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BOOK OF ABSTRACTS

SPANISH-PORTUGUESE INDUSTRY-ACADEMIA AEROGEL MEETING

AERoGELS
COST ACTION CA18125
environment | medicine | biotech | industry | academia

1-2 March 2022

COIMBRA | PORTUGAL

Grupo I.L.C.

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SPANISH-PORTUGUESE INDUSTRY-ACADEMIA AEROGELS FORUM
1-2 March 2022
Faculty of Sciences and Technology
UNIVERSIDADE DE COIMBRA

CREATED BY:
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WELCOME

On behalf of the Organizing Committee, we are pleased to warmly welcome you to the "Spanish-Portuguese Industry-Academia Aerogel Meeting", held on 1-2 March 2021, in Coimbra, Portugal.

This meeting is organized by AERoGELS COST Action (https://cost-aerogels.eu), the Chemical Process Engineering and Forest Products Research Centre (University of Coimbra, Portugal), the I+D Farma research group (University of Santiago de Compostela, Spain) and Flucomp (Asociación de Expertos en Fluidos Comprimidos, Spain).

We expect that the contacts and collaborations established during this meeting can potentiate a synergistic development of research on aerogels in the Iberian Peninsula.

Prof. Dr. Luísa Durães
Chair

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AERoGELS

AIMS & SCOPE

Aerogels are unique nanostructured porous materials with special properties adapted to fit a multitude of advanced applications. Their extremely high porosity and diverse chemical compositions lead to unusual performance as thermal and acoustic insulators, energy storage materials, catalysts, sensors, adsorbents of pollutants, drug carriers, wound dressings and bone grafts, among other uses.

Further advances on aerogels and exploitation of their wide application potential need the overcoming of some of their structural limitations and fading of constrains for large production. This may be achieved by novel material designs, synthesis processes intensification, effective production layouts, also supported by modelling tools and adapted characterization techniques.

This meeting, framed under the scope of COST Action AERoGELS (CA18125) - "*Advanced Engineering and Research of aeroGels for Environment and Life Sciences*", aims at gathering, integrating and strengthening the scientific-technological-production knowledge in aerogels developed in the Iberian Peninsula. It also aims to create new collaboration bridges in the Portuguese and Spanish aerogel community to foster synergies for production development and leading roles enhancement in the European/World context.

The meeting promotes the networking among participants through several sessions for oral presentations from Iberian academia groups and industry players, posters discussion, one Iberian meeting table for analysis of the status and role of Portugal and Spain in the aerogels community, and one Early Career Investigator's Forum that gives the floor to new ideas and promote training sustainability of aerogels development in the Iberian Peninsula.

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PROGRAMME

TUESDAY 1st March

14:00 - 14:30 **REGISTRATION**

14:30 - 15:30 **PLENARY LECTURE** **Chair:** Carlos García-González

Eunate Goiti *(Tecnalia)*

Francisco Ruiz *(Keey aerogel)*

"Sustainable aerogel production using SICLA technology"

ORAL SESSION 1

Chair: Catarina Reis

15:30 - 15:45 **Miguel Sánchez-Soto** *(Univ. Politécnica de Catalunya)*
"Aerogels for construction materials"

15:45 - 16:00 **Miguel Pérez** *(Univ. Valladolid)*
"PU aerogels for thermal insulations"

16:00 - 16:15 **Anna Roig** *(ICMAB-CSIC)*
"Cellulose and nanocellulose aerogel applications"

16:15 - 16:30 **Inês Flores-Colen** *(IST, Univ. Lisboa)*
"LCA and LCC of aerogels and renders"

16: 30 - 17:00 **Coffee Break | POSTER SESSION**

ORAL SESSION 2

Chair: Miguel Sánchez-Soto

17:00 - 17:15 **Corne White** *(Armacell)*
"ArmaGel insulation in the industrial market"

17:15 - 17:30 **Fernando Simões** *(Active aerogels)*
"Silica aerogel production scale-up: technical, environmental, and financial challenges"

17:30 - 17:45 **Amparo López Rubio** *(IATA-CSIC)*
"Aerogels for valorization of wastes"

18:00 - 20:00 **HISTORICAL TOUR**

20:00 - 22:00 **SOCIAL DINNER**

WEDNESDAY
2nd March

09:00 - 10:00 **IBERIAN MEETING**

ORAL SESSION 3

Chair: Ana Borba

10:00 – 10:15 **Marta Corvo** *(I3N, Univ. Nova de Lisboa)*
"Aerogels for CO_2 capture"

10:15 – 10:30 **Pedro Simões** *(Univ. Coimbra)*
"Molecular modeling of aerogels"

10:30 – 10:45 **Clara López-Iglesias** *(Univ. Santiago de Compostela)*
"Aerogels for biomedical applications"

11:00 - 11:30 **Coffee Break | POSTER SESSION**

11:30 - 12:30 **EARLY CAREER INVESTIGATORS' FORUM**

Chairs: Clara López-Iglesias | Joana Barros

Six 10min presentations of PhD and Post-Doc researchers

12:30 - 13:00 **CLOSING SESSION**

AUTHORS' INDEX


AERoGELS

Abstracts of INVITED LECTURES


AERoGELS

PL

Sustainable aerogel production using SICLA technology

Eunate Goiti[a,*], Francisco Ruiz[b,*], Marta Ocejo[a], Kanda Philippe[b]
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GRAPHICAL ABSTRACT

Schematic of closed-loop circular economy business model of high-added value products for energy efficiency.

ABSTRACT

Climate change and environmental degradation represent a threat that Europe and the rest of the world are facing. In this regard, Europe has defined a new sustainable strategy called the European Green Deal [1] with the aim of transforming the European Union (EU) into a modern, resource-efficient and competitive economy, where there are no net greenhouse gas (GHG) emissions by 2050 and where economic growth is decoupled from resource use. Therefore, EU has established a roadmap with actions that aim to: i) boost resource efficiency by shifting to a clean and circular economy and ii) restore biodiversity and reduce pollution.

The principal goal is to make the European Union (EU) climate neutral by 2050. This requires actions in all sectors of the economy, including: ***(i)*** EU climate ambition for 2030 and 2050. In this sencse,the new Circular Economy Action Plan has been launched [2]. ***(ii)*** Energy and resource efficient building and renovation: ensuring that buildings are more energy efficient.

Buildings account for 40% of total energy consumption and about 75% of them are energy inefficient. The European Commission is increasingly committed to the energy rehabilitation of the Member States' building stock with the aim of reducing the EU's total energy consumption by 5-6% and reducing CO2 emissions by 5% [3]. One of the simplest ways to achieve this "energy performance" target is to reduce their energy consumption by decreasing heating and cooling energy demands. Thus, building effective insulation represents an immense market worldwide, for both new constructions and for renovation.

In this perception, silica aerogel, the most effective materials known for thermal insulation at ambient conditions, stands out as the most promising high-performance insulation material. However, the market for aerogels as building-insulation materials remains largely underdeveloped due to the high cost of precursor (~80%) associated with industrial scale production. Thus, price of silica aerogel is clearly the biggest entry barrier of this material in the sector.

KEEY Aerogel and TECNALIA present a **novel sustainable aerogel production technology** called **SICLA™** that follows a closed-loop circular economy model: high-performance building insulation material is manufactured from silica containing waste materials. Silica aerogel materials with exceptional thermal conductivities (λ<0.015 W/mK) are achieved through this novel process. Moreover, the **SICLA™** technology guarantees at least a 40% cost reduction of the aerogel production process.

The present communication attempts to describe different aspects of the process and its implementation, comprising:

- The technology behind the recycling process and manufacture of superinsulating aerogel materials.
- The application of **SICLA™** on the design and development of intermediate and semi-finish insulation products for the retrofitting and new insulation market.

ACKNOWLEDGEMENTS

This work was partly supported by the project "Cost-effective recycling of CDW in high added value energy efficient prefabricated concrete components for massive retrofitting of our built environment" -VEEP- (Project number: 723582) and is financed by the European Union under the Horizon 2020 Framework Programme.

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IL1.1

Aerogels for Construction Materials

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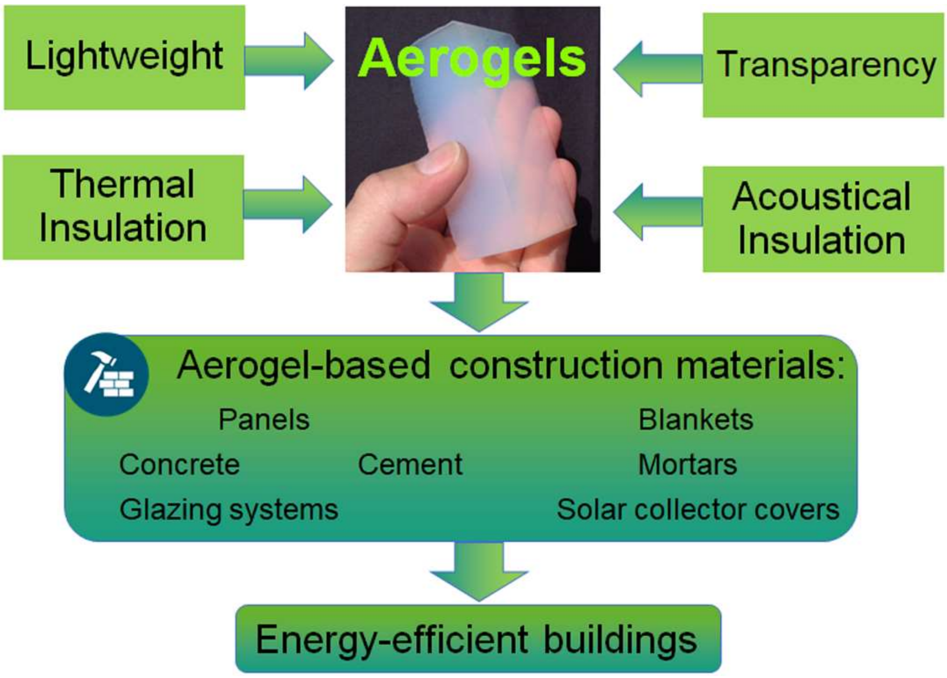
GRAPHICAL ABSTRACT



ABSTRACT

Nowadays, the worldwide overall energy consumption is growing and a 53% increase is forecasted by 2030. In 2016, the global building sector consumed around 30% of the overall energy and contributed with 28% of global energy-related CO2 emissions. Consequently, efforts are made to enhance the thermal performance of building's envelope such as floors, roofs and walls. Incorporating thermal insulation materials is one of the most effective ways to ensure energy savings, being capable of mitigating outward heat losses [1-2]. In order to achieve a further improvement of the building's envelope, traditional insulation materials are used in thicker or multiple layers, resulting in more complex constructions and an adverse net-to-gross floor area. Therefore, there is an urgent need for better thermal insulation solutions.

Aerogels are open-porous, high-performance thermal insulation materials which makes them one of the most promising materials of the last decades. Outstanding low thermal conductivities (even lower than the one of still air) can be achieved in aerogels if their pore size is smaller than the mean free path length of air (~70 nm) due to the Knudsen effect. Therefore, aerogels can be used for very thin building insulations [3]. Moreover, aerogels have applications in structural materials such as the so-called Aerobrick, an aerogel-filled insulating brick with 35% reduced thermal conductivity as compared to conventional perlite-filled bricks [4]. Aerogel granules can be mixed with concrete, cement or mortar in order to achieve a greater porosity and hence a lower thermal conductivity. Since some aerogels are translucent they can be incorporated in glazing systems where they provide a better thermal insulation [5]. Besides, they can be employed for capturing solar thermal energy more effectively [6]. Additionally, aerogels exhibit a low sound velocity of 100 m/s due to their high porosity which gives them acoustical insulation properties [7]. To conclude, if the economic and environmental costs of aerogel production can be further decreased then aerogel insulation may become an appropriate alternative to current traditional building insulation materials. This work aims to review the state of the art of aerogel building applications.

ACKNOWLEDGEMENTS

Work carried out in the frame of the COST-Action "Advanced Engineering of aeroGels for Environment and Life Sciences" (AERoGELS, ref. CA18125) funded by the European Commission.

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IL1.2

PU aerogels for thermal insulation

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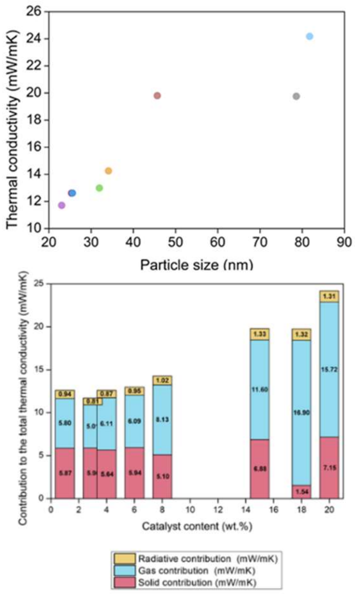
GRAPHICAL ABSTRACT

PU AEROGELS WITH DIFFERENT PARTICLE SIZE



ABSTRACT

With the European Green Deal, the EU is increasing its climate ambition and aims at becoming the first climate-neutral continent by 2050. To meet the new EU climate targets, energy efficiency needs to be prioritized [1]. Improving energy efficiency in buildings is mandatory to mitigate climate change and reduce pollution. Buildings are responsible for 40% of the global energy consumption and 36% of the emitted greenhouse gases [2], thus, the European Commission established a legislative framework to boost their energy performance [3]. Enhanced thermal insulation is thus a powerful tool in combating climate change and a very important step forward in the ecological transition. One of the most promising materials to improve thermal insulation in many applications are aerogels due to their extremely low thermal conductivity in comparison with traditional thermal insulating materials [4].

Since the first aerogels were developed by Kistler in 1931[5], the subsequent studies were focused on silica aerogels because of their versatility and incredibly promising properties. However, it was in 1989 when organic aerogels started to be explored by Pekala [15]. Organic aerogels provide a synthetic control of the porous structure as well as an increased toughness in comparison with inorganic aerogels [7]. For these reasons, organic aerogels have reached a high relevance in the nanoporous materials field and there are numerous works analyzing their synthesis and characterization

In this work, we have focused our attention in PUR-PIR based aerogels. Transparent PUR-PIR based gels have been synthesized and supercritically dried obtaining a family of aerogels characterized by a similar porosity but a clearly different internal structure with different particle size and pore size. The thermal conductivity of these materials have been measured at different temperatures (from 10ºC to 40ºC) obtaining a clear trend with the initial formulation and porous structure. The thermal conductivity measured at 10ºC ranged between 12 and 24 mW/mK, so it has been possible reaching very small values. In addition, the thermal conductivity has been modelled as a function of the structural parameters of the aerogels to quantify the different heat flow transfer mechanisms in these materials. It has been demonstrated that the differences observed between materials are mainly due to the Knudsen effect, which is more intense for those materials having a reduced pore size. Moreover, their mechanical properties have been studied. The manufactured aerogels showed tunable stiffness through the change in the catalyst concentration and a significant elasticity. Thus, super-insulating transparent PUR-PIR aerogels with tailored mechanical properties have been produced.

ACKNOWLEDGEMENTS

Financial support from the FPU grant FPU17/03299 (Beatriz Merillas) from the Ministerio de Ciencia, Innovación y Universidades (RTI2018-098749-B-I00) is grateful acknowledged. Financial assistance from the Junta de Castilla y León (VA202P20) and the "Ente Público Regional de la Energía de Castilla y León" (EREN_2019_L4_UVA) are gratefully acknowledged.

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IL1.3

Cellulose and nanocellulose aerogel applications

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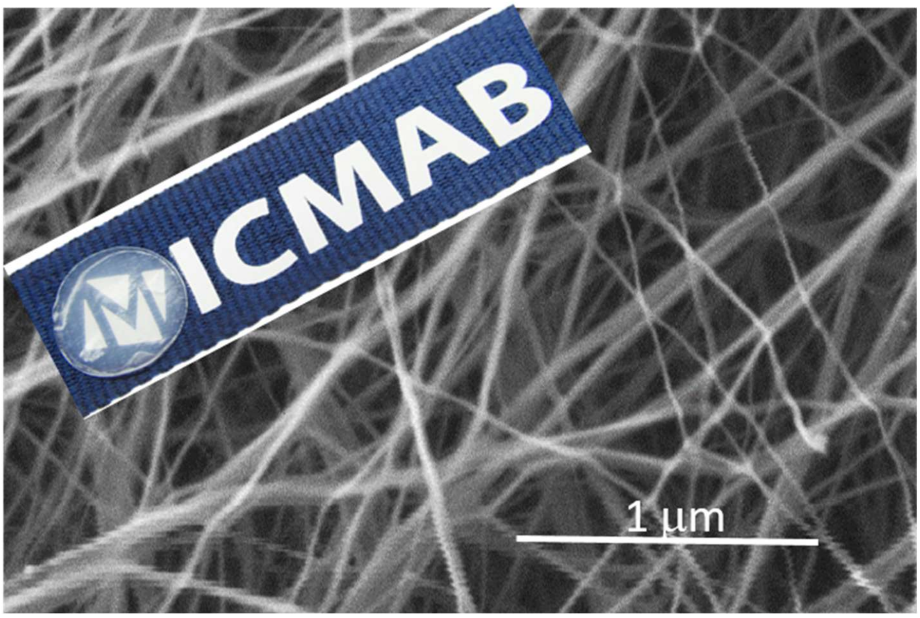
GRAPHICAL ABSTRACT



ABSTRACT

The different governmental policies and the society as a whole are pressuring to address climate change and plastic pollution and move towards a more circular bioeconomy by replacing petrol-based products with bio-based and biodegradable materials. Cellulose is increasingly viewed as a versatile biopolymer with renewable, biodegradable, sustainable and even biocompatible characteristics [1]. As wood, cellulose has provided shelter and was used as a heat source by our ancestors and in the form of paper has facilitated the knowledge transmission for centuries. Only in the last century, cellulose and cellulose aerogels have been used to fabricate advanced materials and products [2].

The term aerogel, which for many years was synonymous with a very specific type of low-density mesoporous silica, has now grown to encompass an extensive family of other types of materials. Some members of this family include aerogels from a panoply of carbon allotropes, aerogels made of bio-based polymers (cellulose, pectin, or chitosan), metallic foams and nanoparticle-based aerogels obtained from the assembly of pre-formed nanoparticles. In a

sense, the term aerogel has evolved from describing a type of material to describing a new state of matter, the scope of which will only continuously evolve.

Cellulose aerogels are ultra-light-weight, open porous materials with high surface area. There is a broad range of possibilities to synthesize cellulose aerogels. For instance, regarding the creation of a gel state, two different strategies can be followed. A "colloidal approach", and a "molecular approach". The first strategy uses cellulose nanocrystals (CNC) or cellulose nanofibers (CNF) and the gel formation is promoted by physical entanglement, transfer for dispersion to solid-state, or modification of particles interactions by changes in the pH, ionic strength or temperature [3]. The "molecular approach" is based on cellulose dissolution and coagulation routes [4]. The combination of those two approximations to gels added with the several possibilities of drying the gels to achieve aerogels (supercritically and freeze-drying or freeze casting) makes that the characteristics of the resulting materials encompass values in wide ranges. The surface area goes up to hundreds m^2/g, density from a fraction of g/m^3 to 1 g/m^3 and the average pore size from thousands to a few nm; this is also true for the mechanical properties that usually scale as E [2]. The range of properties and functionalities of cellulose aerogels can be further extended by the growing family of cellulose-based hybrid aerogels in which organic and inorganic components are incorporated in the cellulose network to developed materials with tunable functionalities [5]. Few examples of cellulose aerogels applications in fields including healthcare [6,7], environmental remediation and monitoring, and energy-storing will be discussed.

ACKNOWLEDGEMENTS

The author acknowledges financial support from the Spanish Ministry of Science and Innovation through the RTI2018-096273-B-I00 project, the 'Severo Ochoa' Programme for Centres of Excellence in R&D (CEX2019-000917-S) and the Generalitat de Catalunya 2017SGR765 grant. The author participates in the CSIC InterdisciplinaryPlatform for Sustainable Plastics towards a Circular Economy, SUSPLAST. Work also carried out in the frame of the COST-Action "Advanced Engineering of aeroGels for Environment and Life Sciences" (AERoGELS, ref. CA18125) funded by the European Commission."

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IL1.4

LCA and LCC of aerogels and renders

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GRAPHICAL ABSTRACT

ABSTRACT

Aerogels are classified as nanostructured superinsulation materials. Silica aerogels have been used in a wide range of scientific and technological applications, such as in mortars coating. Aerogels are typically prepared using a multi-step synthesis process whereby the 3D network of the material with a high proportion of porosity is preserved. Despite the numerous types of aerogels, for the synthesis of almost all aerogels three main steps are involved: i) gel preparation; ii) ageing; and iii) drying. Hydrophilic aerogels have high structural sensitivity to water, being thus unsuitable for incorporation into mortars. In fact, internal fractures appear in the aerogel leading to its collapse, with poor adhesion between the aerogel and the remaining mortar paste, resulting in a mortar with poor cohesion at the hardened state. Therefore, the aerogel surface is modified during or after drying, producing hydrophobic aerogels. This property can be achieved by chemical modification (sialylation) of the pores surface, allowing the preservation of the porous structure, and can be carried out both after and during the synthesis process of the aerogels. Supercritical and ambient processes are the drying methods commonly used. Supercritical drying has been used for a large-scale commercialization and in the production of aerogels, despite the high cost and safety risks to the manufacturers (high pressure equipment). New trends followed subcritical drying processes, being cost-effective and easy to produce. Moreover, the subcritical hybrid aerogel is less brittle and its

hydrophobicity is better controlled, and the particle size distribution can be controlled by grinding and sieving. However, it has usually a higher density, influencing the mortar properties. For those reasons tailored-made (functionalized) aerogels have an important role in the final rendering mortar properties. In a life cycle cost analysis (LCC) for several aerogel synthesis at a laboratory scale, the results showed that the raw materials acquisition stage is the most relevant phase in terms of costs, because it represents more than 97% of the total costs (reactants costs). When the drying process is longer the expenses associated with energy consumption are significant. The environmental evaluation using the Life Cycle Assessment (LCA) for the previous synthesis, using a cradle to gate approach, also emphasized the impact of raw materials; the environmental impacts, and the production in the lab (high energy consumption in longer drying process). Two indicators were studied in a cradle-to-gate analysis: AdP-ff (MJ), Abiotic Depletion Potential from fossil fuels as an indicator of the use of non-renewable energy sources; and GWP (kg CO2 eq) associated to greenhouse gases production. The results showed that the main contributors to the subcritical aerogel environmental performance were isopropanol (the main solvent used) and electricity (related to the drying process). Further, TEOS also had a significant contribution, however, with lower overall impact. Nevertheless, a sensitivity analysis was carried out based on other works, including the variation of reagents recycling and energy optimization in the drying process (less drying period), showing a significant reduction of environmental impacts in an optimized synthesis (e.g. 98% recycling of reagents and 92% of optimization of electricity use). This presentation focuses on one single part [1-4] of the work that has been developed in the last ten years in CERIS / IST, under the scope of Nanorender and PEP research projects (concluded), three PhD theses (António Soares, Marco Pedroso and Maria Júlio) and two MSc dissertations (Rita Garrido and Isabel Pinto).

ACKNOWLEDGEMENTS

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IL2.1

ArmaGel insulation in the industrial market

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GRAPHICAL ABSTRACT

ABSTRACT

The dedicated Energy segment of Armacell provides thermal and acoustic insulation systems designed to deliver performance advantage, project, operational and energy savings, asset integrity and safety in the harshest of environments.

The use of Aerogel powders in the design and fabrication of thermal and acoustic insulation blankets has resulted in many strong advantages for plant owners and system installers. Advantages that include protection against CUI (corrosion under insulation), drastic reduction of overall insulation thickness in both acoustic and thermal insulation applications and total cost of ownership reduction for plant operators.

Armacell's brief presentation in March will provide an overview of applications insulated with our ArmaGel aerogel insulation blankets.

ACKNOWLEDGEMENTS

All R&D costs related to the development of 'ArmaGel' have been paid for by the company.

REFERENCES

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IL2.2

Silica aerogel production scale up: technical, environmental, and financial challenges

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GRAPHICAL ABSTRACT

ABSTRACT

Aerogels are extraordinary materials capable of combining unique characteristics, namely their thermal, mechanical, electrical, and optical properties. At *Active Aerogels*, we have developed multiple textures (rigid and flexible panels, granular, foam, powder) of silica-based aerogels for a wide range of applications in different sectors of activity. However, moving from laboratory research to mass production and commercialization is among the most ambitious and challenging endeavours. Here we present the most important issues regarding the scale-up and process industrialization of our super insulating silica aerogel suitable for industrial and housing applications.


AERoGELS

IL2.3

Aerogels from waste valorisation

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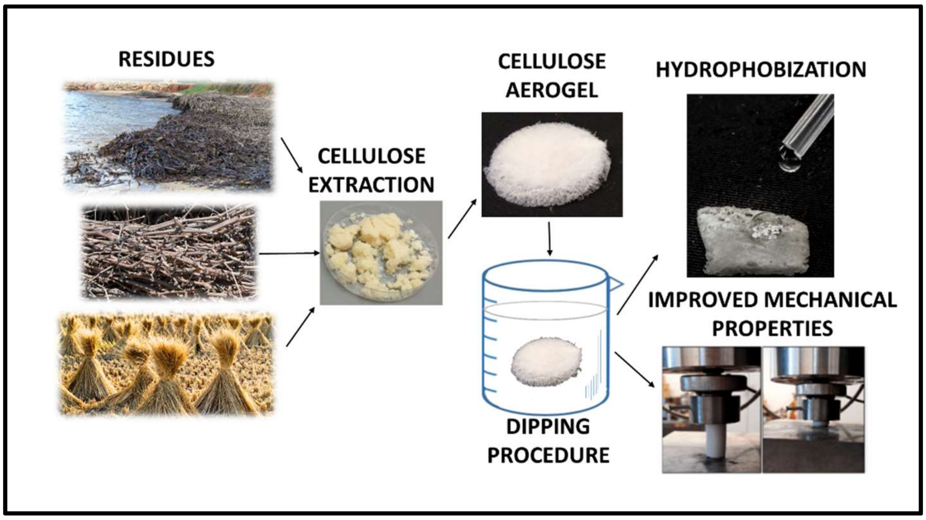
GRAPHICAL ABSTRACT



ABSTRACT

Cellulose is a promising raw material for the preparation of aerogels for a broad variety of applications. This biopolymer is the most abundant carbohydrate on Earth and it can be extracted from a wide variety of agroindustrial residues. Furthermore, cellulose presents excellent biocompatibility, biodegradability, and non-toxicity, as well as high susceptibility to chemical modifications, which makes them ideal carriers for drug delivery systems. However, one of the main issues associated to cellulose aerogels are their high moisture sensitivity and relatively poor mechanical properties both hindering their use for many applications in which silica aerogels, for instance, are nowadays being used. In our research group we have been exploring, on the one hand, simplified methods for the extraction of cellulose-rich fractions

from a broad variety of agroindustrial residues [1-3] and, on the other hand, strategies to improve cellulose aerogel performance [4, 5]. Interestingly, cellulose aerogels prepared from less purified cellulosic fractions, apart from being more cost-competitive, also have improved sorption performance. This is mainly explained by the more porous structure generated as a consequence of having hemicelluloses present in their composition. Regarding the strategies used to improve cellulose aerogel performance, we have patented a very simple coating method [5], which apart from enhancing the mechanical properties of the materials, allows them to keep their integrity when immersed in water. Moreover, very interestingly, if these coated cellulose aerogels are immersed in a solution containing both water and oil, they preferentially sorb oil, thus making them attractive for water purification applications. In this oral presentation, several examples dealing with the preparation of cellulose aerogels from waste biomass will be shown, together with the characterization and advantages of the strategies developed.

ACKNOWLEDGEMENTS

The authors acknowledge grant RTI2018-094268-B-C22 funded by MICIN/AEI/10.13039/501100011033/ and by "ERDF A way of making Europe". This work was also carried out in the frame of the COST-Action "Advanced Engineering of aeroGels for Environment and Life Sciences" (AERoGELS, ref. CA18125) funded by the European Commission.

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IL3.1

Aerogels for CO_2 capture

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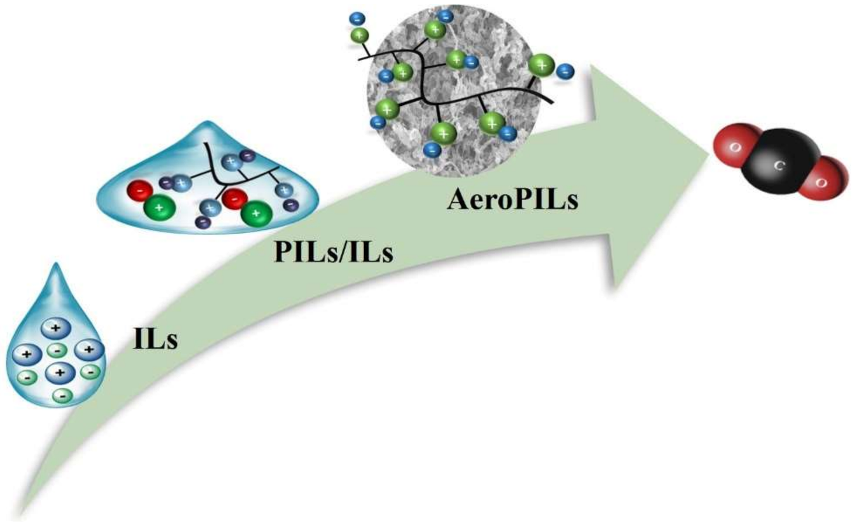
GRAPHICAL ABSTRACT



ABSTRACT

Carbon dioxide atmospheric levels are currently above the 400 ppm threshold. Being the most common greenhouse gas produced by human activities, and the major cause for manmade global warming, CO_2 capture (CC) is an urgent matter of global need. Conventional CC focuses on solvent scrubbing with chemical absorption of CO_2, however, disadvantages as solvent loss through evaporation, formation of corrosive byproducts, and high energy consumption during regeneration, demand optimized alternatives. Given the magnitude of global CO_2 emissions, the energy input for recycling capture materials is a key factor for efficiency and cost of the overall process [1].

Ionic liquids (ILs), organic salts with melting points below 100 °C have been proposed as alternative solvents for CC due to their stability and high selectivity for CO_2 absorption. The possibility of manipulating ILs properties has made ILs extremely versatile materials with several applications from reaction media, to catalysis and electrochemistry. Nuclear Magnetic Resonance (NMR) is a privileged technique for tailoring ILs, as it provides atomic resolution at the same time as enables the study of dynamic information [2]. The search for superior IL systems has taken us on a journey that started on the optimization of ILs properties for CC, departing to polymeric systems, to combine the unique characteristics of ILs with macromolecular frameworks, and arriving at aerogels from polyILs (aeroPILs). This quest has led us to the design of both the chemical structure and the morphology of materials capable of capturing and converting CO_2 [3-5].

ACKNOWLEDGEMENTS

We are thankful for financial aid from FEDER funds through COMPETE 2020 Programme and National Funds through FCT (Portuguese Foundation for Science and Technology-POCI-01-0145-FEDER-007688, UIDB/50025/2020-2023; PTDC/QUI-QFI/31508/2017; PINFRA/22161/2016), and Xunta de Galicia [ED431C 2020/17], MICINN [PID2020-120010RB-I00], Agencia Estatal de Investigación [AEI]. RVB.; CL-I; MZ; and MCC acknowledge FCT for the SFRH/BD/150662/2020 PhD fellowship; Xunta de Galicia (Consellería de Cultura, Educación e Ordenación Universitaria) for a postdoctoral fellowship [ED481B-2021-008]; European Union's Horizon 2020 research and innovation program under the Marie Skłodowska-Curie grant agreement Nº101026335, and PTNMR&i3N for the researcher contract, respectively. Work partially carried out in the frame of the COST-Action "Advanced Engineering of aeroGels for Environment and Life Sciences" (AERoGELS, ref. CA18125) funded by the European Commission.

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IL3.2

Molecular modeling of aerogels

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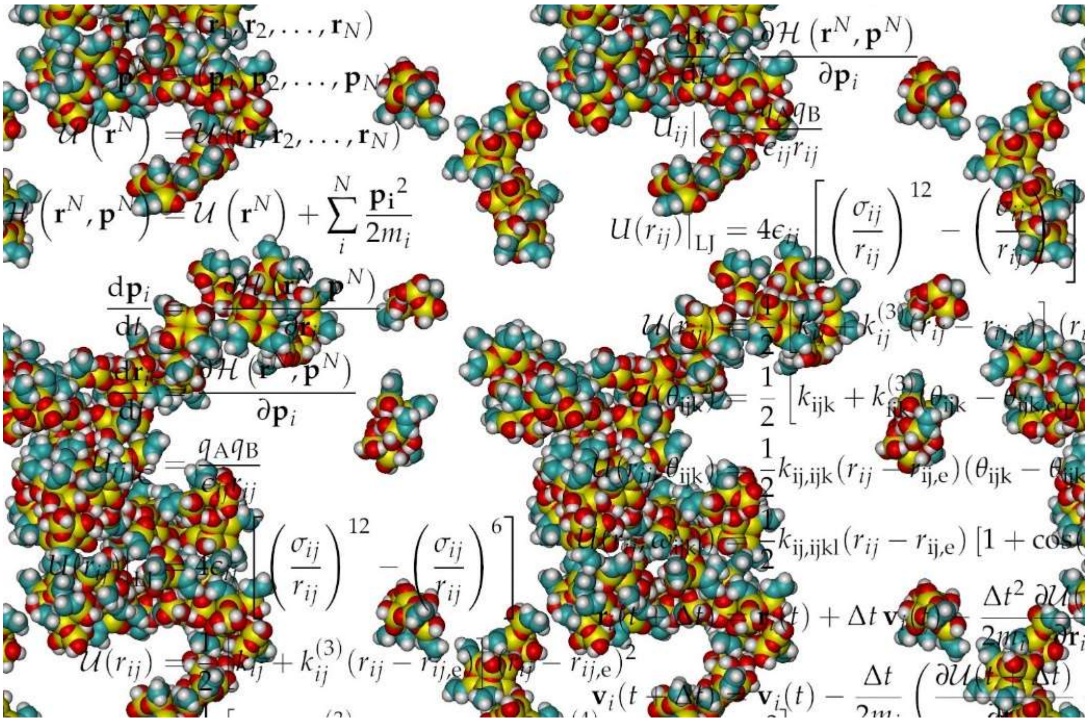
GRAPHICAL ABSTRACT



ABSTRACT

Aerogels are multifaceted materials, the most representative species being silica-based aerogels, which reveal a remarkable potential for various commercial applications. The chemical and physical phenomena underlying the sol-gel process span a variety of scales, from molecular precursors, through the secondary particles, to the final aerogel network. An in-depth knowledge of the interrelationship between these phenomena and between them and the properties of the aerogel is still lacking. In this context, molecular modeling and simulation has emerged as a crucial technique to unravel mechanistic features of the sol-gel process with atomistic detail, hardly accessible by strictly experimental means.

This talk aims to provide a short overview of relevant atomistic models and simulation approaches to computationally study aerogels, together with relevant physical-chemical properties attainable from them. The focus will be on silica-based aerogels as the prominent class. A bottom-up multiscale approach will be considered, from the (sub)atomic scale up to the nanoscale, highlighting how they can intersect in view of an integrated strategy. Quantum mechanics-based calculations will be discussed at first as a key method to obtain the structure of primary particles and to predict the mechanism and energetics of the hydrolysis and condensation reactions of the initial sol-gel stages, thus at subnanometric scale. After, models of the fractal structure of the aerogel network targeted to classical molecular dynamics (MD) nanoscale simulations will be discussed. [1]

ACKNOWLEDGEMENTS

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IL3.3

Bio-based aerogels for drug delivery and tissue engineering

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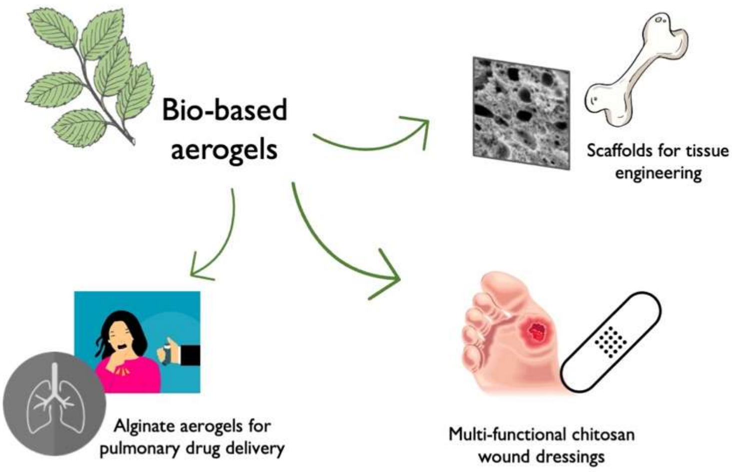
GRAPHICAL ABSTRACT



ABSTRACT

Aerogels find a wide range of applications in the biomedical field due to their outstanding textural properties. Aerogels based on biomolecules, such as polysaccharides and proteins, are especially promising since there are obtained from highly abundant sources and provide excellent biocompatibility and biodegradability [1]. In this work, recent advances on bio-based aerogels are presented, with special focus on their application as tissue engineering scaffolds, wound dressings, and pulmonary drug delivery formulations.

Aerogels present a structure similar to the extracellular matrix where cells can adhere and proliferate, making them suitable candidates for tissue engineering applications. However, most aerogels prepared from polysaccharides present a mesoporous structure, which can limit their use as scaffolds. In recent work, zein was included in a starch aerogel formulation as a porogen agent to successfully introduce macroporosity in the polymer network [2].

Furthermore, zein increased the stiffness of the aerogels and presented a protective effect during the storage period, preventing mechanical changes. The fabrication process was also combined with a supercritical CO_2 sterilization to prepare ready-to-implant scaffolds, which were further characterized in terms of sterility and cytocompatibility.

Aerogels can also be used for drug delivery purposes since their high surface area allows for high drug load yield, which is especially relevant for drugs with poor solubility or bioavailability. In the field of wound healing, aerogels also present high sorption capacity due to their high porosity, being able to absorb wound exudate [3]. In recent work, chitosan-based aerogels were prepared and loaded with an antibiotic for the prevention and treatment of chronic wound infection. The combination of the intrinsic properties of chitosan with the properties of an aerogel material resulted in a multifunctional material promising for wound healing purposes. The formulation was further optimized in terms of particle size and drug load efficiency by combining the aerogel technology with novel techniques such as jet cutting and 3D-printing.

Finally, in the field of pulmonary delivery porous particles are being sought to overcome the common limitations of traditional dry powder formulations, such as poor flow properties and reduced effective dose [4]. In this sense, aerogels can reduce the density of the particles, allowing for larger particle size while maintaining suitable aerodynamic diameters. To obtain particles with suitable sizes, alginate aerogels were prepared combining supercritical drying with the inkjet printing technology, leading to powder formulations with improved performance in pulmonary administration.

ACKNOWLEDGEMENTS

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Abstracts of ORAL PRESENTATIONS

Early Career Investigators' Forum


AERoGELS

O1

A technological combination to obtain 3D-printed and sterile bioaerogel scaffolds composites

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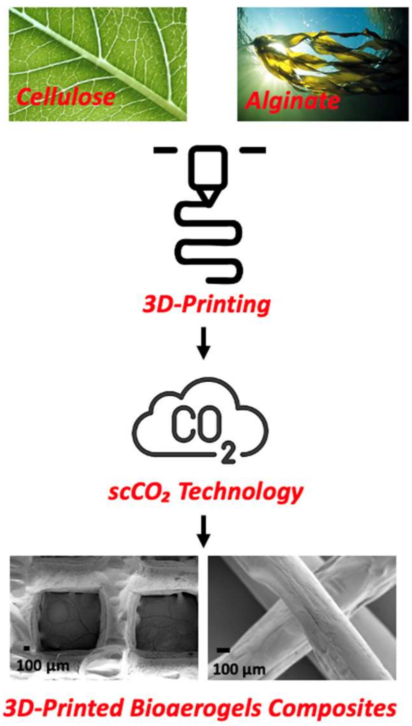
GRAPHICAL ABSTRACT



ABSTRACT

3D printing is a cutting-edge technology that enables the fabrication of structures with a personalized external morphology adaptable to the specific needs of patients. The preparation of scaffolds with a customized external shape and a dual internal structure (mesoporous and macroporous) is essential for tissue engineering and can be achieved by the novel combination of 3D-printing and supercritical CO2 (scCO2) drying [1]. For this purpose, a polymeric ink is printed to obtain a gel of personalized shape and endowed with attractive structural and

biological properties. Then, the gel solvent is removed by the green supercritical fluids technology, preserving all gel properties and resulting in 3D-printed bioaerogel scaffolds.

The formulation of an adequate ink is crucial in 3D-printing. Alginate is a low-cost material commonly employed in bioinks because it is a biocompatible, non-immunogenic and biodegradable material that supports cell growth. Moreover, molecules can be trapped inside the alginate matrix being still able to diffuse. This feature makes alginate hydrogels ideal for bioink formulations, usually containing bioactive molecules. Cellulose is also a natural and non-toxic polysaccharide, easily mass produced and usually employed in 3D-printing for tissue engineering applications. It presents a large capacity of water absorption and excellent mechanical properties [2].

Prior to implantation, the clinical-grade sterility of bioaerogel scaffolds must be ensured [3]. The sterilization implies the complete destruction of life, including fungi, bacteria and viruses and it is commonly achieved by heat, chemicals, irradiation, pressure, or filtration. A low-temperature and radiation-free alternative is the use of scCO2 technology [4]. In this work, alginate and cellulose-based aerogel scaffolds were obtained by the combination of 3D-printing and supercritical CO2 technology for bone tissue engineering applications. Their macro and microstructure as well as textural properties were evaluated by adsorption/ desorption analysis (BET) and by SEM imaging. The suitability of the scCO2 post-treatment to achieve the sterility of bioaerogel scaffolds is evaluated by microbiological tests and by changes in textural parameters with respect to those obtained before the sterilization procedure.

ACKNOWLEDGEMENTS

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O2

Reinforced silica aerogels by using open cell polyurethane foams as skeleton: thermal and mechanical properties

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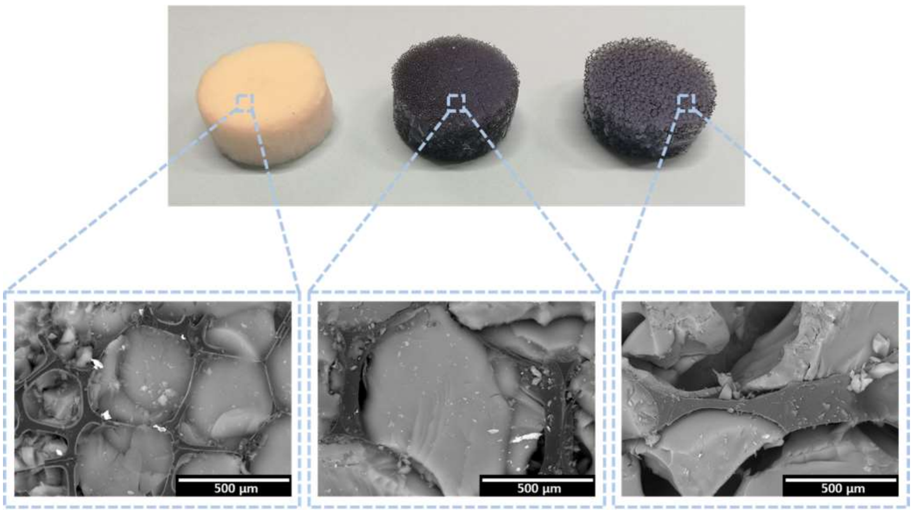
GRAPHICAL ABSTRACT



ABSTRACT

In the recent years, there exists a growing interest on the development of super-insulating materials. Aerogels have mainly gathered the attention due to their exceptional properties such as light weight, low speed of sound, huge surface areas and incredibly high insulating performance [1]. Silica aerogels are one of the most explored aerogels because of their outstanding characteristics, however, their brittleness and reduced mechanical properties significantly limit their applications. This fact leads to the employment of different reinforcing strategies[2]: several studies have modified their flexibility and stiffness by using other silica

precursors [3], via polymeric cross-linking of the silica skeleton [4] or even by fiber reinforcement [5], [6].

In this work, a novel reinforcing strategy based on reticulated polyurethane foams has been employed. Three foams having different pore sizes have been used as support of silica aerogels. A surface modification with HMZD was performed for some composites to assess its effect on the final properties. Additionally, two drying methods were employed: ambient pressure drying and supercritical drying for each formulation. In this way, a continuous network of silica aerogel was obtained while completely filling the foam inner pores. Despite the increase in the final density that the fabricated composites show, their thermal conductivity is notably reduced in comparison with the initial PU foams because of the sharp decrease in the pore size and the corresponding Knudsen effect. Moreover, the mechanical properties of the silica aerogel have been improved by increasing their stiffness while keeping a high recovery ratio after several compression-decompression cycles.

Finally, the effect of the PU foam pore size, silica modification through silylation and type of drying were analyzed obtaining the optimum features and conditions for achieving the lowest thermal conductivity and enhanced mechanical properties.

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O03

Controlling the post-structure properties of metal-crosslinked carbon-aerogels from alginate for emerging technologies

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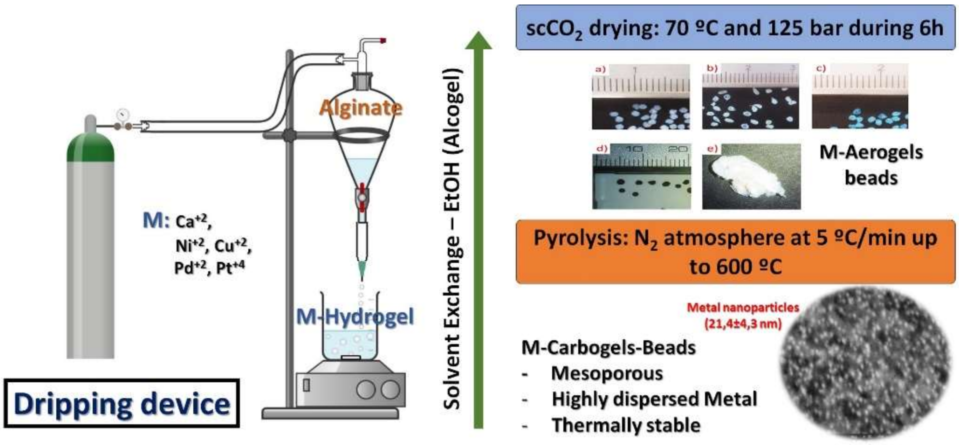
GRAPHICAL ABSTRACT



ABSTRACT

Polysaccharides have gained much interest as an inexpensive and abundant carbon source for aerogels production, especially those derived from species with large capability of fixing carbon dioxide, such as the brown algae [1]. This represents an opportunity in the context of sustainable development, by encouraging the algae CO_2 capture with added value. Up to 60% of the total sugars in brown algae are alginate, an anionic biopolymer that can be easily crosslinked with cations for the obtention of metal-hydrogels [2], and also for capturing pollutant cations as adsorbent of heavy metals from contaminated environments [3][4]. For its part, carbon aerogels (carbogels) are a promising alternative for natural carbonaceous materials in the fields of catalysis, environmental remediation and energy storage [5]. In the present work, a series of metal-crosslinked aerogels (MCAs) and carbogels (MCCs) with catalytic activity prospective were synthesized via a facile and unexpensive strategy from the

crosslinking of anionic alginate with selected metals (Ca^{2+}, Ni^{2+}, Cu^{2+}, Pd^{2+}, Pt^{4+}). The evolution of the aerogels into carbogels was studied based on a thorough characterization, comprising techniques like ICP-OES, CamSizer, TGA/DTG, UHV-XPS, H_2-TPR, XRD, SEM-BSE, and N_2-adsorption (BET/BJH). The increase of metal precursor bath concentration (from 17 mM to 380 mM) didn't yield a significant metal content in the hydrogel. From the TGA, the MCAs presented a DTG peak between 235-285 ºC, carrying the major mass loss (55-65%), associated to the biopolymer decomposition to form a metal-carbonate [6]. Among the MCCs obtained by pyrolysis, Pd-carbogel remarkably lost the least, with only 58 %, up to 600 ºC. The textural features of the MCAs showed specific surface area ranging from 480 to 687 m^2g^{-1}, and pore sizes in the mesoporous range ($5<Pr<25$ nm). Through different pyrolysis temperatures (150-600ºC) there was a decreasing trend of the textural features, with a huge change after approximately 285 °C, associated to the expected shrinkage, by the reduction of mesopore-radius between % 66 and 90%. Interestingly, for Pd the thermal treatment between 130-265 ºC provoked the opposite effect than the other metals, thus gaining pore size, but still in the mesoporous range. SEM-BSE images of Ni-Carbogel showed metallic-clusters evolution through the different pyrolysis temperatures, developing its best shape at 600 ºC, with a well-defined round Ni nanoparticle of 21,4±4,3 nm size, highly dispersed across the inner pore structure of the carbogel.

ACKNOWLEDGEMENTS

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O04

Bioinspired preparation of alginate aerogels for wound treatment

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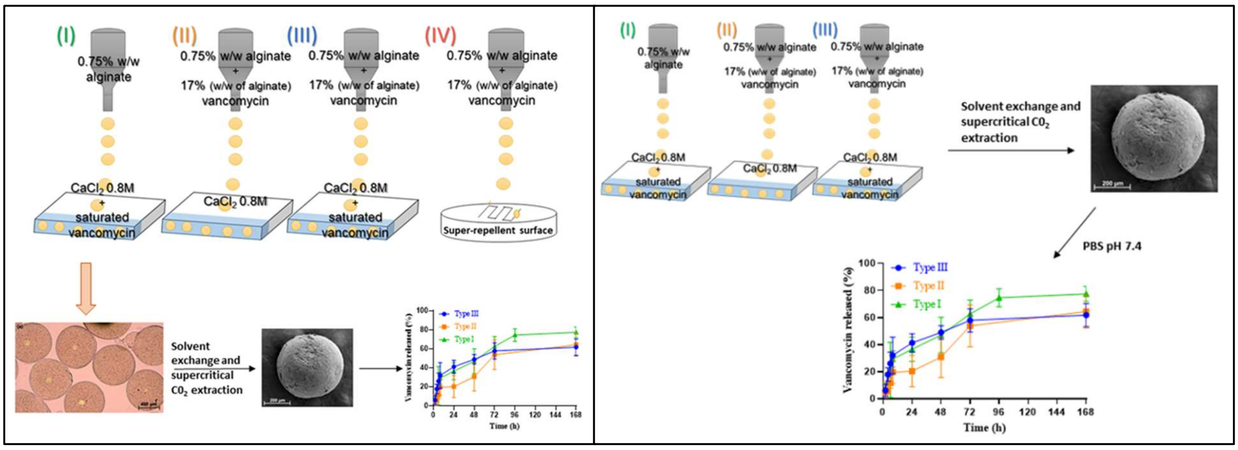
GRAPHICAL ABSTRACT



ABSTRACT

Chronic or hard-to-heal wounds represent poor health-related quality of life of patients and a huge economic burden for the National Health Systems. As example, in the United States there are more than 6.5 million patients with wounds which means an estimated cost for health care system of around US$25 billion per year [1].

Novel techniques for the manufacturing of aerogels are currently getting increasing attention. In the case of aerogels prepared with polysaccharides, they could lead to drug delivery systems with extraordinary compatibility with tissues and cells, alongside their biodegradation and high swelling[3]. These characteristics could be effective in the case of chronic wounds. Implementation of 2D- and 3D-printing platforms could be a very interesting pathway to develop this type of materials [2.3]. Combining this technique with the use of solvent-repellent surfaces (with the same behavior of the lotus leaf when repels the drops of water in Nature) could lead to aerogel microparticles with high reproducibility and improved drug loading yields [4]. The solvent-repellent surfaces are able to overcome the issues of conventional

encapsulation methods, which usually include (i) the use of two or more liquid phases, typically one an organic solvents, and (ii) hard conditions, compromising the stability of labile drugs and the encapsulation yield due to diffusion events between the different phases[4].

In this work, we developed vancomycin-loaded alginate aerogel microparticles, implementing supercritical CO_2 extraction and the combination of printing and solvent-repellent surfaces. Aerogel particle size was measured by optical microscope, the textural characteristics were determined by nitrogen adsorption-desorption analysis and surface structure was studied by Scanning Electronic Microscopy. Loading and release of vancomycin in PBS pH 7.4 were monitored by HPLC. As result, we obtained alginate aerogel microparticles with high surface area, adequate porosity, and spherical shape, with a high reproducibility because of the use of 3D printing techniques. The employment of solvent-repellent surfaces led to an extraordinary encapsulation yield and uniform spherical shape of microparticles. Vancomycin from the microparticles at concentrations above the MIC value for *Staphylococcus aureus* was present in the release medium after 2 h, demonstrating a sustained delivery during 1 week. Overall, this ecofriendly technique could open novel possibilities for the treatment of chronic wounds and related pathologies.

ACKNOWLEDGEMENTS

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O05

Aerogel-based patches as part of a hybrid gold nanoparticle-based photothermal therapy approach

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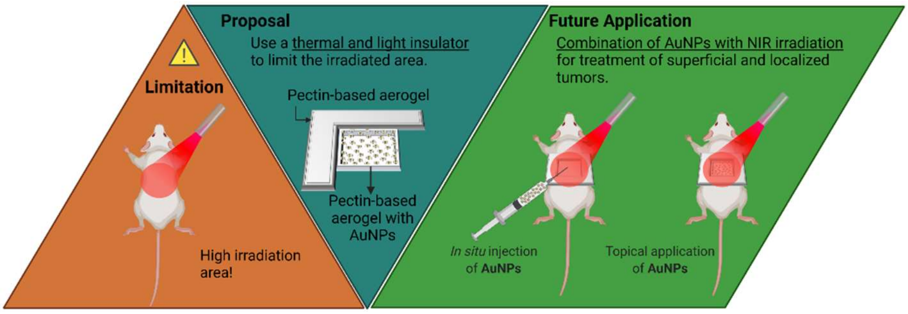
GRAPHICAL ABSTRACT



ABSTRACT

Aerogels have singular properties such as very low density (< 0.5 g.cm^{-3}), high surface area, high porosity (>80 %), tunable surface chemistry, as well as low thermal conductivity, refractive index and dielectric constant [1,2]. All these features attracted attention from different fields and made possible to use aerogels in several applications, namely thermal insulation, catalysis, environmental clean-up, food packaging and cosmetics [2]. In addition, aerogels also present great biodegradability, biocompatibility and ability to mimic biological structures [2], which make them ideal candidates for biomedical applications.

Photothermal therapy (PTT) presents innumerous advantages for the treatment of superficial and localized tumors. However, it requires a great control over the light source used to enhance the depth reached by the radiation. One option to improve PTT efficacy relies on the use of near-infrared (NIR) radiation, once it is less absorbed by the tissues [3]. In this strategy, the concomitant use of gold nanoparticles (AuNPs) also plays an important role for photothermal

enhancement [4]. Herein, we assessed the potential of bio-based aerogels made of pectin as frames for thermal and light insulation in PTT systems for superficial lesions. Pectin is considered as one of the best thermal insulating materials presenting excellent biocompatibility [5]. In this work, aerogels made of only pectin or of pectin incorporating AuNPs were prepared using 3D-printing technology. The gelation of pectin was achieved by combining temperature and ethanol-induction gelation. Aerogels were characterized in terms of macroscopic aspect and textural properties.

The pectin-based aerogels showed good textural properties, which did not seem to be affected by the addition of AuNPs. A preliminary *in vivo* safety assay was also conducted with BALB/c mice to assess the potential use of pectin-based aerogels incorporating or not AuNPs, as part of a PTT system. All produced aerogels were topically applied on dorsal part of the mice and perpendicularly irradiated with a NIR laser. The final goal was to evaluate if the use of aerogels of only pectin led some protection against skin burns potentially induced by laser irradiation and if, by contrast, the use of pectin-based aerogels incorporating AuNPs potentiate the formation of skin burns. This study macroscopically confirms the biocompatibility of the aerogels. Future studies will be required in order to test this PTT systems in localized tumors.

ACKNOWLEDGEMENTS

This work was supported by Fundação para a Ciência e Tecnologia (FCT), Portugal, under the references SFRH/BD/147306/2019, UIDB/00645/2020, UIDB/04138/2020 and UIDP/04138/2020 and by MICINN [PID2020-120010RB-I00], Agencia Estatal de Investigación [AEI] and FEDER funds. T. F.-G. also acknowledges to the COST Action “Advanced Engineering and Research on AeroGels for Environment and Life Sciences” (AERoGELS, Ref. CA18125), funded by the European Commission, for the granted Short Term Scientific Mission to perform the aerogels synthesis at Universidade de Santiago de Compostela, Spain. A.I.-M. acknowledges to Xunta de Galicia for her predoctoral research fellowship [ED481A- 2020/104].

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O06

Multipurpose silica aerogel strengthened with textile fibres waste

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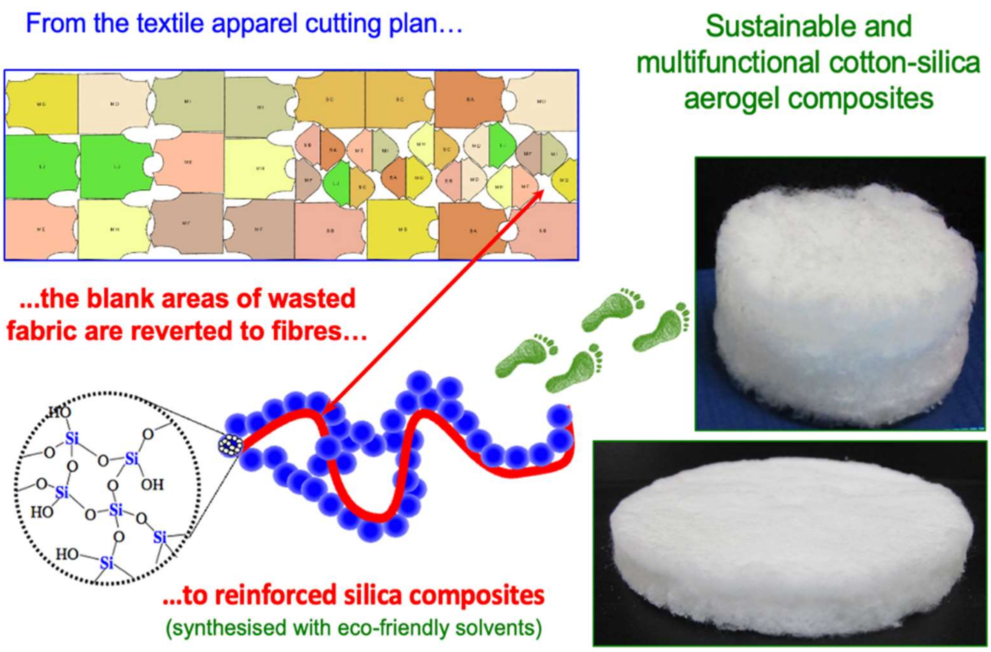
GRAPHICAL ABSTRACT



ABSTRACT

This work describes a sustainability concept relying on the utilization of textile industrial wastes and by-products as raw material for developing silica-based value-added products, namely aerogel composites reinforced with reclaimed fibres, applying environmentally favourable processing routes, such as APD. Also, ethyl acetate was here introduced as a green solvent used in their manufacturing.

Aerogels are unique materials due to their uncommon features, with emphasis on the extreme porosity, putting them at the lower end of densities range for solid man-made materials [1] and rendering them a remarkable insulation performance [2]. However, the highly porous ceramic nanostructure that grants their uniqueness, is as well responsible for their intrinsic

brittleness, which makes difficult their processing and handling [3]. The embedment of fibres into the aerogel structure is broadly recognized as an effective technique for aerogels reinforcement [4].

As per our research in local textile facilities, also in line with the values found in the literature [5], 15 to 20% of the required fabric is wasted in the clothing industry cutting rooms. The here used reclaimed cotton fibres were obtained from those wasted fabric, without the use of chemicals, but utterly through mechanical processes.

Textile fibres are quite startling materials, with an almost endless set of properties. Depending on their characteristics, they range from hydrophilic to hydrophobic, from huge elasticity to extreme stiffness, or from easily flammable to impressively fire resistant. The characteristics of each fibre can be harnessed to precise functionalities, as it is in the case of cotton.

By engineering work, multifunctional attributes were gathered after the development of silica aerogel composites embedded with reclaimed cotton fibres, featuring good thermal conductivity (21 mK m K^{-1}), good acoustic attenuation (with a sound absorption coefficient ~ 0.8), and moisture regulation ability.

ACKNOWLEDGEMENTS

Teresa Linhares gratefully acknowledges *Fundação para a Ciência e a Tecnologia* for the Doctoral Grant SFRH/BD/131819/2017. This work was also supported by the European Regional Development Fund (ERDF), through COMPETE 2020, combined with Portuguese National Funds, through *Fundação para a Ciência e Tecnologia, I.P.* [POCI-01-0145-FEDER-006910 (UIDB/EQU/00102/2020); POCI-01-0145-FEDER-007136 (UID/CTM/00264/2020)]. This work was also carried out in the frame of the COST-Action "Advanced Engineering of aeroGels for Environment and Life Sciences" (AERoGELS, ref. CA18125) funded by the European Commission.

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Abstracts of POSTER PRESENTATIONS


AERoGELS

P01

Synthesis of Starch/κ-Carrageenan/PVASbQ cryogels as potential scaffolds for drug delivery and tissue engineering applications

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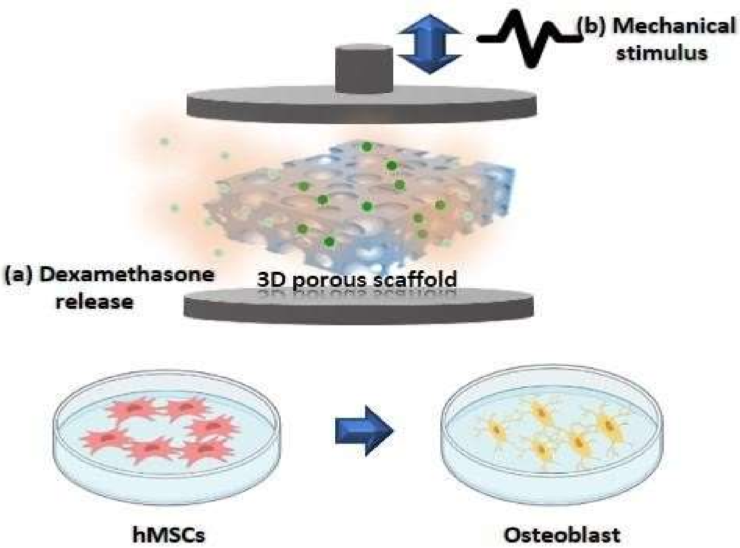
GRAPHICAL ABSTRACT



ABSTRACT

The extracellular matrix (ECM) is a three-dimensional structure which acts as structural support for cells, tissue integrity and elasticity. The diversity of ECM components and their interaction with cells receptors makes possible the regulation of cells behavior [1]-[4]. Those properties make ECM a target during cell tissue engineering studies, highlighting the interest of the development of ECM-like biomaterials for cell colonization.

Specifically, the potential biomedical application of biopolymer-based cryogels as extracellular matrices (ECM) and as drug delivery systems are of great interest due to their three-dimensional structure, composition, and the potential optimization of their physical, mechanical, and electrical properties, making possible the regulation of cells microenvironment and generating diverse biological responses [1]-[3].

In this work, starch-based cryogels were obtained by freeze drying method. κ-Carrageenan and poly (vinyl alcohol) bearing styrylpyridinium groups (PVASbQ) were incorporated to the cryogel formulation in order to evaluate their effect in the physical properties of the structure, the mechanical properties at macroscale were also studied. In general, SbQ groups acted as a crosslinking agent on porous matrices, causing an impact on the cryogel integral structure and affecting the swelling and degradation rates of the matrices after exposure to cell culture incubation conditions.

The immobilization of dexamethasone phosphate at diverse concentrations was promoted during the synthesis of the cryogel, and their presence in the final structure was confirmed using Fourier-transform infrared spectroscopy. The porous matrices containing dexamethasone were used finally as drug delivery systems. The total amount of dexamethasone released was monitored using High-Resolution mass spectrometry and micro infrared, which confirmed the release of the drug at therapeutic levels and provided the cryogels as potential systems to be used during human mesenchymal stem cells differentiation experiments. Finally, biocompatibility of the cryogels was evaluated using C2C12 cell line, the viability of the cells exposed to all scaffolds formulations was remained near of 90%, demonstrating the biocompatibility of the porous structures.

ACKNOWLEDGEMENTS

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P02

Synthesis & Characterization of Micro- and Meso-porous Carbon Microparticles by Emulsion-Gelation Method

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GRAPHICAL ABSTRACT

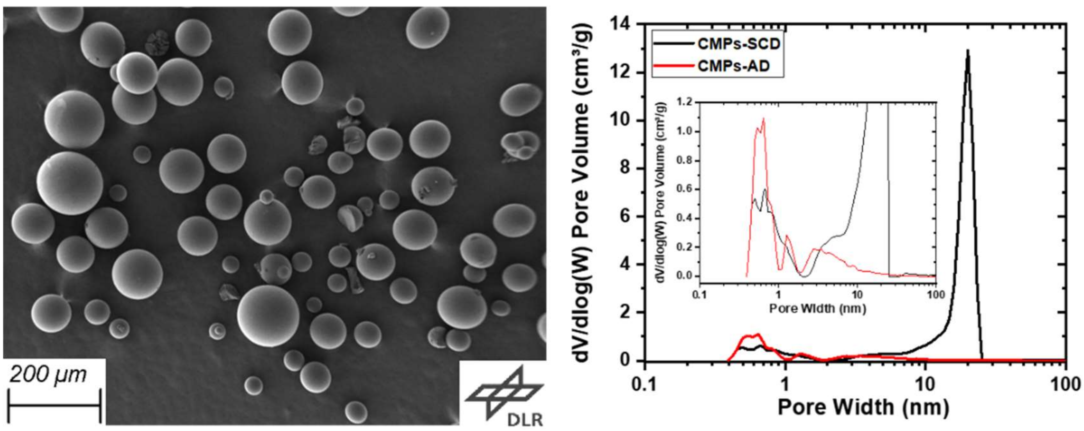

Figure: SEM image of super-critically dried microparticles (left) and DFT pore size analysis of supercritically and ambient dried microparticles (right).

ABSTRACT

Aerogels are a new class of highly porous materials, especially carbon aerogels having high surface area (400-900 m^2/g) and ultrafine pore size (< 100 nm) [1]. These porous carbon materials can be applied in electrochemical energy storage, catalysis, adsorption, fuel cells, etc [1]. Carbon monolithic aerogels are synthesized conventionally by the sol-gel polymerization of Resorcinol (R) and Formaldehyde (F) in a basic medium followed by supercritical CO_2 drying and further carbonization in an inert atmosphere [2]. Compared to carbon monoliths, microparticles have attracted several researchers over the years because of their smooth surface, mechanical strength, microstructures, high packing density and easy processability [3]. They find an extensive application in various fields such as catalysis, health care, energy, environment, electronics, etc [4].

The main objective of this work was to synthesize RF aerogel microparticles by using an emulsion-gelation method [5] followed by supercritical CO_2 drying (SCD) and ambient drying (AD) of the microparticles produced. The dried RF microparticles were carbonized under an inert atmosphere to form carbon microparticles in the size range of 150-200 μm. The as prepared carbon microparticles were characterized using different characterization methods such as gas pycnometry, N_2 adsorption isotherm, XRD and SEM. The supercritically dried microparticles have surface areas greater than 1000 m^2/g with pores in the sizes of micro- & meso- regions whereas ambient dried microparticles have surface areas of around 800 m^2/g with pores in the range of micro- region. The morphologies of the different carbon microparticles were quite similar to that of their RF microparticles [5]. Further studies are in progress to understand their properties for various applications and also to produce them in large scale.

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P03

Chitosan-silica hybrid aerogels for pollutants sorption

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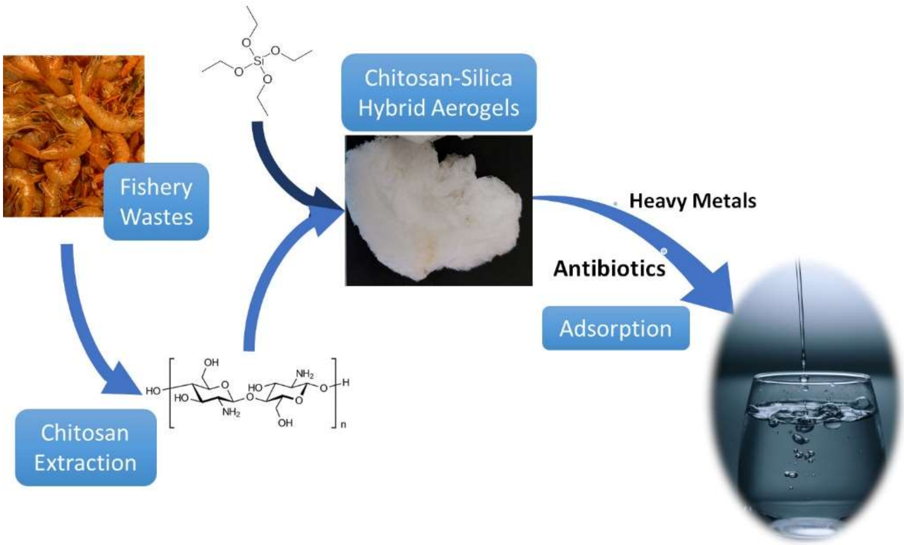
GRAPHICAL ABSTRACT



ABSTRACT

One of the current global issues refers to minimizing waste production. In this regard, wastes from agriculture and fishery, which have environmental impact, can be recycled and given new uses [1]. Antibiotics in wastewaters are not regulated in the EU, and its presence might lead bacterial pathogens to develop antibiotic resistance genes (ARGs) [2]. Moreover, heavy metals are among the most harmful non-microbial pollutants due to their toxicity to humans. These were found to be the most significant pollutants in soils and groundwater in the EU [3]. In this work, chitosan and silica are used to create hybrid aerogels for the removal of pollutants belonging to the aforementioned groups from wastewaters.

Chitosan is one of the most applied polysaccharides in hydrogel synthesis due to its antimicrobial activity, biodegradability and biocompatibility for living beings. In this work, the aerogels are obtained by reticulating the polymeric chitosan chains with silica alkoxide moieties to form cohesive gels, followed by freeze-drying. The obtained gels are characterized by a set of techniques.

Two different kinds of chitosan were tested: raw chitosan (with low molecular weight) and chitosan modified with salicylaldehyde followed by its reduction with sodium borohydride. It should be highlighted that the latter already shows a removal efficiency towards Cu(II) ions of ca. 100 %. The impact of the addition of the silica phase is evaluated, in comparison to chitosan gels. The adsorption performance of these different gels towards antibiotics (such as, tetracycline and sulphonamides) and metal ions (as, for example, copper and nickel) is evaluated through sorption isotherms and kinetics.

ACKNOWLEDGEMENTS

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P04

Development of a novel collagen-NADES aerogel

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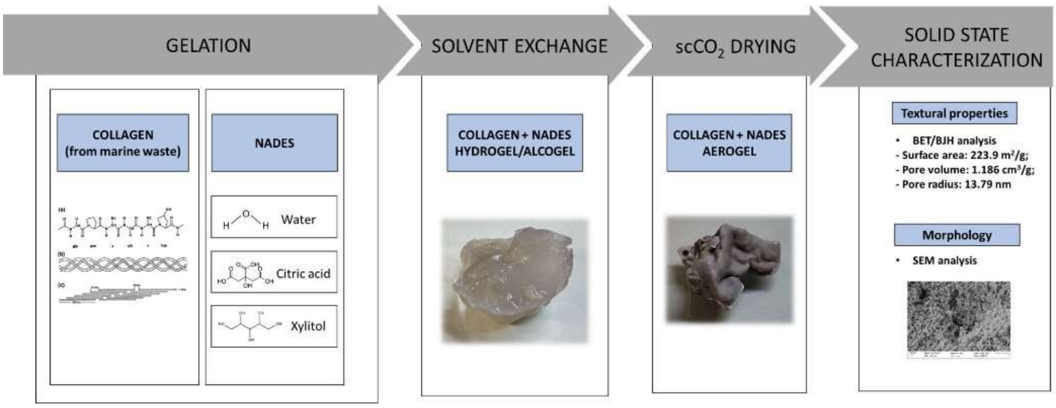
GRAPHICAL ABSTRACT



ABSTRACT

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 also 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 desired properties for a broad range of economically attractive applications [3].

This work aimed to develop a new route towards hybrid collagen-NADES aerogel and perform solid-state characterization. The influence of NADES content and solvent exchange approach on the aerogel characteristics was evaluated. To obtain the aerogel, a hydrogel of collagen and NADES was produced. Hydrogels were converted into alcogels through solvent exchange and then dried with supercritical CO_2. Once the aerogels were produced, the morphology was studied by Scanning Electron Microscopy (SEM), whereas the specific surface area and the pore volume were obtained through Brunauer–Emmett–Teller (BET) and Barrett-Joyner-Halenda (BJH) methods, respectively. The resulting aerogels were light-weight, spongy with a relatively dense meso to microporous network and a specific surface area of 223.9 m^2/g, specific pore volume of 1.186 cm^3/g and a pore radius of 13.79 nm. This work demonstrated that a novel aerogel with a spongy-like structure was successfully obtained through supercritical drying of a collagen-NADES hydrogel, with textural analysis revealing a high specific surface area and pore volume. Future works evolve biocompatibility and bioactivity evaluation of this new material, exploiting the attractive properties of aerogels, collagen and NADES for potential wound healing applications. The relationship of the material shrinkage with the aerogel's NADES content will also be studied.

ACKNOWLEDGEMENTS

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P05

Protein aerogels as functional ingredients able to replace fat and modulate lipid digestion

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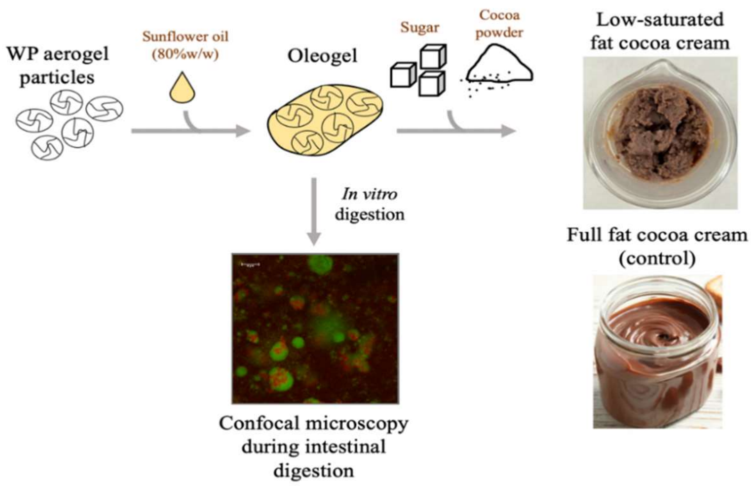
GRAPHICAL ABSTRACT



ABSTRACT

Introduction: The direct relation between saturated fat consumption and chronic diseases such as cardiovascular diseases, diabetes type II and obesity is nowadays well-established (Zhu et al., 2019). However, fat substitution in foods is not simple, due to the important structural and sensorial properties of solid fat, which are hardly replicated by liquid oil. Nevertheless, oil can be structured into semi-solid materials (oleogels) by different "oleogelation" strategies. Oleogels not only mimic the structural properties of fats, but have been also shown to be useful in the modulation of lipid digestion (Calligaris et al., 2020). The aerogel-template approach, is a recent oleogelation strategy, based on the ability of aerogels to absorb oil in their porous network. In particular, food-grade protein aerogel particles have shown the peculiar ability to structure huge oil amounts into plastic systems presenting the mechanical properties of traditional fats (Plazzotta et al., 2020). The aim of the present study was to assess the effect of aerogel-template oleogelation on lipid digestibility and to investigate the possibility to use aerogel-templated oleogels in the preparation of low-saturated fat cocoa creams.

Experimental Methods: Whey protein (WP) aerogel particles were prepared by grinding a heat-set WP hydrogel (20% w/w, pH=5.7), which was then subjected to ethanol exchange and supercritical-CO_2 drying (SCD). Oleogels were than obtained by absorption of sunflower oil (SO) into aerogel particles. Lipid digestibility of the oleogel containing 80% (w/w) SO and 20% WP aerogel (w/w) was assessed by *in vitro* digestion, according to the INFOGEST protocol (Brodkorb et al., 2019). Lipid digestibility was expressed as free fatty acids (FFA %),

assessed by pH-stat method, i.e., by measuring the volume of NaOH (0.25 M) required to maintain the pH at 8.00 during digestion occurring in the small intestine. The choice of using pH 8.00 instead of 7.00 was based on the technical specifications of the used lipase. The digestate samples were analyzed by using dynamic light scattering (DLS) and confocal microscopy. The WP aerogel particles were then used to prepare cocoa creams containing sunflower oil (SO), icing sugar and cocoa powder. Different oil amounts were tested (40-65% w/w), while maintaining constant the ratio among the dried ingredients (WP aerogel:sugar:cocoa =1.5:5:1). Additional control samples were prepared by using native WP. The obtained creams were analyzed for oil release and rheological properties and compared to cocoa spreads available on the market.

Results and discussion: WP aerogel particles were used to structure SO into an oleogel, whose digestibility was then assessed. The lipid digestibility of SO and of the oleogel resulted respectively of 70% and 80%. These results can be attributed to the ability of aerogel protein particles to improve the emulsification of oil in the intestinal digestive mixture, leading to an enhanced activity of lipolytic enzymes. DLS, in fact, evidenced that the lipidic micelles formed during intestinal digestion of the oleogel resulted significantly smaller than those formed during SO digestion. This is probably attributable to the surface activity of WP aerogels, which are able to cover and stabilize the oil droplets in the digestive mixture. The applicability of WP aerogel particles as key ingredients for the preparation of low-saturated fat cocoa creams was then demonstrated, combining WP aerogel particles with SO in presence of sugar and cocoa powder. Native WP did not show oil structuring ability, leading to liquid-like cocoa creams, showing an apparent viscosity lower than 2 Pa·s (50 1/s) and evident oil release upon resting at room temperature. By contrast, aerogel particles produced thicker creams, showing no flow under gravity, a significantly higher viscosity (50 Pa·s), and no oil release under standard storage conditions. This was attributed to the modifications undergone by WP during conversion into porous aerogel particles. The range of rheological properties covered by the WP aerogel cocoa creams resulted comparable with a wide variety of commercial products (e.g. sauces and batters).

Conclusions: This work demonstrates the potentialities of WP aerogel particles as oil structuring agents, exploitable in the formulation of healthier food products with a reduced amount of saturated fatty acids. Such formulation strategy would not compromise the lipolytic action during digestion, making aerogels suitable carriers of bioactive molecules in the gastrointestinal tract.

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