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

UNIVERSITY of DEBRECEN

Workshop on Aerogels Characterization and Modelling

29 - 31 March 2023

Debrecen, Hungary

BOOK OF ABSTRACTS

Workshop on Aerogels Characterization and Modelling

29-31 March 2023

Debrecen, Hungary

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Workshop on Aerogels Characterization and Modelling

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Welcome address

As the Chair of the Workshop on Aerogels Characterization and Modelling 2023 and on behalf of the organizing committee, I am delighted to warmly welcome you all in Debrecen for this major event held under the auspices of the AERoGELS COST Action and hosted by the Faculty of Science and Technology of the University of Debrecen.

The workshop covers the fundamental characterization, the potential applications and the functional models of porous materials in the broadest sense, it covers topics from molecular spectroscopy to surface analytical techniques, from calculations on the atomic level to process modelling. Beside assembling and integrating the most recent scientific and technological knowledge on aerogels and other nanostructured materials, the workshop also aims at strengthening and creating new ties and partnerships in the aerogel community.

In line with the mission of AERoGELS COST Action, the conference will host over 70 participants from 16 countries and 2 continents, ensuring a vibrant and productive meeting that will provide a solid background for sharing fresh ideas and initiating new collaborations. As always within the AERoGELS COST Action, PhD students and young postdoctoral researchers are key participants in this conference.

We gratefully acknowledge all authors for their contributions and all individuals, organizations and companies who have supported the conference.

We wish you all an excellent conference both scientifically and socially, and an enjoyable stay in Debrecen.

István Fábián

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ABOUT AERoGELS CA18125 COST ACTION

COST (*European Cooperation in Science and Technology*) is a funding agency for research and innovation networks. Our Actions help connect research initiatives across Europe and enable scientists to grow their ideas by sharing them with their peers. This boosts their research, career and innovation.

AERoGELS (*Advanced Engineering and Research of aeroGels for Environment and Life Sciences*) COST Action intends to bring together the knowledge on research and technology of aerogels at the European level from academia, industry and regulatory experts.

AERoGELS Action constitutes a scientific-technological platform on aerogels to generate added value solutions in terms of scientific knowledge, high-performance materials and efficient, health compliant and environmentally responsible technologies.

AERoGELS embraces more than 200 public research institutions and companies from 47 countries. The **Main Aim** of AERoGELS Action is to boost the development of aerogel-based products for biomedical and environmental applications by promoting, disseminating and sharing knowledge on aerogels technology through 5 Working Groups (WG) which are highly interlinked to reach the Action objectives.

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Extended Programme

	Wednesday 29th of March			
Time	Code	Title	Prensting author	
11:30-12:50	Registration & Lunch			
12:50-13:10	Opening ceremony			
13:10-13:50	KN-01	Modelling of aerogels: What do we know and what's next?	Ameya Rege	DE
13:50-14:10	OP-01	Scaling the Elastic Properties of Silica Aerogels: A Modelling Insight	Prakul Pandit	DE
14:10-14:30	OP-02	Tailoring organosilica aerogel based materials for application requirements	Bartosz Nowak	PL
14:30-15:10	KN-02	From lab to pilot scale: How to overcome the valley of death?	Barbara Milow	DE
15:10-15:40	Coffee break			
15:40-16:20	KN-03	Perspectives of the aerogel market	Michael O'Connor	FR
16:20-17:00	KN-04	Engineering of porous materials: from microscale to applications	Pavel Gurikov	DE
17:00-17:20	OP-03	The AEROPILs evolution: Poly(ionic liquid)-based aerogels towards CO_2 capture and conversion	Raquel V. Barrulas	PT
17:20-17:40	OP-04	Fabrication of nanoparticle agglomerate films by spark ablation and their application in surface-enhanced Raman spectroscopy	István Csarnovics	HU
17:40-18:40	Poster session			
19:30	Opening Dinner			
	Thursday 30th of March			
9:00-9:40	KN-05	Numerical modeling of kinetics of aerogel synthesis	Jakub M. Gac	PL
9:40-10:00	OP-05	Modelling and characterization of carbon aerogels	Hemangi Patel	DE
10:00-10:20	OP-06	Methyl functionality of monolithic silica xerogels synthesized via co-gelation approach combined with surface silylation	Selay Sert Cok	TR
10:20-10:40	OP-07	Nucleation-growth type models of nanoparticle formation: deterministic and stochastic approaches	Gábor Lente	HU
10:40-11:10	Coffee break			
11:10-11:50	KN-06	Evaluation of Bioaerogels for Biomedical Applications	Carlos A. Garcia-Gonzalez	ES
11:50-12:10	OP-08	In vitro assessment of Silk Fibroin Aerogel Particles loaded with Adenosine for Wound Healing	Beatriz G. Bernardes	PT
12:10-12:30	OP-09	Multiscale mechanics of native arteries and porous collagen constructs	Florian Fage	FR
12:30-13:50	Lunch			
13:50-14:10	OP-10	Characterization of alginate-based hydrogels aimed at biomedical applications	Igor Lacik	SK
14:10-14:50	KN-07	The versatility of carbon aerogels	Krisztina László	HU
14:50-15:10	OP-11	Synthesis of dual (N, S) and graphene oxide doped marine biomass derived porous carbon aerogel	Samantha K. Samaniego Andrade	HU
15:10-15:20	Anton-Paar - Company Presentation			
15:20-16:00	Coffee break			
16:00-16:40	KN-08	The electrical impedance of carbon xerogel hierarchical electrodes	Cedric J. Gommes	BE
16:40-17:00	OP-12	Application of NMR relaxation methods for aerogels and other porous materials	Mónika Kéri	HU
17:00-17:20	OP-13	K-Wave modelling of ultrasound wave propagation in aerogels and the effect of physical parameters on attenuation and loss	Firouzeh Sabri	US
17:20-18:10	Poster session	AERoGELS COST Action Workgroup Meeting		
19:30	Gala Dinner			

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	Friday 31st of March			
9:00-9:40	KN-09	In between ice crystals: correlative approaches to unveil the local pressure and composition surrounding cells during directional freezing	Francisco M. Fernandes	FR
9:40-10:00	OP-14	Preparation of 3D metal oxide nanostructures	Gergő Vecsei	HU
10:00-10:20	OP-15	Measuring the conditions of gelation of vapor-grown 1-D nanoparticles	Nabil Abomailek	ES
10:20-10:40	OP-16	Covalently immobilized copper(II) complexes as novel nanoenzymes with superoxide dismutase activity	Norbert Lihi	HU
10:40-11:10	Coffee break			
11:10-11:50	KN-10	Polyurea-crosslinked biopolymer aerogels as a versatile platform for design and synthesis of nanostructured materials for environmental applications	Patrina Paraskevoupoulou	GR
11:50-12:10	OP-17	Towards CO_2 upcycling with porous carbon materials	Marta Corvo	PT
12:10-12:30	OP-18	Silica based organic–inorganic hybrid xerogels and aerogels: synthesis, structure and applications	Zoltán Dudás	HU
12:30-13:50	Lunch			
13:50-14:30	KN-11	Thermal properties of aerogels as a function of porosity and density	Zoran Novak	SL
14:30-14:50	OP-19	Sustainable silica aerogel synthesized from waste glass via the ambient pressure drying method	Marina Borzova	NL
14:50-15:30	KN-12	Thermal stability investigations of different aerogel blankets	Ákos Lakatos	HU
15:30	Closing ceremony & Awards			
	Poster Presentations			
	CODE	TITLE	Presenting author	
	PP1	Influence of the initial synthesis chemical composition on the gelation kinetics of MTMS-based aerogels	Aleksandra M. Pisarek	PL
	PP2	Glutaraldehyde crosslinked aerogel for the selective sorption of aqueous Pd(II)	Balázs József Bukta	HU
	PP3	Microbiological and morphological characterization of bio-based aerogels after supercritical CO2 sterilization	María Carracedo-Pérez	ES
	PP4	Synthesis and characterization of gelatin, and crosslinked gelatin aerogels	Madalina Ranga	HU
	PP5	Impacts of Chitosan's Intrinsic Properties on Aerogel Structure	Serap Namli	TR
	PP6	What can liquid-phase NMR tell us about porous materials?	Vanda Papp	HU
	PP7	Small-angle neutron scattering (SANS) investigation of functionalized and hybrid silica aerogels	Zoltán Balogh	HU
	PP8	The effect of ionic liquid on the morphology and surface properties of RF carbon aerogels by NMR	Dávid Nyúl	HU
	PP9	Vinyl modified silica aerogel coated glasses for the thermal insulation applications	Fatoş Koç	TR
	PP10	Exploring thermal conductivity of aerogels through DSC analysis	Gabrijela Horvat	SL
	PP11	A simple kinetic model to explain the solubilizing spring effect in aerogel drug delivery systems	László I. Orosz	HU
	PP12	Investigation of the growth kinetics of $ZnAl_2O_4$ spinel phase in cylindrical geometry	Laura Juhász	HU
	PP13	Mechanical characterization of cellulose aerogels	Max Zinke	DE
	PP14	Thiol functionalized mesoporous silica sorbent for selective sorption of aqueous Ag(I)	Dániel Pércsi	HU
	PP15	Hydration mechanism of borosilicate-PVA aerogels	Bertold Ecsédi	HU
	PP16	Syntheses and Characterization of Flexible Polyimide Aerogels	Armela Ademi, Oyun-Erdene Odongerel	HU
	PP17	Self-sterilizing PVA electrospun membranes	Eszter Kiss	HU

KEYNOTE LECTURES

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KN-01 Modelling of aerogels: What do we know and what's next?

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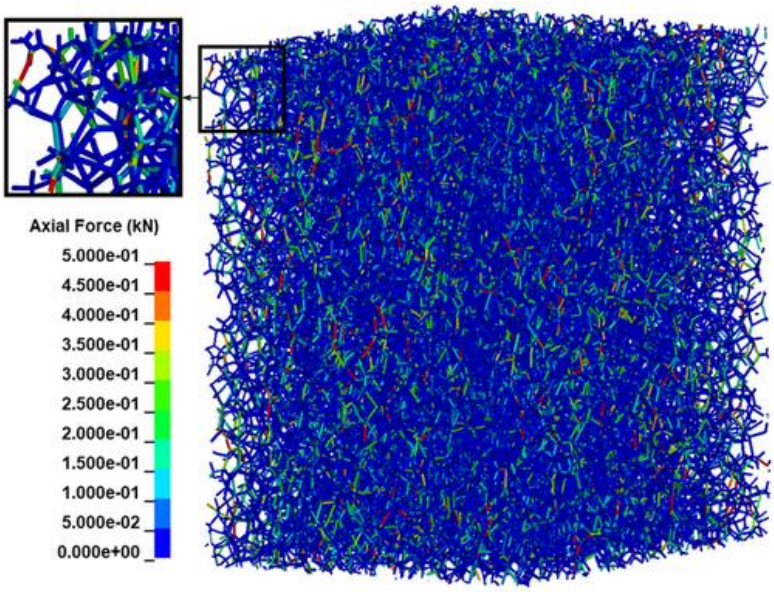

ABSTRACT

The nanostructured open-porous morphology of aerogels can be characterised by computational methods to understand their structure-property relations. Silica aerogels have been modelled by means of the diffusion-limited cluster-cluster aggregation (DLCA), a method which captures the fractal morphology of these aerogels. Does DLCA mimic the gelation process accurately? Organic aerogels have gained interest in recent times. However, very few studies have reported on their computational description. Gaussian random fields have been exploited to characterise their porous networks. How accurate are these approaches? Not all aerogels show a colloidal-like morphology. Several biopolymer aerogels demonstrate a fibrillar morphology. Radical Voronoi tessellations have proven to be very effective for designing the morphology and predicting structure-property relations. Can this approach be used for reverse engineering of the aerogel design process? What alternative approaches exist? The literature on aerogel modelling, while gaining increased significance, is still fragmented. A perspective on this subject can be found in a recent publication [1], on which this contribution will be based. Here, we shall discuss the open-ended questions and aim at drawing a path towards more clarity on the subject.

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KN-02 From lab to pilot scale: How to overcome the valley of death?

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ABSTRACT

In the meantime, aerogels have been researched for many years. A large number of applications of aerogels have been identified. Most of them contribute to the reduction of greenhouse gases and can be useful for the 1.5° Celsius target to stop climate change.

However, their commercial breakthrough has not yet been fully achieved. They still play a role almost exclusively in niche markets, or represent only a very expensive exclusive solution.

Due to their fascinating combination of properties, which can be adjusted by a "simple" sol-gel synthesis and the choice of chemical base materials, aerogels and aerogel composites should play a much larger role in the future.

Therefore, we need to figure out what challenges need to be overcome, and what is really needed to move forward.

Let us talk about the needs to be done to give aerogels a chance on the market?

The presentation will outline future challenges and discuss the approach of setting up pilot plants in a large-scale research facility as a possible solution.

Let's find out if the establishment of an Aerogel Launch Factory can really bring the necessary turnaround?

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KN-03 Perspectives of the aerogel market

Michael O'Connor

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GRAPHICAL ABSTRACT

ABSTRACT

Presentation of the Global commercial opportunity for aerogels over the next 5 – 10 years .

Focusing on the opportunities by geography, market and aerogel type.

Introduction to the Advapor association (advanced porous materials association)

An introduction to Keey Aerogel and the steps we are taking to create a new approach to aerogel production.

ACKNOWLEDGEMENTS

Source of market data IDTechex report September 2021.

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KN-04 Engineering of porous materials: from microscale to applications

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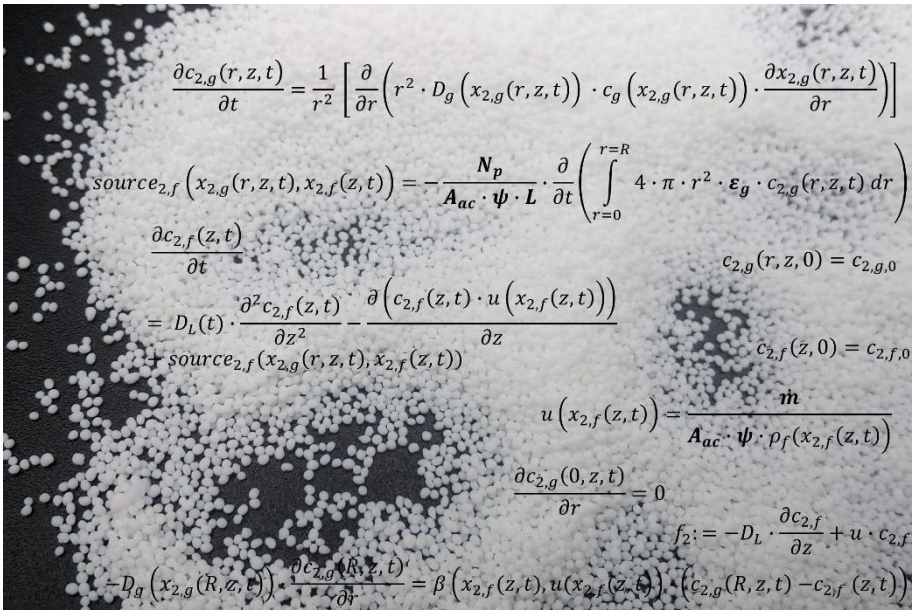
GRAPHICAL ABSTRACT



ABSTRACT

The contribution presents recent advances in the processing of biopolymer aerogels with a focus on production at a large scale and modeling of the fabrication process. We begin with a short introduction into fundamentals of the gelation [1], solvent exchange [2] and supercritical drying. We proceed with existing and emerging concepts towards large-scale production of biopolymer aerogels [3] and address the question what models are required to engineer and optimize the process. In the concluding part, we discuss open scientific and engineering questions related to the topic.

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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KN-05 Numerical modeling of kinetics of aerogel synthesis

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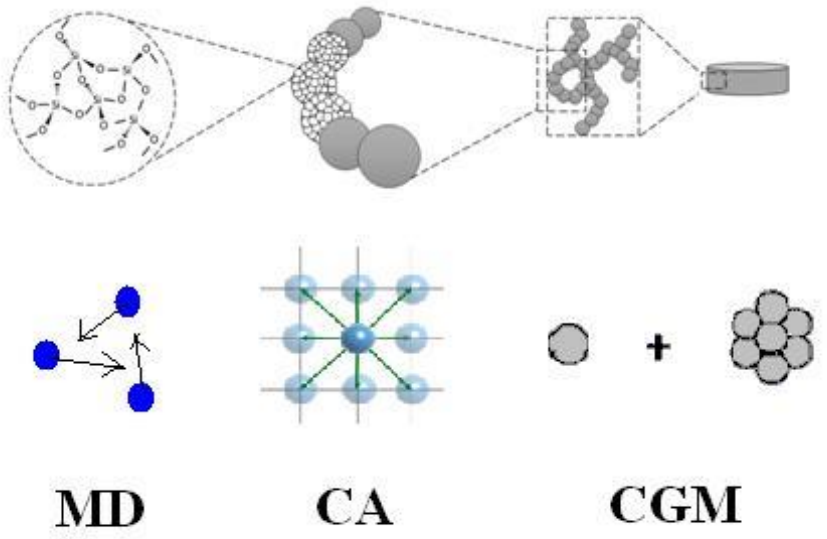
GRAPHICAL ABSTRACT



ABSTRACT

With the growing number of applications of aerogels in various industries, there was a need to improve the methods of their preparation - so as to obtain materials with strictly desired characteristics (porosity, pore morphology, surface area). It was also important to determine the expected time of the entire synthesis as well as the selection of appropriate process conditions. Obtaining this information is, of course, possible based on the results of many conducted experiments, but it is a great help to initially estimate the appropriate conditions and relationships by performing numerical simulations of the aerogel formation process or its individual stages.

The first approach to numerically describing the synthesis of aerogels was a description based on kinetic equations [1] - as is the case with many other chemical reactions. It allows to estimate the speed of the gelation reaction (and possibly its changes), as well as other parameters related to the reaction itself, e.g. activation energy. However, if a more detailed knowledge of the process itself is needed, and especially to relate the process to the

morphology of the resulting aerogel - a more detailed approach to numerical modeling is necessary.

A special feature of the structure of aerogels is their hierarchical nature. This also results in the multi-scale nature of the processes taking place, and the numerical modeling of the synthesis of these materials must also be multi-scale. Hence the need for different approaches for different spatial and temporal scales present in the process. For example, molecular dynamics (MD) can be used to consider interactions between precursor molecules and the formation of primary particles [2]; models based on the concept of cellular automata (CA) can be used to model the interactions of primary particles leading to the creation of more complex structures[3-5]; finally, to reproduce the weight gain and specific surface area development, the use of a cluster growth model (CGM) is appropriate[6].

The aim of the presented lecture will be a critical review of commonly used numerical models of aerogel synthesis, together with an indication of their place in the multi-scale process, the results that can be obtained with their help and the limitations to which individual numerical approaches are subject.

ACKNOWLEDGEMENTS

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KN-06 Evaluation of Bioaerogels for Biomedical Applications

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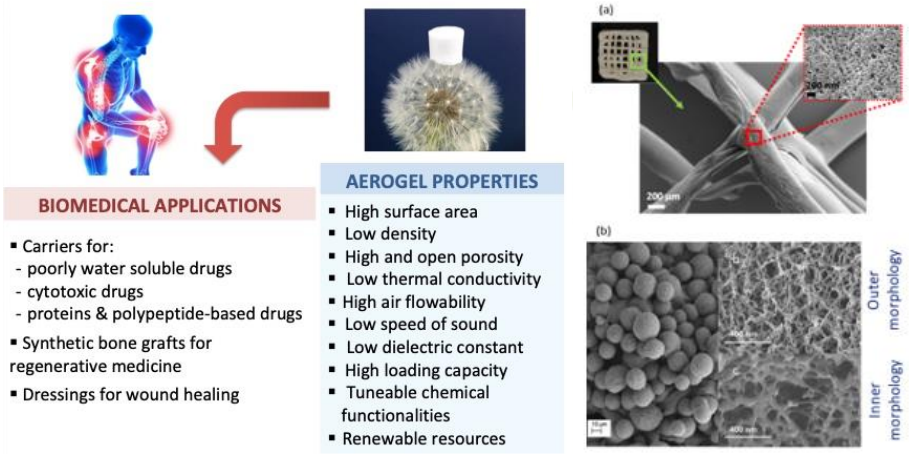
GRAPHICAL ABSTRACT



ABSTRACT

High and open mesoporosity, high specific surface area and lightweight are attractive and unique physicochemical properties of bioaerogels that can be exploited for biomedical applications, including drug delivery, wound treatments and regenerative medicine [1]. The use of aerogels as carriers of bioactive compounds may result in an increase in their solubility and in their modified release. Moreover, aerogels are particularly favorable for oral and mucosal administration routes [2,3]. In terms of production, the aerogels can be easily scaled-up using supercritical CO_2 and the manufacturing of aerogel-based drug products under good manufacturing practices (GMP) is possible. For wound treatments, aerogels have outstanding properties for wound fluid management thus contributing to haemostasis, healing and regeneration of the skin. The local release of bioactive compounds from aerogels at the wound site can be specifically used for the treatment of wounds under different healing phases [4]. Finally, for regenerative medicine, aerogels can mimic the extracellular matrix and the correct choice of the source can have relevant biological implications to promote tissue regeneration, such as the promotion of the attachment and growth of cells or the promotion of cell

differentiation towards certain lineages. The technological combination of aerogel processing with 3D-printing technology has been recently implemented to provide enhanced performances to aerogels for regenerative medicine purposes [5]. In this work, recent advances and uses of aerogels for biomedical applications are herein evaluated and discussed.

ACKNOWLEDGEMENTS

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KN-07 The versatility of carbon aerogels

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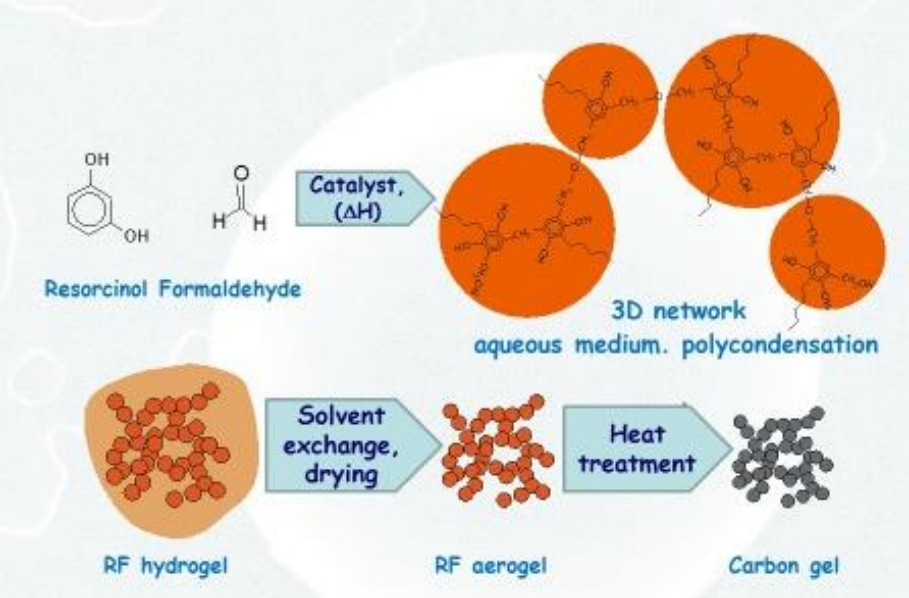
GRAPHICAL ABSTRACT



ABSTRACT

For more than two decades sol-gel techniques have been employed to prepare polymer aerogels with tailored structure, which are excellent precursors for porous carbon. The outstanding properties of carbon aerogels compared to metal oxide and polymer ones is that there are thermal insulators AND electric conductors at the same time [1]. Carbon aerogel (CA) precursors are generally obtained by catalytic polycondensation reactions between synthetic or natural (poly)hydroxy benzenes and formaldehyde, most often in aqueous medium [1–3]. Tuning the fundamental properties of the carbon precursor hydrogels is achieved by adjusting the reaction parameters: molar ratio of the reagents, pH, choice of

catalyst, inclusion of further additives, overall concentrations, temperature trajectory, and duration of aging [2–5]. An additional parameter is the chemical nature of the reaction medium [6]. Each of these factors, either individually or in combination, influences the structure of the hydrogel. An additional tool in the box of tuning parameters is the means of solvent removal, which can yield cryo-, aero- or xerogels [7]. The final form of the carbon aerogels is obtained by pyrolysis, sometimes followed by activation.
Resorcinol and formaldehyde have been the most widely used monomers. The versatility of the sol-gel technology and the auxiliary techniques used in the preparation allows fine tuning of both the pore morphology and the surface chemistry. Non-metallic (N) and metallic (Mo) heteroatoms, as well as nanoparticles (graphene oxide), can be included during the synthesis [8, 9]. Post-synthesis alterations are also straightforward. Examples to be presented range from gas storage to (photo)chemical and electrochemical catalysis [8–10].

ACKNOWLEDGEMENTS

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KN-08 The electrical impedance of carbon xerogel hierarchical electrodes

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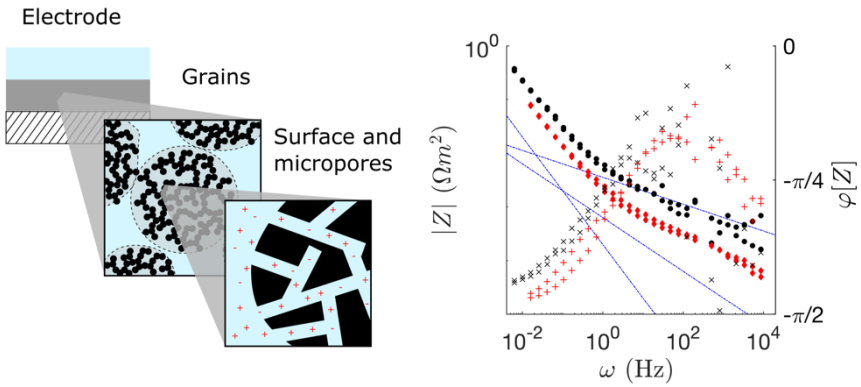

ABSTRACT

Porous materials have many applications in the field of electrochemical energy storage, notably as battery electrode materials or as supercapacitors. In this context, it is central to understand the transport and storage of electrical charges (ions and electrons) in the materials. In many practical situations, the electrode is prepared by grinding the active material into a powder and depositing a layer of it on a current collector, which leads to hierarchical structure. The largest scale is that of the macroscopic layer. The intermediate scale is that of the micrometer-sized porous grains that make up the layer. The smallest scale is that of the nanostructured skeleton of the porous grains. The storage of the electrical charges happens at the smallest scale (in micropores and on the mesoporous surface), but their transport takes places simultaneously at all scales.

We discuss electrical impedance experiments performed on carbon xerogel electrodes soaked in KCl aqueous solutions [1]. The impedance characterizes the relation between the electrical current and at an applied voltage at increasing frequencies. Low-frequency measurements allow the electrical charges to reach their equilibrium configuration, and the central electrode characteristic in that context is it capacitance. When increasing the frequency, the measurements become increasingly limited by the transport of electrons and ions.

Considering impedance over a wide range of frequency ω therefore offers a unique opportunity to study charge storage and transport in porous materials.

Specifically, we present impedance results obtained on a variety of carbon xerogels differing by their mesopore size, and by their microporous volumes. Moreover, nonane adsorption is used for the preparation of some electrodes to selectively plug micropores and make them inaccessible to the ions. We also develop a mathematical model to analyze the data, assuming electrical-double-layer (EDL) mechanism for charge storage and a Nernst-Planck equation for ion transport.

The impedance data exhibit the expected ω^{-1} scaling at low frequency, which is typical of capacitive behavior. The values of the capacitance show that the ions barely enter into the micropores, so that it is mostly the mesoporous surface area that contributes to the storage capacity. At high-frequency, a scaling of the type $\omega^{-1/4}$ is observed, which differs from the classical Warburg behavior in $\omega^{-1/2}$ that is usually expected for porous electrodes [2]. The mathematical modelling shows that this unusual exponent ¼ corresponds to a situation where charge transport is rate-limiting at two scales simultaneously, in the macroscopic layer and in the micrometer-sized grains. It is therefore unique to hierarchical structures.

ACKNOWLEDGEMENTS

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KN-09 In between ice crystals: correlative approaches to unveil the local pressure and composition surrounding cells during directional freezing

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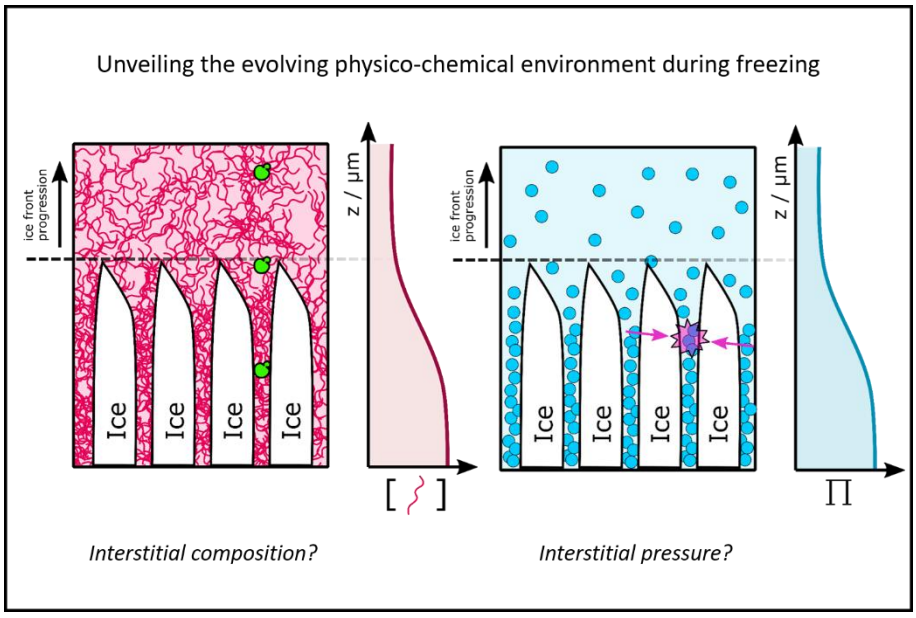
GRAPHICAL ABSTRACT



ABSTRACT

Cryopreservation is the only fully established procedure to extend the lifespan of living cells and tissues, a key to activities spanning from fundamental biology to clinical practice. Despite its prevalence and impact, central aspects of cryopreservation, such as the cells' physico-chemical environment during freezing or the pressure generated in the interstitial space in between ice crystals, remain elusive. Quantifying the pressure generated during freezing is expected to provide much needed insight into the local stress endured by cells. Similarly, being able to determine the local concentration of the freezing medium during freezing process could provide central cues to understand the fate of cryopreserved cells.

To tackle these open questions, we have recurred to directional freezing, a technique that allows to precisely define the thermal boundaries during freezing. Coupling the confocal imaging system developed by Dedovets *et al.*[1] with the phase diagram of a simple cryopreservation medium, sodium alginate, allowed us to extract its spatial distribution during

freezing, providing a tool to describe the cell vicinity at any point in cryopreservation. We show that two major events define the cells' local environment over time: the interactions with the moving ice front and with the vitreous moving front – a concept we introduced recently.[2]

In a second correlative approach, we merge data from an ice-templated-resolved *in situ* synchrotron small-angle X-ray scattering (SAXS) with data obtained from controlled adiabatic desiccation experiments. These results enabled to draw, for the first time, a quantitative picture of the local pressure in between ice crystal, that can reach the kbar range. [3]

Our results reinforce the importance of the ice front velocity – rather than the ubiquitous cooling rate – as a determinant criterion of cell viability during freezing. Moreover, being able to describe the local environment around cells during cryopreservation is expected to help designing more efficient, less toxic solutions to preserve living biological matter.

ACKNOWLEDGEMENTS

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KN-10 Polyurea-crosslinked biopolymer aerogels as a versatile platform for design and synthesis of nanostructured materials for environmental applications

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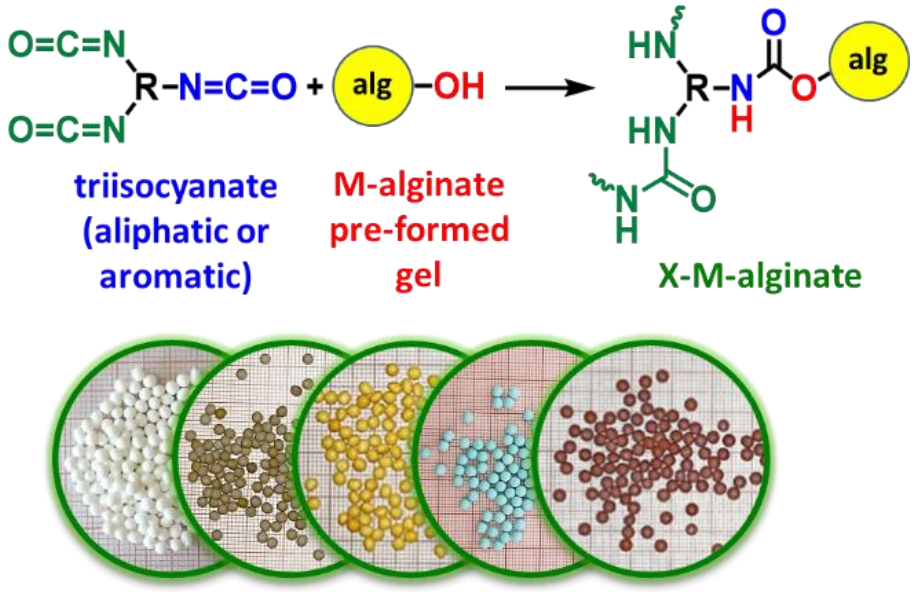
GRAPHICAL ABSTRACT



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 were rectified recently with the synthesis 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 one third of their density. They are also extremely stable in all aquatic environments with pH in the range of 3-9, including seawater and various wastewaters. These properties allow their application to environmental remediation, as adsorbents of heavy metals, or in biomedicine, as candidate materials for implants. In addition, certain X-alginate aerogels can be pyrolyzed to metal- and N-doped carbon aerogels in good yields, suitable for

applications as porous carbon electrodes and catalytic systems analogous to catalytic converters.

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KN-11 Thermal properties of aerogels as a function of porosity and density

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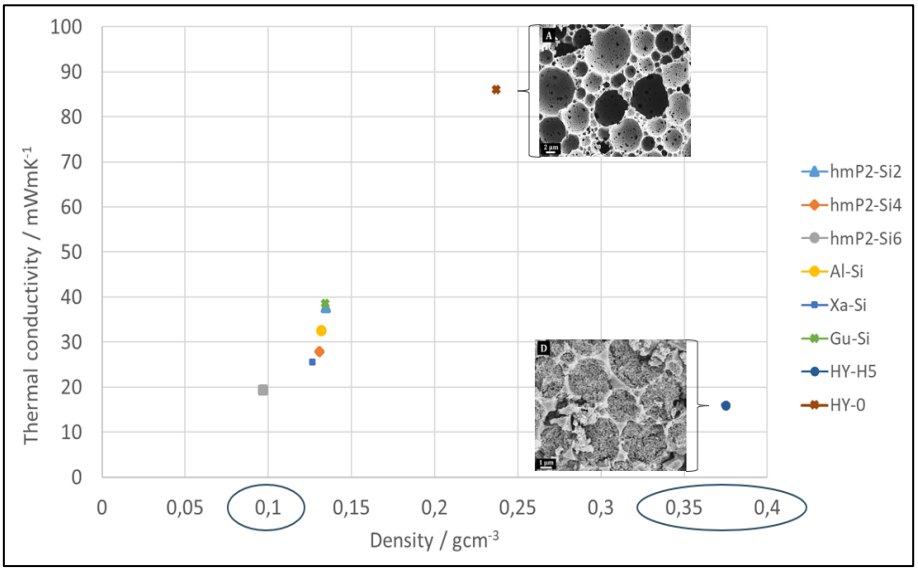
GRAPHICAL ABSTRACT



ABSTRACT

Silica aerogel is a type of aerogel, which is a highly porous and lightweight material. It is one of the lightest materials known, with a density of 0.001 g/cm^3. Silica aerogel has very low thermal conductivity, which can vary depending on factors such as the specific composition and density of the aerogel, as well as the temperature and pressure conditions in which it is used. However, in general, silica aerogel has a thermal conductivity of less than 0.020 W/mK at room temperature, much lower than traditional insulation materials like fiberglass or foam.

In fact, silica aerogel has the lowest thermal conductivity of any solid material known, which makes it an excellent insulating material for applications that require high thermal resistance with minimal thickness or weight. The drawback is that it is very fragile and brittle [1]. Its mechanical properties, such as its tensile strength, compressive strength, and elastic modulus, are much lower than those of conventional materials like metals or plastics. To overcome the brittleness of silica aerogel and improve its mechanical properties, researchers have developed several techniques, such as reinforcing it with fibers or nanoparticles, or combining it with other materials to form composites.

These methods can help to enhance the strength and durability of silica aerogel while still maintaining its low thermal conductivity and other unique properties [2,3].

Reinforcement with polysaccharide aerogels leads to mechanically stronger materials while preserving low thermal conductivity. Pectin, xanthan, alginate and guar were used for the reinforcement of silica aerogels, prepared from tetramethyl orthosilicate precursor. Hybrid materials exhibited a highly porous structure with porosity above 90%. Their surface area ranged between that of neat silica (973 m^2/g) and neat polysaccharides (103 -384 m^2/g). The surface area of guar-silica aerogels (678 m^2/g) increased the most compared to neat guar aerogel (103 m^2/g). Silica aerogel fills the macropores in the hybrid materials, thus lowering thermal conductivity, which showed to be the function of density. The lowest thermal conductivity was observed by pectin-silica aerogels (0.019 W/mK).

A promising approach for enhancing the properties of silica aerogels involves combining them with polyHIPEs. The one-pot synthesis method, in which the polymerization of monomers in the external phase and the condensation of TMOS in the internal phase occur simultaneously, was employed for the first time. The resulting hybrid polyHIPE materials exhibited significantly improved mechanical properties, with the compressive modulus reaching 98 MPa in the hybrid polyHIPE with the highest silica aerogel content. Additionally, the thermal conductivity of polyHIPE filled with 5M silica aerogel was found to be as low as pure silica aerogel (0.017 W/mK). Remarkably, this result was even lower than that of pectin-silica aerogel, despite the higher density of the material due to the weight of polyHIPEs. These findings demonstrate that both approaches can yield final materials with enhanced properties, presenting new hybrid materials that could be useful for diverse applications, such as insulation, catalysis, or energy storage.

ACKNOWLEDGEMENTS

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KN-12 Thermal stability investigations of different aerogel blankets

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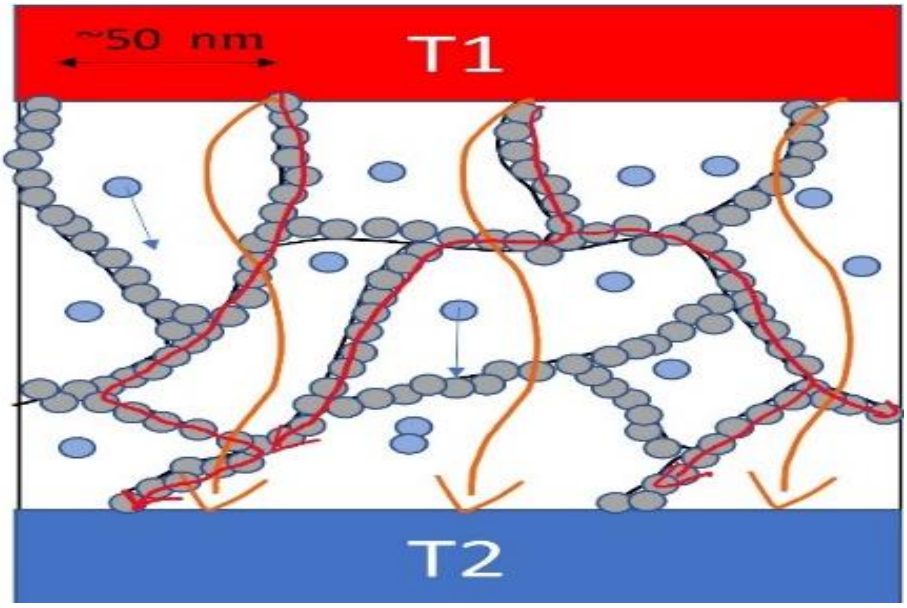

ABSTRACT

In the world, about 75% of total energy use comes from the energetic sector. Energy use can be significantly decreased by insulations in both building and vehicle sectors. Today, in many cases, it is no longer possible to use traditional insulation materials (polystyrene or wool sheets), but new solutions and new types of thermal insulation materials are needed, such as aerogel or vacuum thermal insulation panels as well as thermal insulation materials doped with graphite. Nowadays, these materials are often referred to "Super Insulation Materials" such as aerogels and vacuum insulation panels. The mentioned products have much better thermal insulation properties, but most of the long-term thermal parameters are unknown. In the presentation we will talk about the investigations of the thermal stability of different aerogel blanket samples ageing through heat treatments. The changes in both the thermal properties (thermal conductivity, specific heat capacity) and the structure will be followed with different methods [1, 2].

ACKNOWLEDGEMENTS

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ORAL SESSIONS

Workshop on Aerogels Characterization and Modelling
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OP-01 Scaling the Elastic Properties of Silica Aerogels: A Modelling Insight

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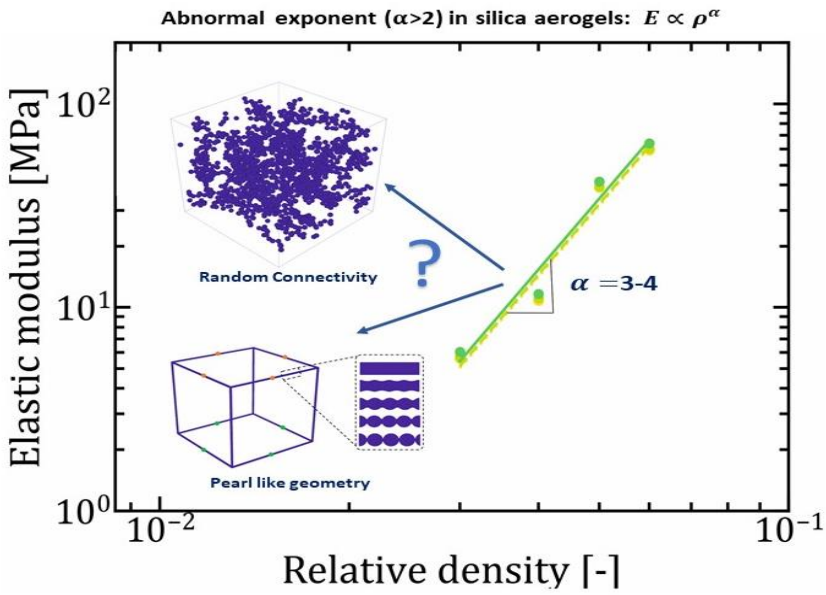
GRAPHICAL ABSTRACT



ABSTRACT

The computational modelling of silica aerogels has the potential to investigate mesoporous phenomena that are difficult to accurately probe from experimental investigations. One such phenomena, the scaling behavior of the elastic modulus to density ($E \propto \rho^{\alpha}$) in silica aerogels has been a keen research topic. Conventional open-porous materials like foams typically exhibit exponent $\alpha = 2$, however, for silica aerogels the value of the exponent α is typically observed to be between 3-4 [1]. While there have been several insights and discussions into the possible explanation behind this observed extraordinarily large exponent; the effect of spatial arrangement of the particles and the pearl-like necklace morphology of the pore wall remain unexplored. This influence can be effectively modelled with computational modelling of the silica aerogel microstructure and then performing finite element calculation for their mechanical properties. As such, while some research has focused on application of Brownian motion-based diffusion limited cluster-cluster aggregation (DLCA) algorithm to model the silica aerogel morphology [2], other works have looked into the application of ballistic cluster-cluster aggregation (BLCA) algorithms [3] for the same.

In this contribution, a hybrid cluster-cluster aggregation (HCA) model is presented to model the silica aerogel microstructure, which provides a directionality bias factor, thus a partial ballistic nature along with the pure Brownian motion. This is critical to understand the influence of ballistic motion on the morphology of the silica aerogel microstructure and its mechanical properties. With regard to the high scaling exponent observed, to understand the effect of the pore-wall structure and the random network connectivity on the scaling mechanical properties, the phenomena are investigated individually simulating compression, once by unit structure with corrugated pore walls (having different particle neck sizes) and the other by a micro structure modeled with HCA model.

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OP-02 Tailoring organosilica aeogel-based materials for application requirements

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ABSTRACT

The potential of aerogel-based materials, dormant for decades, seems to be on the verge of full expansion in many fields. Thanks to the continuous development of new precursors, exploring the phenomena accompanying synthesis and optimizing unit processes used in production, we can better control and adapt materials to the requirements of specific applications [1]. The material tailoring, however, must go hand in hand with understanding the destination process - the purpose and conditions in which it is carried out, and the resulting limitations.

Phase-Change Materials (PCM) are used in the thermostabilizing solar distillation process by absorbing excessive heat during high solar irradiance and decreasing the heat losses from the absorber to the environment. However, accompanying solid-liquid transition forces PCM immobilization, preferably in a highly-porous skeleton – assuring a high PCM loading, with controllable pore size and affinity to both PCM and water [2]. MTMS-based material hydrophobicity, flexibility and good affinity to paraffin-based PCM, obtained by the presence of methyl group, make them a suitable material for the shape-stabilization of PCM. Morphology control serves to maximize PCM loading while minimizing leakage during the solid-liquid transition.

Unique biosynthetic abilities revealed by plants determine *in vitro* cultures of hairy roots as a suitable source of pharmaceutically relevant bioactive compounds. However, biomaterial fragility and toxicity of producing naphthoquinones drive the search for bi-functional platforms, suitable for simultaneous immobilization of hairy roots and in situ extraction of extracellularly secreted bioproducts. In this context, organosilica-based aerogels with their hallmark wide range of surface modifications – both in chemical composition and morphology - allow to provide unique conjunction of such features and create an opportunity for directed design and adjustment of interactions between plant biomass, secondary metabolites and applied aerogel-based construct [3, 4].

Often nature itself sends the best ideas that only need to be reproduced in the laboratory. Almost all body tissue cells arrange in a complex 3D solid matrix with a wide distribution of various size gaps that are responsible for chemical and physical signals transmission, providing orientation in the microenvironment. Aerogel-based HIPE material combines excellent features and structure control possibilities of both emulsions templating and sol-gel chemistry. The HIPE technique allows control of the macro-pore structure by obtaining emulsion with the desired droplet size and interconnectivity, while changes in chemical composition and pH during the sol-gel reaction are used to adjust the mesoscale structure.

The aerogel-modified filters exhibit intensified surface phenomena and increased collector surface area, much needed for efficient oil-mist separation from gas streams. However, the modification conditions were designed in what would seem a contradiction to the common practice, to achieve the combination of macropores - to prevent extensive airflow resistance, with mesoporous structure deposited only on the fibre's surface [5]. Understanding the behaviouristic model of filtration dynamics was used to propose a multi-layer separation material [6].

Regardless of the application, the adjustment procedure ought to result from process requirements understanding and be well-grounded in the basic research on aerogel synthesis.

ACKNOWLEDGEMENTS

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OP-03 The *AEROPILs* evolution: Poly(ionic liquid)-based aerogels towards CO_2 capture and conversion

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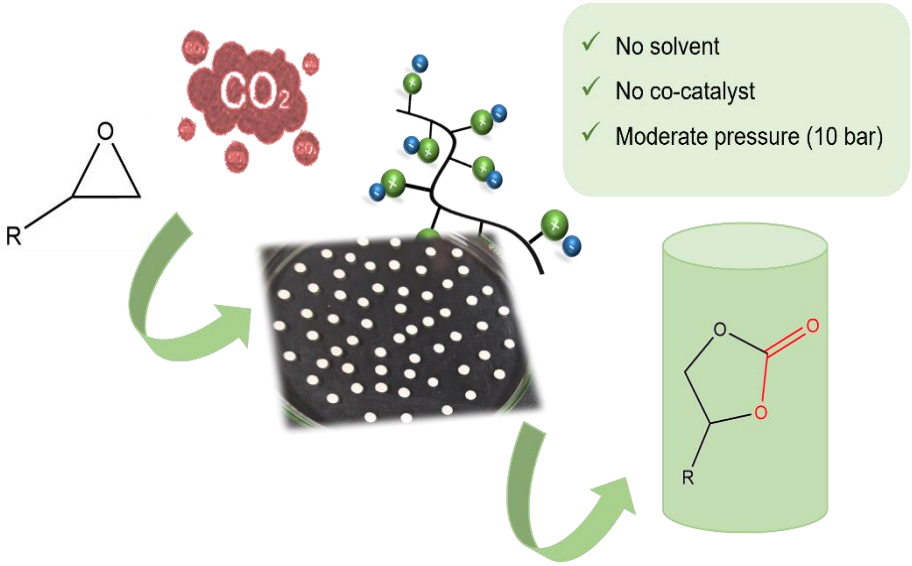

ABSTRACT

The growing need for climate change solutions requires improved CO_2 capture 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 through the production of aerogels from chitosan and poly (ionic liquid)s (PILs), introducing a design focused simultaneously on the morphology and the chemical identity of the CO_2 sorbent/catalyst [2,3].

PILs derive from ionic liquid (IL) monomers comprised of organic cations with organic or inorganic anions, whose properties are tunable towards the final applications. PILs combine the unique characteristics of ILs with a macromolecular framework and have been in the spotlight for a plethora of applications [2]. They can enhance CO_2 sorption capacity, but tailoring the porosity is still a challenge. Aerogels are light-weight nanostructured materials with high porosity and specific surface area, that can be obtained from biopolymers such as chitosan, using a biomass residue as starting material [4,5]. *AEROPILs* in the form of beads were effectively obtained with high porosity (88.1-97.0 %) and surface areas (183-744 m^2/g), which were then applied for the first time 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 in its infancy, *AEROPILs* showed very promising catalytic activity towards CO_2 addition to epoxides, at relatively low pressure and in the absence of solvent and co-catalyst.

ACKNOWLEDGEMENTS

This research was funded by National Funds through FCT – 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 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 funding from European Union's Horizon 2020 research and innovation programme 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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Workshop on Aerogels Characterization and Modelling
29-31 March 2023
Debrecen, Hungary

OP-04 Fabrication of nanoparticle agglomerate films by spark ablation and their application in surface-enhanced Raman spectroscopy

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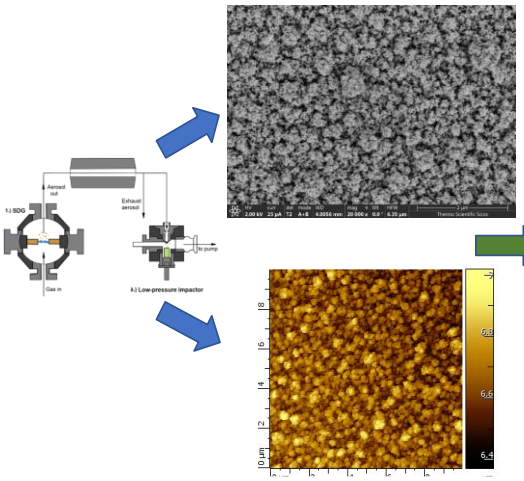
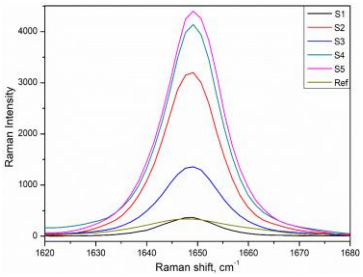


The experimental setup was used for the fabrication of NP films by spark ablation

SEM and AFM images of the created Au-Ag nanostructures



Raman spectra of rhodamin 6G on different Au-Ag nanostructures and on pure glass

ABSTRACT

This paper presents a systematic study of the investigation of nanoparticle (NP) agglomerate films fabricated via depositing spark-generated Au, Ag, and Au/Ag NPs onto quartz microscope coverslips in a low-pressure inertial impactor. The primary focus of the study is to characterize these nanostructures and to examine their potential application in surface-enhanced Raman spectroscopy (SERS). The characterization of the produced nanostructures was carried out by performing optical absorbance measurements, morphology, and composition analysis, and also testing the SERS performance of the NP films at three different excitation laser wavelengths in the visible range. The study aims to investigate the relationship between the optical properties, the morphology, and the enhancement of the produced samples at different excitations, and the results are presented

and discussed. The study highlights the potential of using spark ablation and inertial impaction-based deposition as a method for producing nanoparticle films for SERS.

In the present work, we used the spark ablation technique to produce Au, Ag, and Au/Ag nanoparticle agglomerates and applied a low-pressure inertial impactor to deposit them onto quartz surfaces to fabricate NP films. The produced films were characterized in terms of their morphology, composition, and optical properties. The fabricated nanostructures were also applied in surface-enhanced Raman spectroscopy, by measuring the Raman spectra of Rhodamine 6G as a probe molecule. The average composition of the fabricated nanostructures and the wavelength of the excitation laser was varied to characterize the enhancement properties of the nanostructures at different gold-to-silver ratios. It was found that the samples fabricated via the deposition of Au/Ag NPs of different compositions not only differ in their optical properties (i.e., their absorbance spectrum) but there is a slight variation in the average surface roughness of the structures as well. Therefore, these factors could both contribute to the observed surface enhancement of the different samples. As an attempt to decouple the effect of the surface roughness from the material-related optical properties of the samples, spatially resolved Raman mapping of the substrates was carried out at different excitation wavelengths. From the resulting data, the contribution of surface roughness-related effects to the total enhancement was estimated and found to be ca. 2 to 30 times smaller than that of the effect of average composition depending on the excitation wavelength.

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 research leading to these results has received funding from the TKP2021-NVA-19 project. Petra Pál and Viktória Horváth are grateful for the support from the ÚNKP-22-3-II-DE-53 and the ÚNKP-22-3-SZTE-410 projects, respectively, of the New National Excellence Program of the Ministry for Culture and Innovation from the source of the National Research, Development and Innovation Fund. Istvan Csarnovics is grateful for the support of the János Bolyai Research Scholarship of the Hungarian Academy of Sciences (BO/348/20) and the support through the New National Excellence Program of the Ministry of Human Capacities (ÚNKP-22-5-DE-407). Attila Kohut is grateful to the Ministry of Innovation and Technology of Hungary for the funding provided by the National Research, Development, and Innovation Fund under the PD_21 OTKA funding scheme (PD 139077 project).

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OP-05 Modelling and characterization of carbon aerogels

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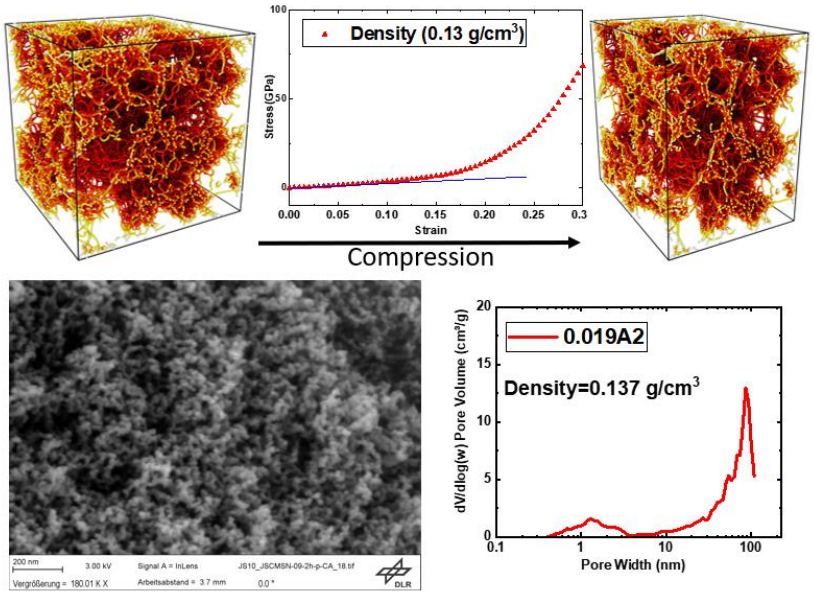
GRAPHICAL ABSTRACT



ABSTRACT

Carbon aerogels are highly open-porous solid materials in which a gas occupies more than 90% of their volume. Therefore, they show a low density, a large inner surface area, a high pore volume and high electrical conductivity. These properties make carbon aerogels an excellent candidate for a cathode material for metal-sulphur batteries. The conversion reaction of sulphur and the formation of Li_2S cause a large volume expansion, which results in a reduction of electron transport paths and thus a decrease in kinetics [1]. Flexible carbon aerogels with tailored microstructure can accommodate volume expansion due to their ability for reversible deformation up to a certain degree during cycling, thereby resulting in high-performance cells [2]. In this work, we present first experimental results on the synthesis and characterization of stiff and flexibel carbon aerogels. Carbon aerogels are synthesized by carbonization of resorcinol-formaldehyde aerogels produced by sol-gel process. Density, pore

size and inner surface area of these aerogels are measured to study their potential applications in metal-sulphur batteries.

First look into the methodology and results on a first-of-its-kind molecular study on a stiff carbon aerogel-like nanostructures will also be presented. Molecular dynamics (MD) studies present a detailed insight into the atomic-scale phenomena that underlie the formation of the porous network of carbon aerogels. The AIREBO potential [3], which is shown to accurately interpret carbon-carbon interactions, is used to simulate the nanoporous carbon network. The mechanical properties of these initial carbon networks are studied under uniaxial compression.

ACKNOWLEDGEMENTS

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Workshop on Aerogels Characterization and Modelling
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OP-06 Methyl functionality of monolithic silica xerogels synthesized via co-gelation approach combined with surface silylation

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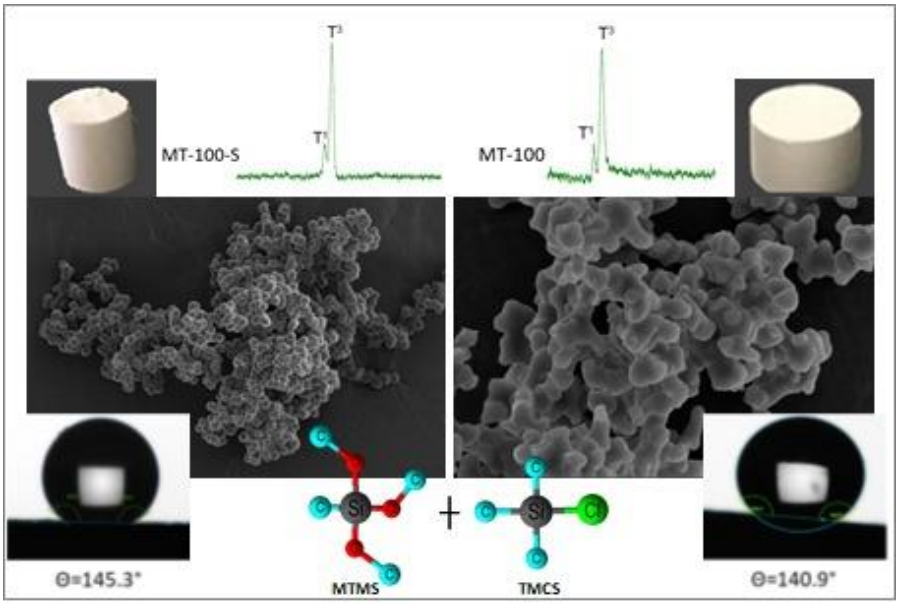
GRAPHICAL ABSTRACT



ABSTRACT

Silica-based xerogels are sol-gel derived porous materials synthesized in ambient conditions. Depending on their morphology, they can combine many exceptional properties such as high apparent surface area, low density, high porosity, and low thermal conductivity, and therefore, they have been proven to be promising materials in diverse applications [1,2]. However, the moisture sensitivity of the xerogels is one of the major obstacles that hinder the practical applications of these materials. One can obtain hydrophobic silica aerogels either by performing the silylation before gelation or by the co-precursor method. In both strategies, organically modified silica precursors (alkylsilanes or arylsilanes (of the type R4-xSi(OR')x)) are served as chemical modifiers of the silica particles derived from silicon alkoxide ($Si(OR')_4$)) precursor. Although it can be a powerful strategy to combine both in-situ (co-precursor method) and ex-situ silylation for an effective methyl substitution to the xerogel network, the

existing studies on this issue are very limited [3-5]. The possible synergetic effect of combining these two approaches is still vague and is needed to be explored systematically. This study was performed to analyze the degree of methyl substitution of silica based xerogels synthesized via co-gelation approach in achieving monolithic structure and to understand the synergetic impacts of co-gelation and ex-situ surface silylation methods on the surface and structural properties of these silica xerogels. For this purpose, a trifunctional organosilane methyltrimethoxysilane (MTMS) were utilized as silica co-precursor along with traditionally used tetraethylorthosilicate (TEOS). To investigate the changes in the materials' morphology and surface characteristics with the methyl substitution, the molar ratio of MTMS/TEOS was varied, and performing extra silylation with a monofunctional silane (trimethylchlorosilane (TMCS)) was also studied as a second parameter. Results have revealed that silylation has a direct effect on enhancing the methylation of the silica surfaces both confirmed by FTIR and ^{29}Si-MAS-NMR analyses. SEM micrographs showed that by adjusting the MTMS content, phase separation tendency and thus the macroporous structure of the material can easily be tuned. When the methyl content has increased, texture of the xerogels has changed from micro-/mesoporous nature to hierarchical macro/mesoporous nature as proven by the N_2 sorption analysis. In the samples containing MTMS as a single or major precursor, crosslinking degree and network compactness have significantly been reduced (MT-75, MT-100, MT-75-S and MT-100-S) and the samples have been obtained in a monolithic form with very low densities. Wettability studies have also supported the surface methylation with high contact angle values (>140°). High hydrophobicity, lightweight and monolithic form of these four samples can make those materials practical candidates for potential environmental and biomedical applications in future.

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OP-07 Nucleation-growth type models of nanoparticle formation: deterministic and stochastic approaches

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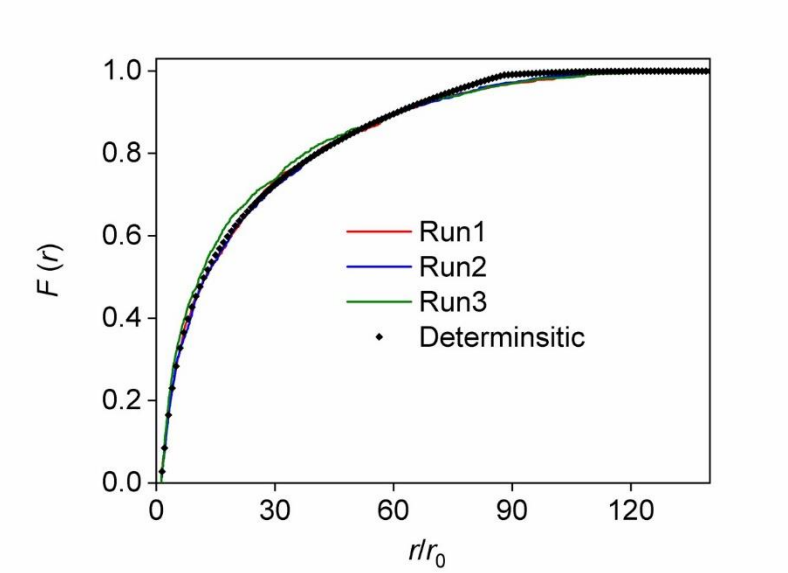
GRAPHICAL ABSTRACT



ABSTRACT

A kinetic model describing nanoparticle formation is presented using both the deterministic and stochastic approaches. The model starts from a monomer unit (M), two of which combine in a slow second-order seed formation reaction. The other process is second-order particle growth between a particle (C_i) and a monomer unit, the rate constant of which is proportional to the mass of the growing nanoparticle:

$$n\mathrm{M} \xrightarrow{k_n} \mathrm{C}_n$$

$$\mathrm{C}_i + \mathrm{M} \xrightarrow{k_{g,i}} \mathrm{C}_{i+1} \qquad i \geq n \qquad k_{g,i} = K(i)k_g$$

Here n is the number of monomeric units necessary for the formation of a viable seed, $K(i)$ is a kernel function that characterizes the size dependence of the reactivity of a growing nanoparticle. Four different kernels were employed: $K(i) = i$ mass kernel, $K(i) = i^{2/3}$ surface kernel, $K(i) = i^{1/3}$ Brown kernel, and $K(i) = 1$ diffusion kernel. Exact analytical solutions were derived for the time dependence of the concentrations [1-2] of all different kinds of nanoparticles in this model for $n = 1$, and 2 of the mass kernel, and $n = 1$ of the diffusion kernel. For example, for $n = 2$ of the mass kernel, the solutions is:

$$[\mathrm{M}]=\frac{[\mathrm{M}]_0 2k_\mathrm{g}^{i-2}k_\mathrm{n}^2(i-1)!(-1)^{i-1}\left(e^{-k_\mathrm{g}[\mathrm{C}_1]_0 t}-1\right)}{\left[2k_\mathrm{n}+\left(k_\mathrm{g}-2k_\mathrm{n}\right)e^{-k_\mathrm{g}[\mathrm{M}]_0 t}\right]\prod_{j=2}^{i}\left(2k_\mathrm{n}-k_\mathrm{g}j-k_\mathrm{g}\right)}+$$

$$[\mathrm{M}]_0\sum_{j=2}^{i}\frac{k_\mathrm{n}\left(j^2-1\right)}{\left(2k_\mathrm{n}-k_\mathrm{g}j-k_\mathrm{g}\right)j}\binom{i-1}{j-1}(-1)^j\left[\left(\frac{k_\mathrm{g}}{2k_\mathrm{n}+\left(k_\mathrm{g}-2k_\mathrm{n}\right)e^{-k_\mathrm{g}[\mathrm{C}_1]_0 t}}\right)^{k_\mathrm{g}j/\left(2k_\mathrm{n}-k_\mathrm{g}\right)}-1\right]$$

In this case, the cube-root number-average size of the nanoparticles can also be obtained analytically:

$$\langle r\rangle = r_0\sqrt[3]{\frac{k_\mathrm{g}-2k_\mathrm{n}}{-k_\mathrm{n}+\frac{2k_\mathrm{g}k_\mathrm{n}+k_\mathrm{g}\left(k_\mathrm{g}-2k_\mathrm{n}\right)e^{-k_\mathrm{g}[\mathrm{C}_1]_0 t}}{2\left(k_\mathrm{g}-2k_\mathrm{n}\right)\left(1-e^{-k_\mathrm{g}[\mathrm{C}_1]_0 t}\right)}\ln\left(\frac{k_\mathrm{g}}{2k_\mathrm{n}+\left(k_\mathrm{g}-2k_\mathrm{n}\right)e^{-k_\mathrm{g}[\mathrm{C}_1]_0 t}}\right)}}$$

For other kernels and *n* values, approximation methods were developed for handling the model in the deterministic approach [3-4]. Stochastic simulations on the molecular level (with the Gillespie algorithm[5]) were employed to validate the approximations developed here [3-4].

ACKNOWLEDGEMENTS

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Workshop on Aerogels Characterization and Modelling
29-31 March 2023
Debrecen, Hungary

OP-08 *In vitro* assessment of Silk Fibroin Aerogel Particles loaded with Adenosine for Wound Healing

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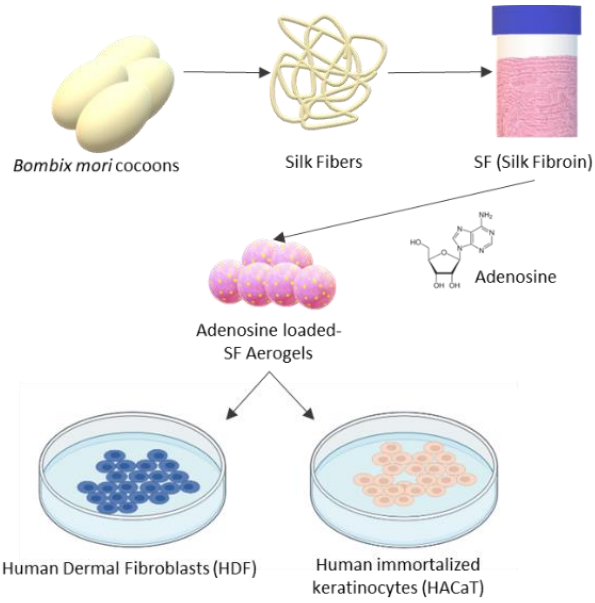
GRAPHICAL ABSTRACT



ABSTRACT

Chronic wounds represent a significant challenge in the fields of healthcare and therapy. These types of wounds do not heal within a typical timeframe and require a specific and targeted approach to their treatment. Bio-based aerogels, from natural polymer sources, can provide advanced performance for wound healing due to their high porosity and large surface area, which can be tailored for a fast and directional fluid transfer of the exudate; also, they

can act as carriers for bioactive compounds.[1] Silk fibroin (SF) protein is an excellent biomaterial for aerogel production. It can serve as a carrier of bioactive compounds while supporting cell proliferation, being presently used in wound healing and regeneration. Keratinocytes and fibroblasts are two types of cells that play important roles in the process of wound healing and regeneration, assisting in the construction of a new matrix and skin tissue repair.[1,2] Keratinocytes are the most abundant type of skin cells and can be found in the external layer of the skin.[2] Fibroblasts are connective tissue cells that are responsible for collagen production.

In this work, we study the *in vitro* behaviour of Human Dermal Fibroblasts (HDF) and Human immortalized keratinocytes (HACaT) when in contact with SF aerogel particles loaded with Adenosine (ADO). ADO is a nucleoside that promotes angiogenesis and regeneration.[3] For particle production, different concentrations of SF (3%, 5% and 7%(w/v)) and different ratios of ADO were used. ADO and ADO-loaded aerogel particle biocompatibility was evaluated by direct contact with HDFs and HaCats. Quantitative data were subjected to an analysis of variance (one-way ANOVA, Tukey's test; α=0.05). Different concentrations of ADO (0.1-2 mg/ml) were used to understand the viability of cells. Preliminary findings suggest that as the concentration of ADO increases, it enhances the cellular growth of HDF. However, for HaCat, the lowest concentrations of ADO promote cellular growth. The preliminary results also indicate that the ADO-loaded aerogel particles exhibit similar behavior. Considering the antagonistic behavior of the two cell lines in response to ADO, it is plausible that the developed particles have the potential to treat both deep and shallow wounds, depending on the dosage of ADO incorporated in each particle. In the future, further tests will be carried out by studying the particles in endothelial cells.

ACKNOWLEDGEMENTS

This research was funded by MICINN [PID2020-120010RB-I00], Xunta de Galicia [ED431C 2020/17], Agencia Estatal de Investigación [AEI] and FEDER funds. This work was also supported by National Funds from Fundação para a Ciência e a Tecnologia (FCT), through project UID/Multi/50016/2020, Doctoral Research Grant 2021.05717.BD. Work carried out in the framework of the COST Action CA18125 "Advanced Engineering and Research of aeroGels for Environment and Life Sciences" (AERoGELS), supported by COST (European Cooperation in Science and Technology); and project TEX4WOUNDS (POCI-01-0247-FEDER-047029), financed under the Incentive System for Research and Technological Development, R&DT Projects in co-promotion (Notice SI/17/2019).

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OP-09 Multiscale mechanics of native arteries and porous collagen constructs

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GRAPHICAL ABSTRACT

ABSTRACT

Atherosclerosis is the leading cause of death worldwide[1]. If not diagnosed and correctly treated, it can lead to ischemia and to irreversible damages to the non-vascularized organs. However, synthetic materials currently used to replace the defective vessels fall short in terms of their biointegration, leading to reduced endothelialisation and thrombosis. Another major drawback of currently used materials is the mismatch between their mechanical properties

and those of the native vessels, causing major complications like intima hyperplasia. In fact, while the stress-strain-curve in synthetic grafts is linear and the grafts are relatively stiff, native blood vessels are soft and have a non-linear, strain-stiffening mechanical behaviour.
Here we will report the development of a common tool to probe the mechanical response of native arteries but also those of newly designed macroporous collagen grafts developed by our team. In particular we will discuss the macroscopic properties of these materials such as the radial compliance, circumferential and axial Young's modulus, pressurized burst pressure — both in static and pulsatile conditions — and the suture retention strength. These developments allowed to decouple luminal pressure from axial elongation by using a feedback loop, thus reproducing the physiological constraints endured by the materials after implantation. Finally, we will refer to the local properties of native arteries and those of the macroporous collagen grafts using atomic force microscopy and bioindentation, in order to capture the mechanics of the cellular environment, a key factor behind cellularisation.
Our findings show that macroporous collagen grafts have not only similar stiffness to native arteries but also share their characteristic non-linear, strain-stiffening behaviour. Finally, we will discuss the role of porosity induced by ice templating in regulating stiffness of biomimetic vascular grafts at different scales.

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Workshop on Aerogels Characterization and Modelling
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OP-10 Characterization of alginate-based hydrogels aimed at biomedical applications

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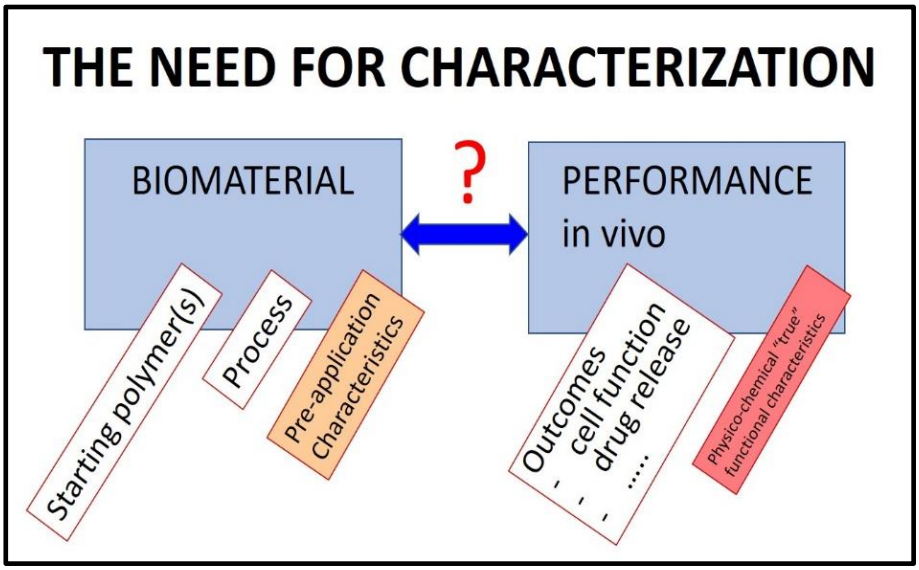
GRAPHICAL ABSTRACT



ABSTRACT

The premise of this contribution is to convey the message that the functional characteristics of alginate-based biomaterials need to be better recognized.

The alginate-based biomaterials, hydrogels as well as aerogels, are based on the electrostatically stabilized network. The number and position of crosslinks fluctuate with time and depend on the type of the environment to which a biomaterial is placed. Then, the functional characteristics, i.e., characteristics determining the *in vivo* performance in case of cell therapies, drug delivery, tissue engineering and others, may significantly differ compared to the characteristics determined after preparation or after *in vitro* conditioning.

The network of alginate-based biomaterials upon implantation is challenged from the outside by the host factors, including ions, proteins and cells, and from the inside by an encapsulated cargo. These factors influence the electrostatic interactions and the network experiences re-distribution of polymers. This has been surprisingly neglected in the development of

biomaterials for clinical applications. Consequently, even nowadays, the correct interpretation of proper performance and/or failures cannot be done properly.

The study subject of this contribution are the alginate-based microspheres developed for immunoprotection of transplanted cells in diabetes treatment. Our laboratory has been active in this topic for a number of years with a globally recognized expertise on polymer chemistry, process of cell encapsulation and characterization of alginate-based microspheres [1-3].

Table 1 lists the experimental methods for characterization of microspheres, which are carried out on microspheres in the physiological environment, as any pre-treatment may lead to artefacts due to the polymer network re-arrangement. These methods provide the information on macro up to nanoscale characteristics with respect to surface, chemical and mechanical stability, permeability, and spatial distribution of polymers. This information is then used for (i) correlation between the process conditions, (ii) understanding of the mechanisms controlling the microsphere formation, and (iii) for optimization of microsphere properties for a given application. This contribution will devote a special attention to determination of the spatial distribution of polymers, the characteristic responsible for chemical and mechanical stability, molar mass cut-off, diffusion properties, and cell microenvironment, by recently implemented confocal Raman microscopy [4].

Table 1. *Methods for characterization of alginate-based microspheres under physiological conditions*

METHOD	OBTAINED INFORMATION	
Optical microscopy	Size, shape	
Atomic force microscopy	Surface topography and local mechanical properties	surface
Zeta potential	Surface charge	
Texture analyzer	Mechanical stability (compression resistance)	stability
Osmotic stress / explosion assay	Resistance against complex destabilization	
Inverse size-exclusion chromatography	Permeability, molecular weight cut-off and pore size distribution	permeability
Confocal laser scanning microscopy	Polymer spatial distribution and diffusion of labeled compounds of interest	spatial distribution of polymers
Confocal Raman microscopy	Polymer spatial distribution	

In conclusion, most of these methods are applicable for characterization of explanted biomaterials. Thus, there are no barriers to understand the functional properties of this class of biomaterials developed for current and future biomedical applications.

ACKNOWLEDGEMENTS

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Workshop on Aerogels Characterization and Modelling
29-31 March 2023
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OP-11 Synthesis of dual (N, S) and graphene oxide doped marine biomass derived porous carbon aerogel

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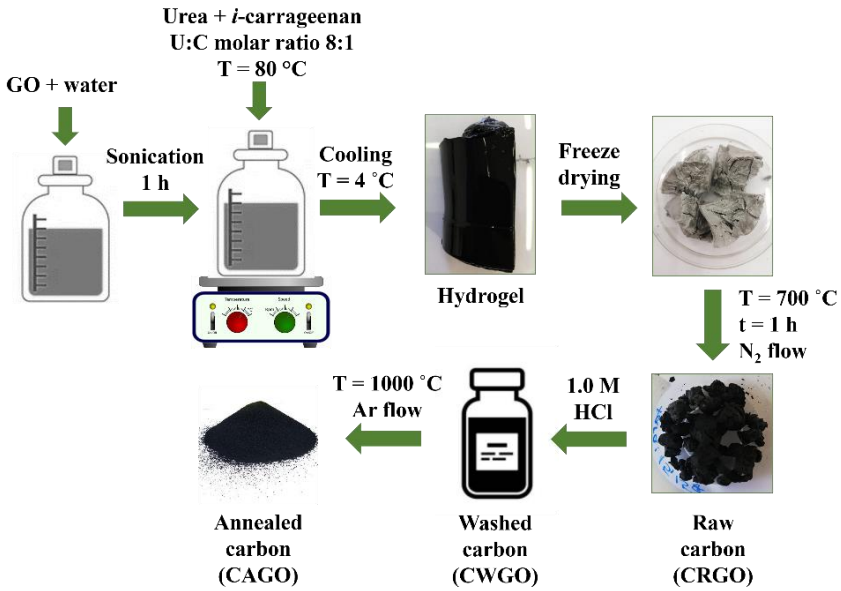
GRAPHICAL ABSTRACT



ABSTRACT

Exhaustive research has been done in the last decades to explore novel energy sources which are as or more efficient than the non-renewable fossil fuels and less dangerous to our planet. Such sources must be abundant, renewable, and not expensive and here is where biomass materials fit very well. Biomass residues such as seaweed derivatives, rice husk, crops residues, food waste, etc. offer great potential as prominent precursors in the fabrication of high-performance porous carbon materials. The outstanding microstructure and surface properties of biomass-derived carbons make them promising materials for their application in

fuel cells [1 - 2], supercapacitors [3], alkaline ion batteries [4]. Compared to terrestrial biomass, marine biomass offers also great potential, but it has been less studied despite its abundance. N-containing chitin, or S-containing seaweed derivatives i.e., ι-carrageenan could serve perfectly to synthesize N or S self-doped porous carbons [5-7].

The outstanding properties of graphene such as surface area, high electrical and thermal conductivity and good mechanical strength are considered for the development of electrode materials needed in the above-mentioned applications. Due to its hydrophilicity, graphene oxide (GO) has been used previously as platform for preparing N-doped graphene-like catalysts where urea is added as N doping agent, and GO is easily dispersed in aqueous media [8]. Later in the synthesis, GO is converted to reduced graphene oxide (rGO) and partly recovers the desired sheet-like structure and physico-chemical properties of graphene.

In our work, the synthesis of a dual doped N, S marine biomass derived porous carbon was studied. Moreover, the effect of adding rGO onto the porous carbon was investigated. Briefly, the dual doped N, S carbon aerogel was obtained from the mixture of 2 g urea and 2 g ι-carrageenan with 100 mL of water at 80 °C. The obtained hydrogel was freeze-dried and pyrolized at 700 °C (20 °C/min) in dry N_2 flow (25 L/h) for 1 h. After removing the inorganic impurities with aq. 1.0 M HCl, the sample was annealed at 1000 ˚C in Ar flow for 1 h. To study the effect of rGO, a 100 mL GO aqueous suspension (containing 50, 100 and 200 mg GO, respectively) was used instead of the initial 100 mL water. The resulting carbon aerogels were labeled as CA, CAGO50, CAGO100 and CAGO200.

ACKNOWLEDGEMENTS

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Workshop on Aerogels Characterization and Modelling
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OP-12 Application of NMR Relaxation Methods for Aerogels and Other Porous Materials

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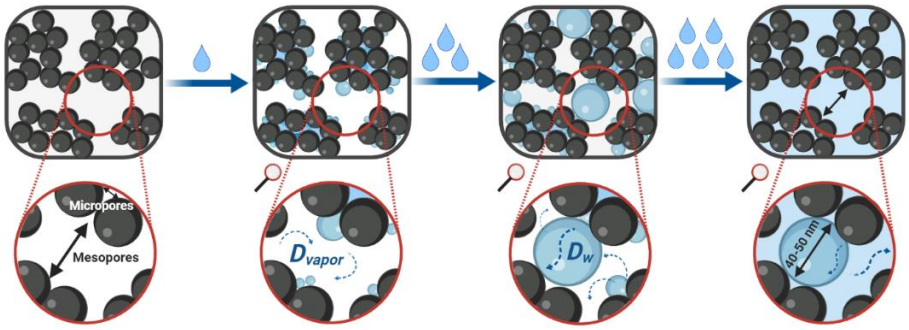
GRAPHICAL ABSTRACT



ABSTRACT

There are several well-known, conventional techniques for the structural characterization of aerogels and other porous materials. However, from the point of view of possible applications of these materials, mainly immersed in liquid, characterizing the size, shape and accessibility of the pores, as well as studying the interaction with the liquid medium, the solid-liquid interface reactions, or the description of the structural changes occurring under the influence of the liquid, are of primary importance. Liquid phase nuclear magnetic resonance (NMR, *N*uclear *M*agnetic *R*esonance) spectroscopy offers a joint solution for this, providing the opportunity for the non-destructive examination of the solid phase through the investigation of the liquid medium. The behavior of the liquid that partially or completely fills the pores provides information about the solid framework structure, the wetting of the pore surface, and last but not least, the mobility of the liquid in the pore system. There are several questions that NMR relaxometry, cryoporometry, diffusometry, or their complex use (like in the graphical abstract) can answer:

-Does the morphology and the composition change under the circumstances of application? Cryoporometry gives the answer for the pore size [1], but the relaxation time and the rate of restriction in diffusion are also dependent on the pore size.

-What kind of liquid domains are there in the porous structure, and how is their distribution? According to their T_2 transverse relaxation times liquid domains can be differentiated, and 2D correlation maps show their connectivity.
-How does the filling of the pores happen, what is the mechanism for that? Relaxation times of liquid domains with the filling degree can show the pore filling mechanism.[2,3]
-How does the solid surface behave by wetting, how much is it hydrophobic or hydrophilic? T_2 relaxation measurements and cryoporometry of test liquids of different polarity filling the pore structure can solve these questions.
-How does the liquid move in the porous structure? Diffusion measurements determine the (restricted) self-diffusion coefficients of solvent molecules, indicate the permeability of the structure, and also vapor-phase diffusion can be detected in partially filled samples.
In the presentation these questions and answers are demonstrated through several spectacular examples, measured on mainly carbon aerogels [4,5], and other porous materials, like rocks, cement and geopolymer binding materials. The theory of the methods and further details will be presented in the poster of V. Papp et al.

ACKNOWLEDGEMENTS

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OP-13 K-Wave Modelling of Ultrasound Wave Propagation in Aerogels and the Effect of Physical Parameters on Attenuation and Loss

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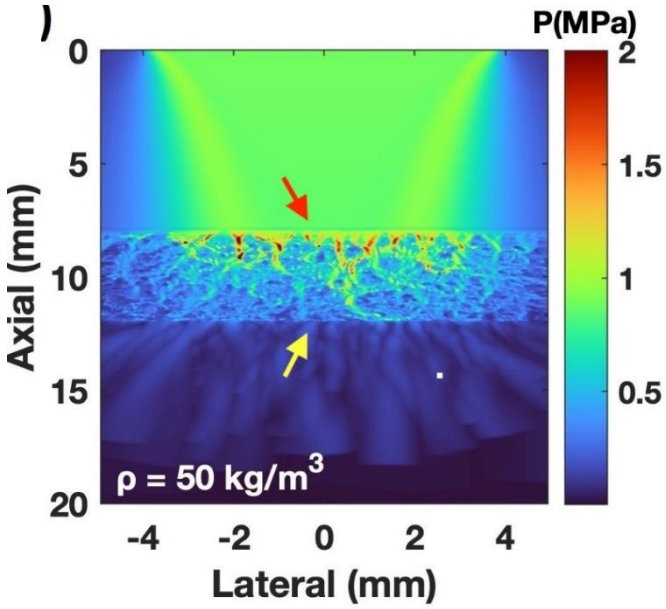

ABSTRACT

In recent years, aerogels have emerged as the material of choice for anechoic applications and interest in aerogels is continuously growing [1]. Due to the high tortuosity of the aerogel structure, these materials have demonstrated superior behavior as sound absorbers compared to other materials. Most of the published literature however explores the experimental use of aerogels for sound applications mostly in the audible range (0 - 4000 Hz) and is thus very limited in scope. Some studies have explored higher frequency ranges including the ultrasound region (> 20 kHz) where the main interest has been developing acoustic metamaterials [3]. Progress in optimizing material properties for these applications is hindered by the lack of a reliable model that can predict the response of these materials in different environments and can be addressed to some extent by the work presented here. Aerogels are also gaining traction as the material of choice for many biomedical applications such as neuronal scaffold, dental implants, and vascular implants to name a few. Given that aerogels have a high acoustic impedance mismatch (compared to soft tissue in the body),

ultrasonic detection can be reliably utilized for noninvasive rapid detection of aerogels in vivo which is made possible by the acoustic signature of these implants.

This was demonstrated for the first time experimentally in 2013 and serves as the inspiration for the work presented here. The summary provided above attempts to capture the versatile nature of aerogels and the many applications that they have been developed for to date. In many cases, whether embedded in an aqueous or non-aqueous environment, rapid and non-invasive detection and tracking of aerogels is an important part of the conversation and sound-based techniques offer a clear advantage over other techniques such as those that rely on ionizing radiation. Diagnostic techniques that exploit non-ionizing radiation (NIR) are preferred and sound-based techniques are among the most popular ones. The study of wave propagation is a complicated process because of complex intrinsic and extrinsic aerogel properties. This may lead to difficulty in acquisition and interpretation of data from acoustic measurements. The difficulty is often due to the varying size of the pores, their distribution, and the density, stiffness, and surface roughness of the aerogel structure. These all affect the wave propagation in different ways.

A computational approach to investigating wave-aerogel interaction allows for control over these parameters and the ability to study one parameter at a time in a manner that is not necessarily possible experimentally and forms the foundation of the work presented here. Nonlinear wave equations are the preferred method of choice for simulating wave propagation in heterogenous media [2]. Silica aerogels have been the focus of prior computational analysis by our group with an emphasis on developing a genetic algorithm to reconstruct defects in silica aerogels and how they affect thermal behavior of the aerogels [3]. The motivation behind the work presented here is to achieve real time visualization of wave interactions with aerogels in two different media (aqueous and non-aqueous) using k-Wave tool, and by recording the regions of maximum and minimum pressure at the interfaces. Our earlier work demonstrated that aerogels show interesting acoustic properties in response to ultrasound waves and give rise to distinct B-mode when used as implants in biological media . We have successfully applied through transmission technique to study the acoustic behavior of aerogels while accounting for different intrinsic (density, pore geometry, speed), as well as extrinsic (frequency, scanning angle) material properties.

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OP-14 Preparation of 3D metal oxide nanostructures

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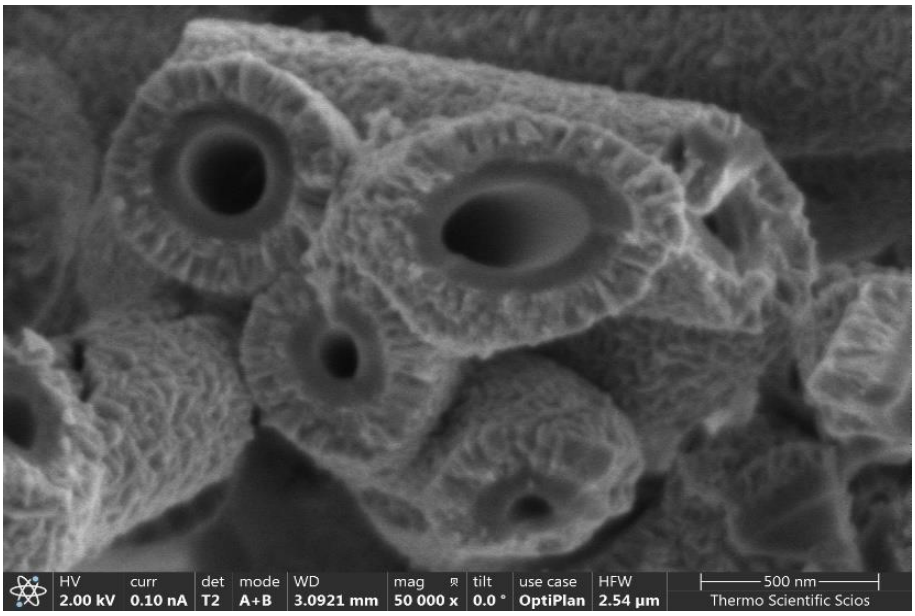
GRAPHICAL ABSTRACT



ABSTRACT

Metal oxide cylindrical nanostructures were prepared for diffusional studies. The samples can be classified into double and triple-layered nanotubes (DLNT/TLNT) with a hollow structure and double-layered nanocylinders (DLNC) with a core-shell structure.

Al_2O_3, and ZnO layers were deposited to different substrate materials by Atomic Layer Deposition (ALD) method. Trimethylaluminum (TMA) and water precursors were used to produce Al_2O_3, and Diethylzinc (DEZ) and water precursors for the ZnOlayer. Polymer nanofibers and platinum nanopillars were used as a substrate material for DLNT/TLNTs, and DLNCs respectively.

The polymer fibers were removed by a two-step annealing (burn out) in an ambient atmosphere in a tube furnace to create hollow structures.

Platinum nanopillars were produced by Gas Injection System (GIS) combined with a dual beam Scanning Electron Microscope (FIB/SEM).

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OP-15 Measuring the conditions of gelation of vapor-grown 1-D nanoparticles

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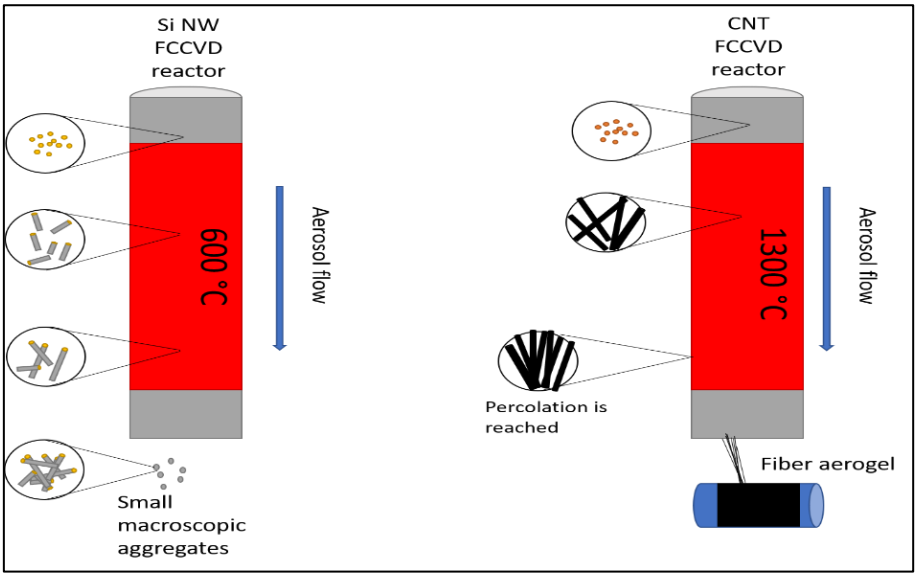
GRAPHICAL ABSTRACT



ABSTRACT

A usual drawback of nanomaterial synthesis is that the fabrication processes are slow, long and involve multiple steps. In order to overcome this problem, vapor phase synthesis methods have emerged as an alternative. For instance, Floating catalyst chemical vapor deposition (FCCVD) is a synthesis method for ultrafast synthesis of 1-D nanoparticles such as carbon nanotubes and inorganic nanowires [1]. Specifically, FCCVD process works as follows: a nanoparticle catalyst is produced in the aerosol phase while gas phase precursors are added, by these means, 1-D nanomaterials are grown from the surface of the catalysts as they are floating through the reactor. This approach allows growth rates that are orders of magnitude higher than the ones of standard wet methods. Moreover, an interesting and relatively unexplored phenomenon of this synthesis is that, as these 1-D nanoparticles are synthesized as concentrated aerosols, they can aggregate and spontaneously form macroscopic aerogels that can be collected from the reactor outlet. In this work, we study the aerogels generated

in the FCCVD synthesis process of silicon nanowires (Si NWs) and carbon nanotubes (CNTs), respectively. In Si NW synthesis, the nanowires form macroscopic aggregates, similar to small porous flakes consisting of non-oriented nanowires [2]. For CNT synthesis, instead, a single continuous aerogel is formed. This aerogel consists of aligned CNT bundles that can be directly drawn out from the reactor and spun as yarns or mats [3]. When these CNT aerogels are drawn of the reactor and densified, they resemble nanotextiles. We have previously studied their pore structure by BET gas adsorption, and find that they have a fracture mesoporous structure stemming from the imperfect packing of 1D nanoparticles and the formation of elongated open pores resulting from them [4].

Now, we have estimated the length and diameter distributions of the FCCVD-produced CNTs and Si NWs by SEM analysis. From these distributions, we have calculated the percolation threshold of each system considering the length and diameter polydispersity of the 1-D nanoparticles using a reported theoretical formula [5]. Interestingly, we find that, when comparing the percolation threshold of CNTs and Si NWs with their respective volume fractions in the reactor, CNTs volume fractions are much closer to the percolation threshold than Si NWs. This explains the appearance of the CNT aerogel as a single continuous structure compared to the small porous ensembles formed in the Si NW synthesis.

ACKNOWLEDGEMENTS

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Workshop on Aerogels Characterization and Modelling
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OP-16 Covalently immobilized copper(II) complexes as novel nanoenzymes with superoxide dismutase activity

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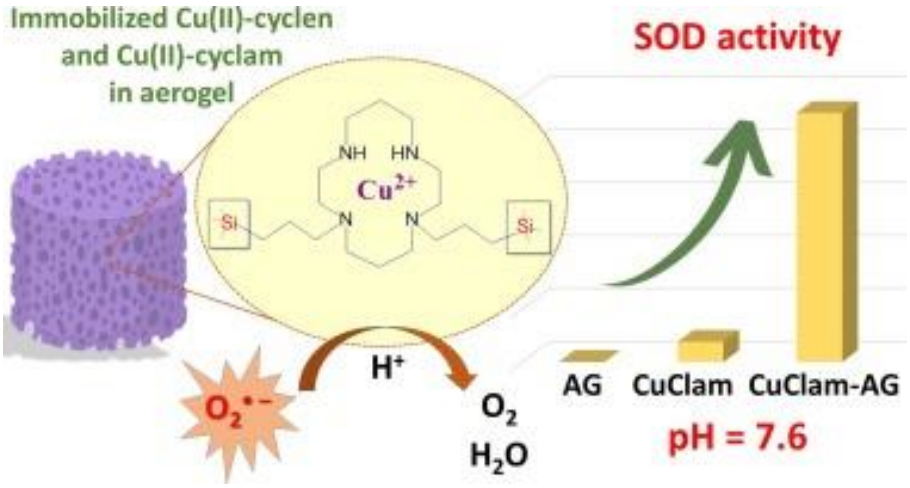
GRAPHICAL ABSTRACT



ABSTRACT

Antioxidant enzymes such as superoxide dismutase (SOD) and glutathione peroxidases are required to keep the concentration of superoxide anion radical at controlled low limits under physiological conditions. From these enzymes, SODs are capable to assist the decomposition of superoxide anion radical to hydrogen peroxide and molecular oxygen. [1] The absence of these enzymes or the elevated level of reactive oxygen species cause oxidative stress and inflammatory diseases. [2] Since the direct utilization of SODs in therapy is not feasible due to its low solution stability and membrane permeability, significant efforts have been made to develop efficient SOD mimics as novel therapeutic agents against oxidative stress. To improve the stability of SOD mimics, the immobilization in solid supports provides a promising strategy.

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]

In the quest for developing functional antioxidant nanosystems, we have covalently conjugated the copper(II) complexes of 1,4,7,10-tetraazacyclododecane (cyclen) and 1,4,8,11-tetraazacyclotetradecane (cyclam) into mesoporous silica aerogels. The chemical structures of the functionalized aerogels were elucidated based on the combined mass spectrometry (MS) and nuclear magnetic resonance spectroscopy (NMR) characterization of the synthetic precursors, and the electron paramagnetic resonance (EPR) spectroscopy characterization of the as-prepared aerogels. Contrast variation small angle neutron scattering (SANS) and EPR measurements unambiguously prove that the Cu(II)-complexes are not aggregated, but molecularly dispersed in the silica networks. The functionalized aerogel microparticles show excellent catalytic activities in the dismutation reaction of the superoxide anion mimicking the superoxide dismutase enzymes. The covalent immobilization drastically increases the SOD activities of both Cu(II) complexes compared to the aqueous parent complexes obtained by the xanthine/xanthine oxidase/NBT model system. [4]

ACKNOWLEDGEMENTS

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OP-17 Towards CO_2 upcycling with porous carbon materials

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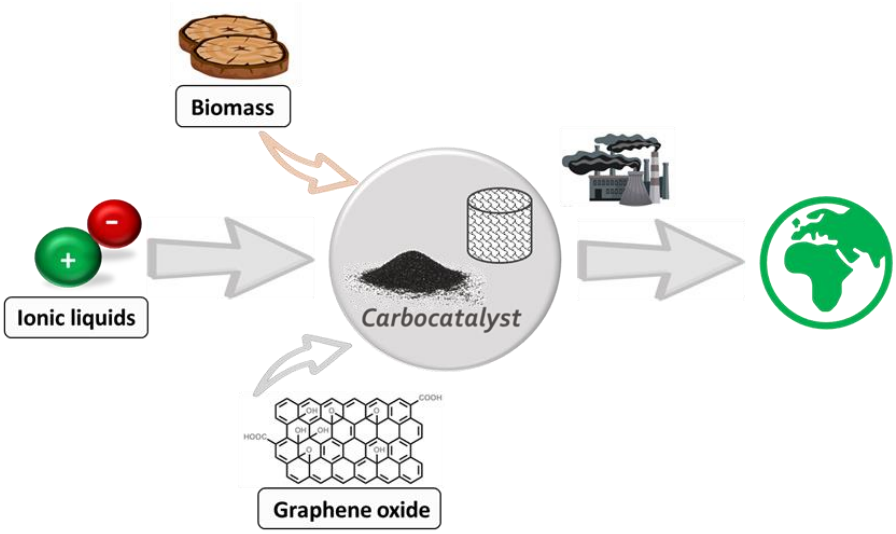
GRAPHICAL ABSTRACT



ABSTRACT

Carbon dioxide has become the focus of attention for being a major anthropogenic greenhouse gas associated to climate change. To decrease the concentration of CO_2 in the atmosphere, CO_2 capture (CC) technologies based on physisorption, chemisorption and membrane separation have been developed. The industrial most advanced technology is still based on aqueous monoethanolamine (MEA) solutions. However, the drawbacks of this process such as the corrosiveness, volatility of the substances and the energy penalty in recycling cause environmental and economic problems.

Carbon materials have been proposed as good candidates for CO_2 sorption and ionic liquids (ILs) are known to have not only good sorption, but also selectivity for CO_2 among other gases [1].

Moreover, the abundance of CO_2 makes the capture of this gas an opportunity, as it is a C1 building block source capable of promoting a circular carbon economy. Despite the large number of efforts related to CC, a viable solution with potential industrial applicability is still missing, namely through the design of effective sorbent and catalyst materials able to work at

low pressures. In the last years, we have been dedicated to the optimization of IL derived materials for CC and conversion, having developed nuclear magnetic resonance (NMR) protocols to study CO_2 sorption both at atmospheric and higher pressures [2,3]. The use of carbon-based composites for this purpose allowed us to achieve highly selective CO_2 conversion reactions in significant yields, while it has emphasized the need for well-defined porosities [4,5]. Herein we present our latest results on the synthesis and characterization of heterogenous catalysts for CO_2 conversion, and the application of these materials, that range from biomass derived carbons to poly(IL)-graphene aerogels to model CO_2 conversion reactions.

ACKNOWLEDGEMENTS

We are thankful for financial aid from FEDER funds through COMPETE 2020 Program and National Funds through FCT- Fundação para a Ciência e a Tecnologia, I.P., in the scope of projects LA/P/0037/2020, UIDP/50025/2020 and UIDB/50025/2020 of the Associate Laboratory i3N, and PTDC/QUI-QFI/31508/2017 and PINFRA/22161/2016. MCC acknowledges FCT for the researcher contract (2021.03255.CEECIND); MZ acknowledges the European Union's Horizon 2020 research and innovation program under the Marie Skłodowska-Curie grant agreement №101026335.

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Workshop on Aerogels Characterization and Modelling
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OP-18 Silica based organic–inorganic hybrid xerogels and aerogels: synthesis, structure and applications

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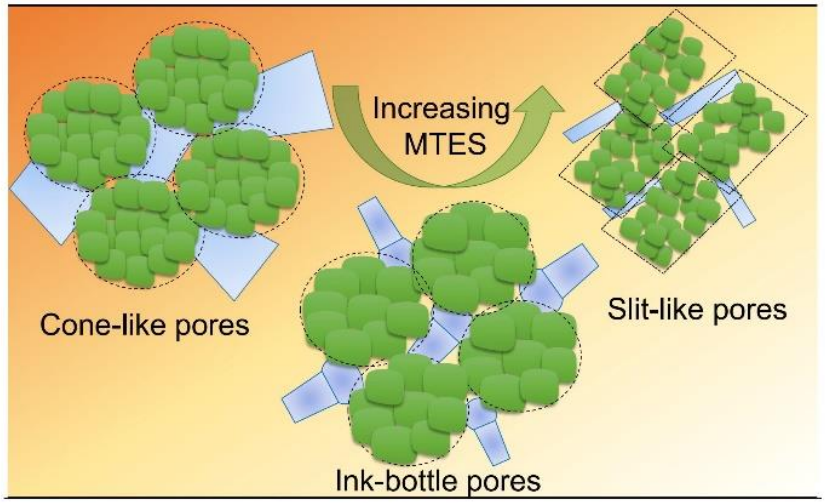
GRAPHICAL ABSTRACT



ABSTRACT

Silica xerogels and aerogels are unique class of mesoporous solid materials which can be synthesized using several methods [1]. Among the others, the sol-gel technique is one of the most popular methods to obtain mesoporous materials [2]. The structural properties have direct effect on the materials functionality, small changes in the synthesis parameters can cause distinctive structural properties [3-4]. In order to design advanced materials with proper structural properties the relationship among the materials and processing structure and properties is the most important factor [5-6].

In this work we aim to present an overview about the recent research progress on hybrid silica xero-/aerogels synthesis, structural characterization and their environmental, medical and catalytic applications. The structural characterization of porous inorganic and organic hybrid materials at the nano-, micro- and macro level were done by using a multi-technique approach. The morphological characterization was carried out by adopting electron

microscopy (TEM and SEM), the textural properties were revealed by nitrogen porosimetry and small angle scattering (neutron and X-ray), the chemical composition by solid state NMR and FT-IR spectroscopy.

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Workshop on Aerogels Characterization and Modelling
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OP-19 Sustainable silica aerogel synthesized from waste glass via the ambient pressure drying method

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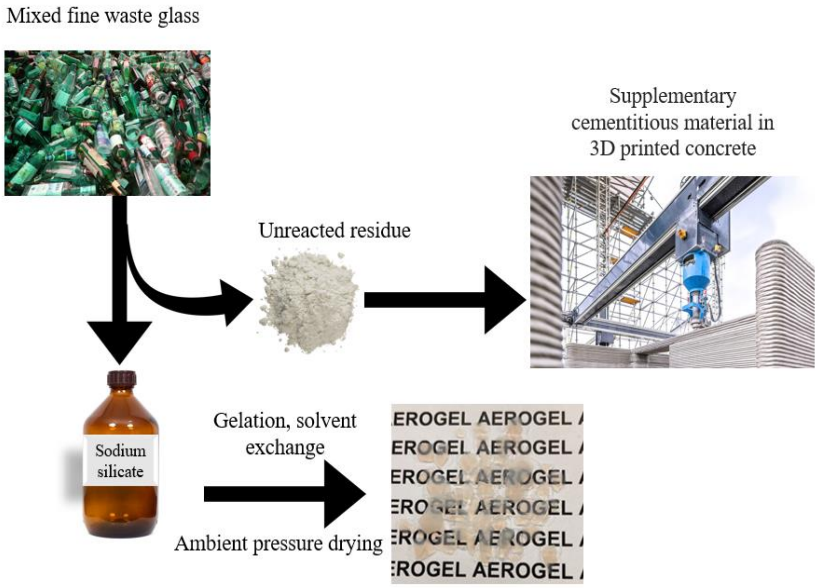
GRAPHICAL ABSTRACT



ABSTRACT

To make the building materials industry more circular recycling of waste glass has been investigated in the past.[1] Researchers have used it as a supplementary cementitious material or added it to geopolymers.[2] This work describes a new protocol for a complete synthesis of silica aerogel, which is an outstanding insulation material, via ambient pressure drying (APD) method from mixed fine glass waste as a starting material. While a large percentage of waste glass is recycled (around 50% worldwide)[3], there are difficulties which may result in landfilling 10-30% of waste glass that is e.g. broken down too finely or mixed to the extent where it is not feasible to separate it.[4][5] The produced silica aerogel particles obtained nearly super hydrophobic property (149.5°) and sufficiently low thermal conductivity (0.027 W/m·K). This research aims to adapt the synthesis route and the final material to the needs of the insulation material industry, so the process can be reproduced and utilized at a larger scale. The parameters were optimized to maximize the extraction efficiency with

regards to resources such as time and materials spent on the process, as well as the quality and reliability of the properties of the silica aerogel. Additionally, the method prioritizes sustainability and circularity, which is why the energy intensive supercritical drying method [6] was replaced by APD and solvent exchange, and the unreacted glass residue was used as a supplementary cementitious material in 3D printed concrete.[7] The overall aim of this work is to reuse and recycle as much waste as possible while creating an affordable highly efficient insulation product.

ACKNOWLEDGEMENTS

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POSTER PRESENTATIONS

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PP-01 Influence of the initial synthesis chemical composition on the gelation kinetics of MTMS-based aerogels

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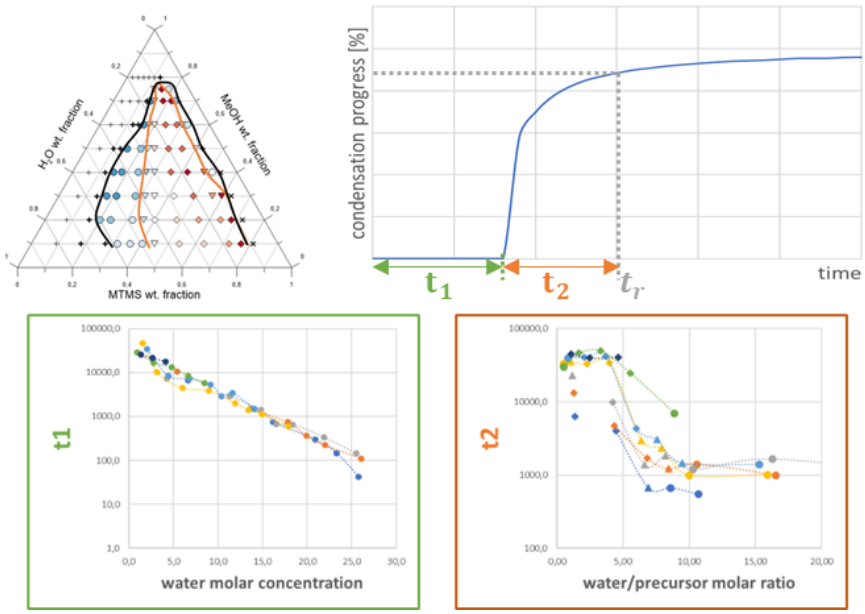
GRAPHICAL ABSTRACT



ABSTRACT

Silica aerogels have become technologically and scientifically popular due to their unique properties, such as high porosity, low density, high specific surface area, and low thermal conductivity [1]. They are usually obtained by sol-gel synthesis combined with a specific drying method. Particularly important for this type of synthesis is the condensation step which is directly responsible for the aerogel final structure formation [2]. However, condensation kinetics is significantly complicated due to many individual reaction rates and occurring phase separation [3]. One of the promising research tools for studying these kinetics is UV-Vis spectrophotometry in which the measured absorbance is proportional to the change of condensing mass in the solution, as has been shown by Borzęcka et al. [4].

The presented research was carried out to understand the influence of the synthesis solution composition on the gelation kinetics. The initial components ratio is described based on a ternary system – methyltrimethoxysilane, methanol, and water. As the synthesis procedure, the two-step, acid-base, sol-gel method was used, in which during the 2nd step – condensation – the samples were examined by UV-Vis spectrophotometry. After that, to prepare aerogels,

the ambient pressure drying method was employed. Additionally, the scanning electron microscope (SEM) observation allowed for the morphologies classification as one of the four distinguished in literature types of structures [5].

During the kinetics results analysis, three values were defined: duration of reaction (t_r) – the value of time which corresponded to 97% of the maximum absorbance of the sample (this value was also compared to gelation time obtained by the "tilting" method), duration of nucleation process (t_1) - the value of time which corresponded to the beginning of intensive mass growth, and the duration of microscopic phase separation process (t_2) – the value which was the subtraction of t_r and t_1.

It was observed that the time t_1 depends mainly on the water content in the reaction mixture. The t_1 values decrease with increasing water content, which means that water accelerates the nucleation process. The relationship between t_1 and water concentration is exponential and monotonic. Whereas the value of t_2 depends on a much larger number of parameters than t_1. Comparison of t_2 with the structure classification allows concluding that for the particle aggregates structure (being an effect of nucleation and growth phase separation mechanism in a thermodynamically metastable region), t_2 reaches relatively constant low values (fast reactions). While in the case of other structure types (formed by spinodal decomposition mechanism that occurs in a thermodynamically unstable region), t_2 has much higher values and reaches its maximum for the water/precursor ratio equals three. Maximum corresponds to the stoichiometric ratio (3 alkoxy groups in the precursor molecule). The reaction duration (t_r) is the sum of t_1 and t_2, so it is the result of the above-mentioned factors. Additionally, t_2 and t_r values are affected in the same way by the methanol content – increasing the solvent (methanol) concentration dilutes the solution, thus increasing the condensation duration.

These studies have the potential to advance the state of the art by providing knowledge about controlling silica aerogel syntheses. Gaining knowledge of condensation kinetics complexity is of significant importance due to its crucial impact on aerogel structure formation.

ACKNOWLEDGEMENTS

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PP-02 Glutaraldehyde crosslinked aerogel for the selective sorption of aqueous Pd(II)

Balázs József Bukta [a*], Petra Herman[a], Dániel Pércsi[a,b], Madalina Ranga[a], Attila Forgács[a], István Fábián[a], József Kalmár[a]

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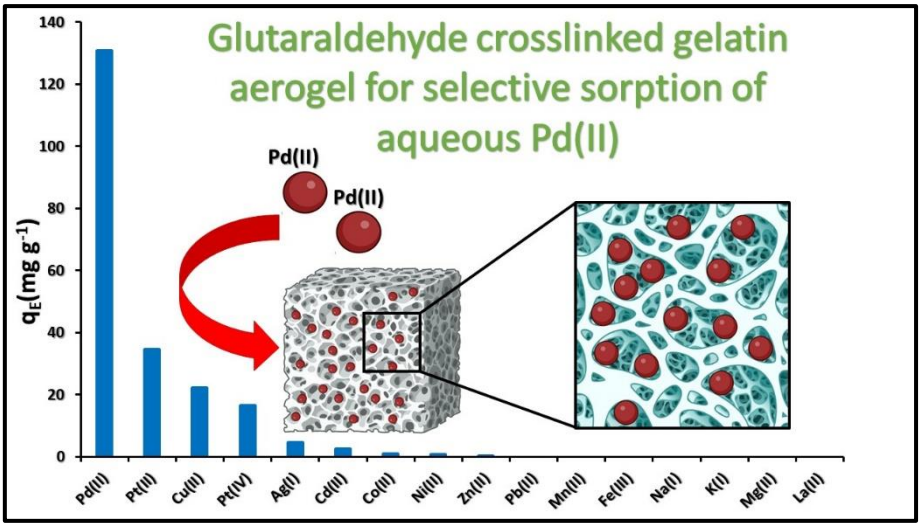
GRAPHICAL ABSTRACT



ABSTRACT

The removal of toxic and/or precious metal ions from aqueous media is a great environmental as well as economical challange. Palladium and its compounds have high technological importance and extensive use in many fields of industry, principally in catalysis [1]. Because of the increasing demand for palladium its recovery is crucial. Among the various methods reported for the removal of heavy metal ions, sorption stands out because of its high performance and cost effectiveness [2].

Gelatin containing porous solids have shown promising results in the selective sorption of aqueous metal ions [3]. In the present study, we aimed to prepare a mesoporous aerogel sorbent by crosslinking the polypeptide chains of the gelatin for the high capacity selective sorption of aqueous Pd(II). The gelatin backbone provides the functional groups for the sorption of Pd(II) while glutaraldehyde serves as a crosslinking and structure stabilizing agent.

The structure of the aerogel was characterized using low voltage scanning electron microscopy (LV-SEM) and nitrogen sorption porosimetry. These results confirm a mesoporous structure and a narrow pore size distribution.

The aerogel has a remarkable selectivity for binding aqueous Pd(II) in acidic solutions in the simultaneous presence of Pt(II), Pt(IV), Cu(II), Pb(II), Hg(II), Zn(II), Ag(I), Ni(II), Cd(II), Co(II), Mn(II), Fe(II), Na(I), K(I), Mg(II). The optimum pH for Pd(II) sorption is 2.0. The sorption capacity is ca. 200 mg g^{-1} Pd(II) at pH = 2.0, using 0.32 g L^{-1} aerogel. Kinetic studies demonstrate that the sorption equilibrium is reached in 1 h contact time. The quantitative dissolution of the Pd(II) and the regeneration of the sorbent was realized using 5 mM methionine solution. 99±2 % of the initial Pd(II) concentration was successfully recovered. The results prove that the synthesized aerogel is a potential candidate for large-scale aqueous Pd(II) sorption.

ACKNOWLEDGEMENTS

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Workshop on Aerogels Characterization and Modelling
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PP-03 Microbiological and morphological characterization of bio-based aerogels after supercritical CO_2 sterilization

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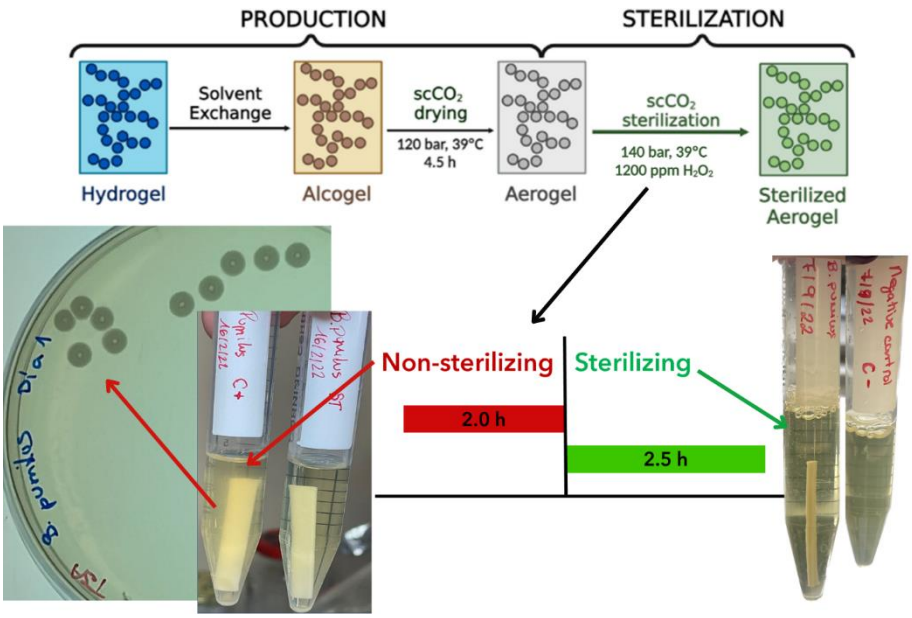
GRAPHICAL ABSTRACT



ABSTRACT

The use of aerogels of biological origin, such as polysaccharides or proteins, is of particular interest in the biomedical field due to their bioactivity, biocompatibility and biodegradability combined with the physicochemical properties of aerogels. Applications of aerogels include wound healing, drug delivery and bone regeneration. However, for the commercialization of certain medical devices and drug products, sterility assurance level (SAL) standards, which quantifies the sterility of the material, must be passed with a value of SAL 6 [1].

Conventional sterilization techniques, such as gamma radiation or dry heat, produce morphological and physicochemical changes in the structure of the bio-based aerogels, whereas ethylene oxide can leave cytotoxic residues that are hazardous to patients. Sterilization with supercritical carbon dioxide ($scCO_2$) emerges as an alternative since the

mixed gas-liquid behavior and the high penetrability of this type of fluid and the microbial inactivation power of CO_2 make the sterilization of porous and complex-structured materials feasible [2]. The inactivation mechanism of $scCO_2$ sterilization starts with the acidification of the extracellular medium by CO_2 solubilization. This decrease in pH not only affects membrane lipids, due to the lipophilic nature of CO_2, but also increases membrane permeability favoring the accumulation of CO_2 in the intracellular medium. The presence of CO_2 inside the cells will not only decrease the pH but will also inhibit or inactivate the metabolic enzymatic activity [1].

In this work, alginate aerogel microspheres and starch aerogel monoliths have been subjected to different sterilization conditions. The microbiological evaluation was carried out by subjecting bioindicators to the same sterilization conditions. Bioindicators are different for each type of sterilization and consist of dried spore strips of the most resistant microorganism. In the case of $scCO_2$ sterilization, the bioindicators are strips of 10^6 dried endospores of *Bacillus pumilus*, which proved to be the most resistant to this sterilization technique compared to *Bacillus sterarothermophilus* and *Bacillus atrophaeus*, bioindicators for steam and radiation sterilization, respectively. After supercritical treatment, spore strips were seeded in trypticase soy broth and trypticase soy agar culture medium. Conditions were considered as sterilizing after 7 days in the absence of microbial growth. Finally, the possible physicochemical changes of the aerogels after the sterilization treatment were characterized by N_2 adsorption-desorption tests and helium gas pycnometry.

ACKNOWLEDGEMENTS

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PP-04 Synthesis and characterization of gelatin, and crosslinked gelatin aerogels

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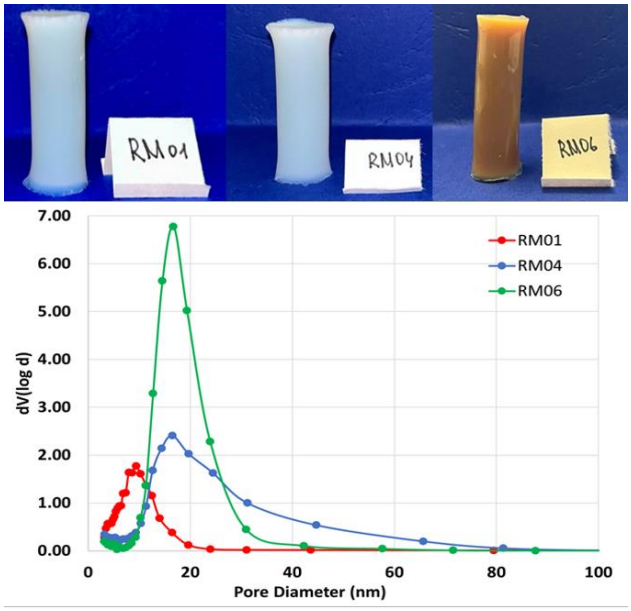

ABSTRACT

This research aims to characterize newly synthesized pure high gelatin content aerogels, and the crosslinked version of the gelatin aerogels with glutaraldehyde (GTA).

Biopolymer aerogel field is a growing branch which is gaining scientific interest due to its superior properties over the traditional synthetic polymers, the non-toxic nature and biocompatibility allowing their safe usage in environmental, food and pharmaceutical industry.[1]

The cross-linking process was achieved by the interaction of the gelatin gel with the crosslinker (GTA) and using a subsequent supercritical CO_2 drying process.[2] Three different aerogels

with the following composition will be studied: 30 wt.% gelatin/water (RM01); 20 wt.% gelatin/water (RM04); and 20 wt.% gelatin/water crosslinked with 2.5 wt. % GTA (RM06). In this research the focus is the characterization of newly synthesized advanced materials by the conventional characterization techniques (SEM, N_2-porosimetry, IR) as well as compressive strength tests. The SEM pictures illustrate the dense fibrils like structure of the pure gelatin samples and the altered one of the GTA-gelatin crosslinked. This feature has an impact on the strength of the two types of aerogels. Moreover, with increasing gelatin content we can observe a trend in increasing the mechanical properties which give these materials the ability to withstand high stress. Aerogels used in biomedical studies should be biocompatible and biodegradable. Biological macromolecule-based aerogels are particularly suitable for implants such as membranes, tissue growth scaffolds. Next these aerogels will be studied for specific applications such as: thermal insulation, promising applications in filtration (selective ions adsorption: Hg, Pt, Pd), catalysis, and medicine (drug delivery, wound healing and tissue engineering).

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PP-05 Impacts of Chitosan's Intrinsic Properties on Aerogel Structure

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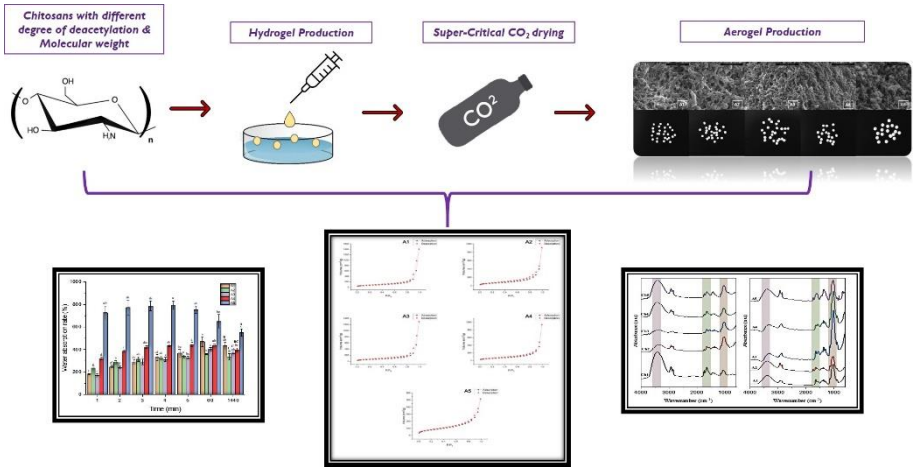
GRAPHICAL ABSTRACT



ABSTRACT

In the presented study, chitosan, a natural polysaccharide was used to make biobased aerogels. Since chitosan is a positively charged polysaccharide, its interaction with negatively charged substances such as fats and proteins makes it an important hydrocolloid for food applications. Although chitosan aerogels have a large surface area and high porosity, intrinsic properties of chitosan such as molecular weight (MW) and degree of deacetylation (DDA) greatly impacted the characteristics of chitosan aerogels. Therefore, aerogels made from chitosans having different MW and DDA were evaluated in terms of bulk density, pore characteristics, and surface area by BET and BJH methods. SEM images were used to comment on morphologies of aerogels while structural changes were observed by FTIR Spectroscopy. It was found that the lower MW with higher DDA increased the aerogel diameters while more compact structures were obtained by higher MW chitosan. The minimum bulk density and the highest water absorption rate were observed with the chitosan having the highest DDA with the lowest MW.

ACKNOWLEDGEMENTS

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PP-06 What can liquid-phase NMR tell us about porous materials?

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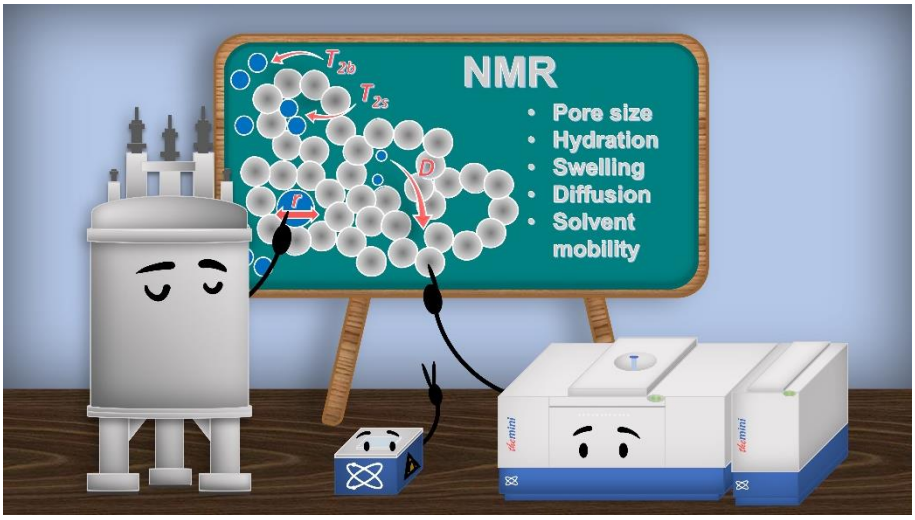
GRAPHICAL ABSTRACT



ABSTRACT

Liquid-phase NMR (***N**uclear **M**agnetic **R**esonance Spectroscopy*) can provide indirect information about the solid material itself based on the changed magnetic properties of the liquid within the porous system. The basic of NMR is that the nucleus with magnetic dipole moment, like protons in water, can be excited with radio frequency irradiation in magnetic field.

In **Low- field NMR relaxometry** we follow the return of the excited magnetic spins of the hydrogen nuclei (protons) in water molecules to the equilibrium state. This process can be described with relaxation time constants, the value of which depends on the mobility of the protons, thus the water types of different mobility in the sample can be distinguished. [1-3] Monitoring the change in relaxation times as a function of various parameters (such as water content, time) gives the opportunity to follow the hydration, swelling, degradation, or even formation of the porous system. The size of the pores, the properties of the surface like hydrophobicity also can be deduced from the measured relaxation time constants. Observing the correlation between the longitudinal and transverse relaxation times (T_1-T_2 *correlation*) can specify the permeability and connection between the liquid domains with different

mobility.

The size and the shape of the pores of nanometer range can be determined by **NMR cryoporometry** as well. The basis of the method is that the freezing and the melting point of the liquid in the pores are lower than in the bulk phase. This temperature shift and the difference between the freezing and melting processes inside the pores give information about the above-mentioned properties. This measurement can be carried out in high- and low- magnetic field. The last gives an opportunity to follow the melting and freezing of different liquid domains. [1-3]

NMR diffusometry enlightens us about the movement of the solvent influenced by the pore structure. From the inhibited diffusion process the size of the inhibiting space can be calculated. If we were to follow the movement of the solvent in a sample containing magnetic inhomogeneity, which is difficult to examine with NMR, we could follow it with a special H_2O-D_2O exchange diffusion technique. [4]

NMR MoUSE (***Mo****bile* ***U****niversal* ***S****urface* ***E****xplorer*) is a small tabletop magnet that can be used "on-field" on which a larger sample can be placed, and thus the sample can be measured as a whole, without destruction or preparation. Due to the one-sided magnet, there is a gradient in the magnetic field going up from the surface of the magnet, because of which diffusion can also be measured, like the mobility of different liquid domains in the human skin.

With the combination of these techniques, we can get a comprehensive picture of the behavior of our porous system in liquid media, which is an extremely important parameter for their wide-scale application.

ACKNOWLEDGEMENTS

The research was supported by the National Research, Development and Innovation Office – NKFIH [PD 135169, K 131989] and was carried out within the framework of the RHK contract. Project no. RH/322-2/2022 has been implemented with the support provided by the Ministry of Culture and Innovation of Hungary from the National Research, Development and Innovation Fund, financed under the KDP-2021 funding scheme. The research was also financed by the European Union and the State of Hungary, co-financed by the European Regional Development Fund in the project of GINOP-2.3.4-15-2020-00007.

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Workshop on Aerogels Characterization and Modelling
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PP-07 Small-angle neutron scattering (SANS) investigation of functionalized and hybrid silica aerogels

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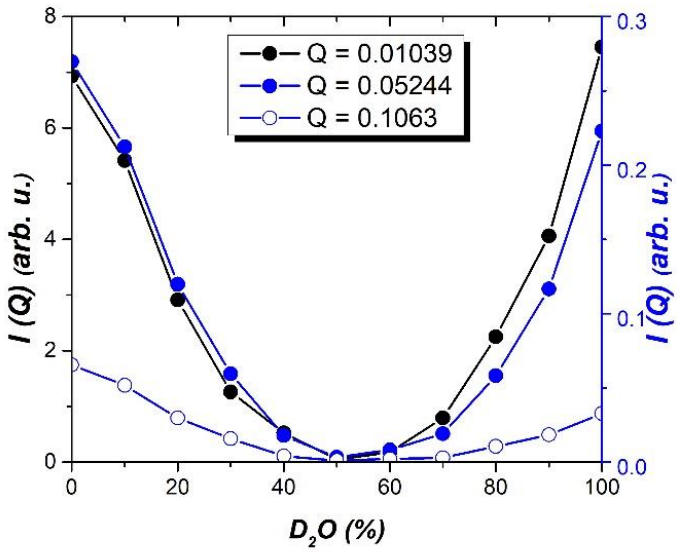
GRAPHICAL ABSTRACT



ABSTRACT

Small angle neutron scattering (SANS) is a non-destructive investigation method which can be used to obtain information on nanostructural inhomogeneities in the size range of 1-100 nm within a material. [1] Aerogels are porous materials in which two major phases can be distinguished: the gel backbone and the pores. SANS measurements can be used to obtain size and morphological information on these phases. If the matrix of the gel backbone is built up by different components, more than two phases may appear in the solid network. If the size of these phases are in the SANS measurement range, information can be obtained on them using contrast variation measurements. [2-4]

Contrast variation SANS measurement were performed on silica aerogels functionalized with Cu(II) cyclen and cyclam to study the incorporation of the Cu(II) complexes into the gel backbone. [3]

In another study borosilicate aerogels hybridized with polyvinyl alcohol were investigated to prove the formation of the hybrid structure. Different Ca(II) sources ($CaCl_2$, β-$Ca_3(PO_4)_2$ and $Ca_{10}(PO_4)_6(OH)_2$ (hydroxyapatite)) were built into the hybrid structure and the size of these particles was investigated using contrast variation SANS. We were able to obtain information on the average size of the hydroxyapatite particles incorporated into the gel backbone.

ACKNOWLEDGEMENTS

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

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PP-08 The effect of ionic liquid on the morphology and surface properties of RF carbon aerogels by NMR

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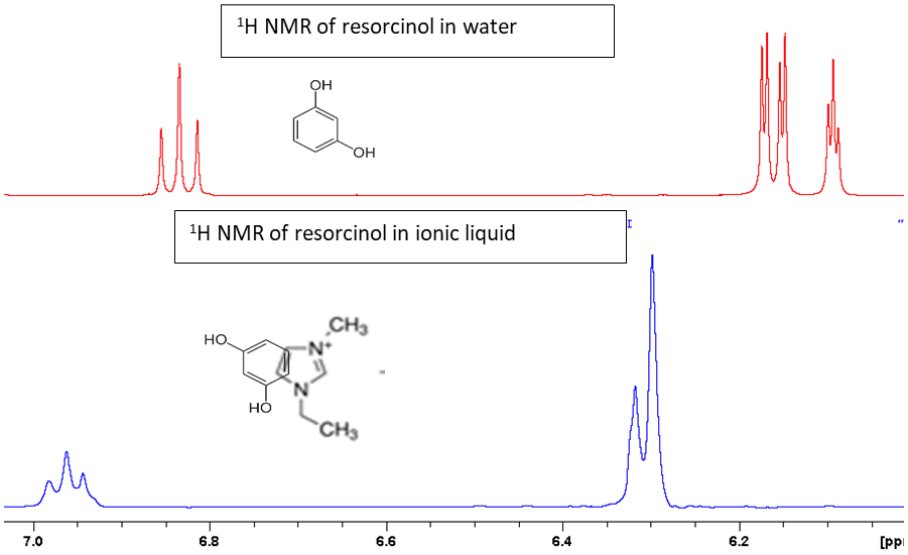
GRAPHICAL ABSTRACT



ABSTRACT

Resorcinol-formaldehyde (RF) based carbon aerogels are widely used materials as catalyst, electrodes or adsorbents because of their high surface area, good electrical conductivity and tunable pore size. During the synthesis many factors can affect the resulting pore structure including the concentration and molar ratio of the reagents, the pH and the temperature. Beside the reaction conditions, porogens (solvents, additives, catalysts) have a crucial role in the resulting pore system.[1] Ionic liquids in the RF precursor solution can serve not only as a porogen, but as a template or a source of nitrogen or carbon. Recently the effect of several room temperature ionic liquids as co-solvent of water and their concentration was studied on the pore structure of the synthesized resorcinol-formaldehyde polymer and carbon aerogels. [2,3] The applied 1-ethyl-3-methylimidazolium ethyl sulphate ionic liquid mainly acted as a catalyst for the polymerization reaction, thus the pore morphology could be tuned by the water to IL ratio. The supramolecular system of water and IL also contributed to the formed aerogel texture.

Some liquid phase nuclear magnetic resonance (NMR) methods are well applicable for the characterization of porous materials like aerogels in wet state. With the use of NMR cryoporometry, relaxometry and diffusiometry the whole wetting mechanism of carbon

aerogels, from hydration to immersion can be described, and contrary to other methods, the differences in the hydration properties can also be pointed out. [4]

In our work, we used the mentioned NMR techniques to study the morphology of carbon aerogels, synthesized with changing IL/water ratio [3], in aqueous medium. It is confirmed that the pore size of the resulted carbon aerogels can be tailored by the IL/water ratio during the synthesis. Moreover, we discovered that the IL modifies the hydration properties of the aerogels. The explanation is supported with the high-field NMR analysis of the monomer/IL/water solutions.

ACKNOWLEDGEMENTS

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PP-09 Vinyl modified silica aerogel coated glasses for the thermal insulation applications

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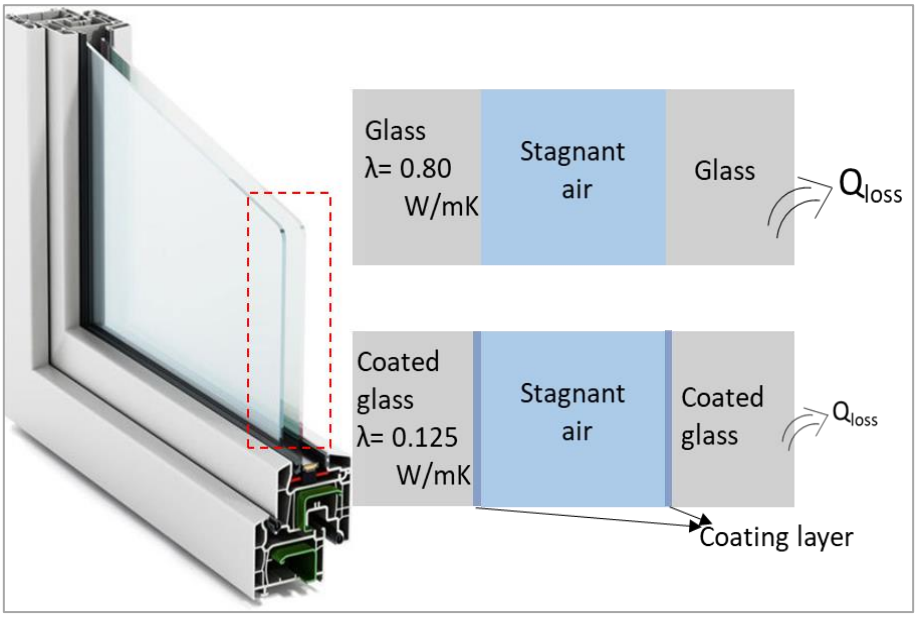
GRAPHICAL ABSTRACT



ABSTRACT

As a result of the growing industry with the developing technology, energy demands have reached maximum levels. In recent years, it has become very important to protect energy as well as to seek ways to meet energy needs. In this context, it has come to the fore to take action to reduce energy loss [1]. Glass surfaces are the materials that lose the most energy in buildings and equipment's. Insulation applications of glass surfaces are essential in many respects. For this purpose, glass productions of different sizes, thicknesses, and types, double/triple/five glass modules have been designed and started to be used. The main purpose of these systems is to reduce the effective thermal conductivity coefficient of the system by trapping gases (air, argon, etc.) with a very low thermal conductivity coefficient between the glasses with a high heat transmission coefficient (0.80 W/mK) [2]. Although these modules significantly reduce energy loss, they considerably increase the system's weight and pose a problem for many applications. In smaller applications (front windows of refrigerators and freezers), the limitation is that these modules are quite costly. Moreover, the cost of inert

gases trapped in these systems, the optimization of the space between the glasses, and the safety concerns in the preparation, distribution, and assembly of the modules bring with them. On the other hand, applying a thermal conductivity values is considered as an effective way to improve the thermal insulation propeties of the surfaces. In this study, sol-gel derived aerogel coatings with very low density (0.01-0.3 g/cm^3), high porosity (>90%) were developed to reduce the thermal conductivity values of the glass substrates [3,4]. Bilayer coating was composed of hydrophilic first later and the hydrophobic top layer, which provides thermal insulation properties and acts as a protective layer barrier against water at the same time. In the solution of the first layer, silica nanoparticle, TEOS (tetraethylorthosilicate) as sol precursor, and ethanol/water mixture as a solvent were used to provide a homogeneous silica structure. In the second layer coating solution, TEOS was used as silica precursor, VTMS (vinyltrimethoxysilane) as co-precursor, ethanol and water as solvent, and 0.01 M HCl acid as catalyst. Coating solutions prepared at an interval of 30 minutes were applied to the glasses cleaned with acetone, ethanol, and distilled water, respectively. In order to prevent cracking during the drying of the surfaces, surface modification was applied by preparing TMCS-hexane solution and the glass surfaces were dried overnight under ambient conditions. The thermal conductivity coefficient of the coated glasses prepared with the developed sol-gel coating technique was measured as 0.125 W/mK and the contact angle as 88°. The experimental results showed that 85% decrease in thermal conductivity of glass substrate (from 0.80 to 0.125 W/mK) was obtained by applying bilayer silica aerogel coating.

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PP-10 Exploring thermal conductivity of aerogels through DSC analysis

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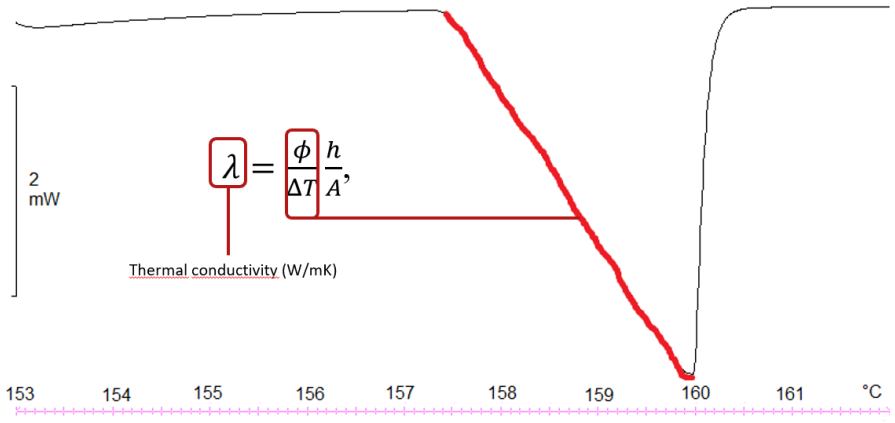
GRAPHICAL ABSTRACT



ABSTRACT

Many instruments are nowadays available to determine the thermal conductivity of materials. One of them is DSC, which was first proposed by Hakvoort and van Reijen [1]. In this method, the melting behaviour of a pure metal (e.g. indium) on top of a cylindrical sample or disk is measured. During heating, the metal reaches its melting point and the temperature remains constant while the metal melts. The temperature difference between lower end surface of the disk and the heat flowing into the disk are measured by the DSC and the thermal conductivity is calculated from the temperature difference and the heat flow. The research work presented in the paper describes this method for measuring the thermal conductivity of novel advanced materials – aerogels. Aerogels are materials with outstanding properties, high porosity, large surface area and low density. They are used in many application, like thermal insulators, capacitors, pharmaceuticals, food additives etc..

Different types of aerogels and aerogel-hybrids were prepared. Polysaccharide aerogels (pectin, alginate, xanthan and guar) were prepared by a crosslinker-free method using ethanol as an antisolvent. Then the same polysaccharides were used to prepare hybrid materials with silica aerogels. Lastly, silica aerogels were reinforced with polyHIPEs (high internal phase emulsion templating). Prepared aerogels were then measured by the HPDSC1 to determine their thermal conductivity. Aerogels were prepared in the form of disks with a height of 0.5 to 1.5 mm. The sample was then placed directly on the sensor and the crucible was placed at the

top of the sample. Crucible was filled with 80 mg of indium. The sample was heated from 153 to 162 °C, 0.5K/min.

Low thermal conductivity was observed for all samples. Thermal conductivity of pure polysaccharide aerogels was in the range of 20 – 100 $Wm^{-1}K^{-1}$. This value was improved by making polysaccharide-silica aerogels, where thermal conductivity decreased to 19 $Wm^{-1}K^{-1}$ Silica aerogels were then reinforced with polyHIPEs and their thermal conductivity reached very low values of 17 $Wm^{-1}K^{-1}$.

This research shows that DSC is not only a great method to determine the nature of the materials (amorphous, crystalline) and their degradation, but according to those results also the method for the precise determination of thermal conductivity of insulating materials.

ACKNOWLEDGEMENTS

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PP-11 A simple kinetic model to explain the solubilizing spring effect in aerogel drug delivery systems

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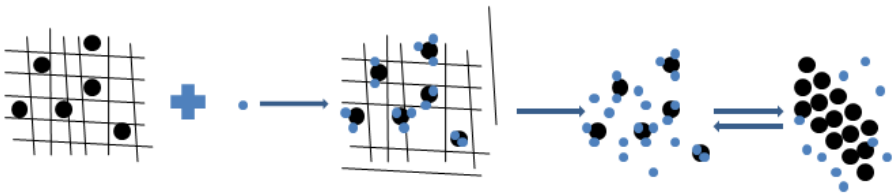
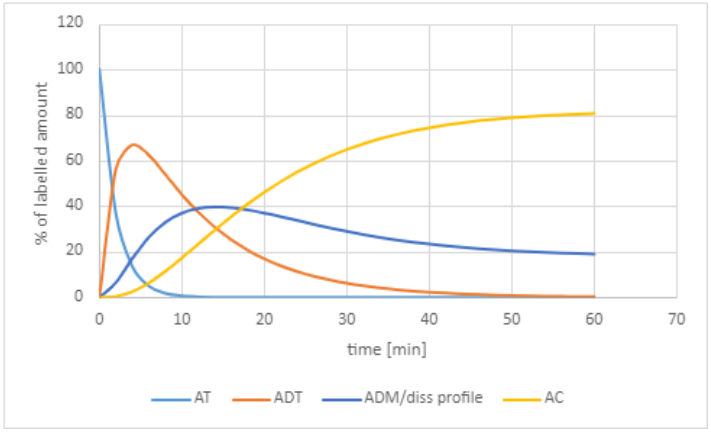


AT: dry API (Active pharmaceutical ingredients) in "Tablet"; ADT: solubilized API in formula; ADM: API Dissolved in Media; AC:API Crystallized



ABSTRACT

In the case of Active Pharmaceutical Ingredients (API) with high permeability & low solubility (Biopharmaceutics Classification system (BCS) II) and low permeability low solubility (BCS IV)[1], increasing the bioavailability is a great challenge. The main reason for this is the low solubility[2] of the API, which can be increased by keeping it in an amorphous form in the final product. The use of aerogels in pharmaceutical formulations as solubilizing aids provides a good platform to keep poorly soluble active substances in an amorphous form increasing their apparent solubility[3], thus increasing their bioavailability[4]. However, the consequence of this is a unique drug release mechanism and kinetic profile, which has a high temporal concentration maximum in the supersaturation range. This phenomenon is termed in the

literature as the ”spring effect”. A mechanistically realistic kinetic model is essential to describe the release profile for drug formulation development which enables a Quality By Design (QBD) approach. The presented new model divides the drug release into three consecutive processes. The first step is the hydration of the carrier and the dissolution of the entrapped drug. The second step is the liberation of the dissolved drug from the carrier. The last step is the dissolution equilibrium of the thermodynamically stable (crystalline) form of the drug. By fitting the model to experimental dissolution curves, it was found suitable to determine the relationships between the structural characteristics of the pharmaceutical formulations and the descriptive t_{max} and c_{max} values of their release profiles. The model was solved and release profiles were simulated by numerical integration, matrix exponent calculations and calculus solutions that support parameter search via derivation.

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PP-12 Investigation of the growth kinetics of $ZnAl_2O_4$ spinel phase in cylindrical geometry

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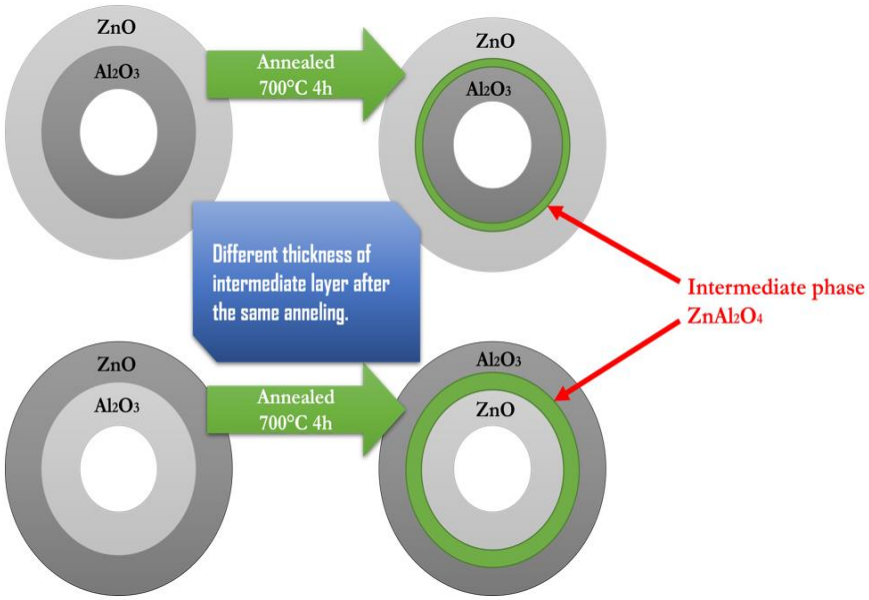
GRAPHICAL ABSTRACT



ABSTRACT

Zinc-aluminate can be used in several application for instance as a catalyst in the reduction of air polluting agents (NOx molecules) [1] and it has a significant potential for photocatalytic air purification [2]. Growth kinetics of $ZnAl_2O_4$ (zinc aluminate) phase was followed in cylindrical geometry. Al_2O_3 and ZnO were deposited on poly(vinyl alcohol) (PVA) nanofibers in two different layer sequences. The influence of closed geometry and stacking order on spinel growth between two oxide layers has been experimentally investigated. PVA nanofibers prepared via electrospinning method [3] were used as templates to produce bilayered nanowires. ZnO/Al_2O_3 and Al_2O_3/ZnO bilayers were deposited on the substrate via atomic layer deposition (ALD) to investigate the influence of stacking order. The samples were annealed at high temperatures (700°C) in air and after that they were investigated using scanning and transmission electronmicroscopy as well as X-ray diffractrometry to follow the growth kinetics of $ZnAl_2O_4$.

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PP-13 Mechanical characterization of cellulose aerogels

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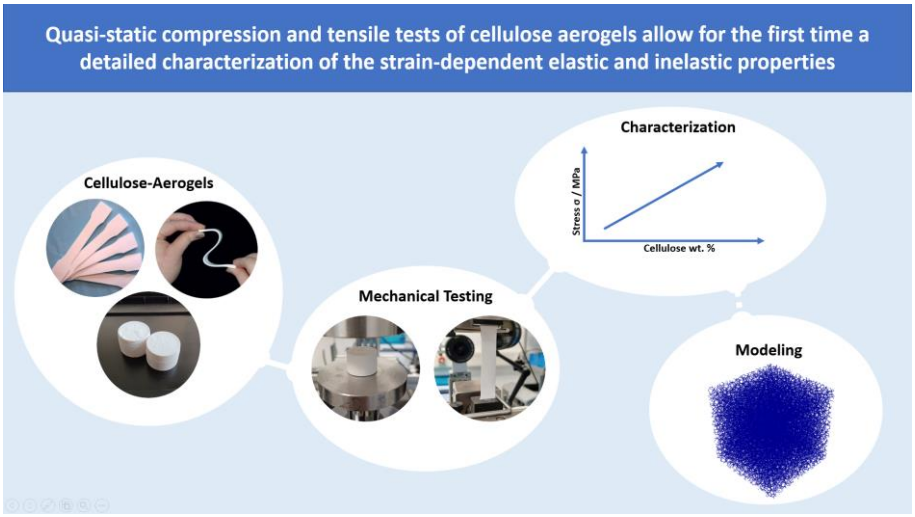
GRAPHICAL ABSTRACT



ABSTRACT

Due to dwindling fossil resources, bio-based cellulose aerogels, whose three-dimensionally structured networks are characterized by nanoscale fibrils, have been of particular interest in recent years. They can be produced by bringing the polymer chains into solution and subsequent regeneration processes and offer the low density and thermal conductivity typical of aerogels. Their bulk properties depend on their nano- and microstructure, which is influenced by their manufacturing process [1]. For practical applications of cellulose aerogels, insights into their elastic and inelastic mechanical properties are desired. To the best of our knowledge, the reports in the literature merely describe the stress-strain curves under monotonic uniaxial compressive loading [2] without exploring the inelastic features. This work aims at extending the state-of-the-art know-how on mechanical characterization of cellulose aerogels within this context. For this purpose, cellulose aerogels having different cellulose concentrations synthesized using $ZnCl_2$ as solvent, salt hydrate – routine [3] were subjected to an intensive mechanical characterization. This included quasi-static compression and tensile tests, which for the first time allow a detailed characterization of their strain-dependent elastic as well as inelastic properties. Furthermore, the results will be illustrated in the context of computational design of their microstructure with already established approaches [4] to better investigate structure-property relations in the future.

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PP-14 Thiol functionalized mesoporous silica sorbent for selective sorption of aqueous Ag(I)

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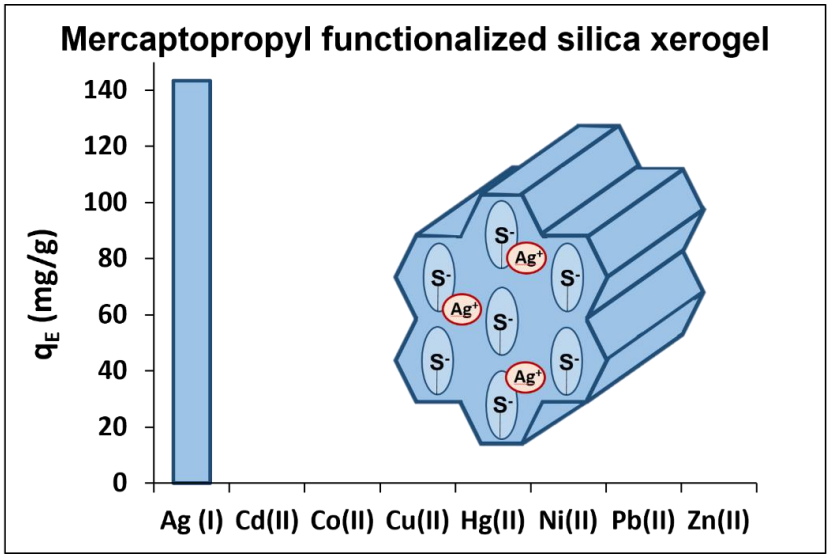
Graphical abstract



ABSTRACT

In the last decades, the concentration of silver compounds in surface waters has increased significantly due to their wide range of technological applications. The high concentration of silver compounds may cause toxic effects on the human body: irritate eye, skin, breathe, digestive system and altering blood cells. Due to its unique, advantageous properties silver is an invaluable substance in many industrial applications. The recovery of silver ions from aqueous solutions containing other metal compounds is an important task of environmental technology. One solution is the development of selective sorbents by the appropriate design

of surface functionality. For this purpose, a mesoporous, mercaptopropyl functionalized silica xerogel was prepared by the sol-gel method. It is characterized by low voltage scanning electron microscopy (LV-SEM), N_2-sorption porosimetry, EDX analysis and X-ray photoelectron spectroscopy (XPS). Its aqueous phase Zeta potential was investigated as a function of pH. The xerogel has excellent selectivity for binding Ag(I) in the simultaneous presence of seven other metal ions at pH = 5.0, with a very high sorption capacity of 238 mg g^{-1}. The sorption equilibrium establishes extremely fast, after 15 min which is a major advantage regarding the practical functionality of the present xerogel sorbents. The quantitative recovery of Ag(I) and the regeneration of the sorbent is possible using 10.0 mM $Na_2S_2O_3$ solution. The mechanism of binding can be explained by the formation of Ag(I) clusters on the surface, which is usual for silver ions in the presence of thiol groups. This explains the excellent selectivity of the sorbent for silver ions.

ACKNOWLEDGEMENTS

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

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PP-15 Hydration mechanism of borosilicate-PVA aerogels

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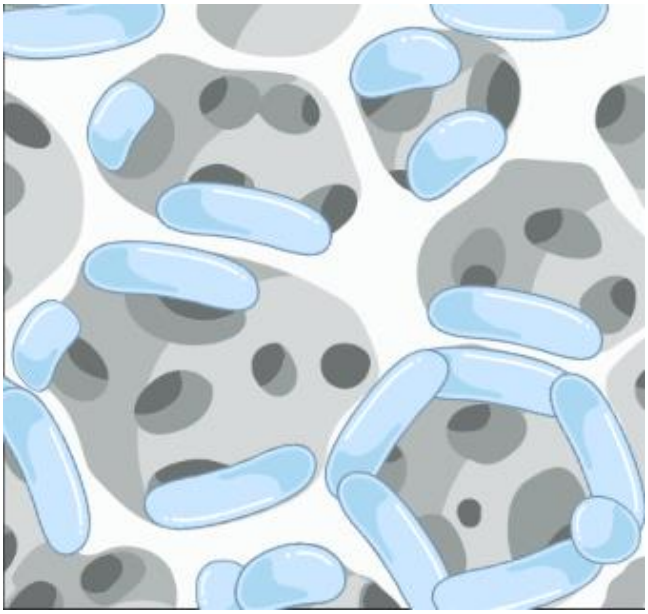
GRAPHICAL ABSTRACT



ABSTRACT

Understanding the hydration mechanism of the aerogels is essential because they are commonly used in aqueous media. In this study we investigated the hydration of two types of hybrid aerogels, using different liquid phase ^{1}H-NMR techniques like NMR relaxometry, NMR cryo-porometry and NMR diffusiometry. Both of the investigated aerogels are borosilicate-PVA hybrid aerogels, and one of them was functionalized with hydroxyapatite.

First of all, NMR relaxometry was used to model the hydration mechanism of these materials [1]. To investigate the hydration mechanism, the samples were gradually filled with distilled water. After that NMR relaxometry was used to measure the T_2 values of the aerogels. To measure the spin-spin relaxation times of water molecules in the pores we used CPMG method.

The water molecules can be located in different section of the pores, e.g., on the surface of pores or in bulk water, far from the surface. Water can be found in different relaxation

domains in a sample. Each relaxation domain contributes to the measured signal by a single exponential decay, and the observed signal is the sum of these decays.

The two investigated aerogels hydration mechanism was similar. In each case there were two domains with small T_2 values at low water content. One of these domains is where the water molecules attached to the borosilicate structure, the other domain was attached to the PVA part. The third domain appeared when the filling factor was about 0.5 g/g for both aerogel. This domain is probably forming small puddles with higher relaxation times. At higher water content, the fourth domain appeared with much higher T_2 values. This is the signal of the bulk water. In both case the pores were totally filled in 2.0 g/g water-aerogel ratio.

During the NMR-cryoporometry measurements the hydrated aerogels were measured by a freezing-melting method where the pore geometry and the pore distribution of the wet aerogels were examined [2]. According to this experiment, the pores are almost four times larger in the functionalized aerogels than in the non-functionalised one, and this result is similar to the nitrogen sorption porosimetry experiment. The pore geometry was spherical in the non-functionalized and cylindrical in the functionalized aerogel. From these results it is clear that there is no morphological change due to the hydration in the case of both aerogels.[3].

The hydration properties of the aerogels are similar, and the functionalized one can be a good bone replacement material in the future.

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PP-16 Syntheses and Characterization of Flexible Polyimide Aerogels

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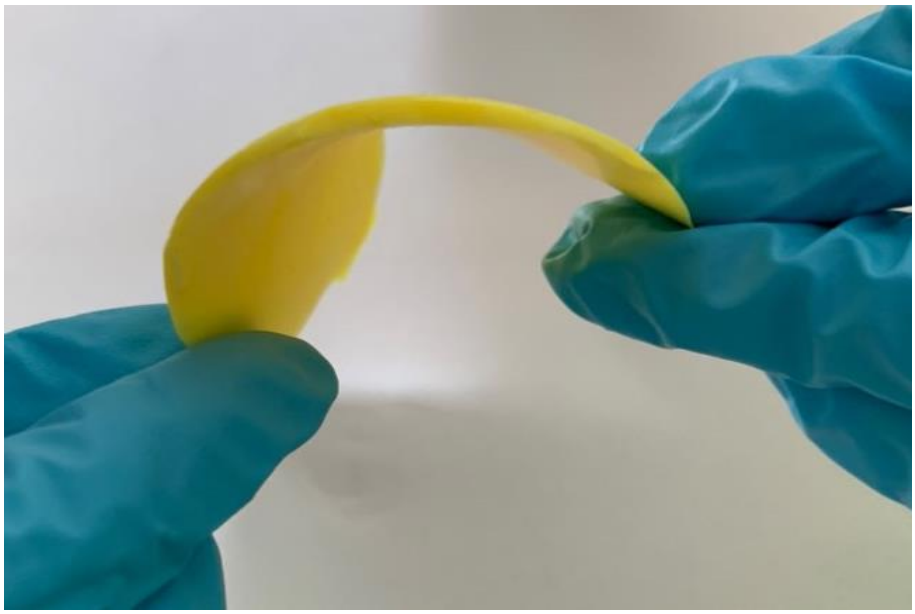
GRAPHICAL ABSTRACT



ABSTRACT

Polyimide aerogels are mesoporous materials characterized by low densities, low thermal conductivities, good mechanical properties and relatively high thermal stabilities. [1] These properties can be utilized in many applications. Here, their appliance as thermal insulators are to be underlined. In the past decade polyimide aerogels have gained considerable interest in the airspace industry, after NASA exemplified the prospect of their utilization as thermal insulators for Entry, Descent, and Landing (EDL) systems. Detailed analysis of the structure, the physical and mechanical properties of these materials is essential to ascertain their usage suitability, especially as part of insulation layers.

For the synthesis of polyimide aerogels, the acid dianhydride BPDA (biphenyl-3,3',4,4'-tetracarboxylic dianhydride) and the diamine DMBZ (2,2'-Dimethylbenzidine)/ODA (4,4'-oxydianiline) dissolved in NMP (N-methylpyrrolidinone) are mixed in a 1:1 stoichiometric ratio,

at room temperature. [2] The polyamic acid oligomers driven by this reaction, are cross-linked in a three-dimensional structure with the aid of TAB, and the mixture is chemically imidized upon the addition of acetic anhydride and pyridine. In the final step, the gels are supercritically dried with liquid CO_2. This particular synthesis route yields very little shrinkage and provides the optimal combination of light specific weight, high strength and high thermal stability. Most notably, these aerogels can be fabricated into thin layers, which resulted in being flexible and foldable, making polyimide aerogels the materials of choice when it comes to EDL system insulators, for which the flexibility is an essential requirement.

To get a clear picture of the polyimide aerogel structures, SEM images were taken, without coating the sample with a conductive layer and by applying acceleration voltage of 1-2kV when imaging. [3] In addition, N_2-adsorption and desorption isotherms were measured and surface areas as high as 660m^2/g were calculated. [4] As the last step, the variation of Young's Modulus with increasing humidity level conditioning was measured. A significant correlation was observed between these parameters in the case of the ODA-derived aerogels. The Young's modulus first increases from 4 MPa with increasing humidity level, then shows a maximum with 38 MPa at medium levels of humidity, and finally decreases at high humidity conditioning.

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PP-17 Self-sterilizing PVA electrospun membranes

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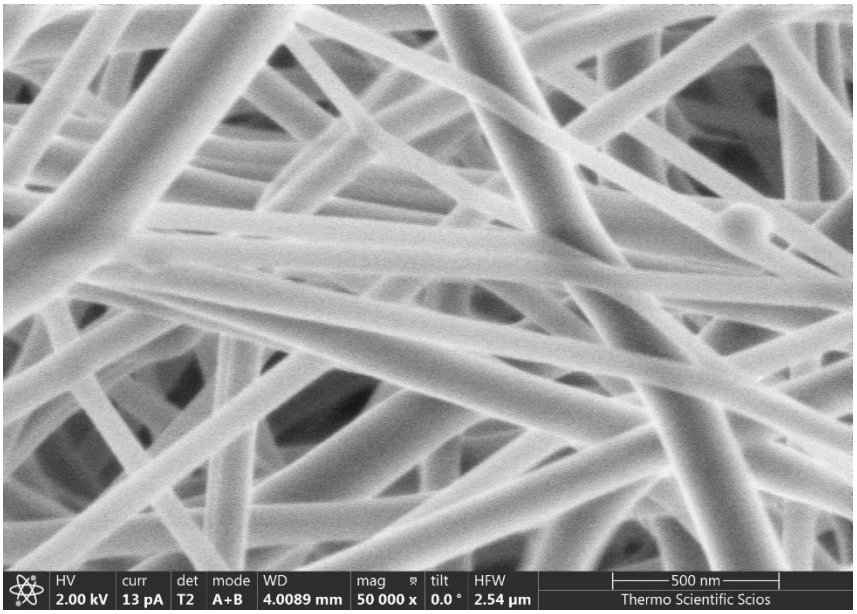
GRAPHICAL ABSTRACT



SEM image of PVA fibers prepared with sodium chlorite.

ABSTRACT

Nanomaterials produced by electrospinning method are extensively used in numerous medical applications. They are also widely utilized in biomedicine, tissue and filtration technology, air and water purification, sensor technology and cosmetics.[1] According to their drug-carrying properties, they are also suitable for targeted delivery of antibiotics, anti-cancer drugs, antioxidants, proteins and DNA molecules within the body. During the use of fiber-drawn materials as drug carriers, the high surface/volume ratio enables rapid release of active ingredient.[2,3]

With electrospinning, polymer fibers with different chemical compositions can be manufactured in a variety of shapes. A significant limitation of the use of biocompatible polymers for human medicinal purposes is that they can only be applied under sterile conditions. Traditional sterilization methods are not suitable for disinfecting these materials because the inner surface of their porous structure is not easily accessible and these procedures can change the physico-chemical properties of the polymers.

Now, we report novel PVA fibrous materials prepared with a well-known disinfectant, sodium chlorite. SEM images reveal that uniform porous structures form with 160 nm fibers. We analyzed the chlorite ion content of these materials with ion chromatography and investigated the long-term stability of chlorite ion.

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