accordance with the principles of Green Chemistry [3]. The sustainability of the optimized strategy has been measured through the application of different Green Metrics.

Acknowledgments

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Expresión, Purificación y Caracterización de una Formiato Deshidrogenasa

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Actualmente, la única formiato deshidrogenasa (FDH) accesible comercialmente es la expresada por la levadura *Candida boidinii* (CbFDH), cuya baja eficiencia catalítica respecto de la reducción de CO₂ supone una importante limitación en su aplicación práctica. Existen otras variedades mucho más eficientes, aunque no se encuentran disponibles en el mercado. En particular, la FDH expresada por la bacteria *Thiobacillus sp* KNK65MA (*TsFDH*) posee unas características cinéticas atractivas respecto de la fijación de CO₂ a ácido fórmico [1].

En este trabajo, se ha diseñado el gen que codifica la enzima *TsFDH* y se ha insertado en un plásmido comercial para su transformación en *Escherichia coli*. Tras la optimización de las condiciones de cultivo, se ha expresado y purificado exitosamente la enzima recombinante (Figura 1), incrementando el rendimiento de la producción de *TsFDH* hasta 11 veces respecto de las condiciones de partida.

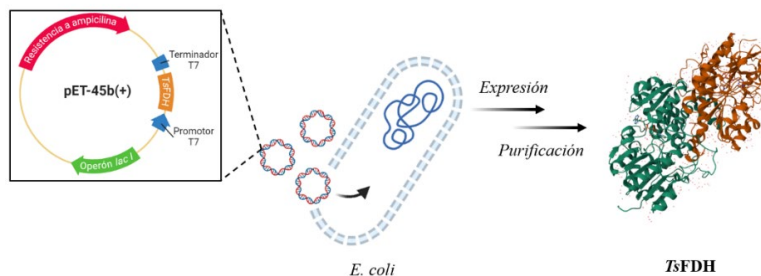


Figura 1. Esquema de la obtención de *TsFDH* recombinante.

Se han determinado algunas características estructurales mediante la aplicación de técnicas bioquímicas y espectroscópicas, tales como la electroforesis en gel de poliacrilamida y las espectroscopías de fluorescencia, resonancia magnética nuclear de ¹H y espectroscopía de infrarrojo. Los resultados obtenidos de estos análisis confirman tanto la identidad de la proteína como su adecuación estructural para su uso en sistemas de captación de CO₂ [2]. Asimismo, se ha determinado la actividad específica en la catálisis de la reacción de oxidación de ácido fórmico a CO₂ y se han comparado los valores obtenidos con los de la enzima CbFDH concluyendo que *TsFDH* es un orden de magnitud más eficiente en el rango de pH ensayado.

Este trabajo ha sido realizado gracias a la subvención de la Fundación Séneca (21884/PI/22) y a MICINN/MRR Next Generation (PID2021-124695OB-C21).

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Creating Hybrid Systems of Functionalized Polymeric Ionic Liquids and Metallic Nanoparticles Using Thiolactone Chemistry

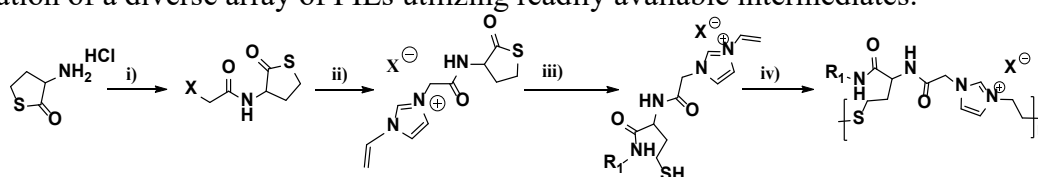
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Ionic liquids (ILs) possess a remarkably adaptable structure, enabling the fine-tuning of their physicochemical characteristics for diverse applications [1]. The bioactivity of ILs is closely intertwined with this structural versatility. Derived from ILs, polymeric ionic liquids (PILs) exhibit promising potential in augmenting these traits. This investigation delineates a synthesis route for PILs employing click chemistry, specifically utilizing amino-thiolactone [2]. Noted for its simplicity and versatility, this approach entails the synthesis of the alkyl-imidazolium salt of amino-thiolactone, followed by its ring-opening via aminolysis, culminating in thiol-ene photopolymerization to yield various PILs. This scalable synthesis methodology facilitates the generation of a diverse array of PILs utilizing readily available intermediates.



Scheme 1. Synthesis (i) Bromoacetyl bromide, K_2CO_3 , CH_2Cl_2 , 1 h, r.t., 60–65%; (ii) ACN, 1-vinylimidazole, 16 h, 45°C, 80–85%; (iii) 3-(Dimethylamino)-1-propylamine/ H_2O , 24 h, r.t.; (iv) Lithium phenyl-2,4,6-trimethylbenzoylphosphinate (LPA), 0.5 h, r.t.

This study presents a novel approach to enhance the bioactivity of Polymeric Ionic Liquids (PILs) against tumor cells and bacterial strains by integrating nanoparticles such as Silver (AgNPs) and Gold (AuNPs). Utilizing synthesized PILs, the research successfully developed nanoparticle-PIL (NP@PIL) hybrid systems through varying metal/polymer molar ratios. The methodology involved rapid nanoparticle production via metal solution introduction into a $NaBH_4$ -polymer solution, indicated by observable color changes. These hybrid systems demonstrated promising efficacy against bacterial strains and tumor cells, expanding their potential applications.

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Nowadays the entire population makes use of at least one cosmetic product, regardless of age or gender which has contribute to the expansion of cosmetic industries. Due to the high competitiveness within this sector, the search for new products is essential and today the focus is set on the development of new products with bioactive properties that fight against the skin problems at their molecular origin. Precisely, cosmetics is a representative field where sustainable and safe synthesis plays a key role due to character of the products and their consumption as commodities, which is manifested through the express commitment of the vast majority of companies to more sustainable production and distribution processes.¹ Hence, the use of enzymes as natural catalysts to achieve the synthesis or transformation of raw materials is highly suitable to achieve this purpose, in combination with non-contaminant and innocuous neoteric solvents.

In this work, the commercial immobilized lipase N435 and hydrophobic ionic liquids with sponge-like behavior (SLILs) have demonstrated their excellent synergy in the esterification of panthenol (pro-vitamin B5, 69 °C m.p.) with different (hydroxy)cinnamic acids (HCAs, 133-225 °C m.p.) with high selectivity and optimum yields (30-75%) under soft reaction conditions (70 °C, 8-48 h, 1:6 mol/mol P:HCA) achieving a great atom efficiency. Indeed, the physical chemical properties of the SLIL permits the fast and easy extraction of products and non-reacted substrates in pure form, avoiding a costly purification step.

So far, the esterification of panthenol² and HCAs³ has been reported, however this strategy permits to obtain new bioactive products (Figure 1), still non available in the market, that unite the bioactive properties of the former raw materials, i.e. panthenol (humectant, moisturizing, anti-inflammatory, wound-healing, anti-aging...)^{iError! Marcador no definido.} and (hydroxy)cinnamic acids (UV-filter, anti-oxidant, anti-inflammatory, antimicrobial...)^{iError! Marcador no definido.}

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Intensificación de procesos biocatalíticos sostenibles para la captura de CO₂, la síntesis de cosmeceúticos y la despolimerización del poliuretano.

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El desarrollo de procesos químicos sostenibles es una necesidad para el mantenimiento del bienestar en nuestra sociedad y para crear una industria química competitiva, compatible con una conciencia medioambiental muy desarrollada. Para que esto sea posible, es necesario allanar el camino desde los conocimientos básicos adquiridos en las dos últimas décadas en el campo de la Química Verde hasta las aplicaciones industriales

Esta comunicación abunda en la intensificación de los procesos desarrollados con diseño y aplicación de nuevas herramientas verdes avanzadas. Dichas herramientas implican el uso de quimio y biocatalizadores activos y estables en sistemas inteligentes basados en ILs, DESs, agua o medios de reacción/separación sin disolventes, así como su implementación tecnológica para la conversión de CO₂ y materias primas renovables en productos químicos de valor añadido, además de la preparación de nuevos cosmeceúticos y la despolimerización catalítica del poliuretano

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Hydrolysis of polyurethane foam wastes using ionic liquid-superbase catalytic systems

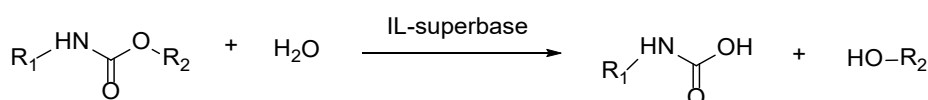
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The fossil origin of over 98% of today's produced polymers and the prevalent disposal method through incineration play a substantial role in contributing to CO₂ emissions throughout the life cycle. Addressing these challenges and minimizing the environmental impact of polymer waste needs the development of novel chemical and biochemical conversions for efficient recovery and recycling. According to Plastics Europe (2022), Europe produced 57.2 Mt of plastics in 2021 and generated 29.5 Mt of plastic waste in 2020. It is estimated that 65% of this plastic waste was landfilled or incinerated and only 35% was sent to recycling. Polyurethane (PU) have a yearly production tonnage of 18.6 MMT/year. As a result of the absence of reuse procedures, a large amount of PU foam wastes from mattress has been thrown out by simple landfilling with dramatical impact on the environment. However, the current strategies towards a fully circular plastic economy are not yet economically viable [1]. Thus, novel chemical re/upcycling strategies to depolymerize PU into valuable monomers to re-synthesize the same plastic or to convert it into a different material are foreseen.[2] It has been described that the combination of the ionic liquid technology,[3] and (bio)catalysis, has been shown as a suitable approach to carry out the easy and green depolymerization of PUF wastes (<100 °C, 1 bar, 6 h),[4] opposite to the classical glycolysis in reaction media based on a large excess in glycols, which occurs under harsh operation conditions (up to 180-250 °C, and pressures up to 50 bar), and long reaction times (24-48 h). We present here the use of ionic liquid-superbase combination technologies for the depolymerization of polyurethane foam wastes under mild conditions.



Scheme 1. Hydrolysis reaction of polyurethane.

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One-pot growth of MOFs on 3D-printed polymers for catalytic performance enhancement

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Metal-Organic Frameworks (MOFs) are materials which have emerged as a class of crystalline solids with enormous surface areas and porosity. Despite of their unprecedented chemical and structural tunability and excellent catalytic performance in liquid and gases reactions[3], it is challenging to build customizable MOF shapes with tunable mechanical properties because of their rigid and brittle powder morphology. Additive manufacturing (3D printing) is a useful technique that allows the design and construction of polymeric scaffolds improving the capacity to be used as a material for enhancement of reactivity in different catalytic reactions.[1] Growing of MOFs onto 3D printed polymeric materials could improve the typical agglomeration and mechanical attrition during the performance of catalytic cycles. In this contribution, one-pot growing of MOFs (e.g. UiO66-NH₂ and UiO67) onto the surface of 3D-printed polymers are reported. These novel MOF-3D printed composites have been characterized and applied as active catalytic materials for cycloaddition of CO₂ to epoxides under free additive and mild pressure conditions. [2]

Typical experimental procedure was divided into two steps: growing of MOFs into 3D printed polymeric epoxy devices and then its performance in the cycloaddition of CO₂ to epoxides. As a first step, ring-opening of epoxides (of the 3D-printed device) were performed by using 2-amino terephthalic acid and then incorporation of the ligand (biphenyl-4,4'-dicarboxylic acid) and zirconium chloride. Characterization of the materials was performed by XRD, XPS, ICP-MS, Raman and FTIR spectroscopies. The catalytic performance of these systems was evaluated for catalytic cycloaddition of CO₂ to epichlorohydrin at 80°C and CO₂ balloon. Kinetic curves were elucidated by using both NMR-¹H and FTIR. Reuse of reactors was also performed together with their use in a flow catalytic system.

The crystalline structure of the bulk and the *in-situ* grown UiO66-NH₂ and UiO67 onto 3D polymers showed the typical expected UiO-pattern, while the XPS results showed that the presence of certain amount of free carboxylate groups together with the coordination of the Zr. Raman spectroscopy in depth suggested more than two phases in the cases where the 3D-printing device was submitted to a MOF growing. Catalytic performance showed that MOF-3D printing composites lead to the cyclic carbonate with excellent yield (up to 99%) and with productivity values up to 225 mol_{epoxide}/mol_{Zr}.h surpassing the results previously achieved by analogous MOF. These devices also are robust and can be reused for several times without an apparent loss of their catalytic activity. Finally, its application under flow continuous conditions has been also assayed suggesting its potential for practical applications.

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Controlled Composition of Panthenyl Monoacyl Ester Mixtures for the Development of Emulsified Formulations

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Esters of polyhydroxylated compounds like panthenol are widely used in the cosmetic and pharmaceutical industries due to their bioactive properties, including humectant, moisturizing, anti-aging, and wound-healing effects.[1]

Esters are currently synthesized through chemical or enzymatic catalysis, offering a clean and sustainable process for industrial scaling up in organic solvent-free media. Introducing a biocatalyst, such as an enzyme, into the eutectic mixture enables the selective lipophilization of panthenol with free fatty acids of varying alkyl-chain lengths.[2] The resulting panthenyl monoacyl ester (PME) exhibits amphipathic properties.

In this context, the amphiphilic nature of panthenyl esters facilitates self-assembly, enhancing gel and emulsion formation. We report on the ability of modified panthenol derivatives to form oil-in-water emulsions (Figure 1) and examine how the composition and proportion of panthenyl monoacyl esters influence emulsion formation. Additionally, the rheological properties of oil-in-water emulsions have been identified as an important factor in the preparation of creams and gels for various topical applications.

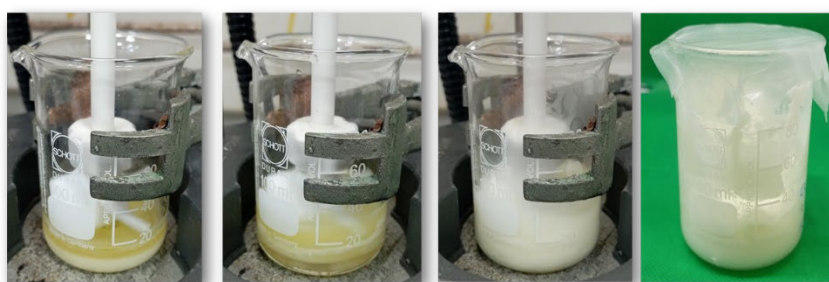


Figure 1. Protocol for oil-in-water emulsion formation.

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New biodegradable hydrogels from renewable raw materials for water absorption

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Nowadays there is a growing interest in hydrogels because of their multiple applications that range from drugs delivery or food additives to soil amendment.[i] According to their aim, different requirements and properties are demanded, like the natural composition and synthesis, their physical state (solid/liquid-phase or even stimuli-responsive) or a superabsorbent capacity. To date, non-biodegradable polymers lead this profitable market contributing to waste pollution, highlighting the need for more sustainable alternatives.

Glycerol is the byproduct in the synthesis of biodiesel to which the policy of circular economy has found a great range of applications.[ii]

In this work, different hydrogels have been synthesized from glycerol through its combination with citric acid, lactic acid and cellulose by means of eutectic mixtures. The stable net was obtained after a process of incubation at moderated temperatures followed by a curing step.

The water absorbing capacity of the obtained hydrogels has been determined at different pH, reaching values for water absorption index up to 960 %.

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Continuous flow chemo-enzymatic reactor for the sustainable synthesis of cyclic carbonates from CO₂

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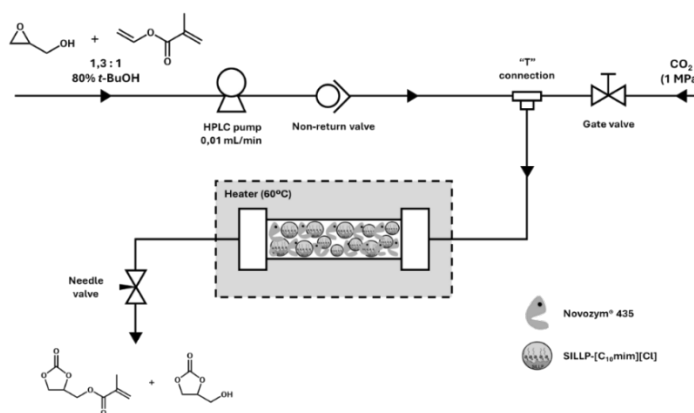
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Global warming is one of the greatest challenges which humanity must face nowadays, with CO₂ being one of the main gases causing the increase in the greenhouse effect. Thus, a key strategy to reduce its concentration in the atmosphere is its incorporation for the synthesis of value-added products in the chemical industry. Moreover, polyurethane is the sixth most widely used plastic material globally, whose traditional synthesis requires the use of highly toxic compounds such as phosgene or isocyanate, so it is vital to design new sustainable alternatives for its large-scale production. In this context, one of the most interesting sustainable strategies for CO₂ valorisation is the preparation of Non-Isocyanate Polyurethanes (NIPUs), following the principles of Green Chemistry. [1] Thus, it is worth highlighting the synthesis of glycidyl esters and the subsequent CO₂ cycloaddition on the epoxides to form the corresponding cyclic carbonates, which are monomers susceptible to react with polyamines to obtain the mentioned NIPUs. [2]

In this work, it is demonstrated the development of a continuous flow (bio)catalytic reactor system, using an immobilised commercial lipase and a supported ionic liquid as sustainable catalysts to produce glycerol carbonate methacrylate, a monomer with potential application in the synthesis of NIPUs.



Scheme 1. Continuous flow system designed for the chemo-enzymatic synthesis of glycerol carbonate methacrylate from CO₂.

Acknowledgment

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Multifunctional Ionic liquid for the preparation of catalytic micellar systems

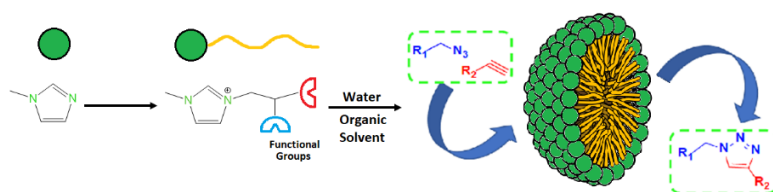
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In search of more environmental acceptable chemical transformations, the replacement of organic solvents by water is highly attractive as water is safe and benign medium. However, the low solubility of organic compounds hampers its wider application. Ionic liquids (ILs) with amphiphilic character have been used as surface-active ionic surfactants (Surface-Active Ionic Liquids, SAILs). Here we reported the preparation of multifunctional SAILs to create catalytic micelles. A family of novel surface-active surfactants will be prepared via -homocysteine thiolactone in the ILs scaffold that allows the “orthogonal” introduction of different moieties by double modification through aminolysis and subsequent thiol-click reaction. This route opens the way to the preparation of a large variety of amphiphilic functionalized ILs through the synthetic manipulation of a reduced number of common simple intermediates easily available even in large scale.[1] In this way, we have developed a family of ILs presenting at least co-existence of an ionic hydrophilic head group, an additional unit (e.g. amine groups) acting as metal ligand and a long hydrophobic tail favoring its self-aggregation in water/Me-THF mixtures. This functionalized SAILs in presence can act as an efficient micellar systems for Cu-catalyzed azide terminal alkyne cycloaddition (Cu-AAC) for synthesis of 1,2,3-triazoles (Figure 1). Under the optimized reaction condition, the catalyst showed efficient for the synthesis of 1,2,3-triazole with respect to the various acetylenes and halides. The systems here reported exhibit three main advantages: (i) high catalytic efficiency at low catalyst loading, (ii) recyclable, and (iii) lower E factors.



Scheme 1. IL synthesis and micellar formation in the mixture of organic and aqueous solvent.

Acknowledgements

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Síntesis sostenibles de carbonatos (poli)cíclicos a partir de CO₂ en medios no convencionales

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La emisión de dióxido de carbono (CO₂) es una de las principales causas del calentamiento global, así como la producción de materiales plásticos recalcitrantes, que suelen ser enterrados en vertederos o incinerados, causando un enorme daño ambiental. Se ha demostrado que las moléculas de carbonato de mono- y di-glicerol son monómeros útiles para la síntesis de poliuretano no isocianato (NIPU) de base biológica mediante una reacción de aminólisis con poliaminas, evitando la síntesis tradicional de poliuretano basada en fosgeno e isocianato, que son compuestos altamente tóxicos. [1] Sin embargo, la síntesis industrial de estos monómeros a partir de CO₂ requiere condiciones rigurosas de alta presión y temperatura (por ejemplo, 180°C y 50 bar), típicamente catalizadas por sales de amonio cuaternario. [2]

En este trabajo se ha descrito una estrategia sostenible basada en la síntesis quimioenzimática de carbonatos (poli)cíclicos a partir de aceites vegetales mediante dos etapas: una primera reacción de epoxidación sobre las insaturaciones catalizado por una lipase, seguido de una reacción de cicloadición sobre los grupos oxirano para la síntesis del carbonato cíclico catalizado por un líquido iónico soportado. Así mismo, se plantea un sistema de flujo en continuo a partir de vinil metacrilato, glicidol y CO₂ para la síntesis de glicerol carbonato metacrilato. Para la obtención de estos precursores para la síntesis de NIPUs (del inglés, “*Non-Isocyanate Polyurethanes*”) se han empleado biocatalizadores y líquidos iónicos, fomentando la filosofía de la Química Verde. [3]

Agradecimientos: Este trabajo fue parcialmente apoyado por MICINN-FEDER-AEI 10.13039/501100011033 (PID2021-124695OB-C21/C22 y PDC2022-133313-C21/C22), MICINN – Unión Europea Next Generation EU-PRTR (TED2021-129626B-C21/C22) y SENECA (21884/PI/22) para apoyo financiero.

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Sustainable depolymerization strategies for polyurethane foam

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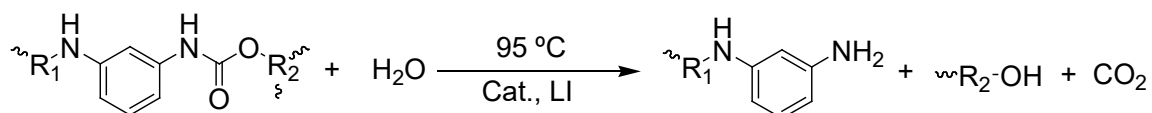
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The increase in the production of plastics has become one of the main turning points of modern society. Among plastic materials, polyurethane (PU) reached a production of 21.2 Mt in 2022 owing to their high resilience, durability, and versatility which ultimately leads to the generation of huge volumes of plastic waste.[1] PU is synthesized from two monomers, a diol, and a highly toxic isocyanate, providing the characteristic urethane bond (-NHCOO-). Nevertheless, the chemical methods for recycling PUs are not suitable because of the use of severe reaction conditions (i.e. >200 °C).[2]

Herein, this study aimed to develop a depolymerization strategy for PU foam (PUF) under mild reaction conditions by using an active reaction media based on Ionic Liquids (ILs) and a superbase catalyst (see Scheme 1). The hydrolysis of PUF was achieved at temperature below 100 °C, obtaining monomeric compounds (recycled polyols) that can be separated from the active media, thus demonstrating the industrial potential of this process.[3]



Scheme 1. Schema for the chemical hydrolysis of PUFs.

Acknowledgments: This work was partially supported by MICINN-FEDER-AEI 10.13039/501100011033 (PID2021-124695OB-C21/C22 and PDC2022-133313-C21/C22), MICINN–European Union Next Generation EU-PRTR (TED2021-129626B-C21/C22) and SENECA (21884/PI/22) for financial support.

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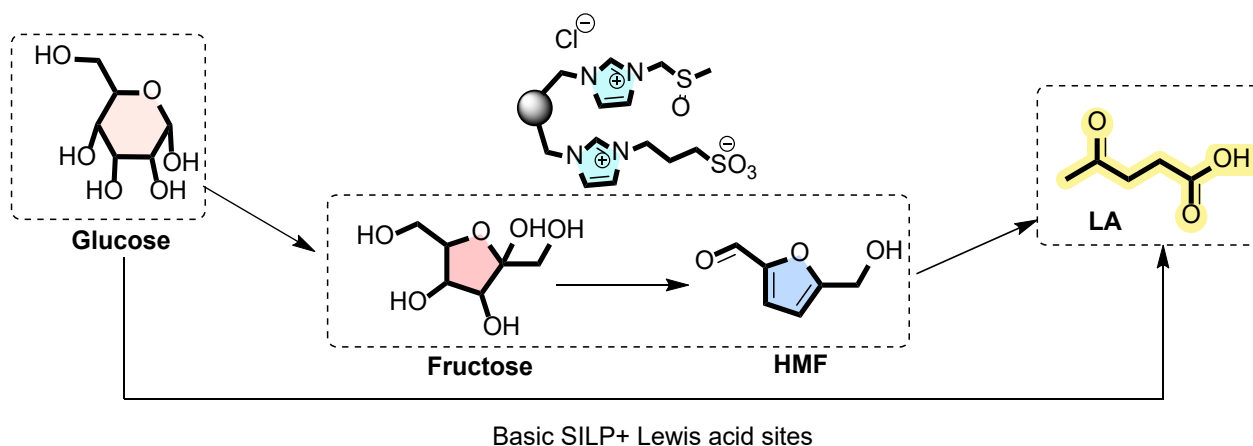
A sweet transformation: Synthesis of Sugar Derivatives by Using Supporting Ionic Liquid Phases

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Interest in biomass is growing due to its potential to replace petroleum-derived chemicals and its renewability.[1] Sugars offer a promising alternative for producing valuable industrial chemicals.[2] This study explores transforming sugars (glucose and fructose) into either 5-hydroxymethylfurfural (HMF) or levulinic acid (LA) using supported ionic liquid phases (SILPs). First, fructose dehydration to HMF was achieved at mild conditions with a bifunctional SILP containing both dimethyl sulfoxide (DMSO) and sulfonic groups. This catalyst yielded up to 90% HMF on the gram scale and remained stable for three consecutive uses. Secondly, cascade conversion of glucose to LA was achieved using a basic SILP impregnated with copper (Cu) as a Lewis acid site. The basic SILP alone only isomerized the glucose, but Cu significantly increased reaction rate and yield of LA. The Cu and basic SILP can be reused for multiple cycles. Adjusting reaction conditions like Cu loading, time, or temperature allows selective control over HMF or LA production, achieving good to excellent yields for both products. Both LA and HMF can also be used as chemical intermediates for the synthesis of potential surfactants by using esterification or possible monomers for the synthesis of biopolymers. This research exemplifies the use of SILP for the valorization of sugar-derivative biomass.



Scheme 1. Sugar transformation (glucose and fructose) into chemicals using SILPs.

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(Bio)catalytic applications of water-soluble palladium complexes

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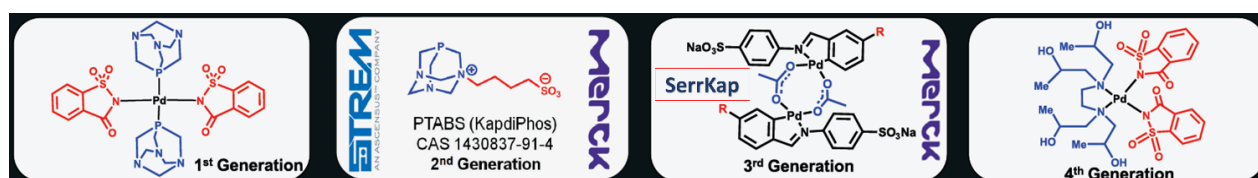
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The use of water-soluble catalysts in biphasic systems/neat water has received increasing attention, since they promote environmentally respectful processes with additional practical advantages, like a simple extractive work-up. The incorporation around the metallic centre of hydrophilic ligands, that usually achieve such property through hydrophilic substituents, has been the preferred strategy to stabilize water-soluble homogeneous catalysts [1]. We initiated in 2014 the exploration of phosphine ligands and phosphine containing complexes like those displayed as 1st and 2nd generation in the scheme, and have devoted the last few years to the synthesis of phosphine-free palladium complexes able to catalyze the room-temperature modification of nucleoside analogues in water through cross-coupling reactions [2]. The preliminary cytotoxic activity of the four generations of Pd-complexes is currently being tested, and this and other recent advances in its use as catalysts in different processes as the ones described below will be presented.

Thus, 3rd generation SerrKap palladacycle has been used in the development of a rapid flow-based cross-coupling synthesis protocol for a variety of C5-pyrimidine substituted nucleosides. The scale-up potential of this methodology approach was also demonstrated on a multigram synthesis of the antiviral nucleoside Brivudine.



Scheme 1.

Recent confinement into mesoporous silica support of 1st generation complex [Pd(sacc)₂(PTA)₂] has shown to increase the activity and stability of the palladium catalytic species for the room-temperature aqueous hydrogenation of either bicarbonate or CO₂ into formic acid.

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Ionic Liquids as sustainable tools for polyurethane foam depolymerization

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Polyurethanes (PUs) are a unique family of polymers with exceptional physico-chemical properties, which allow them to be used in a wide range of applications (e.g. coatings, binders, foams for mattresses, or vehicle seats.). This versatility makes PU the 6th most produced plastic in the world,[1] accounting for almost 25% of plastic waste management in the European Union. In this context, polyurethane foams (PUFs) are a type of thermosetting polymer whose enormous recalcitrant nature greatly complicates their recycling. In fact, no efficient strategy for their depolymerization has been reported to date. This study aimed to develop such a strategy by coupling Ionic Liquids (ILs) technologies and superbasic catalysts (see Figure 1).[2]

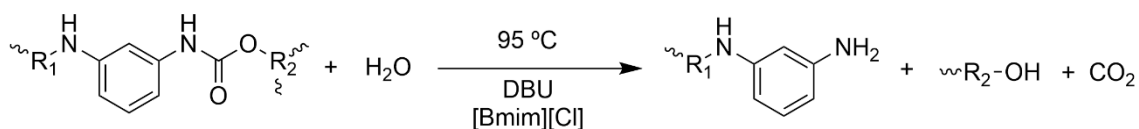


Figure 1. Schema for the chemical hydrolysis of PUFs, obtaining the corresponding amine intermediates, polyols, and CO₂.

Furthermore, unlike chemical depolymerization strategies described in the literature, which use organic solvents and extreme temperature (200 – 400 °C) and pressure (15 – 50 bar) conditions,[3] the proposed depolymerization protocol is carried out under mild reaction conditions (95 °C and atmospheric pressure) using easily recoverable and completely reusable non-conventional solvents, such as ILs.[4] Throughout the experimentation, the influence of different ILs and superbases as catalysts was assessed by characterizing the resulting products (recycled polyols) through solubility tests in dimethyl sulfoxide (DMSO), analysis of the hydroxyl index, and spectroscopic techniques such as nuclear magnetic resonance (NMR) or Fourier-transform infrared (FTIR). Finally, the feasibility of scaling up the process (up to 200 times) was demonstrated.

Acknowledgements

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Sustainable chemo-enzymatic processes for CO₂ capture

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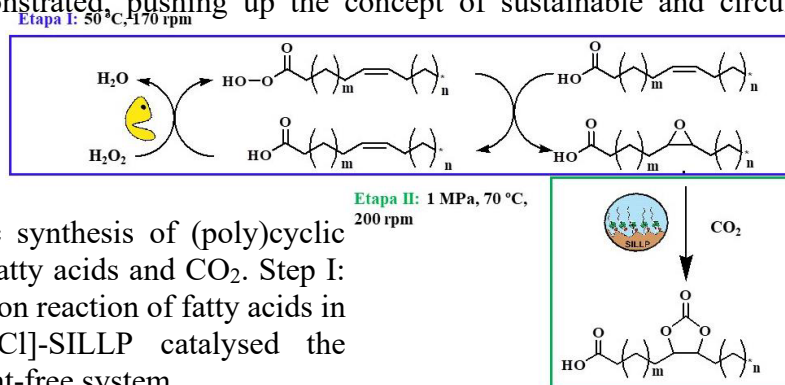
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The increased demand for polyurethane (PU) requires the development of new pathways that avoid the use of phosgene and isocyanates, which are highly toxic compounds. [1] In this context, the synthesis of non-isocyanate polyurethane (NIPUs) by means of an aminolysis reaction from cyclic carbonates and polyamines is one of the most relevant alternatives to traditional PUs. [2] In addition, the synthesis of cyclic carbonates can be carried out by incorporating CO₂ into epoxides subsequent cycloaddition reaction. [3]

In this work, a sustainable chemo-enzymatic approach for the synthesis of cyclic carbonates from unsaturated fatty acids and CO₂ has been successfully developed. The process involves the enzymatic epoxidation of the double bonds present in different fatty acids (e.g., oleic acid) and the subsequent CO₂ cycloaddition reaction by taking the advantages of ILs technologies (See Scheme 1). Furthermore, the efficiency and synergy when combining Novozym 435 as a biocatalyst and ILs for obtaining cyclic carbonate derivatives have been demonstrated, achieving conversion values up to 100%. Finally, the full recovery and reuse of both catalysts have been successfully demonstrated, pushing up the concept of sustainable and circular chemistry.



Scheme 1. Chemo-enzymatic synthesis of (poly)cyclic carbonates from unsaturated fatty acids and CO₂. Step I: Lipase-catalysed the epoxidation reaction of fatty acids in SLIL; Step II: [C10mim][Cl]-SILLP catalysed the cycloaddition of CO₂ in solvent-free system.

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