8th YOUNG POLYMER SCIENTISTS SEMINAR

29th October 2024



Book of Abstracts













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INDEX OF CONTENTS

Scientific program
Invited Speakers7
Polymers and Polymer-Based Composites for Additive Manufacturing
Thanks to the Spanish Research Council, my journey, storytelling9
Oral Communications
Crystallization-Induced Hydrogels: Modulating Mechanical and Thermal Properties for Smart Materials
Energetics and structure of Poly(trifluoroethylene): A DFT study
¹ H NMR as quantitative approaches to study molecular structure and dynamics on rubbers I 3
Biological Properties of Cannabidiol-Loaded PLGA Nanoparticles Engineered by Microfluidics 14
Temperature-sensitive and DNA-coating Polymers by an Aza-Michael Reaction
Thermosensitive hydrogels for skin regeneration with applications in drug delivery and gene therapy
Conformational and Microrheological study of novel biopolymers
Plasma-derived fibrin hydrogels with perfusable microchannels: Fabrication using sacrificial materials
Biobased polymers hydrogels for sustainable energy applications
Bio-composite innovation: merging biopolymers and agricultural-waste for novel materials20
Synthesis of polymeric matrices doped with fluorescent silica nanoparticles (Dye@SiO2) for polymer optical fibers (POFs) development as future solar concentrators (LSCs)21
Tailoring n-type thermoelectric properties in lignin-derived hydrogel through amination22
Ionic cellulose aerogels for industrial wastewater treatment
Optimization of the VARI process and recycling of laminates produced with Akelite resin24
Development of Sustainable Electrochemical Separators Based on Polylactic Acid Membranes for Secondary Batteries
Modification of the wettability of polymers by laser irradiation with nanosecond and femtosecond pulses
Advanced 3D Printing Techniques for fabricating Hierarchically Porous Membranes for Efficient Oil-Water Separation
There is no glitch in the matrix: Different elastomeric matrices and self-healing capability28
Reprocessing of biopolymer-based aerogels
Quinone-based hyper-crosslinked polymers as photocatalysts for valorization of glycerol30



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SusPlast

Flash Presentations	31
Comparative study of vulcanization systems for more sustainable materials	32
Comparative study of pla with microcellulose fibers obtained by melt and solution electrospinning	
Towards a reversible self-crosslinkable propylene-anthracene copolymers with improve thermal and dielectric stability	d 34
Effect of Alginate from Rugulopteryx Okamurae Seaweed on the 3D-Printing of Soy Pro Based Materials	otein 35
Composite aerogels based on cellulose fibres and acid collagen for potential applications wound dressings	s as 36
Bacterial cellulose and collagen bilayer systems for the controlled release of bioactive compounds	
Development of biomaterial inks based on pectin and hyaluronic acid hydrogels for 3D	printing 38
ECO-strategies to obtain sterile aerogels	
Posters	40
Improving the Stability of Guar Gum-Based Hydrogels through Semi-Interpenetrating Po Networks for Biomedical Applications	olymer 41
Synthesis of Biodegradable, Super-Porous Polymeric Networks Based on Guar Gum for Gastroretentive Controlled Drug Release	
Development of multilayer films based on recycled pla and caseinate reinforced with bac cellulose from kombucha fermentation	cterial 43
Self-healing polymers as electrical insulation	44
Characterization of High Impact Polystyrene Materials for Potential Use in Food Packagi	ing45



8th YOUNG POLYMER SCIENTISTS SEMINAR, SEJIPOL2024

29th October 2024, Sala 110, ICTP-CSIC

Scientific program

- 8:45 9:00 Registration
- 9:00 9:15 Welcome
- 9:15 9:45 Invited Speaker Alberto Sanz de León, Universidad de Cadiz Polymers and Polymer-Based Composites for Additive Manufacturing
- 9:45 10:30 ADVANCES IN POLYMER SYNTHESIS AND CHARACTERIZATION Chair: Anselmo del Prado
- 9:45 10:00 Alexis Alvear, POLYMAT/ICTP-CSIC Crystallization-Induced Hydrogels: Modulating Mechanical and Thermal Properties for Smart Materials
- 10:00 10:15 Carlos García Arcos, UNED Energetics and structure of Poly(trifluoroethylene): A DFT study
- 10:15 10:30 Fernando Martin Salamanca, ICTP-CSIC IH NMR as quantitative approaches to study molecular structure and dynamics on rubbers

10:30 – 10:50 FLASH PRESENTATIONS

Chair: Rosa Barranco

Juan Carlos Chicharro Sestines, ICTP-CSIC Comparative study of vulcanization systems for more sustainable materials

Elena Navas Ortiz, ICTP-CSIC Comparative study of PLA with microcellulose fibers obtained by melt and solution electrospinning

Angélica Martin Lorenzo, ICTP-CSIC

Towards a reversible self-crosslinkable propylene-anthracene copolymers with improved thermal and dielectric stability

Mauricio Vera Arévalo, Universidad de Sevilla Effect of Alginate from Rugulopteryx Okamurae Seaweed on the 3D-Printing of Soy

Protein Based Materials











TRSEQ

11:30 – 13:15 BIODEGRADABLE POLYMERS AND BIOMATERIALS Chairs: Sergio Martín-Saldaña

- 11:30 11:45 Pedro Luis Echevarría Torres, ICTP-CSIC Biological Properties of Cannabidiol-Loaded PLGA Nanoparticles Engineered by Microfluidics
- 11:45 12:00 Santiago Sarasa San José, INMA Temperature-sensitive and DNA-coating Polymers by an Aza-Michael Reaction
- 12:00 12:15 Ana García Crespo, ICTP-CSIC

Thermosensitive hydrogels for skin regeneration with applications in drug delivery and gene therapy

- 12:15 12:30 Andrés Cardil Tornos, IEM-CSIC Conformational and Microrheological study of novel biopolymers
- 12:30 12:45 Alejandro Hernández Sosa, ICTP-CSIC Plasma-derived fibrin hydrogels with perfusable microchannels: Fabrication using sacrificial materials
- 12:45 13:00 Sergio Javier Peñas Nuñez, POLYMAT Bio-based polymer hydrogels for sustainable energy applications
- 13:00 13:15 Laura Cabrera Villamizar, IATA-CSIC Bio-composite innovation: merging biopolymers and agricultural-waste for novel materials
- 13:15 14:15 LUNCH
- 14:15 14:45 Invited Speaker

Marian Cruz Tejedor, REPSOL

Thanks to the Spanish Research Council, my journey, storytelling.

- 14:45 16:30 POLYMERS AND COMPOSITES FOR ADVANCED APPLICATIONS Chairs: Enrique Blázquez
- 14:45 15:00 Miriam Guadaño Sánchez, ICTP-CSIC

Synthesis of polymeric matrices doped with fluorescent silica nanoparticles (Dye@SiO₂) for polymer optical fibers POFs) development as future solar concentrators (LSCs)

- 15:00 15:15 Nazish Jabeen, University of Valencia Tailoring n-type thermoelectric properties in lignin-derived hydrogel through amination
- 15:15 15:30 Álvaro Vázquez Romero, ICTP-CSIC Ionic cellulose aerogels for industrial wastewater treatment
- 15:30 15:45 David Martin Crespo, ICTP-CSIC Optimization of the VARI process and recycling of laminates produced with Akelite resin





TRSEQ

Sección

Territoria

polymers

15:45 – 16:00 Clemente Rodríguez, ICTP-CSIC

Development of Sustainable Electrochemical Separators Based on Polylactic Acid Membranes for Secondary Batteries

16:00 - 16:15 Patricia Martínez García, NANOesMAT, UNED

Modification of the wettability of polymers by laser irradiation with nanosecond and femtosecond pulses

16:15 - 16:30 Rayane Akoumeh, Qatar University

Advanced 3D Printing Techniques for fabricating Hierarchically Porous Membranes for Efficient Oil-Water Separation

16:30 – 16:50 FLASH PRESENTATION Chairs: Daniel Domingo

Paula García Abril, Aerofybers Technologies SL

Composite aerogels based on cellulose fibres and acid collagen for potential applications as wound dressings

Daniel Lechuga Cruz, ICTP-CSIC

Bacterial cellulose and collagen bilayer systems for the controlled release of bioactive compounds

Jorge Mercado Rico, ICTP-CSIC

Development of biomaterial inks based on pectin and hyaluronic acid hydrogels for 3D printing

María Carracedo Pérez, Universidad Santiago de Compostela ECO-strategies to obtain sterile aerogels

16:50 – 17:35 SUSTAINABILITY, VALORIZATION AND POLYMER RECYCLING Chair: Suman Thakur

16:50 - 17:05 Itziar Mas Giner, ICTP-CSIC

There is no glitch in the matrix: Different elastomeric matrices and self-healing capability

17:05 – 17:20 María Blanco Vales, Universidade de Santiago de Compostela Reprocessing of biopolymer-based aerogels

17:20 – 17:35 Angela Matarin Serrano, ICMM-CSIC Quinone-based hyper-crosslinked polymers as photocatalysts for valorization of glycerol







Territorial de Madrid polymers

TRSEQ

Invited Speakers



Polymers and Polymer-Based Composites for Additive Manufacturing

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<u>Abstract</u>

Additive manufacturing consists of a number of fabrication technologies in which objects are built layer by layer from a computer aided design. Unlike traditional subtractive manufacturing methods (such as machining or milling), where material is removed from a solid block, additive manufacturing adds material only where it is needed, minimizing waste. This process, commonly known as 3D printing, uses a wide variety of materials, including plastics, metals, or ceramics, and is applied across different industries, from nanotechnology to the transportation sector.

Herein will be presented the most relevant results of our research on polymers and polymer matrix composites, covering topics from material synthesis and characterization to the development of functional prototypes and final products. It will be demonstrated how the additive manufacturing process and the mechanical properties of the fabricated objects are influenced by the selection of polymers and additives at the macro, micro, and nano levels, as well as their chemical structure. In particular, results obtained using fused filament and granular fabrication (FFF and FGF), stereolithography (SL), and two-photon polymerization (2PP) technologies will be discussed.



Thanks to the Spanish Research Council, my journey, storytelling

Marian Cruz Tejedor

REPSOL

Abstract

Thinking out of the box, this is what R&D means and what we are proud of as scientists. There is not Artificial Intelligence model that can generate so many new ideas based on human experience and well-trained brains, maybe in the future, who knows. let's celebrate together how we grew up, embracing change, playing with our hands and thinking of innovation every day, reading books and learning from each other without the internet or youtube tutorials.

When we work together, we will travel long distances, if we were alone, it would be more difficult to reach the top. This is my journey, storytelling.



Oral Communications



Crystallization-Induced Hydrogels: Modulating Mechanical and Thermal Properties for Smart Materials

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Abstract

Over the last few years, there has been an increasing interest on the development of hydrogels with self-healing and shape memory properties for applications in additive manufacturing techniques (3D and 4D (bio) printing). Very recently, a polyacrylic acid with crystallizable hexadecyl side chains was synthesized and a proof of concept of its employment in 4D printing was shown. [1] Our group has recently reported on the self-assembly of block copolymers with crystallizable blocks to form hydrogels, studying their structural organization as a function of copolymer architecture and paving their way to their application as polymer biomaterial inks for 3D direct printing. [2]

In this communication, we will show recent results regarding the development of crystallizationinduced hydrogels in which mechanical and thermal properties can be modulated through the crystallinity of both the polymer backbone and the side chains. To this aim, polyvinyl alcohol (PVA), a semicrystalline polymer able to form physical hydrogels through freezing and thawing cycles was chosen.[3] PVA offers inherent advantages, like hydrophilicity, biodegradability, and biocompatibility. However, it produces physical hydrogels that are mechanically weak. To address this limitation, and increase the functionality, We propose the fabrication of thermoreversible hydrogels based on polyvinyl alcohol (PVA) mixed with alkyl chains with the objective is to explore how the incorporation of alkyl chains affects the crystal formation and the thermal response of the hydrogels, with potential applications in controlled drug delivery systems and smart materials.



Energetics and structure of Poly(trifluoroethylene): A DFT study

Carlos G. Arcos (UNED. Ponente), Oscar Toledano (IEM-CSIC), Oscar Gálvez (UNED)

UNED

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Abstract

Polytrifluoroethylene (PTrFE) is an organofluorinated polymer that plays a key role in the formation of copolymers. Its bonding with polyvinylidene fluoride (PVDF) facilitates the orientation of this polymer towards the formation and stability of its phases with ferro- and piezoelectric properties with respect to the homopolymer. These phases are also of great interest for many types of applications, ranging from the pressure and temperature sensor industry to signal transduction and the creation of novel energy harvesting devices. While the structural arrangement of PVDF is well known, the structure of TrFE has been studied by a very small number of authors and its structural conformation has not yet been agreed upon. For this reason, here we present an approach to its characterization based on high-level ab initio calculations using DFT methods, with the aim of evaluating the structural stability of the different PTrFE conformers from their energy profile and the simulation of their X-ray diffraction pattern



¹H NMR as quantitative approaches to study molecular structure and dynamics on rubbers

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Abstract

The elasticity of rubber compounds is determined by the molecular parameters that define the network structure. Various experimental techniques have been applied to obtain these parameters. The quantification of those parameters allows us to understand at a deeper level the elasticity in rubber materials. Here, in the last years, several nuclear magnetic resonance (NMR) approaches appeared which allow to expand our knowledge.

Multiple Quantum Nuclear Magnetic Resonance (MQ-NMR) is a well-established technique to characterize the average molecular structure of rubber materials based on the correlation function and spin-spin relaxation. The residual dipolar coupling is the main observable that quantifies the constraint density at a molecular level. This approach is also sensitive to the non-elastically active fraction of material, i.e. the fraction of defects.

Field cycling NMR has been widely applied to study standard polymers. Molecular dynamics can be directly probed by measuring the spin-lattice relaxation rate over a wide range of Larmor frequencies. Using an advanced home-built relaxometer and appealing to frequency-temperature superposition, relaxation spectra are revealed which cover about ten decades in frequency. They are interpreted in terms or Rouse and entanglement dynamics. Their spectral changes are monitored when rubbers with different crosslink densities and swelling degrees are studied.

By means of both NMR approaches we can evaluate the effect of crosslink density to NMR signals, obtaining unique information about molecular structure and dynamics on rubbers. This project elucidates the importance of applying quantitative methods to characterize rubbers, enhancing the value of a unified theoretical framework for this purpose.



Biological Properties of Cannabidiol-Loaded PLGA Nanoparticles Engineered by Microfluidics

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Abstract

The study focuses on developing cannabidiol-loaded poly(lactic-co-glycolic acid) (PLGA) nanoparticles (CBD-NPs) using microfluidic technology. Cannabidiol (CBD), a non-psychoactive cannabinoid, is known for its anti-inflammatory, antioxidant, and antibacterial properties, making it a promising compound for wound healing. However, its low solubility and bioavailability limit its clinical applications. This research aims to overcome these limitations by synthesizing CBD-loaded PLGA nanoparticles that enable controlled release, enhancing CBD's therapeutic efficacy.

The CBD-NPs demonstrated high encapsulation efficiency, stable hydrodynamic sizes, and consistent zeta potentials, retaining stability over 56 days. Antioxidant assays showed enhanced radical scavenging activity, while antibacterial tests against Staphylococcus aureus identified a minimum inhibitory concentration of 1.25 mg/mL. Importantly, cytotoxicity assays revealed reduced cytotoxic effects of the nanoparticles compared to free CBD, promoting higher cell viability in macrophages, keratinocytes, and fibroblasts—critical cells in wound healing.

This study highlights the potential of CBD-NPs to improve wound healing outcomes through their antioxidant, antibacterial, and biocompatibility properties.



Temperature-sensitive and DNA-coating Polymers by an Aza-Michael Reaction

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Abstract

In this project, we investigate the synthesis and application of novel biodegradable polymers

through an aza-Michael addition reaction. We have developed a set of poly- β -(hydrazide ester)s with an amphiphilic chemical structure exhibiting thermoresponsive behavior and liquid-liquid phase separation, a phenomenon which depends on the length of their hydrophobic side chains. On the other hand, we have synthesized a series of cationic poly(β -amino ester)s derivatives that have shown some interaction with DNA-based nanomaterials with potential for improving their therapeutic performance in biological environments.



Thermosensitive hydrogels for skin regeneration with applications in drug delivery and gene therapy

Ana García-Crespo, Juan Rodríguez-Hernández, Helmut Reinecke, Alberto Gallardo, Carlos Elvira, Enrique Martínez-Campos.

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Abstract

Despite recent advances in biomedicine, cell therapy for skin regeneration faces several challenges such as limited cell integration. Transplanted cells often have difficulties to adhere, proliferate and differentiate properly at the damaged site, which compromises the efficacy of the therapy. In this context, polyvinylcaprolactam (poly-VCL) hydrogels are polymeric networks with thermosensitive properties that allow cell culture and controlled detachment as a monolayer by temperature decrease. These monolayers could play a crucial role in skin's regeneration, specifically most external layers, facilitating better cell integration into the damaged tissue.

These hydrogels can be applied as free membranes and dressings (free hydrogels) or as coatings on multiwell plates, highlighting their versatility in diverse biomedical applications. In addition, this technology offers the possibility to be customised by incorporating drugs, such as antibiotics or growth/differentiation factors, in the form of poly-VCL-based particulate systems, obtained using supercritical CO2 technology, and due to their swelling and thermosensitive properties, poly-VCL hydrogel systems allow for controlled drug release. Similarly, it is possible to form polymer-RNA complexes based on cationic polymers, that can be integrated into the hydrogel matrix, protecting the RNA from degradation and facilitating its introduction into the target cells. These systems would enable the development of more precise gene therapies for treating genetic or degenerative diseases of skin layers.



Conformational and Microrheological study of novel biopolymers

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Abstract

Bacteria can produce a wide range of biopolymers. They present a huge variety of possible chemical structures and compositions, depending on the bacteria and its growing conditions [1]. Bacterial biopolymers are very interesting materials for industrial application due to their rheological and chemical properties. Obtaining has many advantages over other bio-based sources, like plants or algae, thanks to the stability of production under controlled conditions [1]. Even so, the range of bacterial biopolymers is sorely underexplored [2].

From a biotechnological standpoint, secreted polysaccharides are the most promising. They have been applied in different industrial sectors as textiles, adhesives, alimentary additives, or spillage treatments for example [3]. We focused our attention on a novel polysaccharide with potential biotechnological applications. This biopolymer is (1-3)(1-4)- β -glucane (mixed linkage β -glucane, MLG) [4].

We have studied the properties of MLG using a variety of characterization techniques (FTIR, DSC, GPC/SEC). The viscoelastic properties of polysaccharide solutions depend on its concentration, molecular weight and chain rigidity. To study the conformational nature of MLG we measured the hydrodynamic size using dynamic light scattering (DLS) and its intrinsic viscosity using triple-detection GPC/SEC.

We compared the behaviour of MLG with other polysaccharides with varying amounts of $\beta(I-3)$ y $\beta(I-4)$ links, like Curdlan, Barley, Lichenan Pullulan and Hydroxypropylcellulose. Microrheological measurements were performed to compare their viscoelastic properties.

- [1] Rehm, B.H.A. Nature Reviews Microbiology (2010)
- [2] Pérez-Mendoza, D., et al. Curr Opin Microbiol (2016)
- [3] Nwodo, U.U.;et al. International Journal of Molecular Sciences (2012)
- [4] Pérez-Mendoza, Det al. Proc Natl Acad Sci USA (2015)



Plasma-derived fibrin hydrogels with perfusable microchannels: Fabrication using sacrificial materials

Alejandro Hernández-Sosa, Cristina Quílez, A. Benéitez Fernández, M.C. Muñoz Turrillas, Diego Velasco, Rebeca Hernández

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Abstract

Skin disorders significantly impact patients' well-being, and there are few available medications. Recently, advances have been made in developing in vitro human skin models for testing cosmetic and pharmaceutical products. However, these models lack appendages like blood vessels, sweat glands, or hair follicles, limiting their ability to represent normal human physiology. Current research focuses on creating more advanced models with perfusable vasculature, essential for treating severe skin injuries like burns.

Fibrin, a protein derived from blood, it is an useful biomaterial to fabricate skin models since it can form hydrogels with relevant bioactive responses. Nowadays, the goal is to pattern these fibrin hydrogels so that they have structures that mimick vasculature.

Methods to generate channels inside fibrin hydrogels include removable structures, laser ablation, soft lithography, and 3D printing. 3D printing has proven effective in creating vascularized hydrogels using materials like PVA or gelatin, which can be removed without toxic effects. This process has been successful in forming interconnected channels.

In this work, we present advances using sacrificial materials to generate channels inside fibrin hydrogels. We will focus on the use of sacrificial spacers, such as nylon wires, and on removable materials such as PVA and gelatin. Overall, we were able to generate fibrin hydrogels with interconnected perfusable channels of milimetric size.



Biobased polymers hydrogels for sustainable energy applications

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<u>Abstract</u>

Introduction

Batteries are a fundamental technology to drive the transition towards sustainability, boost green transportation, reduce the carbon footprint, and promote the goal of achieving climate neutrality.. They are employed for a plethora of applications ranging from vehicles, mobile phones, or home appliances up to medical devices such as human recording electrodes and biosensors. In this regard, power sources that are free from shape constraints and can conform to various shapes are highly desired to facilitate the integration of the energy storage system into the device. Nevertheless, the environmental impact of the usage of conventional batteries is a major concern and the development of sustainable batteries has become a challenge for researchers. In this work, natural polymer-based hydrogels are investigated as injectable matrixes to host carbonaceous electrically conductive materials leading to shape-defined semisolid electrodes with the potential to reduce the environmental impact of the batteries of the future. This novel material is developed to be part of the battery's electrodes and electrolyte, and can be an alternative to conventional systems for the transition to more sustainable energy devices.

Methodology

Supramolecular hydrogels based on natural polymers, alginate and/or chitosan, crosslinked with potassium chloride were employed as matrixes to embed carbon black and Prussian blue giving rise to injectable electrodes. The hydrogel formation was confirmed by inverted tube tests and the chemical interactions were determined by infrared spectroscopy. The injectability properties were assessed by syringe injection tests and rheological characterization. Ionic and electronic conductivity measurements were also performed.



Bio-composite innovation: merging biopolymers and agricultural-waste for novel materials

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Abstract

Developing sustainable alternatives to conventional plastics has been a necessary research area in recent years. Thus, as the primary focus of this study, the development of degradable and more bio-based composites was addressed. We explored the development of biodegradable and bio-based composites using polyhydroxybutyrate-co-valerate (PHBV) and poly(butylene adipateco-terephthalate) (PBAT). To enhance sustainability, rice straw, a low-cost and eco-friendly agro-industrial residue, was incorporated as a filler. Various PHBV:PBAT ratios (80:20, 50:50 and 20:80) and rice straw loadings (20, 30 and 40%) were investigated, along with different particle sizes (\leq 250µm and \leq 500µm). The materials were characterized for mechanical properties, thermal stability, water vapor permeability, and disintegration according to UNE-EN ISO 20200:2016 over 90 days.

Results showed that rice straw particle size had minimal impact on mechanical properties and contact angle. However, increasing filler content led to higher Young's modulus but decreased tensile strength and elongation. Thermal profiles were also influenced by filler incorporation. Detailed characterization of biocomposites with 80:20 and 50:50 PHBV:PBAT ratios, with and without 40% rice straw filler showed that composites with higher PHBV content exhibited lower permeability, while filler addition increased permeability. Disintegration studies revealed that 80:20 PHBV:PBAT formulations achieved over 93% disintegration, while 50:50 blends showed less than 90% disintegration.

These results underscore the potential of using agricultural waste as a sustainable resource in the development of biopolymer composites. They also provide crucial insights into the formulation and optimization of environmentally friendly materials, paving the way for the advancement of sustainable alternatives to conventional plastics.



Synthesis of polymeric matrices doped with fluorescent silica nanoparticles (Dye@SiO₂) for polymer optical fibers (POFs) development as future solar concentrators (LSCs)

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Abstract

This work focuses on the synthesis of polymeric preforms with optical capabilities by inserting nanoparticles with fluorescent properties for the development of polymer optical fibers (POFs) for uses as solar concentrator. The preforms were developed with coaxial structure through free radical polymerisation of methacrylate-derived polymers. These polymers exhibit high hardness properties and favourable processing conditions for the POFs manufacturing, as well as high resistance to solar exposure. Furthermore, the coating was conducted in the presence of silica nanoparticles (NPs) doped with perylene. Silica doped nanoparticles (Dye@SiO2) were synthesised by mixing two different types of silanes that encapsulated the dye inside. The characterisation of the polymers was performed by gel permeation chromatography and differential scanning calorimetry obtaining optimal molecular weight values of 100000 - 120000 g/mol and a glass transition of 100 $^{\circ}$ C allowing the resulting preforms to be not only tough but also malleable for manufacturing POFs. Moreover, the size and morphology of the NPs was determined by TEM providing a particle size around 20 nm and a highly homogeneous size distribution. Finally, their optical properties were characterised by fluorescence and ultraviolet visible spectroscopy and the amount of dye encapsulated in the silica nanoparticles was determined, obtaining concentrations of 11.2 ± 4.8 mg of perylene per gram of silica.

Acknowledgment

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Tailoring n-type thermoelectric properties in lignin-derived hydrogel through amination

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Abstract

Fossil fuel depletion and climate disasters have sparked interest in capturing low-grade heat and converting it into electricity to address the energy crisis. Ionic thermoelectric (i-TE) materials have garnered significant attention due to their unique properties such as excellent Seebeck coefficients, low thermal conductivity, and high electrical conductivity. These i-TE materials utilize ions as charge carriers, enabling the conversion of heat into electricity. Lignin presents an innovative approach to develop hydrogels as sustainable materials for application in ionic thermoelectricity. This study focuses on the synthesis of lignin-derived hydrogels through chemical crosslinking and then amination with amine group (switching p-type to n-type i-TE material). For functionalization, this research utilizes a green synthesis method. The amination of crosslinked hydrogel with 2-Chloroethylamine hydrochloride as greatly affected by the optimized reaction condition. The synthesized crosslinked hydrogels exhibit lower value of Seebeck coefficient (1.614 mVK-1), ionic conductivity (4.04 mScm-1) and thermal conductivity (0.22 Wm-1. K), that give rise to small value of ionic figure of merit (0.0013). However, functionalized hydrogels result in high negative Seebeck coefficient (-7.873 mVK-1), superior ionic conductivity (6.09 mScm-1) and thermal conductivity (0.184 Wm-1. K) that leads to enhance ionic figure of merit 0.056 as compared to crosslinked hydrogel, making them suitable i-TE material for various applications such as energy harvesting, waste heat recovery and thermal management systems.



Ionic cellulose aerogels for industrial wastewater treatment

Álvaro Vázquez, María Hernández, Aránzazu Martínez-Gómez, Nuria García, Pilar Tiemblo.

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<u>Abstract</u>

One of the most serious threats to the environment is the dumping of industrial wastewater containing contaminants such as textile dyes or oily residues, among others. To solve this problem several strategies have been explored. The use of materials able to adsorb these wastes is an attractive idea. Cellulose is an interesting raw material for this application because it is the most abundant biopolymer in nature and can biodegrade.

In this work, ionically modified cellulose aerogels will be studied as materials for this purpose. The first step of the synthetic route is the oxidation of cellulose to give 2,3-dialdehyde cellulose (DAC). In this research, DAC products were obtained in a huge range of oxidation degrees, and they were characterized using I3C-RMN, SEM and IR. Subsequently, DAC were used to obtain the anionic modification of cellulose (ADAC) by the introduction of sulfonic moieties and the cationic modification (hPEI-DAC) with branched polyethyleneimine. Both materials were characterized using IR to elucidate their structures, microanalysis for determining their compositions and SEM to explore their morphological features.

Finally, the application of these aerogels as wastes adsorbents was studied by using castor oil as a model substance for oily waste, and some dyes such as methylene blue or rose bengal.



Optimization of the VARI process and recycling of laminates produced with Akelite resin

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Abstract

Composite materials are now a days the material of choice over traditional materials, such as metals or ceramics, for their unique combination of strength and lightness. Their market has mostly been dominated by thermoset resins, that harden permanently thanks to a crosslinking mechanism. This mechanism facilitates the processability and provides its excellent mechanical performance. However, this crosslinking also results in their main disadvantage: once processed, they cannot be transformed again or recycled. Meanwhile, thermoplastic composites are gaining traction because they solve this problem but they require a profound change in the processing methodology, due to their extremely high viscosity in the molten state. To solve this problem, we have developed a new liquid thermoplastic resin, AKELITE, patented by the CSIC group, capable of producing sustainable and 100% circular composite materials, without changing the production processes.

This presentation will explain the Vacuum-Assisted Resin Infusion (VARI) process for manufacturing high-performance laminates using Akelite resin. Key parameters influencing the quality and performance of the laminates will be discussed, along with the strategies implemented to enhance the efficiency of the process. Moreover, we will address the recyclability of the Akelite-based laminates, detailing the recycling method and its successful application to a real-world component. This dual focus on process optimization and recyclability positions Akelite as a sustainable alternative for industries reliant on polymeric composites.



Development of Sustainable Electrochemical Separators Based on Polylactic Acid Membranes for Secondary Batteries

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Abstract

Electrochemical secondary batteries are at the center of research and development due to their fundamental role in the transition towards a more sustainable economy. One of the principal challenges in this field is improving the safety and durability of electrochemical cells, as it is necessary to prevent risks such as short circuits or leaks of toxic and flammable liquids. To achieve this, separators play a key role, as long as they meet a series of chemical, mechanical, thermal, and transport properties.

In comparison to inorganic separators, polymeric ones are light and flexible, particularly those made of polyolefins. As in many other industrial fields, the reduction of the dependence on petroleum-based polymers is a very desired aim in battery technology, and consequently, separators based on other polymers are being developed, for instance cellulose-based ones¹.

In this work, separators made of polylactic acid (PLA) blends prepared making by a modified Non-Solvent Induced Phase Separation (NIPS) method, employing only green solvents, are presented. The use of PLA presents challenges derived from balancing adequate porous membrane-formation and thermal and mechanical stability. However, PLA microstructural variation is rich which can be advantageous to tune the desired properties, as explored in this research. The separators presented in this work are characterized in terms of their thermal properties (DSC, TGA), chemical structure and morphology (FTIR, SEM), mechanical properties (DMA, nanoindentation) and their chemical stability against sodium electrolytes.

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Modification of the wettability of polymers by laser irradiation with nanosecond and femtosecond pulses

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Abstract

The topography and chemical composition of the surface determine the wettability of materials. Some examples can be found in nature, such as the lotus leaf, which due to its topography and composition has superhydrophobic properties [1], inspiring structuring techniques to modify and control wettability. In the case of polymers, laser techniques can be used to modify their surfaces, such as Laser Interference Lithography, Laser Induced Periodic Surface Structures (LIPSS) or ablation [2]. It has been observed that the formation of LIPSS on polymers induces topographical changes and slight oxidation, typically reducing their contact angle [3]. To generate hydrophobic surfaces a possible alternative could be laser ablation or foaming [4]. Parameters such as repetition rate, pulse duration, irradiation wavelength, fluence and number of pulses are are essential factors that influence ablation [5]. This study investigates the wettability of different polymers with different initial wettability after laser irradiation using both femtosecond (fs) and nanosecond (ns) lasers. For the case of fs pulses the polymers have been irradiated at a wavelength of 795 nm at different fluences and overlapping, and in the case of ns three different wavelengths (266 nm, 532 nm and 1064 nm) and different numbers of pulses have been used. Subsequently, a study of how this affects their wettability has been carried out by measuring the contact angle, and it has been analyzed whether the changes are due to chemical or topographical modifications by means of X-ray Photoelectron Spectroscopy and Scanning Electron Microscopy.

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Advanced 3D Printing Techniques for fabricating Hierarchically Porous Membranes for Efficient Oil-Water Separation

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<u>Abstract</u>

Due to its revolutionizing production efficiency, Additive manufacturing (AM) enables the creation of complex geometries with intricate hollow features. By leveraging the unique capabilities of Direct Ink Writing (DIW) and Multiphase Direct Ink Writing (MDIW), we present for the first time the fabrication of hierarchical multilayer porous membranes. DIW facilitates the vertical layer-by-layer printing of thin, porous membranes (~250 μ m) that mimic natural soil systems for efficient water purification. Whereas MDIW allows the horizontal layering of membranes (~200 μ m) by matching the viscosities of two solutions: polymer/polymer composite, achieving individual lines composed of 32 sublayers. The properties and performance of these membranes will be rigorously tested for oil-water separation to assess their separation efficiency and durability. This research highlights a promising approach for scalable 3D printing of multilayer membranes, showcasing a novel AM technique to produce membranes with tailored porosity and high-performance filtration characteristics, thereby opening new possibilities for customized membrane design and production in wastewater remediation.



There is no glitch in the matrix: Different elastomeric matrices and self-healing capability

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Abstract

The intricate composition of polymeric materials, often incorporating various additives such as fillers, antidegradants, and processing oils, frequently renders many of these waste products unsuitable for secondary use. In response to this challenge, our research focuses on the development of self-healing thermoplastic elastomers (TPEs) derived from natural rubber sourced from conventional sustainable sources such as Hevea Brasiliensis, as well as alternatives like Russian Dandelion and Guayule. Furthermore, the integration of biobased fillers from natural sources, agricultural or forestry waste such as cellulose, lignin, or silk, is explored for rubber reinforcement, aiming to establish a fully sustainable system. The selection of these additives and the chemical interactions among them will be essential to confer self-healing capability to the material. The first results of our study have focused on synthetic systems resembling natural rubber, employing substances like epoxidized natural rubber (ENR) and polyisoprene (IR) with cellulose as a filler, achieving promising results regarding their healing capabilities. Additionally, the inherent thermoplastic nature of our TPEs ensures their reprocessability and recyclability. These promising results will definitely contribute to waste reduction , extending the lifetime of the rubber materials.



Reprocessing of biopolymer-based aerogels

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<u>Abstract</u>

Aerogels are a class of innovative lightweight materials with high specific surface area and open mesoporosity. Biopolymeric aerogels, those based on natural proteins and polysaccharides, have advantageous characteristics such as excellent permeability, biocompatibility, and bioactivity. These properties make them particularly attractive for biomedical and pharmaceutical applications [1,2]. Furthermore, they are increasingly viewed as promising candidates for integration into a circular economy.

In this work, the production of corn starch aerogels and their subsequent reprocessing has been carried out to yield new aerogels with satisfactory textural properties. Both the original and the reprocessed aerogels are fabricated using supercritical CO2 drying, which uses mild operating conditions, preserves the structural integrity of the materials and leaves no toxic residues. Additionally, the reprocessing methods were simple, efficient, and did not require toxic reagents/solvents or energy-intensive techniques, making them environmentally friendly procedures.

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Quinone-based hyper-crosslinked polymers as photocatalysts for valorization of glycerol

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Abstract

The high price of oil and the decline of its reserves are focusing attention on biomass, as an alternative to energy and chemical production. Biodiesel, produced from biomass, generates glycerol as a by-product. This excess of glycerol can be converted into high-value oxygenated fuel additives, such as solketal, which is in high demand due to their non-toxic, biodegradable, and renewable nature.[1] In addition, photocatalysis offers a green alternative for glycerol valorization by using solar energy to produce oxygenated bio-additives for biofuels.[2]

In this work, it is present the acetalization of glycerol by means of photocatalysis using two hyper-crosslinked polymers: one based in anthraquinone (HCP-AQ-2Ph) and the other based in anthracene (HCP-AN-2Ph). They were synthesized by knitting strategy following a Friedel-Craft-type reaction with anthracene or anthraquinone, respectively, and biphenyl as a comonomer. These new polymers were fully characterized by elemental analysis, I3C-NMR in the solid state, FT-IR spectroscopy, thermogravimetric analysis, N2 adsorption/desorption isotherms, and UV-visible spectroscopy.

The catalytic activity was evaluated in the photocatalytic acetalization of glycerol to obtain solketal. At first glance, HCP-AN-2Ph needed more time to complete the reaction than HCP-AQ-2Ph. However, subsequent runs indicated that anthracene units were oxidized, which resulted in a catalyst (HCP-AN(OX)-2Ph) just as effective as HCP-AQ-2Ph. The complete and selective conversion of glycerol into solketal was reached in 3h using 4 wt% catalyst and maintained this catalytic performance for at least 5 cycles.

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Flash Presentations



Comparative study of vulcanization systems for more sustainable materials

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Abstract

This work explores a novel approach to develop sustainable elastomers using itaconic acid as a bio-based vulcanizing agent. The focus is to achieve improved physicochemical properties without compromising environmental sustainability.

The research compares traditional vulcanization methods using sulfur and dicumyl peroxide (DCP) with the proposed itaconic acid strategy. Sulfur, the industry standard, serves as a benchmark. DCP, known for its fast cure rates, is included for comparison. Itaconic acid, a bioderived diene monomer, offers a sustainable alternative for vulcanization.

By replacing traditional curing agents with a bio-based alternative, the research contributes to the development of sustainable elastomers with reduced environmental impact. The ultimate goal of the work is to provide these elastomers with self-healing capabilities through transesterification reactions.



Comparative study of pla with microcellulose fibers obtained by melt and solution electrospinning

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Abstract

In this study fibers are developed with the goal of creating a dressing for would healing using biodegradable polymers as a base, specifically poly(lactic acid) (PLA) and microcellulose, which have been also selected for their biocompatibility and bio-based origin. Cellulose is used as a support in the PLA matrix to improve the mechanical and functional properties of the composite material. The microcellulose will act like a structural support due to its high aspect ratio and stiffness, whereas the PLA will provide good thermal properties. Additionally, a plasticizer, poly(ethylene glycol) (PEG), will be incorporated into the system for improve processability and reduce the stiffness of PLA. Two methods will be used for the production of the fibers: melt electrospinning and solution electrospinning. In both cases, microcellulose will be added in different proportions: 1, 3 and 5 wt%, and 15 wt% of PEG. The structural and thermal characterization of obtained composite materials have been conducted.



Towards a reversible self-crosslinkable propylene-anthracene copolymers with improved thermal and dielectric stability

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<u>Abstract</u>

Polypropylene (PP) is a widely used polyolefin in electrical and electronic applications due to its non-polar character, high insulating properties, and tunable flexibility. These characteristics make PP an ideal host for incorporating functional molecules to enhance the performance of energy storage devices such as batteries, capacitors, and supercapacitors. The work carried out by Chung et al. [1] have demonstrated the potential of this methodology to obtain high-performance PP. Our research group has expanded on this issue by developing PP with improved thermal stability and dielectric properties using various lab-synthesized olefins carrying pyrrole, carbazole, or fluorene polar groups [2-5].

This study focuses on the synthesis of functionalized polypropylene via metallocene catalysis, specifically through the copolymerization of propylene with an anthracene derivative with the goal of creating polymers capable of self-crosslinking reversibly through the action of light or thermically. The development of this study involves the synthesis of the anthracene-derivative co-monomer, and its co-polymerization with propylene. Then, the molecular microstructure and semicrystalline morphology of the copolymers will be characterized as well as the reversibility of crosslinking. Furthermore, thermal stability will be investigated by thermogravimetric analysis, in addition to the dielectric, mechano-dynamic and photo-physical properties.

Consequently, the general objective of this work is to explore the potential of polypropylenes functionalized with polar groups, as electroactive materials in electrical and electronic components with high-performance.



Effect of Alginate from Rugulopteryx Okamurae Seaweed on the 3D-Printing of Soy Protein Based Materials

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Abstract

Sodium alginate can be efficiently extracted from the brown seaweed Rugulopteryx okamurae (RO), which is an invasive species in the south of Spain. In the present work, different bioinks based on soy protein isolate (SPI) and RO alginate have been developed for 3D printing, which is an economical, sustainable, simple, and versatile technique that allows the design of complex structures. In those formulations, glycerol and polyvinyl alcohol are used as additives to improve processability and mechanical properties. First, the formulation was optimized employing commercial alginate, being their rheological properties correlated to their printability. The optimized formulation, including 7.5% extracted alginate, was selected for RO alginate containing bioinks, obtaining analogous results. Furthermore, the consistency of the printed structures was improved by storing at 105°C for 24 h, which increased their mechanical properties and integrity after water immersion. Consequently, the use of extracted alginate from RO emerges as a feasible ingredient for bioinks which could be better used in several fields (i.e., scaffolds, horticulture), while providing an alternative for using the waste of Rugulopteryx okamurae seaweed that accumulates on Mediterranean coasts. This has proven not to compromise the processability, quality and properties of neither the bioink not the resulting printed product.



Composite aerogels based on cellulose fibres and acid collagen for potential applications as wound dressings

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Abstract

Aerogels have emerged as promising materials for the development of wound dressings due to their high porosity and absorptive capacity. In this study, aerogels were prepared by combining vine shoot and eucalyptus cellulose (CEL) with acid collagen (COL) to investigate their mechanical properties. One of the main limitations of COL-CEL aerogels is their low water resistance. To address this issue, a coating of polylactic acid (PLA) was applied to increase the water sorption capacity. Different formulations of COL-CEL aerogels were prepared with densities ranging from 12 to 22 g/cm³. A deformation test was performed by compressing the aerogels to 50% of their original height, and their ability to recover this height was evaluated, showing recovery rates between 78% and 89.8%. The water sorption capacity of the PLA coated aerogels showed values between 500% and 2500%. Among the tested formulations, the combination of 1% COL and 0.25% CEL showed a low density of 14.9 g/cm³, the highest recovery rate and the highest sorption capacity. These results suggest that the PLA-coated COL-CEL aerogels have significant potential for applications in wound dressing development.



Bacterial cellulose and collagen bilayer systems for the controlled release of bioactive compounds

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<u>Abstract</u>

This study investigates the fabrication and characterization of bilayer systems based on bacterial cellulose, and collagen -hyaluronic acid. The systems were designed to serve both as protective wound dressings and as platforms for the controlled release of therapeutic agents, such as drugs, peptides, dendrimers or adenoviruses, which can improve the healing process.

A key focus of the research was the evaluation of the drug release capabilities, specifically the controlled release of an antioxidant dendrimer functionalized with caffeic acid moieties. Release studies demonstrated that the bilayer system provided a sustained and effective release of the dendrimer, which is critical for maximizing therapeutic efficacy in wound healing.

Rheological analyses were also conducted to assess the viscoelastic properties of the bilayers, with particular attention to the role of varying concentrations of hyaluronic acid (HA) mixed with collagen. The results revealed significant differences in the behaviour of the collagen networks depending on HA concentration. At lower HA levels, the properties of the collagen networks in water and TRIS buffer were comparable, showing no notable structural differences. However, at higher HA concentrations, the collagen network in TRIS exhibited greater stability compared to that in water, whereas the degree of hydration in TRIS may be higher, facilitating stronger intermolecular interactions (hydrogen bonds), giving rise to more structured and stable network.

These findings suggest that the concentration of HA and the solvent medium are critical factors influencing both the mechanical properties and drug release capacity of the bilayer systems.



Development of biomaterial inks based on pectin and hyaluronic acid hydrogels for 3D printing

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Abstract

Hydrogels obtained from natural polymers, polysaccharides and proteins, are widely employed as biomaterial inks in 3D extrusion (bio)printing, an additive manufacturing technique which holds significant potential in the biomedical field for applications such as drug delivery or tissue engineering among others. As most of the hydrogel-based biomaterial inks are physical gels, one of the main challenges is the optimization of their rheological properties for printing and their final mechanical stability. In this study, we propose an innovative approach based on 3D extrusion printing of chemical crosslinked hydrogels obtained from natural polymers aimed to increase mechanical and stability properties of the resulting hydrogels. To this aim, high molecular weight pectin and hyaluronic acid were chemically modified with bis(3-aminopropyl) amine (APA) and subsequently crosslinked with genipin at different molar ratios. The resulting hydrogels were fully characterized as to their viscoelastic properties, stability, and weight variation as a function of pH. Biphasic hydrogels were formulated following a procedure reported by our group and successfully manufactured through 3D extrusion printing into polymer scaffolds with high hydrolytic stability and pH response which are currently being evaluated for drug release applications.



ECO-strategies to obtain sterile aerogels

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Abstract

Advanced biomaterials are under development for grafting procedures in tissue engineering. Bioaerogels combine the advantageous properties of aerogels, such as high specific surface area, exceptional porosity, and lightweight structure, with the inherent benefits of natural polymers, including biocompatibility, bioactivity, and biodegradability [1]. However, aseptic environments and sterile materials are required to prevent post-surgical infections. Conventional sterilization techniques can cause physicochemical changes in the biomaterials and medical devices or leave cytotoxic residues [2]. Supercritical carbon dioxide (scCO2) sterilization is an alternative technique for nanoporous and other complex materials, due to the high permeability of this type of fluids along with the microbial inactivation power of CO2.

In this work, sterile starch cylinders and alginate aerogel beads were produced using scCO2 technology. The sterility efficacy of the process was evaluated using three standardized bioindicators. The obtained aerogels were evaluated regarding their texture (He pycnometry, N2 adsorption-desorption analysis, SEM) and biological performances in comparison to their non-sterile aerogel counterparts [3].

Acknowledgements

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TRSEQ

Sección

Territorial de Madrid polymers

Posters



Improving the Stability of Guar Gum-Based Hydrogels through Semi-Interpenetrating Polymer Networks for Biomedical Applications

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Abstract

Guar gum (GG) is a natural polysaccharide extracted from the seeds of Cyamopsis tetragonoloba, which has attracted considerable attention due to its promising potential in biomedical applications. The chemical structure of this polymer contributes to its substantial thickening, viscosity and mucoadhesive properties. However, GG hydrogels tend to degrade rapidly. To overcome this drawback, the formation of semi-interpenetrating polymer networks (semi-IPNs) has been proposed.

This study presents the preparation of two third-generation semi-IPNs, composed of two polymers: one synthesized from two monomers based on previous works and a novel crosslinking agent, while the second polymer used was GG. The systems differed in the degree of crosslinking in polymer I.

Additionally, we examined the stability of these GG-based semi-IPNs obtained over time and under varying temperature conditions by rheological studies, comparing the results with those of GG-only hydrogels. Strain and frequency sweeps were employed to observe changes in the elastic and viscous modulus over time, which showed a substantial decrease in both modulus for the GG hydrogel, whereas the semi-IPNs maintained their gel structure for extended periods. Thermal stability was assessed through heating-cooling tests at ambient and physiological temperatures (24 °C to 25 °C, and 36 °C to 37 °C; and vice versa) as well as across a broader range (25 to 70 °C, and vice versa).

In conclusion, our findings demonstrate that the incorporation of a synthetic polymer to form semi-IPNs with GG markedly improves the structural stability of the hydrogels, allowing them to retain their gel-like properties for several months.



Synthesis of Biodegradable, Super-Porous Polymeric Networks Based on Guar Gum for Gastroretentive Controlled Drug Release

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Abstract

The increase in antibiotic resistance exhibited by microorganisms such as Helicobacter pylori has made the optimization of antibacterial therapies very neccesary, especially those aimed at preventing their colonization of sensitive organs such as the stomach. A plethora of publications highlight amoxicillin, formulated in gastroretentive drug delivery systems (GRDDSs), as the compound of choice when trying to improve the pharmacokinetics of oral treatments.

The present work focuses on the synthesis and characterization of semi-interpenetrating polymer networks that can function as superporous hydrogels with high efficiency in drug encapsulation and optimal mechanical strength when exposed to gastric movement. The resulting semi-IPNs, in particular those displaying low ecological footprint, ease of synthesis, floating properties and mucoadhesivity, may, therefore, be use as GRDDSs in the treatment of bacterial infections with antibiotics like amoxicillin.

The obtained polymers are biodegradable and contain labile acetal groups, generated through thiol-ene click reactions (polymer A) with guar gum (polymer B, a natural polysaccharide with mucoadhesive properties). This one-pot polymerization process also involves a crosslinking agent. The resulting semi-IPNs exhibit different degradation patterns and have proven to be excellent matrices for the controlled release of drugs such as amoxicillin in the gastrointestinal tract.



Development of multilayer films based on recycled pla and caseinate reinforced with bacterial cellulose from kombucha fermentation

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Abstract

This study focused on developing three-layer films using mechanically recycled poly(lactic acid) (rPLA) for food packaging applications. The films comprised an inner layer of caseinate sodium (SC), which was plasticized with glycerol (G) to enhance compatibility with the outer rPLA layers. Additionally, the inner rPLA layer was loaded with 0.5% and 1% kombucha-derived bacterial cellulose (KC) to create antioxidant active packaging formulations. The films were characterized in terms of thermal, mechanical, and water vapor barrier properties.

Bacterial cellulose was produced from kombucha fermented in a coffee residue infusion and then incorporated into rPLA layers. These films showed good adhesion between the PLA and SC layers, attributed to hydrogen bonding interactions.

Mechanical properties show that SC-G films demonstrated greater ductility than the more fragile rPLA films. However, the addition of KC improved the thermal stability and mechanical properties of the multi-layer films. The incorporation of KC improved the thermal properties, mechanical strength, and reduced the water vapor permeability compared to single-layer SC-G films. The tri-layer system, particularly those with KC-loaded composites, exhibited strong potential as a sustainable solution for biodegradable active food packaging, aligning with the goals of a circular bioeconomy.

In conclusion, the tri-layer film system, particularly with KC reinforcement, proved to be a promising biodegradable and sustainable packaging material with antioxidant properties. This study highlights the potential of using recycled PLA and bio-based materials like kombucha cellulose in active food packaging, contributing to the advancement of circular economy practices.



Self-healing polymers as electrical insulation

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Abstract

Polymers are currently the dominant materials used in electrical insulation technology. In general, they offer much higher durability, better electrical insulation properties or higher resistance to moisture and mechanical stress compared to material structures based, for example, on cellulose. Innovative polymer materials are therefore one of the key elements for progress in electrical and power engineering, as they can increase the efficiency and reliability of electrical equipment. Reduced risk of failure and longer equipment life contribute to sustainability and reduced maintenance costs. In summary, the motivation for pursuing innovative electrical insulation materials is to improve the safety, efficiency and sustainability of electrical systems, which has a significant impact on modern society and the future of the energy sector.

However, even polymeric materials are subject to ageing during their lifetime, leading to an irreversible loss of their original electrical insulation properties. In this context, the term 'self-healing (S-H) polymers' has become increasingly common in recent years. So far, it has been demonstrated in practice that these materials are able to partially recover their mechanical properties after mechanical damage to their internal structure. This ability could be of great interest for electrical engineering applications. However, damage to a solid material by an applied electric field (e.g. electrical breakdown) involves a much more complex set of mechanisms and raises many unanswered questions compared to mechanical damage (e.g. scalpel cut). This is the main motivation for the planned dissertation, which will attempt to elucidate some of the previously undescribed mechanisms.



Characterization of High Impact Polystyrene Materials for Potential Use in Food Packaging

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Abstract

High-impact polystyrene (HIPS) is a material widely used in food packaging. An example is its use in dairy products, which are more recognized in yogurt containers thanks to their processability and permeability [1]. Currently, although other countries have authorized the use of recycled material in contact with food, in systems with higher demands, such as the one developed by the European Commission, there are still no decontamination technologies authorized for this purpose, which limits the applications to which these materials can opt after being recycled. It is key to reverse this situation by opting for a greater demand for material that benefits its use and favors the transition to a circular economy. In this context, innovation in recycling and decontamination methods is fundamental to increasing the sustainability and safety of packaging. In addition, research on the characteristics of the raw materials to be used and different formulations to be incorporated can contribute to its acceptance in the market, thus promoting the efficient use of resources and the reduction of plastic waste. This work studies the effect of reincorporating high-impact polystyrene (HIPS) reducing the use of virgin polymer for potential use in food packaging. The effect of its origin and proportion in each mixture on the mechanical, structural, and optical properties is analyzed.

