INTERNATIONAL SOL-GEL SOCIETY

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eMarathon

13 March 2025

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Following the success of the inaugural ISGS PhD Student eMarathon in March 2024, the ISGS will hold the second PhD Student eMarathon on 13 March 2025. The eMarathon will involve a series of lectures by students who are currently undertaking their PhD in sol-gel science and technology, with each lecture being of 30 minutes duration and an additional 10 minutes for questions. Our goal is to showcase the depth and diversity of the sol-gel research being undertaken by our students in academia, industry and/or national laboratories, and to provide an opportunity for them to present their work to an international audience in a relaxed, online environment.

We look forward to your participation in the inaugural event and to your support of our PhD students, the future leaders of the sol-gel community of practice.

Date: **Thursday 13 March 2025**

Time:

01:50 - USA (East Coast)	03:50 - Chile, Argentina and Brazil (São Paulo)
06:50 - UTC	06:50 - UK, Portugal
07:50 - Switzerland, Central Europe, Sweden	08:50 - Romania, Israel, Lithuania, Ukraine
09:50 - Russia (Moscow, St Petersburg)	14:50 - China, Singapore, Malaysia
15:50 - Japan, Korea	16:50 - Australia (Queensland)

ZOOM: https://us05web.zoom.us/j/8867581791?pwd=T0c2REcyL2prNy9EZXNVWEwrZ1JiUT09&omn=82741308381

Meeting ID: 886 758 1791
Passcode: 1jDKEJ

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Program

Speaker	UTC	Europe/ Tunisia	Israel/ Lithuania	Brazil/ Argentina	New Zealand	Australia (Qld)
John Bartlett (*ISGS*) **Opening Comments**	06:50-07:00	07:50-08:00	08:50-09:00	03:50-04:00	19:50-20:00	**16:50-17:00**
Kate Wislang (*University of Canterbury, New Zealand*) **Electrical Studies of Ga_2O_3 Thin Films Produced Using Sol-Gel Techniques**	07:00-07:40	08:00-08:40	09:00-09:40	04:00-04:40	**20:00-20:40**	17:00-17:40
May Yam Moshkovitz-Douvdevany (*The Hebrew University of Jerusalem, Israel*) **Alumina Structures Crafted with Light and Chemistry**	07:40-08:20	08:40-09:20	**09:40-10:20**	04:40-05:20	20:40-21:20	17:40-18:20
Olzhas Shalkhar (*Vilnius University, Lithuania*) **Exploring the Optical Properties of YAGG:Ce Co-Doped with Lanthanides through Sol-Gel Processing**	08:20-09:00	09:20-10:00	**10:20-11:00**	05:20-06:00	21:20-22:00	18:20-19:00
Break	09:00-09:30	10:00-10:30	11:00-11:30	06:00-06:30	22:00-22:30	19:00-19:30
Willi Max Leopold (*TU Bergakademie Freiberg, Germany*) **Enhanced Mechanical and Anti-Corrosive Properties of Hybrid Sol-Gel Coatings Through Statistical Optimization Methods**	09:30-10:10	**10:30-11:10**	11:30-12:10	06:30-07:10	22:30-23:10	19:30-20:10
Giovanna Li Petri (*CNR, Palermo, Italy*) **Advancing SilverSil Sol-Gel Chemistry Towards Novel Antibacterial Coatings**	10:10-10:50	**11:10-11:50**	12:10-12:50	07:10-07:50	23:10-23:50	20:10-20:50
Randy Haddad (*Institute Polytechnique de Paris, France*) **Omniphobic coatings based on raspberry-like nanoparticles and their applications on cotton**	10:50-11:30	**11:50-12:30**	12:50-13:30	07:50-08:30	23:50-00:30	20:50-21:30

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Program (continued)

Speaker	UTC	Europe/ Tunisia	Israel/ Lithuania	Brazil/ Argentina	New Zealand	Australia (Qld)
Break	11:30-12:00	12:30-13:00	13:30-14:00	08:30-09:00	00:30-01:00	21:30-22:00
Jihane Khadhraoui (*University of Sousse, Tunisia*) **Synthesis and characterization of Cellulose-based nanocomposites**	12:00-12:40	**13:00-13:40**	14:00-14:40	09:00-09:40	01:00-01:40	22:00-22:40
Guiseppe Angellotti (*CNR, Palermo, Italy*) **Sol-Gel Encapsulation of Essential Oils for Enhanced Stability and Insecticidal Activity**	12:40-13:20	**13:40-14:20**	14:40-15:20	09:40-10:20	01:40-02:20	22:40-23:20
Ángela Solis-Garrido (*Carlos III University of Madrid, Spain*) **Sol-Gel Siloxane Networks for Rapid Bacteria Detection and Identification**	13:20-14:00	**14:20-15:00**	15:20-16:00	10:20-11:00	02:20-03:00	23:20-00:00
Break	14:00-14:30	15:00-15:30	16:00-16:30	11:00-11:30	03:00-03:30	00:00-00:30
Thais C.A. da Silva (*Universitat Jaume I, Castelló, Spain*) **Microwave-Assisted Synthesis of $CsPbBr_3$ Perovskite Nanocrystals: Towards Efficient and Scalable Light-Emitting Applications**	14:30-15:10	**15:30-16:10**	16:30-17:10	11:30-12:10	03:30-04:10	00:30-01:10
Judith González-Lavin (*INCAR-CSIC, Oviedo, Spain*) **Microwave-Assisted Sol-Gel Synthesis of Transition Metal Aerogels: Unlocking Advanced Property Tunability by Innovative Heating Techniques**	15:10-15:50	**16:10-16:50**	17:10-17:50	12:00-12:50	04:10-04:50	01:10-01:50
Leonardo Marchiori (*UNESP, Araraquara, Brazil*) **Aerogel Materials for Photocatalysis, a PhD story**	15:50-16:30	16:50-17:30	17:50-18:30	**12:50-13:30**	04:50-05:30	01:50-02:30
Close	16:30	17:30	18:30	13:30	05:30	02:30

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Electrical Studies of Ga_2O_3 Thin Films Produced Using Sol-Gel Techniques

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Abstract

Ga_2O_3 is an ultra-wide bandgap semiconductor with high thermal stability and breakdown strength attracting considerable interest for use in power electronics and solar-blind UV photodetectors [1]. Several common techniques to produce Ga_2O_3 exist, however, long growth times, low throughput, and high cost prevent Ga_2O_3 from reaching widespread adoption. Using the Sol-Gel technique, transparent and smooth Ga_2O_3 thin films were produced orders of magnitude faster growth rate and at a fraction of the cost and energy demands. Economical Ga_2O_3 fabrication using Sol-Gel will change the landscape of electrical device manufacturing, however, similar electrical properties must first be achieved.

Doping, increasing film thickness, and high-temperature annealing were used to improve the electrical properties of the as-grown Sol-Gel films. Sn- and Si-doped Sol-Gel Ga_2O_3 films were fabricated on *c*-plane sapphire and quartz substrates. This process was optimised to produce films up to 500 nm in thickness with 100% visible transparency, and low surface roughness (0.3 nm rms). Moss-Burstein type shifts were observed for both dopants, with significant increases in optical bandgap with doping concentration, a maximum shift of 0.29 eV for the Sn-doped material.

The electrical properties of 100 nm Sn- and Si-doped Ga_2O_3 films were measured using Ti/Au Transmission Line Rings (TLR), with gap sizes of 10-75 µm. While the films conducted, modest performance suggested low activated dopants mobility. 500 nm multi-layered films were produced to overcome surface depletion layer effects and thereby increasing the carrier quantity. TLRs showed increased performance to 10^{-6} A at 10 V, 15 µm gap size. PtOx/Pt Schottky diode devices were fabricated and showed strong rectifying behaviour [2]. H donation was demonstrated via forming gas annealing, though the effect was temporary due to H_2 being highly mobile.

High-temperature annealing (500°C to 1100°C) in N_2 gas, formed polycrystalline β-Ga_2O_3 in the amorphous as-prepared films. TLRs showed the electrical properties were not greatly improved and reduced as the annealing temperature increased, possibly due to grain boundaries and potential diffusion from the substrate.

Ongoing investigations into improved Ga_2O_3 dopants show fluoride having a promising theoretical potential as a donor [3]. Preliminary results are encouraging, 100 nm 0.01% SnF_2-doped films produced a current of 10^{-10} A at 10 V, orders of magnitude higher than Sn- and Si-doped films.

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Alumina Structures Crafted with Light and Chemistry

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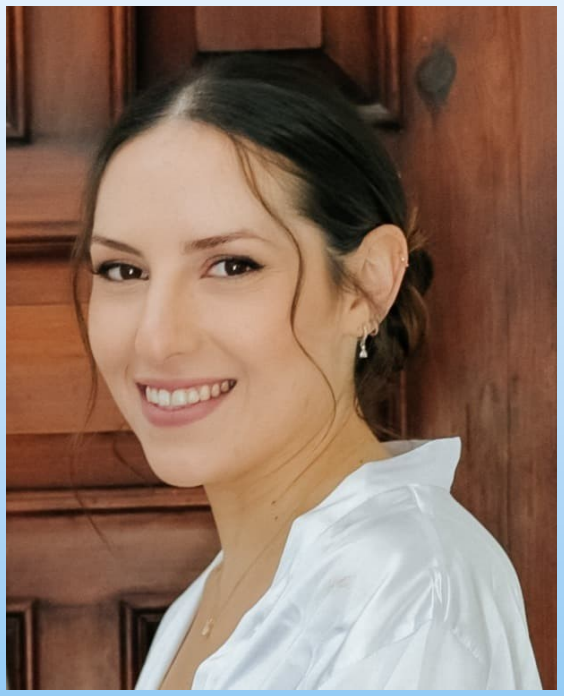
Abstract

Ceramic manufacturing has long been constrained by the limitations of traditional methods, particularly when it comes to producing intricate, high-precision designs. Conventional ceramic 3D printing often faces significant challenges, such as particle settling, light scattering, and the high costs of nanoparticle-based materials.

This research addresses these challenges with a novel method for fabricating alumina-based ceramics by combining photopolymerization with sol-gel processes. Photopolymerization develops complex structures from a particle-free solution of soluble metal salt precursors. The subsequent sol-gel process transforms these precursors into alumina, eliminating nanoparticle need and reducing production costs.

This approach enables control over the composition and microstructure of alumina-based ceramics. We have successfully 3D-printed transparent, porous γ-alumina structures with controlled porosity and optical clarity and developed a particle-free method for printing luminescent chromium-doped α-alumina (Ruby), demonstrating crystallization control, making them ideal for applications in aerospace, biomedical engineering, optics, electronics, and defense.[1,2]



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Exploring the Optical Properties of YAGG:Ce Co-Doped with Lanthanides through Sol-Gel Processing

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Abstract

This study investigates the sol-gel synthesis and photoluminescence properties of $Y_3Ga_3Al_2O_{12}:Ce^{3+}$ (YAGG:Ce) garnet phosphors co-doped with a series of lanthanide ions. The sol-gel method was chosen due to its advantages over conventional solid-state reactions such as effective chemical homogeneity, stoichiometric control, and production of highly uniform and nanoscale particles at lower temperatures. These traits are of utmost importance for enhancing the luminescent performance of phosphor materials since they affect the crystallinity, phase purity, and defect concentration of the host lattice [1,2].

X-ray diffraction was employed to characterize the synthesized phosphors in order to verify the formation of the garnet phase and scanning electron microscopy was employed to gain insights into their morphology and particle size. The emission properties and energy transfer mechanisms between Ce^{3+} and the co-doped lanthanide ions were analyzed using photoluminescence spectroscopy.

The findings show that incorporating lanthanides into the host matrix changes the luminescent behavior, with specific dopants enhancing the emission intensity and others affecting a spectral shift due to variations in ionic radius and electronic configuration. The nature of the dopant had been found to have a significant impact on energy transfer efficiency and quenching effects, emphasizing the role of sol-gel synthesis in adjusting optical characteristics [3,4].

This study demonstrates how well the sol-gel method works as a flexible technique for preparing unique phosphor materials. Sol-gel synthesis makes it easier to optimize YAG:Ce-based phosphors to be used in radiation detection, display technologies, and LEDs by enabling uniform microstructures and controlled doping [5,6].

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Enhanced Mechanical and Anti-Corrosive Properties of Hybrid Sol-Gel Coatings Through Statistical Optimization Methods

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Abstract

This study presents a systematic investigation of hybrid sol-gel coatings as an environmentally friendly alternative to conventional chromium-based corrosion protection systems. The sol-gel process was used to develop versatile coatings that adhere to a variety of surfaces without extensive pretreatment. The formulations were synthesized using tri- and tetra-functional silanes with metal-organic titanium and zirconium derivatives as curing agents, with fillers such as talcum, zinc dust and micaceous iron ore incorporated to enhance the mechanical and anti-corrosive properties.

A definitive screening design (DSD) was used to systematically evaluate key synthesis parameters, including temperature, pH, reactant ratios, and water content. Zinc dust fillers provided active corrosion protection via sacrificial anodic mechanisms, while talcum-filled formulations exhibited improved adhesion, providing an alternative barrier protection strategy.

Nine months of outdoor weathering tests demonstrated the durability of the coatings, with impedance spectroscopy confirming stable performance in zirconium cured formulations. Initial results indicate that these hybrid coatings provide a combination of barrier and active protection while maintaining mechanical stability. The inorganic network exhibits resistance to UV degradation and improved temperature stability compared to purely organic coatings.

A comprehensive characterization approach, including impedance spectroscopy, infrared spectroscopy, and scanning electron microscopy, provided insight into coating performance and degradation mechanisms. Statistical analysis identified non-linear interactions between synthesis parameters and obtained analytical results, allowing data-driven optimization of mechanical and anti-corrosive properties.

These results support the development of sustainable, high-performance anti-corrosion coatings and highlight the effectiveness of statistical design tools in materials optimization.

Acknowledgment

This work was funded in part by public funds from the Federal Ministry of Education and Research and the State of Saxony (Project Number: SN0390002). The support of the European Social Fund (ESF) and the Sächsische Aufbaubank (SAB) for the doctoral research associated with this study is gratefully acknowledged.

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Advancing SilverSil Sol-Gel Chemistry Towards Novel Antibacterial Coatings

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Abstract

Organically modified silicates (ORMOSIL) are hybrid materials synthesized via the hydrolytic polycondensation of silanes bearing organic functional groups, often combined with traditional alkoxide precursors.[1] Due to their tunable properties, which are enabled by the powerful and versatile sol-gel process, ORMOSIL-based materials have attracted significant interest across diverse fields, including optics, electronics, energy, environmental science, and biomedicine.[2] Among various microencapsulation strategies, the sol-gel entrapment of metal nanoparticles (NPs) within ORMOSIL matrices provides key advantages, such as enhanced nanoparticle stabilization and the formation of functionalized materials in different formats, including powders, films, coatings, and monoliths, making it versatile for multiple applications.[3]

SilverSil, a sol-gel-based material introduced in 2020, integrates silver nanoparticles (AgNPs) within an ORMOSIL matrix, demonstrating broad-spectrum antibacterial activity.[4] In this lecture, I will present the synthesis and characterization of new SilverSil materials developed through a two-step sol-gel hydrolytic polycondensation of tetraethyl orthosilicate (TEOS) and organically modified silanes, including methyltriethoxysilane (MTES) and (3-aminopropyl)trimethoxysilane (APTMS), with and without different concentrations of $AgNO_3$. The influence of key parameters, including alkoxide/water ratio, ethanol-free formulations, gel aging time, and alcogel/hydrogel drying conditions, on the final material properties was systematically investigated.[5]

Both blank and Ag-doped SilverSil materials, appropriately dispersed in two different bio-based resins and an acrylic-based thickener, were successfully deposited onto cotton fabrics via a printing technique. Antibacterial assessments demonstrated up to 90% efficacy against both gram-positive (*S. aureus*) and gram-negative (*E. coli*) bacterial strains, highlighting SilverSil's potential for antimicrobial textile applications, including medical garments, protective clothing, and other functional fabrics requiring long-lasting antibacterial protection.

Acknowledgements

This study was carried out within the MICS (Made in Italy – Circular and Sustainable) Extended Partnership and received funding from the European Union Next-GenerationEU – Piano Nazionale di ripresa e resilienza (PNRR) – Mission 4 Component 2, Investment 1.3 – D.D. 1551.11-10-2022

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Omniphobic coatings based on raspberry-like nanoparticles and their applications on cotton

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Abstract

To date, polyfluorinated molecules (PFAS) are employed industrially to confer water repelling and oleophobic properties in food packaging, antifogging glass and textile surfaces treatments. However, there are increasing concerns regarding their bioaccumulation and many severe environmental issues. This motivates very active research toward the development of environmentally friendly alternatives.

We here focus on the case of amphiphobic textiles that requires the surface treatments of polymer fibers. The developed strategy is both to achieve the appropriate surface modification with molecular species alternative to PFAS and the engineering the optimal hierarchical roughness known to allow some enhancement of omniphobic properties.

The first phase of the process involves epoxying and activating the surface of cellulose fibers by grafting an organic monolayer, providing epoxy groups for covalently bonding nanoparticles to the textile fibers. Subsequently, by mimicking the topology of naturally self-cleaning Lotus plant leaves, engineered hierarchical surface roughness is ensured employing raspberry-like silica particles. The obtained surface is then treated through the dendrimeric polymerization of precursors, generating branched alcoholic chains for multiple-anchor grafting thus leading to optimized density of surface functional groups. Finally, silicone-type polymers based on polydimethylsiloxane chains are grafted thus revealing hydrophobic and water-repellent properties.

The presentation will describe the global strategy and discuss the different parameters allowing for the engineering of textiles with optimal wetting properties. The results will be compared to the previous strategy using PFAS. We will also show that the method finds straightforward and adaptable applications for the industrial high scale processing of textile, and discussion will be complemented by results regarding the stability of the treatment toward ageing and multiple washing as in real conditions of textile use.

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Synthesis and characterization of cellulose-based nanocomposites

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Abstract

The development of suitable materials for a wide range of applications, such as energy storage and conversion systems, gas sensors, dielectric components, and insulation materials, along with the responsibility to address environmental issues, are two vital reasons behind research efforts. The primary objective of this research is to explore the potential of cellulose-based nanocomposites, aiming to combine the eco-friendly nature of cellulose with the superior functional properties of graphene and graphene derivatives. To optimize the synthesized materials for specific applications, morphology and structure were studied through scanning electron microscopy (SEM), Transmission electron microscopy (TEM), X-ray diffraction (XRD), and Fourier Transform infrared spectroscopy (FTIR). Electrochemical studies such as Cyclic Voltammetry (CV), Electrochemical Impedance Spectroscopy, and Galvanostatic Charge discharge analysis (GCD) have shown a pseudocapacitive behavior of the nanocomposites.

Keywords: Cellulosic fibers, reduced graphene oxide, Metal/Oxide nanoparticles, energy storage systems

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Sol-Gel Encapsulation of Essential Oils for Enhanced Stability and Insecticidal Activity

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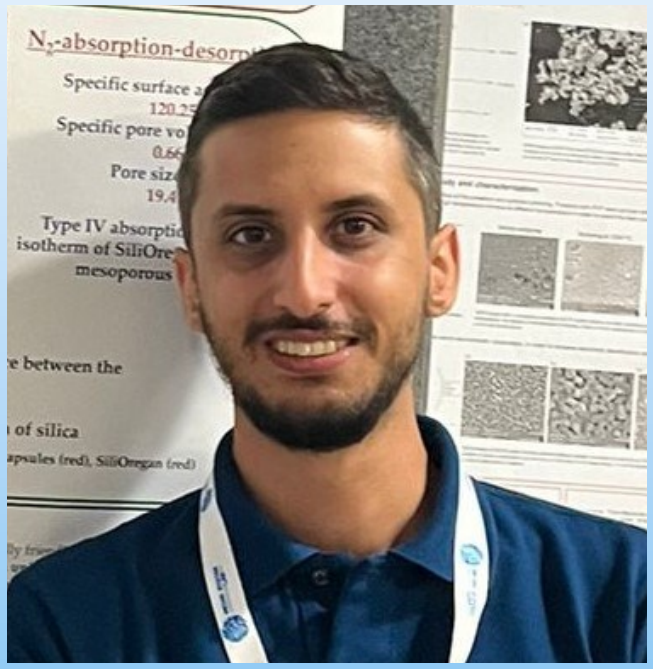
Abstract

The widespread use of synthetic pesticides poses significant threats to both ecosystems and human health, with climate change further intensifying challenges in agriculture and pest management. Globally, approximately 3.5 billion kilograms of synthetic pesticides are applied, contributing to environmental contamination and long-term health concerns. In response, essential oils (EOs) have gained attention as natural alternatives due to their insecticidal properties. Extracted from plants such as *Citrus*, *Origanum vulgare*, and *Rosmarinus officinalis*, EOs exhibit strong larvicidal, insecticidal, and repellent effects. However, their high volatility and instability in environmental conditions hinder practical field applications [1]. To overcome these limitations, a microencapsulation strategy was developed. This involved the preparation of oil-in-water (O/W) emulsions of EOs using surfactants, which acted as microreactors for the sol-gel polymerization of silicon alkoxides, leading to the formation of mesoporous SiO_2 shells. This encapsulation process improved the stability of EOs, prolonged their shelf life, and enabled a controlled release mechanism [2-3]. The resulting microcapsules displayed submicrometric sizes, high surface areas, and mesoporous structures. Scanning electron microscopy (SEM) confirmed their spherical shape, uniform size distribution, and the presence of internal cavities indicative of in situ EO encapsulation. Quantitative assessments demonstrated high encapsulation efficiency, validating the suitability of the sol-gel method for EO preservation. Release studies further confirmed a controlled and sustained release of EOs over time. Additionally, the pesticide efficacy of these microcapsules was tested against *Spodoptera littoralis*, a common agricultural pest, showing promising results for pest control applications.



Acknowledgments

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Sol-Gel Siloxane Networks for Rapid Bacteria Detection and Identification

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Abstract

Almost one million patients get an arthroplasty in the USA per year [1] and the possibility of infection impacts around 2% of the cases [2]. There are also removable devices such as catheters, used daily, that are also prone to infection. Therefore, pathogens identification, to overcome and treat the issue, is essential and always associated with long waiting times. When combined with the rising incidence of polymicrobial infections and the emergence of new drug-resistant strains, this important problem encourages the developing of new technologies to try to palliate them. For this reason, this research proposes the development of a novel device for rapid bacteria detection and identification, competitive with traditional procedures. This early diagnosis method could be promising as a novel alternative to early detection and identification.

In this context, the development of a sol-gel-derived detector material based on a siloxane network and synthesised by sol-gel technology was carried out [3],[4]. Sol-gel siloxane networks can be used as a carrier to locate chromogenic molecules without interfering with the network itself. These molecules are able to colour when reacting selectively in the presence of bacteria and, therefore, act as a rapid detector. For this purpose, this work is focused on *Staphylococcus aureus* detection, since as seen in prior etiological studies, *it* is one of the most encountered bacteria found in biodevices infections.

In the first place, synthesis and deposition methods on the substrate were optimised. The formulations prepared contain potassium tellurite (TEL) as chromogenic molecule, as it can react with *S. aureus* presenting a black colour. TEL was physically and superficially trapped into the sol-gel network, as verified by SEM and the degradation of the carrier in the presence of infected corporal fluids was evaluated by EIS techniques. Also *in vitro* assays were carried out, checking colouring ability towards *S. aureus* cultures and determining the kinetics colof ouration. The *in vitro* assays include specific tests to prove that this chromogen is only susceptible to *S. aureus* strains, by performing similar assays with other strains (*P. aeruginosa*, *S. epidermidis*, *E. faecalis* y *E. coli*).

The developed material proved to be functional and specific towards *S. aureus* detection in less than 10 hours. Also, the addition of TEL does not affect the formation of the polysiloxane network.

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[2] N. Benito *et al.*, *J Clin Med*, vol. 8, no. 5, p. 673, 2019, doi: 10.3390/jcm8050673.
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[4] A. Garcia-Casas *et al.*, *Colloids Surf B Biointerfaces*, vol. 181, pp. 973–980, Sep. 2019, doi: 10.1016/j.colsurfb.2019.06.042.

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Microwave-Assisted Synthesis of $CsPbBr_3$ Perovskite Nanocrystals: Towards Efficient and Scalable Light-Emitting Applications

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Abstract

Cesium lead bromide ($CsPbBr_3$) perovskite nanocrystals (PeNCs) have emerged as promising materials for optoelectronic applications due to their outstanding optical properties, includinghigh photoluminescence quantum yield (PLQY) and narrow emission linewidth. However, achieving high-quality NCs with controlled size, morphology, and composition while maintaining scalability remains a challenge. In this work, we explore a microwave-assisted synthesis approach to enhance the reproducibility and efficiency of $CsPbBr_3$ NC fabrication. This method offers rapid heating, uniform temperature distribution, and precise reaction control, leading to improved crystallinity and reduced defect density.

Despite not being a conventional sol-gel system, our study presents **interdisciplinary connections** with sol-gel chemistry, particularly in solution-based processing and controlled nucleation and growth mechanisms. By tuning reaction parameters such as precursor concentration, solvent environment, and microwave power, we optimize the synthesis to obtain highly stable and luminescent perovskite NCs. The potential of this approach for large-scale production and integration into light-emitting devices (LEDs) will be discussed.

This presentation aims to foster discussions on the intersection between perovskite nanocrystals and sol-gel techniques, highlighting novel strategies for material design and processing.

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Microwave-Assisted Sol-Gel Synthesis of Transition Metal Aerogels: Unlocking Advanced Property Tunability by Innovative Heating Techniques

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Abstract

Addressing climate change is one of the most pressing challenges of the century. The transition to sustainable energy solutions is essential to reduce greenhouse gas emissions and mitigate global warming. One promising pathway is substituting traditional fossil fuels conversion with hydrogen-based systems, which offer a clean and renewable alternative. Hydrogen fuel cells, in particular, have gained significant attention due to their high energy density and zero-emission operation, producing only water as a byproduct. Despite their potential, the widespread adoption of hydrogen fuel cells is limited by the reliance on noble metal electrocatalysts, and critical materials. These are costly, scarce, and geographically concentrated, making them unsuitable for large-scale use. Developing alternative electrocatalysts using transition metals offers an affordable and sustainable solution. Transition metals like iron, cobalt, nickel, and manganese exhibit diverse catalytic properties and hold promise for key reactions such as hydrogen evolution and oxygen reduction—key processes in fuel cell operation. However, their catalytic activity and stability need to be significantly enhanced to substitute noble metals. A critical strategy to achieve this is by increasing the active surface area and creating abundant catalytic sites.

Aerogels present a compelling solution to this challenge. Their ultra-low density, high porosity, and large surface area maximize the exposure of catalytic sites and facilitate efficient charge and mass transport. To further enhance their catalytic performance, the synthesis of transition metal aerogels (TMA) must be carefully controlled to tailor their properties, such as surface area, porosity, and composition. The sol-gel process emerges as a green and versatile chemical route for the synthesis of aerogels. This method involves the formation of a gel network from a solution, followed by drying to preserve the porous structure. The sol-gel process is inherently adaptable, allowing for the incorporation of various transition metals and the fine-tuning of their interactions within the gel matrix. A significant advancement in the sol-gel synthesis of TMA is the use of microwave heating to accelerate the gelation process and reduce energy consumption. Moreover, microwaves provide rapid and uniform heating, forming particles with homogeneous sizes and shapes. However, factors such as operating conditions (i.e., reaction time, temperature), or certain details of the process design (i.e., ratio volume/power) have a direct influence on the final properties of the TMA. For this reason, it is essential to understand the fundamentals of the process, so that structural and compositional changes can be foreseen and aerogels can be custom-designed for various electrocatalytic applications and, in particular, for fuel cell reactions. Furthermore, combining different transition metals during the synthesis of aerogels creates synergistic effects that enhance catalytic activity and stability. For example, iron can boost oxygen reduction reaction (ORR), while nickel excels in hydrogen oxidation reaction (HOR).

Despite the advantages of microwave-assisted sol-gel synthesis, one of the challenges lies in completely understanding the synthesis pathway. Therefore, this work proposes an explanation based on measurements during the process and the physicochemical characterization of the final aerogel. This study was focussed on monometallic iron (FeA) and nickel (NiA) aerogels, besides a bimetallic aerogel composed of these two transition metals (FeNiA). The three aerogels were synthesized at a temperature of 70°C for 1 hour in a total volume of 80 mL. The reaction of the precursors occurs in multiple stages, yielding water and CO_2 as by-products. SEM images of these materials reveal a nodular structure characteristic of aerogels, with particle sizes ranging from 40 to 70 nm and BET surface areas between 100 and 200 m^2/g, which are exceptionally high values for metallic materials. X-ray diffraction results show that the reaction products are oxidized phases of the metal or bimetal, while the supernatant consists of dissolved salts. Additionally, their electrochemical properties were studied through cyclic voltammetry (CV), and their stability was evaluated using chronoamperometry. TMA demonstrated good electroactivity and remained stable after more than 10000 seconds of operation.

In conclusion, understanding the synthesis process of aerogels through microwave-assisted sol-gel synthesis is essential for designing and tailoring TMA with specific properties. Moreover, the green chemistry principles underlying their synthesis ensure that their environmental impact is minimized, offering a ground-breaking solution to the limitations of current fuel cell technologies. As research in this field continues to advance, the widespread adoption of these innovative materials could play a pivotal role in the transition to sustainable and affordable energy supply.

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Aerogel Materials for Photocatalysis, a PhD story

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Abstract

Aerogel materials are mostly known for their high surface area and high porosity, although exclusively inorganic aerogels lack mechanical strength, hindering their range of applications. Supporting the inorganic phase on bacterial cellulose, for example, can overcome the main disadvantages of inorganic aerogels, producing a porous and mechanically stable material, resulting in an organic-inorganic hybrid aerogel. These high-surface-area hybrid materials are suitable for many applications, such as in-flow photo-assisted water purification, where the biocompatibility of the organic framework can be combined with the photocatalytic activity of inorganic coatings to create a high-performance filter for different organic and/or inorganic contaminants.

Here, we will present a summary of two recent articles developed in our group that highlights the beneficial combination on hybrid aerogels, with the support material being bacterial cellulose and the coatings made of SiO_2, TiO_2, and MoS_2. The target applications will include photocatalysis and sorption, with a focus on structure-properties relationships. The first article (10.1007/s10971-024-06380-2) explores MoS_2 supported on bacterial cellulose for dye degradation and highlights the importance of proper drying technique, either on membranes and sphere-like bacterial cellulose, and how the drying approach to create an aerogel, cryogel or xerogel, will impact the photocatalytic and sorption properties. The second article (10.1021/acsami.3c02008) shows the impact of a silica layer coating to enhance the photocatalytic activity and thermal stability of titania, both supported on bacterial cellulose. We demonstrate that controlling the amount of silica not only improves the dye degradation, but also helps the titania organization and controls the amount of titania that can be supported. With both articles and a brief introduction about aerogels, the authors hope to focus the audience's attention on materials' structure-properties relationships, and show recent work developed in our research group.

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