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LNLS Users Group: Young Research Award

PLENARY LECTURES

Assessment of silica nanoparticle hydrodynamic diameter in complex media by means of X-Ray Photon Correlation Spectroscopy (XPCS)

Agustin Picco

The constant growth of nanomaterial applications in various fields, including medicine, agriculture, and energy, has created a need for the characterization of nanoparticle properties in challenging environments. In this regard, scattering techniques, both static (e.g. SLS, SAXS) and dynamic ones (e.g. DLS, FCS), are commonly employed to determine size, shape, colloidal stability and interactions of nanoparticles in diverse media. In recent years, synchrotron-based X-ray Photon Correlation Spectroscopy (XPCS) has gained attention as a valuable technique for studying nanoparticle diffusion and deriving their hydrodynamic diameter in complex enviroments, where light-based methods often fail to provide reliable data owing to high scattering and absorption from the media.

This study presents an initial evaluation of the capabilities of the Caterete beamline at LNLS-Sirius to study nanoparticle diffusion in biologically relevant media by means of XPCS. To this end, silica nanoparticles (SNPs) with sizes ranging from 200 to 700 nm were examined in various media including phosphate buffer saline (PBS), albumin solutions and PBS supplemented with fetal bovine serum (FBS). Additionally, the influence of an antifouling and colloidal stabilizing coating (PEG, Mn=2000) on selected nanoparticle sizes was investigated. Different models were tested to analyze the XPCS derived autocorrelation functions and obtain information on SNP diffusion and hydrodynamic diameters, including single exponential, KKW model, and cumulant analysis. Furthermore, all samples were measured by SAXS and DLS concomitantly and a comprehensive comparison was made focusing on the influence of protein adsorption and colloidal stability/instability on the obtained results from each of the three techniques (XPCS, SAXS and DLS).

Challenge and Opportunities of the Sirius Bean-lines: From Crystal Structures to Quantum Crystallography

Javier Ellena

The present and the future of the Bean-lines at SIRIUS open great opportunities for the solid-state study of a wide range of different kind of materials, from Minerals to Natural products, from Metal Organic Frameworks to Active Pharmaceutics Ingredients. New experiments can be design to understand the mater more deeply than ever before. However it also represents a big challenge to develop new and innovative ways to analyses the materials designing observational protocols at a completely new level. In this presentation we will describe the different experiment that can be designed at the current stage of the Sirius's Bean-lines as well as problems that will be able to be faced in the near future. In this presentation we will also discuss the recently obtained results involving a large number of problematic cases ranging from micro crystals to massive structures, process observation, etc. It will follow a discussion of new implementation that can easily been perform at the Sirius beam-lines together with the future implementation that could open new doors to different new kind of investigations such as high-resolution single crystal x ray diffraction to assess the electronic and magnetic structure of the molecules.

Formation of skyrmions in metallic interfaces: a theoretical-experimental collaboration

Helena Maria Petrilli

We will show some examples of a very fruitful collaboration with the experimental researchers Julio K. Cesar and Jeovani Brandão performed by the theoretical groups of Angela B. Klautau(Belém do Pará) and Helena M. Petrilli(IF-USP, São Paulo). The origins of skyrmions in seemingly symmetric stackings are explored using the Pd/Co/Pd system as an example.

The effects of interfacial atomic intermixing in the Dzyaloshiinski-Moryia interaction behavior is discussed . Also, magnetic properties of the synthetic ferrimagnet multilayers are currently being investigated.

Innovative Smart Diagnostics for Early Detection of Pre-Cachexia in Patients with Advanced Cancer

Yara Paiva Maia

The study aims to develop an automated diagnostic tool for pre-cachexia cancer patients using a machine learning algorithm. The algorithm will be trained with infrared spectrum wavenumbers obtained by Attenuated Total Reflection (ATR) – Fourier Transform Infrared (FTIR) spectroscopy in two groups of cancer patients in palliative care, classified as without cachexia and pre-cachexia. The study included 44 advanced cancer patients, of which 29 had no cachexia, and 15 had precachexia. The spectra were measured in the 4000 to 650 cm-1 wavenumber regions using a FTIR spectrometer Agilent Cary 600 Series coupled with MCT detector. The spectrum of air was used as a background before each sample analysis. Sample spectra were taken in triplicate, at a spectral resolution of 4 cm-1, and to each measurement 64 scans were performed. The infrared spectra were analyzed after undergoing pre-processing, which involved positive Rubberband baseline normalization, normalization by minimum and maximum, and second derivative by the Savitzky-Golay filter. The main spectral regions detected in the serum were 945-918 cm-1 and 2875-2869 cm-1. These wavenumbers represent the vibrational modes of PO3 2- symmetric stretching and CH3 symmetric stretching, respectively, which have been previously linked to DNA/RNA ribose and lipids. The feasibility of developing an algorithm for the automated diagnosis of pre-cachexia was examined based on the identified wavenumbers. The Naive Bayes classifier was employed to differentiate between the two study groups, exhibiting an accuracy of 0.77, sensitivity of 0.87, and specificity of 0.72. The changes in biochemical constituents that occur in serum as a result of pre-cachexia are related to the infrared spectrum obtained by ATR-FTIR, featuring a molecular fingerprint. The implementation of this approach in clinical practice may automate the diagnosis of pre-cachexia in advanced cancer patients, monitoring whether changes in therapy or interventions are necessary throughout cancer treatment. To the best of our knowledge, this is the first study to propose a method for the diagnosis of pre-cachexia cancer using a machine learning approach trained with infrared spectrum wavenumbers obtained by ATR-FTIR with blood serum, providing a new avenue for future research in this field.

Modeling the surface structural of two-dimensional materials by Crystal Truncation Rods analysis

Angelo Malachias

In this work, processes for modeling the surface electronic density of two-dimensional materials with lamellar stacking using Crystal Truncation Rods are discussed. This method allow for checking intercalation and diffucion profiles in systems such as topological insulators, synthesized graphene bilayers and exfoliated minerals. Examples of these three cases will be discussed with simplified models.

Multiple surface states and Electronic Confinement in Modulated Topological Materials

Rogerio Paniago

Topological materials are well-known for their surface states with different electronic properties . The electronic topology of Bi4Te3, composed of alternating Bi2 and Bi2Te3 layers, was investigated by density functional theory and angle-resolved photoemission spectroscopy (ARPES). We find, remarkably, that there are three adjacent strong topological gaps with associated protected surface states within a 2-eV range of the Fermi level. The existence of three consecutive Dirac cones in k space gives promise for alternative phenomena and applications.

Another topological material, Sb4Te3 consists of a stacking of two distinct topological materials: the 3D topological insulator Sb2Te3 (111) and the 2D topological insulator Sb (111). By comparing the ARPES results with the bulk bands and quantum wells of Sb as reported in the literature, we observed a confined electronic state between the Sb4Te3 bulk and the Sb termination. The photon-energy independence of this state indicates confinement along the stacking direction. Furthermore, the topological behavior exhibited by Sb2Te3 and Sb persists, characterized by states with hexagonal warping associated with time reversal symmetry and photon-energy independence in these surface states.

Polarised Soft X-ray Imaging of Quantum Materials

Larissa Sayuri Ishibe Veiga

In order to explore the novel functionalities of quantum materials, a comprehensive understanding of their microstructure is essential. Macroscopic probes provide an average assessment of the material's properties and offer indirect insights into the pivotal roles played by magnetic and microstructural inhomogeneities. The array of polarized soft X-ray spectroscopic techniques available at the I06 beamline of Diamond Light Source (UK) can be seamlessly integrated with Photoemission Electron Microscopy (PEEM) or coherent diffraction imaging, enabling high-resolution imaging of fundamental phenomena governing the properties of functional and quantum materials.

In this talk, I will provide an overview of the key scientific areas addressed by the beamline, with a particular emphasis on the in-situ manipulation of quantum materials. This manipulation encompasses the control of domains and domain walls in antiferromagnets, magnetic domain imaging of novel topological spin textures, and the investigation of light-induced phenomena in strongly correlated materials.

Probing charge density waves using dark field X-ray microscopy

Eduardo Bittar

Charge density waves (CDW) are a collective phenomenon where, below a specific transition temperature, there is a modulation of conduction electrons accompanied by a distortion in the crystal lattice. Experimental signatures of CDWs can be observed even in macroscopic measurements such as thermal and electrical transport and X-ray diffraction, where forbidden reflections appear in the diffraction pattern below the transition temperature. We employ the dark-field X-ray microscopy technique to explore and characterize the mesoscopic and macroscopic properties of CDW in the intermetallic compound Sr0.4Ca0.6Rh4Sn13.

Resonant X-ray Scattering Studies of Charge Density Wave Correlations in the Cuprates

Eduardo Silva Neto

Resonant X-ray Scattering (RXS) is a photon scattering technique that can be used for the study of electron states in quantum materials. In the last 15 years we have witnessed the development of ever more advanced RXS synchrotron beamlines working in the soP x-ray regime (energies below 2 keV) and the many important scientific findings enabled by this technique. The study of charge density waves (CDWs) in cuprate high-temperature superconductors over the last 12 years has evolved in tandem with the technologica improvements of RXS instruments, from the early stages of detecting CDW correlations with energy-integrated RXS (EI-RXS) to the study of dynamic CDW correlations with the newest resonant inelastic x-ray scattering (RIXS) instruments that can resolve the energy and polarization of the scattered photons. I will present an overview of various RXS studies of the CDW in the cuprates and the resulting insights about the superconductivity, pseudogap state and strange metal behavior in those systems, as well as the form of the effective interaction between electrons.

Search for new electride high temperature superconductors

Edison Zacarias da Silva

In this century a new class of materials have been shown to present superconductivity. Under pressure, many new electride superconductors have been discovered. The present work discusses two new materials proposed as new high temperature superconductors, one under pressure, Li5C, the other, Mo2N at ambient pressure. Using density functional theory (DFT) and particle swarm search method (PSO), we discovered the electride superconductor, Li5C, stable for pressures from 50 to 210 GPa. Li5C has a significant electron-phonon coupling (EPC) with superconducting critical temperature Tc = 48.3 K at pressure 210 GPa. We also discuss superconductivity in the 2-D electrene Mo2N. Using DFT and Eliasberg approach, we show how biaxial strain affects superconductivity in Mo2N. Results indicate that Mo2N presents strong (EPC) with large anisotropy in the superconducting energy gap. This material shows superconductivity and calculations point to Tc = 24.7 K.

Search for the Kitaev quantum spin liquid state in honeycomb iridates at high pressures

Gilberto Fernandes Lopes Fabrris

Compounds with 5d transition metal ions have attracted recent attention due to the prediction and observation of novel forms of topological magnetic and electronic states. Among these, particular attention has been given to the Kitaev quantum spin liquid state that is expected to occur in honeycomb iridates, and which is a potential candidate for topologically protected quantum computing. However, the presence of a spin liquid in these materials rely on the delicate balance between Kitaev and Heisenberg exchange interactions; such balance is very sensitive to structural deviations away from a perfect honeycomb motif. In this talk I will present our efforts in the study of honeycomb iridates using several x-ray techniques at high pressure to control and probe their structure, as well as electronic and magnetic ground states. Particular focus will be given to our recent results on Cu2IrO3 and Na2IrO3, highlighting their distinct and complex phase diagram.

Spin-State Ordering and Intermediate States in a Mixed-Valence Cobalt Oxyborate with Spin Crossover

Eduardo Granado

Spin-state ordering – a periodic pattern of ions with different spin-state configurations along a crystal lattice – is a rare phenomenon, and its possible interrelation with other electronic degrees of freedom remains little explored. Here we perform a structural investigation of the mixed-valence Co homometallic ludwigite Co22+Co3+O2BO3. A superstructure consistent with a long-range Co3+ spin-state ordering is observed between T4=580 K and T3=510 K (see Fig. 1). Intermediate states with mesoscopic correlations are detected below T3 down to T1=480 K with a change of dimensionality at T2=495 K. The spin-state correlations are connected to the charge sector as revealed by the abrupt changes in the electrical resistance at T1 and T2. The evolution of the structural parameters below T1 indicate that the spin crossover is ignited by a moderate degree of thermally-induced Co2+/Co3+ charge disorder. Charge and spin-state degrees of freedom can be interrelated in mixed-valence spin-crossover materials, leading to sharp transitions involving intermediate spin-state/charge correlated states at the mesoscale.

The polymorphous nature of halide perovskites

Gustavo Dalpian

Many halide perovskites exhibit a cubic crystal structure (Pm-3m) at elevated temperatures but transition to lower symmetry structures such as orthorhombic or tetragonal at lower temperatures. Recent theoretical findings have cast doubts upon the cubic structure, as it may exhibit negative phonon modes, unusual band gap trends, and incomplete alignment with PDF measurements. In this context, we propose the concept of polymorphous structures, suggesting that the cubic structure emerges as a result of temporal and spatial averaging of lower symmetry structures. To substantiate this idea, we will employ ab initio and molecular dynamics simulations.

The specific, non-specific interaction between dendrimers and HIV gp120 protein

Nathan Cowieson

Dendrimers are branched polymers that are of interest as anti-microbials or anti-cancer drugs, vectors for drug delivery and other applications in the health sciences. In this study we investigate poly-lysine dendrimers as topical inhibitors of HIV infection. We have used SAXS and other biochemical analyses to study the structure of the dendrimers and their interaction with the HIV gp120 protein that is essential for viral infection.

Previously published molecular modelling results suggested that these dendrimers may collapse making packing interactions within the particle to form a structure with a defined shape and surface that may be capable of making specific interactions in a protein-like manner1. Our SAXS results show that charge repulsion between the head groups cause the dendrimers to adopt a very much larger structure that shrinks and grows in response to changes in salt concentration suggesting a less well-defined structure.

Conversely, measuring particle size by SAXS while titrating the dendrimers onto gp120 show an interaction with a defined stoichiometry that is quite distinct from related unbranched polymers. These results suggest a more specific mode of interaction.

Finally, it does not seem right to model these interactions in a discrete, atomistic way that would be typical for specific interactions or simply in terms of bulk properties as would be typical for non-specific interactions. I will present my preliminary approach at modelling this interesting system.

1) Roberts, Benjamin & Scanlon, Martin & Krippner, Guy & Chalmers, David. (2009). Molecular Dynamics of Poly(L-lysine) Dendrimers with Naphthalene Disulfonate Caps. Macromolecules. 42. 2775-2783. 10.1021/Ma802154e.

Thermal transport and structural transitions in Barium Bismuthate

Valentina Marteli

Perovskite-type complex oxides are a family of compounds that have attracted growing interest because of the variety of tunable physical properties making them attractive for technological applications in different areas [1]. At LQMEC, we are interested in investigating how structural properties and transitions impact the evolution of heat transport in some selected perovskites from bulk to thin films.

In this talk, I will present and discuss our recent investigations of thermal transport in a representative of this class of oxides: the Barium Bismuthate BaBiO3 (BBO) [2]. BBO exhibits an insulating ground state with a still debated origin, and a superconducting state upon hole-doping, besides being predicted to host a topological insulating (TI) state upon electron doping [3]. A complex relation between electronic and lattice degrees of freedom has been called into question in the attempt to explain the electronic states of this compound [4]. In our thermal conductivity experiments, we found and unexpected $\sim T^2$ power law at low temperatures, reminiscent of a glass-like behavior, and possible interpretations will be discussed in terms of the temperature -dependent structural phase diagram.

Finally, I will discuss our perspective as users on experiments we expect to carry out at the Synchrotron light source Sirius, in BBO and in other compounds, to bring further evidence to the interpretation of our (electrical, thermal, thermoelectric) transport experiments.

Unveiling emergent phenomena in Bi2Se3 and V5S8 single crystals through structure-property relationships

Marcos Avila

This talk will highlight three recent works currently submitted for publication, conducted by our research group and collaborators on the title binary compounds. With a large band gap and a single Dirac cone responsible for the topological surface states, Bi2Se3 is widely regarded as a prototypical 3D topological insulator. We have used the self-flux method to obtain large, high-quality Bi2Se3 single crystals in the entire concentration range available for such on the binary phase diagram. By combining basic structural characterization with resistivity, Hall effect and Shubnikov-de Haas (SdH) quantum oscillations, different types of lattice defects are identified and their detailed role on the bulk transport are investigated. Previous open questions on Bi2Se3, such as the different scattering times in transport and quantum oscillations, and the presence of additional low mobility bands, are addressed. For V5S8, we have grown high-quality single crystals by chemical vapour transport (CVT) and report the observation of an unexpected phase transition at high magnetic fields between the spin-flop and spin-flip transitions in this d-electron antiferromagnetic quantum material. High-precision magnetic, thermal and electrical transport measurements enable us to track the transitions up to fields as high as 35 T and at temperatures down to the millikelvin range, revealing three distinct magnetic quantum phase transitions. We present a model that finds agreement with our observation of a triad of spin transitions involving two sublattices with frustrated inter- and intra-sublattice spin couplings. Additionally, our heat capacity studies on V5S8 have revealed an unexpected and highly unusual (for a bulk material) quadratic dependence of the specific heat at low temperatures. The regime is independent of applied magnetic field and therefore not magnetic in origin, but rather related to the system's particular layered structure, as part of the broader chalcogenide family VxS8 (x=4,5,6,8). We present a model wherein the anomalous heat capacity is described with an unconventional acoustic phonon spectrum, which is linear in wavevector in the c direction, but quadratic in the a-b plane, indicating a form of geometrical elastic criticality.

Use of nanotechnology in studies of urban aquifer contamination in the São Paulo State

Claudia Varnier

Nitrate contamination is almost ubiquitous in urban aquifers, becoming a challenge for the São Paulo State, where more than 80% of its services are supplied totally or partially by groundwater. Despite the several existing studies, there are critical scientific challenges that overcoming will contribute to the environmental management of extensive contaminated urban areas. Recently, advanced research groups have suggested the joint use of new tools, which would integrate with traditional hydrogeochemical techniques, including nanotechnology (synchrotron-based high-resolution and time-resolved X-ray imaging). This work proposes to apply, in a pioneering way, this tool in a very detailed study in a contaminated area in the city of Bauru (SP). For this purpose, the following activities are planned:

i) registration and treatment of previous data;

ii) determination of BAS flow conditions;

iii) reassessment of previous and recent nitrate concentrations;

v) installation of multi-level monitoring wells;

iv) sampling for physicochemical, chemical, isotopic, gas, microbiological, artificial sweetener analyzes of surface water, groundwater, rainwater, and wastewater samples;

v) collection of sediment/rock samples for porous media characterization and chemical, microbiological, isotopic and nanotechnology analyses. Undisturbed samples

collected from multilevel wells drilling will be sent to the Brazilian Synchrotron Light

Laboratory (LNLS) for the porous medium characterization on a micro scale.

The techniques adopted will be X-ray micro and nanotomography, using the synchrotron light source. In this beamline, high-resolution three-dimensional images are obtained from soil/rock samples with a maximum diameter of 1.5”. This technique allows for the study of the same sample at different resolutions without additional preparation, and 3D models of the porous medium can be made, which will provide subsidies for pore-scale modeling studies. Thus, it is expected that understanding nitrogen dynamics in the subsurface and the factors that control it will help public policies for dealing with urban aquifer contamination.

Dynamics in soft matter probed by X-ray photon correlation spectroscopy
Aline Ribeiro Passos

Formation, stability and crystallization in metal halide perovskites analyzed by in situ experiments
Ana Flavia Nogueira

Highlights of the Hierarchical and Heterogeneous Matter Division
Hélio Cesar Nogueira Tolentino

How tomography helps to solve geological puzzles
Carolina Camarda

In operando studies for X-ray mapping in cathodic materials in Li-ion cells. Effects of chemical heterogeneities
Felix Requejo

In situ Monitoring of Exsolving Nanoparticles from Perovskite Oxides using Synchrotron-based XRD and XAS
Swathi Raju

Multispectral studies of metal halide perovskites: simultaneous x-ray ptychography, x-ray fluorescence, and x-ray excited optical luminescence experiments
Francisco M. C. da Silva

Polarized X-ray absorption spectroscopy applied to novel materials
Cinthia Piamonteze

Science opportunities with scanning microscopy at new generation X-ray sources
Gerardina Carbone

The domain wall between a Mott insulator and a metal is an anomalous metal
Eduardo Miranda

Three-Dimensional Imaging of Hierarchically Mesoporous Catalysts with Coherent X-ray Diffractive Imaging
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Tracing paleoenvironmental and preservational aspects of 3.4 billion years old fossils from Pilbara region, Western Australia by using synchrotron-based X-rays techniques
Flávia Callefo

Unveiling the Versatility of X-ray Crystallography: Applications in Structural Biology, Biochemistry, and Medicinal Chemistry
Maria Cristina Nonato

Women's presence in synchrotron research at Sirius
Ingrid Barcelos

Women's presence in synchrotron research at Sirius
Regina Cely Barroso

ORAL PRESENTATIONS

4D Study of Groundwater Remediation Techniques at Pore-scale

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Groundwater remediation is a pressing issue in the modern world. In Brazil, almost 37% of the cities are supplied exclusively with groundwater. Study by Lunardi et al., (2021) highlighted high susceptibility to groundwater pollution of regions, where pollutant source, such as industry, is present. The studies performed on methods like ones studied in this work usually do not study what is happening on pore-scale. Such study is performed by Pandey, Sharma and Saha (2022) on nZVI nanoparticle production techniques, or by Chen et al., (2021) on slow-release potassium permanganate. This highlights a knowledge gap in the modern understanding of these remediation techniques. In this work, a dataset on nZVI nanoparticle reaction with TCE (trichloroethane) is studied. TCE is a DNAPL – Dense non-aqueous phase liquid. These compounds are challenging to be removed from groundwater reservoirs via conventional means, as they are almost immiscible in water, and are difficult to remove from the porous medium. Therefore, nanoparticles used for remediation of such reservoirs must be able to reach the contaminant. The dataset was obtained via X-ray microtomographic scanning (X-ray micro-CT) and allows for 4D (3D time) study of the processes, happening on the pore-scale. The dataset was captured at Diamond Light Source by Dr. Tannaz Pak. This study is performed via Fiji (ImageJ), Avizo and Dragonfly. In addition to this, column experiments on different porous materials are performed to assess nZVI particle distribution through the column. Particle distribution throughout several injections was assessed via magnetic susceptibility sensor. The experimental data of the breakthrough curves is then analysed with MnMs, to confirm the results of the experiment and get a better insight into nanoparticle mobility. Latest experiment performed on this column involved injecting same nanoparticle suspension, which was used in the in-situ experiment, studied at Diamond Light Source.

Biosamples analysis using synchrotron radiation multi-techniques at Sirius: new trends and challenges

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Modern synchrotron radiation sources have significantly advanced biological analysis. Mainly due to the tissues exhibit complicated structures from the organ level to the subcellular level, new scientific issues can now be tackled with synchrotron X-ray microbeams and nanobeams at Siurius. The main objective of this study is to characterize different biosamples using synchrotron multi-technique approaches including high-resolution X-ray microtomography, X-ray nanospectroscopy and coherent diffraction imaging. In this context, the present work aims to present three case-studies: i) The use of CDI at the Cateretê beamline in different Rhodnius prolixus tissues to provide detailed morphological information of the effects of Azadirachtin on the insects system and therefore new insights on Chagas Disease control; ii) Compare the coronary arteries of health rats with the irradiated group composed by rats submitted to thoracic radiotherapy and the group of rats treated with losartan X-ray fluorescence with nanometer-scale spatial resolution at CARNAUBA beamline, in order to identify morphological and elemental changes in the tissues and iii) Microtomographic visualization followed by 3D analysis at submicrometer resolution at MOGNO beamline allow acquiring images with high quality, low scatter and dose reduction, rand facilitates the use of monochromatic X-rays. Visualization of 3D structures at submicrometer resolution of soft tissue samples and hard tissue samples such as rare fossil and extant taxa may allow virtual histology as well as virtual section planes of various orientations and thicknesses.

References: From micro to nano and time resolved X ray computed tomography: Bio-based applications, synchrotron capabilities, and data driven processing. 10.1063/5.0129324

Phase-contrast X-ray microtomography links Cretaceous seeds with Gnetales and Bennettitales. 10.1038/nature06278

Combined μ-FTIR and sXRF towards space-resolved localisation of mineral nutrients and protein in soybean seeds: a one-sample strategy at Imbuia and Carnaúba beamlines

Gabriel Sgarbiero Montanha[1], Lucas Coan Perez[1], Nicolas Gustavo da Cruz da Silva[1], João Paulo Rodrigues Marques[1], HUDSON WALLACE Pereira de Carvalho[2]

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Soybean (Glycine max) seeds exhibit an outstanding concentration of proteins (ca. 35 wt.%) and are currently responsible for granting ca. 70% of the world's protein meal[1]. Most of the soybean proteome encompasses storage proteins, which are strategically located in specialised vesicles of cotyledonary tissue cells to be used as a nitrogen source during early seedling development[2]. Despite having a balanced amino acid composition, soybean storage proteins are remarkably deficient in sulphur amino acids such as methionine, whose biosynthesis is reported to be Zn-dependent[3]. Although a positive association between Zn and proteins has been observed across soybean seed varieties cultivated in Brazil[4], their distribution within the seed cotyledonary cells, i.e., those where the storage proteins are located, has never been properly assessed. In this regard, the cotyledonary tissues from four genetically differing genotypes exhibiting contrasting total protein and Zn concentrations collected from a field experiment in the 2022/2023 season were cross-sectioned using a glass-knife ultramicrotome, and the resulting 5 μm-thick cross-sections were immediately transferred either to a Si/Au wafer and sequentially analysed through FTIR using a benchtop FTIR system at the Imbuia beamline and through XRF at Tarumã endstation of the Carnaúba beamline of the Brazilian Synchrotron Light Source. The results revealed the feasibility of coupling FTIR and XRF using the same seed sample and indicated a higher absorbance of the amide I band (1545 cm-1) in the protein storage vesicles of the soybean genotypes which exhibited higher XRF Zn intensities, thereby indicating a cellular-level association between storage protein and Zn concentration in soybean seed cotyledons. These results revealed that Zn is a limiting factor for protein biosynthesis in soybean seeds and might foster nutritional-based strategies for boosting both the storage protein content and quality in this species.

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Development of flexible composite scintillator films

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Scintillators materials convert X-ray photons into visible light, and should have high X-ray absorption, high density, large effective Z, radiation hardness, high conversion efficiency and low afterglow. Inorganic scintillators are used in many applications, from particle physics to medical imaging[1]. LiYF4 is a promising candidate due to high visible light transparency, large band gap and as good host for trivalent rare-earth ions (RE). LiYF4:RE emission is within the spectral sensitivity of CCD detectors [2]. CdSiO3:Mn is another scintillator material, whose light emission is due to the Mn2 dopant around 600nm[3], close to the maximum efficiency of SiPM detectors [4]. CdSiO3:Mn has the advantages of not depending on RE doping, reducing drastically the production costs. LiYF4: RE, on the other hand, has the advantages of accommodating different RE allowing the choice of the light emission according to the desired application. In the present work 2 types of composite films were produced using polystyrene (PS) as the organic structural matrix, the first one using LiYF4 co-doped with Ce3 and Tb3 (Ln = Ce, Tb) and, the second one, using CdSiO3:Mn as the inorganic load. The films were investigated in view of high efficiency X-ray detection. The luminescent properties of the films were done by PL with excitation at UV-VIS range. STXM, XRF and XEOL 2D mapping were performed at CARNAÚBA beamline to investigate the dispersion of the inorganic load in the PS matrix. The valence of the dopants was analysed via XANES done at the EMA beamline. The results showed that the PS concentration directly affects the particles distribution changing strongly the efficiency of emission process. In addition, the images reveal that in the LiYF-PS films the inorganic powder are mainly deposited in the bottom of the films leading to different RL and PL responses for each side. Whereas, for the CdSiO3-PS films, there is a homogeneous distribution of the inorganic scintillator in the film.

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Development of microfluidic devices compatible with synchrotron techniques for in-situ monitoring of mineral precipitation on rocks

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Microfluidic devices have emerged as an essential tool for reproducing large-scale phenomena in a controlled environment. Their main characteristics are the reduced size, low weight, and high throughput, as well as the capacity to operate small amounts of samples through micrometer and/or sub-micrometer channels. In this sense, devices for reproducing flow in simplified pore structures have been fabricated using engineered materials such as silicon, glass, and PDMS. However, these devices present inherent limitations once they do not fully reproduce complex micropore structures and do not replicate the natural chemical reactivity of the real rock surface. As a result, these micromodels do not fully evaluate the fundamental mechanisms of flow, transport, and reactions within the context of an actual reservoir. To overcome these limitations, two microfluidic devices were developed considering a real rock as the sample. In addition, these devices are compatible with synchrotron-based techniques which propitiates higher spatial and temporal resolutions. The first device is composed of a 5 mm thick rock matrix with a channel 1 mm wide, 0.5 mm deep, and 10 mm long in one of the surfaces. This configuration allows the injection of acid solution along the channel and in-situ monitoring of the reaction's evolution by performing X-ray fluorescence maps and X-ray absorption with time in the Carnauba beamline. The second is a three-dimensional printed device designed to hold a 2.5 mm diameter cylinder-shaped rock and set a fluid flow along it. By performing time-resolved tomography measurements at the Mogno beamline, this device allows to track the morphology evolution during the injection. Both setups presented promising results in the first test, permitting to observe the mineral dissolution as well as the pore size distribution changes. Next step is to unify these devices to obtain both chemical and structural information from the same sample.

Effect of high pressure on phase transition of glassy systems

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Glasses are defined as amorphous solids obtained from melting-quenching, resulting in isotropic solid materials. When glasses are heated to the glass transition (Tg) region, the atomic mobility increases, leading to the formation of crystalline nuclei embedded in the amorphous matrix. Depending on the thermal treatment, these nuclei can grow and give rise to a glass-ceramic materials above the crystallization temperature (Tc). With extremely high loads in the GPa region, static pressures can exert an actual catalyst effect on the short-range order of glass at room temperature. The lithium disilicate and soda-lime silica glass and glass ceramic have been widely studied at high pressure in high volume samples (ex-situ characterization), presenting formation of polyamorphism due to the induced densification, and the structure of glass-ceramic formed is dependent of pressure and temperature applied. In this work we discuss the in-situ analyses of Raman spectroscopy and x-ray diffraction (XRD) of both amorphous and crystalline lithium disilicate, lithium germane silicate and soda-lime silica at pressures up to 75 GPa performed at EMA beamline. Results of XRD and Raman measurements carried out at high pressure and room temperature and also at high pressure and high temperature using Laser Heating will be presented. Crystalline-crystalline, crystalline-amorphous and amorphous-amorphous transitions will be discussed. Reversible and irreversible transitions will be presented. Furthermore, it is worth noting that suitable methodology was developed for in situ XRD measurements of high-quality glasses, suitable for calculations of the structure factor and consequently analysis of the short-range order through the total pair distribution function, G(r). This study focused on investigating the short and medium-range structure of glasses and explore the effects of high pressure on the elastic-plastic deformation domains.

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Evolution of the supramolecular arrangement during extrusion 3D printing of micellar inks.

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Pluronic is a class of poly(ethylene oxide)-poly(propylene oxide)-poly(ethylene oxide) triblock copolymers which, in concentrated aqueous solutions, can form thermoreversible hydrogels based on the micellar packing into cubic phases [1]. The incorporation of methacrylate groups at the Pluronic chain ends is a strategy to confer photocrosslinking capability, leading inter and intramicellar covalent bonds. Pluronic with methacrylate terminal groups are being widely used, alone or with other materials, for the 3D printing of biomaterials [2], under the assumption that the micellar packing in preserved after printing and photocrosslinking. The retainment of the supramolecular arrangement in these gels is desirable, because it is associated with biomaterials' elasticity and drug delivery control. However, shearing during extrusion printing and subsequent photocrosslinking can disrupt the ordering and/or structure of the micelles, compromising these properties. We performed small-angle X-ray scattering (SAXS) measurements to monitor the time evolution of the nanostructure of printed filaments fabricated at different rates, needle diameters, and ink compositions and we evaluated the photocrosslinking effect through light exposure after printing. The experiments we performed at the CATERETÊ synchrotron beamline (Sirius, Brazil) show that shearing during 3D printing disturbs the gel nanostructure, but it can be recovered over time, while the significant reduction of the long-range ordering and partial micellar disruption after photocrosslinking is irreversible. In future perspectives, we aim to elucidate the mechanical impact of these nanostructural changes performing SAXS measurements during in-situ tensile tests of these 3D printed photocrosslinked filaments. X-ray photon correlation spectroscopy (XPCS) will also be performed to correlate the nanostructural changes with changes in polymer mobility and dynamics that could have an impact on their biomechanical performance.

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High-pressure and High-temperature Fluid Flow system in the MOGNO Beamline for Time-Resolved Tomography

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The fourth-generation synchrotron facility provides X-rays from low to high energy with a high flux of photons that, coupled with advanced detector technology, allow routine acquisition of high-resolution tomograms in a few seconds. In addition to high-throughput experiments, in this case, computed tomography can also be resolved in time, which is the 4D CT scan. Among the wide range of interesting physical phenomena to be solved in time by three-dimensional images, the fluid flow in porous materials is one that is present in several areas, such as oil industry, agriculture, and environmental science. In particular, the flow of fluids in very deep reservoirs, where the porous material is under very high pressure (HP) and, normally, also at high temperatures (HT), is an important scientific case. With that in mind, the MOGNO group, in partnership with the energy company Equinor and Petrobras, is installing an HPHT Fluid Flow system at the beamline. This work aims to show the scientific community the system to be installed, promote discussions about the device and future experiments, such as time-resolved fluid flow in porous media, and take this perspective to other areas of knowledge.

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High resolution X-ray diffraction experiments and Rietveld refinement of exsolved perovskites.

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Paineira beamline is dedicated to perform X-ray diffraction (XRD) experiments of polycrystalline materials. Currently Paineira is under scientific commissioning, and the present work exhibits the fisrt measurements and characterizations developed at the beamline. Two samples of lanthanum chromite perovskite doped with ruthenium LaCr0.8Ru0.2O3 were synthesized as ceramic powders using the Pechini method. One of the samples underwent a thermochemical treatment, in order to exsolve nanoparticles of ruthenium on its surface [1]. They were measured at Paineira [2] and later subjected to Rietveld Refinement. The measurements were collected with the high-resolution detector MAC FMB-Oxford, energy of 19.505KeV, angular range of $5° < 2\theta < 35°$, scan step of 0.008° and counting time of 0.3 seconds at each step (flyscan mode). The characterization was successful and the crystallite size, microstrain, atomic positions and Ru-fractions were determined via Rietvield refinement for both samples. The correct percentage of ruthenium in the perovskite structure was found to be 15%. For the exsolved sample, only 8% of the ruthenium remained in the perovskite structure, indicating that 7% of it resulted in exsolved nanoparticles. No other phases were detected, suggesting that the exsolved ruthenium and ruthenium oxide nanoparticles are amorphous or have a small size that cannot be detected via XRD. Transmission electron microscopy (TEM) images of both samples were collected at LNNano facility. The images confirmed both samples were highly crystalline. The sample that didn't experience the thermochemical treatment has a porous nanostructure with average size higher than the sample that underwent the thermochemical treatment. The thermochemical treatment acted to favor the ruthenium exsolutions and formed nanoparticles with average size smaller than 5 nm. The latter nanostructures are smaller in size, rough and are absent of pores.

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How tomography helps to solve geological puzzles

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Diamonds originate from deep within the Earth. When they crystallize, they trap other deep Earth's minerals, conserving their shape and size, and preserving their chemical composition. These trapped minerals may record various processes, such as exsolution reactions. Here we show the results of the investigations of J1 diamond mineral inclusions. At the MOGNO beamline, the initial step involved performing a μ-tomographic scan of the diamond to detect the inclusions. Subsequently, specific regions of interest were selected for further analysis, where a tomographic scan employing X-ray nanofluorescence contrast was executed at the CARNAÚBA beamline. Nano-tomography data for each inclusion required ~8 hours to acquire, and the data reconstruction was undertaken by the Scientific Computing Group. The chosen inclusions are composed mainly of Fe, Cu, and Ni, and one is mainly composed of Fe. With XRD analysis at EMA beamline, they were classified as sulfides and hematite-goethite, respectively. Sulfides exhibit dimensions of 70μm and 20μm, displaying twinning and an exsolution texture transitioning from pentlandite ((Fe,Ni)9S8) to pyrrhotite (Fe1-xS), with calchopyrite (CuFeS2) covering the crystals. The Fe-rich oxide inclusion was identified as hematite-goethite-type (Fe2O3OH) with a size of 35μm. This inclusion exhibits irregular holes that were subsequently filled by the diamond. To explain the OH, we suggest that OH-rich fluids reacted with the Fe2O3, leading to the formation of a FeOOH phase. In addition, during the data segmentation process, Fe intensities were differentiated into high and low values to enable quantitative analysis. We conclude that XRF tomography assists as an invaluable tool for comprehending and visualizing the spatial 3D distribution of elements, which may help to solve numerous geological puzzles to better comprehend what is inside of our planet.

Identifying the active site of Cu/Cu2O for electrocatalytic nitrate reduction reaction to ammonia with in-situ spectroscopies

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The electrochemical nitrate reduction reaction (NO3RR) is an alternative for wastewater remediation and electrosynthesis of ammonia can be explored to obtain a high value-added product. Among transition metals, copper-based catalysts are studied for NO3RR due to their favorable interaction with nitrate ions, attributed to the similar energy levels between highly occupied d-orbitals of Cu and the lowest unoccupied π^* orbital of NO3-.2 Copper oxides are attracting attention due to their high selectivity towards ammonia formation compared to unmodified Cu. However, the active site of copper oxides for NO3RR to ammonia is still under debate, where 3 possible ones are postulated: Cu2O-metal interfaces, oxygen vacancies generated by Cu2O reduction, and metallic Cu.2 Herein, we conduct a systematic study kinetically and in situ behavior and composition of Cu/Cu2O composite under different potentials for NO3RR to identify its active site for ammonia synthesis. Combining in-situ Raman, X-ray near edge absorption structure (XANES) and Fourier-transform infrared (FTIR) spectroscopies, we correlate the activity to NH3 with the previous reduction of Cu2O. We detect the reduction of Cu2O at -0.6 V vs. SHE followed by the formation of hydroxylamine at -0.7 V vs. SHE a key intermediate for the production NH3 from NO3RR. We map and track the distribution of Cu and Cu2O domains in situ with XANES spectroscopies conducted in Carnauba beamline. We also evaluate the nitrate rate orders of different degrees of reduced Cu2O surfaces to correlate the catalyst surface with its performance for NO3RR to ammonia. We found that there is a dependence of the applied potential with the active site that boosts ammonia formation at Cu2O-based catalysts. Thus, we were able to give important clues towards the design and performance of Cu2O-based catalysts for NO3RR.

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In situ Monitoring of Exsolving Nanoparticles from Perovskite Oxides using Synchrotron-based XRD and XAS

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The exsolution of nanoparticles (NPs) from perovskite oxides (PO) has emerged as a promising strategy to synthesize stable metal NPs (highly anchored NP-substrate interaction) with tunable electronic and catalytic properties along with minimizing the use of noble metals (1). Generally, the exsolution occurs via a reduction process, where the B-site of PO diffuses to the surface, followed by reduction, nucleation, and growth (2). In this study, we will induce the exsolution process by applying a reducing atmosphere (at 400 0C with 5% H2 gas flow) to different metal doped strontium titanate POs. By monitoring the process in situ using synchrotron-based X-ray diffraction (XRD), we will certainly be able to follow the reaction mechanism by monitoring the structural changes of PO as a function of temperature. Additionally, these findings from XRD technique can be complemented by employing XAS technique, which allows us to monitor how the local B-site coordination geometry and electronic properties of different elements (both PO and exsolved NPs) change after the process. Together, they provide invaluable information on the influence of temperature and non-stoichiometry of both the sites of the POs. Therefore, X-ray-based techniques are promising tools that permit us to obtain unprecedented information in this field, tracking the exsolution of NPs by following their structural and electronic changes. For these experiments at Sirius, we have submitted proposals to Paineira and Ema beamline.

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Investigating the association between the tumor cell glycocalyx and metal ions on tumor progression

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The tumor microenvironment comprises components that promote tumor progression. Among these, we highlight the glycocalyx and metal ions, which are relevant to modulate cell behavior. Healthy and tumor cells present very different glycocalyces, whereas tumor cells produce a richer and denser, glycan-decorated, cell surface that affects tumor growth. Glycoconjugates act as co-receptors and modulate protein conformation and activity, while metal ions are relevant as cofactors to enzymes and signal transduction. Besides, metal ions can interact with polysaccharides promoting changes in polysaccharides chain structure and rigidity. Previous data from our group showed that manganese is a relevant element in the acquisition of malignancy, promoting invasiveness related to cell surface changes after manganese exposure. In this work, we aim to investigate changes in plasma membrane morphology throughout the association between the glycocalyx and divalent metals. In our approach we will simulate changes in tumor microenvironment metallomics by exposing LLC (Lewis Lung Carcinoma) cells to different divalent metals (Mn, Fe, Cu and Zn) in vitro. We plan to evaluate cell morphology changes and metal accumulation through live-imaging optical microscopy, ICP-OES (Inductively Coupled Plasma Optical Emission Spectrometry) and X-Ray fluorescence nanoimaging and ptychography at Carnauba beamline. Our preliminary data indicates that cell exposure to manganese induce early changes in cell morphology, enhancing cell complexity, indicating that manganese effect on tumor cells may be related to in metal-glycocalyx interactions that affect plasma membrane mechanical properties. In conclusion, we have strong evidence indicating that metal-glycocalyx interactions influence cell morphology and expect that such changes may affect tumor cell behavior with the formation of membrane processes that promote invasiveness and modulate tumor cell behavior in accordance with our previous findings

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MICRO AND NANO X-RAY TOMOGRAPHY APPLIED IN SOIL SCIENCES

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This talk aims at presenting current efforts using X-ray micro and nano tomography at Sirius to investigate Soil related processes and properties. It will showcase an image-based strategy to determine the permeability of soil aggregates (i.e., at the microscale). This strategy was applied to a study of soil aggregates from different land-use systems such as forest, no-tillage, minimum tillage, and conventional tillage. Another ongoing study seeks to apply the zoom-tomography capability from MOGNO beamline to understand the role of different soil pores in greenhouse gas emissions, depending on the type of land-use employed. In addition, a sample environment designed for in-situ and time-resolved experiment for investigations of the soil water retention at the MOGNO beamline will be presented. Water retention and movement in soils are highly relevant to evaluate the physical conditions in which plant roots grow. Particularly, this sample environment will be used in studies of cohesive soils from the Coastal Tablelands of Brazil, which are difficult to cultivate due to its remarkable hardness under low water content. With these diverse examples, it is hoped to stimulate ideas of potential experiments at the MOGNO beamline to understand important phenomena occurring in Soils.

Acknowledgements:

References:

Model for energetic dependence of the Eu emission in $NaMgF_3$ fluorperovskites based on synchrotron techniques.

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Lanthanide(Ln)-doped NaMgF3 (NMF) presents interesting luminescent properties, such as optically stimulated luminescence and radiophotoluminescence [1,2]. Due to similarity between Na (with an ionic radius of 118 pm and 8-fold coordination) and Mg2 (with an ionic radius of 72 pm and 6-fold coordination), there is an open question in the literature where the Ln ion is incorporated and which valence will take over in the host with evidence of both 2 and 3 emissions [1-3]. In this context, Photoluminescence (PL) studies in the vacuum ultraviolet (VUV) and visible (VIS) regions combined with XAFS techniques can answer these questions. In this work, Eu-doped NMF samples were produced by microwave assisted hydrothermal method (MAHM). This route is known by produce fluorites compounds with low oxygen and hydroxyl contamination, short synthesis time and low synthesis temperature [4]. X-ray diffraction measurements (XRD) confirmed NMF phase as well as complete dissolution of Eu ions in the matrix. In excitation region below the bandgap (10.5 eV), emission spectra showed both valence of Eu ions with dominant 5D0→7F2 transition (centred around 613 nm) for Eu3 and a weak 5d→4f broadband (from 350 up to 450 nm) for Eu2 ions. For region above bandgap, the emission spectra switch to a violet colour due to increase of Eu2 blue band. Furthermore, 5D0→7F1 of Eu3 starting to dominate in the red region of emission spectra when photon excitation is above 10.5 eV. Results of X-ray absorption near edge structure (XANES) from EMA beamline (proposal - 20221991) demonstrated both valences for Eu ions, confirming PL-VUV measurements. While Extended X-ray absorption fine structure (EXAFS) demonstrated that Eu ions occupy both cationic sites, which can explain the energetic dependence of the emission spectra.

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Mogno beamline - zoom-in and time-resolved experiments of x-ray tomography

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The MOGNO beamline is a high-energy imaging beamline dedicated to in situ and operando experiments in heterogeneous and hierarchical samples, and one of the beamlines of the first phase of SIRIUS, the 4th generation storage ring at the LNLS. The beamline is currently under commissioning and will operate at energies of 21.5, 39.0 and 67.7 keV, using as primary source a 3.2T permanent magnet dipole, with critical energy of 19.15 keV. The beam is demagnified down to a nanometric focal spot (≈120 nm) using a set of three elliptical mirrors, which introduces a divergence and, hence, the beamline operates in a cone beam geometry with variable field-of-view (FoV between 150 μm to 85 mm) and spatial resolution (≈120 nm to 55 um). These conditions are reached with two end-stations, the nanotomography and the microtomography stations. A high-Z photon counting detector, with detection area of ≈85 x 85 mm2, will serve for both the nano and micro-station, but the beamline will also count on an indirect detection system based on a sCMOS camera and a macroscope.

Multispectral studies of metal halide perovskites: simultaneous x-ray ptychography, x-ray fluorescence, and x-ray excited optical luminescence experiments

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The modern lifestyle and the increase in world population have dramatically increased the energy demand, pushing the development of new energy sources. Among new photovoltaic materials, metal halide perovskites (MHP) (ABX3, where A=CH3NH3, CH(NH2)2 or Cs, B=Pb2, X= I- or Br-) are the most promising for solar cells and detectors owing to their low processing cost, easy fabrication, high-power conversion efficiency, and high scintillation yield.[1] Despite that, several aspects of morphology, chemical composition, and optoelectronic properties are to be uncovered from the micro to the nanoscale.[1] X-ray nanoscopy techniques available at the CARNAUBA beamline (SIRIUS/LNLS)[2] are up-and-coming to investigate MHP[3,4], revealing the correlation among morphology (x-ray ptychography), stoichiometric chemical distribution (x-ray fluorescence - XRF), and optoelectronic response (x-ray excited optical luminescence - XEOL) of these materials. We report x-ray nanoscopy experiments, including coherent diffractive imaging (CDI) in flyscan mode. Ptychography in phase contrast (2-atmosphere was essential to mitigate eventual sample damages that are typically the main limiting factor to applying the CDI in such samples. Our multispectral images of hybrid perovskites are a pivotal step in developing and applying the technique in beam-sensitive samples for high-resolution imaging, especially in the case of heterogeneous and hierarchical functional materials in which the multiscale properties determine final device performances.

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New Superconducting Phases in Light Materials

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Despite significant efforts to increase the critical temperature of conventional, cuprates, and iron-pnictides superconductor materials, none of them have yet demonstrated superconductivity at room temperature and pressure, with all critical temperatures remaining below 150 K. Super hydrides compounds have shown potential for room temperature superconductivity, but only under high pressure conditions [1-3]. However, 2023 had renewed the excitement in the field with the report of near-ambient superconductivity in N-doped lutetium hydride [4] and the controversial announcement of room conditions superconductivity in LK-99 [5]. Therefore, the quest for superconductivity in suitable environmental conditions for urgent applications continues. In this talk, I will show that this quest might take new pathways and then we can look at possible superconductor candidates with light elements. Superconductivity was predicted to appear in mild pressure and temperature conditions (50 GPa and 15 K) in an electride compound based on lithium and carbon [6]. Two years after the release of this prediction we have observed for the first-time signs of the expected phase through in-situ extreme conditions synchrotron X-ray diffraction experiment carried out at EMA beamline.

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Predominant surface states of the multiple topological material Sb4Te3

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Topological materials are well-known for their surface states with different electronic properties [1-6]. Recently, we have calculated the electronic structure of Sb4Te3. Remarkably, we have found that this compound possesses several topological states due to complex structure as well as different surface terminations. The goal of this experiment is to determine the predominant surface states of Sb4Te3. We have identified three strong topological gaps in Sb4Te3 , via DFT calculations, that are adjacent to each other, at different energies, and below the Fermi level. These gaps are related to three TSS Dirac cones. The possible energy alignment of electronic states from different structural terminations opens up the possibility of information transfer at the surface of these materials. The atomic structure of Sb4Te3 can be described as a stack of Sb2Te3-Sb2Te3-Sb2-Sb2 layers. The insertion of bilayers in topological insulators creates new surface electronic states . We performed a DFT calculation for these terminations and it is possible to observe different TSS in the band structure. Arpes measurements were performed at BLOCH line in MAX IV synchrotron to verify these states and observe how this material can change properties depending on its termination.

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Studying the electro-oxidation of glycerol on perovskite oxides in situ with X-rays

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Amidst the ongoing global crisis linked to issues like global warming, pollution, and the diminishing reserves of fossil fuels, the imperative need for sustainable energy sources has become an undeniable reality. One promising field poised to make significant contributions to this cause is electrocatalysis. However, the widespread utilization of noble metals in electrocatalysis can pose economic challenges that hinder its broad implementation [1]. In this context, perovskite oxides have gained considerable attention as non-rare metal catalysts. They have already found application in critical processes such as the oxygen evolution reaction (OER) [2] and the electro-oxidation of biomass components like methanol and glycerol [3]. Nevertheless, given the ultimate goal of utilizing these materials in energy-related devices like electrolyzers and fuel cells, it becomes imperative to comprehensively understand the underlying mechanisms governing their operation. This understanding is crucial for enhancing the efficiency of the electrochemical processes in which they are involved. To address this problem, we used X-ray based techniques to capture nanoscale heterogeneities—a pivotal aspect of electrocatalysts [4]. Firstly, we performed in situ X-ray fluorescence to locate the material, images the material grains and analyze whether it leechs during the reaction. Then, we used in situ nano X-ray Absorption Near Edger Spectra (XANES) before and during the electro-oxidation of glycerol to analyze how the B site oxidation state (the one that mostly governs the material reactivity in this kind of perovskite) changes. We observed that the most active compositions would show much higher oxidation state changes in the grain edges during the reaction. This observation shows that nano XANES can be of significant importance towards an in depth understanding of the local mechanism behind the activity of these perovkites towards biomass electro-oxidation.

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Three-Dimensional Imaging of Hierarchically Mesoporous Catalysts with Coherent X-ray Diffractive Imaging

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Zeolites are an important class of materials, known for their microporous structure and high acidity, widely used as acid catalysts and support in several reactions. Key properties of these materials are the diffusion rates through their pore network and the inhomogeneity of their acid sites. Controlling zeolite synthesis is needed to achieve better catalytic properties. Moreover, the formation of mesopores in zeolites modifies their catalytic properties but also delays their deactivation due to coking. Novel 4th generation synchrotron facilities are leading to the development of new microscopy X-ray methods. Among these techniques, coherent diffractive imaging (CDI) is one of the most promising, enabling nanometre-scale imaging of non-crystalline samples. Indeed, new visualisation methods can be used to resolve structures at resolutions that were previously unachievable. Here, I will present the application of coherent diffractive imaging for the three-dimensional visualisation of hierarchical mesoporous catalysts, with a resolution of < 50 nm. Thanks to the high-penetration depth of the X-ray beam, we visualized, in a non-destructive manner, the 3D complex mesoporous structure of 10-micron MCM22 zeolite crystals, with controlled mesoporosity and obtained quantitative information about pore size distribution and pore network interconnectivity across the whole crystal. The non-destructive nature of this method, coupled with its ability to image samples without requiring modification or a high vacuum environment, makes it valuable in the fields of porous- and nano-material sciences enabling imaging under different environmental conditions [1]. The 3D imaging of the zeolite crystals was accomplished in less than two hours data collection time. The temporal resolution of plane-wave CDI will pave the way to novel in situ time-resolved and in operando nanotomography studies exploring real-time phenomena, spanning chemistry, biology, catalysis and materials research fields. These experiments were carried out at the Cateretê beamline [2] at the SIRIUS 4th generation synchrotron source.

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Tracing paleoenvironmental and preservational aspects of 3.4 billion years old fossils from Pilbara region, Western Australia by using synchrotron-based X-rays techniques

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The Dresser Formation (DF), Western Australia, hosts important geoheritage sites that contain one of the oldest records of life on Earth (3.48 billion years old) [1,2]. Accessing any biosignature in such ancient rocks means opening a window to understanding how life successfully colonized the planet. Microbial laminae that form microbial mats and microbialites can exhibit morphological textures and horizontal geochemical patterns, that can be preserved even over billions of years. In these laminae, the biofilm produced by bacteria can serve as a nucleation site for biominerals. The aim of this research was to evaluate the biogenicity of samples of putative stromatolites and microbially induced sedimentary structures (MISS) collected in the surroundings of Buik Geoheritage Reserve in DF. These samples present morphological and microscopic features consistent with microbial structures. Here, synchrotron-based x-ray techniques such as XRF, XANES, XRD and WAXS (experiments performed at the Carnaúba beamline, Sirius, and NanoMAX beamline, MAX IV, coupled to lab-based Raman and photoluminescence spectroscopy) were of fundamental importance to access the inorganic biosignatures with high sensibility and high resolution, enough to resolve nanometric potential microbial fossilized structures. The characterization and evaluation of trace elements in submicrometric putative biominerals is of fundamental importance to attest their biogenicity. The paleoecological and preservational parameters is being evaluated based on the spatial distribution of the mineral assemblage and its associated trace elements, in order to understand the environmental and ecological aspects of early ecosystems and the preservation signatures left by them. The confirmation of the biogenicity of these rocks is essential for the extension of the protected areas around the reserve, in a joint effort with the GSWA, helping to preserve this important site threatened by mining and human activity in general.

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Understanding the Bioelectrocatalytic Mechanisms of Metalloenzymes by using Synchrotron Light

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Multicopper oxidases are metalloenzymes containing four copper ions in their catalytic sites. These enzymes have been applied as bioelectrocatalysists on electrochemical energy conversion systems, as biofuel cells, and on water splitting, as they catalyze the oxygen reduction reaction and the water oxidation reaction at very small overpotentials and under mild conditions. Understanding the electron transfer mechanisms of redox proteins has direct impact on their successful practical applications. X-ray absorption spectroscopy (XAS)is a powerful technique for studies on metalloenzymes, as it can provide information on the oxidation state of redox metal co-factors and their chemical environment, without interferences. In addition, XAS can be couple to electrochemical measurements (in situ XAS) to probe mechanisms of redox catalytic reactions under real reaction conditions. Here, aspects of the electron transfer and bioelectrocatalytic mechanisms of bilirubin oxidase (a multicopper oxidase) toward the oxygen reduction reaction and water oxidation reaction by in situ XAS are shown. Through these measurements was able to probe that copper ions act as a 3D redox active electronic bridges for the electron transfer reaction in the enzyme active site. In addiiton, the driving force and energy balance of the catalytic water oxidation reaction could be experimentally calculated.

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X-Ray Microtomography for Evaluating Groundwater in-situ Chemical Remediation at Pore-Scale

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In situ chemical oxidation (ISCO) has been widely used as a remediation method in contaminated areas, considering its increasingly consolidated effectiveness. The application of the technique using potassium permanganate (KMnO4) for the degradation of trichloroethylene (C2HCl3) has been reported in pilot-scale and field studies, with favorable remediation results. A limitation of the process optimization is the gaps in the knowledge of the reaction at the microscale. Thus, this project aims to investigate the dynamics of the ISCO remediation process of trichloroethylene (TCE) with potassium permanganate (KMnO4), through 3D synchrotron microcomputed tomography (μCT) images resolved in time, from an experiment of injection in porous medium. From the processing and analysis of data, the characterization of the sample saturated in water was made, obtaining information such as the total porosity, the surface area of the grains in the matrix, and the tortuosity of the material. With the analysis of the samples saturated with TCE, and potassium permanganate, we hope to observe the changes in the permeability of the medium and the amount of residual contaminant, in order to conclude about the remediation dynamics at pore scale.

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Zero-valent iron nanoparticle behaviour on sandpack samples: pore-scale insigths using synchrotron X-ray computed microtomography

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Nanoremediation is an emerging technology that consists of treating contaminated aquifers with reactive nanomaterials. Studies at column, pilot, and field scales have reported successful decrease in contaminant concentration upon injection of zero-valent iron nanoparticles (nZVI) in contaminated zones[1-4]. The knowledge gaps surrounding the processes that occur at a pore scale limit our ability to design optimized nanoremediation processes at larger scales [5]. Thus, this study aims to investigate the mobility and entrapment of nZVI in porous media at a pore-scale, using synchrotron X-ray microtomography (μCT) to capture the details of fast processes.

Two sand-packed columns were analysed by μCT imaging. They were first saturated with water and subsequent injected with the nZVI suspension. A post water flushing was done to remove the mobile nZVI. The column was imaged in three segments by moving the μCT stage in the vertical direction, helping to preserve image resolution whilst analysing a relatively large sample, and thus to investigate nZVI mobility along the entire column length at each experiment step. A reduction of about 70% of porosity was observed on both cases after nZVI injection, causing a drastic reduction in permeability. The nZVI suspension is not miscible with the water phase, first occupying the larger pore-spaces. After the last water flush, the trapped nanoparticles were mainly occupying the pore-throats, that is, the narrowest parts of the flow pathways. Most of the original total porosity was recovered on sample 2 after the water flush, whilst sample 1 only recovered about 50% of its initial total porosity.

This experiment shed light on the pore-scale mechanisms involved in nZVI entrapment in porous media. Future studies shall take advantage of the higher spatial and temporal resolution at the new beamline at Sirius, allowing the analysis of the nZVI suspension front during injection in nearly real time.

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POSTER

A Ceramic Moisture Sensor Development (PbTiO3-La) for Precision Agriculture

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Precision agriculture plays a crucial role in optimizing agricultural processes with the aim of increasing productivity and reducing the consumption of natural resources. In this context, soil moisture sensors play a fundamental role in the efficient management of water resources. This study describes the development of a ceramic moisture sensor based on lead lanthanum titanate (PbTiO3-La) designed for precision agriculture applications. The modification of lead titanate with lanthanum was conducted to enhance the sensitivity and stability of the sensor under different soil moisture conditions. The choice of lanthanum as the modification material was based on its unique electrical and structural properties, which can significantly improve the sensor's performance. Furthermore, this study explores the characterization of the sensor using advanced synchrotron light techniques. Synchrotron light provides a highly brilliant and coherent radiation source, providing a detailed analysis of the structure and properties of the sensor material at micro and nanoscales. This synchrotron-level characterization offers valuable insights into the interactions between the sensor material and soil moisture, as well as structural changes that occur during the moisture detection process. The resulting sensor holds promising applications in precision agriculture, allowing for continuous and accurate monitoring of soil moisture. Additionally, characterization in X-ray diffraction techniques provide an innovative approach to understand and optimize the sensor performance at molecular levels. This study contributes to the advancement of agricultural sensing technologies, providing a solid foundation for future research in soil moisture sensing and the monitoring of other micrometeorological variables.

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Advanced collagen-based biocomposites reinforced with *2D nano*-talc for applications in bone tissue engineering

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The advancement of nanobiocomposites reinforced with bidimensional (2D) nanomaterials plays a pivotal role in enhancing bone tissue engineering [1]. In this study, we introduce a novel nanobiocomposite that reinforces bovine collagen with 2D nano-talc, a recently exfoliated nano-mineral [2, 3]. These nanobiocomposites were prepared by blending collagen with varying concentrations of 2D nano-talc, encompassing mono- and few-layer talc from soapstone nanomaterial. Extensive characterization techniques including Atomic Force Microscopy, X-ray photoelectron spectroscopy, nano-FTIR, s-SNOM nanoimaging, Force Spectroscopy, and Peak Force Quantitative Nanomechanical Mapping were employed. The incorporation of 2D nano-talc significantly enhanced the mechanical properties of the nanobiocomposites, resulting in increased stiffness compared to pristine collagen. In vitro studies supported the growth and proliferation of osteoblasts onto 2D nano-talc-reinforced nanobiocomposites, as well as showed the highest mineralization potential. These findings highlight the substantial potential of the developed nanobiocomposite as a scaffold material for bone tissue engineering applications.

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Advanced Structural Investigation of Human Hair

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Human hair has two major morphological regions well distinct: the cuticle and cortex. Between the cells of the cuticle and cortex, cell membrane complexes (CMCs) are present, consisted of proteins and lipid bilayers. The main function of the cuticle, the outer structure, is to provide mechanical protection for the cortex, which contains a helical fraction comprising a crystalline phase (intermediate filaments - IFs) embedded in amorphous matrix that is sensible to water, largely influenced by the relative humidity. Hair treatments with chemical products and the use of thermal devices, can promote important structural changes like protein denaturation, water removal and surface damages on the cuticle. We combined a number of techniques, Small-Angle X-ray Scattering (SAXS) in situ measurements (varying the temperature), thermogravimetry coupled to mass spectrometry (TG/MS), optical microscopy (OP), X-Ray computer tomography (XRCT) and differential scanning calorimetry (DSC) to obtain structural details on the human hair and structural changes upon chemical and thermal treatments [1,2]. Caucasian virgin hair, Caucasian bleached hair, Caucasian straightened hair and Caucasian bleached and straightened hair were investigated. As will be shown, several results could be obtained providing important structural details as changes on porosity of the inner structure of hair, changes on the CMC structure and hair surface characterization. These results are very important for the basic knowledge on hair structure but also for the cosmetic companies and professionals on hair styling to understand the implications of their products and treatments.

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A new *operando* capillary plug-flow cell for QUATI beamline

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A new versatile capillary plug-flow microreactor with controlled temperature has been designed for operando x-ray absorption/fluorescence spectroscopic, Raman and diffraction studies [1] to be used at QUATI beamline [2]. This setup will allow characterize materials in long (XRD) and short (XAFS) range order and additionally follow solid-gas interaction with Raman. The samples could be placed in a quartz or sapphire capillaries up to 2 mm of external diameter in the center of an aluminum oven's cavity between 2 resistive wires (Kanthal), coiled around a ceramic tube, that are connected in series to a direct current supply. A thermocouple placed close to the catalyst bed's back is used to monitor the working temperature. The heating system was designed to increase the heating by focused IR radiation and to minimize axial gradient temperature along the bed. While a Kapton is placed on the front window to minimize convection cooling. The extremities of the capillary are attached, through Swagelok® fittings, to a gas inlet and outlet system, which can be connected to a gas analysis system such as mass spectrometry and gas chromatography to evaluate the catalytic activity. The novelty on this device is that has been built using modular components that facilitate its handling by any user for experimental setup, which mainly aims to reproduce industrial conditions of chemical processes, at a laboratory scale, enabling the establishment of plenty of variety environments (temperature and pressure up to 1273 K and 20 bar, respectively) to explore, phase transition, reaction kinetics of gas-solid interaction, and heterogeneous catalysis.

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A operando XAS cell design for research IT-SOFC at QUATI beamline: an electrochemical cell for operando measurements

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An operando characterization of electrode materials under electrochemical reaction conditions is important for their further development. X-ray absorption spectroscopy (XAS) presents a unique opportunity in this regard, as it allows data to be collected using user-designed operando cells. The development of a device that enables the characterization of several properties simultaneously under controlled atmospheres in a wide range of temperatures represents an advantage over the traditional independent measurements since those properties could be strongly correlated. Here we propose a design and implementation of a new high-temperature cell that allows performing in-situ or operando X-ray Absorption Spectroscopy, X-ray Diffraction or Raman simultaneously with electrochemical characterization at a wide range of temperatures and atmospheres. In this work we present the first design of this device devoted to study of intermediate temperature SOFCs at QUATI beamline [1]. The operando cell contains a sample stage with a tunable head so that XAS data can be collected at different angles between the electrode and the X-ray beam with an accuracy of 0.5. The mechanism to adjust the angle of incidence of the beam on the sample allows control over the depth of penetration of the X-ray photons into the electrode. At low angles, it becomes possible to collect surface sensitive data, which is of great importance as electrochemical processes are believed to take place on the surface of the electrodes. Follow the oxidation state changes occurring in elements in the near-surface region compared to the bulk of the electrode is one of our aims in this cell. Such an ability to distinguish between the surface and bulk properties of the electrode during a real reaction environment will help to understand the underlying phenomena better, which will enable electrode design targeted towards the reactions of interest.

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Automated alignment of optics and sample precession correction at the IPE beamline of Sirius

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The IPE beamline at the Sirius synchrotron light source is currently in the commissioning phase and is set to become a vital tool for material research. To optimize alignment tasks and reduce downtimes and errors, it is necessary to develop automated procedures. One example is automating the beam switch between the two experimental stations RIXS and XPS. Manual execution of this task, involving the translation and fine tuning of a plane mirror's pitch angle, is time-consuming and prone to errors. We developed an iterative method based on optimizing the photon flux through the exit slit by making angular scans the mirror for successively smaller slit apertures. However, due to the long distance between the mirror and the exit slit, the mechanics of the mirror translation, based on the sliding of large granite bases, introduced significant errors in the mirror's rotation, resulting in considerable intensity losses. We fine-tuned the procedure to compensate for the mechanical errors, with the piezoelectric actuator controlling the rotations. This resulted in a more robust method, with the final photon flux after repeated beam switches remaining consistently smaller than 2%. Another interesting problem for automation is the precession of the UHV sample manipulators (in both XPS and RIXS stations), leading to the loss of the focal point of the beam in the sample upon rotation. We showed that this effect could be compensated by making mechanical adjustments and using a circular trajectory of the sample holder, simultaneously with their rotation. The calculations, obtaining empirical parameters for the model, and implementation were all performed at the beamline. These contributions will improve the beamline's performance and make it fully available to users soon.

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CaOCrAl nanocomposite synthed by catalytic combustion

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In this work, the synthesis process was used by combustion in a digital muffle furnace (Solid Steel-SSFM (100°C-1200°C) with temperature control and stable atmosphere. Calcium Chloride (CaCl2.2H2O) P.A., Aluminum Chloride (AlCl3.6H2O) P.A., Chromium Chloride (CrCl3.6H2O) P.A. and the fuel catalyst urea (CH4N2O) P.A. The masses were made in the proportion of 1:1:1:3 respectively. After being homogenized in a mortar, they were inserted into a crucible and went through the catalyst activation process, at temperatures of 150°C, for 20 minutes four times. After this stage, combustion processes were carried out at temperatures of 200°C, 220°C, 280°C for 20 minutes for 3 times, by temperature. The samples, after synthesis, were characterized by XRD, EDS, FTIR, UV-Vis-NIR, TEM and scanning microscopy. Optical microscopy observations were also carried out. XRD measurements showed that the structure (CaOCrAl) was evidenced using the Rietveld refinement method. Fluorescence measurements showed the atomic constituents with their respective energies for Ca, O, Cr and Al [1;2]. FTIR measurements showed the presence of metallic compounds in the low wavenumber region. UV-Vis-NIR measurements showed that GAP energies have energy around 3.5 eV. TEM measurements showed the morphology of the sample and after making lognormal adjustments to the diameter distribution, it presented an average diameter of 25 nm. Scanning microscopy showed that the morphology of the nanoparticles has rodlike surfaces.

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Characterization of microstructures of specialtyoptical fibers for electric-field sensing by propagation-based x-ray phase-contrast microtomography

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In this work, we present a quantitative 3D morphological characterization of optical fibers used in electric-field sensing. The characterization technique employs propagation-based x-ray phase-contrast microcomputed tomography (micro-CT). In particular, we investigate specialty optical fibers that contain microstructured holes that are electro-optically modified by thermal poling to induce second-order nonlinear effects (SONE). The efficiency of the SONE is reflected in the characterization parameter, $V\pi$, which is highly dependent on the dimensions of the fiber. The fiber microstructures must be uniform to support the fabrication of reproducible devices. The results obtained using the micro-CT technique performed in IMX beamline (LNLS) show that uncertainty of $\pm 1.7\%$ arises in the determination of the expected value of the voltage that causes a change in the phase of the electromagnetic wave. We expect, performing measurements at Mogno beamline, to obtain more detailed information of different types of fiber optics used for different kinds of applications. The group has a scientific commissioning beamtime (Mogno-20231815) and during RAU2023 will present the first results.

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Coherent imaging of nanostructures under extreme conditions

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The crystalline structure of nanomaterials under extreme pressure and temperature conditions are affected by processes like phase transition and defect formation, which impact on their basic physical properties. Understanding crystal dynamics in extreme conditions might be the key to further tailoring of potential materials for a range of industrial applications. Sirius, one of the few 4th generation synchrotron sources in the world, allows the use of high brilliant high coherent X-ray beams on experiments able to explore the edges of knowledge in science and technology [1]. The EMA beamline offers the opportunity to work with several synchrotron techniques, like X-ray scattering, diffraction, spectroscopy and fluorescence, while submitting materials to high pressure, as well as high and low temperatures [2].

Coherent imaging techniques use computational routines to overcome the phase problem and retrieve local information of nanomaterials with resolution better than the beam size [3,4]. Although users from several research groups are already taking advantage of EMA´s versatility to extract valuable information on their experiments, the great potential of the beamline for applying coherent imaging on nanomaterials under extreme conditions is so far unexplored. By commissioning coherent imaging at EMA, we aim to offer this state-of-the-art technique to users who are interested in exploring local processes with spatial resolution down to the order of ten nanometers [5]. This can reveal properties so far difficult to access, either because they require extreme ambient conditions or simply cannot be spatially resolved by traditional techniques already available at the beamline.

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Could solvents potentially affect the mobility of water within the confined channels of reverse wormlike micelles?

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This study explores supramolecular structures formed as lecithin dissolves in organic solvents (as alkanes) with trace water, creating reverse wormlike micelles (RWLM) [1] RWLM characteristics are incompletely defined, with alkane choice significantly influencing morphology. Integrating cryo-TEM and SAXS results aims to elucidate mechanisms guiding RWLM formation and growth, alongside the solvent's impact. SAXS experiments confirm solvent-dependent RWLM structure, with higher water increasing channel diameter. The literature-guided electron density difference in RWLMs expands the micelle cross-section with rising water content. In cyclooctane, a trend at higher q-values suggests a structural transition absent in octane. Linear solvents confirm solvent influence on micellar parameters. Despite constant water content, subtle changes at higher q-values and significant changes at low q-values highlight sensitivity to hydrocarbon chain length. For cryo-TEM experiments, liquid ethane, ideal for cryogenic vitrification, has challenges due to its oil-dissolving nature. High viscosity limits plunge freezing in sample prep. To address this, a gel method was introduced: sample dispensing onto the grid box with a syringe/needle, followed by flash-freezing in liquid nitrogen. Two wormlike micellar system prep methods were explored. Immediate freezing resulted in aligned chains; a 180-second waiting period, based on rheological measurements, ensures successful cryo-TEM application in studying oil-continuous microstructures. RWLMs in cyclooctane at various W0 values revealed solvent-related morphology changes. Lower W0 exhibits oriented micelles; higher W0 displays alignment and a fingerprint-like arrangement, marking the first RWLM observations in cyclooctane across diverse W0 values. Notably, no alignment effects were observed in octane despite differing relaxation times in cyclooctane, possibly due to longer relaxation times linked to micelle length.

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Cruzeiro do Sul: base de dados espectroscópica de referências experimentais e teóricas para o SIRIUS

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A técnica de absorção de raios X é muito atrativa devido à sua sensibilidade atômica e versatilidade. Geralmente, é complementada por outras técnicas de caracterização para que a informação fornecida por uma técnica acrescente informações à outra técnica. No entanto, em certos casos, algumas técnicas não são adequadas para serem realizadas conjuntamente devido à natureza do material, o que dificulta a identificação dos espectros apenas pela utilização da técnica de absorção de raios X. Hoje, a comunidade científica tem à disposição ferramentas computacionais para interpretar os espectros de absorção. Entretanto, estas técnicas frequentemente estão sujeitas à consulta de bases de dados experimentais, as quais costumam ser limitadas em volume de dados. No SIRIUS [1], na linha QUATI [2] a ideia de ter um banco de dados experimental é atrativa devido à grande quantidade de dados que serão gerados e processados internamente. Assim, o principal objetivo deste trabalho foi desenvolver um banco de dados com mecanismos simples de consulta pela Internet para facilitar a obtenção de conjuntos de dados espectroscópicos. Para isso, desenvolvemos um banco de dados integrado a um website, o qual armazena espectros fornecidos pelos usuários. Além disso, foi implementado um algoritmo genético para buscar soluções com bases em combinações lineares, uma vez que amostras que contém mais que uma fase cristalina ou diferentes materiais, podem dificultar a análise e identificação de cada componente. Portanto, o usuário pode fornecer um espectro no qual diferentes materiais ou fases cristalinas podem ser identificados, a depender dos materiais presentes na base de dados. Portanto, é de suma importância a colaboração dos usuários, pois ao fornecerem espectros, contribuem para tornar as identificações mais precisas. Promovendo o avanço na pesquisa científica e a compreensão do fenômeno estudado por meio da técnica de absorção de raios X.

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DEVELOPMENT OF AN EFFICIENT CELLULAR MODEL TO STUDY THE BIOLOGICAL EFFECTS OF CONVENTIONAL AND FLASH RADIOTHERAPY.

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Flash radiotherapy (RT) is an innovative and promising RT modality in preclinical studies. The Flash effect, which involves delivering radiation in a very high dose rate at milliseconds, holds the promise of efficiently eliminate tumor tissue while minimizing damage to surrounding healthy tissue. To better understand the biological mechanisms of the Flash effect, this project aims to develop and validate a cellular model of radiotherapy. To reach this goal, we designed and adapted a radiological treatment protocol for conventional RT and Flash RT based on the literature. We also designed and improved cell death quantification protocols using tumor cell viability assays (MTT) and cell survival assays (clonogenic assay). The cell lines to be used in this study are human glioblastoma T98G and U87MG and human neural progenitors. Conventional RT was performed using a clinical linear accelerator and Flash RT will be executed at Sirius. The results of MTT assay revealed that T98G cells were equally affected by different dose rates in conventional RT, with reduced viability at all three dose rates tested (400 cGy/min, 1400 cGy/min and 3300 cGy/min). Interestingly, the clonogenic assay of T98G cells revealed a notable difference, with approximately 30% of cell survival compared to the approximately 85% cell viability observed in the MTT assay. These differences may be attributed to the fast metabolism of tumor cells that influence the MTT assay, while clonogenic assay highlights cells that were able to replicate. Therefore, we conclude that it is possible to simulate radiotherapy using cellular models, but attention to details regarding physical and biological conditions is crucial for obtaining reliable data. Our next steps include adapting our 2D protocol into 3D models to better simulate the tumor microenvironment, as well as executing experiments at Sirius (CNPEM, SP) to achieve high dose rates, compatible with the Flash effect range, using Mogno beamline line.

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Development of microfluidic devices compatible with synchrotron techniques for in-situ monitoring of mineral precipitation on rocks

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Microfluidic devices have emerged as an essential tool for reproducing large-scale phenomena in a controlled environment. Their main characteristics are the reduced size, low weight, and high throughput, as well as the capacity to operate small amounts of samples through micrometer and/or sub-micrometer channels. In this sense, devices for reproducing flow in simplified pore structures have been fabricated using engineered materials such as silicon, glass, and PDMS. However, these devices present inherent limitations once they do not fully reproduce complex micropore structures and do not replicate the natural chemical reactivity of the real rock surface. As a result, these micromodels do not fully evaluate the fundamental mechanisms of flow, transport, and reactions within the context of an actual reservoir. To overcome these limitations, two microfluidic devices were developed considering a real rock as the sample. In addition, these devices are compatible with synchrotron-based techniques which propitiates higher spatial and temporal resolutions. The first device is composed of a 5 mm thick rock matrix with a channel 1 mm wide, 0.5 mm deep, and 10 mm long in one of the surfaces. This configuration allows the injection of acid solution along the channel and in-situ monitoring of the reaction's evolution by performing X-ray fluorescence maps and X-ray absorption with time in the Carnauba beamline. The second is a three-dimensional printed device designed to hold a 2.5 mm diameter cylinder-shaped rock and set a fluid flow along it. By performing time-resolved tomography measurements at the Mogno beamline, this device allows to track the morphology evolution during the injection. Both setups presented promising results in the first test, permitting to observe the mineral dissolution as well as the pore size distribution changes. Next step is to unify these devices to obtain both chemical and structural information from the same sample.

Elemental analysis and surface chemical speciation in the formation of Pantanal aerosols

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Atmospheric aerosols are particles emitted by natural sources (ocean waves, leaf litter and living beings) or anthropogenic (biomass and fossil combustive burning) and remain suspended in the atmosphere until undergoing deposition processes. These particles acquire their own characteristic depending on their sources and participate in reactions with gases. The interest in characterizing aerosols collected in nature is current and seeks to resolve questions about their formation (SANTOS et al., 2021). One of the major uncertainties for climate models still relates to the microphysical parameters of aerosols, such as size, composition, and structure, which govern their optical properties (BZDEK et al., 2020). This work proposes to develop a methodology of aerosol sampling, in short time intervals, to verify the changes on the chemical surface species promoted by the aging of these atmospheric particles. The aim is to infer the formation kinetic using synchrotron XPS, XANES and EXAFS analytical technique (OUF et al., 2016), the great sensibility promote by Sirius will enable this research project, once bench equipment do not have the necessary sensibility to realize the measurements. The results can directly contribute to areas such as climate change, cloud formation, human health, and recent scientific applications such as the impact of aerosols in precision agriculture as part of micrometeorological modeling combined with data from fixed stations.

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Elementary characterization, classification and distribution of atmospheric aerosols.

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This project's main theme is applications that aim to develop precision agriculture. In this context, we bring together several themes that can contribute to the development of this technology in the state of Mato Grosso, Brazil. Monitoring particles in the atmosphere has brought important answers to climate and environmental issues. Air quality has been a great health concern, due to the damage it causes to human health, and more recently, the consequences of aerosols in agriculture have been discussed in order to understand the environmental and atmospheric conditions that affect grain cultivation. To this end, we developed a particle monitor using a Raspberry pi development board, AS726X spectrometer sensor, analog-to-digital converter and signal amplifiers, programming in Python. Bench tests on sensor development showed promising results for applications involving air quality monitoring, the principles of data collection and smart sensors can be used for modeling in Precision Agriculture. Atmospheric aerosols can influence variables related to luminosity, which are essential for optimizing crop management and promoting a more efficient and sustainable agriculture. The validation of the developed sensor will be through the correlation between the data obtained with the sensor and the results of aerosol characterization carried out with Conventional X-ray fluorescence Analysis (EDXRF – Energy Dispersive X-ray Fluorescence) and synchrotron light through TEY, (X-ray absorption spectroscopy – XAS, XANES, EXAFS). The depth of characterization of these techniques is fundamental for validating the developed sensor, with measurements referring to biogenic, biomass burning and urban aerosols. This work is part of the project Micrometeorological Station for Precision Agriculture, Process FAPEMAT-PRO 000328/2023.

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EMA Beamline at Sirius: A Versatile Platform to Probe Glass and Glass Ceramics Under Extreme Thermodynamic Conditions

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Glass and glass ceramics are functional and widespread materials, albeit their structural complexity. Glasses configure non-crystalline states of matter, characterized by their absence of translational symmetry and thermodynamic instability. In turn, glass ceramics are composites formed through the partial and controlled crystallization of the parental glass under treatment, then presenting enhanced properties. As valuable external thermodynamic variables, pressure and temperature act differently and complementarily, prompting distinct structural modifications to their final structures [1]. Synchrotron-based techniques have historically been powerful probes for unraveling the intricate structure of glass and glass ceramic and correlated fine-tuned properties. Such details can be addressed not only at the final stage but also through their spontaneous transformation along the thermodynamic treatments, being highly sensitive to the P,T pathways undertaken. In this regard, the Extreme Condition Beamline (EMA) at the Brazilian Synchrotron Light Source – Sirius, an experimental station dedicated to multiple hard-energy X-ray synchrotron techniques at extreme sample environments [2], may serve as a versatile platform to probe in-situ vitreous systems under varying extreme P,T conditions, encompassing $P < 300$ GPa and $0.5\ K < T < 6000\ K$. The selection of examples explored here addresses, not restrictively, some potentialities through X-ray diffraction, X-ray Absorption and X-ray Raman Scattering spectroscopies available for the users at the EMA Beamline.

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Evaluation of protective effects of Losartana in rat coronary arteries submitted to thoracic radiotherapy: A multielemental analysis at CARNAÚBA beamline

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The cardiac regeneration has been studied to better understand the damage that occurs following radiation procedures in the heart tissue after many thoracic cancer treatments. In this proposal we want to compare the coronary arteries of health rats with the irradiated group composed by rats submitted to thoracic radiotherapy and the group of rats treated with losartan using X-ray fluorescence at CARNAUBA beamline, in order to identify morphological and elemental changes in the tissues. For this experiment, eighteen healthy Wistar rats, eight weeks old, were randomly allocated into three groups: control group; a group that received irradiation (IR); and a group that received irradiation and were treated with losartan (IRL). At three months old, the animals from groups IR and IRL were irradiated using the Small Animal Radiotherapy Research Platform. Control rats were submitted to the same procedure, but were not irradiated. After two months of treatment, the hearts were carefully cleaned with sterile saline. The coronary artery was removed, cut, and fixed (10% neutral buffered formalin solution), processed for optical microscopy, and then embedded in Paraplast Plus (Sigma-Aldrich, St. Louis, MO, USA). The measurements were conducted at the CARNAÚBA beamline. Experiments were performed at 9.725 keV in order to access elements like calcium and also zinc. The sample is raster-scanned through the nanoprobe providing two-dimensional maps. XRF images of each sample were acquired in a field of view of 50 µm x 50 µm with a step size of 500 nm. The present study aimed to verify the potential use of losartan in reducing, or even preventing, the radiation-induced coronary damages that can occur after thoracic RT. The use of X-ray fluorescence measurements at the Carnaúba beamline in coronary artery of rats will provide detailed the chemical information of the effects of radiation-induced and the beneficial effect of losantan in this artery.

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Exploring Boundaries in X-ray Photoelectron Spectroscopy: Study of Atmospheric Aerosol Interfaces with an Environmental Chamber

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The atmosphere plays a vital role in climate and the environment, consisting of gasses, aerosols, and clouds. The interaction between aerosols and gasses at a thin interface on the particle surface is essential for processes such as smog and particle formation, impacting properties like chemical activity and hygroscopicity [1]. Studying these interfaces necessitates considering environmental factors due to their sensitivity. X-ray Photoelectron Spectroscopy (XPS) is a sensitive technique for surface analysis, providing information about elements, protonation states, and functional groups [1]. Synchrotron-based XPS offers advantages, enabling high-resolution measurements on aerosol particles. However, the traditionally required ultra-high vacuum (UHV) can be limiting. In this work, we propose the use of an environmental chamber to control pressure, enabling the analysis of aerosol samples collected outside the UHV, particularly those collected during the dry season in the Pantanal biome. This approach, known as ambient-pressure XPS (APXPS), extends the application of XPS to studies of solid-gas, solid-liquid, and vapor-liquid interfaces, with applications spanning environmental chemistry in atmospheric studies [2]. The key feasibility of the APXPS environmental chamber is its capability to withstand helium pressures of up to 20 mbar [2]. We intend to employ synchrotron-based spectroscopic techniques, such as O 1s XPS, as the O 1s photoemission signal area serves as a good indicator of surface fluctuations, along with Auger emission spectra at the O K-edge, aiming to investigate binding and oxidation states, as well as the elemental chemical composition, at the aerosol/gas interface under ambient pressure conditions, including its hygroscopic activity. It is expected that the CNPEM Sirius facilities will enhance atmospheric research, making it more accessible and effective by providing the necessary resources available at synchrotron facilities.

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Exploring the morphology of the Rhodnius prolixus using SR-micro-CT

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High resolution micro-computed tomography (micro-CT) is a very versatile accurate technique for assessing internal structures of biological and biomedical samples without causing damage to them in comparison with histology [1][2]. Application of micro-CT using phase contrast X-ray imaging has been crucial to investigate the anatomy of insects like Rhodnius prolixus that is responsible to carry the vector of Trypanosoma cruzi, the causative agent of Chagas disease [3][4]. In this work, micro-CT experiment were carried out on SYRMEP beamline at Elettra Sincrotrone (Trieste, Italy) to investigate internal structures of two R. prolixus specimens immersed in ethanol. Operating in whitebeam mode, the beam was filtered with 200μm Silicon resulting in 21 keV mean energy. Using phase-contrast regime by propagation-based imaging, the specimens were positioned 150 mm away from detector, and projections were acquired using two different effective pixel sizes (2 μm and 4 μm) with exposure time of 50 ms and 100 ms, respectively. For this experiment, 1800 and 3600 projections were acquired over an angular range of 180º and 360º. STP software developed by the SYRMEP team was used to reconstruct the data scans in which was applied the Transport of Intensity Equation (TIE-Hom) as a phase retrieval method [5] and the Filtered Backprojection (FBP) algorithm. Micro-CT images obtained on the Elettra synchrotron revealed regions of interest in the structure of R. prolixus that are fundamental to understanding its development process, such as its brain and protocerebrum, endo and exo skeletal features, muscles, digestive system. In addition, the results showed that it was possible to observe regions of soft tissue even without using staining methods. For future studies, MOGNO beamline, located at the new Sirius synchrotron light source (Campinas, Brazil) can strongly contribute to the study of zoom tomography in particular regions of the brain responsible for its development.

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Exploring the nanophotonics and the vibrational strong-coupling ability of mid-infrared surface plasmon-polaritons in the polar two-dimensional crystals/gold interface

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Surface plasmon-polaritons (SPP) are quasi-particles created by the coupling of photons to free charge carries [1]. Due to being surface modes with evanescent behavior in the normal direction from the metallic surface, SPP are intrinsically two-dimensional (2D) modes of high importance to nanophotonics for a variety 2D-defined problems [2]. In the mid-infrared (mid-IR) window, SPPs can be used to interrogate interface states associated to molecular vibrations, electronic transitions in the 50 – 300 meV energy range and room temperature thermodynamical reactions involving 2D and/or low-dimensional materials interfacing metal surfaces [3]. To extract such information from mid-IR SPPs, in analogy to information transport from other forms of electromagnetic radiation, it is critically to fully characterize their propagative properties in the near-field zone. We present here the real-space, nanoscale resolved imaging/spectroscopy SPP waves in asymmetric insulator/metal/insulator (IMI) heterostructures by using synchrotron infrared nanospectroscopy and laser-illuminated scattering-type scanning near field optical microscopy. We show that SPP propagation lengths can exceed 20 μm at room temperature in the 750-1450 cm-1 mid-IR range with group velocities reaching 20 % of the light velocity in vacuum and 0.2 – 0.4 ps lifetimes. Moreover, we demonstrate that the SPP waves can interact with polar crystals phonons in the strong coupling regime, a feature that can be further utilized to exploit detection of molecular vibrational systems in the mid-IR.

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FEASIBILITY STUDY OF EVALUATING THE EFFECT OF LOSARTAN ON THE AORTIC ARTERIES OF RATS USING CARNAÚBA XRF LINE

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The treatment and management of malignancies have improved over the last few years, resulting in greater long-term patient survival. Great efforts have been applied to cancer therapy and have been resulted in a growing number of cancer survivors. Radiotherapy-induced vascular disease is emerging as a current cancer treatment consequence. Intimal hyperplasia and accelerated development of atherosclerotic plaques are some examples that can be caused by some types of chronic inflammatory reactions. In this proposal, we used CARNAÚBA (Coherent X-Ray NAnoprobe BeAmline) X-ray nanoprobe beamline at the Sirius storage ring to do elemental maps of aortic arteries of rats. With X-ray fluorescence (XRF), the X-ray emission is sorted by its energy and brings elemental information of the sample. Sirius, the new Brazilian Synchrotron Light Source, is the largest and most complex scientific infrastructure ever built in Brazil and one of the first 4th-generation Synchrotron Light Sources to start operation worldwide. The purpose was to compare aortic arteries from healthy rats with a rat group thats was irradiated with a thoraxic radioteraphy and with another group of irradiated rats treated with losartan medicine. With this purpose, eighteen healthy Wistar rats, with eight weeks old, were divided into three groups: control group (C); irradiated group (IR), wich represents a group that received irradiation; and a group that received irradiation and then it was treated with losartan medicine (IRL). Our samples consist of, after two months of treatment, the entire heart of the rat was removed and cleaned with sterile saline and the aortic artery was removed, cutted and fixed (in 10% neutral buffered formalin solution). Then, it was embedded in Paraplast Plus (Sigma-Aldrich, St. Louis, MO, USA) and processed for optical microscopy. It is worth mentioning that Control rats were submitted to the same procedure, but were not irradiated. At CARNAÚBA beamline, the energy used was 9.725 keV, it is the maximum energy that permits to acess calcium and zinc elements. Through nanoprobes, the sample is scanned and two-dimensional maps of all the elements present in the sample are provided, with a field of view of 50 μm x 50 μm, and with a step of 0,5 μm. These measurements of X-ray fluorescence of aortic artery of rats will provide detail of the chemical information of the effects of radiation-induced and the beneficial effect of losartan in this artery. It will be possible to verify potential use of losartan in reducing, or even in preventing, the radiation-induced damage that can be caused by thoracic radiotherapy.

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FeOAuCo nanocomposite synthed by thermal combustion

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The nanocomposite (FeOAuCo) was obtained by combustion synthesis [1] in a digital muffle furnace (JUNG J200) with temperature control and stable atmosphere. Ferric chloride (FeCl3.6H2O) P.A., Cobalt chloride CoCl2.6H2O) P.A., gold chloride (HAuCl4.3H2O) P.A., and oxalic acid catalyst fuel (C2H2O4.2H2O) P.A. were used. The doughs were made in the ratio of 2: 0.02: 1: 3.3 respectively. After being homogenized in a mortar, they were inserted into a crucible and underwent the catalyst activation process, initially at a temperature of 120°C for 20 minutes four times. After this step, calcination was performed at 150°C once for 40 minutes, 180°C once for 10 minutes and at 200°C for 10 minutes. Samples after synthesis were characterized by XRD, EDS, FTIR, UV-Vis-NIR, TEM and scanning microscopy. Optical microscopy observations were also performed. The XRD measurements showed that the structure (FeOAuCo) was evidenced using the Rietveld refinement method. The fluorescence measurements showed the atomic constituents with their respective energies for Fe, O, Au and Co. FTIR measurements showed the presence of metallic compounds in the low wavenumber region. The UV-Vis-NIR measurements showed that the GAP energies present an energy around 4.3 eV. The TEM measurements showed the morphology of the sample and after making the lognormal adjustments of the diameter distribution, it showed a mean diameter of 25 nm. Scanning microscopy showed that the morphology of the nanoparticles has spheroid surfaces.

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Fricke dosimetry in synchrotron light source

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CARNAÚBA (Coherent X-rAy NAnoprobe BeAmline) presents itself as an interesting beamline for the beginning of a dosimetry proposal. Fricke dosimetry is a chemical dosimetry, based on the oxidation of ferrous ions (Fe2) to ferric ions (Fe3) present in the solution, through interaction with ionizing radiation. Through the interaction of radiation with the dosimeter, water radiolysis occurs, and the products are oxidizing agents, which makes the Fe3 concentration proportional to the radiation dose. The increase in ferric ion concentration is measured spectrophotometrically at a wavelength of 304 nm [1]. The Fricke dosimeter is 96% by weight water, so its dosimetric properties are very similar to water. This dosimeter can be used in a dose range of 5 to 400 Gy, and for dose rates of up to 106 Gy/s and can be applied to photon and electron beams [2]. Due to these properties, the Fricke dosimeter has proven to be an effective method to overcome clinical and laboratory dosimetry difficulties that have previously been well resolved, mainly with the use of diodes and other dosimeters [3]. In order to assist dosimetry in Brazil, the Radiological Sciences Laboratory (LCR/UERJ) and LabFismed (IF/DFAT/UERJ) have been implementing the Fricke dosimeter and a computational infrastructure capable of data analysis with the help of research projects. The solution is produced and read in the LCR and all data analysis is performed in LabFismed. The CARNAÚBA Beamline covers the energy range from 2.05 to 15 keV and includes different techniques with different types of samples, largely biological, characterization based on absorption, scattering and emission of X-rays. Therefore, it is important to verify the dose to evaluate possible damage to the sample and this work hopes to apply the Fricke dosimeter to this radiation field, thus obtaining an absolute absorbed dose in water [4], [5].

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High-pressure and High-temperature Fluid Flow system in the MOGNO Beamline for Time-Resolved Tomography

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The fourth-generation synchrotron facility provides X-rays from low to high energy with a high flux of photons that, coupled with advanced detector technology, allow routine acquisition of high-resolution tomograms in a few seconds. In addition to high-throughput experiments, in this case, computed tomography can also be resolved in time, which is the 4D CT scan. Among the wide range of interesting physical phenomena to be solved in time by three-dimensional images, the fluid flow in porous materials is one that is present in several areas, such as oil industry, agriculture, and environmental science. In particular, the flow of fluids in very deep reservoirs, where the porous material is under very high pressure (HP) and, normally, also at high temperatures (HT), is an important scientific case. With that in mind, the MOGNO group, in partnership with the energy company Equinor and Petrobras, is installing an HPHT Fluid Flow system at the beamline. This work aims to show the scientific community the system to be installed, promote discussions about the device and future experiments, such as time-resolved fluid flow in porous media, and take this perspective to other areas of knowledge.

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High-resolution XRF for assessing the distribution and association of mineral nutrients in organo-mineral fertilisers

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Brazilian agriculture accounts for ca. 8% of the world's food production, and is responsible for feeding 600-800 million people[1]. Current agricultural practices rely on mineral phosphate fertilisers, which have high solubility and low efficiency in tropical soils, as a major fraction of the P released into the soils is fixed by Fe and Al oxides, thereby compromising its availability to plants[2]. In this scenario, the use of organo-mineral fertilisers, which are expected to exhibit a gradual release of several nutrients into the soil solution, is claimed as an alternative to boost the efficiency of P-based fertilisers[3]. Nevertheless, the association between mineral nutrients within these fertiliser granules has not been properly understood. Herein, we explored the nanoprobe XRF facility of the Tarumã endstation from the Carnaúba beamline to assess the distribution of P and its correlation with other plant mineral nutrients in cross-sectioned pellets of organo-mineral fertilisers produced from sewage sludge and tree pruning combined with either soluble (monoammonium phosphate, MAP) or insoluble phosphate sources (AshDec®, originating from the thermochemical treatment of incinerated sewage sludge ash). High-resolution XRF maps were obtained within 400 × 400 μm areas with a 2-μm lateral resolution and indicated a heterogeneous distribution of P across the fertiliser pellet cross-sections. Furthermore, it revealed a positive association between P and K, Ca, and Fe distributions, which could not be observed in benchtop systems. As the spatial localisation of the nutrients in the fertiliser pellet might affect their interaction throughout its solubilisation, this space-resolved characterisation might shed light on the diffusion dynamics of these nutrients into the granulosphere, i.e., the interface between the organo-mineral pellets and the soil, and ultimately, has the potential to contribute to the development of more efficient alternatives to P supply.

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Implementation of Cone-Beam Zoom Tomography Data Completion reconstruction algorithm

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Three dimensional Cone-beam Computed Tomography, compared with the traditional 2D CT, using a synchrotron beamline to achieve X-ray micron-resolution, can provide high resolution sample data, better image constrast, spatial and temporal resolution. Aiming to increase spatial resolution, we zoom into the object and increase the scanner's magnification. The distance of the focal spot to the center of the sample can be decreased to achieve higher resolution and localy image specific features within the sample. Here, we have developed and applied direct three-dimensional reconstruction algorithms in the context of local tomography, for mathematical phantons and experimental data aquired at the MOGNO beamline at SIRIUS - LNLS. Firstly, an overview scan with low spatial resolution and a large field of measurement (FOM) is performed. Sencondly, a high-resolution scan is performed, in which the scanner's magnification is changed, such that the FOM matches the region of interest (ROI) at the cost of truncated projection data. The data completion method provides a projection correction in which line integrals must be measured for a given ROI and also provides a backprojection-filtration reconstruction algorithm that averts the truncation artifacts that typically get in the way of filtered backprojection reconstructions from truncated data. This is demonstrated through simulation studies, with mathematical phantons and with real synchrotron-based micro CT data aquired at the MOGNO beamline. We conclude that applying the data completition method in the data reconstruction pipeline improve the data quality, feature contrast and enable electronic density values, pores and material segmentation analysis better. Thus, to apply this reconstruction method help to get better understanding in geological, chemical, and physical properties of the material under analysis by comprehending their morphology.

Improvement of submicrometer resolution computed tomography for anatomical study of biological specimens(D. melanogaster brain volume)

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Synchrotron light is electromagnetic radiation emitted when particles accelerate to very high speeds, in the range of the speed of light, with the action of intense magnetic fields with their trajectory deviated. It generates an extremely intense beam of X-rays, collimated and with a high degree of spatial coherence, high level of polarization, coherence and brightness, with low emissivity and tunable, allowing the X-ray phase contrast (PhC) imaging state. These characteristics are perfect for obtaining microtomography [1]. With experimental technique in micro-CT beamlines: previously at IMX/UVX and currently at MOGNO/Sirius and SYRMEP/Elettra.In this research, the goal is on analyzing and improving images of soft tissue samples from small biological specimens. Using microcomputed tomography (micro-CT) as a technique, it provides a map of both internally connected tissues and external tissues of these inspected samples. The sample used in the research was Drosophila melanogaster, which is of great importance for basic research. D. melanogaster is a good model used in scientific research due to its short life cycle and well-characterized DNA. This work analyzes the current uses of flies by applying micro-CT imaging to investigate the responses of amyloid-β protein (Abeta) concentration in cells of the nervous system. This protein is naturally produced in the brain but in people with Alzheimer's, it accumulates to form plaques that cause an inflammatory action. The objective is to understand how the presence of this protein can influence the fly's brain volume[2].Segmentation is of great importance for morphometrics, which studies and measures shapes and dimensions. It is essential for understanding the brain region and consequently carrying out quantitative analyses.

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Influence of solvent and methylammonium chloride on 2D and quasi-2D Br-rich perovskites for solar cells

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2D perovskites have become a popular choice for perovskite solar cells because of their enhanced stability. This type of material has a structure in which n layers of inorganic [PbX6]4- (X = Br- or I-) octahedra are separated by two layers of large organic spacer cations. Changing the number of layers between the spacers drastically changes the perovskite optoelectronic properties. However, there is competition between 2D and 3D perovskite crystallization, so the control of phase purity is quite challenging and not yet fully understood. [1], [2] For this, we investigated the formation and properties of the perovskite (PEA)2FAn-1Pbn(Br1/3I2/3)3n-1 (PEA = C6H5CH2CH2NH3 and FA = CH(NH2)2) (n = 1, 5, 10) with two types of co-solvents DMSO ((CH3)2SO) and NMP (OC4H6NCH3) together with the addition of MACl (MA = CH3NH3). Grazing-incidence wide-angle X-ray scattering measurements were performed on the Microdiffraction beamline of the Advanced Light Source (Berkeley-CA) during the spin-coating and thermal annealing steps of the films. The data showed that the co-solvent strongly alters the formation mechanism, with DMSO leading to the formation of more crystalline and oriented perovskite. The addition of MACl drastically changes the crystal orientation for all compositions. Photoluminescence spectra showed that the lateral heterogeneity in the distribution of phases n is reduced when MACl is used. The characterization of the horizontal distribution of halides through nano-X-ray fluorescence carried out on the Carnaúba/Sirius beamline showed that n=1 perovskite films are quite heterogeneous, while for n>5 occurs the halide homogenization with MACl. Our results indicated the strong dependence of the formation and consequent properties of the films on the co-solvent used as well as the use of the MACl additive, which will allow a greater understanding and facilitate the obtaining of quasi-2D perovskites films with improved crystallinity, phase purity and optical properties.

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Initial Advances towards the Preparation of a Solid Oxide Electrolysis Cell based on $Ba_{0.5}Sr_{0.5}Fe_{0.8}Cu_{0.2}O_{3-\delta}$

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Green hydrogen, obtained through water electrolysis using electric energy from renewable sources, is currently the only viable alternative for use as an energy carrier, replacing carbon-based fuels. One type of system developed for water electrolysis to generate green hydrogen is the Solid Oxide Electrolysis Cell (SOEC), composed of a solid electrolyte made of an oxide that conducts either oxygen ions (O2-) or protons (H), connected to two solid electrodes. The oxides used as electrodes must exhibit mixed ionic-electronic conductivity and serve as catalysts for the reactions occurring within them, whether it's the electro-decomposition of water to form H2 at the fuel electrode or the oxidation of oxide anions to form O2 at the oxygen electrode.

At Cryssmat-Lab/DETEMA, a combustion synthesis method has been developed to lower the material preparation temperature for Solid Oxide Cells [1,2]. This enables reducing the assembly temperature of the materials to form the cell, improving the microstructure of the electrodes - a crucial characteristic for their activity within the cell. These materials have been applied to the preparation of Solid Oxide Fuel Cells (SOFC) [3], which are similar to SOEC but operate in reverse (reducing O2 at one electrode to form water from hydrogen at the other, generating electric energy). This will allow testing the same SOFC materials or variations thereof for SOEC construction.

In this work, we present the initial steps of synthesis, structural and electrochemical characterization of a potential material for the oxygen electrode of a SOEC. Among the materials studied this work presents the perovskite Ba0.5Sr0.5Fe0.8Cu0.2O3-δ (BSFCu). Structural characterization using powder X-ray diffraction was performed using 19.5 keV radiation at the PAINEIRA beamline of Sirius (LNLS, Campinas, Brazil) confirming the obtention of the desired structure and microstructure.

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In situ analysis of the bio-nano interface formed by silica nanoparticles and protein media using X-Ray Photon Correlation Spectroscopy

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Silica nanoparticles (SiO2NPs) have shown great promise in the field of nanomedicine, being attractive for various biomedical applications such as diagnostic and therapeutics [1,2]. To harness their full potential for real-world applications, it is imperative to thoroughly characterize these systems in the biological environments, where they will be used. Traditionally, this task is performed by dynamic light scattering (DLS) in very dilute standardized environments, such as in aqueous and buffered media. However, for NPs embedded in complex biological environments (e.g. blood), the application of DLS becomes unfeasible due to the lack of transparency of these media to visible light. In such scenarios, X-Ray Photon Correlation Spectroscopy (XPCS) is a technique fully capable to provide information about the dynamics of the SiO2NPs in biological media and derive information on hydrodynamic diameter, among other parameters. The assessment of data derived from X-ray Photon Correlation Spectroscopy (XPCS) enables the monitoring of both protein adsorption and nanoparticle aggregation processes [3]. Aiming to study SiO2NPs in environments closely resembling biological ones, we conducted XPCS experiments at the Cateretê beamline with a set of bare-SiO2NPs and SiO2NPs-PEG (diameters ranging 200-600 nm, 10 mg/mL) in different media: water, phosphate buffer saline (PBS 100 mM), bovine serum albumin (BSA 5 mg/mL in PBS 100 mM) and fetal serum bovine (FBS 10% in PBS 100 mM). It was observed for all the bare-SiO2NPs an increasing in the relaxation time with the presence of protein media (BSA and FBS), due to the adsorption of protein and/or nanoparticle aggregation. On the other hand, for SiO2NPs-PEG, the relaxation time did not change regardless the media which they are dispersed. These results confirmed that XPCS can provide information of protein corona formation onto and aggregation of SiO2NPs in biological media and the impact of different functionalization.

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In situ experiment to study the structural contribution on the mechanical properties on ceramic composites

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Ceramic materials reinforced with graphene have demonstrated significant improvements in properties such as hardness and fracture toughness [1,2]. These gains are multifactorial, with some studies providing more consistent and easily explainable results than others. Among the observed factors, the diffusion of reinforcing material into the ceramic matrix has been infrequently documented, with the materials being considered as inert to each other [1,2], although it does occur [3], and this study aims to investigate its impact on mechanical properties. To do so, three samples with incremental amounts of multilayer graphene were produced to exhibit incremental carbon diffusion, resulting in phase transitions and an increase in lattice parameters. To investigate how diffusion impacted the rigidity of each phase, we employed a system that allowed for in-situ stress application during X-ray diffraction experiments, enabling a comparative analysis of deformation and microdeformation of each crystalline structure.

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Larvicidal formulation containing N-tosylindole: A viable alternative to chemical control of Aedes aegypti

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Aedes aegypti represents a significant challenge in current public health. This mosquito is responsible for the transmission of infectious diseases that have sparked epidemics on a global scale. Surfactant-stabilized systems, such as microemulsions, liquid-crystalline precursors, and liquid crystals, show promise as sustained delivery methods for both hydrophilic and hydrophobic substances. These systems serve as biocompatible, water-soluble reservoirs for N-tosylindole, which exhibits biological activity against Aedes aegypti Linn. (Diptera: Culicidae) larvae. In the ternary diagram, four distinct regions are evident: microemulsion (ME), liquid crystal (LC), emulsion (EM), and phase separation (PS). The use of polarized light microscopy (PLM) and small-angle X-ray scattering (SAXS) revealed the presence of microemulsions, lamellar, and hexagonal phase liquid crystals. Notably, the system exhibited a lower lethal concentration of 50% (LC50 = 0.1 ppm, 0.36 μM) compared to pure N-tosylindole (0.24 ppm, 0.88 μM), which is less effective in aqueous media. Additionally, this formulation demonstrated no toxicity to Artemia sp., a non-target organism. The system displayed remarkable larvicidal activity, offering an alternative to commercial larvicides that have shown resistance and pose environmental risks when used extensively against Ae. aegypti larvae. Furthermore, a two-fold increase in potency was observed.

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*S*cattering *AP*parat*U*s for *C*omplex *A*pplications and *I*n-situ *A*ssays (SAPUCAIA): The SAXS beamline at Sirius.

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At the Brazilian synchrotron Sirius, the SAPUCAIA beamline is a cutting-edge facility created to maximize the potential of 4th generation synchrotron radiation for cutting-edge small angle X-ray scattering research. With its cutting-edge features and technical specifications, the SAPUCAIA beamline enables researchers to investigate a variety of scientific fields, including biology, chemistry, and materials science. In addition to being simple to use and allowing for any time-and-again changes to the experimental setup, SAPUCAIA was envisioned and created to have the best performance (and high reproducibility of the experiments). Operating in the hard X-ray spectrum, SAPUCAIA has an undulator source installed, which produces X-rays with energy ranging from 5 to 17 keV, enabling research with a variety of sample conditions. Users can access various experimental setups while conducting their experiments and a large q-range window can be accessed. Low parasitic scattering, low beam divergence, and excellent optical component stability were used as the foundation for the beamline design, which will make SAPUCAIA one of the most significant beamlines in the world. These features enable the investigation of particles with sizes ranging from a few nanometers to a few micrometers, making SAPUCAIA a flexible instrument for researchers in a wide range of specialized and interdisciplinary fields.

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Magnetic Properties and Local Structural Investigations of Nanoparticles Based on Cobalt, Zinc, and Copper Mixed Ferrites

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Magnetic liquids based on nanoparticles have gained considerable attention, being developed for current worldwide issues, in technological and environmental advances, production of energy in a sustainable manner, diagnosis and treatment of diseases [1, 2]. They consist of dispersions of magnetic nanoparticles in non-magnetic liquid media. One of the challenges is to obtain high performance nanoparticles by finely tuning both their magnetization and magnetic anisotropy [3]. Here, we propose to modulate the chemical composition and the local structure to elaborate nanoparticles with large values of magnetization and anisotropy. Thus, we synthesize bimagnetic nanoparticles with a core of mixed ferrite based on zinc, copper, and cobalt, in different proportions, surrounded by a protective shell of maghemite. Then these core/shell nanoparticles are dispersed in acidic medium. Several techniques are used to crosscheck their characteristics, the chemical composition has been determined by chemical analysis using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) and Energy Dispersive X-Ray Fluorescence Spectroscopy (EDX). This crossed analysis allows us to precisely determine the proportions of core and shell volumes and the stoichiometry of the core materials in all samples investigated here. The crystalline structure and the nanoparticle sizes were studied by using X-Ray Diffraction (XRD). Transmission Electron Microscopy (TEM) pictures show their morphology and allow a mean size determination, which well matches the one deduced from XRD analysis. The local structure was investigated by X-ray photoelectron spectroscopy (XPS) on the Sirius Ipê beamline. An extensive magnetic characterization was carried out using a SQUID magnetometer. The results show that magnetization and anisotropy can be tuned by the core composition and the core/shell architecture.

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Magnetic structure and component-separated transitions of HoNiSi3: experiment and theory

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Microfluídica aplicada a soluções de ultra-alto vácuo em câmaras experimentais

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Este trabalho apresenta uma abordagem inovadora para resolver problemas de instrumentação em sistemas de ultra-alto vácuo, com foco na otimização da geometria de uma micro válvula de Tesla. A manutenção de ambientes de ultra-alto vácuo e livres quebra de vácuo é fundamental em diversas aplicações principalmente científicas e em alguns casos industriais, como análise de amostras sob vácuo utilizanndo ultra violeta de vácuo (UVX) ou raio X moles e em análise de superfícies limpas. Também em casos de bombeamento diferencial. Para atingir esse objetivo, é crucial controlar o fluxo de ar de forma eficiente. Neste estudo, o gradiente de pressão é mantido fixo, com pressão atmosférica em uma das saídas da válvula e 10^-9 mbar na câmara de vácuo. Para otimizar a geometria da micro válvula de Tesla, utilizamos o software open-source OpenFOAM de fluidodinâmica computacional para simular o comportamento do escoamento em diversas geometrias. Em seguida, empregamos técnicas de machine learning, especificamente redes neurais, para extrair padrões de comportamento do escoamento para diferentes geometrias da válvula. Além disso, implementamos um algoritmo genético para encontrar a geometria ótima da micro válvula de Tesla que maximize a eficiência do controle de fluxo de ar, mantendo o ambiente de vácuo sob pressão de alto vácuo. Essa abordagem multidisciplinar oferece uma contribuição significativa para a resolução de problemas de instrumentação em câmaras de vácuo, com potencial para melhorar a eficiência em uma ampla gama de aplicações científicas e industriais.

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Micrometeorological Station for Precision Agriculture

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Precision agriculture has advanced considerably in different spheres of knowledge. However, it continues to face challenges due to limited exploration in certain areas, which makes it difficult to obtain immediate responses from crops. This is largely due to the lack of availability of micrometeorological data, as well as the lack of analysis within the agricultural context. This study proposes the development of an economically viable micrometeorological/agrometeorological station, capable of measuring and providing information on variables related to agrometeorology. The main objective is to help understand the responses of grain crops in areas practicing precision agriculture. This plays a crucial role in understanding and explaining plant responses to factors such as solar radiation, air temperature, humidity, wind speed, and so on. The implementation of a micrometeorological station, equipped with a wireless data transmission system and powered by solar energy can be economically challenging to establish a comprehensive network of stations for a specific region. However, this infrastructure is essential for any initiative that seeks to establish solid scientific parameters that can contribute to solving challenges in agriculture. The development proposed in this study aims to offer a technology adapted to the specific needs of producers, aiming to improve crop production in the state of Mato Grosso. This approach represents a significant opportunity to improve the efficiency and sustainability of agriculture, promoting a deeper understanding of the agrometeorological variables that affect agricultural crops in the region.

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Nanoscale morphological and mineralogical study of the mineralization of microbial filamentous structures in Holocene lacustrine carbonates

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Microbialites are organo-sedimentary structures formed after the lithification of microbial mats and correspond to the oldest macroscopic evidence of life on Earth1. Their formation can involve the carbonate organomineralization of microbial biofilms or the inorganic mineralization of biological templates2. Since the distinction of features formed by these mechanisms can be obscured due to diagenetic processes the study of recent microbialites is key for the recognition of biosignatures in the geological record or in extraterrestrial materials. Meter-scale carbonate build-ups formed along the margins of Lago Sarmiento (southern Patagonia – 51°S) are composed by millimetric shrubby structures composed by Mg-calcite formed around micrometric hollow filamentous structures of microbial origin. SEM and transmitted light petrography reveal that these structures are entombed by a Si-bearing amorphous carbonate phase and/or by an unknown type of smectite-like clay mineral. Kerolite [$Mg_3Si_4O_{10}(OH)_2 \cdot H_2O$] and stevensite [$(Ca_{0.09}K_{0.01}Sr_{0.01})_{\sum=0.11}(Mg_{2.84}Fe_{0.02}Al_{0.03})_{\sum=2.89}(Si_{3.98}Al_{0.02}O_{10})(OH)_2 \cdot nH_2O$] are types of Mg-clay minerals recognized in microbialites formed in several alkaline lakes3,4. While the first has been reported in Lago Quechulac3, a freshwater lake like Lago Sarmiento, the latter are reported in Lake Clifton4, a brackish to saline lake that houses a similar microbial consortium as Lago Sarmiento5. We propose that the Lago Sarmiento's microbialites are world-class geological materials to study the fossilization of microbial organisms and their diagenetic alterations by using synchrotron-based ptychographic X-ray computed tomography. By comparing the expected results of this research with known locations such as Lago Quechulac and Lake Clifton we can improve our knowledge on the interplay between physicochemical and biological factors influencing the mineralogy and morphology of microbialites and their significance for paleoenvironmental interpretation.

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Ni rich NiTi Alloy Thermomechanical Behavior Under Compression at High Temperature

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NiTi alloys are well known for their unique functional properties, such as shape memory effect and superelasticity. These properties make it highly suitable for various applications, including engineering and biomedical devices. These alloys exhibit notable characteristics, such as high corrosion resistance, biocompatibility, damping capacity and elastic recovery. Such properties are acquired through complex thermomechanical processes, inducing martensitic and reverse phase transformations, whether in a single step or in multiple steps, allowing adjustments in phase transformation temperatures and chemical composition, which must be rigorously controlled, because small changes in these factors influence the functional characteristics.

Raw ingots do not have much ductility and do not exhibit shape memory and superelasticity effects. Therefore, it is necessary to submit these ingots to a hot working process, in order to improve mechanical and functional properties, obtained through compositional and microstructural homogenization, as well as the desired texture.

The microstructures produced from hot working operations requires strict control of temperature and stress. This enables grains refinement, the preservation of shape memory and superelasticity effects, increasing the ductility and obtaining geometries closer to the target geometry. In this study, a physical simulation was carried out to evaluate the microstructural modifications of the remelted and annealed Ni rich NiTi alloy. The sample was compressed with a strain rate (in the order of 10-1 s-1) at 850 ° C, interspersed with cooling at 300 °C, using x-ray diffraction analysis in situ. The tests were carried out on a Gleeble® thermomechanical simulator (XTMS), at the XRD1 beamline, in the Brazilian National Synchrotron Light (LNLS - CNPEM), Campinas- Brazil.

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Novel methodology to determine thermal properties of nanoparticles exclusively based on SAXS measurements applied to Bi nanocrystals and nanodroplets in a glass matrix

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Operando studies of gas sensors using Near Ambient Pressure (NAP) XPS: correlation between morphology and electronic structure with the sensing property of ZnO during exposure to O2 and CO gases at operating temperature.

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Today's gas sensors have received significant attention because of their essential applications in numerous areas, such as environmental monitoring in industry and residential areas, disease diagnosis, agriculture, industrial wastes, food quality monitoring, etc [1,2]. Resistive gas sensors based on semiconductor metal oxides (SMO) in thin-film materials are the most used and studied devices to control toxic and flammable gases. To thoroughly understand the mechanism of gas detection, it is essential to characterize the surface composition and the material electronic structure during the exposure of the material to target gases. Near-ambient pressure X-ray photoelectron spectroscopy (NAP-XPS) provides an option for in-situ analysis under realistic operation conditions relevant to semiconducting metal oxide (SMO) based chemical gas sensors combined with high surface sensitivity and quantitative analysis capability [3]. NAP-XPS experiments of ZnO samples were carried out in the CIRCE beamline (ALBA), exposing the material to different O2, CO, and humidity and heating the sample at temperatures ranging from 25 to 300oC. The electrical resistance of the sample was also measured during the sample exposition to the gases, and the XPS analysis was carried out when the resistance was stable after exposing the material to O2 and CO gases. The correlation between the electronic structure and sensing properties of the ZnO sample was carried out by analyzing the O 1s and Zn 2p electronic levels when the sample was heated to 300oC and exposed to O2 and CO gases, with and without moisture. The XPS data analysis shows that exposure to CO gas and moisture affects the oxygen species on the sensor surface. In contrast, the Zn 2p level is unaffected by any sample condition.

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Operando Studies on Cathodic Active Materials in Li-Ion Cells: Effect of Chemical Heterogeneities on Electrochemical Performance

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Li-ion cells are inherently electrically isolated systems, which presents challenges when attempting to quantify the uniformity of their properties, including chemical composition, current distribution, charge levels, and temperature, particularly under real operating conditions (in operando). The performance and stability of active cathodic materials in Li-ion cells are significantly influenced by both chemical and structural homogeneity at microscopic scales [1]. Currently, only a limited number of methodologies are capable of assessing cell homogeneity during actual operation. Consequently, there is a relatively small body of literature on real cell studies, as the more common practice involves disassembling cells for ex situ or post-mortem characterizations, which may not always accurately represent the cathode's original state during the charging or discharging process. In this contribution, results obtained on Ni and Mn based Li-enriched layered materials (Li-rich) are presented, exploring the chemical and structural nature of the cathode for different states of charge. The possibility of directly tracking the material's characteristics with spatial resolution allows for the specific visualization of the instability in terms of its chemistry and the determination of the involvement of different elements under different charge conditions, which is not possible to ascertain through electrochemical or general ex situ characterizations. Preliminary results from our experiments indicate the high instability of Mn at high potentials in Li-rich samples and provide the first direct evidence of Ni involvement in low-potential regions, which had not been manifested through previous electrochemical characterizations.

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Operando Synchrotron X-ray Diffraction and X-ray Absorption Spectroscopy Studies of $LiCoO_2$ in Aqueous Supercapacitor

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Supercapacitors are ultrafast energy storage devices based on carbon materials such as active carbon, graphene, and carbon nanotubes. To increase the capacitance in supercapacitors, faradaic reactions (redox processes similar to those on batteries) are added on the electrodes, transforming those devices on pseudocapacitors [1]. Those faradaic reactions could be obtained with metal oxides such as LiCoO2 [2,3], which allow ultrafast electron transfer on electrode/electrolyte interfaces [4,5]. The ability of those devices to store energy effectively and efficiently is associated to the chemistry and structure of the electrode and electrodes/electrolytes interfaces [6-8]. This work consists in understanding the structural and electronic changes during the operation process of charge/discharge of the supercapacitors using operando XRD & XAS. Supercapacitor was developed using a coin cell with a Kapton window that allow incoming/outcoming X-rays. The cells were composed of two identical electrodes (symmetric configuration) containing multi-walled carbon nanotubes (25 wt%) and synthesized LiCoO2 (75 wt%) as a composite buckypaper self-supported and filled with a 1.0 M Li2SO4 aqueous solution. The LiCoO2 was analyzed by X-ray diffraction with operando measurements on the DanMAX beamline at MAX IV Laboratory and the data were refined by the Rietveld Method with the GSAS-II software. The structural changes of LiCoO2 during cell cycling showed an increase in parameters c during charge and a decrease in parameters a and b during discharge, this phenomenon is associated with the process of oxidation and reduction of Co. To evaluate this process, XANES measurements were carried out in the Sirius.

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Pressure-induced structural transition in $Sr_2Ir_{0.95}Co_{0.05}O_4$

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Iridates present an array of fascinating physical phenomena such as Kitaev magnetism, spin liquid phases, and superconductivity due to the presence of a strong spin-orbit coupling together with considerable Coulomb interactions, crystalline electric field, and orbital hybridization [1,2]. In particular, Sr2IrO4 is a spin-orbit coupling assisted Mott Insulator that has a noncollinear magnetic structure below TN∼240K as a result of a substantial Dzyaloshinskii-Moriya-type antisymmetric exchange interaction [3, 4]. It also presents exotic states under external pressures, while still maintaining the nonmetallic state up to at least 185 GPa [5, 6]. However, when doped with 5% of Co it effectively presents a metal-insulator quantum critical point with remarkably heavy quantum critical fluctuations, while preserving the magnetic order [7]. Still, there are no investigations about this compound at high pressures, which was shown to be a crucial external parameter in tuning the electronic properties of this kind of material [4]. In this work, we intend to fill this gap by measuring Sr2Ir0.95Co0.05O4 (SICO) under pressure. X-ray Powder Diffraction (XRD) experiments were conducted up to 70 GPa at the EMA beamline of the Brazilian Synchrotron Light Laboratory. Our results show that SICO exhibits similar anomalies as the undoped iridate, such as a structural phase transition at around 55 GPa with phase coexistence up to about 65 GPa. This transition occurs at pressures about 10 GPa higher than in the pure compound, even though both unit cells remain quite similar at lower pressures, a fact that warrants further investigation. A tentative characterization of the new structure is proposed.

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Recent developments to the high-throughput SAXS beamline B21 at Diamond Light Source, UK.

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Since 2012 the B21 high-throughput SAXS beamline has largely focussed on serving the structural biology community providing automation for delivering liquid samples either directly or via an in-line HPLC system to a SAXS instrument that is optimised for measuring protein samples in the range from peptides up to particles the size of ribosomes (1). With a user community that is largely focussed on the health sciences it is becoming more important to be able to tackle drug delivery vectors such as cubic phase lipids or hydrogels as well as synthetic particles such as the encapsulated RNA particles that are now important vaccines and composite particles such as functionalised nanoparticles.

To facilitate this change in use the beamline is developing capabilities for measuring solid and viscous samples (2) for illuminating photoactive samples with UV-VIS light in-situ during SAXS measurements, for measuring simultaneous SAXS and WAXS and for increasing the sample temperature range and rate of change. In this poster I will describe these upgrade projects and give some examples of the science that they have enabled.

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Single-phase Navier-Stokes Methodology for Quantification of Petrophysical Properties from synchrotron-based X-ray microtomography imaging of Pre-Salt Reservoir Rocks

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In this study, we present an innovative methodology for the precise quantification of petrophysical properties of pre-salt reservoir rock samples based on synchrotron hard X-rays microtomography imaging. We leverage reconstructed and segmented 3D volumes obtained through imaging experiments conducted at the MOGNO beamline of the 4th generation Sirius synchrotron to define computational domains essential for Single-phase Navier-Stokes incompressible fluid flow simulations. Our approach employs the Projection Method and Finite Volumes discretization to effectively approximate the governing Navier-Stokes partial differential equations by decoupling velocity and pressure fields through Helmholtz-Hodge decomposition. This enables us to quantitatively assess critical petrophysical properties, such as effective permeability and hydraulic conductivity, and to perform simulations of particle flow. We validate our method by comparing its performance and quantitative results against a well-established reference software, Avizo. The presented preliminary results will be used to advance the understanding and characterization of pre-salt reservoir rocks, offering valuable insights for the porous media research within synchrotron facilities. Moreover, the proposed algorithm is flexible regarding the addition of new conditions to the mathematical model, such as source terms to deal with external forces acting on the flow or different boundary conditions, yielding a powerful tool that could be easily applied to other cases of fluid flow through porous materials.

Site selective electronic exploration of labile complexes

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The study of electronically labile complexes (ELC) has gained attention due to their potential in sensors and devices. These materials exhibit varying electronic properties under external stimuli [1]. Valence and spin states of ELCs are usually determined indirectly using X-ray diffraction (XRD) and X-ray absorption near edge structure (XANES) techniques[1]. However, these techniques can't provide direct electronic information for different atoms of the same element in the structure. Diffraction Anomalous Fine Structure (DAFS) experiments combine XRD's structural data with spectroscopic information by analyzing specific Bragg peaks' (h) intensity changes in energy varying experiments[2]. DAFS allows retrieval of valence and spin states information, provided the phase problem in resonant scattering is solved. In this regime, atom-electron bonding must be accounted, leading to the atomic structure form factor f(h) being divided into three parts: $f(h,E) = f_0(h)\ f'(E)\ if''(E)$, where E is energy, f_0 is the Thompson scattering factor, f' relates to resonance, and f'' is associated with absorption [3]. Meurer et al. [3] recently implemented the olex.refine algorithm within Olex2 software, allowing independent refinement of f' and f'' values. They demonstrated the algorithm's success in modeling the Mo(CO)6 complex when considering anomalous dispersion alongside the non-spherical atoms model [3]. DAFS proves a powerful method for studying structures with multiple metallic ions in different valence states, as seen in Co(o-dioxolene)2(pyridine)2. This crystal houses two independent Co ions exhibiting diverse pathways during valence tautomerism interconversion. Our goal is to apply DAFS to Co(o-dioxolene)2(pyridine)2, using recent techniques to analyze each Cobalt atom's behavior within the structure.

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SrMnZnO nanocomposite synthed by thermal combustion

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The nanocomposite (SrMnZnO) was obtained by combustion synthesis [1] in a digital muffle furnace (JUNG J200) with temperature control and stable atmosphere. The reagents were strontium chloride (SrCl2.6H2O) P.A., potassium permanganate (KMnO4) P.A., zinc sulfate (ZnSO4.7H2O) P.A., and the catalyst fuel oxalic acid (CH4N2O) P.A. The doughs were made in the ratio of 1:1:1:3 respectively. After being homogenized in a mortar, they were inserted into a crucible and underwent the catalyst activation process, initially at a temperature of 100°C for 40 minutes four times. After this step, calcination was performed at 150°C, 180°C and 200°C once for 10 minutes and at 220°C for 5 minutes. Samples after synthesis were characterized by XRD, EDS, FTIR, UV-Vis-NIR, TEM and scanning microscopy. Optical microscopy observations were also performed. The XRD measurements showed that the structure (SrMnZnO) was evidenced using the Rietveld refinement method. The fluorescence measurements showed the atomic constituents with their respective energies for Sr, O, Mn and Zn. FTIR measurements showed the presence of metallic compounds in the low wavenumber region. The UV-Vis-NIR measurements showed that the GAP energies present energy around 2.9 eV. The TEM measurements showed the morphology of the sample and, after making the lognormal adjustments of the diameter distribution, it showed a mean diameter of 18 nm. Scanning microscopy showed that the morphology of the nanoparticles has spheroid surfaces.

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Strategies for high-resolution XRF analysis of plant materials: a study case from the Carnaúba beamline

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X-ray fluorescence spectroscopy (XRF) is an important technique for exploring the composition and distribution of elements in plant materials[1]. The Carnaúba beamline of the Brazilian Synchrotron Light Laboratory provides unique features for assessing the distribution of nutrients in plant tissues at the subcellular level[2]. However, the high photon flux intensity and localized nanoprobe X-ray beam pose challenges when analyzing biological materials. Therefore, establishing proper sample preparation strategies is crucial to avoid possible artifacts induced by X-ray exposure[1,3]. Herein, a fresh sample preparation strategy was employed at the nanoprobe Tarumã endstation of the Carnaúba beamline to evaluate the possibility of analyzing plant materials without cryofixation. In this regard, the petioles of soybean (Glycine max) and coffee (Coffea arabica) plants were manually cut using a razor blade, and the resulting ~ 100 μm cross-sections were fixed on a polyamide tape and mounted on 6 × 6 mm plastic frames covered by a 4-μm thick Ultralene® film either containing a 1-μL deionised water droplet added and then directly measured at 10300 eV and 800-nm lateral resolution. The XRF mappings allied to the light microscope assessment revealed that the hydrated samples measured within 240 × 240 μm did not present signals of elemental redistribution or structural changes, whereas those obtained within 400 × 400 μm and 320 × 320 μm exhibited clear dehydration and morphological changes in the analyzed region. This indicates an intricate association between exposure time and sample integrity. It also showed that the thickness of the sample can compromise the quality of the XRF measurements. The results obtained in this study might facilitate simpler and faster sample preparation methodologies for high-resolution synchrotron-based XRF analyses of plant materials.

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STRUCTURAL MODIFICATIONS INDUCED BY HYDROSTATIC PRESSURE ON LEAD METASILICATE PHASES

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Among the silicates, lead silicates stand out because of the possibility of making glass and glass ceramics with a wide range of compositions, which enables a great variety of applications ranging from domestic to high technology. For most of them, the control of the nucleation-growth-crystallization process is essential to determine the final physical properties of the resulting glass-ceramic. Nonetheless, the crystallization of silicates is not a straightforward process, but the system reaches intermediate metastable stages before reaching the most stable conformation. Due to the broad range of applications of the lead silicates and its high sensitivity to in situ structural analysis, the lead metasilicate PbSiO3 presents itself as a model system for the investigation of the structural evolution with pressure and temperature, allowing the evaluation of the thermodynamic quantities involved in this complex process, which are crucial to the physical description of the crystallization of the glasses presenting heterogeneous nucleation. Additionally, from the application point of view, the application of pressures capable of inducing changes in the energy of such phases can be viewed as a potential method to intervene in the stages of the overall crystallization, opening pathways to new properties for the resulting glass-ceramic. In this sense, we report on in situ high-pressure Raman and in-situ X-ray diffraction analyses of stable and metastable phases of PS, crystalized in a monoclinic and a hexagonal structure, respectively. The progressive redshift of the Raman peaks up to 8 GPa indicates that the structures are highly sensitive to the application of hydrostatic pressure. By following the diffraction patterns we confirm the structural evolution induced by pressure, especially the abrupt shift of the main diffraction peaks for $P > 16$GPa, which indicates a dramatic softening of the structure at this pressure regime.

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Synchrotron 3D virtual histology of zooarchaeological materials

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Modern synchrotron radiation (SR) sources have significantly advanced biological imaging (Betz et al., 2007). The application of SR X-ray microtomography has been reported for imaging a broad range of samples, from dense to soft materials. However, hard tissues, such as archaeological materials, often exhibit complex structures down to the subcellular level. In this study, we demonstrate that new archaeological questions can be pursued using synchrotron X-ray microtomography, a unique and effective non-invasive methodology for examining their morphology and histology at a three-dimensional (sub) microscale (Cupello et al., 2022). Measurements were taken during the MOGNO commissioning (proposal 20231815), and the results will unlock new possibilities in the field of Zooarchaeology, shedding light on the potential of this methodology in the study of 3D virtual histology of archaeological samples. Here, we discuss our main results, which include hard tissues like otoliths (small structures found in the inner ear of teleost fish, as described by Disspain et al., 2016). Virtual transverse sections of an archaeological specimen of Micropogonias undulatus imaged during the experiment display annual cycle bands that indicate the age of the fish. The size and age of individuals are important characteristics for estimating changes in human predation behavior and its impact on aquatic ecosystems (Disspain et al., 2016).

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Synchrotron microtomography applied to the morphological studies of *Aedes aegypti*

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Synchrotron microtomography is a powerful technique in the study of internal microstructures with low attenuating without staining then with a contrast medium. A specimen with high sanitary importance to study is the Aedes aegypti insect due to its anility to infect humans with several diseases as dengue, chikungunya yellow fever and zika. Studies about the morphology of Aedes aegypti mosquitoes are still lacking and visualize in detail the internal and external structures are of great importance in vector control, enabling the analysis of potential morphological alterations caused by chemical agents with insecticidal action, such as essential oils. In this regard, this work aims to show the ability of synchrotron microtomography for the visualization of soft tissues and internal microstructures in the aedes aegypti using differents setup available in the Mogno beamline on Sirius Synchrotron. The Mogno is a beamline able to study the sample with zoom-tomography technique in addition to the use of phase cotrast, varying the setup according to the structure to be viewed. Therefore, the work will contribute to advancing our knowledge of morphological studies, and physiological alterations of the Aedes aegypti insect, through the application of advanced computed microtomography techniques.

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Synchrotron Nano-FTIR at Imbuia beamline - Unraveling the Nanoparticle-Bacteria interface

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Nanotechnology has emerged as a promising solution to combat the escalating challenge of antimicrobial resistance (AMR) [1]. However, the current comprehension of the intricate interaction mechanisms between nanoparticles (NPs) and bacteria remains restricted, primarily due to the spatial limitations inherent in traditional surface characterization methods such as Fourier transform infrared (FTIR) and Raman spectroscopies [1-2]. The aim of this work is to understand the surface interaction between Escherichia coli (E. coli) and silica-based NPs (SiO2NPs) coated with carbohydrates, using a cutting-edge approach that combines Scanning Near-field Optical Microscopy (s-SNOM) with broadband synchrotron infrared nanospectroscopy (nano-FTIR). To enhance the interaction between the NPs and the bacterial membrane at the nanoscale, SiO2NPs were coated with the carbohydrates mannose, maltose or trehalose, since Gram-negative bacteria are known to recognize and interact with glycans [3]. Physical-chemical characterization techniques were performed in order to confirm the presence of carbohydrates, and the interaction between NP's and bacteria were studied by biological in vitro assays and scanning electron microscopy. This work presents a unique approach for studying the nano-bio interface using the Nano-FTIR technique, which enables the chemical and morphological analysis of a single cell

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Terahertz nano-optics features of graphene revealed by nanoimaging

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The well-established two-dimensional (2D) material graphene is anticipated to feature extraordinary near-field optical properties in the terahertz (THz) frequency range [1]. However, the experimental interrogation of such phenomena is non-trivial due to the intrinsic dimensionality mismatch between atomically thin graphene and the wavelength of the THz radiation reaching hundreds of micrometers. This task becomes even more challenging in the THz gap (0.1 – 10 THz) wherein powerful light sources are scarce [2]. Nevertheless, modern nanoscopy techniques coupled to unique, highly brilliant THz light sources have been demonstrated to accomplish an experimental investigation of such 2D materials [3]. In this work, we apply a scattering-scanning near-field optical microscope (s-SNOM) illuminated by THz radiation of a tunable free-electron laser (FEL) to investigate the near-field optical response of graphene within the THz gap (1.5 - 6.0 THz). We firstly demonstrate that graphene takes on an optically metallic character at around 2 THz. In this frequency range, the driving THz field enables a quasi-static response associated to the relaxation of the Gr charge carriers. Secondly, we find the graphene to feature a distinct field-enhancement effect (FEE) at 3.8 THz, meaning a significantly increased optical contrast and sharpness of the near-field optical images. Through theoretical analysis based on the polariton dispersion relation and near-field modeling, the FEE can be attributed to a plasmonic resonance of graphene near 3.0 – 6.0 THz. We believe these novel aspects of graphene nanooptics in the THz range to prove valuable for future graphene-device-based studies and technological applications.

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THE DEVELOPMENT OF S4CI: THE SAPUCAIA SOFTWARE SOLUTION FOR SAXS CUSTOMIZED INTERFACE

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The SAXS users' community is already well-established in Brazil. At UVX, the old synchrotron, around 160 proposals were applied per year for using this technique. Many of them, however, were submitted by users who, despite having high-impact research, did not have much experience with SAXS and did not take full advantage of this powerful technique. Aiming to help these users in this task, at SAPUCAIA beamline will be available a software solution that will ease the visualization, simulation, and simple SAXS data analyses. The software is a graphical user interface written in Python. Its main function is helping users to analyze shapes and sizes of particles dispersed in a homogenous medium by using already existent scattering object models. It simulates the scattering intensity using the parameter values controlled by sliders on a graphical interface. Moreover, this tool eases the fit convergence, since the initial guess can be very close to optimal values, which is crucial for non-linear least-squares minimization procedures. An arbitrary number of experimental data files can be imported and adjusted to the models selected by the user, taking as initial guesses the current values of the sliders. It is also possible to link different parameters with mathematical expressions. Data such as the fitted curve, parameter values, and goodness-of-fit statistics for all performed fits are shown and can be saved. We hope that, with the software available at the beamline, even inexperienced users would be able to leave the beamtime with preprocessed data which can be useful not only for publication purposes but also during the beamtime itself, helping the users to decide the directions the experiments will take, making beamtime more fruitful.

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The endolymphatic calcium deposits of Thoropa miliaris (Anura: Cycloramphidae) larvae and their skeletal development accessed through synchrotron microtomography images

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The metamorphic climax in anurans is a period marked by a series of complex morphological changes. Although it is mainly marked by the reabsorption of the tail, and disappearing of several larval traits, it is also the period where several adult features are more noticeable, externally, and features such as cranial, axial and/or appendicular bones begin to ossify. Surprisingly, and less explored by research, the skeletal mineralization process has its onset while the tadpole's mouth parts are degenerating, and the larvae are not feeding. Therefore, one has to assume, that the source of calcium for the bones, may be obtained by a process other than feeding. The answer for this question relates to the ontogeny of a relatively overlooked structure in frogs, the endolymphatic calcium deposits (ECD). Known by frog morphologist since the end of XVIII century, the the ECD is a system described for diverse groups vertebrate and was the focus of several studies distinct research fields, since morphology and ultrastructure until physiology and medicine. Internally, this system of vesicles is filled by a milky substance containing small aragonite crystals (CaCO3). For this study the sample included specimens on nine distinct larval and metamorphic stages of development. The samples are part of the Herpetological collection of the Federal Rural University of Rio de Janeiro, and were returned to the collection after the experimental procedure by tomographic scan. Herein we present an evaluation of using SR-microCT image reconstruction. Measurements were carried out previouly at IMX (proprosals 20160615 and 20180540) and XRD1 (proposal 20180534) facilities, and more recently during the MOGNO commissioning (proposal 20231815). The results will allow us to understand the osteogenesis process between larval and adult stages, such as transformation from ECD to bone tissue.

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The influence of the crystallite size on the phase diagrams of nanomaterials: another important lesson from Aldo Félix Craievich

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When, on April 24th of this year, we received the very sad news about the passing of Dr. Aldo Félix Craievich, a very important era for science in Latin America came to an end. Only a few yaars before, in 2019, the first source of LNLS, UVX, had already ceased to operate after 22 years of receiving users from all over the region. Subsequently, in 2020, Dr. Ricardo Rodrígues, the brilliant builder of that machine (and also of the SIRIUS source), passed away. Dr. Craievich also played a leading role in the construction of the LNLS, serving as its first deputy director and head of the scientific deparment.

In this work, I will review one of the topics that Prof. Aldo Craievich studied intensily throughout his career: the thermodynamic properties of nanomaterials. In particular, many of his investigations focused on illustrating the changes that phase diagrams undergo when reducing crystal sizes to nanometric scales. For instance, together with Prof. Guinther Kellermann, they conducted several studies on the size effect on phase transitions of spherical metallic nanoparticles embedded in a vitreous matrix. In my case, I had the honor of collaborating with Prof. Craievich for approximately 20 years in studying various nanostructured oxides of interest for solid oxide electrochemical cell applications. Through powder X-ray diffraction and EXAFS spectroscopy, we also explored the relationship between crystalline structure and local atomic order. Particularly interesting was the case of the ZrO2-Sc2O3 system, where notable changes in the phase diagram were observed, avoiding phases of low electrochemical performance. These materials garnered much interest recently when they were used in a solid oxide electrolyzer to generate oxygen on Mars from the CO2(g) in its atmosphere. Currently, the next challenge will be to understand the properties of materials under operating conditions, with the help of the beam lines that are under construction in the new SIRIUS source.

Acknowledgements: This work is a tribute to Prof. Aldo Félix Craievich, who did a hard work to establish and strengthen the LNLS user community and was a mentor to many of us. I also thank the LNLS staff who worked tirelessly on UVX during its 22 years of operation. I also appreciate the financial support from the agencies that funded our research over 20 years: MinCyT, CONICET, Agencia IDi, CAPES, CNPq and CLAF.

Thickness-Dependent Ferroelectricity of Few-Layer Germanium Sulfide Observed by Scanning Tunneling Spectroscopy

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Layered materials with van der Waals interaction have great interesting properties to study because new physical phenomena arise from few layers. When we are approaching the 2D limit, certain properties may appear or change. We are interested in seeing how properties can change on the thin thickness of the material, such properties as ferroelectricity [1]. Our study material will be a group IV monochalcogenides, Germanium Sulfide (GeS). This material has a stacking in layers separated by the van der Waals interaction, low toxicity, robustness at room temperature and is a ferroelectric material at low dimensionality [2]. Our work consists of measuring the ferroelectric behavior of the Germanium Sulfide when the 2D limit is approached and was possible to verify using Scanning Tunneling Spectroscopy (STS) a hysteresis curve and a thickness-dependence of the I-V curve. This is the first measurement of ferroelectricity change with the thickness using STS and this work was submitted to Physical Chemistry Letters and resubmitted recently with the referee corrections addressed.

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Three-Dimensional Visualisation of Hierarchically NanoPorous Materials with Coherent X-ray Diffractive Imaging @ the Cateretê Beamline

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The novel 4th generation synchrotron facilities and x-ray free-electron lasers are leading to the development of new X-ray methods of microscopy. Among these techniques, coherent diffractive imaging (CDI) is one of the most promising, enabling nanometre-scale imaging of non-crystallographic samples. Indeed, new visualisation methods can be used to resolve structures at resolutions that were previously unachievable. Here, I will present the application of coherent diffractive imaging and ptychographic X-ray computed tomography for the visualisation of zeolites and polymeric ultrafiltration membranes with a resolution of ~26 nm. Zeolites are an important class of materials, known for their microporous structure and high acidity, widely used in industry as acid catalysts, absorbents, water filtration systems … Thanks to the high-penetration depth of the X-ray beam, we visualised the 3D complex structures in a non-destructive manner and obtain quantitative information about pore size distribution and pore network interconnectivity across the whole membrane wall and of the zeolite. The non-destructive nature of this method, coupled with its ability to image samples without requiring modification or a high vacuum environment, makes it valuable in the fields of porous- and nano-material sciences enabling imaging under different environmental conditions. The method is implemented at the Cateretê beamline [1,2] at the SIRIUS 4th generation synchrotron source based multibend achromat lattice [3].

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Time-resolved synchrotron light source X-ray detection with Low-Gain Avalanche Diodes

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Low Gain Avalanche Diodes (LGADs) represent the state-of-the-art in timing measurements, and will instrument future timing detectors of the High Luminosity Large Hadron Collider (HL-LHC) experiments. While initially conceived as a sensor for charged particles, the intrinsic gain of LGADs makes it possible to detect low energy X-rays with good energy resolution and excellent timing (tens of picoseconds). Using the Stanford Synchrotron Radiation Lightsource (SSRL) at SLAC, several LGADs designs were characterized with energies from 10 to 70 keV. The SSRL provides 10 ps pulsed X-ray bunches separated by 2 ns intervals, and with an energy dispersion $\Delta E/E$ of $10-4$. LGADs from Hamamatsu Photonics (HPK) and Brookhaven National Laboratory (BNL) with different thicknesses ranging from 20 μm to 50 μm and different gain layer designs were read out using fast amplification boards and digitized with a high bandwidth and high sampling rate oscilloscope. PiN devices from HPK and AC-LGADs from BNL were characterized as well. A systematic and detailed characterization of the devices' energy linearity, resolution and timing resolution as a function of X-ray energy was performed for different biasing voltages at room temperature and will be reported in this presentation. The charge collection and multiplication mechanism were simulated using GEANT4 and TCAD Sentaurus, providing an important handle for interpreting the data.

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Ultrasonic Anemometer in the Analysis of Aerosol Dispersion in Precision Agriculture

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This study focuses on the application of ultrasonic anemometers in precision agriculture, with an emphasis on the analysis of aerosol atmospheric dispersion. These devices use piezoelectric transducers to measure wind speed, providing real-time data that is relevant in various agricultural applications.

The ultrasonic anemometer is integrated into autonomous weather stations, allowing for precise wind speed measurements at different heights. This information is crucial for optimizing crop management, such as the dispersion of agrochemicals, adjustment of fertilizer and pesticide application rates, and the prediction of local weather conditions. In this study, we present the integration of the ultrasonic anemometer with particle analysis techniques, enabling the evaluation of aerosol dispersion in agricultural areas. The wind speed data collected by the anemometer is correlated with the spatial distribution of aerosols, providing crucial insights into the influence of wind on particle dispersion. It is expected that this study will provide valuable insights for precision agriculture, contributing to informed decision-making. The results can assist in the optimization of agricultural practices, reducing costs, minimizing environmental impact, and improving air quality in rural areas.

The ultrasonic anemometer plays an essential role in precision agriculture, and its integration with particle analysis techniques offers a multidisciplinary approach to understanding aerosol dispersion in agricultural environments. The combination of these technologies promotes more efficient and sustainable agricultural practices, with the potential to reduce costs and minimize environmental impacts.

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USAXS Analysis of Ionic Liquid-based Electrolyte for Li-O2 Battery Application

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The versatility and superior stability of ionic liquids (ILs) have motivated their use as additives for electrolytes in electrochemical devices. Based on that, this work proposed the use of a multifunctional ionic liquid crystal (ILC), C16mimBr (1-hexadecyl-3-methylimidazolium bromide), as additive for DMSO/LiClO4 electrolyte. The cation [C16mim] combines the electrochemical stability of ionic liquids with the structural character of liquid crystals to provide a better performance of Li-O2 batteries. The cycling profile of the Li-O2 cell appears to be correlated with the packaging of the bromide ions. This was elucidated by ex situ characterization of the electrolytes via ultra-small angle X-ray scattering (USAXS) using synchrotron light source. In diluted systems, from 0 to 0.35 M ILC, it is suggested the formation of small clusters in the electrolyte structure, while from intermediate to saturated electrolyte, from 1.02 to 2.36 M ILC, a well-defined structure that resembles a core-shell is formed. Besides, as more concentrated is this system, the bigger is the formed structure, reaching micron scale.

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Utilizing Brazilian Sewage Sludge as Feedstock for Biofuel Production: Advancing Towards a Circular Economy

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In Brazil, the improper disposal of sewage sludge poses a significant environmental challenge due to its contamination with various metals. However, a novel approach has emerged, focusing on utilizing this inorganic/organic resource as a catalyst feedstock. Through pyrolysis, sewage sludge can be transformed into an efficient material for converting lignocellulosic biomass (using platform molecules) into biofuels and value-added components for pharmaceutical and fine-chemical industries. One platform molecule, furfural, derived from lignocellulosic biomass, offers a cost-effective and globally available option. Traditionally, furfural conversion into biofuels relies on noble metals, contributing to elevated carbon footprints during catalyst production. Therefore, incorporating biochar from sewage sludge presents a compelling opportunity to reduce costs in both the biofuel and fine-chemical sectors while addressing environmental concerns. This study extensively assessed the utility of two sewage sludge-derived biochars as catalysts for furfural conversion, revealing promising applications for these materials. Comprehensive characterization revealed the presence of acid and basic sites, and these biochars exhibited remarkable activity in converting furfural, with high selectivity towards furfuryl alcohol. In summary, this research highlights the advantageous properties of sewage sludge-derived biochars as catalysts for enhancing lignocellulosic biomass-derived compounds, particularly the versatile furfural molecule. Moreover, by exploring innovative applications for sewage sludge, this work contributes to addressing environmental concerns and advances the development of more sustainable and cost-effective catalysts for bio-derived compound production, aligning with the principles of a circular economy.

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Virtual histology and 3D reconstructions of Drosophila melanogaster brain

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The synchrotron radiation microtomography imaging is an important tool of biomedical science. The technique enables 3D quantitative analysis of structures of biological samples and can be used to measure tissue volumes and tissue densities. Samples with low x-ray absorption it is necessary to use phase retrieval imaging mode. The propagation-based phase contrast is often used and enables to differentiate internal structures without a contrast agents. The fruit fly Drosophila melanogaster is a versatile model organism that has been used in biological research. However, to have a successful tissue segmentation, an optimal imaging technique is necessary. In this work, we analyzed reconstructions of Drosophila melanogaster using the phase retrieval method by Paganin, et al (2002; 2020). We scaled the level of phase retrieval by adjusting the different ratios of the dispersive and absorptive aspects of the wave-matter interaction to discuss the significant impact reconstruction parameters analyzing the brain of fruit fly. The Drosophila melanogaster was scanned in the phase-contrast regimen with a 10 cm sample-detector distance in the Syrmep beamline of Elettra-Sincrotrone. The setup used filtered polychromatic radiation with mean energy of 16,7 keV, pixel size of 1,5 μm, 1200 projections, 180º of scan range. We compared reconstruction parameters to validate specific contrast methods to have the best contrast of the fruit fly's brain with the less loss of information possible. The possibility of use of a 4th generation of synchrotron-light source would contribute a lot to this topic. The Mogno beamline at Sirius enables an exploration of propagation-based phase contrast imaging modes using a monochromatic beam to better study the parameters of phase retrieval. Different image regimes can be explored in phase contrast imaging modes and the zoom tomography can be very useful to explore the same sample in different aspects and under other image regime conditions.

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X-Ray Diffraction Pattern Analysis with Data-driven Methods

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X-ray diffraction (XRD) is the main experiment that allows the investigation of materials' crystalline structures. Advances in instrumentation allow modern facilities to execute high-throughput experiments, but XRD patterns analysis has yet to be developed in tandem, creating a bottleneck for materials research and discovery. Analysis difficulty increases when: 1. a mixture of different phases is present, and 2. patterns deviate from ideal references due to sample defects or impurities, a common scenerio in inorganic materials synthesis.

Artificial intelligence (AI) methods have recently made possible faster and more accurate approaches for XRD pattern analyses. Phase separation and novelty identification can be modeled as a Machine Learning (ML) classification problem. Significant works were developed in this direction by Maffetone et al. [1], and by Szymanski et al. [2]. They use physically informed data augmentations to include defects and impurities in simulated XRD patterns, which are used to train Neural Network models to classify experimental patterns. They achieved the same accuracy as crystallographic experts but in real-time with the measurements. However, differentiation between similar compositional phases and patterns measured under extreme pressures remains challenging.

This work aims to improve the results obtained by applying instance selection algorithms to the synthetic dataset. This preprocessing task performs intelligent operations of instance categorization to edit the data. It cleans noisy and redundant instances and sets the focal point on the critical part of the data. Reducing the dataset size and retaining only the best samples for the problem can diminish training time and improve classification accuracy under still challenging scenarios.

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LNLS USERS GROUP: YOUNG RESEARCH AWARD

Understanding the Bioelectrocatalytic Mechanisms of Metalloenzymes by using Synchrotron Light

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Multicopper oxidases are metalloenzymes containing four copper ions in their catalytic sites. These enzymes have been applied as bioelectrocatalysists on electrochemical energy conversion systems, as biofuel cells, and on water splitting, as they catalyze the oxygen reduction reaction and the water oxidation reaction at very small overpotentials and under mild conditions. Understanding the electron transfer mechanisms of redox proteins has direct impact on their successful practical applications. X-ray absorption spectroscopy (XAS)is a powerful technique for studies on metalloenzymes, as it can provide information on the oxidation state of redox metal co-factors and their chemical environment, without interferences. In addition, XAS can be couple to electrochemical measurements (in situ XAS) to probe mechanisms of redox catalytic reactions under real reaction conditions. Here, aspects of the electron transfer and bioelectrocatalytic mechanisms of bilirubin oxidase (a multicopper oxidase) toward the oxygen reduction reaction and water oxidation reaction by in situ XAS are shown. Through these measurements was able to probe that copper ions act as a 3D redox active electronic bridges for the electron transfer reaction in the enzyme active site. In addiiton, the driving force and energy balance of the catalytic water oxidation reaction could be experimentally calculated.

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Synthesis and structural characterization of $NiSi_2$ nanoplates into silicon [001] wafers

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The work focuses on the development and analysis of nanocomposites consisting of NiSi2 nanocrystals embedded in single-crystalline Si, with potential applications in nanotechnology. Current preparation methods face challenges such as nanocrystal agglomeration and the need for complex instrumentation. The work proposes an alternative method involving a Ni-doped SiO2 thin film deposited on Si(001) wafers through the sol-gel process. Thermal treatments induce Ni diffusion, leading to the formation of ordered NiSi2 nanocrystals. Various techniques, including STEM and GISAXS, are employed to study the nanocomposite

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