3rd International Conference on

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BOOK OF ABSTRACTS

Zoran
NOVAK

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Gabrijela
HORVAT

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Gabrijela Horvat

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AERoGELS

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Convergence of 3D Printing and Food-Grade Aerogels for the Delivery of Bioactive Compounds

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The poor chemical stability and bioavailability of bioactive compounds, such as lycopene, lutein, and curcumin, impede their potential health benefits. Aerogels (i.e., materials with nanoporous structures) have recently received great attention for the delivery of bioactive compounds due to their high surface area and porosity. Therefore, the goal of this study was to develop a novel encapsulation method using 3D food printing, to enhance the chemical stability and bioavailability of a model bioactive compound, namely, lutein. In the first part, porous starch and alginate-pectin beads were formed via an innovative 3D printing approach. The effects were investigated of starch concentration (10, 12.5, and 15%) and nozzle size (0.33, 0.25, 0.15, 0.10, and 0.08 mm) on the particle size [1,2]. Next, the 3D-printability was investigated of corn starches with different amylose contents (25, 55, and 72%). The rheological properties of the starch inks were determined before 3D printing. After the starch beads were created, they were dried using freeze-drying and supercritical carbon dioxide (SC-CO_2) drying. The smallest bead size (~650 μm) was created using the high amylose corn starch with the smallest nozzle size. SC-CO_2 drying of the beads resulted in a nanoporous structure, whereas the porous structure collapsed

upon freeze-drying. The 3D-printed beads dried with SC-CO_2 showed outstanding properties, including a surface area of 175 m^2/g, a pore size of 14 nm, and a pore volume of 0.7 cm^3/g [3]. In the second part, coaxial extrusion 3D printing was employed using lutein-loaded ethyl cellulose as the inner flow (core) material and corn starch as the outer flow (shell) material. The 3D printing parameters (i.e., layer height (0.4, 0.7, and 1 mm), ethyl cellulose (6, 8, and 10%, w/v) and starch (9, 10, 11, and 12%, w/w) concentrations, and printing temperature (55, 65, or 75 °C) were investigated and optimized for the best printability and encapsulation. The rheological properties of inks were determined prior to 3D printing. The 3D-printed shapes were investigated for their microstructure, storage stability, crystallinity, and surface chemistry. The best shape fidelity was obtained using the layer height of 0.7 mm, as determined by their microCT images. In addition, starch concentrations of 10 and 11% provided the best printability, which, in turn, resulted in the highest storage stability of lutein. Encapsulation of lutein via 3D printing increased the lutein retention indexes significantly from 24 and 10% (control crude lutein) to 70 and 48% (encapsulated via 3D printing) after 21 days of storage at 25 and 50 °C, respectively. Thus, the proposed 3D printing approach, along with SC-CO_2 drying, can provide high precision and accuracy over the particle size and composition, while preserving the porous structure and eliminating the need for organic solvents. Overall, 3D printing can be an alternative approach to generating bioactive compound delivery systems for the food and pharmaceutical industries.

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A Novel Aerogel from a Collagen-NADES Extract for Potential Topical Biomedical Applications

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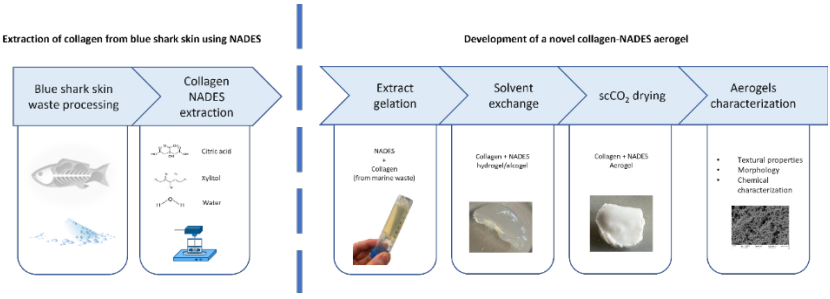

Figure 1: Graphical abstract
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Integrating natural deep eutectic solvents (NADES) extraction technologies to recover collagen from marine waste sources is an ongoing topic in our laboratory. Developing new composite biomaterials with these extracted biomolecules represents a valuable resource for an integrated and product-focused supply chain contributing to a circular economy. Besides being structural elements of the extracellular matrix of human tissues, these biomolecules have numerous properties, such as gelation capacity, biocompatibility, biodegradability, and antibacterial activity [1, 2]. Due to gelling properties, these compounds are promising candidates for developing new aerogels which have the desired properties for a broad range of biomedical applications [3]. This work aimed to develop new porous material from a collagen-NADES extract and perform solid-state characterization. To obtain the aerogels, the collagen-NADES extract was gelled under 4°C, converted into alcogel through solvent exchange, and dried with supercritical CO_2. The morphology was studied by Scanning Electron Microscopy, whereas the specific surface area and the pore volume were obtained through the Brunauer–Emmett–Teller and Barrett-Joyner-Halenda methods, respectively. The resulting aerogel was lightweight, with a sponge-like structure, a relatively dense meso- to microporous network and a specific surface area of 224 m^2/g, specific pore volume of 1.19 cm^3/g and a pore radius of 13.8 nm. The collagen`s structural integrity was studied by discontinuous electrophoresis, Fourier transformed infrared spectroscopy and circular dichroism. This work demonstrated that a novel aerogel was obtained successfully through supercritical drying of a collagen-NADES gelled extract, revealing a high specific surface area and pore volume material, and no impact of the aerogel production process on the collagen`s structure. Future works evolve to evaluate the material's mechanical behavior, biocompatibility and bioactivity, exploiting the attractive properties of aerogels, collagen and NADES for potential biomedical applications.

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Coating of Whey Protein Aerogels with Alginate, Agar, or Ethylcellulose to Enhance their Functionality in Hydrophilic and Hydrophobic Model Food Systems

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In recent years, aerogels have been attracting great attention in the food sector for the development of ingredients with innovative functionalities [1]. In fact, due to their open porosity (e.g., pores connected and accessible from the surface), aerogels can be loaded with bioactive or flavor compounds, making aerogels potential delivery systems in foods [2]. The unique structure of aerogels could also be exploited to deliver air into food, to reduce the calorie density and increase the perception of sweetness and saltiness [3]. Although open porosity is an essential feature driving aerogel functionality, it might also represent a critical issue in food applications. In fact, the penetration of moisture or reactive molecules into the pores might cause their collapse, as well as undesired modification of the aerogel structure, thus impairing their aerogel properties [4]. Moreover, upon impregnation with liquids (i.e., water and oil used in food formulations, and digestive fluids), able to solvate or solubilize the polymers they are made thereof, aerogels might undergo collapse, swelling and dispersion or solubilization, losing their functionalities [5]. In

this context, the application of an external protective layer on the aerogel surface can represent a promising strategy to protect the inner structure of the aerogels. Nevertheless, limited studies reported the successful application of a coating material on bioaerogel surfaces to prepare food-grade delivery systems [6, 7]. Besides, these papers only refer to aerogel coating with hydrophobic molecules, potentially able to control the structure of aerogels when placed in contact with water. Nevertheless, food systems might also include a relevant amount of lipid components such as oils.

Based on these considerations, the present work aims to widen the knowledge on aerogel coating, to increase the functionality of these innovative materials as food ingredients.

To this aim, whey protein aerogels (20% w/w, pH 4.8) were coated by dipping with alginate (AL, 3% w/w) or agar (AG, 3 % w/w) aqueous solutions, and ethylcellulose (EC, 10% w/w) alcoholic solution, selected as food-grade polymers presenting different polarities, hydro- and lipophilic, respectively. The coated aerogels were analysed for structural properties (SEM microstructure, volume shrinkage, firmness) and their ability to interact with food systems characterised by different compositions and increasing complexity (water vapour, water, oil, flour-water batter, coconut stearin-oil mixture) by assessing the ad/absorption capacity.

The obtained results showed how an AL coating application induced the collapse of the aerogel structure, caused by the absorption of the coating solution into the aerogel pores. By contrast, AG and EC formed homogeneous coating layers of 65 and 100 μm thickness, respectively. In these cases, the absorption of the coating solution was probably prevented by the rapid setting of the polymers onto the aerogel surface. The moisture adsorption of aerogels exposed to 100% relative humidity was affected by the presence and the nature of the coating. EC coating resulted in the lowest moisture adsorption, likely due to EC`s hydrophobicity. When samples were dipped in water and oil, the uncoated aerogel absorbed both liquids readily. In contrast, liquid absorption was delayed significantly in the AG- and EC-coated samples. In particular, the AG-coated aerogel showed an overall water and oil uptake about 40 and 60% lower than the uncoated sample. Similarly, the EC-coated aerogel showed a water uptake 30% lower than the uncoated aerogel. In contrast, the EC coating was not as effective in reducing the overall oil uptake, probably due to the solubilisation of EC in oil. The interaction of AG- and EC-

coated aerogels with more complex model food systems was evaluated based on these results. Specifically, the AG-coated aerogel was included in a lipophilic food system (i.e., a coconut stearin-sunflower oil mixture), whereas the EC-coated sample was immersed in an aqueous one (i.e., a water-flour batter). The results demonstrated the efficacy of AG and EC coating in limiting aerogel liquid absorption in both food systems.

These results indicate the potentiality of tailored coating applications to enhance aerogel functionality in food.

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Comparison of Drug Release Kinetics Between Carboxymethyl Cellulose Aerogels and Cryogels

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Porous carboxymethyl cellulose (CMC) materials as potential wound healing materials were prepared from CMC solutions, followed by solvent exchange, and then drying with supercritical CO_2 and freeze drying, leading to "aerogels" and "cryogels", respectively. The hydrophilic drug L-Ascorbic acid 2-phosphate sesquimagnesium salt hydrate (Asc-2P) was used as the model drug, as it is known to possess antioxidant activity and collagen synthesis. The drug was dissolved in a CMC solution, and release from aerogels or cryogels was studied in a simulated wound exudate medium. The goal was to compare the *in vitro* release behavior and drug loading efficiency from CMC aerogels and cryogels as a function of the drying methods and the physicochemical properties (CMC degree of substitution, density, specific surface area and morphology) of materials. Higher water absorption was obtained for the CMC cryogels (up to 2500%) as compared to aerogels (100-1700%). The cryogels showed a higher loading efficiency (up to 100%) and fast release and dissolution time in comparison with the aerogels. The drug-loaded aerogels showed controllable and sustained release of Asc-2P. The behavior of drug release is dependent on the CMC degree of substitution and density in the case of aerogels. In contrast, for cryogels, the degree of substitution and density didn't influence the release kinetics noticeably. The sustained release from the aerogels based on CMC

of low degree of substitution (0.7) was able to reach 48 h in a simulated wound exudate medium.

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Advanced Polysaccharides-based Materials from Wastes as Carriers of Active Molecules for Biomedical Applications

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Polysaccharide-based materials have attracted the attention of the Biomedical industry for the production of drug delivery systems, as, by definition, they are biodegradable and biocompatible natural polymers [1]. In this work, hydrogels for potential biomedical applications were produced from structurally modified starch. Agri-food wastes were used as feedstock of starch, contributing to the "Circular Economy" practice [2]. Two sources of starch with a different amylose/amylopectin ratio were oxidized with sodium periodate ($NaIO_4$) in water and mild conditions, generating aldehyde derivatives involved in the Schiff bases reaction [3], as well as, in the crosslink process, able to generate after a freezing and thawing procedure a stable three-dimensional hydrogel network. All the materials were characterized

deeply by means of several techniques, evidencing a high swelling degree; a homogeneous fibre arrangement, and an interesting uploading/releasing drug profile. The hydrogels were "loaded" with selected active molecules, specifically, peppermint essential oils, diacetyl and methylglyoxal, known for their common beneficial functions, such as antiviral, antimicrobial, anti-inflammatory and antioxidant effects [4], following two approaches: *i)* By the absorption of selected molecules; *ii)* By chemical grafting. The drug-carrier capability and the drug release profiles were obtained through qNMR spectroscopy [5]. The antibacterial activity of the loaded hydrogels was then proven against *Escherichia coli* and *Staphylococcus aureus* strains, evidencing good results, (Figure 1). Furthermore, the cytotoxicity was evaluated by performing LDH and MTS assays on CAL27 cell lines, and the results of the loaded hydrogels were surprising, because they evidenced a sort of "frozen proliferative state" for the growing of cell lines, probably correlated to the effects of the completely swollen hydrogels into the cell medium after 24 h of incubation at 37° C. Finally, Flow Cytometry experiments demonstrated that the produced starch hydrogels induced less "early apoptosis" and more "late apoptosis" in cells, compared to the untreated control.

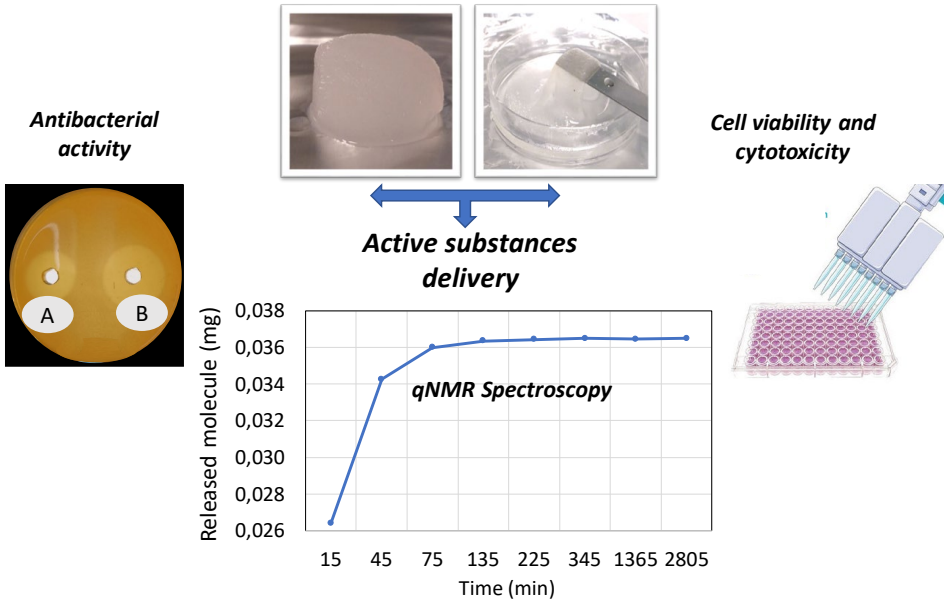

Figure 1: Starch hydrogels as carriers of antibacterial and antiproliferative molecules

Source: own.

In conclusion, the obtained results make the synthesized hydrogels as promising drug delivery systems for biomedical applications, thus contributing to the current challenges in the search of new formulations with even more increasing therapeutic effects on cell migration and tissue regeneration.

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Biopolymer-derived Carbon Aerogels as Catalyst Support for Hydrogen Evolution Reaction

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Hydrogen plays a key role in mastering the energy transition and, thus, contributing to the environmental goals of the EU. In Power-to-X concepts, renewable energies are utilized to generate green hydrogen by the electrolysis of water. PEM electrolyzers typically work with Platinum (Pt) as catalysts on carbon supports for the hydrogen evolution reaction (HER). In order to achieve high activities with Pt/C catalysts, tailoring the properties of both, the Pt-nanoparticles, as well as carbon support is necessary. In particular: 1) The Pt-nanoparticles` size, distribution, loading and shape need to be controlled, whereas small nanoparticles are beneficial, in order to increase the surface to volume ratio [1]. 2) The textural properties of the carbon support should provide sufficient internal surface area and allow good mass transport inside the carbon matrix [2]. Carbon aerogels with significant micro- and mesopore content are promising materials in this regard, since they combine the beneficial properties of both pore-size regimes.

The main objective of the project is to boost the development of highly active and stable HER electrocatalysts with low Pt-loadings, in an attempt to reduce the cost of water electrolyzer systems. To achieve this goal, it is important that Pt is distributed evenly in the carbon carrier, with optimal metal nanoparticle sizes of just a few nm, avoiding further aggregation. For this purpose, highly porous carbon aerogels (CAs) will be derived from biopolymer aerogels obtained from sustainable sources. These will, further, be loaded with Pt by the use of innovative supercritical deposition technologies, and tested according to their HER performance. Evaluation of the production parameters shall enable rational design of highly active and stable CA-based, low Pt-content HER electrocatalysts.

During a Short-Term Scientific Mission (STSM) from 26/03/2022 to 09/04/2022 at Koç University, by the group of Prof. Erkey, it was demonstrated that a novel electrocatalyst material (Figure 1) for application in green hydrogen generation starting from a biopolymer-based aerogel, could outperform a commercial fossil-based Pt-C electrocatalyst in Linear-Sweep-Voltammetry tests as the first benchmark test. This finding is in-line with the goals of WG2 (Environmental Applications of Aerogels) and WG3 (Materials` engineering and characterization) of the AERoGELS COST Action.

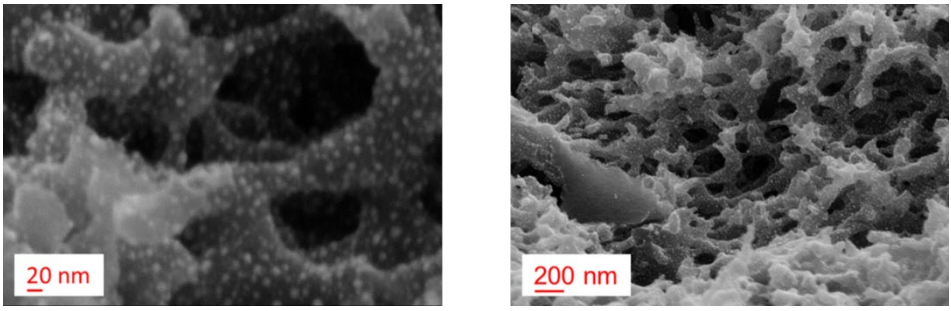

Figure 1: SEM images of Pt nanoparticles (3 - 12 nm) deposited homogeneously on carbon aerogels derived from cellulose aerogels.

Source: own.

The work resulted in a successful follow-up proposal, and is ongoing in the IraSME project "CarboCAT - Nanostructured and Platinum Loaded Carbon Aerogels for Hydrogen Economy", a joint project including the participation of working groups of the TUHH and Koç University. The scope of this project includes the production

of similarly produced Pt-C catalysts, starting from cellulose aerogels, with further variation of the different postprocessing steps (pyrolysis, plasma-doping/etching, Pt-deposition) up to the manufacture and testing of the material in Membrane-Electrode-Assemblies in PEM Electrolysers of the W to kW range.

Acknowledgements

This publication is based upon work from COST Action "Advanced Engineering of aeroGels for Environment and Life Sciences" (AERoGELS, ref. CA18125), supported by COST (European Cooperation in Science and Technology).

The related international project "CarboCAT" is funded by ZIM (Central Innovation Programme for Small and Medium-sized Enterprises (SMEs)) and TÜBİTAK (The Technological and Scientific Research Council of Turkey) as an IraSME project (International Research Activites by Small and Medium-sized Enterprises).

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Galactomannan-based Aerogel Microparticles for Potential Pulmonary Delivery

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Bio-aerogels, namely, polysaccharide-based aerogels, are particularly promising for biomedical applications. The biocompatibility and biodegradability of polysaccharides, combined with the high porosity and very low-density characteristics of aerogels, have been reported to be advantageous for pulmonary delivery [1]. Galactomannans are polysaccharides composed of a backbone of mannose (M) with single galactose (G) side chains, with applicability in biomedicine [2].

Locust bean gum (LBG)-based aerogel microparticles were developed in this work. Commercial LBG (M:G ratio »4:1) was oxidized enzymatically using galactose oxidase, horseradish peroxidase, and catalase, to be able to form crosslinks [3]. The oxidized galactomannan solution was used to form microparticles that were dried supercritically to obtain the respective aerogels. The size and morphology of the aerogel microparticles were assessed using Scanning Electron Microscopy, and their density using helium pycnometry.

The oxidation of the galactomannans was confirmed by observation of the formation of a weak hydrogel after freezing, which does not occur for unoxidized galactomannans. The developed aerogel microparticles had geometric diameters over 5 μm and spherical shape, with an aerodynamic diameter expected to be between 1-5 μm. These characteristics indicate that the galactomannan-based aerogel microparticles will be able to reach the lower airways and avoid macrophage clearance, thus having the potential to be used for pulmonary delivery.

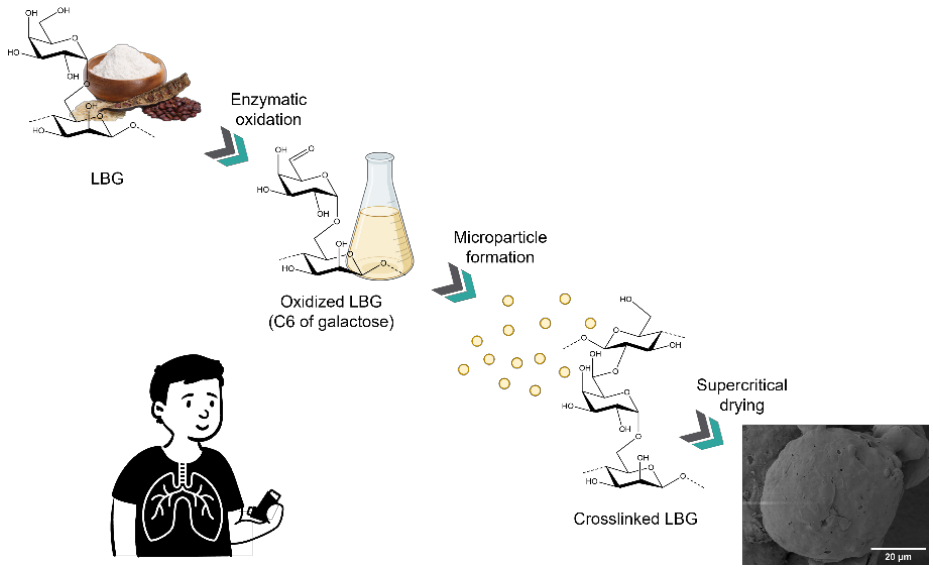

Figure 1: Scheme of the preparation of galactomannan-based aerogels microparticles.
Source: own.

Acknowledgements

This work was supported and carried out in the framework of COST Action CA18125 "Advanced Engineering and Research of aeroGels for Environment and Life Sciences" (AERoGELS, https://www.cost.eu/actions/CA18125), funded by the European Commission. Thanks are due to the University of Aveiro and FCT/MCT for the financial support to the LAQV-REQUIMTE research units (FCT UIDB/50006/2020) and CICECO - Aveiro Institute of Materials, UIDB/50011/2020 & UIDP/50011/2020 through national funds, and, where applicable, co-financed by the FEDER, within the PT2020 Partnership Agreement. Cláudia Passos`s contract (CEECIND/01873/2017) and Sara Valente7s PhD grant (DFA/BD/5430/2020) were supported by FCT. The research contract of Ricardo Pinto was funded by national funds (OE), through FCT in the scope of the framework contract foreseen in the numbers 4, 5, and 6 of the Article 23, of the Decree-Law 57/2016, of 29 August, changed by Law 57/2017, of 19 July.

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Biological Thermal Performance of Aerogel Patches for Light-based Therapies

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Cancer is a major worldwide concern with a high economic and social burden. It is accountable for the death of one in six people, which encourages the search for novel therapeutic options. Over recent years light-based therapies have been explored, as an attempt to surpass the main limitations associated with conventional therapies. Promising results for photodynamic and photothermal therapies have been obtained, but several challenges still persist, like hindering of the light absorption by tissues, and some undesirable effects, such as skin burns. Radiation within the optical therapeutic window (600 nm < wavelength < 1200 nm) has been explored widely, to enhance the penetration capability of the beam, as it is absorbed less and scattered by the tissues. Moreover, frame-like structures based on a light and thermal insulator material can be used to delimitate the irradiated area, as well as to avoid undesirable burns. Aerogels are a class of materials fulfilling the main requirements for the aforementioned application; they are known to be lightweight, porous, thermal and light insulators, biocompatible, and biodegradable [1]. Among the vast library of aerogel materials, silica and pectin-based aerogels are recognized as the best thermal insulators made of inorganic and biological organic materials, respectively [2,3]. In accordance with these data, the thermal performances of silica-based aerogels reinforced with cotton fibers and pectin-based aerogels were assessed, in combination with irradiation with a near infrared laser, for their potential use as light delimiters for light-based therapies [4]. Silica and pectin-based structures were prepared and characterized physico-chemically in terms of specific surface area, pore volume and pore diameter, by N_2 adsorption-desorption analysis. Later, the thermal performance of aerogels in combination with NIR irradiation was assessed in three experimental models: agar phantoms, *ex vivo* pork skin, and *ex vivo* human skin. Finally, the skin compatibility of both aerogels was evaluated in human volunteers. Both aerogels showed promising textural properties, with silica-based structures presenting more auspicious features. Moreover, silica-based aerogels conferred enhanced thermal protection in the two *ex vivo* skin graft models tested. Finally, after proving to be safe *ex vivo* in both pork and human skins, both aerogel structures exhibited excellent skin biocompatibility in human volunteers. Overall, herein the high potential is reinforced of silica-based aerogels as non-ionizing radiation delimiters and thermal insulators in light-based therapies.

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Thermal Superinsulating Pectin Aerogels

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Around thirty-five percent of the total greenhouse gases worldwide are emitted because of insufficient thermal insulation in buildings. Aerogels, apart from vacuum insulation panels, are the only "intrinsically" thermal superinsulating materials with thermal conductivity far below that of air at ambient conditions (< 0.025 $W \cdot m^{-1}K^{-1}$), due mainly to their mesoporous structure and low density. A low carbon footprint and sustainability are important factors to consider, along with high performance. Among the "bio-aerogel family", aerogels based on pectin, which is a natural polysaccharide, are very promising new materials for thermal insulation applications with very low thermal conductivity (0.015 – 0.020 $W \cdot m^{-1}K^{-1}$) [1].

The different process parameters involved in the synthesis of pectin aerogels have a strong influence on the aerogels' morphology as well as properties, and, thus, the thermal conductivity of the materials [2]. In this work, highly porous and nanostructured pectin aerogels were prepared via dissolution in water, followed by a non-solvent induced phase separation and a final drying with supercritical CO_2. Pectins of different degrees of esterification (DS) were used, one high-methylated (DS > 50%) and one low-methylated (DS < 50%). Ethanol or acetone were

investigated as non-solvents, and other parameters were varied, such as the pectin solution pH value and the concentration of the polymer. The influence was studied of pectin characteristics and processing parameters on aerogel morphology, density, specific surface area and the thermal conductivity of aerogels. All the corresponding aerogels were thermal superinsulating materials with thermal conductivity in the range of 0.015 – 0.023 $W \cdot m^{-1}K^{-1}$.

As a next step, pectin hydrophobization was performed, to prevent material aging. Hydrophobization of pectin aerogels resulted in water contact angles in the range of 117 – 130°. The affect of hydrophobization on the thermal conductivity was evaluated, and the evolution of aerogel properties in time as a function of humidity are in progress.

Acknowledgements

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SANS Measurements on Different Vinyl Substituted Silica Xerogels

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The main purpose of this STSM was to learn about advanced characterization techniques and new methodologies as a complementary characterization method for silica-based aerogels. The work carried out within this mission includes the synthesis of vinyl-modified silica xerogels as a potential adsorbent for the removal of oil/organic solvents from aqueous media, and morphological characterizations of these materials via SEM and SANS techniques. The Host Institution operates a SANS (small angle neutron scattering) large-scale facility, coordinated by the Budapest Neutron Centre (BNC). SANS is a beneficial technique to provide complementary information about the nano structure and structural modifications in addition to basic pore characterization techniques, such as N_2 adsorption.

During the synthesis, vinyl-modified xerogels were prepared in two series. In the first series (abbreviated as VM) xerogels were synthesized by using VTMS and MTMS as silica precursors, whereas VTMS and TEOS were utilized as silica precursors in the second one (VT). In both series, 5 samples with gradual vinyl substitution (0%, 25%, 50%, 75%, and 100% VTMS by vol.) were synthesized via the sol-gel method and dried under ambient pressure. Structural modifications induced by the vinyl content were determined both via SEM and SANS analyses and N_2 sorption.

Under the supervision of Dr. Zoltán Dudás and Dr. Adél Len, SANS measurements were performed at the Yellow Submarine instrument. The powder samples were poured in 2mm thick quartz cuvettes and measured at the whole available Q (scattering vector) range: 0.003 - 0.3 $Å^{-1}$at 3 different set-ups: 1. Sample to detector distance: 1.16 m, wavelength: 4.95 Å, 2. Sample to detector distance: 5.26 m, wavelength: 4.95 Å, 3. Sample to detector distance: 5.26 m, wavelength: 9.7 Å. The transmission of the samples, as well as the calibration measurements were also performed (detector cell sensitivity, background noise, quartz cell scattering). The obtained data were analyzed by Beaucage and Power-Law model approximations. For each series, scattering intensity I(Q) vs. scattering vector (Q) graphs were drawn, as displayed in the following Figure.

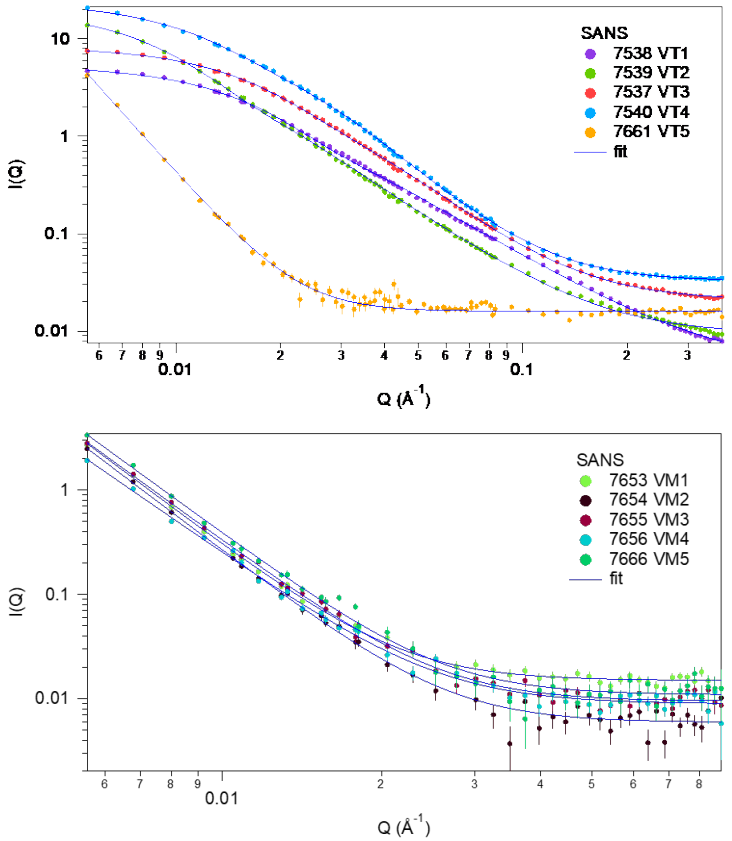

Figure 1: SANS curves of the series VT and VM

Source: own.

In the VM series, the gradual increase of the vinyl content exhibited very limited effects on the silica backbone modification, probably due to the similar steric effects of the selected organosilanes. In the VT series, on the other hand, in accordance with the results obtained in N_2 porosimetry and SEM analysis, SANS measurements confirmed the variation in nanostructure, depending on the vinyl functionalization. In the VT series, the increasing quantity of the vinyl functionalization caused an increase in the scattering objects` sizes and achieved a maximum for VT-50. However, in the N_2 sorption, the obtained pore sizes within the series did not change drastically with the vinyl substitution. Hence, the increased measured size in SANS can be explained by the distinct agglomeration of small primary silica particles, resulting in average cluster sizes which varied between 26-51 nm in this series. The obtained power-law exponents (p) for the VT series varied between 2-3, showing a characteristic volume fractal-like structure, whereas, for the VM series the p values were close to 4 in all content, indicating the smoother surfaces, as also proven by the SEM micrographs.

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Artificial Bone Substitution with Bioactive Silica-calcium Phosphate Composite Aerogels

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Artificial bone substitution is a constant and still growing task in all areas of orthopedic, dental and maxillofacial surgery, due to the limited availability of natural

resources. Autologous tissues are frequently unavailable in the necessary quantity or quality. In the clinical practice, several types of artificial materials are used routinely or tested experimentally. Most recently, resorbable materials are favored to regenerate the original bone structure.

Orthosilicate ions promote the formation of Type I collagen in the bone healing process. By combining the bioactive material beta-tricalcium phosphate (TCP) or hydroxyapatite (HA) with silica aerogel, we have prepared new resorbable bioactive composite materials, which proved to be bioactive and may find an application in dental, maxillofacial or orthopedic surgery in the future. [1–3]

In the presentation, we show the special technique used for the synthesis, as well as the physical, structural, and biological testing of the new silica aerogel-based bone substitute materials.

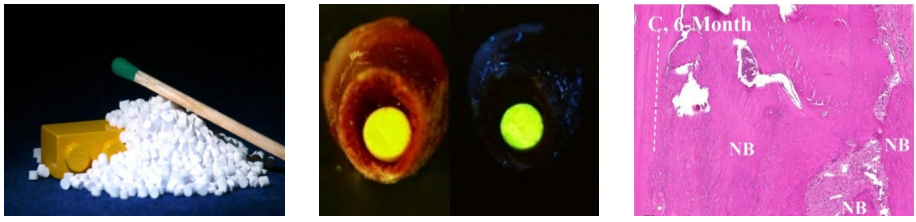

Figure 1: Left: Heat-treated cylindrical form of the silica-TCP composite; Center: FITC-labeled aerogel particles implanted in a bone model under visible and 365 nm UV light; Right: Extensive new bone (NB) formation and complete resorption in a rat calvaria critical size experiment 6 months afer implantation.

Source: own.

A summary will be presented of the laboratory and small-scale synthesis in batch and continuous mode, as well as the techniques of post-drying pore generation and scaffold-making of the material. In vitro experiments with cell lines SAOS-2 and MG63 proved cell adhesion, proliferation, bioactivity, and the advantageous effect of the combination of silica aerogel with the TCP composite. Small animal studies will be discussed that proved the resorbable nature and bone healing potential of the new materials, as well as the details of bone regeneration properties.

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Design and Evaluation of UCNPs-loaded Aerogels for Bioimaging Applications

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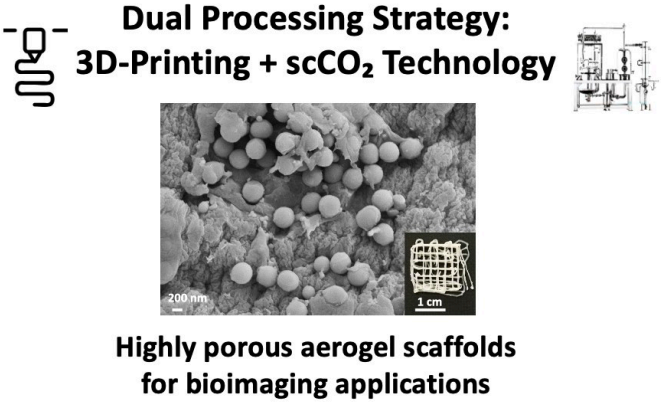

Figure 1

Aerogels are very attractive structures for the biomedical field because of their outstanding physicochemical characteristics like high porosity and surface area, low density and tunable mechanical properties and composition [1]. Nevertheless, tracking them and their degradation products through an *in vivo* imaging technique after administration still represents an important challenge today. In this context, upconversion nanoparticles (UCNPs), represent an efficient alternative as bioimaging agents, to monitor aerogel-based medical devices after implantation in a safe and non-invasive way. UCNPs are regarded as the "new generation fluorophores", because they present unique bioimaging properties like excellent detection sensitivity, deep penetration into biological tissues, physicochemical stability, and low toxicity [2]. As an example, lanthanide doped UCNPs are well-known fluorescent bioimaging labels, with great potential for *in vitro* and *in vivo* biodetection applications [3].

In this work, UCNPs were synthesized and characterized regarding their upconversion spectra, chemical structure (XRD) and morphological properties (TEM). The UCNPs were then incorporated into 3D-printed aerogels obtained by a dual processing strategy [4], and evaluated regarding biomedical applications by biocompatibility (cell studies), textural properties (BET and SEM) and fluorescence (confocal microscopy). As future work, in vivo evaluation must be carried out, to assess the possibility of monitoring aerogel-based structures after implantation. Overall, UCNPs are promising labels to localize aerogels and their degradation products *in vitro* and *in vivo* by a non-invasive imaging method in a safe and cost-effective procedure.

Acknowledgements

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Fabrication of Antibacterial, Osteo-Inductor 3D Printed Aerogel-Based Scaffolds by Incorporation of Drug Laden Hollow Mesoporous Silica Microparticles into the Self-Assembled Silk Fibroin Biopolymer

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In the current study, three-dimensional (3D)-printed aerogel-based scaffolds with hierarchical porosities were developed by incorporating drug-loaded hollow mesoporous silica microcapsules (HMSC) into the self-assembled silk fibroin (SF) biopolymer. The biomimetic aerogels were studied as potential bone-tissue engineering (BTE) material for their in vitro anti-infectivity, osteoconductivity, and

osteoinductivity properties, confirming their great potential as an ideal scaffold for bone healing applications [1].

The study includes synthesis and research of the photo-cross-linkable methacrylated silk fibroin (SF-MA) biopolymer. Such gel can act as an ink, suitable for printing via direct extrusion to the complex 3D macroscopic shapes, followed by its subsequent transition into the porous aerogel scaffolds by the freeze-casting and freeze-drying methods (cf. Fig. 1). Here, while the printed scaffolds possess controllable hierarchically organized pore sizes with a high extent of porosity and degradability [2], the photo-crosslinking of the printed gel brings about mechanical stiffness into the final scaffolds. Moreover, photo-crosslinking contributes to the covalent bonding of the dispersed methacrylated hollow mesoporous silica microcapsules (HMSC-MA) as a drug carrier to the SFMA gel network, which allows it to maintain a sustained ciprofloxacin release, which renders final scaffolds with antibacterial properties.

Importantly, HMSC incorporated scaffolds promote cellular ingrowth and proliferation and osteoblastic differentiation by inducing the expression of osteogenic markers and matrix mineralization (cf. Fig. 2)

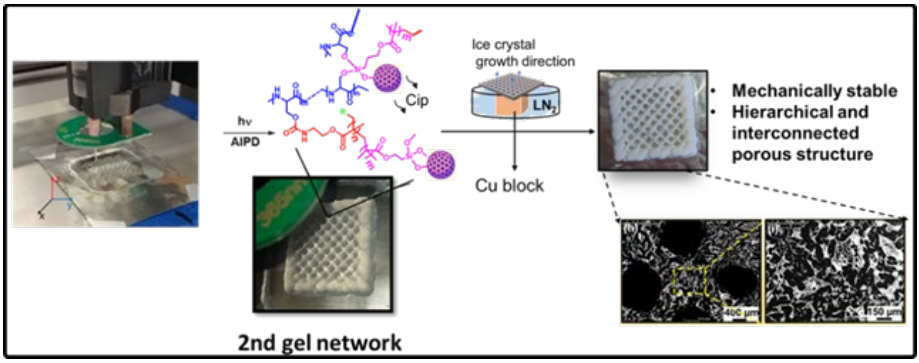

Figure 1: Printing and photo-crosslinking of hydrogel constructs using micro-extrusion-based 3D printingfollowed by unidirectional freeze-casting and freeze-drying.
Source: own.

Finally, the osteoconductive, -inductive, and anti-infective composite aerogels are expected to act as an excellent bone implanting material, with an extra feature of local and sustained release of a drug for efficient therapy of bone-related diseases.

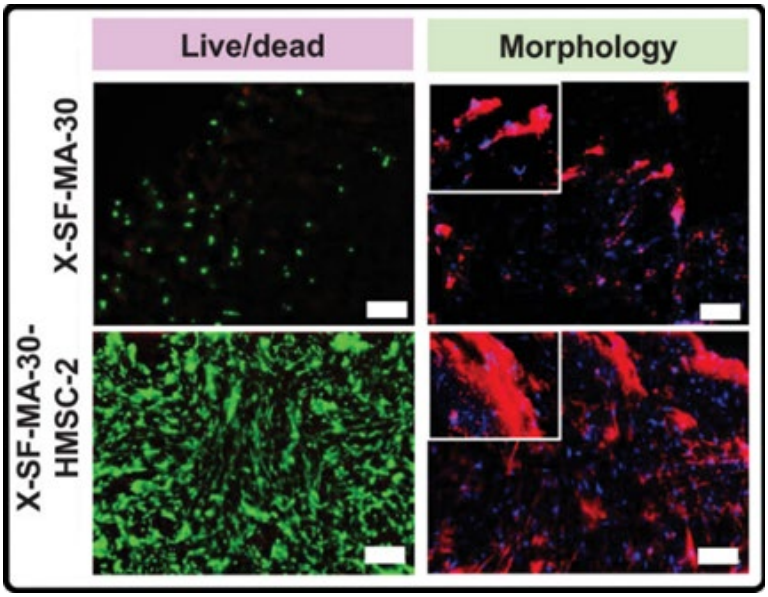

Figure 2: Fluorescence images of Human Mesenchymal Stem Cells after 21 d in culture under BASAL conditions.
Left panel: Live cells are stained by calcein-AM (green) and dead cells by propidium iodide (red). Scale bars: 200 µm.
Right panel: DNA is stained by DAPI (blue), and F-actin filaments are stained by Phalloidin (red). Scale bars: 100 µm.

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DAY 2

AERoGELS
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The IUPAC-Aerogels Connection: Aerogels as one of the IUPAC Top Ten Emerging Technologies in Chemistry in 2022

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The International Union of Pure and Applied Chemistry (IUPAC) is known to all chemists as a worldwide authority established over a century ago "to unite a fragmented, global chemistry community for the advancement of the chemical sciences via collaboration and the free exchange of scientific information" [1].

The IUPAC activities encompass all the areas of the global world of chemistry, providing (i) The guidelines for the common language by unifying the chemical nomenclature and terminology, including new elements, (ii) The guidelines on standardized measurements, processes and procedures to obtain and evaluate chemical data with the "IUPAC" label, (iii) The objective scientific expertise for all the aspects of chemistry and their societal impact, (iv) The support of projects on any topic in chemistry requiring a global view, (v) The floor for critical scientific discussions, and (vi) Many others.

In 2018, the IUPAC newspaper Chemistry International proposed to identify the Top ten emerging chemistry technologies to inform about new discoveries and exciting contributions of chemistry to the society. This selection started in 2019. In 2022 the aerogels received this prestigious recognition, which gives to aerogels a well-deserved and high-reputation position among other topics in the world of chemistry.

This contribution will inform about the IUPAC in general and its role for society, about the Top ten emerging chemistry technologies as one of the tools to demonstrate how chemistry is useful for solving the pressing societal issues, and about the activities of the IUPAC Polymer Division [2].

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[1] www.iupac.org
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The Feasibility of Cellulose Porous Materials as Oleogel-templates for the Development of Fat-replacers

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Cellulose, the most abundant biopolymer on Earth, is naturally present, or added as an ingredient, in many food products. Consumers increasingly appreciate this component, due to its plant origin and gut-protecting role. Interestingly, cellulose can be obtained from agro-industrial vegetable side streams, in a closed loop avoiding the generation of large quantities of waste, thus promoting the circular economy [1].

It has recently been demonstrated that food-grade aerogels and cryogels, with an interesting capacity to absorb large quantities of oil, can be produced by freeze-drying or CO2-critical drying [2]. This oil entrapping ability could be exploited to structure liquid oils, converting them into materials, called oleogels [3], which are particularly interesting as a saturated fats-substitute in food formulation [4]. To date, to our knowledge, no literature on cellulose-based oleogels is available.

The aim of this study was to understand the feasibility of cellulose porous materials being used as an oleogel-structuring template. For this purpose, cellulose was first ground before freeze-drying, to obtain a porous powder. The latter was characterized by SEM, density, specific surface area, particle dimensions, and porosity.

Upon progressive oil addition, the powder was able to absorb 2.4 - 2.6 goil/gdm of sunflower oil, which was effectively retained, leading to a pseudoplastic material with an oil holding capacity > 99%. These systems can be regarded as oleogels, being weak gels with G' higher than G", and showing rheological parameters in the range of that of commercial plastic fats [5]. It can be hypothesized that the oil absorption into the porous pore particles allowed the formation of a network among cellulose particles via capillary forces.

This study demonstrates the feasibility of using cellulose to structure liquid oils into oleogels, with interesting possible applicability in different food categories as fat replacers.

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Mesoporous Silica Aerogels for Sunflower Oil Refining and Investigation of their Adsorption Performance

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Silica aerogels are composed of cross-linked silica chains (SiO_2) and a high number of air-filled pores within a size range of 5-100 nm [1,2]. Silica aerogels are effective adsorbents for various materials, and have also been shown to be effective for the adsorption of various heavy metals from water [3]. Refining processes such as degumming, deacidification, neutralization, decolorization and deodorization are generally applied in edible oil production in the food industry. Impurities such as fatty acids, phospholipids, heavy metals, peroxides, etc. are removed at these stages: In sticky substance removal; phospholipids and trace metals, in the neutralization stage; free fatty acids, phospholipids, in decolorization; color substances, soap phase, in deodorization; impurities such as aldehydes, ketones, free fatty acids (FFA), polyaromatic hydrocarbons (PAH) are removed that give bad odor to the oil [4]. Bleaching soils and silica aerogel+bleaching soils combinations are used in the

refining process and decolorization stage in the oil industry [5]. The refining process is necessary to enhance quality, stability, and the shelf life of crude oils.

In this study, a sodium silicate solution was obtained from rice husk ash (RHA), and mesoporous silica aerogel (MSA) was synthesized by the sol-gel method, to be evaluated in the oil refining process of sunflower oil. The chemical structure and surface properties of the aerogel were determined by Scanning Electron Microscopy (SEM)/Energy Dispersive X-ray (EDX), Fourier Transform Infrared Spectroscopy (FTIR), Surface Area and Porosity Analyzer (BET). The efficiency of the adsorption performance of MSA was determined by the refining of sunflower oil, and compared with commercial aerogel, bleaching soil (Bentonit) and commercial silica. Adsorption studies were carried out of crude and neutralized sunflower oil with 1-3 wt.% MSA, bentonite, commercial silica, bentonite+MSA, bentonite+commercial silica combinations at 90°C for 30 minutes. The best result in acid removal among all the adsorbents was found when 1-3 wt.% MSA were used. The outcomes showed that FFA removal (above 30%) using MSA at 90°C with a 3 wt.% ratio was highly successful. While it was seen that commercial silica gave the best result in peroxide removal, it was observed that the phosphorus in the crude oil was removed completely by MSA and commercial silica. In the bleaching process, 1 wt.% bentonite + a commercial aerogel mixture gave the most effective results. Overall, the study suggests that MSA synthesized from RHA has a high potential as an adsorbent in food refining processes, due to its high adsorption capacity.

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Vinyl Modified Aerogel-Like Materials as Efficient Sorbents for Oil/Organic Solvent Removal from Aqueous Media

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Accidental oil and organic solvent spills possess accumulative and long-term hazardous impacts, and pose a serious threat to marine life [1]. For that reason, developing efficient strategies to remove petroleum-based products and solvents from seawater is a crucial issue for the sake of the aquatic environment. The adsorption method is usually considered as a reliable technique to address problems of marine oil spills. Among many alternatives, having special wetting properties, silica-based porous sorbents such as aerogels/xerogels, aerogel-like sponges and their hybrids, are considered highly promising materials for this purpose [2]. Especially, organic-inorganic hybrid silica materials, occupy a significant interest in the literature, owing to their relatively high sorption capacity, high selectivity, and recyclability. For inorganic-organic hybrid silica systems, oil sorption studies were usually conducted by utilizing methyl-based silane (methyltrimethoxy/ethoxysilane (MTMS/MTES)) derived materials and their combination with classical tetraalkoxysilanes (tetraethyl/methylorthosilicate (TEOS/TMOS)) [3,4]. On the

other hand, only a limited number of studies reporting the adsorptive performances of vinyltrimethoxy/ethoxy silane (VTMS/VTES) - based silica hybrids are available in the current literature [4].

The present work aims to synthesize lightweight silica-based aerogel-like porous monoliths with reduced shrinkage via the sol-gel method under ambient conditions. Vinyl and methyl functionalized silica materials were prepared by using VTMS as a silica precursor in combination with MTMS (VM series) and TEOS (VT series) in different series. In both series, the wet gels were silylated by trimethylchlorosilane (TMCS), to increase the degree of methylation, and vinyl content was investigated as a sol-gel parameter. The synergistic impacts of these silanes on the underlying silica network, especially on the pore structure, chemical and surface properties, are revealed by detailed analyses such as ^{29} Si-MAS NMR, SANS and SAXS, along with the conventional characterization techniques such as FTIR, SEM and N_2 sorption. Regardless of the applied precursor combination, all samples have exhibited a high degree of polycondensation of the silanes and successful functionalization of the silica surfaces with vinyl and methyl radical groups. In the VM series, the vinyl content had limited effects on the network formation, as both precursors used in this system were functionalizing silanes that possess similar steric impacts on the material's microstructure. In the VT series, on the other hand, with increasing the vinyl content, a transition was observed in the material's texture from micro/meso porous to a dominantly macroporous structure. All the samples in the VM series exhibited a sponge-like monolithic form, whereas, in the VT series, only the samples with high vinyl content preserved their structural integrity. The obtained densities (0.072-0.131 g/cm^3) and porosities (90.8-94.9 %) were similar in each series. Owing to their vinyl and methyl content, all the samples showed a high degree of hydrophobicity, with Contact Angle values ranging from 133 to 147. An easy-to- handle monolithic form with mechanical durability and intrinsic hydrophobicity, seems to make these materials perfect candidates to be employed as sorbents for the quick removal of oil/organic solvents from water. Therefore, a batch sorption study was presented, to determine the sorption capacities of the produced materials for the removal of different types of organic solvents or oil pollutants. It was seen that, in different series, VM-50 and VT-100 can separate oil or organic solvents from water selectively, and the oil adsorption capacity of the samples can reach 11.63 g/g and 10.92 g/g for VM-50 and VT-100, respectively. They also exhibited an enduring sorption property for organic solvents

after nine cycles, which can prove their possibility to be used as separation media in oil spill clean-up practices in the future.

Acknowledgements

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Aerogel-like Cellulose Monoliths and Beads Without Supercritical- or Freeze-drying

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Bio-based aerogels (here, based on polysaccharides) are nanostructured materials with low density and high specific surface area of several hundreds of m^2/g. To obtain such properties, the solvent from the aerogel precursor is usually extracted under supercritical conditions, which is the best way to preserve the network`s morphology. Another drying option, which avoids capillary pressure, is freeze-drying; however, the growth of ice crystals usually deforms the polymer network strongly, leading to materials with large pores and low surface area [1, 2]. Both drying options mentioned above involve high- or low-pressure technology; the development of alternative drying pathways, still preserving bio-aerogel precursor morphology, are, thus, of great interest.

We prepared cellulose aerogel-like materials via cellulose dissolution and low-vacuum drying from ethanol [3]. The density of the materials was around 0.1 – 0.2 g/cm^3 and specific surface area 200 – 300 m^2/g, which is very similar to the characteristics of cellulose II aerogels obtained via drying with supercritical CO_2. The influence of the processing conditions on material morphology and properties will be discussed, together with open questions.

Acknowledgements

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Optimization and Characterization of Reinforced Biodegradable Cellulose-based Aerogels via PLA/PHB Coating for Agronomic Applications

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The research interest in cellulose-based foams and aerogels is recent, but growing rapidly. The combination of performance, tunable porous architecture and sustainability makes them of interest for a wide range of applications, including, e.g., biomedical scaffolds, thermal insulation and agronomic applications, due to their biocompatibility, biodegradability and composability [1], [2].

In this work, vine shoots (VS) and waste eucalyptus paperboard (IT) were used as cellulose sources for developing cellulose-based porous materials. Two different cellulosic concentrations (0.5% and 2% w/v) and freezing temperatures (-20 ºC and -80 ºC) were tested, in order to evaluate the differences in the porosity of the materials via BET and thermal conductivity analyses. In addition, a supplementary

coating was applied to the raw aerogels by means of adding either PLA or PHB solutions (1% w/v) to their matrix, in order to improve their inherent fragile performance derived from the freeze-dried cellulose.

Their microstructure was observed via SEM (Figure 1), and their reinforcing capacity was measured by means of mechanical compressive tests (~10-fold improvement) and water resistance (Contact Angle >100º).

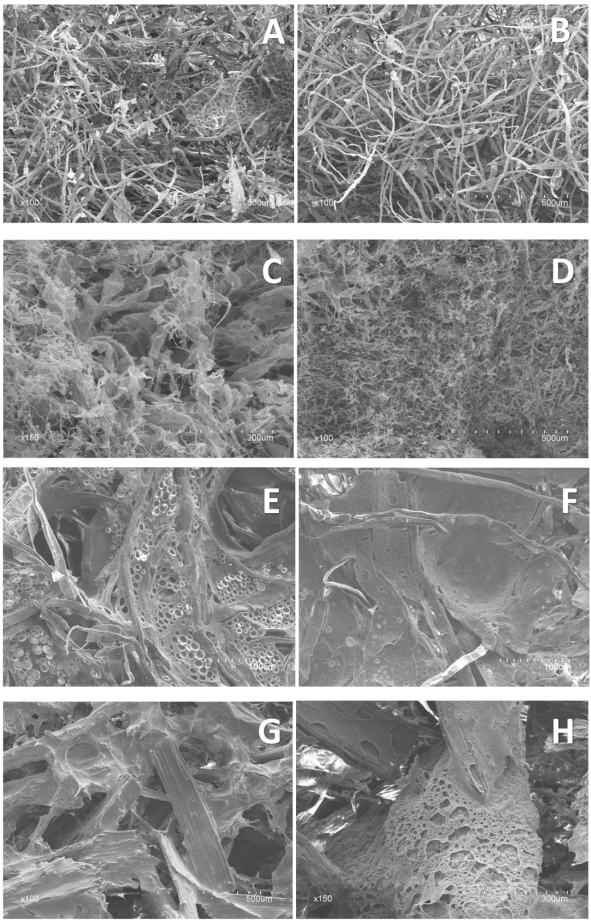

Figure 1: Surface SEM images of (A) IT 0.5% (-20ºC), (B) IT 0.5% (-80ºC), (C) VS 0.5% (-20ºC), (D) VS 0.5% (-80ºC), (E) IT 0.5% + PLA, (F) IT 0.5% + PHB, (G) VS 0.5% + PLA and (H) VS 0.5% +PHB

Source: own.

Finally, the biodegradability of the tested cellulosic sources and developed porous materials was also assessed according to the Standard ISO 20200, thus providing a sustainable and high-performance alternative to conventional materials, also following the circular economy principles (Figure 2). These results enable alternative applications for the developed porous materials, such as the agronomic field, where they can act as water retainers when incorporated in novel soil formulations, thus palliating drought effects. In addition, the possibility of incorporating both hydrophilic and lipophilic compounds (such as biostimulants and/or mycorrhizal preparations) which can be released steadily thanks to the biopolymeric coating [3], can improve the crops` growth without compromising the sustainability of the environment, as no further treatment is required for these porous materials.

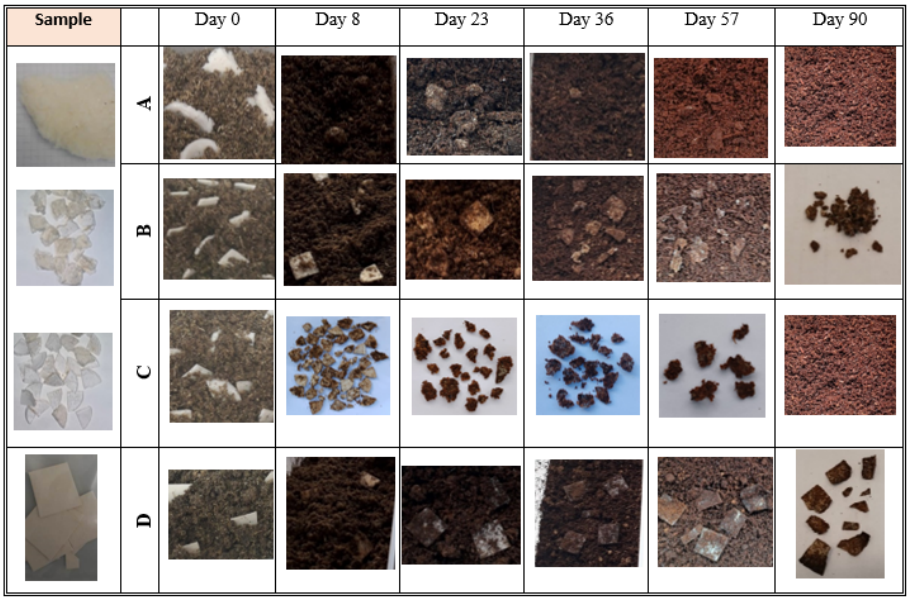

Figure 2: Visual appearance of the samples before, during and at the end of the disintegration process (90 days) being (A) 2% IT, (B) 2% IT + PLA, (C) 2% IT + PHB and (D) raw IT cardboard.

Source: own.

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Transparent Cellulose Aerogels from Concentrated Salt Solutions

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Mesoporous aerogels are promising materials for numerous applications, e.g. in the fields of Adsorption, Pharma and the Food industry, and as lightweight thermal insulating materials. The combination of typical aerogel features with optical transparency has been, up to now, governed mainly by silica aerogels, and opens up additional fields, e.g. as transparent insulators in advanced glazing units. Even though silica aerogels show high potential as transparent insulators due to their high transmittance in the visible region, brittleness and fragility caused by their nanocolloidal structure limits their commercial applicability.[1] In contrast, biopolymer aerogels` microstructure shows, in most cases, a fibrillar morphology with a biopolymer network consisting of flexible, three-dimensionally entangled fibers, which results in a foam like morphology and better mechanical properties. However, randomly connected fibrous structures generally result in opaque products, which do not match the requirements which are necessary to avoid light scattering (Figure 1). The latter is caused by interaction of light with the aerogel structure, in particular, by interaction of light with the spatial inhomogeneities of the refractive index n, which occur due to the porous nature of the material: e.g. air filled pores ($n \approx 1$) have a different refractive index as compared to biopolymer-fibers.[2]

Consequently, the fiber diameter and presence of macropores have a significant influence on light scattering in porous materials.

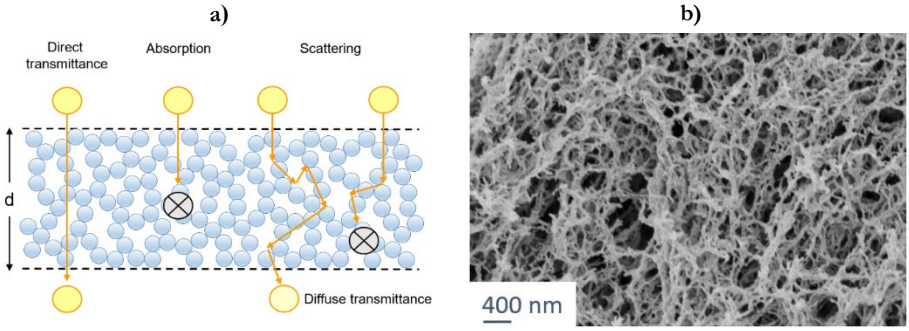

Figure 1: a) Schematic presentation of light transport/scattering in porous materials. b) Typical fibrous pore structure of an opaque cellulose aerogel.

Source: own.

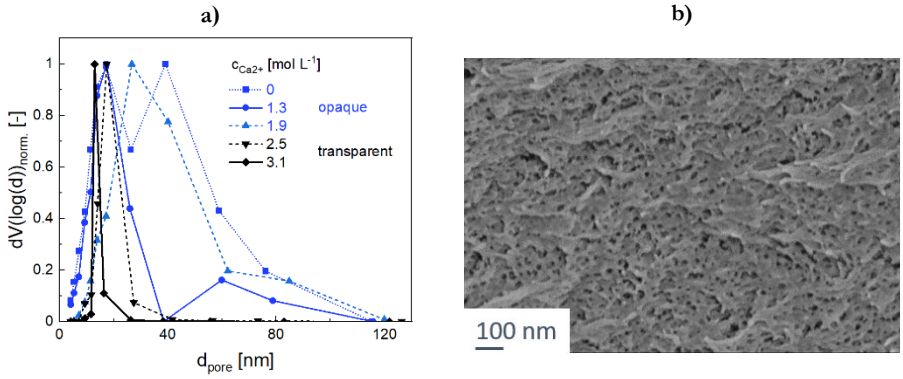

Figure 2: a) Mesopore size distributions of cellulose aerogels produced at different Ca^{2+} concentrations in the gelation bath. b) SEM picture of a transparent cellulose aerogel slab.

Source: own.

In light of the aspects given above, the following prerequisites can be formulated for biopolymer aerogels to achieve high transparency: 1) Small mesopores with uniform size distribution 2) Homogeneous pore networks containing no defects or larger structures. In order to obtain cellulose aerogels with the desired properties, a novel approach based on Ca^{2+}-induced crosslinking of aqueous cellulose-$ZnCl_2$ is presented, which allows precise control of the mesopore size distribution in a wide

range via variation of the process parameters` Ca^{2+}-/cellulose concentrations (Figure 2, a). The resulting aerogels show high optical transmittances (up to 80%) and a non-fibrous mesoporous structure (Figure 2, b), resulting in specific surface areas up to 655 $m^2 g^{-1}$.

It is notable that no costly/time consuming pre-oxidation steps of cellulose (e.g. TEMPO oxidation) and use of expensive/harmful solvents are necessary to achieve these properties via the presented salt-induced approach.

Acknowledgements

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Pickering Emulsion-templated Lignocellulosic Adsorbents for Pharmaceutical Pollutants

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Pickering emulsions are emulsions that used solid particles as stabilizers of the oil-water interface [1]. They are effective templates for porous beads or monoliths because of their highly stable droplet structure, which can be maintained even after the removal of the liquid phases [2,3].

In this study, oil-in-water Pickering emulsions were prepared using nanocellulose and lignin nanoparticles (LNPs) as solid stabilizers. The different factors affecting the stability of the Pickering emulsions were investigated, including the amount and charge of LNPs, pH of the aqueous phase, and the order of mixing the stabilizers. Stable Pickering emulsions characterized by small droplets and a slow rate of creaming, were favored with increasing LNP content, at pH 5 and 8, and with

simultaneous mixing, or with LNPs being added first. Cationization of the LNPs, emulsification at pH 3 and sequential mixing, with nanocellulose being added before the LNPs, resulted in large droplets prone to creaming. Freeze drying the stabilized Pickering emulsions enabled the production of macroporous materials (Fig. 1), having pore cell structures dependent on the droplet morphology. Smaller and more homogeneous pore cells were obtained in emulsions with combined LNPs and nanocellulose as stabilizers than with only nanocellulose. Finally, the Pickering emulsions-templated macroporous materials were tested as adsorbents for pharmaceutical pollutants, which are emerging aquatic contaminants causing global concern. The results showed that the addition of LNPs promoted the removal of aromatic ring-containing pharmaceuticals with different ionic characters.

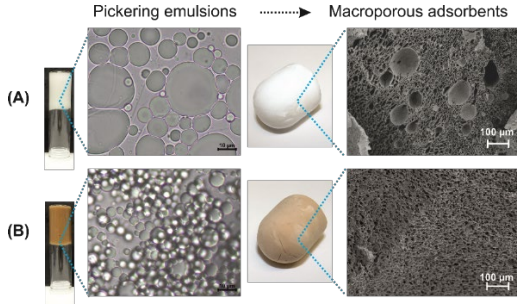

Figure 1: Oil-in-water Pickering emulsions stabilized by only nanocellulose (A) and in combination with lignin nanoparticles (B) producing macroporous adsorbents with freeze drying.

Source: own.

Overall, the study demonstrated the potential of LNPs as co-stabilizers of nanocellulose-based Pickering emulsions, at the same time, as modifying agents to the subsequent macroporous materials.

Acknowledgements

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Upscaling of Cellulose Extraction from Hemp Bast Fibers and Production of Cellulose Aerogel Beads: An Industrial Case

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Academia and industry have been developing ways to produce novel materials from renewable bio-based sources. Hemp is cultivated mainly for fiber production, due to its outstanding durability, absorbency, anti-mildew, and anti-microbial activity.[1-2] We have been developing strategies to upscale the extraction of cellulose from waste hemp fibers, in order to create cellulose aerogel beads. Alkali hydrolysis and bleaching established a simple method to extract high-grade cellulose from hemp waste fibers at the lab-scale.[3-4] Parameters such as the degree of fiber milling and time of residence have been modified, in order to adapt this method to a 2L reactor for industrial purposes. The quality of the extracted cellulose was analyzed by X-ray diffraction and FTIR.

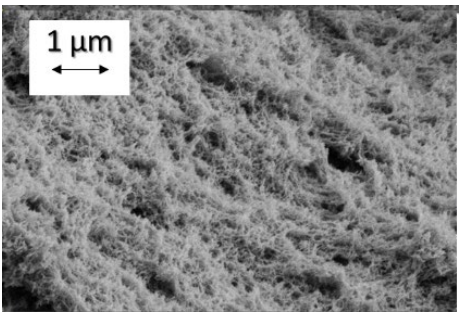

Figure 1: SEM picture of the internal microstructure of a cellulose aerogel.
Source: own.

The extracted cellulose was dissolved using a mixture of NaOH, urea, and water as a solvent.[5-7] The cellulose aerogels beads were produced using the Jet-Cutter® from commercial and extracted cellulose. Those samples were characterized, and compared via nitrogen adsorption-desorption isotherm, BJH pore data analyses, density analyses, FTIR and Scanning Electron Meicroscopy. Highly porous materials were produced from both sources of cellulose, which expanded the use of agricultural wastes at the industrial scale.

Acknowledgements

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Evaluation of Calcium Silica Aerogels as an Anticaking Agent for Food Powders

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Preservative additives are of critical importance in terms of extending the shelf life of foods and preserving their taste and structure. The aggregation of powdered foods during processing, transportation, and storage is a major problem that must be prevented. Anti-caking agents increase the fluidity of powders by reducing the stickiness and compressibility of interparticle forces. Anti-caking agents have a high surface area, allowing for the adsorption of a significant amount of ambient water However, anti-caking agents should have a more fine particle size than the main food powder particle size [1], [2]. In the food industry, silica-based powders are used widely as anti-caking agents, thanks to their superior surface area and particle size properties [3].Here, we present mesoporous calcium silica aerogels that can be employed as anti-caking food additives. With this aim, calcium silica aerogels were produced and optimized according to independent parameters (Si: Ca ratio, feeding time, and aging temperature) by using response surface methodology (RSM) and the Box−Behnken approach. The effect of the independent parameters on the BET

surface area and water vapor adsorption capacity was determined by performing ANOVA analysis [4].

Successively, the optimized calcium silica aerogel samples are characterized by means of N_2 desorption-adsorption analysis, inductively coupled plasma optical emission spectrometry (ICP-OES), particle size analysis, Fourier transform infrared spectroscopy (FT-IR), and Scanning Electron Microscopy (SEM). A digestive system model was created to evaluate the optimized products, and the amount of dissolution was determined after contact of the samples with artificial body fluids. *In vitro* cytotoxicity studies were performed by applying the MTT (3-(4,5-Dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide) cell viability test and Lactate Dehydrogenase (LDH) test using HEK-293 and Caco-2 cell lines. *In vivo* acute systemic toxicity analysis was performed on mice by administering the limit dose of 5000 mg/kg orally to CD1 mice.

In conclusion, calcium silica aerogels were added to the model food at a 1% ratio, which improved powder flow in the free flow region. The ion release of calcium silica aerogels is suitable for daily consumption in food. The MTT test results showed that calcium silica aerogels have low cytotoxicity. As the concentration decreases, the cell viability rate increases. The *in vivo* acute systemic toxicity analyses showed no toxicity. It was concluded that the produced silica aerogels did not have any toxic effect.

Acknowledgements

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Covalent Immobilization of Copper(II) Complexes on Silica Aerogels has High Impact on Their Catalytic Activity in Redox Processes

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In a quest for studying heterogeneous catalysis in advanced oxidation processes and developing novel antioxidant systems, mesoporous silica aerogels were synthesized, functionalized covalently with copper(II) complexes of cyclen and cyclam. These functionalized materials were characterized by SEM, N_2 adsorption-desorption porosimetry, ICP-OES and SANS measurements. The chemical structures of the precursors were characterized by mass spectrometry (MS) and nuclear magnetic resonance spectroscopic (NMR) techniques. Electron paramagnetic resonance (EPR) spectroscopy was used to study the coordination environment of the copper(II) complexes immobilized in the aerogels. Contrast variation small angle neutron scattering (SANS) and EPR measurements proved unambiguously that the Cu(II)-complexes were not aggregated and molecularly dispersed in the silica networks.

The immobilized copper(II) cyclen complex effectively catalyzes the oxidation of phenol by H_2O_2. In depth analysis of the kinetic and spectroscopic data confirmed that a unified kinetic model is adequate for the interpretation of the data,, and describes both the homogeneous and heterogeneous catalytic systems. [1] The results showed that the specific activity of the immobilized catalyst was slightly lower than that of the homogeneous counterpart, which is most probably due to the hindered mass transport. However, functionalization provided a higher rate for the activation of the catalyst, and ensured the conservation of the catalytic activity of the complexes. [2]

Another aspect is that the functionalized aerogel microparticles showed excellent catalytic activities in the dismutation reaction of the superoxide anion, thus mimicking the superoxide dismutase enzymes. The covalent immobilization increased the SOD activities of both Cu(II) complexes drastically compared to the aqueous parent complexes measured using the conventional xanthine / xanthine oxidase / NBT model system. This can be explained by the formation of a new chemical environment upon immobilization, the effective separation of the copper(II) centers, and the confinement effect operative in the silica backbone. Since silica aerogels are biocompatible and can normally be secreted by animals, antioxidant aerogel microparticles can be useful for the treatment of oxidative stress related conditions. [3]

Figure 1
Source: own.

Acknowledgements

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Optical Sensing of Contaminates in Water by Metallic Aerogels

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Contamination in water can occur through natural processes, such as leaching of minerals from soil, or through industrial waste. It is needless to mention that it has serious impacts of human health, as well as on the environment. Hence, the development of analytical sensors to detect those contaminations is highly important.

We developed a facile method for fabrication of aerogel-like metallic networks. Our method is based on physical vapor deposition (PVD) onto a charged substrate. The resulting aerogel-like metallic network contains a 3D carpet of 'hot-spots' at a broad range of optical regime, and, thus, leads to enhancement of the electro-magnetic (EM) field and optical processes. As a result of the strong, broadband, and deep sub-wavelength confinement of the EM field, inherently weak nonlinear optical processes, such as Raman scattering, can be boosted by orders of magnitude by this metallic network interface. Herein, we present aerogel-like metallic networks for optical sensing of contaminates in water. In addition, we show its application as a photocatalytic 3D structure.

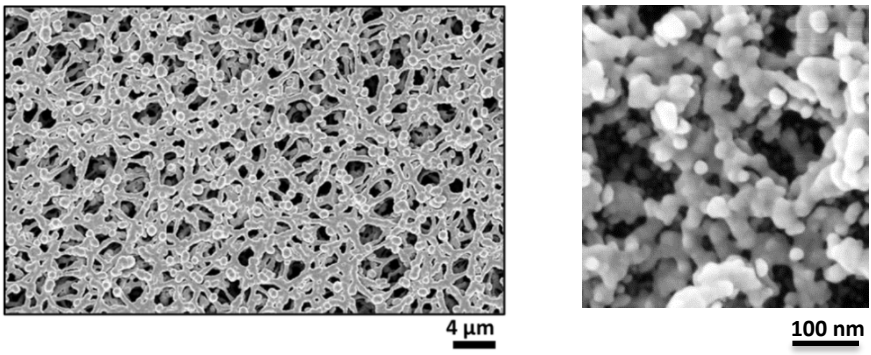

Figure 1. Large scale metallic aerogel-like networks made of Ag in a single fabrication step. By modifying the physical parameters of the fabrication process, one can control the average size of the ligament/building blocks.

Source: own.

Fabrication of metallic aerogels via traditional sol-gel synthesis is challenging, and highly dependent on the chemistry of the metal of interest, and unachieved for some metals. As such, alternate synthetic strategies for synthesizing metal aerogels are highly desirable. Herein, we present the fabrication of aerogel-like metals grown by a direct PVD technique in the free space above a charged substrate, guided by electrostatic field lines extending off the substrate. The combination of nanosized metallic elements with three-dimensional organization over macroscopic dimensions of these materials shows an ample range of environmental applications, including photo-catalysis, light harvesting, chemical purification, optical sensors, and batteries. [1]

The random architecture of such metallic aerogel-like structure results in electromagnetic field enhancements at hot-pots over a wide optical range, since a network combines a broad range of shapes and nanosizes of particles and holes structurally, and both are optically active elements.[2]

Being large enough in size, these interconnected networks become optically robust materials so that local damages do not lead to a system failure. As a result of the strong, broadband and confined EM field, inherently weak nonlinear optical processes, such as Raman scattering and second harmonic generation (SHG), are boosted by orders of magnitude.

Surface-enhanced Raman spectroscopy (SERS) is a powerful analytical technique that can detect trace amounts of chemicals with high sensitivity and specificity. There have been several developments in SERS technology in recent years, that have improved its performance and expanded its capabilities. However, being a nonlinear process it is negligible, and the high numerical aperture (NA) objective is needed to collect the scattered signal. Using our metallic aerogel we enable detecting analytes at low concentration with a low NA objective of about 0.3.

Herein, we develop SERS based sensors for the detection of different families of chemical pollutants in water;(i) Piperidine and its derivatives (Pharmaceutical waste) (ii) Pesticide (iii) Dioxins & Polychlorinated biphenyls (PCBs). [3]

Acknowledgements

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Sustainable Production of Aerogels from Recycled Materials, A Lifecycle Analysis

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The production of aerogels has been of significant interest, due to their unique properties, including high thermal insulation and high surface area, making them useful in a variety of applications. In particular, aerogels for this research are produced for the purpose of being an insultation material for buildings. Utilizing aerogels as a thermal insulation solution would help address the issue of energy inefficiency in homes in the Netherlands[1].

However, the traditional method of producing aerogels, high-temperature supercritical drying (HTSCD), has proven to be expensive and energy-intensive, resulting in significant carbon emissions. Low Temperature Supercritical drying (LTSCD) has been found to have an even bigger CO_2 footprint, due to the amount of methanol and liquid CO_2 required for the process[2]. As an alternative, the study investigates the use of ambient pressure drying (APD) as an alternative production process for aerogels. APD is of particular interest, since the supercritical drying step is one of the most energy-intensive components of the aerogel production process. The use of waste glass as a replacement for commercial silica will be investigated additionally. By combining alternative source materials with this alternative process,

the research aims to reduce the negative environmental impact of aerogel production.

The study conducts a simplified life cycle assessment of the APD aerogel production process, to determine the overall environmental impact. This will help determine how much more sustainable the APD process compared to the HTSCD process actually is, as well as giving insights as to which the biggest emission producing steps are. Surprisingly, there is limited literature on the lifecycle assessments of aerogels, which underscores the need for further research in this area. By conducting a lifecycle assessment, this study aims to identify areas for improvement and guide future research in the field.

Acknowledgements

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Environmentally Friendly Composites Based on Agricultural and Plastic Waste: Production Methods, Current Progresses and Challenges

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In the last decade the development of new composite materials governed by the criteria of industry, technical-economic and environmental criteria are more focused on obtaining materials with good mechanical properties with low cost, and "eco-friendly" for the environment. Environmental hazards imposed by the accumulation of plastic waste and reduction of natural resources, oil and all raw materials related to the further production of monomers, etc., prompted the need for the application of waste and natural materials, especially those from renewable resources, in obtaining new composites and providing a return flow of reactants. Nowadays, the pressure increases more on manufacturers of materials and finished products to take into account the impact of their products on the environment, starting from the manufacturing process, the cycle of application and their final removal. This "sustainable design" has become a philosophy that is used intensively to a growing number of materials and finished products. These requirements, in combination with unnecessary production costs, recently prompted substantial interest in

obtaining new composite materials, so the academic world and many industries are more and more focused on new sustainable technologies and ensuring a sustainable environment. The combination of good mechanical and physical properties of plastic waste, natural materials from renewable sources, along with their "friendly" relation to the environment, prompted various activities in the area of sustainable technologies. The selection of technologies and processing conditions for obtaining a composite material from agricultural and plastic/recyclable waste and natural components from renewable resources is of particular importance, because of the significant effect on the properties of the resulting material and on the price of the final product.

For all processing techniques of composite materials it is necessary to apply sufficient pressure at a certain temperature and for a limited time, in order to obtain a composite with stable dimensions from the liquid mass reinforcement - matrix. Therefore, research of technologies for obtaining the composites from waste, recycled and natural components with maximum use of them and by providing a return flow of unuseful reactants, has been challenge. Also, a scientific and practical challenge is their characterization and the possibility of their recycling and reuse.

The environmental production of various construction materials for application in agriculture, construction, the furniture industry and others has been investigated in this paper. The processes for compression and injection molding have been applied, and, as a basic material plastic/recyclable waste has been used, as well as biodegradable materials from renewable resources in our country. The possibility of introducing a sustainable technological procedure for the production of new composites from multiple recycled material has also been investigated additionally. Interest about new composites based on waste/recycled materials is growing rapidly, because of their higher environmental standards, the potentially good properties of these composites, low density and cost, the opportunity for maximum utilization of the reactants, and providing a return flow of waste material.

In the framework of these investigations, we have used different types of plastic recyclable wastes and natural materials from renewable resources as a matrix, and various types of agricultural waste as reinforcements. The composites containing 30 wt % reinforcement were manufactured by compression and injection molding, and their mechanical and thermal properties were compared. It was found that the

applied techniques for manufacturing of the environmental friendly composites under certain processing conditions did not induce significant changes of the mechanical properties. The optimal processing conditions for both techniques have been determined based on the results. The experimental results suggest that compression and injection molding are promising techniques for processing of environmental friendly composites. We tested the recycling ability of the produced composites by using multiple recycled material, and their applicability - by comparison with traditional composites intended as construction materials in various industries. These new produced composite parts are "friendly" to the environment, with a low price, good quality and technical performance on the one hand, and, on the other - cheaper and competitive for import parts.

This research may causeencouragement for the development of sustainable technologies mainly in agriculture, but also in construction, furniture, the automotive and other industries, and also the application of recyclable and waste material would be wider and the quality of life in terms of environmental protection would be better.

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Biomimetic Assembly of Artificial Leaves and Stems of Plants Using Polysaccharide for Water Desalination

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Plants have attracted enormous attention as biomimetic models because of their remarkable combination of mechanical strength and light weight. The hierarchical cellular structure of highly-oriented cellulose fibrils in the matrix (hemicellulose and lignin) is largely responsible for such properties in plant tissues.

Beyond the structural aspects, plants perform a myriad of functions. In particular, liquid transport between the roots and the leaves is an inspiration to design new desalination devices. The driving force behind this function is transpiration, which determines water transport from the roots through the stems' macropores (xylem) up to the surface of its leaves, driven by sunlight. One of the consequences of transpiration is the separation of the water from its solutes. Especially, plants like

mangroves, willows and reeds are able to generate clean water from seawater and wastewater through the solar power as a sole inexhaustible energy source [1].

Here, we propose an analogous system able of reproducing the functions and structure of the plant stem and leaves, ultimately capable of water desalination via sun-powered evaporation. The isotropic hydrogels corresponding to the "stem" were obtained by ice templating, a materials processing technique that enables the formation of aligned macropores by controlling ice growth precisely in solutions and/or suspensions [2,3]. By freezing oxidized cellulose dispersion in an alginate matrix and stabilizing them through calcium ion-crosslinking, we aim at reproducing the vascular structure and function of the plant stem.

Furthermore, hybrid buckypapers (carbon-clay black paper-like material) were prepared to act as artificial "leaves" by vacuum filtration [4], and placed on top of the anisotropic hydrogels. Because buckypapers have excellent photo-thermal properties, they accelerate the capillary liquid transport behavior of the hydrogels in the presence of the artificial solar light.

Herein, we will discuss the influence of the alginate moiety on the mechanical properties and morphologies of cellulose-based hydrogels through compression tests and SEM observation. Besides, we will describe the role of ice growth velocities on the morphology (pore size and pore shape) of cellulose-based hydrogels on the capillary transport properties and intrinsic diffusion coefficients. Finally, we will report the assembly of the buckypaper as "leaves" and the alginate/oxidized cellulose hydrogel as "stem". This assembly irradiated under solar light irradiation for 6 hours yielded low salinity with 0.0072 wt% from 3.5 wt% initial salt water, which is much below the salinity levels stipulated by the World Health Organization (WHO).

In summary, these results highlight their potential applicability for new bioinspired materials that combine relevant mechanical properties, effective capillary transport, and a remarkable ability to desalinate for water purification.

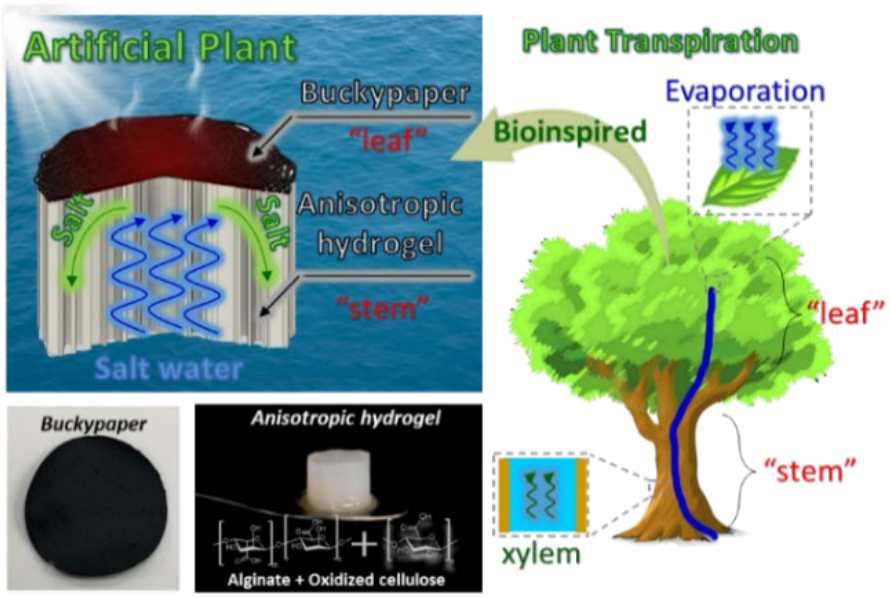

Figure 1: Working principle of cellulose-alginate macroporous hydrogels and their similarity to native plants

Source: own.

Acknowledgements

"This work has been supported by a French Government Scholarship 2021-2022 and the Île-de-France Region through RESPORE (Network of Excellence in Porous Solids)."

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An Overview of the Environmental Impacts of Aerogels with the Life Cycle Assessment Approach

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Aerogels are unique nanoporous materials with multiple superior properties, such as low thermal conductivity, extremely low density, low dielectric constant, high porosity, and a large surface area. The combination of these properties makes aerogels excellent candidates for a wide range of applications, and, in addition, increases their commercialization potential. Aerogels have been produced on a lab scale for a long time; however, some silica, organic, and carbon aerogel types have been commercialized in recent years [1]. Growing concerns about environmental sustainability make researchers and industrialists take more environmentally friendly decisions at every stage of production, including product design, material selection, production processes, consumer delivery and support, and product end-of-life management after its useful life has ended [2, 3]. The life cycle assessment (LCA), a thorough process for investigating both energy and material flows, examines the

effects of resource consumption, the generation of products, by-products, waste, and emission release on the environment.

This study aims to review the available literature on the LCA of aerogel materials, and to assess the methodology used, as well as the results of the LCAs and recommendations to mitigate environmental devastation. In order to elaborate aerogel LCA studies, the system boundaries and functional unit (FU) will need to be consistent, in line with the goal and scope set by ISO 14040-44 [4, 5] for the comparisons. Most of the existing aerogel studies are in the field of Thermal Insulation. Füchsl et al. [6] reported that the use of thermal resistance as FU instead of the existing m^3 would be more appropriate for comparison so that it can meet the scope fully. Besides, most of the aerogel LCA studies have been conducted from the cradle to the laboratory gate. Cradle-to-grave LCA has been proposed for materials such as aerogel, because many auxiliary substances can be recycled in the use and end-of-life stages. Additionally, mass or volume allocation is generally preferred, while economic allocation should be done where the amount of the main product is low compared to the by-products [6].

The Global Warming Potential (GWP) of the studies in the literature based on adsorption and thermal insulation differs from 16.9–4830.0 kg CO_2eq/kg, and the GWP of drug delivery studies differs from 127.0–6970.0 CO_2eq/kg. Other impacts, such as Acidification Potential and Eutrophication Potential, differentiate between 0.19–50.90 kgSO_2eq/kg and 0.03–2.56 kgPO_4^{-3}eq/kg, respectively. Many studies have high burdens because they were analyzed at a lab scale. On the other hand, in the industrial-scale simulation of these studies, it is observed that all environmental impacts are reduced by 95%. SimaPro software and the Ecoinvent database have been used in almost every study. However, a variety of impact assessment methods and a lack of life cycle data have led to inconsistencies. Forthcoming studies should be more transparent, in order to evaluate aerogel LCA comprehensively.

The results come up with the most influential parameters in the environmental impact categories being the precursors and solvents (TEOS, ammonia, ethanol, heptane, etc.) used in the gel formation and aging steps, and the energies of the equipment (stirrer, air dryer, freeze dryer, supercritical dryer, etc.) used in the aging and drying processes, depending on the temperature and time conditions. In light of these concerns, it is suggested that the precursors and chemicals should be replaced

with greener alternatives or produced from waste, or that the production methods for these chemicals should be greener. However, in the worst-case scenario, it is predicted that the most effective environmental impact reduction strategy will be the recycling of these chemicals. The significance of geography should not be ignored in environmental impact studies from the point of view of both energy consumption and transport. In EU countries, the environmental impact may be quite low in countries where the consumed energy is more renewable and biofuel is used in transport, while the environmental impact is high in countries that are far from raw materials, use petroleum-based fuels, and obtain most of their energy from fossil fuels. The findings further recommend that the environmental impact can be reduced by using equipment with higher energy efficiency, using renewable energy sources, reducing the processing time and temperature, greater product batches and continuous flow processes by transition to a large scale.

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A Closed-loop Recycling and Post-synthetic Reprogramming of Organic Aerogels

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Aerogels are advanced structural materials based on their low density, high porosity, and mesoporous structure with high specific surface area. Thanks to their excellent characteristics, aerogels are attractive for various applications, such as thermal insulation, energy storage, drug delivery, and chemical adsorption.[1] However, due to their highly cross-linked and irreversible chemical networks, recycling of organic aerogels is a scientific challenge.

To counter this we used reversible imine linkages to prepare chemically recyclable aerogels. The imine bonds can be hydrolyzed under ambient acidic conditions, and the monomers can subsequently be recovered with high yields and purities. This approach allows for preparation of fresh aerogels from the retrieved building blocks, thus demonstrating efficient closed-loop recycling.[2] In addition, the dynamicity of the imine bonds has been utilized to reprogram the properties of aerogels after their synthesis.[3,4] Based on the associative equilibrium between primary amines and imines, the addition of amines results in the breakdown of the cross-linked polyimines into soluble oligomer structures (Figure 1). This dissolution can be used to reform original gels by adding fresh aldehydes. Consequently, a newly generated aerogel can be prepared by supercritical CO_2 drying. Depending on the choice of

amine reagents, the pristine aerogels can also be altered by introducing diverse material properties, such as increased hydrophobicity and mechanical performances. This unique approach provides us with the possibility to repair damaged aerogels, or implement different functionalities into existing organic aerogels after their preparation.

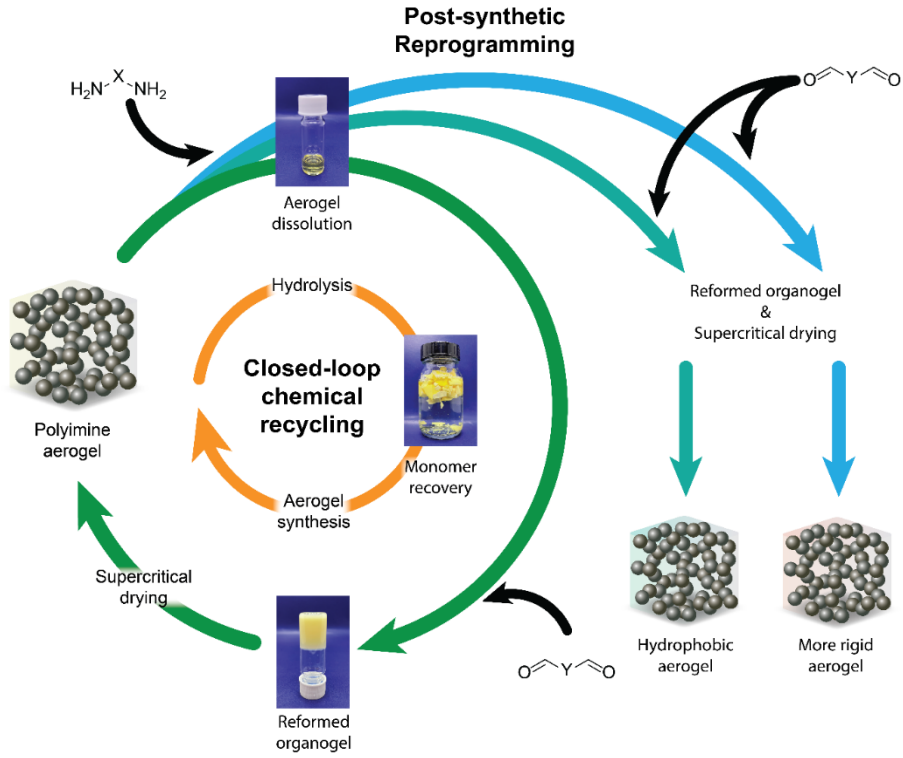

Figure 1: Closed-loop recycling and post-synthetic reprogramming of polyimine aerogels
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Acknowledgements

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Utilization of Lignin Aerogels Into Targeted Industrial Applications Blow-in Insulation as a Prototype

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Many climate change agreements, including the Paris Agreement and European Union 2020, emphasized the importance of improving the energy efficiency of buildings as a critical measure to limit global warming, thus increasing the need to develop energy-efficient and low-carbon solutions for buildings and construction [1], [2]. Biopolymer aerogels represent innovative insulation materials due to their distinctive properties, such as low density, high surface area, and very low thermal conductivity (e.g. 0.017 $W.m^{-1}.K^{-1}$) [3]. However, some factors delay their utilization in industrial applications, such as high price, sensitivity to moisture, low mechanical stability (in some cases), and challenges associated with scale-up [4].

Lignin represents a renewable biopolymer, which, when incorporated with aerogels, led to a mechanically stable biopolymer aerogel with higher hydrophobicity and less moisture sensitivity [5]. Therefore, this study aims to evaluate the potential of lignin as an additive in alginate aerogels, to enhance application-relevant properties.

In this study, lignin microparticles were mixed with aqueous alginate solutions, and hydrogel particles were produced via the dripping method in a calcium chloride solution, to facilitate ion-induced gelation of alginate. Thereafter, the CO_2 supercritical dried alginate-lignin hybrid aerogel particles were mixed with cellulose fibers, and then blown into hollow wood frames using the blow-in technique. The aerogel particles were evaluated in terms of particle morphology and porous structure, while the aerogel cellulose fiber mixtures were evaluated in terms of homogeneity and thermal conductivity.

The preliminary results showed that alginate-lignin hybrid aerogels are sufficiently stable, and, thus, suitable to be used as a raw material for blow-in insulation. The replacement of cellulose fibers by lignin aerogel particles up to 32 wt % decreased the thermal conductivity by 9% and achieved a lower thermal conductivity of 34.09 $W.m^{-1}.K^{-1}$. The targeted application is thermal insulation material for building elements.

Acknowledgements

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Hybrid Porous materials, Peptide- Alginate /poly(itaconic anhydride-co-3,9-divinyl-2,4,8,10-tetraoxaspiro[5.5]undecane) Based with Controllable Properties

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Over the years, biopolymer-based aerogels have attracted particular attention in numerous biomedical and environmental applications, due to structural features like high porosity, extremely low density and large specific surface area[1]. Nevertheless, the hybrid materials have exhibited remarkable properties, achieved via a synergistic combination between two or more compounds [2]. This study reports a strategy for the development of hybrid networks based on natural/synthetic polymers (Figure 1). Thus, the use of Fmoc-Lys-Fmoc amino acid and Fmoc-Gly-Gly-Gly tripeptide fragments for the development of a self-assembled system (1st network) provides

excellent biocompatibility, biodegradability and specific bioactivity, while the network based on alginate and poly(itaconic anhydride-co-3,9-divinyl-2,4,8,10-tetraoxaspiro[5.5]undecane) (PItAU)(the 2nd network) offers adjustable rheological properties [3,4].

The preparation of the hybrid network took place in three steps: (i) Preparation and characterization of the supramolecular co-assembled 1st network; (ii) Synthesis of the PItAU copolymer and NaAlg/PItAU bioconjugate formation; (iii) *in situ* preparation of the hybrid networks.

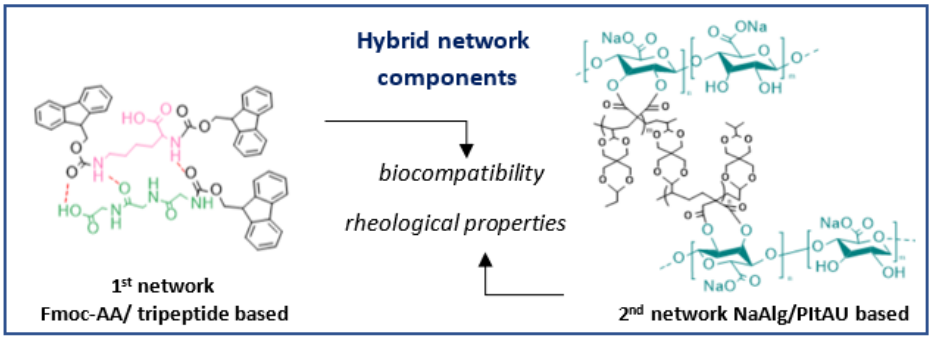

Figure 1: Chemical structures of the hybrid network components.
Source: own.

The supramolecular co-assembled system was characterized by circular dichroism, fluorescence and ultraviolet–visible (UV-vis) spectroscopy, to reveal the molecular arrangements between Fmoc-Lys-Fmoc and Fmoc-Gly-Gly-Gly. The copolymer structure and the NaAlg/PItAU bioconjugate formation were confirmed by spectroscopic analyses (^{1}H- NMR, FT-IR). The hybrid compound was characterized by dynamic vapor sorption (DVS) measurements, FT-IR, SEM and rheological analysis, to provide an insight into the hybrid structure, as well as to conclude upon its final properties.

The study results evidenced that a synergistic hybrid network generated by the physical interactions between the 1st Fmoc-AA/tripeptide system and the 2nd NaAlg/PItAU network had been prepared successfully, also providing multifunctional behavior. The average pore size and BET surface area obtained from

the DVS sorption/desorption isotherms have shown that the Fmoc-AA/tripeptide_NaAlg/PItAU hybrid network presents a porous specific surface characteristic to aerogels.

Acknowledgements

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Supercritical CO_2 Extraction from Bilberry (Vaccinium myrtillus) Fruit and Impregnation of the Obtained Extract Onto Starch Aerogel

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Bilberries (*Vaccinium myrtillus*) have been used in traditional medicine to reduce inflammation and protect against diseases associated with oxidative stress, such as cardiovascular disease, diabetes, and age-related cognitive decline, as well as a remedy for eye conditions. The biological activity of bilberries is ascribed mainly to their high content of anthocyanins, but also to other valuable phytochemicals, including flavonols, phenolic acids, and tannins [1,2]. The enhanced stability and bioavailability of bioactive components from plant material could be accomplished by their incorporation into a polymeric carrier, using the promising integrated processes of supercritical fluid extraction (SFE) and supercritical solvent impregnation (SSI).

In the present study, starch aerogels were functionalized with bilberry fruit extract using the SFE-SSI process. The process conditions were optimized, based on the yield and chemical profile of the extracts and impregnated compounds, as well as the loading amount of extract in the aerogels. The starch aerogels were prepared as

previously reported [3] from an aqueous solution of cornstarch (1:10 w/v). After the replacement of water with acetone, by a successive increase of its concentration until 100%, the formed acetogel was subjected to drying using supercritical CO_2 ($scCO_2$) at 45 °C and 10 MPa. Aerogel was obtained, characterized by a high specific surface area of 208.6 m^2/g, porosity of 81.8% and 273 kg/m^3.

Initially, supercritical CO_2 ($scCO_2$) extraction was investigated as a method for isolating the extract from bilberry fruit at the temperature of 70 °C and pressures of 15 and 30 MPa, with and without the ethanol as a co-solvent. The obtained extraction yields ranged from 1.2 to 7%, with total phenolic content (TPC) values ranging from 250 to 380 mg GAE/g of the extract. The highest yield was obtained at a higher pressure of 30 MPa, while the addition of ethanol had a positive effect on the TPC. HPLC analysis revealed a high content of Procyanidin B1 in all the extracts.

The aerogel was impregnated further with bilberry fruit extract using the integrated SFE-SSI process. The influence of different parameters, including pressure, the addition of ethanol and plant material to the aerogel mass ratio was analyzed, resulting in impregnation yields around 32.4%. The presence of the extract on the surface of impregnated samples was confirmed by FTIR analysis (Figure 1).

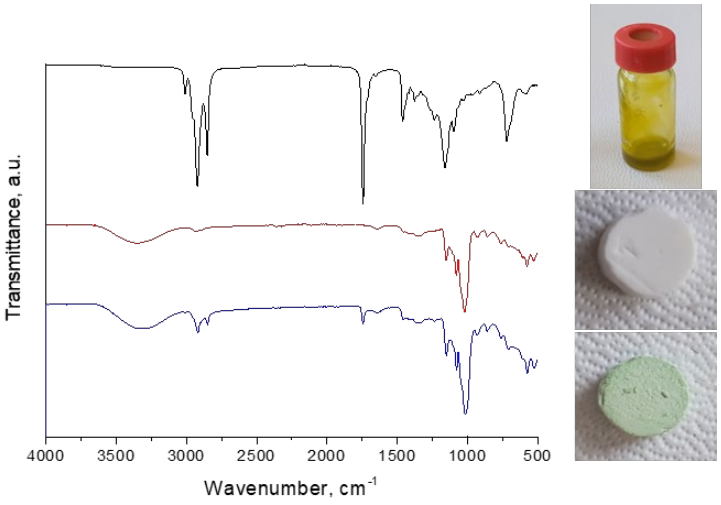

Figure 1: FTIR spectra of the extract and aerogel before and after impregnation
Source: own.

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Interaction of Aqueous Bovine Serum Albumin with Silica Aerogel Microparticles

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Silica aerogels are used widely as drug delivery systems, medical scaffolds and sorbents.[1-3] Understanding the nature of the interactions of biomolecules with these porous nanostructured materials is essential for achieving optimum performance in the targeted applications. In this study, we focus on the adsorptive properties of silica aerogel microparticles in aqueous suspension. The second component of the system is BSA. Due to its 98% similarity to HSA, it is a choice of preference for modeling human biomedical applications.

Aqueous BSA was mixed with suspended silica aerogel microparticles, and the colloid system was monitored on-line by UV–vis spectrophotometry and turbidimetry. Kinetic measurements on the absorption process were carried out at two different pH: 4.6 and 6.4, in acetate and phosphate buffers (I=0.10 M), respectively. The BSA and the silica aerogel concentration dependence of the process was investigated in detail. Additionally, Zeta potential measurements were performed, to determine the isoelectric points of the silica aerogel and the BSA. The global mathematical analysis of the time-resolved data revealed that the fast sorption of the protein on the aerogel microparticles corresponds to a multistep binding

mechanism. The extensive sorption of the protein eventually induced the aggregation of the covered aerogel, due to the alteration of the electrical double layer of the particles. The interaction of the BSA and silica aerogel was the strongest between pH = 4.0 and 5.0, because their native surface charges are the opposite in this pH range, as indicated by their respective zeta potentials.[4]

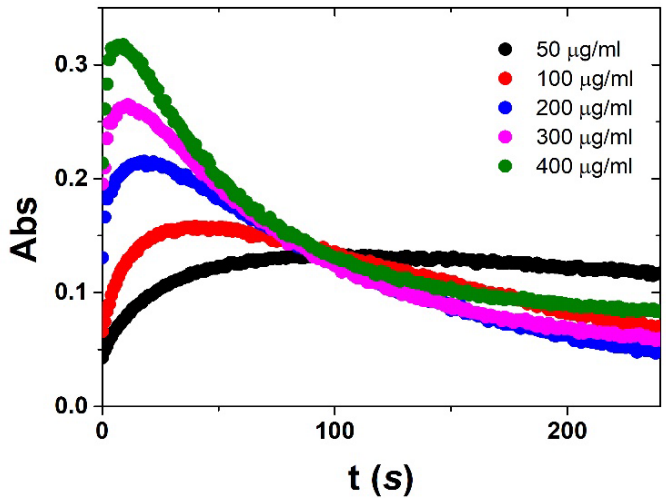

Figure 1: Adsorbance of BSA on 100 μg/ml silica aerogel in acetate buffer pH=4.6
Source: own.

Acknowledgements

The research has been supported financially by the National Research, Development and Innovation Office (NKFIH), Hungarian Science Foundation (OTKA: FK_17-124571 and K_21-139140).

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Using Ice to Mimic the Extra-cellular Matrix of Arterial Tissue: The Role of Topotactic Fibrillogenesis

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Arteries, and especially small-sized arteries, lack biocompatible replacement solutions [1]. Despite providing a similar shape and mechanical properties to native tissues, current grafts fail to reach their biological composition and organization complexity, which leads to aggregation and occlusion issues.

The arterial wall is composed of an assembly of glycosaminoglycans and proteins, mainly type I collagen [2], assembled into an extra-cellular matrix (ECM) that surrounds the cells. When decellularized, the ECM left reveals a porous 3D collagenous structure, whose reconstitution has remained elusive, despite the advances in biofabrication.

Our group has pioneered the elaboration of porous materials using biologically derived polymers. In particular, we have developed a successful method to control the hierarchical structure of 3D dense collagen materials [2], [3]. Such biomaterials recapitulate the structural, biological and functional features of the cells' microenvironment. Herein, we detail how to design those grafts that mimic arteries' ECM by combining ice-templating and collagen fibrillogenesis. Highly concentrated collagen solutions are ice-templated to allow ice crystals` growth and subsequent collagen segregation in-between the crystals. The ice crystals are then melted slowly at a low temperature to reveal pores, while collagen packing is maintained and its self-assembly induced simultaneously. Two fibrillogenesis methods are compared, to study their influence on the porous network evolution through *in situ* cryoconfocal microscopy, as well as the collagen molecules' arrangement (SAXS, PLOM), and their influence on the biological and mechanical properties (Figure 1).

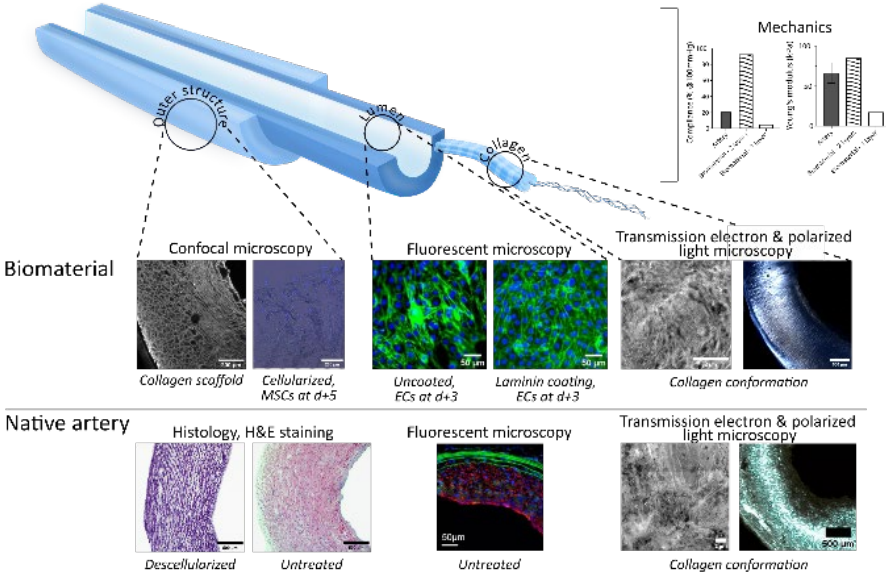

Figure 1: Mimicking arteries' ECM through ice-templating and innovative self-assembly techniques.

Source: own.

By exploring the physics of ice and innovative collagen self-assembly techniques, we can tailor: the collagen conformation and the textural aspects of the material's interfaces, to direct its interactions with recipient cells (endothelial cells and smooth

muscle cells), the number of structural layers to reproduce the architecture of arteries, and the various mechanical features to reach those of native tissues. In this communication we will focus on the impact of these novel fibrillogenesis routes to tailor new biomimetic materials, from their molecular organization up to their macroscopic mechanical functions [4].

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DAY 3

AERoGELS

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Surface Analysis for Biomaterials

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Many analyses focus on bulk material. However, in certain cases, the properties of the surface (the topmost position of the sample) may be more important. The surface analysis involves the investigation of a few nm thick layers on the material surface (5–10 nm). The most commonly used techniques for surface analysis are X-ray Photoelectron Spectroscopy (XPS) and Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS).

XPS can be used to determine information about the elemental composition of the surface, the composition of the thin layered structures on the material`s surface, and, more importantly, the binding of certain species (the environment of a particular element). Moreover, with the development of a gas cluster ion beam sputter source, XPS analysis is no longer limited to a few nm thick analysis regions of organic materials, but it enables the analysis deeper into the subsurface region (a few micrometers deep in the organic material) [1-3].

On the other hand, the ToF-SIMS technique allows the acquisition of molecular-specific information in addition to the elemental composition. In particular, the ToF-SIMS technique is very powerful in 2D and 3D imaging (Figure 1), to determine the spatial distribution of different species simultaneously.

When performing XPS and ToF-SIMS analyses, and when samples are not prepared under inert conditions or in a vacuum, in most cases, environmental species from the surrounding atmosphere, i.e., adventitious carbonaceous species, are adsorbed onto the sample`s surface. This can be problematic for fragment determination when interpreting the ToF-SIMS spectra. To solve this problem a second SIMS analyzer can be used, i.e., the use of a tandem ToF-SIMS technique.

In this work, various aerogels and biomaterials will be analyzed using surface analytical techniques, focusing on the use of ToF-SIMS, XPS, and atomic force microscopy (AFM) as the analytical tools to acquire information not possible to obtain with conventional analytical techniques. Both the results of the successful and unsuccessful development of biomaterials will be presented.

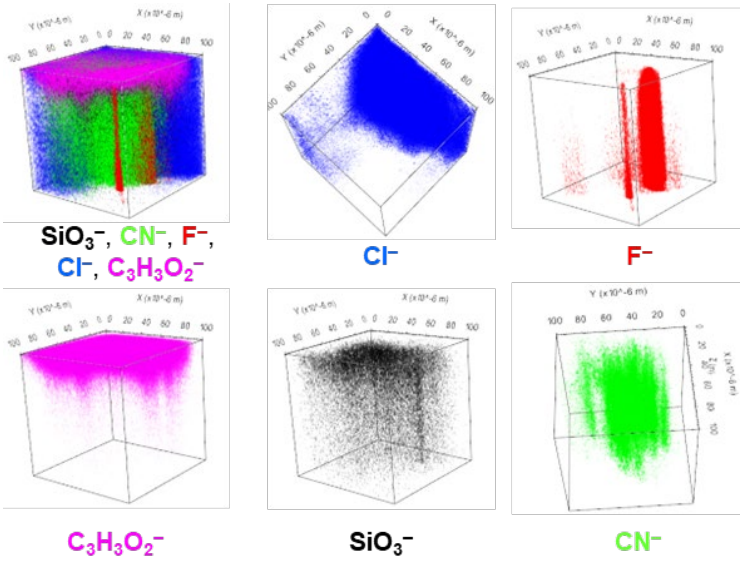

Figure 1: 3D ToF-SIMS image of the surface coating

Source: own.

Acknowledgements

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Polyurea-crosslinked Alginate Aerogels – the Design and Synthesis of Nanostructured Materials with Diverse Applications

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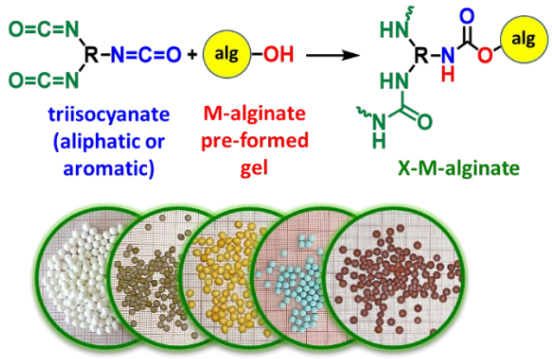

Figure 1: Graphical abstract.
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Alginate aerogels are very attractive materials, because they: (a) Come from renewable (natural) resources; (b) Are prepared in water; (c) Bear a large number of functional groups available for coordination to metal ions, hydrogen bonding,

functionalization, etc.; (d) Are biocompatible, biodegradable and non-toxic; and, (e) Can be converted pyrolytically to carbon aerogels with high open porosities and surface areas. However, their main drawbacks are that they are mechanically weak and extremely hydrophilic materials. These issues have been rectified with the introduction of polyurea-crosslinked alginate (X-alginate) aerogels [1-5].

In terms of their mechanical properties, X-alginate aerogels can be as stiff as the best organic aerogels [6] at half or the one third of their density. They are also extremely stable in all aquatic environments, with a pH in the range of 2-9, including seawater and various wastewaters. These properties, along with their ability to bind different metal cations, have allowed their application to:

a) Environmental remediation, as adsorbents of heavy metals, organic solvents and dyes;
b) Biomedicine, as candidate materials for implants; and,
c) The preparation of metal- and N-doped carbon aerogels in good yields, suitable for porous carbon electrodes and supercapacitors.

Acknowledgements

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Ice-templated Gelatin Aerogel Composites

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Gelatin (G) is an inexpensive water-soluble protein that can be obtained from byproducts or wastes from different industries. Due to its natural origin, it is a renewable and biodegradable material,, which makes it attractive as an ecofriendly alternative in multiple economic sectors. However, its relatively low mechanical properties and water sensitivity have limited its applications. The abundant amino and carboxyl groups in gelatin offer numerous physical and chemical cross-linking reactive sites, that can be used to obtain high-performance biodegradable composites with enhanced properties. Due to its high efficiency, chemical compounds such as glutaraldehyde have been used traditionally [1]. However, alternative bio-based substances can be employed to avoid glutaraldehyde toxicity, e.g., during biodegradation.

Natural tannic acid (TA) can strengthen the mechanical properties of the gelatin effectively, either by hydrogen bonding or by inducing a chemical reaction between the hydroxyls of the tannic acid and the gelatin amino groups (Figure 1a), usually under oxidative conditions [2]. Moreover, the high nitrogen content in the amino acids of gelatin and the combined charring action of tannic acid and montmorillonite clay (MMT) allow obtaining a low-flammability composite aerogel [3-4]. Thus,

combining these three components properly may allow obtaining high-performance sustainable composite aerogels capable of substituting traditional petrol-based foamed materials.

In the present work, the properties of freeze-dried gelatin aerogels modified with tannic acid and clay have been studied as a function of TA content. A high porosity and low densities characterized the composite aerogels. Moreover, exceptional stiffness was obtained, reaching values of specific compressive modulus as high as 150 MPa, which was a 9-fold increase compared to a pure gelatin aerogel. The significant increase in mechanical properties was attributed to the creation of a strong network of covalent C-N bonds between the TA and gelatin through Schiff's base and Michael's addition reactions. The thermal resistance of the composites also increased, as deduced by a decrease in the thermal degradation speed. The protecting action provided by the MMT clay and the charring action of TA allowed obtaining flame-retardant foam-like materials that were characterized by a low peak of heat release rate (PHRR) of 65 kW/m² when exposed to a 50 kW/m² irradiation, corresponding to a well-developed fire (Figure 1b). Finally, hydrophobicity and resistance to harsh environments were obtained by simply immersing the aerogels in a tetrahydrofuran solution containing polydimethylsiloxane (PDMS) and SiO_2 nanoparticles. These multifunctional gelatin composite aerogels are promising ecofriendly alternatives to synthetic polymer-based foams suitable for engineering applications.

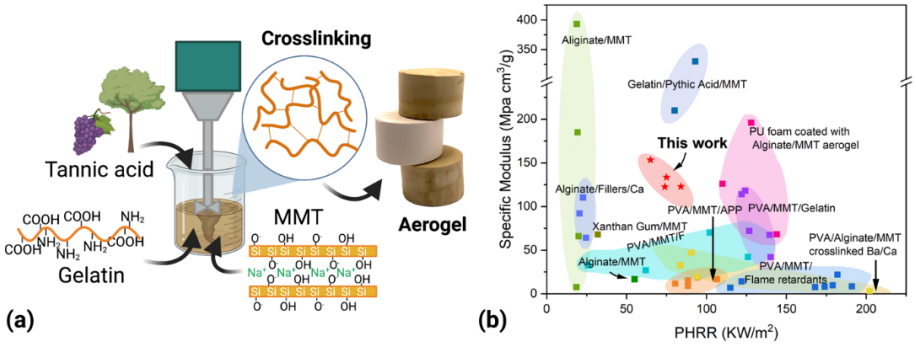

Figure 1: Schematic illustration of (a) Aerogel preparation and the obtained monoliths (b) A comparative chart of specific modulus versus the peak of heat release rate (PHRR) for different polymer/MMT clay aerogels.

Source: own.

Acknowledgements

The authors acknowledge the financial support of this work given by the Ministerio de Ciencia e Innovación (Spain) through the project "UpECOBLENDS" with reference PID2019-106518RB-I00. The authors also thank the Generalitat de Catalunya for the Grant 2021SGR01042. Lucia G. De la Cruz acknowledges the Grant (No. 729201) received from the Consejo Nacional de Ciencia y Tecnología CONACYT (México). The Aerogels Cost Action CA18125 is also acknowledged.

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Novel Production Process of Sterile Bioaerogels Using Supercritical CO_2 Technology

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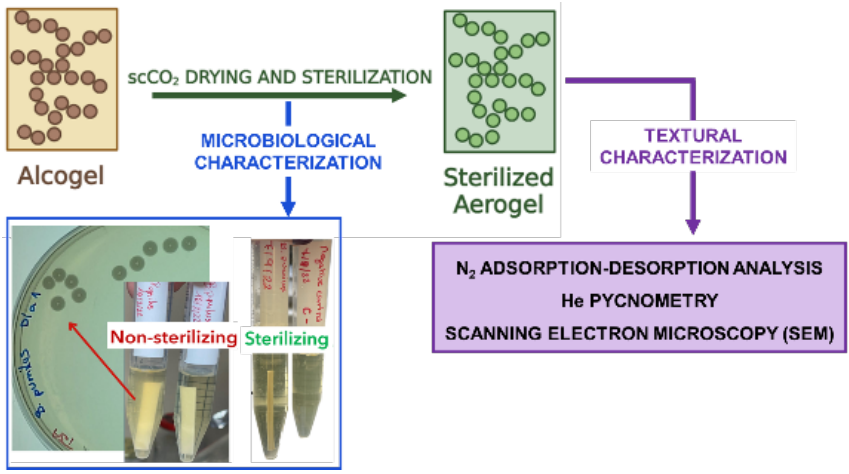

Figure 1

The numerous sources for aerogels and the possibility of getting personalized morphologies enable these materials for a broad range of applications from aerospace engineering to biomedicine. For the latter field, the high bioactivity, biocompatibility and biodegradability of aerogels made from natural polymers (bioaerogels) are interesting properties for grafting, drug delivery or wound healing purposes. Despite these outstanding material properties, the main handicap is the difficulty to obtain these materials under sterile conditions, and thus guarantee the safety levels to avoid life-threatening infections, while maintaining their future functionality. Current hospital sterilization techniques (steam, gamma radiation, ethylene oxide -EO-) cause severe changes in the physicochemical properties of aerogels, or result in the presence of toxic residues after the process. Supercritical carbon dioxide ($scCO_2$) was proposed as an environmentally friendly and efficient sterilization technique, which works under mild working conditions and leaves no residues in the final product [1].

In this work, sterile aerogels of two different biopolymers, starch and alginate, were obtained using $scCO_2$ technology in two different morphologies, monoliths and beads, respectively. The treatment was characterized microbiologically using standardized bioindicators of the steam sterilization (*Bacillus stearothermophilus*) and EO and dry heat sterilization (*Bacillus atrophaeus*) techniques to verify the effectiveness of the sterilization. In addition, commercial strips of *Bacillus pumilus* spores, that are used to monitor the efficiency of radiation sterilization, were also used, since this microorganism is recognized as the most resistant for this sterilization technique [2] After supercritical treatment, the spore strips were seeded in trypticase soy broth and trypticase soy agar culture medium. The conditions were considered sterile after 7 days in the absence of microbial growth. On the other hand, the physicochemical properties of the aerogels and sterilized aerogels were characterized by N_2 adsorption-desorption tests, helium pycnometry and Scanning Electron Microscopy (SEM), to evaluate any relevant changes in the nanostructure. The results show an effective sterilization of the material after the supercritical treatment, and varying physicochemical changes, depending on the polysaccharide source, always within the accepted values for their consideration as aerogels.

Acknowledgements

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Testing of the Acoustic Properties of Cellulose Aero- and Cryogels

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Nowadays, noise reduction has been known as a huge priority, and the necessity to develop advanced materials is causing concern. In addition to common materials used for noise reduction, such as fibrous and nanofibrous materials, great attention is being devoted to different composite materials, such as organic/inorganic ones. [1] A special class of advanced porous materials, i.e. aerogels, can be prepared from inorganic matter, polymers, as well as inorganic/organic composites. Aerogels, as open pores solid networks with very high porosity, specific surface area, and containing mesoporous and small macropores, display very high thermal insulation properties, and are appropriate for different applications, such as catalysts, catalyst supports, etc. [2] Moreover, materials with these properties could potentially serve as very efficient sound absorbers, increasing all three known sound attenuation mechanisms. [3] The mechanisms of sound absorption differ in the aerogel samples significantly from the already highly investigated bulk systems, where pore sizes and

specific surface area could be correlated with the absorption mechanism and other properties of the samples. [4]

In this work, special emphasis is given to bio-aerogels which are biomass-based, i.e. cellulose-based. This type of aerogels is very attractive, as it promotes concepts of renewable, widely available, non-toxic, and biocompatible materials. Their preparation does not involve any toxic components which makes bio-aerogels environmentally friendly. Specifically, different series of samples were investigated: The ones prepared using NaOH solvent with different ratios of microcrystalline cellulose (MCC), and the ones prepared by adding foaming agents (i.e. $Na_2SO_4 \times 10H_2O$) in order to gain different pore distribution. The samples were dried by supercritical CO_2 drying and were compact. Thus, the morphology of the samples, as well as the specific surface area, were analysed by SEM and BET. The samples were shaped to fit an impedance tube to be tested as sound absorbers. Frequency sweeps were performed, and data were recorded from 500 to 6000 Hz. Additionally, the samples mixed with sodium sulphate had better sound insulation properties, i.e. the preparation procedure impacted the pore size.

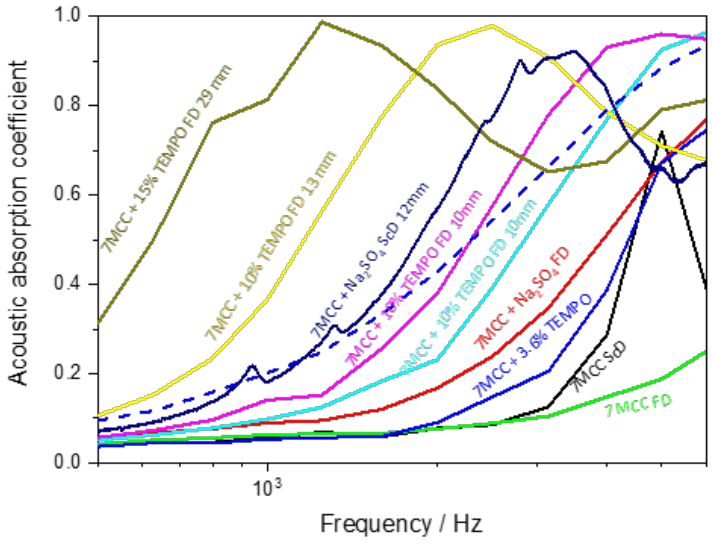

Figure 1: Acoustic absorption coefficient of the prepared samples.
Source: own.

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Biomimetic Silk Macroporous Materials for Drug Delivery Obtained via Ice Templating

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Silk from *Bombyx mori* is one of the most exciting materials in nature. The apparently simple arrangement of its two major components—two parallel filaments of silk fibroin (SF) coated by a common sericin (SS) sheath—provides a combination of mechanical and surface properties able to protect the moth during its most vulnerable phase, the pupal stage. Here, we recapitulate the topology of native silk fibers, but shape them into three-dimensional porous constructs using an unprecedented design strategy. We demonstrate, for the first time, the potential of these macroporous native silk foams as dermal patches for wound protection, and for the controlled delivery of Rifamycin (Rif), a model antibiotic.

The constructs produced here were obtained via a four-step process (Figure 1): *i) Bombyx Mori* silk degumming; *ii)* Ice-templating of an aqueous SF solution, ensuring a controlled anisotropic macroporous structure; *iii)* Methanol (MeOH)-induced enhancement of the β-sheet domain content of SF; and *iv)* Coating of SF with an SS

sheath to yield a reconstructed 3D macroporous silk material. [1] The SS@SF core-shell system obtained represents a suitable 3D cage to encapsulate and deliver Rifamycin (Rif), a bactericidal hydrophilic molecule that works by binding to—and inhibiting—the DNA-dependent RNA polymerase.

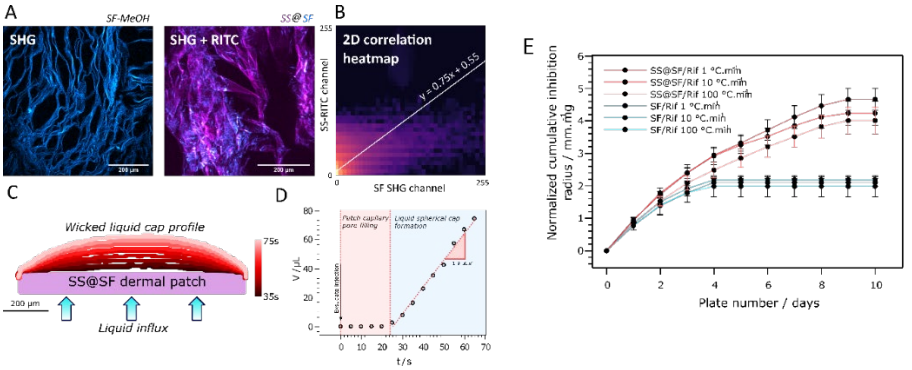

Figure 1: (A) Confocal microscopy of MeOH treated SF (left panel) and SS@SF (right panel) foams fabricated at 10 °C.min^{-1}. Scale bar, 100 µm. The blue channel was obtained from the second harmonic generation (SHG) signal from the β-sheet domains in the SF. The magenta channel codes for SS grafted with RITC dye. (B) 2D correlation heatmap between the blue (SF) and magenta (SS) channels. (C) Profile of the water cap above the SS@SF patch, obtained at 10 °C.min^{-1} in wicking experiments. (F) Wicked water cap volume as a function of time. (E) Kirby–Bauer test of foams of SS@SF/Rif (red tones) and SF/Rif (blue tones), prepared at 1, 10 and 100 °C.min^{-1}, showing the cumulative inhibition zones' radius at each plate. The blue and red lines represent the release from SF/Rif and SS@SF/Rif, respectively. The darker lines correspond to foams prepared at 1 °C.min^{-1}, the intermediate lines to 10 °C.min^{-1} and the lighter lines to 100 °C.min^{-1}. The lines between the experimental data points are exclusively a guide to the eye.

Source: own.

Ice templating, a technique initially developed for the elaboration of macroporous ceramics, has recently been finding increasing relevance in the development of biomaterials, due to the ability to tailor the texture of biological materials [2,3] without inducing denaturation. [1] To support this approach we have performed extensive characterization of the textural (SEM and confocal microscopy), spectroscopic (FTIR), mechanical (compliance in dermal stretch equivalent), liquid transport (wicking experiments) properties, to highlight its relevance in the field of cControlled delivery of active principles. We have further described the release properties of Rifamycine, both in solution and in aKirby Bauer setup, showing the ability of each 1 mm-high patch to SS@SF patch reported here can accommodate a

liquid exudate rate close to 170 $mL.cm^{-2}.day^{-1}$ (1.9 $\mu L.s^{-1}$ per sample surface area, ca. 0.95 cm^2), equivalent to over 60 days of exudate production. This liquid wicking capacity comforts the long-term application of the SS@SF patches, as determined by the antibiotic release studies that suggests their active drug release for 9 days.

This new approach may extend to designing other biomaterials with application in the clinical context requiring a combination of liquid transport, biocompatibility, and controlled release properties, but could virtually also be of pivotal interest for many other areas where fluid transport is critical.

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Unlocking New Possibilities: The Synergy Between Ionic Liquids and Porous M0aterials

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Currently, atmospheric levels of carbon dioxide have surpassed the 400 parts per million threshold. This gas, which is generated predominantly by human activities, is the primary cause of anthropogenic global warming. As a result, there is a pressing global need for CO_2 capture (CC) to mitigate its harmful effects. Traditional CC methods typically rely on solvent scrubbing and chemical absorption of CO_2, but they have several drawbacks, including solvent evaporation, formation of corrosive byproducts, and high energy consumption during regeneration. Therefore,

optimized alternatives are required. Given the significant amount of CO_2 emissions worldwide, the energy required for recycling capture materials plays a crucial role in the overall efficiency and cost of the process. [1].

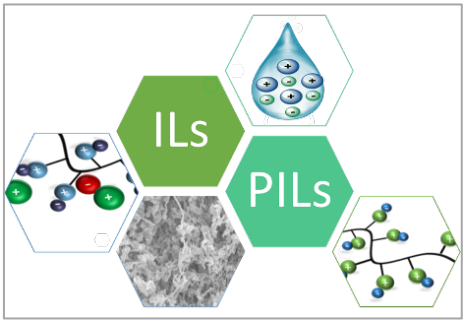

Figure 1: From ionic liquids to porous materials
Source: own.

Ionic liquids (ILs), which are organic salts with melting points below 100°C, have emerged as a promising option for CC, due to their stability and high selectivity for CO_2 absorption. These versatile materials can be manipulated to exhibit a range of properties, and have found numerous applications, from reaction media to catalysis, electrochemistry and tissue engineering. Nuclear Magnetic Resonance (NMR) has proven to be an effective tool for tailoring ILs, providing atomic resolution and dynamic information simultaneously [2]. The search for improved IL systems has led researchers on a journey from optimizing IL properties for CC to exploring polymeric systems that combine the unique characteristics of ILs with macromolecular frameworks- polyILs (Fig.1). This pursuit has culminated in the development of aerogels derived from polyILs (AeroPILs) that are capable of capturing and converting CO_2 [3-5].

Herein, we present our latest results on the use of ionic liquids and ionic liquid derived materials towards CO_2 capture and conversion using an NMR guided approach that includes solution and solid-state techniques, and give an overview of the several ongoing applications. The design of both the chemical structure and morphology of these materials represents a significant step forward in the quest for effective solutions.

Acknowledgements

We are thankful for financial aid from the FEDER funds through the COMPETE 2020 Programme and National Funds through the Portuguese Foundation for Science and Technology (FCT) in the scope of Projects LA/P/0037/2020, UIDP/50025/2020 and UIDB/50025/2020 (for i3N), PTDC/QUI-QFI/31508/2017, and PINFRA/22161/2016 (for PTNMR), Xunta de Galicia [ED431C 2020/17], MICINN [PID2020-120010RB-I00], Agencia Estatal de Investigación [AEI]. MZ thanks the European Union's Horizon 2020 Research and Innovation Orogram under the Marie Skłodowska-Curie Grant Agreement Nº101026335, RVB and MCC acknowledge FCT for the SFRH/BD/150662/2020 PhD Fellowship and the Researcher Contract (2021.03255.CEECIND), respectively. Work was carried out partially in the frame of COST Action "Advanced Engineering of aeroGels for Environment and Life Sciences" (AERoGELS, ref. CA18125), supported by COST (European Cooperation in Science and Technology)."

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PGX-Technology: Bioaerogels and Exfoliated Nanocomposites for Biomedical Applications

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A novel process for generating bio-aerogel powders, fibrils and composites over a range of morphologies based on a disruptive patented technology is presented [1,2]. The technology is called Pressurized Gas eXpanded liquid (PGX) Technology, and utilizes supercritical CO_2-expanded ethanol (the PGX fluid) to precipitate polymers from aqueous solutions, while also removing the water from the formed open-porous morphology. PGX Technology can be used to process, in a single step in one vessel, one or several temperature-sensitive, high-molecular-weight, water-soluble biopolymers quickly into high-value, ultra-light, open-porous, homogenous, fine-structured polymer aerogels [3-8] (Fig. 1) on a semi-continuous basis.

In PGX Technology processing an aqueous polymer slurry or solution is injected, together with the PGX fluid, typically at 40°C and 100 bars, through a coaxial nozzle into a collection chamber. The polymer is precipitated rapidly, and the water is removed with the PGX fluid by changing its composition gradually while avoiding the two-phase region.

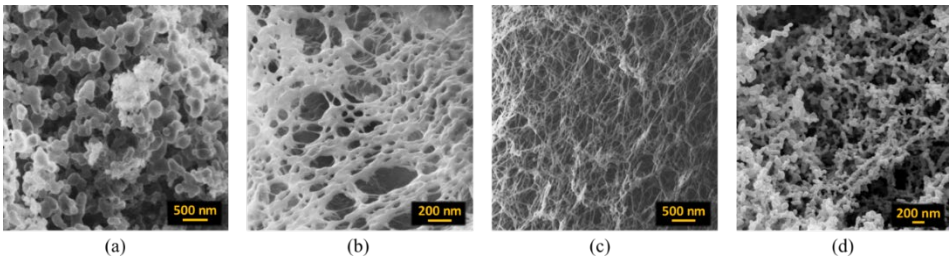

Figure 1: Helium Ion Microscope images of aerogels produced by the PGX Technology. (a) Gum Arabic (65 m^2/g), (b) Oat β-glucan (24 m^2/g), (c) Sodium Alginate (164 m^2/g), (d) Corn Starch (17 m^2/g).

Source: own.

The PGX Technology also facilitates the generation of novel bioactive aerogel composites. For that purpose, aqueous polymer solutions or suspensions can simply be mixed prior to PGX processing at any polymer ratio, leading to the generation of different morphologies and composites, such as intertwined fibrils (Fig. 2a) or exfoliated nanocomposites (Figs. 2b, 2c). PGX aerogel fibrils and composites can be shaped into flat sheets or thin strips (Fig. 3, left), maintaining the porous structure (Fig. 3, right), and loaded with bioactives utilizing adsorptive precipitation [3] for generation of novel nutraceuticals [4,6], drug delivery, or wound healing applications [7]. While PGX Technology does not require cross-linking, nor gelation of polymers to generate porous structures, this can also be implemented to form hydrogels with tunable drug release profiles [7].

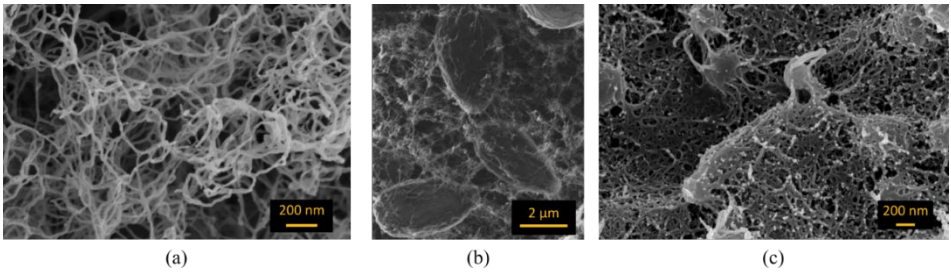

Figure 2: PGX processed aerogel composites: (a) Sodium Alginate and Pectin, (b) Sodium Alginate and Yeast Beta Glucan ghost cells, (c) Nanocrystalline Cellulose and Polyhydroxybutyrate.

Source: own.

Figure 3: Fast dissolving oral strips (size 2x3 cm) made from PGX Sodium Alginate fibrils, (100 to 200 m^2/gram) – the shown strip has 5 m^2 - (left). The open-porous structure is maintained in the compressed strip (right).

Source: own.

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Development of Ionic Liquid Functionalized Silica Xerogels as an Amine-containing Adsorbent for the Fixed Bed CO_2 Adsorption

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Increasing environmental problems and distortion of ecological balance make the sustainable approaches inevitable. Especially CO_2, types of gases like CH_4, N_2O, O_3, and CFC, are known as greenhouse gases, and are released to the atmosphere through activities such as combustion, industrial emission, or anaerobic decomposition. Global emissions of CO2 show a constant increase at an accelerating rate, resulting in the concentration of atmospheric CO_2 reaching 407 ppm in 2018 compared to 280 ppm in preindustrial times [1]. In the special report by "The Intergovernmental Panel on Climate Change" (IPCC), this excessive CO_2 emission is considered as the major source of the greenhouse gases that causes various environmental problems due to global warming and climate change [2]. Hence, there is a crucial need to develop new strategies to reduce CO_2 emissions. Currently, CO2 capture and storage (CCS) is regarded as an important technology for the mitigation of CO_2 emissions [3-5].

The present work aims to develop an adsorption-based process using ionic liquid functionalized silica xerogels to capture CO_2 from post-combustion gas streams. For this purpose, silica xerogels were synthesized by following a one-pot sol-gel method using (3-Aminopropyl) triethoxysilane (APTES) as an amine source, along with the conventional silica precursor of tetraethyl orthosilicate (TEOS). To enhance the CO_2 capture performances of the prepared xerogels, room temperature ionic liquid (1-Ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide) ($EMIMTF_2N$) was also used as another amine source, due to high CO_2 solubility. The molar ratios of the amine functional silica co-precursor to the ionic liquid were investigated as a process parameter, to reveal their correlations with CO_2 sorption performance by a series of fixed bed column-mode studies (Figure 1.). Comprehensive investigations of the pore structure, morphological properties and chemical structure were also achieved, to clarify the dominant sorption mechanisms. The results have shown that, among the prepared xerogels, the sample of $TA_{0.24}IL_{0.28}$ exhibited superior adsorption behavior with a maximum CO_2 uptake capacity of 243.32 mg CO_2/g (5.53 mmol/g) and fast kinetics (90% of its capacity achieved within an early two minutes) [6]. The excellent sorption performance of the synthesized silica xerogels is attributed to the synergistic effect of different sorption mechanisms, either physisorption or chemisorption, arising from the diversity in the porous and molecular structure.

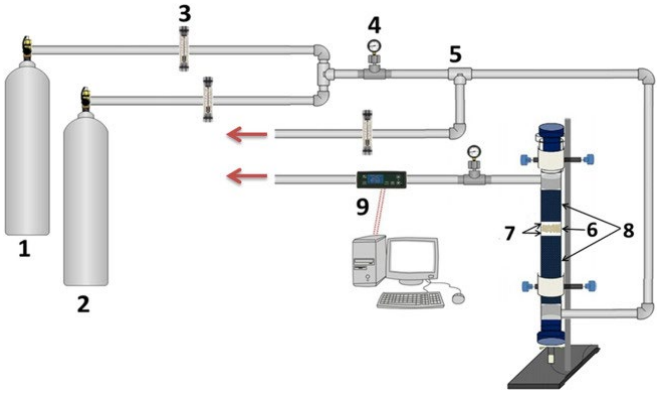

Figure 1: Schematic diagram of a CO2 capture set-up: 1) CO2 tube; 2) N2 tube; 3) Flow meters; 4) Manometer; 5) Three-way valve; 6) Sorbent bed; 7) Glass wool support 8) Quartz bed 9) Gas analyzer with data logger.

Source: own.

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Poly(ionic liquid)-based Aerogels for CO_2 Capture and Conversion: The Ionic Liquid Tour Through the Periodic Table

RAQUEL ALEXANDRA VALADARES BARRULAS,[1] MARCILEIA ZANATTA,[2] MANUELA RIBEIRO CARROT,[3] CARLOS A. GARCÍA-GONZÁLEZ,[4] MARTA CRISTINA PARRACHO CANÇADO CORVO [1]

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The growing need for climate change solutions requires advanced CO_2 capture (CC) techniques [1]. In this work, the ideal CO_2 sorbent/catalyst was envisioned, combining porosity and high surface areas with CO_2 sorption and conversion capacity. This material was obtained by combining poly(ionic liquid)s (PILs) with chitosan aerogel structures – *AEROPILs* [2,3].

PILs arise from ionic liquid (IL) monomers, combining organic cations with organic or inorganic anions, whose properties are tunable towards the final applications [2].

In this work, the screening of the molecular interaction's trends in IL monomers and their corresponding PILs while introducing structural changes focused on elemental variations in the ionic moieties, allowed a systematic view on the influence of the Periodic Table (Figure 1). A rationalization of the structure/property relations using liquid NMR enabled the refinement of the *AEROPIL* structures. Variations in terms of atomic radius, ionization energy, electronegativity, metallic/non-metallic character were identified, and the effect of these properties on intermolecular interactions (for CC) and catalytic activity (in CO_2 reuse).

AEROPILs in the form of beads were obtained with high porosity (88.1-97.0 %) and surface areas (183-744 m^2/g), which were then applied as CO_2 sorbents and catalysts for CO_2 valorization. The maximum CO_2 capture capacity was obtained for the CHT:P[DADMA]$Cl_{30\%}$ *AEROPIL* (0.70 mmol g^{-1}, at 25 °C and 1 bar). Moreover, a higher PIL content led to a higher CO_2 sorption, making this effect more noticeable for PIL chlorides than PIL acetates [3]. Despite this being an ongoing study, *AEROPILs* showed very promising catalytic activities towards epoxides at relatively low pressure and in the absence of a solvent and co-catalyst.

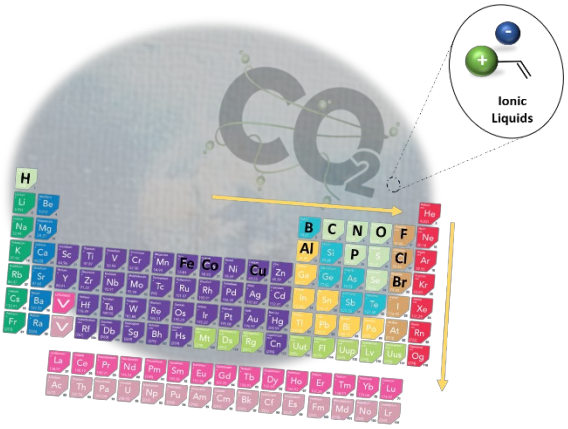

Figure 1: Graphical representation of the methodology adopted in terms of chemical elements` variations and porosity induction in the AEROPILs.

Source: own.

Acknowledgements

This research was funded by National Funds through FCT – the Portuguese Foundation for Science and Technology, through Projects: UIDB/50025/2020-2023, UID/QUI/50006/2019, UIDB/50006/2020|UIDP/50006/2020, PTDC/QUI-QFI/31508/2017, PTNMR-ROTEIRO/0031/2013 and PINFRA/22161/2016, co-financed by ERDF through COMPETE 2020, PT2020, POCI and PORL and FCT through PIDDAC (POCI-01-0145-FEDER-007688, POCI-01-0145-FEDER-007265). Work was supported by Xunta de Galicia [ED431C 2020/17], MICINN [PID2020-120010RB-I00], Agencia Estatal de Investigación [AEI] and FEDER funds. R.V.B. acknowledges FCT for the SFRH/BD/150662/2020 PhD Fellowship. M.C.C. acknowledges FCT for the Researcher Contract (2021.03255.CEECIND). M.Z. acknowledges the funding from the European Union's Horizon 2020 Research and Innovation Program under the Marie Skłodowska-Curie Grant Agreement No 101026335. This publication is based upon work from COST Action "Advanced Engineering of aeroGels for Environment and Life Sciences" (AERoGELS, ref. CA18125), supported by COST (European Cooperation in Science and Technology).

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Multipurpose Cotton-silica Aerogel Composites Fabricated with Textile Fibres` Waste

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This work describes the utilisation of textile industrial wastes as a raw material for developing reclaimed fibres-strengthened silica aerogels, and applying Ambient Pressure Drying. Ethyl acetate was also introduced as a green solvent in the manufacturing of the aerogels. Thus, the new aerogels were designed following sustainability and circular economy concepts.

Aerogels are unique materials due to their extreme interconnected porosity, putting them at the lower end of the bulk densities range for solid man-made materials [1]. This feature provides them with a remarkable insulation performance [2]. Still, they are intrinsically brittle, making their processing and handling difficult [3]. The embedment of fibres in the aerogels` matrix is recognised as an elegant and effective method for avoiding their shrinkage during drying [4]. On the other hand, considering the local industry and literature data, 15 to 20% of the fabric required for clothes` fabrication is wasted in the cutting rooms [5]. The reclaimed cotton fibres used in this work were obtained from those wasted fabrics, only through mechanical processes.

The silica aerogel composites developed here, embedded with reclaimed cotton fibres, feature a thermal conductivity as low as 21 mK m K^{-1}, a good acoustic attenuation, with a sound absorption coefficient of ~ 0.8, and moisture regulation ability [6]. The latter two characteristics were granted by the presence of the cotton fibres in the composite, thus enabling multifunctional attributes to this material.

Acknowledgements

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Adsorption of Organic Components from Fluid Mixtures on Functionalized Mesoporous Materials: Experiments and Simulation

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Inverse supercritical fluid chromatography (iSFC) is a method for characterizing the molecular interactions of dissolved substances of different polarity (mobile phase) and solids (stationary phase). Since a supercritical mobile phase is used, it is particularly suitable for the study of nonporous materials such as aerogels. The aim of this work is the first chromatographic characterization of the interactions between native and enzymatic-modified biopolymer aerogels and organic substances, to optimize loading and release processes, as well as to assess the enzymatic activity in supercritical CO_2.

Selected highly porous polysaccharide aerogels (based on alginate, cellulose and chitosan) and protein aerogels (based on potato protein and whey protein isolate) were synthesized via gelation, solvent exchange and supercritical drying, and used as stationary phases in iSFC. During the aerogel production step, the enzymatic surface modification of the biopolymer aerogels was carried out using *Candida antarctica*: lipase B (Novozym CalB L®) as a non-toxic, eco-friendly biocatalyst for industrial applications (e.g. the food industry, flavor synthesis, pharmacy and biodiesel applications) [1].

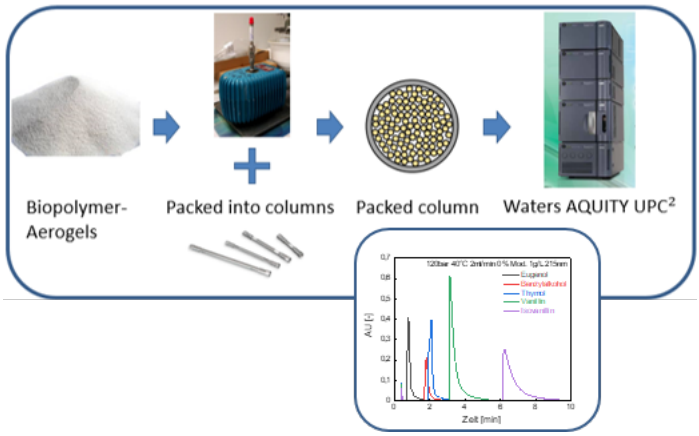

Figure 1: Preparation process and analysis of the aerogel stationary phases in iSFC.
Source: own.

Different techniques were used for immobilization of the CalB on the aerogels` surface: adsorptive immobilization was carried out in hydrogel and alcogel states by mixing wet gels with enzyme containing solvents. Covalent immobilization was carried out by the coupling of CalB via glutaraldehyde with chitosan amine groups during the hydrogel state. CalB-loaded, nanoporous aerogels were characterized (gas sorption, SEM, FTIR) and enzymatic activity after supercritical drying was determined via spectrometric measurements using a 4-nitrophenyl acetate assay.

Furthermore, this test method was transferred to iSFC, where the surface interactions and enzymatic activity of native and loaded aerogel microparticles were elucidated using 4-nitrophenyl acetate as an analyte. An interpretation of the interactions taking place between the mobile and stationary phases was carried out,

based on the experimentally determined chromatographic retention parameters (e.g. dead time, retention time, and factor) and calculated thermodynamic parameters (enthalpy of adsorption) [2].

A simulative approach was used to validate the adsorption behavior. Thereby, the influence of the packing behavior (e.g. inhomogeneity of the packing density, column porosity, diffusion behavior of the analytes) of chromatographic aerogel columns could be considered, using the Lattice-Boltzmann theory to model the highly porous column packing. Additionally, the adsorption process of the analytes within the mobile CO_2-stream phase was simulated in a cellular automata model. In general, the simulative representation allowed a broader consideration of column-specific factors, which could not be controlled or included specifically in the experimental tests [3].

Acknowledgements

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Supercritical Solvent Impregnation Technique for the Development of Antimicrobial Starch-based Aerogels

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Natural compounds found in different plant extracts can show numerous biological activities and be used as health promoters. For instance, thymol, carvacrol, citronellol, and eugenol (found in extracts of thyme, oregano, lemongrass, and clove, respectively) can be used as anti-inflammatory, antiviral, and antimicrobial agents [1-4]. The high volatility of the mentioned natural bioactive compounds (NBCs), which restricts their broader application, can be managed by the incorporation of NBCs into polymer matrices. Therefore, intending to develop devices that release antimicrobial substances in a controlled manner, this study tested the applicability of the supercritical solvent impregnation (SSI) technique for the incorporation of NBCs into starch aerogels. First, the hydrogels were prepared from corn starch. The water in the hydrogels was replaced by ethanol, and the obtained alcogels were dried using the supercritical drying (SCD) technique. The parameters of the aerogels` preparation were optimized previously [5].Later, SSI was performed at 150 bar and 35 °C for 2 h. The obtained materials were analyzed using FTIR, to determine the potential interactions between the NBCs and polymer that could determine the rate of controlled release and antimicrobial activity. Controlled release of NBCs from the aerogels was tested in phosphate-buffered saline solution at 37 °C. The antimicrobial

activity of the impregnated aerogels was tested against the Gram-negative bacteria *E. coli* ATCC 25922, Gram-positive bacteria *S. aureus* ATCC 25923, and the fungi *C. albicans* ATCC 24433, using an agar disk diffusion standard method. Freshly grown microbial cultures were diluted in a sterile physiological saline solution to obtain the inoculum with an initial number of cells of *ca.* 10^6 CFU/mL for *E. coli* and *S. aureus* and 10^5 CFU/mL for *C. albicans*. The diameter of the inhibition zones (DIZs) was determined after 24 h of incubation at 37 °C.

The SCD technique allowed the preparation of aerogels with diameters of 10 mm, density of 291 kg/m^3, and porosity of 81%. The loading of aerogels was in the range from 9.4 to 11.1%, being the lowest for thymol and the highest for eugenol. The disk diffusion test revealed that DIZs for samples impregnated with thymol and carvacrol were larger compared to samples impregnated with citronellol and eugenol (Figure 1). It was interesting to note that DIZs for *C. albicans* could be determined only for the eugenol and citronellol samples, while the zones were overlapped in the cases of thymol and carvacrol. This could be due to the smaller number of cells in the inoculum, or better susceptibility of yeast towards impregnated extracts. Additionally, it was observed that the antibacterial activity of aerogels with carvacrol, eugenol, and citronellol was the same against Gram-positive and Gram-negative bacteria, achieving a DIZ of 27, 19, and 12 mm, respectively. On the other hand, the aerogels with thymol showed stronger antibacterial activity against Gram-positive bacteria, achieving a DIZ of 55 mm.

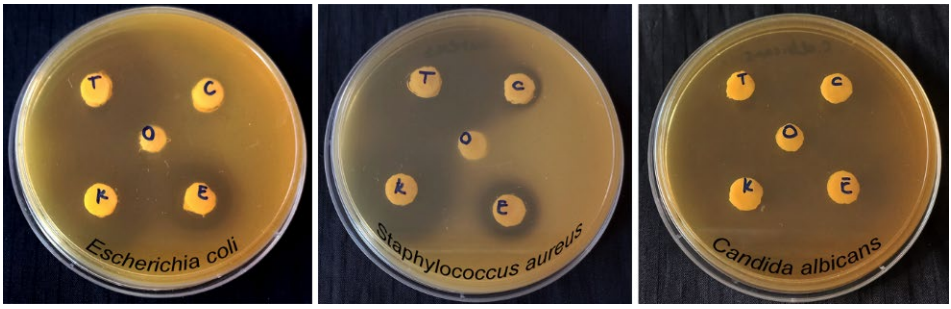

Figure 1: Antimicrobial activity of starch aerogels: neat (O) and impregnated with thymol (T), carvacrol (K), citronellol (C), and eugenol (E)

Source: own.

Acknowledgements

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Spark Ablation as a Potential Method of Metal Oxide Aerogel Formation for Energy Applications

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Nanostructured and nanoporous metal oxides have been showing, for nearly a decade, great proof of concept application in green energy [1]. However, most of the methods to produce nanostructured materials aren't scalable, or their scale-up requires a very large amount of toxic solvents and rigorous procedures [2]. By utilizing spark plasma, through careful disintegration of an electrode, one can produce stable nanoparticles of primary sizes as small as <5 nm, which is well below the quantum confinement range [3,4]. This work investigated the formation of porous metal oxide thin films for photovoltaic energy conversion and photocatalysis. Out of the potential candidates, titanium oxide has shown to be the most prominent material for metal oxide aerogel formation. Microscopy revealed the existence of self-assembled, highly mezo- and microporous structures, while diffraction indicated that the material varies from completely amorphous to nanocrystalline, depending on the synthesis and deposition parameters. Developing a solid state synthesis route for obtaining a low temperature titanium oxide aerogel could prove superior for photocatalytic application.

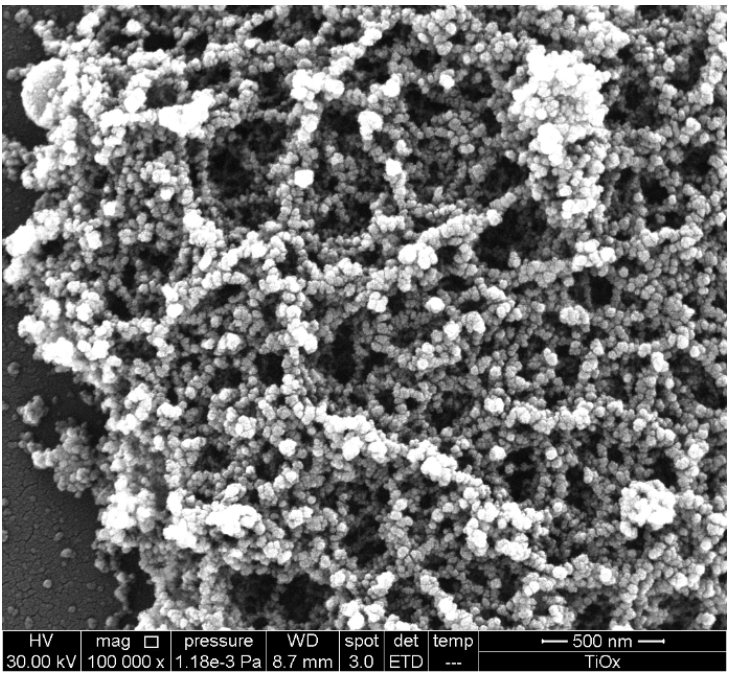

Figure 1: Self-assembled mesoporous titanium oxide powder.
Source: own.

Acknowledgements

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Reinforcement of MTMS/PDMS Silica Aerogel with Modified Nano Fibrillated Cellulose

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Silica aerogel is an outstanding lightweight insulation material, and it is getting scientific and industrial attention in practical applications of housing insulation.[1] Its downfall however is the price and low mechanical strength, which is why, for such applications, it is usually used together with other materials. To increase the sustainability and price of the silica aerogels, alternatives to supercritical drying such as ambient pressure drying (APD) were investigated, and when used for sodium silicate precursor were successful, but required a lengthy solvent exchange process. An alternative silicon source – MTMS (methyltrimethoxysilane) possesses the hydrophobic groups and can withstand APD directly after gelation, with no additional steps, which is beneficial, as this advantage decreases the production time from days to hours. The extreme brittleness of MTMS aerogels hinders their use in real life applications, and this research focuses on investigating a potential solution. Prior research attempted reinforcing such aerogels with Nano Fibrillated Cellulose (NFC) [2] ,and an increase was achieved in mechanical strength. This study focuses on bonding the NFC chemically by means of modifying it first with silane functions.[3,4] Such incorporation (shown in Figure 1) is expected to improve the aerogel strength further.

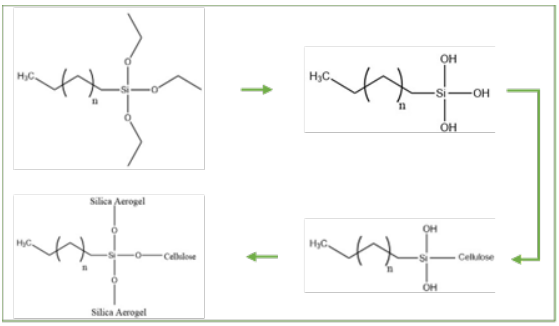

Figure 1: Reinforcement of silica aerogel with modified NFC

Additionally, other parameters were investigated, to optimize the properties of the resulting aerogel – most importantly, increasing the strength without a significant increase in thermal conductivity. The amount of cellulose, solvents, aging time and others were varied, and the product was tested to find the most efficient protocol. Environmental considerations were also taken into account, so no harsh solvents or surface modificators were utilized, and the APD method was used.

Acknowledgements

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Design of Agarose-based Aerogels with Potential Application as Wound Dressings

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In recent decades, many kinds of wound dressings have been developed successfully. Aerogels-based dressings not only have highly porous network, but also possess adjustable surface properties and tunable pore size, which make them promising candidates for wound healing [1-2].

Considering that an ideal aerogel should be nontoxic, biocompatible and biodegradable, agarose is a good option, and has been studied intensively as a constituent for aerogels` fabrication [3].

Agarose is a polysaccharide that is derived from seaweed (red algae). It has great biocompatibility, as well as non-immunogenic properties, making it a good choice for use in wound dressings [3]. Agarose has an adjustable ability for adsorbing water, giving cells an ideal milieu for biological activity.

In this work, we propose the preparation of an agarose-based aerogel as wound dressing material. The aerogels were prepared from the co-assembled mixture between agarose (AG) and poly(itaconic anhydride-co-3,9-divinyl-2,4,8,10-tetraoxaspiro (5,5) undecane) (PITAU), a copolymer with an antioxidant character, followed by the vacuum freeze-drying technique to obtain the aerogels. The materials based on AG and PITAU had a better thermal stability when the percentage of PITAU increased. The sample with a ratio AG/PITAU of 1:1 (AG_PITAU_1_1) presented the best thermal stability, due to the greater number of physical bonds like hydrogen or Van der Walls type, which are manifested between the two constituents.

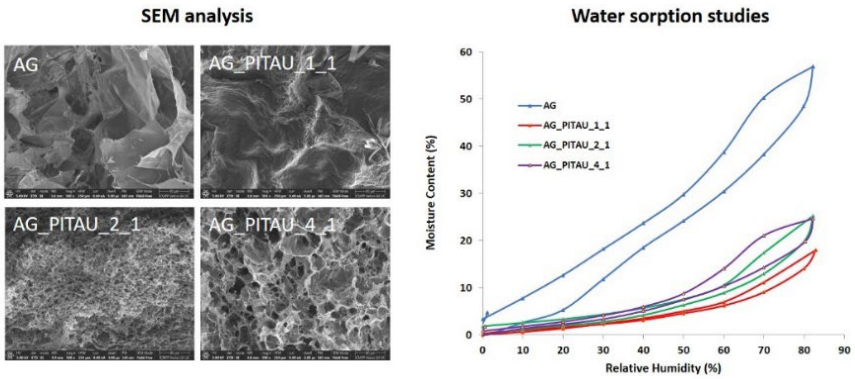

Figure 1: SEM images (left) and water sorption graphs for AG_PITAU aerogels (right).
Source: own.

The effects were investigated of the compounds` ratio on the dynamic vapor sorption capacity of the aerogels (Figure 1). The sorption kinetic studies revealed that the associated isotherms for each material had a hysteresis that can be interpreted as being characteristic to porous surfaces. As compared to AG, each aerogel had a lower BET surface area and a larger average pore diameter. The SEM and porosity results revealed that the increase of PITAU amount within the aerogel matrix led to a decrease in the pore dimensions of the aerogel material, due also to

the greater number of intervening physical bonds. These properties are useful in the process of loading and controlled release of bioactive extracts. Therefore, all the results indicated that the new agarose/PITAU aerogels are promising materials for wound management.

Acknowledgements

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This work was supported by a Grant from the Ministry of Research, Innovation and Digitization, CCCDI - UEFISCDI, Project number PN-III-P2-2.1-PED-2021-2229, within PNCDI III.

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Synthesis of Gold Nanoparticles and Their Immobilization in Aerogels

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Nanotechnology has become a rapidly developing, promising field of research in the last few decades. Metal nanoparticles have great importance, due to their unique optical, physical and chemical properties compared to bulk materials. Metal nanoparticles function as beneficial catalysts in chemical reactions, but their extraction from reaction mixtures for further use or regeneration is not elementary. Furthermore, unsupported metal nanoparticles form aggregates easily and are prone to sintering, which leads to the formation of larger particles and reduces their catalytic performance. Thus, supported nanoparticles are preferred for catalytic reactions [1]. Due to their structure, aerogels are suitable for forming nanocomposites with metal nanoparticles. The immobilization of metal nanoparticles in a chemically resistant aerogel matrix offers the possibility of combining the advantages of an immobilized catalyst and a highly porous and easily permeable solid support.

Both theoretically and practically, gold nanoparticles are in the center of interest, as a wide variety of nanoparticles can be synthetized and studied concerning size and shape [2]. During our research gold nanoparticles were prepared via Turkevich synthesis [3], which has recently gained significant attention, as it is considered to be a simple and effective method resulting in gold nanoparticles with a diameter of

approximately 10 nm. In this bottom-up technique chloroauric acid is reduced and stabilized by sodium-citrate.

Gold nanoparticles were added to the reaction mixture during the sol-gel synthesis, so they are present during the hydrolysis and polymerization processes that ensure gelation. The advantage of this method is that the nanoparticles can be distributed evenly in the matrix and their quantity can be determined easily, however the stabilizer of the nanoparticles can influence gelation. During our experiments it was found that the conditions of the sol-gel synthesis led to rapid aggregation of citrate-stabilized nanoparticles, and, thus, to the loss of their catalytic activity [4]. During our further studies we found that organic solvents and atmospheric CO_2 have a significant effect on aggregation, but these factors cannot be excluded completely under the standard synthetic conditions of aerogels. As a result of testing several stabilizing agents, polyvinylpyrrolidone (PVP) proved to be suitable under the conditions of sol-gel synthesis and drying with supercritical CO_2. In the course of our research we developed synthesis methods for nanogold-containing silica (Figure 1.) and alumina aerogels, during which the nanoparticles aggregated to a negligible extent. The catalytic activity of the as-prepared aerogels was tested by sodium hydroborate reduction of p-nitro phenol to p-amino phenol. P-Nitrophenol is an environmental pollutant in water, and can cause significant environmental and health risks. The catalytic model reaction was carried out at room temperature, and was monitored by UV-Vis spectroscopy. The details of the studies of catalytic activity will be presented on the poster.

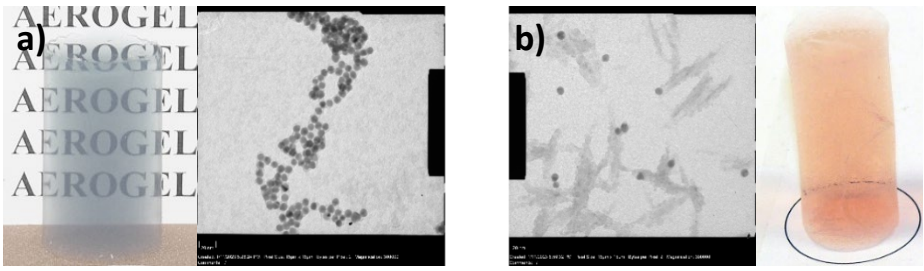

Figure 1: Gold nanoparticle containing silica aerogels with TEM pictures of (a) Aggregated, and (b) Non-aggregated gold nanoparticles

Source: own.

Acknowledgements

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3D-printed and Vancomycin Loaded Aerogel Scaffolds for Advanced Bone Tissue Engineering

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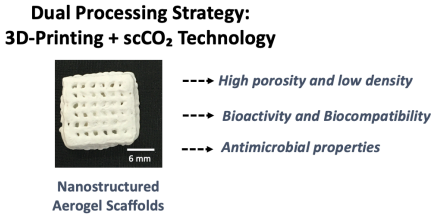

Figure 1
Source: own.

Aerogel technology endow biomaterials with the nanostructuration, hierarchical structure, and dual porosity essential to stimulate bone regeneration [1]. 3D-printing is also a powerful tool employed in regenerative medicine to achieve the

personalization of structures. A dual processing strategy combining both techniques was recently proposed for the manufacturing of 3D-printed alginate scaffolds for bone tissue engineering [2]. Nevertheless, bone defects are commonly accompanied by microorganism colonization, caused mainly by *Staphylococcus aureus,* and leading to infection and inflammation lesions in the damaged area [3]. For this reason, drug-loaded implants are being developed to provide local antibiotic concentrations at the wound place [4].

In this work, methylcellulose (MC)-hydroxyapatite (HA) aerogels were loaded with vancomycin (VAN) and manufactured by the dual processing strategy, combining 3D-printing and supercritical CO_2 technology. The textural characteristics (BET and SEM analysis), biological properties (cell and antimicrobial tests), and mechanical performance (DMA assay) of the aerogel structures were evaluated regarding their potential application in advanced bone tissue engineering. Aerogel scaffolds were fabricated with high specific surface area and dual porosity. The apatite depositions obtained on the MC-HA composites after 26 days suggest the achievement of the long-term bioactivity required for bone tissue engineering. Moreover, a clear inhibition zone was detected for VAN-loaded aerogels after contact with *S. aureus,* indicating an effective antimicrobial effect. Compressive modulus of the scaffolds showed an increment in the mechanical performance with the increasing HA content. Overall, MC-HA VAN loaded aerogel scaffolds are promising candidates for personalized bone tissue engineering and bone infection treatment simultaneously.

Acknowledgements

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Upscaling of Cellulose Aerogel Sheets from Hemp Fibers

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The aim of this work was to produce cellulose aerogel sheets from hemp-extracted cellulose by using the CaProLi® (**C**ellulose **A**erogel **P**roduction **L**ine) as an up-scaling device. Cellulose aerogel sheets have the potential to be used as a sustainable thermal insulation material [1]. Furthermore, they can be used as a material for wound dressing and healing [2]. Two different commercial celluloses were used as references, and compared with the samples prepared from hemp fibers. In addition, they were used to investigate a useful viscosity range for the upscaling process. Suitable recipes were prepared by the addition of sodium salts. Cellulose was dissolved in a mixture of water, NaOH and urea. The samples were dried supercritically with CO_2, after neutralization and solvent exchange to ethanol. The aerogels were characterized for their porosity, morphology and internal structure (BET, SEM, total pore volume and pore size distribution). Additionally, the influence was investigated of the sodium salt addition on the gelation time. The cellulose extracted from the hemp presented good quality, and the hemp cellulose aerogels showed the lowest envelope density and therefore the highest porosity of all the cellulose aerogels. The nitrogen adsorption-desorption analyses showed BET surface area values in the range of 194-288 m^2/g. The effects on the reduction of the gel point in the cellulose solution were also observed with the addition of sodium

salts. A continuous production process of cellulose gel sheets was established (conveyer belt speed of 0.56 cm/s) which is an important step in the up-scaling of this technology.

Acknowledgements

This project has received funding from the European Union's Horizon 2020 Research and Innovation Program under Marie Skolodowska-Curie Grant Agreement ID: 956621. It is carried out in a partnership between the German Aerospace Center and the industrial partner KEEY Aerogel, Habsheim, France.

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Application of Different Drying Techniques to Turn Pea Waste Into Aerogel-like Ingredients with Tailored Functionalities

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A pea is an agricultural commodity belonging to the Leguminosae family, and one of the most important nutritional crops grown across the World and the EU. When green peas are processed into food products, about 15% (w/w) of them are discarded due to substandard properties (yellow color, the presence of stains, substandard size). Substandard green peas represent up to 0.135 million tons/year in the EU only, and are nowadays subjected mainly to anaerobic digestion for the production of biogas [1], resulting in the loss of valuable compounds such as fiber, sugars, and proteins. Wasted peas have the same composition as the fresh ones, which are rich in proteins (15.3-21.9%) and fibers (10.4-30.7%). Different strategies have been proposed to turn this waste into value-added fiber or protein-rich powders intended for food applications. However, the pea protein fractionation requires the disruption of the pea matrix, the recovery of the protein fraction, and its final drying. The process is, thus, particularly energy-intensive and water-

consuming, besides leading to not negligible plant residues, which contribute negatively to the environmental impact [2].

Thus, the development of technological interventions for pea waste recovery and transformation enabling them to be up-cycled into food ingredients with tailored functionalities is in high demand for increasing the value-chain of this commodity [3]. A possible solution is represented by the transformation of pea waste into value-added fiber and protein-rich powders. As is well known, different drying techniques can be applied to this aim today. However, remarkable changes of powder functional attributes are expected, depending on the drying technique applied [3,4].

Air-drying based on the contact of wet materials with a hot air flow is the most commonly applied food drying technique and has limited investment costs. However, its application is associated with material shrinkage, hardness, poor appearance, reduced ability to rehydrate and bioactive loss. Contrarily, the application of freeze-drying produces high-quality dried products, due to water removal by sublimation of the ice crystals, but with higher investment costs [3]. Novel drying techniques, such as supercritical-CO2-drying, have been claimed to increase the environmental sustainability of traditional drying processes, generating highly porous aerogel-like materials with novel functionalities in comparison to air-dried or freeze-dried products [4].

Based on these considerations, the aim of the present study was to compare the structural and technological properties of pea waste turned into dried powders via traditional (air- and freeze-) and novel (supercritical-CO2-) drying techniques. The samples were analyzed for their composition, physical properties (color, bulk density, particle size distribution), microstructure by SEM and interactions with food fluids (solubility, water and oil holding capacity, wettability). Their sensory properties were also assessed finally.

The results demonstrated that the drying technique affected the pea powder composition mainly in terms of lipid and carbohydrate content. This was attributable to the ethanol and CO2 removing capacity of soluble compounds. Beside affecting the chemical composition, the drying technique impacted the bulk density and microstructure of the pea powders. The bulk density decreased in the order AD>FD>SCD, so that the SCD powder maintained mostly the original

microstructure of the pea, being potentially regarded as an aerogel-like material. The differences in the chemical and physical properties of the pea powders obtained upon the application of the different drying techniques also modified the ability of the pea powder to interact with food fluids. In particular, the water and oil holding capacity increased progressively, moving from AD-FD, and, finally, SCD samples, with a completely different wettability. Thus, large amounts of different fluids could easily be entrapped into the pores of the SCD material. Finally, the drying technique also affected the macroscopic properties of the samples. In particular, the SCD sample lost the typical characteristics of green peas in terms of color and flavors. In fact, it resulted as a white powder without the typical pea flavor.

In conclusion, the remarkable effect was confirmed of the drying technique on the chemical, physical and structural properties of pea powders. Among the drying techniques applied, a particularly interesting result was the ability of SCD to produce porous, colorless and taste-free aerogel-like powders, with improved solvent loading ability. All these features could enlarge the possible applicability of these powders into foods.

Acknowledgements

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Graphene-based Aerogels for Electromagnetic Applications

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The use of foams as electromagnetic interference (EMI) shielding materials has been gaining traction in recent years, due to their unique properties. Aerogels possess density orders lower than the bulk polymers that are used widely for composite materials` preparation. Lightweight, high performance, and the ability to tune EMI shielding effectiveness (EMI SE) [1] makes such materials desirable for mobile electronics, aeronautical/aerospace and military applications. The need for EMI shielding materials that absorb primarily electromagnetic energy has grown in recent years, due to the introduction of 5G technology (operates above 24 GHz). The high-level energy of these waves can cause critical EMI pollution, and absorption-dominant shielding materials can be used to avoid it [2].

In this research, the freeze-drying technique was used to make aerogels with three types of nanocarbon fillers, graphene (GR), graphene oxide (GO) and reduced graphene oxide (rGO). Each of these fillers were combined with two polymer types, polyethylene oxide (PEO) and polyvinylalcohol (PVA). Additionally, two samples containing GR were also created, as well as a sample comprising just rGO without a binding polymer.

The electromagnetic properties of the prepared composites were investigated in low frequency (20 Hz – 1 MHz) and microwave (26 – 37 GHz or Ka-band) ranges by means of an HP 4284A LCR-meter and an Elmika R400 series scalar analyzer, respectively. The properties of the investigated materials in both types of measurements were calculated by standard techniques [3,4].

The samples comprising graphene oxide (GO) and a combination of GR+PVA exhibited interesting electromagnetic properties in the investigated frequency range (Figure 1). The most noticeable electromagnetic properties were displayed by the GR+PEO mixtures and those containing rGO. The imaginary part of the permittivity was reduced slightly with the addition of a cross-linker to the GR+PEO samples, while no such effect was prominent with the GR+PVA compositions.

In order to demonstrate pronounced electromagnetic properties in the microwave region the density of an aerogel must be high. Considerable conductivity was shown by the rGO-based samples with ~300 mg/cm^3 density, which is large compared to the other investigated samples. However, the absolute values of the aerogels complex permittivity in the Ka-band did not exceed ~2 - 0.4i, which is similar to the dielectric properties of polymer nanocomposites below the percolation threshold. From another perspective, this initially low value of permittivity may be interesting for elastic aerogels, as their density can be varied through mechanical strain, thus providing a potential avenue for tuning the desired electromagnetic properties.

Comparing the conductivity of the rGO-based samples measured in the low-frequency region, it is possible to see the standard behavior of the composite material above the percolation threshold. In the measured region the absolute value of conductivity was maximal for the rGO+PEO aerogel. Such a trend can be seen in the Ka-band measurements, where the imaginary part of the rGO+PEO aerogel permittivity was higher than that of rGO+PVA.

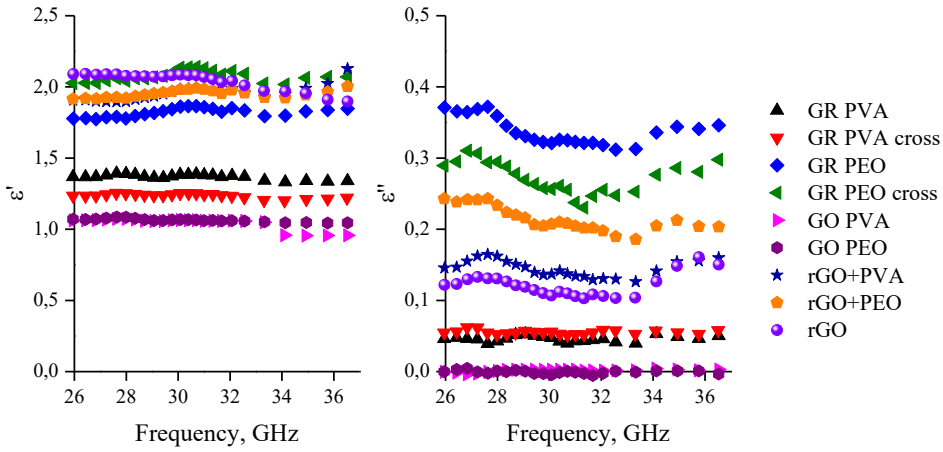

Figure 1: Complex permittivity of the investigated aerogels in the Ka-band

Source: own.

Acknowledgements

This publication is based upon work from COST Action "Advanced Engineering of aeroGels for Environment and Life Sciences" (AERoGELS, ref. CA18125), supported by COST (European Cooperation in Science and Technology)."

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Investigation of Independent Parameters for Customization Properties of Precipitated Mesoporous Silica

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Amorphous precipitated silica (SiO_2) finds wide applications in various industrial fields as a filler in plastics and rubber, an absorbent and drying powder, a substrate for catalysts, and an anticorrosion agent. It serves as an adsorbent for impurities in liquids and gases, a binder in ceramics and concrete, an anti-caking agent in the food industry [1], and an inert filling material in the polymer or pharmaceutical industries [2]. Precipitated silicas are generally produced by destabilizing and precipitating amorphous silica from soluble alkaline silicate using an acid or mineral acids [3]. This process results in the formation of primary particles that tend to associate with each other, forming numerous aggregates without agglomerating into a 3D gel structure. While alkyl orthosilicates are used commonly to prepare mesoporous silica [4], they are not preferred, due to their high cost, flammability, and handling/storage difficulties [5]. Consequently, there is a need for an economical and robust inorganic

silica source as a substitute. For industrial-scale production of mesoporous silica powders, sodium silicate (water-glass) is an excellent silica source [6].

In this work, we present mesoporous precipitated silica production and optimization using a cost-effective commercial sodium silicate solution. Controlling the properties of precipitated silica synthesized by acid additon to sodium silicate solution is a significant challenge. For this purpose, precipitated silica samples were produced and optimized according to independent parameters (temperature, pH, sodium silicate:dH_20 ratio, and acid type) with a modified precipitation tecnique. The dried precipitated mesoporous silica powders were characterized with BET and BJH nitrogen gas adsorption/desorption methods, particle size analysis, Scanning Electron Microscopy (SEM), Fourier transform infrared spectroscopy (FT-IR), density and thermal conductivity analysis. The results revealed that the BET surface area of the prepared precipitated silica increased with the increasing of temperature and decreasing of pH, while the pore size and pore volume were affected. Additionally, the particle size decreased with decreasing of the pH, and particles were produced with an average size of 17-25 μm. However, the sodium silicate:dH_2O ratio also had a critical effect on particle size and surface area, due to dilution of the reaction medium.

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Development of Oxygen-releasing Aerogel Material for Faster Wound Healing

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Wound healing is a biological process by which the body repairs the damaged tissues. It occurs in consecutive and overlapped phases: hemostasis, inflammation and proliferation, maturation or remodelling. The healing process can be impeded or interrupted by a number of factors, such as poor living habits and lifestyle, as well as different medical conditions. These conditions lead to improper wound healing, which results in chronic wounds [1]. Chronic wounds often progress to a state of pathological inflammation, accompanied by bacterial accumulation and the formation of drug-resistant microbial films. Their development is often associated with a state of hypoxia (decrease in oxygen levels) at the wound site, which can impair the healing process and lead to complications such as infection and amputation. Therefore, chronic wounds present a substantial economic burden to healthcare systems all around the World [2].

Aerogels, a class of porous materials with high surface area and low density, have shown potential as oxygen-releasing materials for biomedical applications. They can be engineered to release oxygen in a controlled manner, which can improve oxygen levels at the wound site and promote tissue regeneration [3]. Aerogels made from biodegradable polysaccharides and biopolymers are particularly suitable materials for

wound healing, due to their biocompatibility, biodegradability and high surface area [4].

Figure 1: A process scheme.
Source: own.

In our research we used a natural polysaccharide xanthan gum (Xa) and biopolymer polylactic acid (PLA) to develop oxygen-releasing materials for wound healing. Xanthan gum serves as the matrix material that encapsulates the oxygen-releasing agent, while PLA is used as a reinforcing material to improve the mechanical properties of the oxygen-releasing material.

We prepared various combinations of Xa-PLA hybrid hydrogels by combining PLA in ethyl lactate and Xa in water. The hydrogels were stored in ethanol until supercritical drying, which transformed them into aerogels. These aerogels were then characterised using nitrogen adsorption measurements to determine their surface area (BET), which reached up to 396 $m^2 \cdot g^{-1}$. The BJH method was used to determine the average pore size and pore volume in the mesoporous range. We also performed swelling and stability tests to assess the structural integrity of the materials. The results showed that the combination of xanthan gum and PLA prolonged the stability of the material in simulated body fluid (SBF) for up to 72 hours. The materials with higher PLA content showed very high SBF uptake, up to 67 times its weight, most probably due to the better stability in SBF. Oxygen generation from the aerogels was determined using the standard water displacement method, where the amount of water displaced is directly proportional to the amount of oxygen generated. The measurement was performed from 24 h up to 96 hours, and the results showed that the material released oxygen exponentially over the entire measurement time interval.

Overall, the studies suggest that biodegradable oxygen-releasing materials have the potential to revolutionise wound healing and improve the quality of life for patients suffering from chronic wounds. The development of these materials and their

clinical translation could impact the field of wound care and improve patient outcomes significantly.

Acknowledgements

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Unleashing the Potential of Graphene for CO_2 Capture

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Since the 90s the world's population has become more aware of the problem of environmental pollution. In particular, the greenhouse effect, which, combined with the pressure exerted on polluting countries in the form of Agreements (Paris Agreement, 2015), Protocols (Kyoto Protocol, 1997), and Summits, demanded for solutions to end this crisis. In this context of increasing effects of climate change and of greenhouse gas emissions, especially CO_2, as it is the most abundant anthropogenic gas in the atmosphere, research and industry have sought to mitigate and solve this problem.

Several CO_2 capture (CC) solutions have been presented over the last 30 years, with the most common approaches used in industry being based on aqueous monoethanolamine (MEA) solutions. This type of capture has several drawbacks

and limitations, such as high corrosion, the volatility of the aqueous MEA solutions, and the high energy consumption for recycling these materials [1].

Ionic liquids (ILs) are known for their ability to absorb CO_2 and for their high selectivity for this gas. In our work, we are combining graphene materials with ILs by developing poly(IL)-graphene oxide aerogels [2-4]. To develop this material, we first synthesized the respective precursors - graphene oxide, and poly(ILs). Mixtures of the precursors in different ratios were subjected to hydrothermal treatment, to obtain cryogels (Figure1). The materials were characterized using Fourier transform infrared spectroscopy (FTIR), nuclear magnetic resonance (NMR), X-ray photoelectron spectroscopy (XPS), Scanning and Transmission Electron Microscopy (SEM and TEM), and used as catalysts in CO_2 conversion. The cryogels achieved highly selective CO_2 conversion reactions (close to 100%) with remarkable yields, without the use of co-catalysts or solvents, and under low temperature, low pressures and reduced reaction times. Recycling the cryogels after CO_2 conversion is the last bottleneck, and current efforts are dedicated to optimizing this procedure.

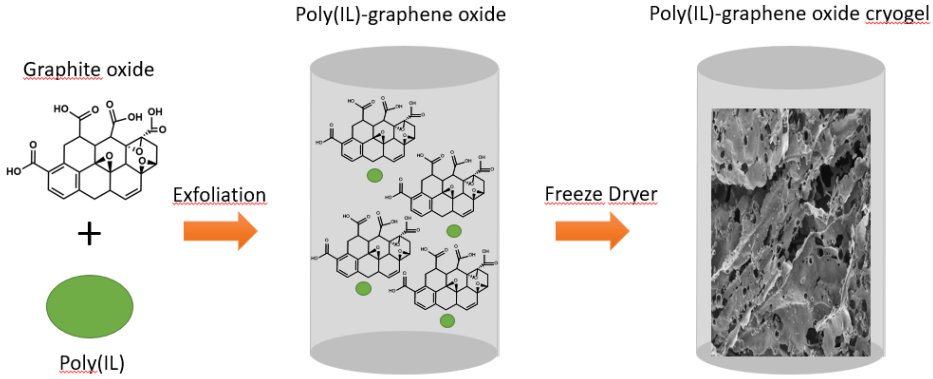

Figure 1: Scheme of the cryogel production process
Source: own.

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Physically Cross-linked Bio-based Hydrogels Prepared by Combining Carboxymethyl Cellulose with Phytic acid

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Carboxymethyl cellulose (CMC) gels have garnered significant attention in recent years, due to their biocompatibility, degradability, availability, tissue resemblance, non-cytotoxicity, and tunable properties [1]. Herein, we present a facile and eco-friendly approach for the synthesis of hydrogels by crosslinking CMC physically with a natural compound known as phytic acid (PA). PA has many hydroxyl-bearing phosphoric groups, can act as a supramolecular crosslinker, and possesses, among others, antioxidant and antimicrobial properties [2]. By incorporating it into various scaffolds, these bioactive properties can be harnessed, allowing the development of hydrogel-based materials with enhanced therapeutic potential. The structural properties, rheological performance, and morphology were investigated systematically by various characterizations. SEM images revealed a highly porous network with fine and interconnected pores. In addition, the hydrogels showed

excellent antibacterial activity against both Escherichia coli and Staphylococcus aureus bacteria. Finally, in vitro studies on fibroblast cells were employed, to assess the practical value of the hydrogel in biomedical applications. Overall, this study suggested a novel, facile and cost-effective approach to achieve a biocompatible hydrogel with favorable properties.

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Plasmonic Based Sensor for Quantification of Chemical Pollutants in Water and its Improvement by Machine Learning

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Chemical pollutants in drinking water can have many sources, such as pharmaceutical waste, agricultural runoff, and industrial discharges[1,2,3]. The development of a reliable, sensitive, and handheld sensor for the detection of a mixture of contaminants is important, both for human health and the environment. Herein, we show the development of a plasmonic sensor for Surface-enhanced Raman spectroscopy (SERS) and colorimetry measurements. Two types of plasmonic surfaces which enhance the electromagnetic field are presented here; (i) Well-defined cavities milled in silver substrates which are covered with 5 nm of SiO_2 for stability. (ii) A scalable metallic-like aerogel network with large surface area, for increasing the sensitivity of our measurements. Three different families of analytes were studied, which can be found in drinking water: Piperidine and its derivatives (Pharmaceutical waste), Dioxins & Polychlorinated biphenyls, Per- and polyfluoroalkyl substances, each of which is toxic, both to the environment and human`s health, even at a low concentration of 30 mg/Kg ($3*10^{-4}$M). Those

molecules are relatively small, and therefore their cross-section for absorption is low, something which makes it difficult for SERS based sensing. Yet, machine learning algorithms are applied, in order to improve the sensitivity and selectivity

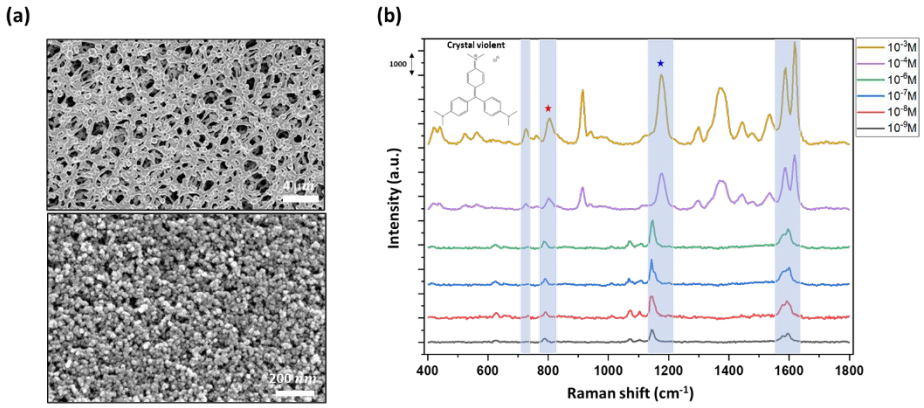

Figure 1: (a) SEM images of a metallic-like aerogel network (b) Raman spectra of Crystal violet dissolved in water in different concentrations10^{-3}M-10^{-9}M.

Source: own.

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Preparation of Scaffolds from Polysaccharide Aerogels and Supercritical Foams for Tissue Engineering

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Scaffolds from synthetic polymers, such as polycaprolactone (PCL), are biocompatible, biodegradable, structurally stable, and have a vast potential for biomedical applications, e.g., tissue engineering [1]. Scaffolds with superior properties can be fabricated using supercritical fluids. The incorporation of other components can, additionally, improve the properties of the scaffolds. Polysaccharide aerogels present attractive materials that can be prepared from renewable, sustainable, and non-toxic sources. They have been considered to have great potential for biomedical applications [2]. Polysaccharide aerogels can be an important mesoporous support for PCL in obtaining hybrid scaffolds [3].

The aim of the following work was to develop a hybrid material – scaffold made from PCL foam and chitosan aerogel beads. The chitosan aerogel beads were prepared by sol-gel synthesis and dried using supercritical carbon dioxide (CO_2). The PCL foams were made by supercritical foaming at different operating conditions (40°C/150bar and 60°C/200bar). The chitosan aerogel beads were embedded into

the foams simultaneously with the foaming process, to obtain hybrid materials, PCL-chitosan scaffolds.

The obtained aerogels, foams, and scaffolds were characterized using N_2 adsorption-desorption analysis, thermogravimetric analysis (TGA/DSC), Scanning Electron Microscopy (SEM), and Fourier transform infrared spectroscopy (FTIR). The chitosan aerogel bead sshowed a high specific surface area, namely, 528.23±7.08 m^2/g. The PCL-chitosan scaffolds had specific surface areas ranging from 2.88±0.14 m^2/g to 13.24±0.43 m^2/g, depending on the ratio of aerogel used. The TGA/DSC analyses showed improved thermal stability of the scaffolds with the addition of aerogels, while the SEM revealed the macroporous and mesoporous structure of the scaffolds. Lastly, the scaffolds were impregnated successfully (confirmed by FTIR) with the chosen model drug, indomethacin.

The prepared scaffolds showed great potential for biomedical applications, due to the combination of properties coming from both the aerogels and supercritical foams.

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Icephobic Characteristics of Organically Functionalized Silica Coatings

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In recent years the surface coatings on glass substrates have occupied severe interest, owing to their high water repellent, non-flammable, self-cleaning, anti-icing, and anti-bacterial properties. Due to the nature of the sol-gel chemistry, silica coatings can be formed on surfaces as a thin film with 3-D, porous, and amorphous structures. So, they have the possibility to be employed in various applications, such as biomedical, anti-fouling/corrosion, and thermal insulation[1–4].

In this study, a thin-film silica coating was prepared by following a one-step sol-gel method with co-precursor and/or seed growth approaches. Using tetraethyl orthosilicate (TEOS), vinyltrimethoxysilane (VTMS), and poly(dimethylsiloxane) (PDMS) as silica precursors, the thin-film layer was formed on glass surfaces, and dried under ambient conditions. In the co-precursor approach, while the silica layers are formed during the co-condensation of TEOS, VTMS, and PDMS, the amount of PDMS was varied in the sol mixture. The curing conditions were also examined as a second parameter. In the seed growth approach, on the other hand, a 3-D silica network of the co-gelated silanes was established on SiO_2 nanoparticles. The content of SiO_2 nanoparticles in the sol mixture was varied, to investigate the changes in the adhesion between the glass and silica layer and their transparency/opacity properties.

Table 1: Sol components of prepared silica layer in all parts (The coated glasses were abbreviated as TxVyPz in the first approach, where T, V and P represented TEOS, VTMS and PDMS; the subscript of x, y, and z referred to their volume ratios. Herein, the x and y ratios were constant as 1 and 1.5, respectively, and z was varied as 1.5, 2.25, and 3. Also, "-C" represents curing. In addition, the coated glasses prepared by the second approach were abbreviated as NPw-TxVyPz, where NP represented the SiO2 nanoparticles; the subscript of w referred to the amount of nanoparticles.)

Strategy	ID	Sol Components					
		SiO_2 NP (g)	Volumetric ratio based on TEOS				
			TEOS	VTMS	PDMS	EtOH	Water
Co-condensation	T1V1.5P1.5	-	1	1.5	1.5	0.5	7
	T1V1.5P2.25	-	1	1.5	2.25	0.5	7
	T1V1.5P3	-	1	1.5	3	0.5	7
	T1V1.5P1.5 -C	-	1	1.5	1.5	0.5	7
	T1V1.5P2.25 -C	-	1	1.5	2.25	0.5	7
	T1V1.5P3 -C	-	1	1.5	3	0.5	7
Seed growth with Co-condensation	NP0.5- T1V1P1	0.5	1	1	1	9.4	-
	NP1- T1V1P1	1	1	1	1	9.4	-
	NP2- T1V1P1	2	1	1	1	9.4	-

Fourier Transform Infrared Spectroscopy (FTIR) analysis was performed to define the chemical structures of the prepared surfaces. Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM) analyses were performed to determine their morphological and textural properties of the silica layers. The hydrophobic properties, which affect the anti-icing behavior directly, were revealed by Contact Angle measurements. Ice adhesion tests were conducted and the results showed that with the addition of low content SiO_2 nanoparticle obtained glass exhibited an almost anti-ice adhesion property with an adhesion break force of 5.6 N in the seed growth studies. In the co-gelation strategy, applied curing has a drastic effect on the relaxation of adhesive forces of iced structures on the surface. Increasing the PDMS content also decreased the magnitude of adhesion, due to the abundancy of the attached methyl groups on the silica structure. Probably because of increased network flexibility due to larger pore formations, the optical properties changed and the coated glass became most transparent at the highest PDMS. These promising results can be indicative for further utilization of vinyl/methyl functionalized silica coated glasses for diverse applications in the future.

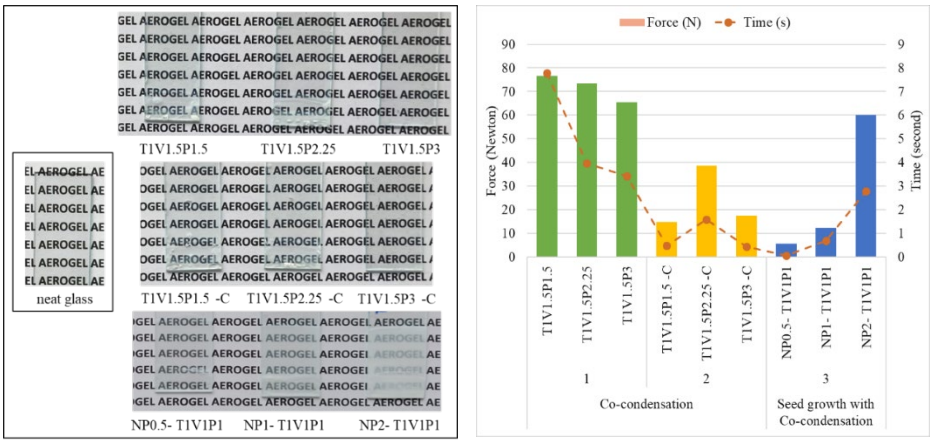

Figure 1: Transparency and ice adhesion test results of the prepared silica layers

Source: own.

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Co-continuous Inorganic–organic Hybrid Materials Through Emulsion–templating

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Emulsion-templating is a promising technique for synthesizing porous polymers, especially through highly concentrated or high-internal-phase-emulsions (HIPEs) as structural templates. After polymerization, HIPEs usually transform into monolithic polymer foams (PFs). However, PFs can also be produced by this technique in a variety of other forms, such as beads, membranes, or wires, as well as in various compositions, such as nanocomposites or hybrids. One of the main advantages of emulsion-templated PFs is their adjustable porosity, e.g. pore volume, size and interconnectivity, which can easily be adjusted by altering the phase volume ratio, surfactant or polymerization chemistry in the emulsion system [1], [2], [3].

In this presentation we will examine different hybrid systems. Most recently, hybrid PFs have been obtained by the bottom-up synthesis approach using different HIPEs as structural templates. In the first case a hydrosol-in-oil HIPE was used, in which monomers were polymerized in the external phase and tetramethyl orthosilicate was condensed hydrolytically in the internal phase, resulting in a highly porous hybrid structure filled with silica aerogel [4]. The resulting co-continuous hybrid structures exhibited hierarchically porous systems with pronounced macro-mesoporosity and

excellent thermal insulation properties, with lambda-values close to those of superinsulators, i.e., 16 ± 3 $mW \cdot m^{-1} \cdot K^{-1}$. In the second example, particle-stabilized water-in-oil HIPEs were used as structural templates, a process known as Pickering stabilization, in which various metal oxides (MO), such as MgO, ZnO, or Co_3O_4 were fixed in the PFs after polymerization [5]. Subsequently, the MOs were transformed in-situ into the corresponding MOF-74 isostructures by the process called secondary-recrystallization occurring at the metal oxide-polymer interface. Successful hybridization resulted in highly porous, co-continuous MOF 74-PF hybrids forming an architectural hierarchy with pronounced macro-microporosity, in which the MOF microporosity was almost completely accessible to gases, i.e., about 87% of the micropores. Further investigation revealed excellent CO_2 capture performance.

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The centre of the Štajerska region in Slovenia is surrounded by the green forests of Pohorje, a sunny wine-growing region and the Drava River that runs through the city. Maribor is a charming city with rich historic and cultural roots. The Guinness world records tell the tale of rich wine tradition, the oldest vine in the world, which is found in the city centre, as well as the wine roads in the vicinity.

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AIMS AND SCOPE

Aerogels are advanced, highly porous materials designed to meet the criteria of biomedical or environmental applications. They can be used as drug carriers, bone grafts, or wound dressings in biomedical applications and as insulators, absorbents, sensors, and catalysts in environmental applications.
This conference aims to assemble and integrate the most recent progress in aerogels' scientific and technological knowledge. The focus will also be on networking, creating, and strengthening collaborations in the aerogel community through poster sessions, social events, coffee breaks, and meals.

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Keywords:
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The 3rd INTERNATIONAl Conference on Aerogels for Biomedical and Environmental Applications, organized by the Faculty of Chemistry and Chemical Engineering University of Maribor and the AERoGELS COST Action, presented the latest research results and applications of aerogels. This publication compiles the abstracts of the conference and highlights the main results and innovations. The abstracts address the use of aerogels in medical, pharmaceutical, and food applications, including drug delivery systems, scaffolds tor tissue engineering, and bioactive coatings. They also explore aerogels as catalysts, insulators, and environmental solutions and investigate improved catalytic activity and thermal insulation properties. The abstracts emphasize interdisciplinary collaboration and provide ideas tor future research directions. This publication provides valuable insights tor researchers and industry professionals interested in harnessing the remarkable properties of aerogels for biomedical and environmental purposes.

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