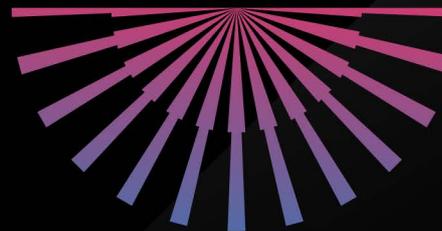


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40 YEARS OF THE FIRST
BRAZILIAN SYNCHROTRON
LIGHT SOURCE PROJECT



ABSTRACT BOOK

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MINISTRY OF
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AND INNOVATION



Laboratório Nacional de Luz Síncrotron (LNLS)

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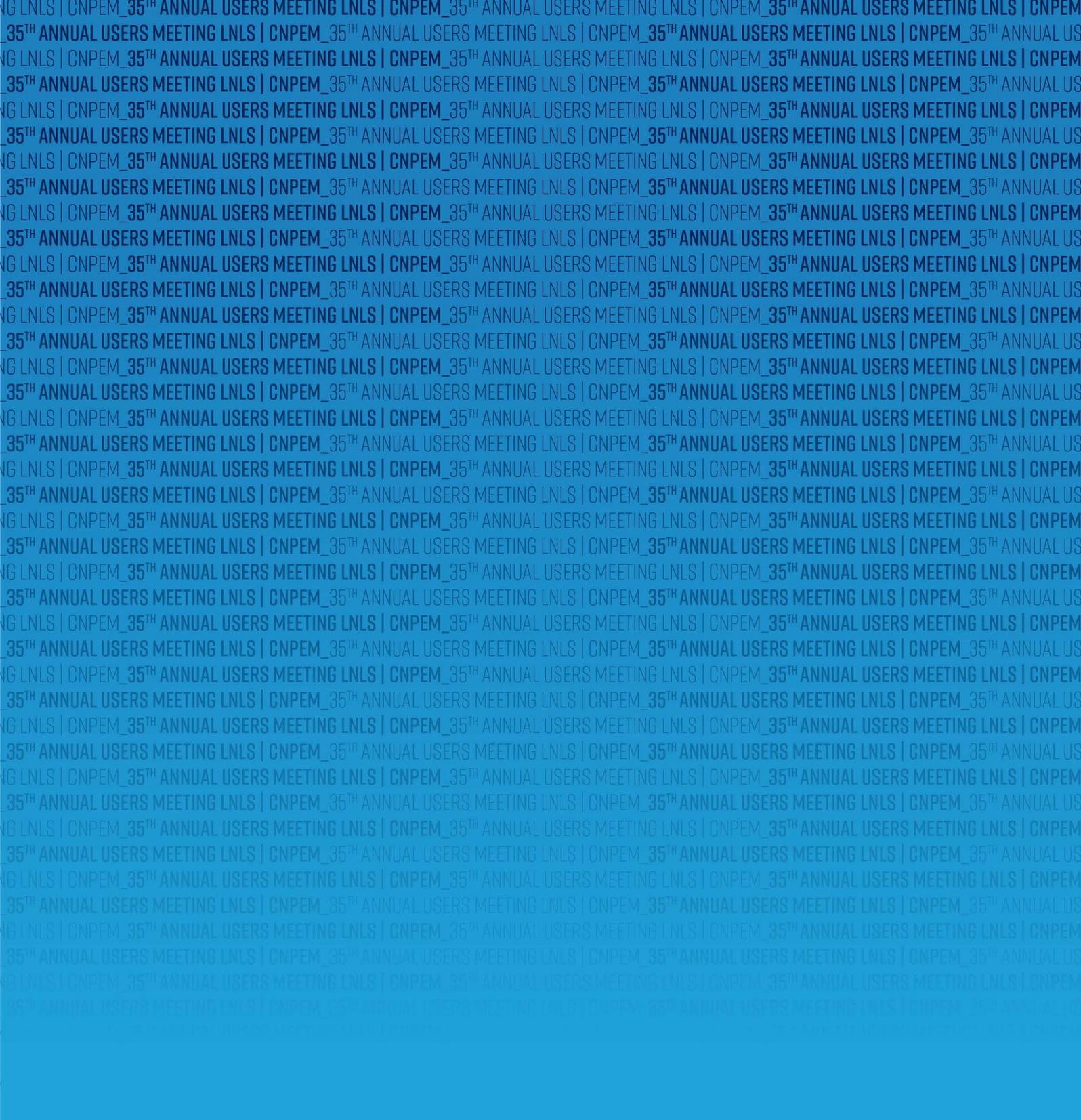
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PLENARY LECTURES





ORAL PRESENTATIONS





POSTERS



Characterization of agarose nanoparticles obtained by w/o/w microemulsion

Andreios Onuzak Aires¹, Nádyá Pesce da Silveira¹, Cilaine Verônica Teixeira¹

¹Universidade Federal do Rio Grande do Sul

e-mail: andreiosonuzak@hotmail.com

The present work investigates the physicochemical behavior of aqueous agarose solutions (0.2-10 mg·mL⁻¹) and agarose nanogels produced by W/O/W emulsions, focusing on main-chain dynamics and double-helix formation leading to gelation. Agar, a mix of cell-wall polysaccharides (agarose and agarpectin), is extracted from red algae (Rhodophyta) [1]; the agarose repeating unit comprises β-d-galactose (1,3) and α-1,3,6-anhydrogalactose (1,4) [2]. Agarose has broad biomedical uses including hydrogels, 3D scaffolds, drug delivery, antibody separation and cell encapsulation [3-9]. In solution agarose adopts a random-coil conformation above the gelation temperature; on cooling some segments form helices that aggregate via hydrogen bonds to create network cross-links [10-11]. SANS suggests relatively rigid chains with large persistence length ($l_p > 9$ nm) and helical motifs in gelation [12]. Experimentally, SAXS and XPCS were measured at synchrotron beamlines, complemented by SLS, DLS, TGA, FTIR and circular dichroism (CD); nanocapsules were prepared by W/O/W with a concentrated agarose phase. SLS/DLS reveal organized aggregates even at low concentrations ($CAC \approx 0-0.2$ mg·mL⁻¹) and a structural crossover near $c^* \approx 0.8$ mg·mL⁻¹. Two R_g populations were observed, linked to individual chains and to aggregates; higher scattering anisotropy (IVH/IVV) appears at lower concentrations. Nanogels show $R_h \approx 400$ nm (PDI ≈ 0.3), zeta ≈ -30 mV; SLS yields fractal dimension ≈ 3.6 , $R_g \approx 89$ nm and shape factor $\approx 0.6 \pm 0.2$, consistent with microgels. FTIR and TGA confirm agarose stabilization in the matrix, while CD evidences helicity onset during cooling. SAXS shows double-helix features with correlation length ≈ 19 nm. XPCS data were acquired and are under analysis to clarify the kinetics and mechanisms of double-helix formation and nano/microstructural self-assembly. These insights will guide design of agarose materials for separation, encapsulation and as soft stabilizers (Mickering).

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- [2] 10.1007/bf00003999
- [3] 10.1016/j.carbpol.2018.01.060
- [4] 10.1016/j.carbpol.2014.04.085
- [5] 10.1021/acs.biomac.6b01401
- [6] 10.3390/gels10110691
- [7] 10.36868/IJCS.2024.03.03

MOGNO Beamline Synchrotron-Based X-Ray Microtomography Applied to Herpetological and Embryological Research

Gustavo Colaço Gonçalves¹, KATRINE DE PAIVA SOARES¹, TAYANE TANURE FRANÇA¹, SAMARA CRISTINA SANTOS DE OLIVEIRA², THAINA CHRISTYNE DE ALVARENGA RIBEIRO¹, MARCOS VINICIUS COLACO GONCALVES¹, Helio Ricardo da Silva³, REGINA CELY RODRIGUES BARROSO¹

¹Universidade do Estado do Rio de Janeiro, ²Universidade Federal do Rio de Janeiro, ³Universidade Federal Rural do Rio de Janeiro

e-mail: gustavcolaco@gmail.com

The introduction of micro-CT techniques in fields of vertebrate zoology, here in special herpetology (study of amphibians and reptiles), was a groundbreaking event, allowing new looks on old problems and even new opportunities of research. The different tissues and organs may source a wide amount of information of different natures, and in this sense and less explored are the ultrastructural and developmental analyses. During a MOGNO commissioning project, we performed a study using two different embryos: an embryo of the direct-developing frog *Eleutherodactylus cochranæ*, species without larval phase and another of the viviparous skink *Brasiliscincus agilis*, species that retains the eggs and developing embryos inside the female's body until development is complete. Although SR μ CT has been used in descriptive research involving herpetofauna members, developmental studies were carried out only recently. The mineralized structures like bones were more easily observable in both specimens. We identified different bones like the cranial, the appendicular and the axial ones and their state of development. MOGNO beamline also allowed the discrimination of soft tissues. For both samples, we observed in detail the eye and its subunits, such as lens, cornea, retina and their layers and the whole central nervous system, including the brain and some nerves. Specific tissues could be better visualized in only one of the specimens, in *B. agilis* we identified the heart, whereas in *E. cochranæ* observed structures of digestive system. The number of studies applying SR μ CT to study embryos and larvae, their development and organogenesis are still scarce. The production of protocols and methodology to measure this type of specimen may open the doors to the use of other model animals among members of herpetofauna in different types of studies and help to reveal more about the biology of these organisms, with applications in morphology, physiology, ecology and evolutionary studies.

The development of this research is funded by Conselho Nacional de Desenvolvimento Científico e Tecnológico and by Fundação de Amparo à Pesquisa do Estado do Rio de Janeiro. This research used facilities of the Brazilian Synchrotron Light Laboratory, part of the Brazilian Center for Research in Energy and Materials. The MOGNO beamline staff is acknowledged for assistance during the experiments.

Tunable of optical properties in quasi-1D V₂O₅ nanostructures by alkali ion intercalation

Marcus Vinicius de Paiva¹, Waldir Avansi Junior², Flavio Henrique Feres¹, Yuri Bernardes Marçal¹, Francisco Carlos Barbosa Maia¹

¹Brazilian Center for Research in Energy and Materials, ²Univerdade Federal de São Carlos (departamento)

e-mail: marcus.paiva@lnls.br

One-dimensional (1D) V₂O₅ nanowires and nanoribbons are gaining increasing interest due to their unique nanophotonic properties in the mid-infrared (mid-IR). These materials can act as subdiffraction waveguides, supporting phonon-polaritons such as those observed in the SnO₂ [1] and α -MoO₃ [2] nanostructure. Furthermore, it was recently predicted that the polaritonic properties of V₂O₅ can be tuned by the intercalation of alkali ions (lithium, sodium, and calcium) into the crystal structure. However, studies on how ion intercalation can modify the nanophotonic properties of 1D-V₂O₅ are lacking. We investigated the polaritonic response of V₂O₅ nanowires and nanoribbons with and without Li and Na intercalation using synchrotron infrared nanospectroscopy (SINS). The materials were synthesized by the hydrothermal method, where the reactions occur in a temperature-controlled reactor. We obtained V₂O₅ nanorods in the orthorhombic phase from an experiment at 200 °C for 48 h, while nanoribbons in the monoclinic phase resulted from an experiment at 120 °C for 24 h. X-ray diffraction and electron microscopy confirmed the formation of rod and ribbon structures. By SINS, we observed that the Li and Na intercalations significantly altered the phononic properties of the nanorods compared to the original V₂O₅ nanostructures. In Li:V₂O₅, the band observed at 1005 cm⁻¹, which is attributed to the V–O bond, is observed at 1005 cm⁻¹, while in non-intercalated materials, it appears at 1020 cm⁻¹. For Na:V₂O₅, we observed that intercalation led to a band shift from the non-intercalated band at 1020 cm⁻¹ to band lengths near 1005 cm⁻¹, also toward shorter band lengths. Based on these results, we discuss the intercalation-induced effects on the polariton bands and evaluate the corresponding polariton properties. These results indicate that intercalated V₂O₅ nanowires and nanoribbons specifically constitute a new class of one-dimensional nanophotonic platforms potential.

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[1] Nature Communications 10, 1-7 (2019)

[2] Nano Letters 18, 3056-3062 (2018)

USAXS and XPCS studies of gold nanoparticles confined into polyelectrolyte complex coacervate

Lucas Aguiar Portela^{1,2}, Watson Loh¹, Aline Ribeiro Passos³

¹Universidade Estadual de Campinas, ²Brazilian Center for Research in Energy and Materials, ³Brazilian Center for Research in Energy and Materials (LNLS)

e-mail: lucas.portela@lnls.br

Complex Coacervates are formed by spontaneous associative liquid-liquid phase separation (LLPS) between oppositely charged polyelectrolytes in aqueous solution, leading to a coacervate-rich phase and a dilute phase [1]. The dense phase provides a confined microenvironment that can affect the diffusion of reactants and the stability of catalysts. Recently, many authors used coacervates to encapsulate enzyme and use them as microreactors [2], [3]. Gold nanoparticles (AuNPs) are well-known for their catalytic properties in reactions such as alcohol oxidation, borohydride-assisted reduction and carbon-carbon coupling [4]. However, their performance can be significantly influenced by the environment in which they are situated. In this work, AuNPs were encapsulated within polyelectrolyte coacervates formed by sodium polyacrylate (PANa) and poly(diallildimethylamonium) chloride (PDADMAC), their catalytic activity was studied using the reduction of 4-nitrophenol as a model reaction. The results were compared to those obtained using free AuNPs in solution. The coacervate were characterized by X-ray Photon Correlation Spectroscopy (XPCS) and Small Angle X-ray Scattering (SAXS) revealing droplets of hundreds of nanometers with high stability, with no coalescence over the probed window. Furthermore, AuNPs can be used as rheological probes to study the viscosity inside the coacervate phase using XPCS and SAXS. The use of coherent X-ray techniques allows us to detect the nanoparticles inside the coacervate phase, giving information about their structure and dynamics in this confined microenvironment. It was observed that the confined environment within the coacervates altered the catalytic reaction kinetics, likely due to changes in local concentration

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X-ray study of surface structural and electronic fluctuations in the two-dimensional magnetic semiconductor Ni₃TeO₆

Luisa Vargas Costa Freitas¹, Eduarda P. M. Campos¹, Rayan Alves da Cruz¹, Angelo Malachias de Souza¹

¹Universidade Federal de Minas Gerais

e-mail: luisavargasfreitas@gmail.com

The compound Ni₃TeO₆ is a semiconductor that has been attracting growing interest due to its promising magnetic and optical properties for various technological applications. This compound has a hexagonal crystal structure, composed of NiO₆ and TeO₆ octahedra. In this work, we investigate the structural and electronic properties of the Ni₃TeO₆ surface. Crystal truncation rods (CTR) analysis, with measurements carried out at the EMA beamline of Sirius, revealed surface desorption of tellurium and the formation of Ni₂ clusters, in addition to the presence of TeO₂. The fitting of the measurements suggests the coexistence of phases and a possible partial reduction of tellurium (Te⁶⁺ → Te⁴⁺), associated with the presence of oxygen vacancies and local reconstructions. We observed the coexistence of three crystallographic sites with distinct octahedral distortions - Ni(I), Ni(II), and Ni(III) - with Ni(III) sites being predominant. A more detailed understanding of the surface defects is carried out using scanning tunneling microscopy and spectroscopy (STM/STS) measurements, which highlighted the electronic changes due to the existence of vacancies in the system. STM measurements indicate a highly ordered surface, but with local structural modifications reflecting the different coordination of the nickel sites. STS measurements revealed variations in the density of electronic states near the surface, directly related to magnetic anisotropy and to the different Ni-O bonds in the various sites, suggesting an impact of these structural features on the local electronic properties. Finally, implications for the surface potential are discussed through the analysis of Kelvin Probe Force Microscopy (KPFM) measurements. The results show that structural defects, such as oxygen vacancies and local disorder due to Te saturation, induce perturbations in the crystal lattice, potentially affecting the electronic and magnetic behavior of the system.

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