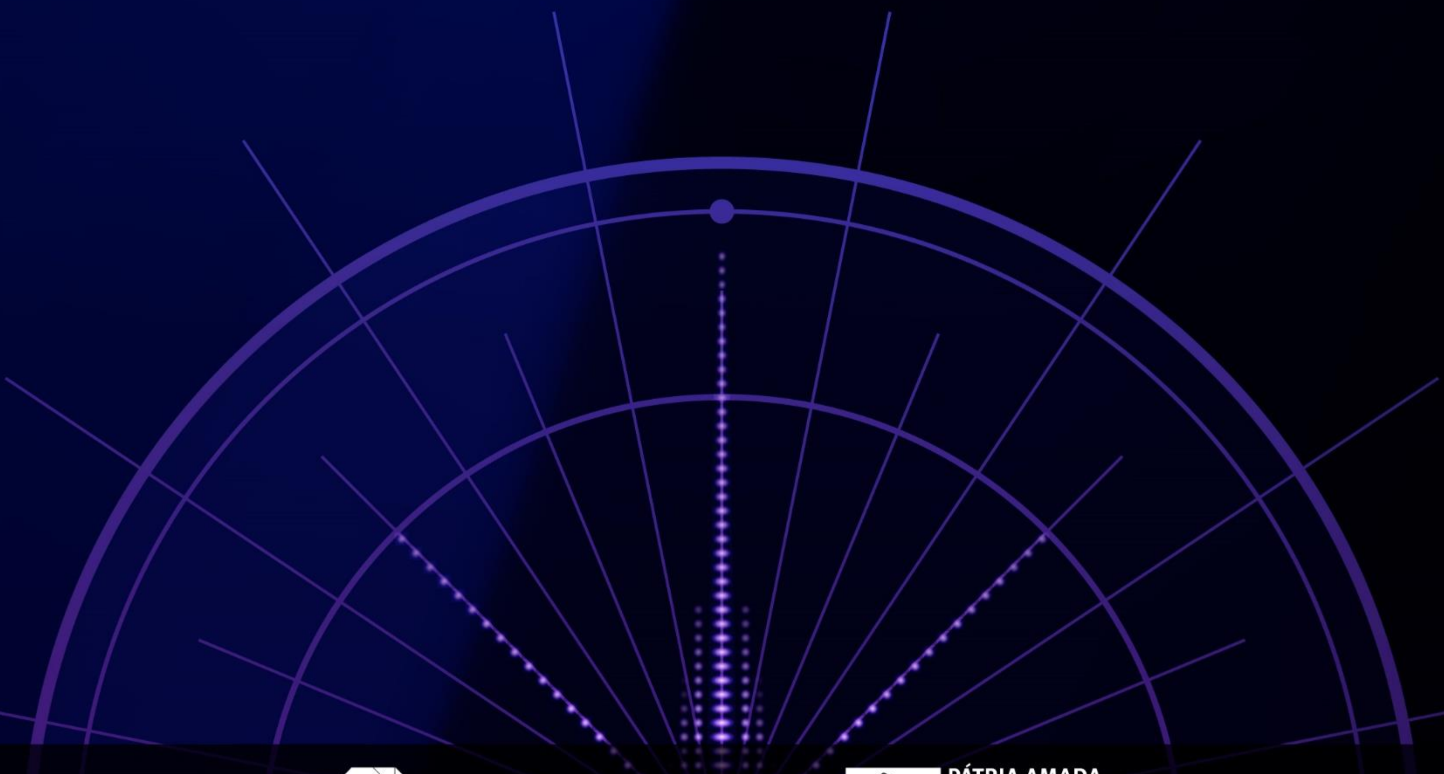
30th RAU

Annual Users Meeting

LNLS | CNPEM

ABSTRACT BOOK



CNPEM
MINISTRY OF SCIENCE, TECHNOLOGY AND INNOVATIONS
PÁTRIA AMADA BRASIL
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30th RAU

Annual Users Meeting

Organization

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Dear colleagues,

On behalf of the Organizing Committee, I am pleased to welcome all the participants to the 30th edition of the Annual Users Meeting of the Brazilian Synchrotron Light Laboratory- RAU/LNLS. I would like to start by wishing you and your families my personal best for your health and safety in these difficult times.

This year has tested us like no other. RAU is a unique opportunity to gather together the Brazilian lightsource community in a single scientific event to disseminate results and exchange ideas. Following much debate, we decided to proceed with the RAU in the now realized alternative format. This was not without its own challenges.

The year 2020 marks the 30th anniversary of this Users Meeting. Although RAU is being run under exceptional circumstances, we have created what we hope will be a RAU to be remembered for many years to come.

30th RAU will be four days long bringing together around 500 researchers from different corners of Brazil and from more than 20 countries around the world. The programme includes 114 poster presentations, 20 oral contributions, 4 keynote talks and 6 invited plenary lectures.

In addition, we will have an overview of LNLS, Sirius virtual tour, meetings with beamline coordinators, and last but not the least, the newly elected User Committee will be presented during the LNLS Users Group assembly.

The structure of this RAU was designed to stimulate discussion and participation. All sessions will have life in them. We certainly encourage all participants to attend and engage with sessions as though you were here in person. We are privileged to hear the highlights from this year’s hard work on Sirius beamlines and to take part in a rich, and highly relevant, discussion on cutting-edge research to stimulate new proposals and ideas. We believe many good experiences will be shared and good lessons learned.

We also want to express our optimism about the future, with the Sirius moving along, ready to go for another 30 years. Sirius currently works with a stable circulating current in the accelerators at around 10% of the current expected for its full operation. In the last few months, the first experiments on the MANACÁ beamline were carried out by external researchers dedicated to studying the molecular mechanisms related to the action of the SARS-CoV-2 virus. The other beamlines of the first phase (CARNAÚBA, CATERETÊ, EMA, IPÊ, MOGNO) are in an advanced stage of assembly. We cannot fail to register our sincere gratitude and our recognition to professor Ricardo Rodrigues.

Finally, we would like to thank this incredible Local Committee and the CNPEM staff for organizing this RAU, ensuring it ran so smoothly. We would also like to take this opportunity to thank our sponsors who supported the move to an online meeting.

Take care of yourselves and stay healthy. Hope to see you all soon!

Regina Cély Barroso
Chair of the 30th RAU

Abstracts

INVITED ORAL PRESENTATIONS

ORAL PRESENTATIONS

POSTER PRESENTATIONS

INVITED ORAL PRESENTATION

3D IMAGING OF BIOLOGICAL TISSUES BY X-RAY PTYCHOGRAPHIC TOMOGRAPHY

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We present state-of-the-art ptychographic tomography of soft biological tissues as performed at the cSAXS beamline at the Swiss Light Source. To this end, we introduce X-ray ptychography, a high-resolution X-ray imaging modality that exploits the high brilliance of 3rd generation synchrotron sources, and its extension to tomography. We show that biological soft tissue extending several tens of micrometers in all directions can be imaged with a 3D resolution down to 50 nm when appropriately stained with heavy elements, while a resolution of about 70 nm can be achieved on hydrated unstained tissue. We will further discuss the advantages of obtaining quantitative density contrast [1] and the limitations and opportunities when attempting to improve the resolution. In doing so, we will cover examples of applications ranging from yeast cells [2] to brain tissue [3]. High-resolution X-ray imaging could be used as a complementary technique to electron microscopy and fluorescence light microscopy to visualize densely packed biological tissues free from cutting artifacts while allowing a quantification of the density of the different subcellular compartments.

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ACKNOWLEDGEMENTS:

We thank users and collaborators at the cSAXS beamline, who will be acknowledged specifically for each project during the presentation. Mariana Verezhak acknowledges funding from the SNF grant No 200021L 169753, ANR-16-CE93-0006 and the European Union's Horizon 2020 research and innovation program under the Marie Sklodowska-Curie grant agreement No 701647.

INVITED ORAL PRESENTATION

ARE YOU MEASURING WHAT YOU THINK YOU'RE MEASURING? RADIATION INDUCED DAMAGE IN X-RAY FLUORESCENCE MICROSCOPY

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where an electron is ejected from its host atom. We call this process the "observer effect", where the very act of making the measurement causes damage to the sample [1]. The observer effect can, in some cases be so obvious to preclude analysis while in other cases it can introduce subtle system wide errors. While often this damage is not observable, it is always present [2], with the aim that the damage to the sample is kept below a threshold that does not influence the results. With a push towards "native state" imaging and imaging with additional dimensions such as rotation [3], energy [4, 5], and time [6], the cumulative effects of X-ray induced radiation damage can become significant, invalidating results. In this presentation I will discuss the effects of radiation induced damage on a variety of radiation sensitive samples, together with a method for measuring radiation induced damage. Strategies for minimising the effect of radiation induced damage are also presented.

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ACKNOWLEDGEMENTS:

This research was undertaken on the X-ray Fluorescence Microscopy (XFM) beamline at the Australian Synchrotron, part of ANSTO.

INVITED ORAL PRESENTATION

BACTERIAL SENSORY MACHINES, WATCH THEM AS THEY MOVE

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Bacteria use protein machineries to sense environmental and intracellular signals, enabling them to respond adaptively. These sensory transduction systems include one-component systems (OCS), two-component systems (TCS), phosphotransferase systems (PTS) and extra-cytoplasmic function (ECF) sigma factors. Our work has contributed to showing that protein malleability is a critical element to allow for signal sensing and output control regulation in TCSs. Extending this general principle to OCSs, we recently solved the crystal structure of Mycobacterium tuberculosis FasR a TetR-like OCS that works as a signal-dependent transcriptional activator of fatty acid synthase I. Solving the structure of FasR in complex with acyl effector ligands and with DNA, uncovered its molecular sensory and switching mechanisms [1]. A tunnel was observed traversing the entire ligand-binding domain, enabling for long- and very long-chain fatty acyl effectors to bind. Only when the tunnel is entirely occupied, FasR adopts a rigid configuration with its DNA-binding domains in an open state, leading to DNA dissociation. Due to the intrinsic symmetry of DNA’s double helix, the FasR-DNA complex exhibited a form of crystal disorder, with the polynucleotide chain oriented in opposite directions among unit cells. Together with the large size of the complex, data collected at a synchrotron facility (Diamond Light Source, UK) was critical to determining this 3D structure, key to understanding the functional mechanism. Finally, a continuous hydrophobic spine was observed connecting the sensory and effector domains within FasR. Such a spine is conserved in a large number of TetR-like regulators, uncovering an efficacious allosteric control mechanism: acting as a true transmission gear, it equips the protein with a signal-binding-dependent engine that modulates its DNA-binding properties. These data showcase the functional relevance and universality of dynamical features in signaling proteins.

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INVITED ORAL PRESENTATION

BREAKTHROUGHS IN MATERIAL CHARACTERIZATION AT EXTREME CONDITIONS WITH SYNCHROTRON AND OPTICAL TECHNIQUES

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In past three decades high pressure research has made breakthrough progress in many fields of science mainly due to significant improvements in both types of high-pressure vessels (diamond anvil cell and large volume press), high x-ray energy sensitive detectors and developments of advanced static and dynamic probes including, high spatial and energy resolution synchrotron-based and optical techniques. Most of the experiments at ultra-extreme pressure and temperature conditions are very challenging and require dedicated synchrotron beamlines, where state-of-the-art high-pressure on- and off-line techniques have been implemented and are currently being developed. Such unique capabilities at ultra-high P-T conditions approaching the warm dense state of matter will open an entire new research area creating a bridge between shockwave and DAC experiments that provides fundamental structural, thermodynamic, and transport property information essential for understanding the composition, origin and evolution of planetary systems. With this technique we have successfully studied a number of unique properties of elements and their compounds synthesized in-situ at ultra-extreme P-T conditions such as transition metals, silicates, various polyhydrides, super-ionic phases of ice etc. Details of recent results and future developments of cutting-edge synchrotron techniques for comprehensive characterization of materials in-situ at extreme conditions in view of planned APS diffraction limited storage ring upgrade will be discussed.

INVITED ORAL PRESENTATION

HETEROGENEOUS CHEMISTRY AT AQUEOUS INTERFACES INVESTIGATED WITH AMBIENT PRESSURE X-RAY PHOTOELECTRON SPECTROSCOPY

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Aqueous interfaces govern important phenomena in technology, catalysis, electrochemistry and the environment [1,2]. These systems include solid-liquid, liquid-vapor and solid-vapor interfaces. Among the questions of interest are the first stages of water adsorption at solid surfaces, which has a strong influence on, e.g., the formation of seeded cloud droplets. Liquid-vapor interfaces hold special interest for the uptake and release of trace gases by aqueous aerosols and CO_2 sequestration by the oceans. Solid-liquid interfaces are at the heart of electrochemical and corrosion phenomena. A detailed understanding of these processes requires the investigation of aqueous interfaces with chemical sensitivity and interface specificity under ambient conditions, i.e., temperatures above 200 K and water vapor pressures in the millibar to tens of millibar pressure range. This talk will discuss opportunities and challenges for the investigations of aqueous interfaces using ambient pressure X-ray photoelectron spectroscopy [3,4]. We will discuss the fundamental challenges to performing XPS experiments at elevated pressures and how to overcome them. Examples of the application of ambient pressure XPS to the study of water adsorption on oxide surfaces, the observation of short-lived reaction intermediates in aqueous solution and the investigation of electrical field gradients at semiconductor-solution interfaces will be presented as well.

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INVITED ORAL PRESENTATION

HIGH RESOLUTION STRUCTURES OF THE SARS-CoV-2 NSP16/NSP10 2'-O-METHYLTRANSFERASE REVEALS STRATEGIES FOR STRUCTURE-BASED INHIBITOR DESIGN

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SARS-CoV-2 is the etiological agent of the respiratory Coronavirus Disease 2019 (COVID-19). The virus has spread rapidly around the world with over 1 million deaths. The Center for Structural Genomics of Infectious Diseases has solved structures of 15 unique proteins, including nucleocapsid domains, proteases, endonucleases, Spike, accessory proteins and components of the replicase. As part of this effort, we conducted an X-ray crystallographic study of the structure of the SARS-CoV-2 2'O-methyltransferase. The non-structural protein 16 (nsp16) methylates Cap-0 viral mRNAs to improve viral protein translation and to avoid host immune detection. The methyltransferase activity of nsp16 is activated upon binding of nsp10, the methyl donor S-adenosylmethionine (SAM), and a viral Cap0-mRNA. We solved structures of the nsp16/nsp10 heterodimer at high resolution (1.8-2.0 Å) in complex with the methyl donor SAM in two different crystal forms. The structure of nsp16 and nsp10 were highly conserved with SARS-CoV, with 100% conservation of the contact interface between nsp16 and the activator protein nsp10. The structure of the heterodimer was also solved in complex with the product of the reaction S-adenosylhomocysteine (SAH) with pan-MTase SAH analog inhibitor sinefungin. Of particular significance, we solved the first SARS structures of nsp16 in complex with the mRNA cap-0 analog m7GpppA and the first structure with Cap1-RNA. In addition to the substrate and SAM binding sites, novel nucleotide interactions were found on the face of the protein opposite the substrate binding. We suggest that these structures can be used to design specific antiviral inhibitors.

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ORAL PRESENTATION

ACTIVITY OF N_2O REDUCTION BY CO AND THE EVOLUTION OF COPPER SPECIES

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One undesired product of the NO reduction by CO is the N_2O pollutant, which is an intermediate of the reaction [1]. There are efforts to minimize its presence by designing more efficient catalysts. The metal copper is a promisor one and it has been explored in some studies of our group [2]. In this work two precursors were prepared by coprecipitation method, one bimetallic (Cu, Al) and another monometallic (Cu). The catalysts were obtained by calcination of the precursors at 600 °C for 4 h with a heating rate of 10 °C min-1 in static conditions (air atmosphere). The catalysts were named CuAlc600 and Cuc600. Afterwards, the catalysts calcined at 600 °C were thermally aged for 12 h at the same conditions (CuAla900 and Cua900). The objective of this work was to compare the effect of Al in the performance and the thermal stability of the catalysts. The reaction of N_2O reduction by CO was carried out using conventional catalytic unit and by XRD in situ, at XPD line at Laboratório Nacional de Luz Síncrotron (Campinas, Brazil). Among the catalysts that contain Al, the catalyst CuAla900 showed the best activity. According to the XRD in situ, throughout the N_2O reduction, this catalyst presented higher formation of metallic copper than the catalyst CuAlc600. Moreover, the intensity of spinel phase peaks along the reaction did not change, it seems that Cu2+ species from that phase do not reduce. In addition, the catalysts with Al showed a particular dynamic between the Cu+ and Cu0 species after 400 °C. The Cu+ species rise gradually whereas Cu0 species decrease; it seems that a redox cycle might be playing a role promoting the N_2O reduction. This behavior was not observed for the aluminum-free catalysts, they lost activity at high temperatures (>250 °C). Thus, after 400 °C, the spinel phase and Cu0 species might play an important role in the N_2O reduction, contributing to the catalytic stability.

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ORAL PRESENTATION

APPLICATION OF X-RAY COMPUTED MICRO-TOMOGRAPHY TECHNIQUE IN UNDERSTANDING THE DYNAMICS OF FLUID FLOW IN POROUS

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In this talk I will present some of the research that I have been doing on multi-phase fluid transport in porous media at microscopic scale, also known as the pore-scale. I will share with you what I have learned from investigating the recovery of trapped organic fluids (solvents or oils) from porous rocks representing the subsurface. These findings have implications for a wide range of applications including: (i) groundwater remediation, (ii) oil recovery from hydrocarbon reservoirs, and (iii) subsurface storage of CO_2 or (more recently) H_2 in geological formations. I will discuss the use of X-ray computed micro-tomography in visualising and quantitatively analysing fluid displacement processes in sedimentary environment. In the past decade, X-ray computed micro-tomography has helped us identify the main events that take place (at pore-scale) during non-reactive [1-4] and reactive [5] fluid transport in porous media. I will discuss that understanding the physics behind such pore-scale processes is the key to conduct successful and effective processes at field scale.

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[4] https://doi.org/10.1038/sdata.2019.4
[5] https://doi.org/10.1073/pnas.1918683117

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KEYWORDS: X-RAY COMPUTED MICRO-TOMOGRAPHY, POROUS MEDIA, GROUNDWATER REMEDIATION, OIL RECOVERY, HYDROCARBON RESERVOIRS, SUBSURFACE STORAGE

ORAL PRESENTATION

ASSESSING THE ROLE OF ELEMENTS IN CANCER PROGRESSION WITH SR-XRF AND DATA MINING

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Cancer is considered one of the most complex and fatal diseases worldwide. New approaches to study tumor progression and growth are relevant subjects of research. In this context, the particular role of elements in cancer progression is a subject still not fully explored that presents opportunities for investigation. The main goal of our study is to assess the distribution of elements and their roles in cancer growth and metastasis. For simulating tumor progression in vivo, murine Lewis lung carcinoma cells were injected in C57BL/6 mice and data indicating the presence, concentration, and location of different elements in distinct tissues, in both control and experimental groups, were obtained in a time frame of 5 weeks of tumor progression. The data were collected via SR-XRF in the Brazilian Synchrotron Light Laboratory (LNLS). These data allow us to observe both the primary tumor and the preparation of distant tissues that the tumor cells may affect. In order to extract relevant information inherent to the voluminous available data, we adopt statistical analysis and data mining techniques, as well as multi-elemental maps. Such techniques allow the analysis of the distribution and role of elements in cancer progression, as well as the discovery of correlations between elements. Our preliminary statistical analysis have highlighted interesting information, such as the impact of animal aging in elemental distribution, as well as the chaotic behavior found within tumor tissue. The elements P, S and K have been found as the most correlated, while the elements Mn, Cu and Zn have shown variability in correlation. Further studies are under development to unveil the relevance of these findings. Our perspectives include performing additional data mining analysis, as well as analyzing multi-elemental maps. We believe data mining is a powerful tool to be applied to SR-XRF data, especially when approaching complex and unpredictable structures, such as cancer samples.

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KEYWORDS: CANCER, X-RAY FLUORESCENCE, DATA MINING, MULTI-ELEMENTAL ANALYSIS, ELEMENTAL CORRELATION

ORAL PRESENTATION

BIOCHEMICAL AND STRUCTURAL CHARACTERIZATION OF LSFA, A 1-CYS PEROXIREDOXIN INVOLVED WITH PSEUDOMONAS AERUGINOSA VIRULENCE

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Pseudomonas aeruginosa is a ubiquitous gamma-proteobacterium that is the main cause of hospital-acquired infections among all pathogens related with pneumonia. Among the host defenses, phagocytic cells release oxidants in an attempt to kill pathogens. LsfA belongs to the peroxiredoxins (Prx) family that comprises Cys-based peroxidases. LsfA belongs to the Prx6 sub-family, which contains mostly enzymes with only one catalytic cysteine (so-called 1-Cys Prx). LsfA is involved in P. aeruginosa virulence, as shown by studies in mice and macrophages models. Therefore, the aim of the present work is the structural and biochemical characterization of LsfA. To determine the second-order rate constants for the reactions of LsfA with several peroxides, we took advantage of the fact that its intrinsic fluorescence of this protein changes in a redox dependent manner. We also employed competitive assays to confirm the data and we observed that LsfA reacted extremely fast with different peroxides (H_2O_2 = 107 $M^{-1}.s^{-1}$; tert-butylhydroperoxide = 106 $M^{-1}.s^{-1}$; peroxynitrite = 107 $M^{-1}.s^{-1}$). Also hyperoxidation rate constants were determined with H_2O_2 (230±2.31 $M^{-1}.s^{-1}$) and t-BOOH (286.9±11.27 $M^{-1}.s^{-1}$) using the fluorimetric approach and confirmed by western blotting. Thus, LsfA rapidly reduces several peroxides, however it is not too susceptible to hiperoxidation. Furthermore, two crystallographic structures of LsfA were elucidated in distinct oxidative states (reduced and sulfonic state at the peroxidatic cysteine). LsfA as a dimer, independently of its redox state, which was confirmed by SAXS analysis. The comparison of the bacterial LsfA and human Prx6 crystal structures revealed some differences at the active site positioning and topology that might be related to differences in substrate accessibility and binding. Thus, important features of LsfA substrates reactiviy and its crystallographic structures may enable the identification of inhibitors for this peroxidase.

ORAL PRESENTATION

DEPICTING X-RAY RADIATION DAMAGE TO IN VIVO PLANT TISSUES: A GLANCE ON ELEMENTAL, ULTRASTRUCTURAL, AND HISTOCHEMICAL RESPONSES

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The X-ray fluorescence spectroscopy (XRF) is a powerful technique for the *in vivo* assessment of plant tissues [1,2]. However, the potential X-ray exposure damages might affect the structure and elemental composition of living plant tissues, hence, implying on artifacts on the recorded data [3]. In this framework, we herein explored the exposure of soybean (*Glycine max* (L.) Merrill) leaves to several X-ray doses on a polychromatic benchtop microprobe X-ray fluorescence spectrometer. We modulated the photon flux by playing with both beam size and focus, as well as the exposure time. Besides, the structure and physiological responses of the irradiated plant tissues were further investigated by transmission electron microscopy (TEM) and histochemical analysis. Depending on the dose, the X-ray exposure induces a sharp decrease of K and the X-ray scattering intensities, whereas Ca, P, and Mn signals increase on soybean leaves. Besides, the presence of necrosis and scorching were also recorded on the irradiated spots. The transmission electron microscopy images reveal the collapse of cytoplasm and cell-wall breaking on the irradiated spots. Furthermore, the histochemical analysis detected the production of reactive oxygen species, callose, and autophagic vacuoles, as well as inhibition of chlorophyll autofluorescence in these areas. Therefore, it highlights that some X-ray exposure conditions, *e.g.*, high photon flux and exposure time affect its structures soybean leaves elemental composition, cellular structures, and then induce programmed death cell. These results might shed light on the characterization of the radiation damage, and thus, help to assess the safety limits and strategies for *in vivo* for XRF analysis.

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KEYWORDS: RADIATION DAMAGE, XRF, TEM, HISTOCHEMISTRY

ORAL PRESENTATION

DYNAMICS OF FERROELASTIC DOMAINS IN $CsPbBr_3$ NANOWIRES IMAGED BY IN SITU NANOFOCUSED SCANNING X-RAY DIFFRACTION

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There is a significant interest of the materials science community in metal halide perovskites (MHPs), for applications ranging from solar cells, laser devices and photodetectors [1]. $CsPbBr_3$ is a material of this class which exhibits good radiation stability, making it extremely promising for a great variety of optoelectronic devices, including X-ray detectors [2]. While there is a wide range of papers reporting MHP's optical and electronic properties, few studies can be found on their crystal structure. Understanding phase transitions and ferroelastic domain formation in perovskite materials is crucial both for basic and applied materials science. We have used the NanoMAX beamline at the MAX IV facility in Lund, Sweden, the first fourth-generation synchrotron in the world, to investigate $CsPbBr_3$ nanowires. Scanning XRD with the 50 nm X-ray focus was used to map the local crystal structure. The measurements show the dynamics of nanoscale ferroelastic domains and multiple phase coexistence along $CsPbBr_3$ nanowires, revealing unique characteristics of this material. Our results answer basic questions regarding its crystal structure, enlightening the role of temperature and pressure on metal halide perovskite phase transitions.

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KEYWORDS: PEROVSKITE, NANOWIRE, X-RAY DIFFRACTION, FERROELASTICITY

ORAL PRESENTATION

FERROMAGNETISM IN TiO_2 AND ZnO OXIDES BY HYDROGEN INCORPORATION

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In the last 20 years the number of non-magnetic solids showing magnetic order induced by some kinds of defects increases continuously. This phenomenon to trigger the magnetic properties by defects was investigated by experiment and theory in a huge amount of papers (see [1] for a short review). This effect was also intensively investigated for ZnO and TiO_2 oxides. In this work we will present results in ZnO [2] and TiO_2 systems that results magnetic due to hydrogen incorporation via hydrogenation (high H_2 pressure in a closed chamber at 500 C) and/or via H+ implantation (at room temperature). The samples were characterized structurally and magnetically by different techniques and particularly by X ray absorption (XAS) and magnetic circular dichroism (XMCD) at Zn-L-edge, O K-edge and/or Ti L-edge. These results combined with ab-initio calculations and spectra simulations with FDMNES contribute to the understanding of the magnetism in these systems.

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ORAL PRESENTATION

FOLLOWING PROTEIN CORONA FORMATION ONTO NANOPARTICLES BY MEANS OF SAXS AND COMPLEMENTARY TECHNIQUES

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Nanoparticles immersed in biological fluids experience protein adsorption. This phenomenon alters the surface properties of nanoparticle and ultimately modifies their interaction with biological systems [1]. Several techniques are used to characterize different aspect of the protein corona like binding parameters (e.g. UV-Vis spectroscopy, Fluorescence, ITC), structural features (e.g. TEM, DLS, circular dichroism) and composition (e.g. SDS-PAGE, LC-MS), among others [2,3]. In this work we show how SAXS can be used to follow the formation of protein corona onto silica nanoparticles in situ. The proposed methodology is model independent and provides a robust way to determine binding parameters. In addition, we compare the methodology with the more traditional DLS characterization of protein corona formation and show pro and cons of each technique. Finally, we complement our results with cryo-TEM experiments for visualizing the protein corona and further confirm SAXS and DLS conclusions.

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KEYWORDS: PROTEIN CORONA, NANOPARTICLE, NANOMEDICINE, PROTEIN ADSORPTION, SAXS

ORAL PRESENTATION

GIWAXS STUDY ON THE ANNEALING TIMES OPTIMIZATION OF CONJUGATED POLYMERS FOR OPV APPLICATION

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Thermal annealing represents an important stage aiming fabrication of active layers for organic solar cells, since crystalline organization improves charge carriers mobility and induces the proper morphology of the electron donor and electron acceptor. In this work, the optimal annealing time for maximization of crystalline volume fraction and the crystallization mechanisms of conjugated polymers films (synthesized in our group) for solar cells application have been determined by Grazing Wide Angle X-ray Scattering (GIWAXS). By following the evolution of the diffraction peak position (Xc), peak area and Full Width at Half Maximum (FWHM), it was possible to determine the proper annealing time and to identify the stages corresponding to crystallites formation and growth for poly (butyloctylbenzodithiophene co benzothiadiazole) (PBOBDTBTD), poly (butyloctylbenzodithiophene co thiophene) (PBOBDTTh) and two different Poly (3-hexylthiophene) (one of lower molar mass synthesized through Kumada mechanism (P3HTA) and other of higher molar mass through Grimm mechanism (P3HTB). The results show that annealing time increases with polymer backbone rigidity, being around 1200s, 800s and 400s for PBOBDTBTD, PBOBDTTh and P3HT, respectively.

ORAL PRESENTATION

HOMOGENEOUS SYNTHESIS OF GOLD NANOPARTICLES: KINETICS, MECHANISMS, AND RELATED ONE-POT COMPOSITES

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Gold nanoparticles (AuNP) are among the most studied noble metal nanoparticles because of their key role in fundamental and applied nanoscience. Several morphologies and sizes could be obtained nowadays, that have been exploited for a great variety of applications, some of which are already available in the market. However, homogeneous synthesis of AuNP has been, by far, less exploited. This approach results excellent to ensure that both the nucleation and growth of the particles take place in the absence of any compositional inhomogeneity in the solution, leading to a more controlled reaction path. Moreover, this approach allows the combination with other chemical reactions to give rise to composites in one step. In this work, we introduce the homogeneous reduction of HAuCl4 into metallic AuNP through the epoxide route.[1] The proposed method takes advantage of the homogenous generation of OH- and reductive moieties driven by epoxide ring-opening, mediated by chloride nucleophilic attack. Once reached alkaline conditions, the reducing medium allows the quantitative formation of AuNP under well-defined kinetic control. The homogeneous growth mechanism was comprehensively studied by means of in situ UV/Vis spectroscopy, small-angle X-ray scattering and pH measurements. The obtained results alert about the non-innocent role of the stabilizing agent in the chemical speciation of Au (III) and its reactivity. Finally, we present a particular application in which the reduction process is sequentially coupled with a non-alkoxidic sol–gel process, leading to nanocomposites (AuNP at inorganic hydrogels) of high optical quality on a one-pot basis. This work paves the way towards the generation of a wide variety of AuNP and composites by one-pot homogenous methods at room temperature. Moreover, it demonstrates the feasibility of using such methods to perform careful physicochemical characterization of the AuNP formation process.

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ORAL PRESENTATION

IMAGING AND ANALYZING NEURAL TUBE DURING MOUSE EMBRYONIC DEVELOPMENT: NEW TOOLS WITH SYNCHROTRON RADIATION TO STUDY THIS COMPLEX 3D STRUCTURE

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Neurulation is a critical process during embryonic development. The neural tube (NT), formed during neurulation, will differentiate into encephalic and medullar structures[1] , thus, impact on neurulation could lead to serious consequences in newborns. The severity of these malformations varies from life impeding (e.g. anencephaly), life threatening (e.g. spina bifida) or might influence cognitive and motor ability[2]. Also, some pathogens such as TORCH agents affects NT development in several degrees. Although severely affected embryos are easily noticed, identification of subtle malformations in a three-dimensionally complex structure like the NT is challenging using only two-dimensional conventional techniques such as histological sections [3], in situ hybridization and immunolabeling for optical or electron microscopy. Here we present preliminary results of an optimized 3D imaging approach, from sample preparation to analysis and quantification, addressing the NT development in mouse model. Whole embryos at 10.5 days post coitum (dpc), preserving NT structural complexity, were harvested and prepared for high resolution synchrotron microtomography at the IMX beamline. The first step in the computational workflow was to correctly separate the NT from all surrounding embryonic tissues. Superpixel-based supervised classification (using in-house developed software called Annotat3D) was applied to correctly identify NT cells in the three-dimensional volume. Next, NT segmentation was refined to fit the NT borders. Later, we developed algorithms to untwist the 3D embryo segmentation and quantify the thickness of neuroepithelium according to the embryo's antero-posterior (AP) axis. The results are promising, with 4 embryos from healthy mouse dams analyzed. In the next steps, we will validate this methodology and findings in embryos from affected dams in flavivirus infection model in order to better understand the impact of subtle malformations to postnatal life.

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KEYWORDS: NEURULATION, IMAGE ANALYSIS, X-RAY TOMOGRAPHY, SEGMENTATION

ORAL PRESENTATION

IN SITU XAS: A POWERFUL INVESTIGATION TOOL FOR PALLADIUM-MEDIATED BIOORTHOGONAL UNCAGING REACTIONS IN CANCER CELLS

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Bioorthogonal chemistry seeks chemical reactions that exhibit selectivity while being performed inside living systems without affecting native bioprocesses [1]. Thus, the use of transition metals that do not exhibit a natural function in biological environments may seem perfectly suited candidates for bioorthogonal applications [2]. In fact, their versatility in making and breaking chemical bonds has led to a rapidly growing interest in this field with purposes varying from cellular imaging to therapy [3, 4]. However, there is a vast gap between the concept and the actual application of these systems in vivo that arises from the loss of efficiency as well as loss of biorthogonality when transferring from reaction flasks to cellular milieu [5]. As a mean to overcome these hurdles, we propose that a better understanding of the mechanism of these reactions in biological media would work as a powerful tool enlightening the path for the rational design of biorthogonal catalysts. The limitations, however, reside in the difficulty to investigate catalytic cycles or lack of thereof in such complex environment as biological media. In this context, the application of X-ray Absorption Spectroscopy (XAS) for mechanistic investigations is extremely appealing because it allows to selectively monitor the metal centre of the catalyst, under turnover conditions, while disregarding organic components in the system. Therefore, we are interested in employing XAS to gather insights about the fate of palladium catalysts during the mediation of uncaging biorthogonal reactions conducted in vitro. The subject of our studies consists of the extra- and intracellular activation of prodrugs by palladium triggered bond cleavage reactions in breast cancer and leukaemia cell lines. The challenge ahead is the experimental setup to acquire electronic and structural information about the palladium accumulated in these cells and how it is affected by the course of the uncaging reaction.

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KEYWORDS: PALLADIUM, BIOOTHOGONAL, UNCAGING, XAS

ORAL PRESENTATION

INVESTIGATION OF EXOGENEOUS PULMONARY SURFACTANTS BY WIDE- AND SMALL-ANGLE X-RAY SCATTERING

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Exogenous lung surfactant (ELS), a surface-active lipid-protein complex that mimics the natural pulmonary surfactant, is used on surfactant replacement therapy, assisting the baby breathing until it can produce these surfactants by itself [1]. Among the ELS commercially available, Curosurf is extracted from lavages of minced porcine lung and further purified by different chromatographic steps to remove cholesterol and other neutral lipids. Its clinical use is closely related to the temperature variation, from storage to being inside the human body, and it is well known that this thermic effect causes alteration in both structure and physical-chemical properties of the ELS. From the perspective of lung surfactant membranes, this temperature variation is large enough to submit them to a melting transition [2] where they display a coexistence of gel (Lβ) and fluid (Lα) phases [3]. In this work we investigate the temperature-induced structural changes of Curosurf samples (in bulk) using simultaneous wide- and small-angle X-ray scattering (WAXS/SAXS) experiments with in situ temperature variation. We found that below ~35 °C, the scattering data indicated the coexistence of two multillamellar phases, characterized by two distinct lamellar periodicities, with different carbon chain organizations (Lβ + Lα). For temperatures above this limit, the coexistence of phases disappears, giving rise to only one multilamelar phase with fluid carbon chains. This process is quasi-reversible under cooling, since advanced data analysis for the scattering data indicated differences in the structural and elastic properties of the pulmonary surfactants. The detailed and systematic investigation shown in this study expands the knowledge on the structure of Curosurf, being relevant from both physiological and biophysical perspectives, and also provides basis for further studies on other types of pulmonary surfactants.

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KEYWORDS: PULMONARY SURFACTANT, WAXS, SAXS, CUROSURF

ORAL PRESENTATION

LOCALIZATION AND SPECIATION OF CARBON OCCLUDED IN SILICA PHYTOLITHS FROM GRASSES AS AN INITIAL STEP TO QUANTIFY ITS GLOBAL POTENTIAL FOR SOIL CARBON SEQUESTRATION STRATEGIES

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Carbon sequestration in soils is one of the main strategies to mitigate global climate change. Carbon is naturally occluded within phytoliths which are abundantly produced by important cereal grasses (rice, wheat) and by energy grasses (sugarcane, switchgrass). Grasses phytoliths are micro-structures (10-30 µm) formed in the epidermis of the plant's shoot. Throughout phytoliths development, a portion of carbon and inorganic elements are incorporated within the amorphous silica matrix. The incorporated carbon within the phytoliths is referred as occluded carbon (PhyOC) [1]. Archaeological evidences confirm that Si-phytoliths are durable bodies that protect PhyOC from natural degradation in soils up to a millennial time scale. The possibility of keeping a fraction of carbon locked up in soils through phytoliths has been proposed as one significant strategy for long-term soil carbon accumulation, possibly at a rate of ~2.4 g C m-2 year-1 [2]. However, PhyOC quantification has been controversial due to chemical-thermal methods currently used to isolate phytoliths from lignocellulosic matter [3]. Non-destructive methods are thus crucial to quantify PhyOC. There are cutting-edge X-ray imaging techniques at Sirius potentially useful to quantify, localize, and chemically characterize PhyOC. We propose strategies to solve the spatial distribution and speciation of PhyOC from sugarcane-phytoliths exploring the potentials of MOGNO, CATERETÊ, CARNAÚBA, and SABIÁ beamlines. Using x-ray microscopy and tomography techniques will allow us to better understand how carbon is sequestered inside phytoliths and eventually lead to new approaches for global strategies to mitigate climate change.

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KEYWORDS: CARBON SEQUESTRATION, CLIMATE CHANGE, PHYTOLITHS, ENERGY CROPS

ORAL PRESENTATION

POLARIZATION-DEPENDENT ARPES ORBITAL STUDIES OF CO AND Cu DOPED $BaFe_2As_2$ BAND STRUCTURE

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Angle-resolved photoemission spectroscopy (ARPES) is consolidated as a reliable tool in the investigation of high-Tc superconductivity. In the case of iron-based superconductors (FeSCs), the multi-orbital multi-band character of the Fermi surface (FS) are key properties, which can be probed using polarization-dependent ARPES. This technique allows the study of the orbital contribution to the FS, of the superconducting (SC) or spin density wave (SDW) gaps, band renormalization, etc [1]. In particular, the role of the different Fe derived 3d orbitals to the SDW or SC transition can be studied. Moreover, the relative orbital weight to electronic correlations, magnetism, and localization can be a key to the minimal model required to explain the difference in the phase diagrams across this family of materials [2]. Among the Fe 3d orbitals, the ones which contribute the most to the FS in the parent compound $BaFe_2As_2$ are the t2g orbitals dxy, dxz, dyz. We performed ARPES, at the PGM beamline, of $Ba(Fe_{0.995}Co_{0.005})_2As_2$ and $Ba(Fe_{0.995}Cu_{0.005})_2As_2$ to study the orbital contribution to the FS in the SDW phase, for different high symmetry directions at 2 different kz values. From our results, no qualitative difference was found between samples, which presented the same polarization dependent features. At Z, the FS also has the same orbital composition, but with larger hole pockets when compared with Γ hole pockets. The electron pocket in M is strongly polarization dependent, with strong dxy contribution. As for the hole pocket, it shows almost no polarization dependency, revealing a main contribution from dxz/dyz orbitals. We could confirm the slightly 3D FS topology for the FeSC, as well as probe the orbital composition to its features. The quantitative analysis of the SDW gap opening is impractical due to the source size and resolution. We expect that these results will bring some insight to probe even better polarization dependent ARPES at SIRIUS hereafter.

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KEYWORDS: ARPES, SUPERCONDUCTIVITY, SDW, ORBITAL, POLARIZATION

ORAL PRESENTATION

PRESSURE-INDUCED STRUCTURAL PHASE TRANSITION AND ANOMALOUS PHOTOLUMINESCENCE BEHAVIOR IN LUMINESCENT BULK Cs$_4$PbBr$_6$ SINGLE CRYSTALS

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In recent years halide perovskites have attracted great interest from the scientific community and have been considered a highly promising and attractive solution for next-generation photovoltaic device applications. Its interesting electrical, magnetic, and optical properties ally to low-cost synthesis technics led these structures to the state-of-the-art in optoelectronic materials [1,2]. In this work, synchrotron X-ray diffraction, Raman spectroscopy, and photoluminescence (PL) measurements were employed to monitor the changes in the structure and in the PL behavior of luminescent Cs$_4$PbBr$_6$ single crystals (SCs) under high-pressure conditions. The synthesized Cs$_4$PbBr$_6$ SCs were luminescent at ambient conditions, showing a strong greenish-light emission centered at 516 nm with a very narrow FWHM of 16 nm, in good agreement with previous reports [3,4]. High-pressure synchrotron X-ray diffraction (SXD) and Raman spectroscopy measurements were collected up to approximately 11.0 GPa. A phase transition at approximately 3.0 GPa from rhombohedral to monoclinic phase was determined by high-pressure synchrotron X-ray diffraction and Raman spectroscopy. At the monoclinic phase, the PL emission is completely suppressed, indicating that the monoclinic phase does not produce a favorable condition for PL phenomena in this structure. Unlike published for nanocrystals [5], in bulk Cs$_4$PbBr$_6$, the monocline phase (phase II) is stable in a very close pressure range. At 4.2 GPa, a new structure appears coexisting with phase II, being the transition completed at 4.7 GPa. The best refinement of phase III was obtained using a trigonal structure with space group P-1. Besides, adding value in the great debate about the origin of the green luminescence in the Cs$_4$PbBr$_6$ systems, our results from Raman mapping analyses suggest that the PL of the luminescent Cs$_4$PbBr$_6$ can be associated with the different size distribution of quantum dots or NCs of CsPbBr$_3$ embedded in Cs$_4$PbBr$_6$ SCs.

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KEYWORDS: PHOTOVOLTAIC COMPOUNDS, HALIDE PEROVSKITES, 0D CS4PBBR6 HALIDE PEROVSKITE, SYNCHROTRON X-RAY DIFFRACTION

ORAL PRESENTATION

STRUCTURAL-DEPENDENT PHOTOLUMINESCENT BEHAVIOR OF $CsPb_2(Br_{0.85}/I_{0.15})_5$

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Metal halide compounds are materials of great interest for optoelectronics because of their enormous potential in solar cells and light-emitting applications. Low-dimensional (0D, 1D, and 2D) metal halide with a variety of compositions present soft lattices allowing modular properties [1], however, the finding of new strategies to further improve such properties is a current research challenge [2]. In this scenario, the 2D perovskite-like $CsPb_2Br_5$ has attracted attention due to its structural stability and optical properties [3]. However, such properties have only been reported for nano or microsystems [4]. In this study, we present the $CsPb_2(Br_{0.85}/I_{0.15})_5$, a 2D perovskite-like structure in which a fractional substitution of Br- by I- ions induces a photoluminescence effect in single crystals at ambient conditions. This material remains the same 2D layered structure reported for $CsPb_2Br_5$, where $[Pb_2(Br/I)_5]$ layers are intercalated by Cs ions along the c axis. High-pressure optical and Synchrotron X-ray diffraction experiments reveal two structural phase transitions around 1 and 5 GPa, whereas the pressure-dependent PL experiments reveal a PL emission quenching occurring around 2.7 GPa. Further analysis of structural features reveals that the PL behavior is directly connected to structural changes observed upon pressure increase.

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ORAL PRESENTATION

STUDY OF INDUCED SPIN POLARIZATION OF Cu ATOMS IN Fe/Co/Cu_3Au(001): AN EXPERIMENTAL AND FIRST-PRINCIPLES INVESTIGATION

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Understanding the complex correlations structure-magnetism and the origins of the magnetic anisotropy in ultrathin magnetic heterostructures is an active research area in physics, given its great potential for new technologies in data storage and processing, and devices. In this work we investigated the induced magnetization of interfacial Cu atoms in ferromagnetic Fe/Co ultrathin films grown on a fcc Cu_3Au(001) substrate. To explore the interfacial nature of the induced magnetic moments and its origin, we probed two structures of five alternate Fe and Co monoatomic layers with either Fe or Co in direct contact with the non-magnetic Cu_3Au(001) surface. X-ray magnetic circular dichroism measurements at the L2,3 edges of Fe, Co and Cu allowed to extract the spin and orbital magnetic moments of Fe and Co in both structures, and to measure extremely low magnetic moments induced in Cu atoms. The samples were grown and probed in-situ at the PGM beamline. The same systems were also investigated by first principles calculations, in order to obtain the spin and orbital moments of the different atoms for both Fe-Co/Cu_3Au(001) stacks, disentangling the origin of magnetic proximity effects at these ferromagnetic/non-magnetic interfaces. The experimental and theoretical results reveal an induced magnetism only in atoms at the interface layer of the ordered Cu_3Au(001) substrate, i.e., the observed Cu XMCD signal is due to only 0.5 ML magnetic Cu atoms. Such results clearly demonstrate the importance of the structural, electronic, and magnetic interface reconstructions on the appearance of magnetic induced moments, which at the end determine the detailed behavior of the magnetic anisotropy.

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ORAL PRESENTATION

SURFACE COMPOSITION OF AEROSOL MODEL SOLUTIONS CONTAINING INORGANIC SALT AND AMINO ACID

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In atmospheric aerosol particles, the chemical surface composition dictates over both heterogeneous chemical reactions with gas-phase species and the ability to act as condensation nuclei for cloud formation. The pH in aerosol droplets can affect these properties, but it is challenging to measure in individual droplets. As a consequence, little is known about its influence on the particle's surface composition. This presentation explores how the surface composition of aqueous model solutions containing inorganic salt and amino acid changes according to the pH using photoelectron spectroscopy. In a submitted manuscript [1], we observe a variation by a factor of 4-5 of the relative distribution of inorganic ions at the surface of a liquid water jet as a function of the solution's pH and dependent on the solvated amino acid. The driving forces for the surface enhancement or depletion are ion pairing and charged layers close to the aqueous surface. Our findings apply to any aqueous interface at which organic species with charged functional groups are present.

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KEYWORDS: ENVIRONMENT, CLOUD FORMATION

ORAL PRESENTATION

SYNCHROTRON RADIATION-BASED X-RAYS FLUORESCENCE AND ABSORPTION SPECTROSCOPIES TO BOOST STUDIES OF AGRONOMIC BIOFORTIFICATION AND METAL SPECIATION IN TROPICAL SOIL ENVIRONMENT

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This abstract shows two possibilities of using synchrotron radiation (SR)-based techniques to boost studies in agri-environmental sciences. The first study aimed to assess elemental mapping of biofortified rice grains with selenium (Se), an essential element for humans, using SR-µXRF. Agronomic biofortification has been considered an efficient strategy for increasing Se contents in cereal grains. However, the spatial distribution of elements in grains is of great relevance, since important nutrients may be lost during the industrial processing (if they are located in external grain region). This analysis of mapping was carried in our study using SR-µXRF (at UVX machine in Brazil) and the spatial distribution of elements in the biofortified rice grains (via agronomic biofortification strategies) varied, with the Se being accumulated mainly in the endosperm (main edible part after the industrial processing) [1]. In the second study, XAS was used in combination with chemical extractions to assess Zn speciation and mobility in Zn-rich soils affected by mining and smelting activities [2,3]. Zn mobility in the tailing sample from the smelting area was much higher (>70%) than that for soils from the mining area (<1%). These differences in Zn mobility was related to the chemical Zn speciation in each site. EXAFS data were analyzed by linear combination fitting (LCF) in k-spacing (2 to 10 $Å^{-1}$) and, in the mining soils, LCF results showed high contribution of Zn species that significantly decrease Zn mobility (e.g., Zn-LDH, Zn-kerolite), while this metal in the tailing sample was mainly associated to weakly bound forms of Zn. In conclusion, SR-based techniques, such as µXRF and XAS, have shown to be powerful tools to assist in studies in agri-environmental sciences. These and other techniques (e.g., SR-XRD) may add substantial values/data to researches in these areas, and we hope that the application of such techniques may be amplified with the 4th generation SR (Sirius).

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KEYWORDS: BRAZILIAN SOILS, ESSENTIAL ELEMENTS, SELENIUM, CHEMICAL SPECIATION, SR-XRF, SR-EXAFS

Oral presentation

THE ACTIVE WOX SITES OF Mn-Na_2WO_4/SiO_2 CATALYST FOR THE OXIDATIVE COUPLING OF METHANE

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The Mn-Na_2WO_4/SiO_2 is the state-of-the-art catalyst for the oxidative coupling of methane (OCM) because it presents high stability (~500 h) and C2 hydrocarbon yield (14-27%). Several works have indicated the synergistic interactions between its components (i.e., WOx, NaOx, and MnOx) and proposed distorted WO4 tetrahedra as the active sites. However, these conclusions were drawn from characterizations obtained at room temperature, which is so far from typical high OCM temperatures (>750 ºC). In this sense, this work aims to establish a structure-activity relationship of the Mn-Na_2WO_4/SiO_2 catalyst for the OCM from experimental evidence collected at relevant reaction conditions. The mean oxidation state and the distortion degree of WOx sites present on trimetallic Mn-Na_2WO_4/SiO_2, bimetallic Na_2WO_4/SiO_2, and monometallic WO_3/SiO_2 catalysts were studied using X-ray diffraction (XRD) and in situ W LIII-edge X-ray absorption near edge structure (XANES) analysis. At room temperature, the XANES results indicate that the mean W oxidation state for all samples is near 6+. Furthermore, the white line shape of the XANES spectra shows that WOx sites are octahedrally coordinated on the monometallic catalyst and tetrahedrally coordinated on both tri- and bimetallic catalysts. These results are consistent with the crystalline phases identified by XRD (i.e., WO_3 for monometallic and Na_2WO_4 for bi and trimetallic catalysts). As increasing temperature, the change in the white line shape suggests a variation of the distortion degree of the WOx sites. For mono- and bimetallic catalysts, the distortion degree increased, while for trimetallic catalyst it decreased presumably due to the interaction with Mn atoms. Therefore, the superior OCM behavior of conventional trimetallic catalyst when compared with bi- and monometallic catalysts is associated with the presence of tetrahedrally coordinated and lowly distorted WOx sites.

Keywords: Oxidative coupling of methane, OCM, Mn-Na2WO4/SiO2, distorted WOx sites, in situ XANES

ORAL PRESENTATION

THE INFLUENCE OF CeO_{2-X} (0 < X < 0.5) PROPERTIES ON THE REACTIVITY OF Cu-CeO_{2-X} NANOPARTICLES TOWARDS THE CO OXIDATION REACTION

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Carbon monoxide (CO) is a poisonous gas, formed by incomplete combustion of organic matter. An efficient way to reduce its emission is by the use of an oxidation reaction, typically performed on expensive noble metals-based catalysts. In a previous work, CeO_{2-x} nanoparticles were synthesized controlling several electronic and structural properties [1]. When exposed to a CO atmosphere, these nanoparticles present a high oxygen vacancy population compared to a commercial CeO_{2-x} standard [1]. In order to study the influence of these properties on the reactivity towards CO oxidation reaction of Cu-CeO_{2-x} systems, Cu nanoparticles were mixed to the synthesized CeO_{2-x} nanoparticles and a commercial standard. The as prepared Cu-CeO_{2-x} nanoparticles were characterized by Transmission Electron Microscopy (TEM), Energy Dispersive Spectroscopy (EDS), X-Ray Diffraction (XRD) and X-Ray Photoemission Spectroscopy (XPS). After, the nanoparticles were heated to 400 °C under a CO atmosphere and then cooled to 250 °C or 150 °C where the CO oxidation reaction occurred. In situ X-ray Absorption Spectroscopy (XAS) and in situ time-resolved XAS measurements probed the Cu K and Ce L3 edges during the full thermal treatment applied. The reactivity of the samples was studied by time-resolved Mass Spectrometry measurements. XPS and EDS-line Scan High Resolution TEM (HRTEM) measurements of the samples after the reduction treatment showed that the Cu nanoparticles spread over the CeO_{2-x} surface during reduction treatment. Moreover, the higher the reactivity of the nanoparticles the higher Ce(III) fraction during the reduction treatment, the higher CuO fraction and the lower Ce(III) fraction during CO oxidation reaction. The reactivity of the samples is also influenced by the local atomic order around Cu atoms during CO oxidation reaction. Lastly, the samples containing the synthesized CeO_{2-x} nanoparticles showed an improved reactivity compared to the samples containing the commercial standard.

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ORAL PRESENTATION

VALENCE ELECTRON EXCITATIONS IN LiH INVESTIGATED BY IXS SPECTROSCOPY

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The simplicity of LiH regarding crystal structure and electronic configuration has established this compound as an ideal system for studying electronic structure and electron excitation in condensed matter while probing different theoretical approaches. The dielectric response of electrons, due to an external perturbation, can be probed by means of inelastic x-ray scattering (IXS) spectroscopy. The information about the excited electron system is obtained by measuring the energy-loss spectrum of scattered hard x-rays, while transferring energy ω and momentum q to the sample. In the present work the effects of electron-hole interaction in the dynamic structure factor S(q,ω) and in the complex dielectric function ε(q,ω) of valence electrons in lithium hydride at finite momentum transfer were investigated by means of IXS spectroscopy and ab initio theoretical methods. Experiments were carried out at the XDS beamline of the Brazilian Synchrotron Light Laboratory (LNLS). Calculations of S(q,ω) and ε(q,ω) were performed within time-dependent density-functional theory (TDDFT) in the the adiabatic local density approximation (ALDA) and many-body perturbation theory (MBPT) based on the Bethe-Salpeter equation (BSE). Our findings reveal that for low-q an explicit treatment of electron-hole interactions by using the BSE formalism slightly modify low-energy structures in S(q,ω) in comparison to ALDA results, but affects strongly the macroscopic dielectric functions. A very good agreement between experimental and theoretical S(q ,ω) and ε(q,ω) in all the range of investigated q values was achieved for calculations based on BSE after taking into account the full excitonic Hamiltonian. Present results [1] demonstrate the potential of approximations based on the Bethe-Salpeter equation to accurately describe the valence excitation spectra, including the near-onset region, and the dielectric response in insulating systems where excitonic effects are relevant.

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ORAL PRESENTATION

X-RAY PROTEIN CRYSTALLOGRAPHY REVEALS UNIQUE FUNCTIONS AND COMPONENTS IN A PHOSPHO-SIGNALING SYSTEM THAT CONTROLS METABOLISM AND VIRULENCE OF *MYCOBACTERIUM TUBERCULOSIS*

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Reversible protein phosphorylation has evolved as a ubiquitous molecular mechanism of protein regulation. Phosphorylation on serine (Ser), threonine (Thr) and tyrosine (Tyr) is central to bacterial physiology and pathogenesis, and the corresponding phospho-systems share similarities to those in eukaryotes [1]. However, as novel functions and components of bacterial O-phosphorylation are identified, distinct differences between pro- and eukaryotic phospho-signaling systems become apparent that could be exploited for drug development. In this presentation I am going to outline recent advances in the study of the signal transduction pathway that involves the Ser/Thr protein kinase PknG [2–4], which senses amino acid availability to control metabolism and virulence of Mycobacterium tuberculosis [5, 6] and has a conserved function in amino acid homeostasis in diverse Actinobacteria [7]. In addition, I will present views and objectives of PlaBEM, the Argentinian Platform for Metabolomics and Structural Biology, regarding X-ray protein crystallography projects. We offer access to our crystallization facilities, coordinate data acquisition sessions in synchrotrons, and organize courses/workshops aimed at training young scientists in integrative structural biology. The results to be present constitute the basis of future proposals for the use of the Manacá beamline of the Sirius synchrotron.

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KEYWORDS: SIGNAL TRANSDUCTION, AMINO ACID METABOLISM, ACTINOBACTERIA

POSTER PRESENTATION

3D-PRINTED MICROFLUIDIC SAMPLE HOLDER DEVICE FOR SERIAL SYNCHROTRON X-RAY CRYSTALLOGRAPHY

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X-ray crystallography is a technique with vast impact on structure-function relationships of biological processes. However, data acquisition so far has been limited by crystal size, fragility, and the need of cryo-cooling. Then, serial X-ray Crystallography emerged at XFELs facilities and recently is pushing protein structural analysis at synchrotron sources to a new avenue without its previous constraints. This approach brought to light the possibility of experiments under room temperature and with much smaller crystals at near-physiological conditions at synchrotron light sources. Anyhow, this new path comes with its own difficulties, as the expansion of sample holders for serial crystallography at synchrotron sources is still a challenge. Therefore, here we report the fabrication, simulation, and proof-of-concept of a 3D-printed microfluidic prototype sample holder for serial crystallography at Manacá beamline/LNLS-Sirius. The sample holder was built using Form 2 3D printer and flow rate experiments were performed using LC-20AD Pump; further, CFD simulations were conducted using ANSYS suite. Our work shows the use of a 3D in-flow geometry of three channels built to improve liquid diffusion. This device was applied in flow-focusing and mixing experiments, including multiple liquid-based mixtures, such as PEGs/water sample carrier media. Hence, the optimal model was obtained using a fork-like geometry, and its characterization was partially performed throughout computational simulations and pressure tests under high and low viscosity states. It has increased flow and mixing efficiency over the orthogonal-base junctions. The flow profile showed parabolic behavior with boundary condition of null velocity at the wall. Additionally, the computed velocity profile was not influenced by pressure or high viscosity media. Consistently, the particle diffusion path follows a laminar flow with particles stream remaining focused at the channel center with uniform velocity.

KEYWORDS: SERIAL CRYSTALLOGRAPHY, SAMPLE HOLDER, SYNCHROTRON X-RAY DIFFRACTION, MACROMOLECULES

POSTER PRESENTATION

A COMPACT SPECTROMETER BASED ON A SEGMENTED CONICAL CRYSTAL ANALYZER

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In this work, we present the commissioning of a spectrometer based on a crystal analyzer with an innovative geometry at the D09B-XRF beamline of the LNLS. The crystal's geometry is composed of several segments of conically bent Si(220) crystals with different radii [1]. Among its advantages are: 1.) It reaches an energy resolution of around 8 eV for the Mn K-alpha1 line, which is at least an order of magnitude better than the commonly used energy dispersive spectrometers; 2.) it allows for the simultaneous aquisition of a 2 keV window, from 5 to 7 keV, being adjustable by changing the crystal's parameters; 3.) it is compact, with a sample-detector distance of only 146 mm. This type of spectrometer may be suitable for different spectroscopic techniques, such as X-Ray Fluorescence, Resonant Inelastic X-Ray Scattering, or X-Ray Emission Spectroscopy.

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POSTER PRESENTATION

A MACHINE LEARNING APPROACH APPLIED TO DETERMINE FORMAL OXIDATION STATE OF 3D COMPOUNDS

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X-ray-absorption K-edge shifts of manganese, cobalt, and copper have been measured in different reference compounds at different structures and in different synchrotron beamlines in order to see if is possible using this edge shifts and machine learning methods to obtain information on the oxidation state of an unknown compound. In all cases, the shifts are the same sign, a fact that points to the absence of a significant uncompensated charge transfer from one elemental constituent to another. Identifying the edge shifts as core-level shifts, the Watson-Hudis-Perlman charge-compensation model is used on these systems, following the method proposed by Capehart et al [1]. We analyze the shift in energy from the pre-peak (taking E = 0; internal reference point) to fulfill a certain fixed area. Due to this method employ an internal reference point, it is independent on the beamline energy calibration. In our first results combining K-edge spectra of Mn, Co and Cu samples at LNLS, ALBA, ESRF and Spring-8, the energy shifts have similarities at the same formal oxidation state. The goal is to get a large number of K-edge spectra obtained from different light sources in order to propose a generalized statistical analysis that calculates the oxidation state of a sample with a certain confidence level using this methodology. This algorithm to calculates oxidation states in now tested with several spectra of references of 3d materials (from Ti-K to Zn-K) and is incorporated into a program that does the estimation independently on the light source and establish limits between which the method is reliable.

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KEYWORDS: XANES, MACHINE LEARNING, CLASSIFICATION ALGORITHM, FORMAL OXIDATION STATE

POSTER PRESENTATION

A NEW QUARTER WAVE PLATE INSTRUMENTATION FOR FAST SWITCHING THE X-RAY HELICITY

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X-ray magnetic circular dichroism (XMCD) is an important element selective technique used to obtain information about magnetic properties of complex materials. XMCD signal is obtained by the difference between two absorption spectra (XAS) of circularly polarized x-rays with opposite helicities from a magnetized sample [1-3]. At EMA beamline (Extreme condition X-ray Methods of Analysis) of Sirius, the change of polarization is controlled by an instrumentation based in quarter wave plates [3]. With this instrumentation the X-ray helicity can be changed as fast as 10 Hz. Taking advantage of the high photon flux of EMA, we propose an upgrade in the current instrumentation in order to increase the frequency of X-ray polarization switch, what would improve quality of XMCD signal. Preliminary tests were conducted in order to assess the current prototype stability in different frequencies and indicated that the maximum helicity shifting frequency is limited by three main factors: the quarter wave plates stability, the mechanical stability of the quarter wave plate chamber, and the control system of the piezoelectric actuator stage. With support of finite element simulations, here we will present a series of mechanical developments that we believe will increase the maximum frequency of helicity switch to at least 100 Hz. This development will allow the measurements of sample with very small magnetization and should open several opportunities of new studies for the Brazilian community.

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POSTER PRESENTATION

ACCELERATING LIGHT IN TWO-DIMENSIONAL CRYSTALS

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By synchrotron infrared nanospectroscopy, we demonstrate modulation of momentum and group velocity of subdiffractional hyperbolic phonon-polaritons (HP2), in hexagonal boron nitride nanocrystals, by varying the SiO_2 film thickness in the hBN/SiO_2/Au heterostructure. We reveal the acceleration of the HP2 pulse in a hBN/(SiO_2 wedge)/Au heterostructure with the gradient of the SiO_2 thickness. The acceleration is explained by semiclassical modeling considering the polariton pulse as a free quantum particle with effective mass dependent on its group velocity. In quantitative agreement with simulations and semiempirical analysis, the modeling predicts an average acceleration of 1.5 × 1018 m·s^{-2} close to that of ∼ 1.45 × 1018 m·s^{-2} obtained from experimental inputs. From the fundamental aspect, the polariton acceleration allows discussing the undulatory-corpuscular behavior of polariton quasi-particles. The acceleration induced by the wedge is a general effect that can provide for control of the polariton pulse dynamics, which is compelling for future polaritonic devices.

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POSTER PRESENTATION

ADSORPTION OF METHYLENE BLUE DYE IN AQUEOUS SOLUTIONS USING NiS_2-NiS NANOCRYSTALLINE COMPOSITE SYNTHESIZED BY MECHANOCHEMISTRY

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NiS2-Nis composite was successfully synthesized by mechanochemistry in argon atmosphere at room temperature starting from $Ni_{34}S_{66}$ powder mixtures. X-ray powder diffraction (XRPD) patterns showed that 9 h of milling are enough to allow the formation of the NiS_2 phase (83%), which is accompanied by a small fraction of the hexagonal NiS (17%) phase. Rietveld analysis indicates that the average crystallite size is about 22 nm for NiS_2 and 12 nm for NiS. Transmission Electron Microscopy (TEM) analyses confirms the nanometric size of the crystalline domains and reveal that the nanocrystals are agregated in large particles of hundreds of nanometers. The as prepared material slowly reacts with air through a complex phase transition scene, being mainly converted to nickel sulfate hexahydrate after long term storage (tens of months) as given by XRPD analyses using sincroton radiation and conventional diffractometer. The fresh and aged composite was used for the adsorption of methylene blue (MB) from aqueous solutions. The effect of different amounts of NiS_2–NiS nanocrystalline composite, adsorption time for dye removal and dye concentration were evaluated, as well as the effect of aging in the adsorption process. Both the fresh and aged samples showed good adsorption kinetics of MB from aqueous solution, and about 40% of the dissolved dye is adsorbed in less than 1 min when 1.88 g L^{-1} of adsorbent is used, reaching 94% after 120 min. Adsorption of MB was faster in the aged material probably due to its higher surface area when compared to the fresh sample.

POSTER PRESENTATION

AGING EFFECT ON VANADIUM OXIDE HYBRID NANOTUBES: A XANES STUDY

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Vanadium oxides present a rich magnetic phase diagram depending on the oxidation state of the V ions. In particular the vanadium oxide nanotubes (VOx NTs) are multiwall nanostructures constituted by alternating layers of VOx and a surfactant. The V-ions are in different oxidation states: +4 (3d1) and +5 (3d0). This coexistence of oxidation states, with different magnetic behavior, confers several promising perspectives for different technological applications for which it is essential to know the oxidation state of V ions, as well as to evaluate the stability with the aging time of the tubes. In this work we present a systematic study of the time evolution of the magnetic properties of VO x NTs. For this complete characterization, we used electron spin resonance (ESR) and dc-susceptibility techniques, which were supplemented with TEM microscopy. By X-ray absorption near edge spectroscopy (XANES) we calculated the amount of V in +4 and +5 oxidation state employing the Wong method. Wong and co-workers correlated the oxidation state and local environment of V compounds to spectral features of XANES signal [1]. In VOx NTs we observed that for aging in normal environmental conditions of pressure, temperature and humidity, the V4+ ions oxidize to V5+. Although the multiwall tubular structure is maintained, this oxidation process produces a marked change in the magnetic properties. We conclude that the aging of the samples affects the V4+/V5+ relationship in the VO x NTs, which may contribute to explain the significant dispersion of data reported in the bibliography [2].

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KEYWORDS: VANADIUM OXIDE NANOTUBES, XANES, AGEING

POSTER PRESENTATION

AN INFRARED NANOSPECTROSCOPY STUDY OF X. VESICATORIA XCV BV5–4A CELL ENVELOPE ALTERATIONS PRODUCED BY *P. FLUORESCENS* SF4C TAILOCINS

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The continuing global spread of antibiotic-resistant bacterial pathogens requires the development of alternative therapeutic strategies and bacteriocins are envisioned as an alternative [1]. Bacteriocins are peptides or proteins produced by bacteria that exhibit narrow or broader activity spectra. *Pseudomonas fluorescens* SF4c produces tailocins, a phage-tail-like bacteriocins with antimicrobial activity against *Xanthomonas vesicatoria* Xcv Bv5-4a, a phytopathogenic strains responsible of bacterial-spot disease in tomato. We demonstrated, that tailocins adhere to the cell envelope of strain Xcv Bv5-4a and cause the cellular death [2]. Although it has been proposed that the action mechanism involve the pore formation in the membrane, direct structural evidence of the damage at the molecular level is missing. Here we report studies using a high resolution microscopy (AFM) in combination with a spectroscopic technique (IR scattering Scanning Near-Field Optical Microscopy, IR s-SNOM) to obtain information of surface topography simultaneously with opto-vibrational information in the near field overcoming the Abbe's diffraction limit of spatial resolution. Besides the morphological differences observed between individuals X. vesicatoria Xcv Bv5-4a cells untreated (control) and treated with tailocins of P. fluorescens SF4c; the principal component analysis (PCA) computed on the set of all acquired spectra allows to differentiate between the treated and control cells. The region from 1800 to 900 cm^{-1} assigned mainly to vibratory modes related to membrane phospholipids, v C=O of lipids (1755 cm^{-1}) and vas P=O of phosphate groups (1240 cm^{-1}) are modified, and can be associated to alterations in the structure of the cell envelope of Xcv Bv5-4a cells when are treated with tailocins. The results demonstrate that IR s-SNOM facilities available at the LNLS enable to study the biochemical nature to single bacterium level at a nanoscale, without any special sample preparation.

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KEYWORDS: PSEUDOMONAS, XANTHOMONAS, BACTERIOCIN, TAILOICIN, BIOCONTROL, AFM, NANO-FTIR

POSTER PRESENTATION

ANALYSIS OF ORMOSIL FILMS BY SYNCHROTRON RADIATION

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Organically modified silicates (ormosil) are systems that possess flexible structures, which allows occlusion of diverse species (ions, molecules, clusters, etc.), contributing to the development of multifunctional nanomaterials with many applications, as for example: coatings, membranes, catalysts, photocatalytic films, optical/photochromic/photonic devices, dosimeters, among others. Ormosil thin films containing phosphotungstic acid (H3PW12O40/PWA) doped with TiO_2 nanoparticles, Zn^{2+} and earth alkaline ions (Mg^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}) were analyzed by Synchrotron Radiation X-ray Fluorescence in Total Reflection (SR-TXRF) and Grazing Angle (SR-GRXRF) modalities, in order to correlate their composition and properties [1-3]. The advantages of analysis of TXRF condition are:1) Self-absorption is almost negligible; 2) Detection limits are improved by decrease the background scattering; 3) In optical flat supports, the X-rays are almost totally reflected. At Grazing Angle X-ray Fluorescence (GRXRF), the X-ray incident beam is almost parallel to the surface of the sample, at angles equal or slightly larger (2-3 times) than the value of the critical angle of sample’s support. GRXRF is very useful for analysis of quasi-homogeneous samples (e.g. films), also avoiding significant self-absorption effects. Synchrotron Radiation X-Ray Fluorescence at modalities of Total Reflection (SR-TXRF) and Grazing Angle (SR-GRXRF) have higher sensitivities than conventional TXRF and GRXRF (laboratory spectrometers) respectively due that: 1) The X-ray intensity of Synchrotron beam is increased by 3-5 orders of magnitude compared to the conventional X-ray tubes. 2) Synchrotron radiation is suitable for energy tuning. 3)The spectral background is minimized because of the polarization, improving the detection limits compared with obtained by TXRF and GRXRF from laboratory [4]. SR-TXRF and SR-GRXRF demonstrated be suitable methods for elemental analysis of ormosil films.

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KEYWORDS: SYNCHROTRON RADIATION X-RAY FLUORESCENCE IN TOTAL REFLECTION MODE (SR-TXRF) SYNCHROTRON RADIATION X-RAY FLUORESCENCE IN GRAZING ANGLE MODE (SR-GRXRF)

POSTER PRESENTATION

ANNULAR TOMOGRAPHY: A NEW METHOD FOR 3D SURFACE INFORMATION

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X-ray Computed Tomography has shown to be a powerfull technique to mapping sample bulks, specially when using synchrotron sources with more advanced contrast like x-ray fluorescence (XFCT) [1]. However, some applications may require specific data acquisition in which part of the sample is either out of reach or should be avoided to minimize dose. In this sence, we present a new type of tomography, named Annular Tomograhy, in which only part of the sample is irradiated for tridimensionally imaging but also leaving the other part of the sample untouched by the x-ray beam. This strategie not only may reduce the radiation dose on sensitive samples, but would also avoid overexposure on XFCT of light elements on thick samples. On this work we propose the use of the Simultaneous Iterative Reconstruction Technique (SIRT) available at the ASTRA toolbox [2] for modeling and tomographic reconstruction. More specifically this implementation aims to adress some specificities of the Carnauba beamline regarding its working energy, resolution, acquisition time and available instrumentation.

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KEYWORDS: FLUORESCENCE COMPUTED TOMOGRAPHY, ALGEBRAIC RECONSTRUCTION, SIRT

POSTER PRESENTATION

ARE DISULFIDE BONDS RESILIENT TO DOUBLE IONIZATION? INSIGHTS FROM COINCIDENCE SPECTROSCOPY AND AB INITIO CALCULATIONS

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Disulfide bonds (–S–S–) are commonly present in biomolecules and have also been detected in astrophysical environments. In this work, the stability of the disulfide bond towards double ionization is investigated using quantum chemical calculations and photoelectron photoion photoion coincidence (PEPIPICO) spectroscopy measurements on the prototype dimethyl disulfide (CH_3SSCH_3, DMDS) molecule. The experiments were performed using high energy synchrotron radiation photons before (2465.0 eV) and at (2470.9 eV) the first sigma resonance around the S 1s edge. We applied the multivariate normal distribution analysis to identify the most plausible ionic fragmentation mechanisms from the doubly ionized DMDS. By mapping the minimum energy structures on the dicationic $C_2H_6S_2^{2+}$ potential energy surface, we show that disulfide bonds are only present in high-lying isomers, in contrast to their analogous neutral systems. Our results also indicate that the number of fragment ions containing a disulfide bond for both photon energies is negligible. Taken together, our results reveal that the disulfide bond is severely damaged as a consequence of sulfur core–shell ionization processes, due to the lowering of its thermodynamic stability in multiply-charged systems.

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POSTER PRESENTATION

AUTOMATION OF DFT CALCULATIONS FOR SUPERCONDUCTORS

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The discovery of a superconductor material in ambient temperature would be revolutionary for all electric technologies; since these materials have zero resistance, there would be no loss of energy by Joule effect. The theoretical calculation of the critical temperature (Tc) is essential to the prediction of superconducting, allowing the analysis of both known and theoretically new structures seeking high Tc. For that, ab initio simulations are used via DFT (Density Functional Theory) to calculate matrix points of electronically-phonon coupling in a specified crystal structure. The Quantum ESPRESSO (QE) package is an integrated open-source suite used for this purpose. Although this great tool is available, it is a difficult process for superconductivity calculation, manual in many objective steps that did not depend on the researcher's judgment. In addition, it was subject to numerous typing errors. Because of these, the preparation time of the several input files was long, and could cost a whole day for only one compound. It was developed a software solution to automate the Tc calculation with python. It provides a friendly platform in command-line with the possibility of one only input: a crystal structure file. It is optional to include a parameter unique file to modify the standard QE variables predetermined in the program. The software allows to create new calculation routines and expand functionalities. It was added a phonon spectra calculation in gamma, this allows to easily determine the modes and frequencies of phonon vibration of a compound. It is also possible to obtain a list of cell structure compressions and equivalent cell energies, this set of points allows an state equation estimation, which would be extremely useful in any experiment in function of pressure. Structures obtained from x-ray diffraction data may be used as input for the program and calculate various properties via DFT, aiding experiment planning and result interpretation.

KEYWORDS: QUANTUM ESPRESSO, SUPERCONDUCTIVITY, DFT

POSTER PRESENTATION

BA-DOPING EFFECTS ON STRUCTURAL, MAGNETIC AND VIBRATIONAL PROPERTIES OF DISORDERED La_2NiMnO_6

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Barium-doped La_2NiMnO_6 double perovskites have been investigated in order to elucidate the role of the B-site doping on its magnetic, dielectric and vibrational properties. In all cases, we have obtained a non- linear dependence with the Barium content signaling that a 5% molar doping renders the maximum B- site ordering. By analyzing a series of gradually doped samples, we have found that the effective magnetic moment in the B-site can be enhanced by a factor of nearly 100% by 5% Ba doping. In order to exclude oxidation state induce changes, XPS and XANES measurements have been carried out concluding that nickel or manganese oxidation states ratio are not modified by Ba doping. Raman spectroscopy correlates Ba doping with vacancies thus attributing to the latter the role of primary source for structural defects in $BaLa_2NiMnO_6$ samples. This manuscript summarizes key consequences of the B-site ordering which are crucial to address the optimization of the magneto-electric coupling in Ba-La_2NiMnO_6 double perovskites.

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KEYWORDS: DOUBLE PEROVSKITE, MAGNETODIELECTRIC, STRUCTURAL ORDERING

POSTER PRESENTATION

CATALYTIC ACTIVITY OF SILVER LOADED ZEOLITES FOR SELECTIVE OXIDATION OF AMMONIA: INFLUENCE OF SILVER SPECIATION

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This work is dedicated to the study of the agglomeration of silver species after hydrogen activation treatments using small-pore zeolites exchanged with silver as a precursor. The influence of the reduction temperature of the material on the formed metallic species using in situ or ex situ characterization methods have been studied. Next, the catalytic consequences of catalysts containing different Ag species in the reaction of SCO-NH_3 (1-3) are discussed. It has been shown that the particle size of the silver species has a fundamental role in the oxidation of ammonia. Specifically, silver species with larger particle sizes (bulk) have better results once they are stable against metal redispersion under the oxidizing conditions of the SCO-NH_3 reaction. Finally, a multipurpose catalytic cell, able to combine XAS+IR, will be proposed (presented) for challenging catalytic experiments to be performed at Sirius.

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KEYWORDS: SILVER NANOPARTICLES, ZEOLITES, XAS

POSTER PRESENTATION

CHEMICAL CHARACTERIZATION OF LYCOPSID FOSSILS FROM TWO DIFFERENT TYPES OF LITHOLOGIES (CORUMBATAÍ FORMATION, ANGATUBA, SP) USING SYNCHROTRON MICRO-XRF

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Fossil lycopsid are common in Corumbataí Formation (Permian Period, Paraná Basin, Brazil). We know a lot regarding their taxonomy, anatomy and some aspects of fossilization process [1,2] But we still lack valuable data about fossildiagenesys (which affects directly paleoenvironment and paleoecological interpretations) [3,4]. In this work, we applied synchrotron micro-XRF to investigate chemical composition between lycopsid preserved in two different lithologies: silicate carbonate and siltstone. Besides the different lithologies of preservation, micro-XRF data showed that lycopsid shared similarities in chemical information that lead us to hypothesized some influence of anoxia or euxinia during fossilization: supported by the detection of intensities of Iron (Fe) and Sulphur (S) in both samples [5,6]. In addition, the presence of Titanium (Ti) and Nickel (Ni) could indicate the action of microorganisms (during the preservation process or weathering?). This chemical characterization can helps us to understand the retention of important information during fossilization. We are still running other complementary analysis (e.g. micro-Raman and MEV-EDS) in order to investigate the relationship between the those chemical information and the fossilization environment of lycopsids.

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POSTER PRESENTATION

CHEMORESISTIVE BUTANONE SENSOR BASED ON Pt-LOADED ZnO TWIN-RODS

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The detection of volatile organic compounds (VOCs) is of major concern due to their impact on human health and the environment. VOCs monitoring can be conducted by using chemoresistive sensors based on semiconducting metal oxides (SMOx) [1]. Although SMOx present satisfactory sensing-properties, such properties are boosted when decorating SMOx with noble metals [2]. Herein, ZnO twin-rod structures were prepared then decorated with Pt nanoparticles (NPs). The VOC-sensing properties of the Pt/ZnO structures were evaluated by measuring the changes in the resistance after the exposure of the VOC [3]. Transmission electron microscopy (TEM) images revealed the twin-rod like morphology of ZnO in addition to the presence of Pt NPs on the ZnO surface. High-resolution TEM, selected area electron diffraction (SAED), and X-ray photoelectron spectroscopy (XPS) confirmed the metallic state of Pt NPs. The VOC-sensing tests showed that all the sensors based on Pt/ZnO heterostructures and pure ZnO were more sensitive to butanone than the other VOCs at 450 °C. However, a 2wt% Pt loading greatly enhanced the sensitivity and selectivity compared to ZnO. Moreover, the baseline resistances of all the Pt/ZnO heterostructures were much higher than that of ZnO in the whole temperature range (150-500 °C). X-ray absorption near edge structure (XANES) spectroscopy was conducted in transmission mode at the Pt L3 edge at the XAFS2 beamline at LNLS. The spectra of the 5wt% Pt/ZnO heterostructure (recorded from 150-500 °C under air flow) revealed that the white line increased and shifted to higher energy values with increasing the temperature, suggesting the oxidation of the Pt NPs. XANES was conducted under more realistic conditions than the ex-situ characterizations (TEM, SAED, XPS), including ambient pressure, presence of oxygen, and temperature. Therefore, combining XANES with the baseline resistance measurements, a mechanism based on the PtOx/ZnO heterojunction was proposed.

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KEYWORDS: SENSORS, ZINC OXIDE, PLATINUM, INTERFACES, XANES

POSTER PRESENTATION

CLINICAL REDIRECTION OF INDO CARRIED AS AN INTEGRAL AND SUPPORTING ANTI-CANCER DRUG IN THE CHEMOTHERAPY TREATMENT OF CANCER

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Indomethacin (INDO) has a mechanism of action based on the inhibition of cyclooxygenase activity of greasy acids during the process of inflammation. Then, its mechanism of action would be related to a possible anticancer activity, however its high toxicity in normal tissues has hindered therapy. The intercalation of the drug in hybrid matrix of Layered Double Hydroxides (LDH) would reduce these unwanted effects by promoting chemotherapy redirection. In this way, a set series of LDH, by using magnesium (Mg) and aluminum (Al) as metals, were synthesized in order to intercalation INDO particles with aging time of 8, 16, 24, 48 and 72 hours; temperatures of 50, 70, and 90 °C; and maintaining the pH value close to 10 and constant. The product was characterized by experimental methods such as: X-ray Diffraction (XRD), Spectroscopy of Fourier Transform infrared (FTIR), Scanning Electron Microscopy (SEM) Absorption Spectroscopy in ultraviolet and Visible (UV-VIS); and Thermogravimetric Analysis and Differential (TGA/DTA). The obtained results, the direct influence of temperature and aging time of particle under constant pH coprecipitation method 10, show the intercalation of INDO on LDH matrices. Moreover different levels of drug were obtained with interleaving time 16h-50 °C, which was the as best condition for intercalation. In vitro results were carried out to prove the potential of cell viability and anticancer activity of the sample LDH-INDO (16h 50ºC) in gastric cancer cell lines, breast cancer, melanoma, lung fibroblast and not neoplastic gastric tissue by MTT assay, it shows the was inhibition of cell proliferation, demonstrating higher and lower toxicity against flowing lines and melanoma, respectively. Based on all the experimental results, we propose a new and unprecedented clinical redirection INDO carried from as anticancer drug and integral adjunct in chemotherapy to cancer.

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POSTER PRESENTATION

COMPARATIVE STUDY OF THE INTERACTIONS BETWEEN FUNGAL TRANSCRIPTION FACTOR NIT-2 WITH MAMMALIAN AND FUNGAL IMPORTIN-ALPHA

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In the classical nuclear import pathway, proteins containing nuclear localization sequences (NLSs) are imported into the nucleus by the importin-α/β heterodimer. Importin-α (Impα) contains the nuclear localization signal (NLS) binding site, while importin-β mediates transport across the pore of the nuclear membrane. Herein, we studied how the NLS from NIT-2 transcription factor of fungus interacts with Impα from *Mus Musculus* and *Neurospora crassa* aiming to understand the specificity of this interaction. N-terminally truncated Impα from *M. Musculus* (MmImpα) and *N. crassa* (NcImpα) were expressed and purified. MmImpα/NIT-2 NLS complexes were co-crystallized and X-ray data collection were collect at Brazilian Synchrotron Light Laboratory followed by data processing, structure elucidation, modeling and refinement. Calorimetric experiments were performed using a MicroCal ITC200 and processed using Origin software. The NIT-2 NLS sequence binds with high affinity to the Impα major binding site from both organisms, but its binding to minor binding sites reveals interesting differences due to the presence of additional interactions of NIT-2-NLS with MmImpα, which corroborates the calorimetric data [1,2]. The higher affinity of N. crassa NIT-2 by MmImpα instead of NcImpα strongly indicates that the major binding site is the site used for the translocation of NIT-2 protein to the nucleus, and we hypothesized that the differential affinity for NLSs at the minor site may be a useful strategy for organisms that only have one Impα isoform to selectively recognize and transport different NLSs.

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KEYWORDS: NUCLEAR IMPORT, CRYSTALLOGRAPHIC STRUCTURES, IMPORTIN ALPHA

POSTER PRESENTATION

COMPETITION BETWEEN THE SHAKE-OFF AND KNOCKOUT MECHANISMS IN THE DOUBLE AND TRIPLE PHOTOIONIZATION OF THE HALOTHANE MOLECULE ($C_2HBrClF_3$)

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The ratios of single, double, and triple ionizations to the total photoionization of the halothane ($C_2HBrClF_3$) molecule have been investigated by a single-photon ionization in the energy range from 21.21 to 320 eV. In the valence region, the multiple ionization results can be described by a sum of contributions generated from the shake-off and the two-step one models. At low photon energies (from the threshold of triple ionization up to 100 eV), the triple photoionization dynamics of halothane can be reasonably well described by a model involving a classical electron impact double ionization of the singly ionized parent ion.

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KEYWORDS: SHAKE-OFF, KNOCK-OUT, DOUBLE PHOTOIONIZATION, TRIPLE PHOTOIONIZATION

POSTER PRESENTATION

CONFORMATIONAL STATES OF LMR-47 A THROMBIN-LIKE ENZYME FROM *L. M. RHOMBETA* VENOM DETERMINED BY MOLECULAR DYNAMIC STIMULATION WITH SMALL-ANGLE X-RAY DATA

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Thrombin-like enzymes (TLEs) are important components of snake venoms due to their involvement in coagulopathies occurring on envenoming. Structural characterization of this group of serine proteases is of utmost importance for better understanding their unique properties. However, the high carbohydrate content of some members of this group prevents successful crystallization for structural determination. Circumventing this difficulty, the structure of Lmr-47 [1], a highly glycosylated TLE from Lachesis m. rhombeata venom was studied in solution. At pH 8.0, where the enzyme displays maximum activity, Lmr-47 has a radius of gyration (Rg) of 37 Å and a maximum dimension (Dmax) of 130 Å as measured by small-angle X-ray scattering (SAXS) and a Stokes radius (SR) of 50 Å according to dynamic light scattering (DLS) data. At the naturally more acidic pH (6.0) of the L*. m. rhombeata* venom Lmr-47 behaves like a more compact particle as evidenced by SAXS (Rg = 27.9 Å and Dmax = 82 Å) and DLS (SR = 30 Å) data. In addition, Kratky plot analysis indicates a rigid shape at pH 8.0 and a flexible shape at pH 6.0. On the other hand, the center of mass of intrinsic fluorescence was not changed while varying pH, possibly indicating the absence of fluorescent amino acids in the regions affected by pH variation. Circular dichroism experiments carried out with Lmr-47 indicate a substantially random coiled secondary structure that is not affected by pH. A low-resolution model of Lmr-47 presented a prolate elongated shape at pH 8.0 and a U-shape at 6.0. Lmr-47 tertiary structure at pH 6.0 was maintained on heating up to 52°C and was completely lost at 75°C. The possible existence of two pH-induced folding states for Lmr-47 and its importance for the biological role and stability of this enzyme was discussed.

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KEYWORDS: LACHELIS M. RHOMBEATA, SNAKE VENOM, SERINE PROTEASE, SVTLE, SOLUTION STRUCTURE, PH-INDUCED CONFORMATIONAL CHANGES, PROTEIN SHAPE

POSTER PRESENTATION

CONTRAST-ENHANCED NANO-CT DENTAL SOFT TISSUES AND CELLULAR STRUCTURES CHARACTERIZATION

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Micro- and nano-CT technologies offer 3D imaging methods, which allow high-resolution examination of bones and teeth, but soft tissue components have weak X-ray attenuation and are not easily visualized in CT images [1]. We introduce a new methodology designed to visualize the dental ultrastructure, including soft tissue components, by utilizing phosphotungstic acid (PTA) as a contrast-enhancement agent. This allowed the three-dimensional imaging of dental cementum and periodontium as well as interior soft tissue components, such as odontoblasts and predentine, with high resolutions. The thickness of the cementum could be computed over the height of the tooth made possible by the PTA-enhanced contrast, and the attached soft tissue components of the interior of the tooth could be shown on the dentine-pulp interface in great detail. Three-dimensional illustrations allowed a histology-like visualization of the sections in all orientations with a single scan and easy sample preparation. Furthermore, the dental tubules, with the characteristic sigmoid curvature, could be visualized. The segmentation of the tubules and the surrounding dentine allowed a three-dimensional investigation of the dentine composition, such as tubular lumen or ratio of tubular lumen area to dentinal surface. The developed methodology may enable new important findings in the field of dental sciences at the interphases between soft and hard tissue, particularly related to endodontic and periodontal research. The possibility of performing this analysis using synchrotron radiation phase-contrast is the next step of this study. We expect this to allow for the identification of the soft tissues attached to the calcified ones keeping the samples in its most natural state, without the chances of structural changes in its structures due to possible alterations caused by the staining solution.

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KEYWORDS: CONTRAST-ENHANCED MICROCT, COMPUTER TOMOGRAPHY, ODONTOBLAST, CEMENTUM

POSTER PRESENTATION

COPPER-MANGANESE CATALYSTS FOR THE METHANOL SYNTHESIS

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Layered double hydroxides (LDH) are minerals from the family of anionic clays. Here, we show a partial result, which is the synthesis of methanol, an intermediate in DME production, an important raw material [1], using catalysts prepared from LDH. MnCuAl LDH precursors were prepared by a co-precipitation method, using Na free reactants. The ratio of Al was fixed at 0.19 and the Cu/Mn ratio at 0.5 to 2.0. For Cu/Mn = 0.5, two pH values were used (6.5 e 8.0). The catalysts were obtained by calcining the precursors at 400 ºC and then reducing them with H_2 at 265 ºC. With the use of the in-situ XRD (XPD beamline of LNLS) it was possible to see the disappearance of the poorly crystalline CuO-like phase in the calcined materials and the appearance of a metallic Cu phase in reduced ones. Transition begins near the end of the temperature ramp (265 ºC) and metallic Cu stays well defined and crystalline during the temperature plateau (for 40 min). X-ray absorption spectroscopic characterization (XANES and EXAFS) were performed on the Cu K-edge at the XAFS2 beamline of LNLS. In the calcined material with pH 8 and Cu/Mn = 0.5, copper appeared to be more electro-deficient than in CuO, probably because of the presence of manganese at a higher oxidation state and/or Al3+ in its vicinity or due to its existence as a defective nanocrystalline phase. In this catalyst, copper appeared to be fourfold coordinated by oxygen probably in a planar configuration, with considerable structure disorder from the second coordination sphere onwards. In the reduced catalysts, no difference in the electronic properties of copper between catalyst and a copper foil standard were observed. The catalyst with the highest manganese content and prepared at higher pH, presented the highest methanol productivity and selectivity. This was credited to the larger surface area of its oxide precursor, which resulted in smaller Cu crystallites and larger copper surface area.

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KEYWORDS: METHANOL SYNTHESIS, LAYERED DOUBLE HYDROXIDES, CUMNAL

POSTER PRESENTATION

CUBIC-TO-INVERTED MICELLAR AND THE CUBICTO-HEXAGONAL-TO-MICELLAR TRANSITIONS ON PHYTANTRIOL-BASED CUBOSOMES INDUCED BY SOLVENTS

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Cubosomes are composed of a mixture of specific lipids with the ability to self-associate, such as phytanthriol (PHY), and polymers that act as a stabilizer, such as poloxamer (F127). [1] These nanoparticles have a high hydrophobic volume, approximately 50%, which makes them promising vehicles for drug delivery of hydrophobic molecules. A challenge for incorporating molecules into nanoparticles is the use of organic solvents in the process. [2] In this study, we investigated the structural influence of four different solvents (acetone, ethanol, chloroform and octane), using low-angle X-ray scattering and cryogenic electron microscopy techniques, aiming to help choose the most appropriate solvent to charge the drug in the cubosome. In the presence of chloroform and acetone, transitions from cubic to micellar phase were observed. Chloroform and octane have different effects on PHY-based cubosomes compared to acetone and ethanol, both of which induced a hexagonal phase transition. These effects are associated with the interaction of the solvent in the hydrophobic phase of the cubosomes, increasing their volume. After the 24-hour incubation period, interesting structural changes were observed in the samples, compared to the freshly prepared ones.

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KEYWORDS: CUBOSOMES, SAXS, SOLVENTS, ORGANIC SOLVENTS, CRYO-TEM, NANOPARTICLES, PHYTANTRIOL, PHASE DIAGRAM, EFFECTS OF SOLVENTS IN NANOPARTICLES

POSTER PRESENTATION

CuO-NiO/CeO_2-ZrO_2-Sc_2O_3 NANOMATERIALS WITH MIXED IONIC-ELECTRONIC CONDUCTIVITY FOR SOFC APPLICATIONS: IN-SITU XANES CHARACTERIZATION

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The development of anode materials for Intermediate Temperature Solid Oxide Fuel Cells (IT-SOFC, 500-700°C) requires tailoring nanomaterials to enhance mixed ionic-electronic conductivity and electrocatalytic activity. The great density of surface defects plays a key role as active sites for fuel oxidation reactions in these nanomaterials. In particular, nanostructured CeO_2-ZrO_2 (CeZr) exhibits electronic conductivity in reducing atmospheres and a high oxygen mobility in the lattice that contribute to suppress carbon formation and/or sulfur present in the fuel. The aim of this work is to study the effect of an aliovalent dopant, Sc^{3+}, on oxygen vacancy generation in the CeZr lattice and its impact in the kinetics of ceria reduction of CuO-NiO/CeO_2-ZrO_2-Sc_2O_3 cermets with different fuels (H_2 or CH_4). Samples were synthesized using the citrate complexation route (CIT) with the following compositions: $Ce_{0,9}Sc_xZr_{0,1-x}O_\delta$, with $0<x<0.1$. Selected compositions were impregnated with CuO-NiO and heated at 500 °C and 1000 °C. In-situ Dispersive X-Ray Absorption Spectroscopy (DXAS) experiments were performed at the Ce LIII-edge of the D06A-DXAS beamline of the LNLS. Temperature programmed reduction (TPR) experiments were performed by heating the samples in a flow of diluted H_2 or diluted CH_4 from room temperature to 800°C. Results indicate that CeZrSc samples exhibit higher Ce reduction values than CeZr and CeSc in both H_2 and CH_4 atmospheres, indicating that oxygen vacancy generation through Sc^{3+} doping promotes Ce reduction, especially, when CuO-NiO is added to the system. In addition, the resistance to C-poisoning was evaluated by an ex-situ treatment of the samples with natural gas, followed by a regeneration process in $5\%O_2/N_2$. In-situ H2-TPR results of these samples indicate a shift on the light-off temperature of Ce reduction toward higher temperatures compared to fresh samples. However, at T>750°C higher total Ce reduction values were achieved in regenerated samples.

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KEYWORDS: CEO2, TPR, XANES, SOFC

POSTER PRESENTATION

CURRENT DEVELOPMENT OF INSTRUMENTATION FOR IN SITU AND IN VIVO EXPERIMENTS AT THE CARNAÚBA BEAMLINE

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Carnaúba was designed to be a multi-technique beamline, capable to operate from 2.05 to 15 keV, exhibiting a precisely focused beam from 100 - 500 nm at the Tarumã station. It will be possible to carry out studies on cells, bacterias, biominerals, materials for energy storage and harvesting, taking advantage of coherent imaging techniques (CDI), nanofluorescence (nano-XRF), nano X-rays excited optical luminescence (nano-XEOL), nanoabsorption (nano-XAS), and nanodiffraction (nano-XRD). One of the prerequisites for in situ experiments at the Trumã is to work with a small amount of samples (solid or liquid) and optimized sample holders. In this presentation, I will give an overview of what we are developing for in situ and in vivo experiments involving electrochemistry, microfluidics, catalysis, heating, and controlled atmosphere (gases). Thus, it is a great opportunity to hear, discuss, and receive suggestions from users about in situ experiments in complex sample environments.

POSTER PRESENTATION

DESIGN OF A DOUBLE CRYSTAL DEFLECTOR FOR XRR STUDIES AT SAGUI BEAMLINE

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X-Ray Scattering, Diffraction and Reflectivity studies of liquid interfaces are becoming well established at Synchrotron Light Sources, allowing the atomic-molecular characterization of new materials in many fields, such as electronics, and pharmaceutical and food industries [1]. However, one of the biggest challenges in the application of these techniques is the variation of the incidence angle of the light beam onto the sample plane, due to the inability of tilting neither the source nor the sample. In this scenario, a specialized instrumentation is being developed at LNLS in order to deflect the incoming beam to the sample surface with high precision. A proof-of-concept prototype in which the liquid surface stays stationary over the whole angular variation was developed and tested, granting the functionality of the deflection device. Now, a new version is being designed to account for the particular requirements of the SAGUI beamline at Sirius. The deflection system uses two crystals with its respective planes of incidence perpendicular to their optical surfaces, positioned like a double crystal monochromator (DCM) with different crystals (eg. Ge[111] and Ge[220]). The sequential Bragg reflections from the double crystal system allows the incident angle at the surface to be varied, while the measuring position remains centered and stationary at the sample surface. Similar deflection systems were implemented on beamlines ID15 and ID10 at ESRF, P08 at PETRA III and I07 at DIAMOND [2], yet this is a new design developed to perfectly fit the existing 6+2 circle diffractometer of the SAGUI beamline, in a way that it can conveniently be used for grazing-incidence X-ray diffraction (GIXD) and grazing incidence small-angle X-ray scattering (GISAXS) in combination with the XRR measurements.

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KEYWORDS: INSTRUMENTATION, XRR, LIQUID INTERFACES

DEVELOPMENT OF A PYTHON-BASED APPROACH AIMING CONTROL AND DATA ANALYSIS FOR HIGH-RESOLUTION X-RAY DIFFRACTION EXPERIMENTS

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Data treatment and setup movement control are important aspects that can improve the success rate of an experiment significantly. A robust and seamless processing of the detected signals into results with a physical meaning leads to a fast understanding of the object of study as well as a decision-making support. Similarly, and friendly and intuitive interface control for experiments that rely on both samples and detectors movement can impact significantly overall experience and optimize the usage of allocated beamtime. Reciprocal Space Maps [1] is one example that could take advantage of such an approach when using the available area detectors at the EMA beamline. In a few words, it requires the transformation of the data from the laboratory-space (angles) into the reciprocal space (Q vector). On top of that, thee experiment controlling tool would also be able to move and probe distinct directions of the reciprocal space depending on the sample features. All of this process has the potential to be performed "under the hood", making it easier for users to control the experiment and have their results already during the beamtime. With this in mind, here is presented some of the progress regarding the customization and integration of python-based tools focused on single-crystals and epitaxial thin films XRD experiments. Will be discussed the current strategy to replace the known beamline controlling software SPEC [2] into Python libraries (such as xrayutilities [3]), particularly for the case of the (4S+2D) Huber diffractometer available at EMA. On the data treatment side, some of the solutions in dealing with non-irregularly spaced data during the development of a 3D-RSM results are also shown. Furthermore, users scripts (or "macros") may also be compatible with this environment by using a popular scientific programming language as Python, at the same time it allows constant software development by the LNLS staff.

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KEYWORDS: PYTHON, RECIPROCAL SPACE MAP, XRD

POSTER PRESENTATION

DEVELOPMENT OF AN AUTOMATIC DATA PROCESSING PIPELINE FOR SERIAL CRYSTALLOGRAPHY ON MANACA BEAMLINE (SIRIUS)

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The recent development of fourth generation synchrotron light sources brought a number of challenges for science and engineering. With regard to data processing, the need for updating data in real time becomes a critical point for immediate verification of the results and quality of the experiment. Therefore, automated and user-friendly platforms are necessary to optimize the beam time per experiment. This work aims to automate the data processing of Sirius' MANACA (MAcromolecular Micro and NAno CrystAllography) beamline. The beamline will be dedicated to crystallography techniques, through which it is possible to visualize the physical chemical structure of macromolecules that make up biological structures, and also small molecules. At MANACÁ, it will be possible to perform the technique of serial crystallography (SSX), which proposes to reveal the structure of microcrystals. SSX, in contrast to conventional crystallography, is based on the diffraction of multiple crystals randomly directed. The joining of thousands of patterns obtained should be done in an automated way, with minimal user interference. From microcrystals (10 um) and with an adequate data treatment, we can reveal the structure of new proteins, such as cell membrane structures, which are difficult to crystallize. Enabling advances in the area of drug engineering and more efficient treatments. The possibility to access information from microcrystals might be of real interest to inorganic/organic small molecule studies, being a complement to powder diffraction technique. SSX also favors the study of protein dynamics at room temperature, being of great interest for the optimization of the enzymatic process in the production of second generation ethanol and the development of drugs from natural products.

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KEYWORDS: SERIAL SYNCHROTRON CRYSTALLOGRAPHY, SMALL MOLECULE, MACROMOLECULAR CRYSTALLOGRAPHY

POSTER PRESENTATION

DISENTANGLING X-RAY AND VISIBLE LIGHT IRRADIATION EFFECTS UNDER CONTROLLABLE MOISTURE CONDITIONS IN HALIDE PEROVSKITES

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Hybrid organic-inorganichalide perovskites are of great interest in applications in optoelectronic devices. $Cs_xFA_{1-x}Pb(I_{1-y}Br_y)_3$ perovskite (PVSK) shows an improvement in the thermal-structural and optical stability, features highly aimed in solar cells and optical devices. Despite this, the comprehension of the impact of environmental factors, such as moisture, temperature and radiation, on its optoelectronic properties remains a challenge. To better understand the evolution of the aforementioned PVSK, we have implemented an experimental setup at the XRD2-LNLS beamline aiming to detailed structural characterization by X-ray diffraction (XRD) while measuring X-ray excited optical luminescence (XEOL) simultaneously, with the sample at a fixed angle. The $Cs_xFA_{1-x}Pb(I_{1-y}Br_y)_3$ perovskites (Cs:Br 10:38 and 40:38) were monitored by XEOL and XRD under controlled conditions (humidity (dry and ambient)) and types of stimuli radiation (X-ray - 7keV - and UV-VIS-NIR)). In agreement with literature [1], 10:38 presented cubic crystal structure while 40:38 the tetragonal phase. For X-ray stimulation experiments in the dry (N2) atmosphere, both samples were completely stable with respect to the XRD and the XEOL. Under ambient atmosphere, both samples suffered a decrease in the quantity of the PVSK phase. The 10:38 PVSK presented a decrease in its peaks, with the arise of a still unidentified phase; the 40:38 peaks suffered a splitting in the XRD peak due to the iodide and bromide rich phases segregation.[2] The XEOL from both samples suffered redshift, particularly for the 10:38 because of the optical split in the PL. Under UV-VIS-NIR stimulation, the samples remained structurally and optically stable under dry atmosphere. Under ambient atmosphere, both perovskites showed variations in XEOL profile (maxima position and bandwidth) and posterior relaxation towards the initial state as the stimulation source was turned off. Despite it, no significant structural changes were observed.

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KEYWORDS: HYBRID PEROVSKITE, PVSK, XRD, XEOL

POSTER PRESENTATION

ELECTRONIC CHARACTERIZATION OF H-PHTHALOCYANINE/$La_{0.67}Sr_{0.34}MnO_3$/$SrTiO_3$ INTERFACE

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Organic semiconductors are promising materials to spintronics applications due to electronic and structural flexibility, and theoretical long spin coherence time (weak spin-orbit and hyperfine interactions). An important role in the device performance is the quality and the properties of the interface between electrodes and molecular film. $La_{0.7}Sr_{0.3}MnO_3$ (LSMO) and Co are the most used electrodes for organic spintronics due to their high magnetic polarization at room temperature. H-Phthalocyanine (HPc) is a quasiplanar heterocyclic macrocycle molecule with high conjugation and in contact with metal substrate may undergo to metallization process, modifying its properties, and favoring the spin polarization of the interface [1,2]. Here, we investigated HPc/LSMO and HPc/Co interfaces by using spectroscopic tools. The interface was prepared using the surface facility tools (Pulser Laser Deposition, Organic and Inorganic Molecular Beam Deposition Chambers) and characterized (X-ray Photoemission and Absorption) in situ at PGM beamline, LNLS. Ultraviolet and X-ray photoemission experiments reveal a significant modification of LSMO valence band by HPc/$La_{0.7}Sr_{0.3}MnO_3$(~10nm) interface formation. This result is promising since can give hint on engineering simple organic-inorganic interfaces with possible spintronic properties even room temperature.

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POSTER PRESENTATION

EVALUATION OF THE ANGIOTENSIN II RECEPTOR BLOCKERS TO PREVENT THE AORTIC ARCH DAMAGE DUE HYPERTENSION

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Hypertension is one of the major public health problems worldwide due to its strong impact on cardiovascular morbidity and mortality. The world is currently suffering from the outbreak of a pandemic caused by the severe acute respiratory syndrome known as COVID-19. Evidence shows that the most common comorbidities associated with increased severity of lung injury and mortality of patients with COVID-19 were hypertension, diabetes and coronary heart disease. Due the severity of this new disease, studies about the hypertension and the efficiency of the medicines in prevent the damage caused by it have become even more important. Angiotensin II has been linked to vascular dysfunction and target organ damage. The efficiency of angiotensin II receptor blocker to protect organs that may be affected has been increasingly studied [1]. Previous studies showed the efficacy of losartan to correct the altered structure and endothelial dysfunction of resistance arteries from patients with essential hypertension [2], and also prevent the progression of coronary atherosclerosis in patients with significant atherosclerotic disease [3]. Determination of the distribution of low-Z elemental constituents as O, Na and Mg in the aortic arteries of hypertensive rats submitted to an antihypertensive treatment with two different ARB agents were previous made by Picker and coauthors [4]. However, the evaluation of heavier elements (P, K, Ca, Mn and others) are extremely important to determine the efficiency of these agents to prevent the artery damage. CARNAÚBA is the tender-to-hard X-ray nanoprobe under construction at the new Brazilian synchrotron light source SIRIUS. The all achromatic CARNAÚBA optics will covers continuously the energy range from 2 to 15 keV [5]. The use of CARNAÚBA beamline will provide both submicrometer spatial resolution and elemental mapping of important elements in coronary artery of hypertensive rats allowing analyze the tissue in a cellular level.

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KEYWORDS: HYPERTENSION, ARBS, ELEMENTAL DISTRIBUTION

POSTER PRESENTATION

EXPERIMENTAL STUDY OF Ni-W NANOSTRUCTURED ALLOYS

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Surface modification by coatings has become an essential step to improve the surface properties of materials. Coatings can protect a substrate by providing a barrier between the metal and its environment. The nanostructured coatings have long been recognized to exhibit remarkable and technologically attractive properties due to their extremely fine microstructure. Nickel and tungsten (Ni-W) alloys are particularly interesting because they exhibit good corrosion resistance in different aqueous media, high hardness and ductility, catalytic and anisotropic magnetic properties. In this work nanocrystalline Ni-W alloys were obtained galvanostatically by pulse electroplating, this method was selected because it allows controlling the structure, morphology and size of the grains and chemical composition in the electrodeposits through the correct adjustment of the system variables (pH, concentration, temperature, pulse frequency). Here we have done an extensive characterization of an electrodeposited Ni-W coating by using surface techniques such as Atomic Force Microscopy (AFM), Scanning Electron Microscopy (SEM), and X-ray Photoelectron Spectroscopy (XPS), and bulk techniques such as energy dispersive X-ray Analysis (EDX), X-ray Diffraction (XRD), X-ray Absorption Near Edge Structure (XANES), Extended X-ray Absorption Fine Structure (EXAFS), and High Resolution Transmission Electron Microscopy (HRTEM).The surface of the coating consists of 10-50 nm crystals forming a cauliflower-like structure protected by a mixture of nickel and tungsten oxides. The cauliflower structure is preserved into the bulk coating that exhibits an average composition □70 at% Ni-30 at% W. Two different phases are observed in the bulk structure: a W-rich amorphous phase (□40 %) and a Ni-rich crystalline phase (□60 %). Results from this extensive characterization shed light on some controversial points related to the chemical composition and structure of this interesting system.

POSTER PRESENTATION

EXPLORING THE EFFECT OF THE AS-BUILT MICROSTRUCTURE ON THE MARTENSITE TO AUSTENITE REVERSION SEQUENCE IN A SELECTIVE LASER MELTED 18Ni MARAGING STEEL

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During selective laser melting, the non-equilibrium rapid solidification conditions promote local elemental segregation, leading to a characteristic microstructure composed of cellular walls. These walls can display either low carbon BCC martensite or FCC retained austenite crystal structures, thus affecting the subsequent isochronal or isothermal martensite to austenite phase transformation mechanism. In the present study, the effect of the non-homogeneous as-built microstructure on the martensite-to-austenite reversion phenomena was studied for a 18Ni maraging steel fabricated by selective laser melting. In-situ synchrotron X-ray diffraction was used to retrieve the austenite volume fraction and lattice parameter evolution during the physical simulation of continuous heating to the fully γ field; and during isothermal tempering cycles throughout the inter-critical α+γ field. The effect of the as-built microstructure on the diffusive- and displacive-driven austenite reversion mechanisms was studied by applying slow (0.167°C.s^{-1}), intermediate (5°C.s^{-1}) and ultra-fast (500°C.s^{-1}) heating rates. For slow diffusive-controlled austenite reversion in the as-built state, the α+γ inter-critical field was expanded when compared to a solution treated sample. This was associated to the synergic effects of compositional segregations (anticipating reversion) and pre-existing retained austenite (delaying solubilization). These two factors, however, were irrelevant when the massive-driven reversion mechanism was dominant upon heating rates at and above 5°C.s^{-1}. Within the α+γ inter-critical field, the as-built state did not fundamentally alter the overall reversion kinetics, the capacity to reach thermodynamic equilibrium nor affected the total volume fraction of reverted austenite formed right before cooling.

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KEYWORDS: AUSTENITE REVERSION, IN-SITU X-RAY DIFFRACTION, MARAGING STEEL, SELECTIVE LASER MELTING, POWDER BED FUSION, ADDITIVE MANUFACTURING

POSTER PRESENTATION

EXPRESSION OF CATECHOL-O-METHYL TRANSFERASE (COMT) FROM *PARACOCCIDIOIDES SPP.* FOR BIOCHEMICAL AND STRUCTURAL CHARACTERIZATION

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Fungi of the genus *Paracoccidioides* are causative agents of systemic mycosis Paracoccidioidomycosis (PCM). PCM is endemic to Latin America, with Brazil accounting for 80% of cases. Treatment with antifungals is prolonged, expensive and has side effects, which discourages patients from following it, increasing the recurrence of the disease, in addition to the already reported cases of resistance. Thus, the catechol-O-methyltransferase (COMT), not yet studied in *Paracoccidioides spp.*, appears to be a promising pharmacological target since in another fungi like *Candida tropicalis* and *Podospora anserina* this protein has functions related to survival in the host. In addition, COMT has low genetic variability between species of *Paracoccidioides* and low similarity with human COMT. For the biochemical and structural characterization of COMT, *Escherichia coli* were transformed with the vector pET28a(TEV) containing the comt gene. Tests on *E. coli* BL21 (DE3) and Rosetta (DE3) under different conditions of temperature and concentration of inducer resulted in expression in the insoluble fraction. So, *E. coli* Arctic Express, which coexpress two chaperonins at 12 ° C, are being used for expression of COMT in the soluble fraction (confirmed by Western blot). COMT was purified by cobalt affinity chromatography, however, SDS-PAGE analyzes showed, in addition to the ~ 34kDa band corresponding to COMT with histidine tail, a ~ 60kDa band in the fractions obtained after elution. Two hypotheses are being evaluated: co-purification with chaperonin Cnp60 or formation of COMT dimers. For this, tests to remove Cnp60 with ATP and more extreme denaturing conditions to break possible COMT aggregates before analysis by SDS-PAGE are being carried out in order to elucidate this enigma and also to obtain COMT pure and functional for activity and structural characterization tests, essential knowledge to better understand this protein.

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KEYWORDS: KEYWORDS: CATECHOL-O-METHYLTRANSFERASE, COMT, PARACOCCIDIOIDES

POSTER PRESENTATION

FUNCTIONALIZED MAGNETITE NANOPARTICLES: STUDY OF ITS PHYSICOCHEMICAL PROPERTIES AND BIOCOMPATIBLE APPLICATIONS

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Nanoparticles of magnetite (NPs-Fe_3O_4) are very versatile systems with multiple applications in science and technology.[1] Surface modification of magnetic NPs with organic molecules present many advantages as low cost of production, physicochemical stability, biocompatibility, safe to the environment and easy coating [2]. This NPs have been the subject of an enormous scientific production in the last decade, and numerous biological applications have been reported. In this work, Fe_3O_4 with different coatings were used in multiple applications as adsorption of various pollutants, effluent remediation, bacteria capture and plant fertilization [3]. NPs-Fe_3O_4 of 11 nm of diameter were synthesized with several coatings such as amino acids (arginine and cysteine), oxalate, polyethylene glycol and humic-acids. Its physicochemical properties were examined from electron microscopy, X-ray diffraction, infrared spectroscopy, magnetization, Z potential, among others. From which size homogeneity, high magnetization, presence of the coatings could be confirmed from the characteristic signals in the infrared, surface charge strongly dependent on the coating, high synthesis yield and surface area. Subsequently, heavy metal adsorption tests such as As, Pb, Cu, Hg were carried out with high removal efficiencies greater than those currently reported in bibliography. The bacteria capture efficiency was 95-97% [4] and the combination of dolomite and magnetic nanoparticles were highly efficient for the removal of phosphates and chromates in effluents. Likewise, the nanoparticles were shown to possess fertilizer characteristics by increasing the quantum and efficiencies yields of photosynthesis in leaves sprayed with NPs-Fe_3O_4. This results shown that NPs-Fe_3O_4 have high potential for bio-applications for adsorption of pollutants and bacteria and as nanofertilizer. However, although the results presented here are promising, more studies are necessary to determine their applicability.

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KEYWORDS: NANOPARTICLES, BIO-APPLICATIONS, POLLUTANTS, SUPERPARAMAGNETIC, REMEDIATION, MAGNETISM

POSTER PRESENTATION

FUNDAMENTAL STUDIES OF MAGNETO-OPTICAL BOROGERMANATE GLASSES AND DERIVED OPTICAL FIBERS CONTAINING Tb^{3+}

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In the last decades, magneto-optical (MO) materials based on the Faraday effect have been studied for numerous authors for cutting edge technologies [1-3]. In this work, new magnetic glass compositions based on the system $(100\text{-}x)(60GeO_2\text{-}25B_2O_3\text{-}4Al_2O_3\text{-}10Na_2O\text{-}1PbO) – xTb_4O_7$ (with $3 \leq x \leq 10$ mol%) were prepared by the melt-quenching technique. The materials were characterized with respect to their structural (XRD, EXAFS, XANES and Raman), thermal, morphological, optical, magnetic and magneto-optical (Faraday rotation) properties. XANES and EXAFS spectra of GBANPb-xTb glasses and the crystalline reference samples, germanium (IV) oxide (rutile phase), Tb_2O_3 and Tb_4O_7 were collected at the XAFS2 beamline at the Brazilian National Synchrotron Light Laboratory (LNLS, Campinas-SP, Brazil) at the Ge K-edge and the Tb-L3 absorption edge at room temperature. Morphological and magnetic measurements show the homogeneous distribution of the Tb^{3+} ions as the magnetic susceptibility increases proportionally to the rare earth content. XANES and EXAFS measurements at the Ge-K edge show that the germanium atoms are always tetrahedrally coordinated, while results at the Tb-L3 edge shows terbium atoms to be eight-coordinated and exclusively present in the oxidation state 3+ independently of the glass composition. There is no evidence of presence of GeO_6 species. Raman spectra indicate that with increasing Tb_4O_7 content the glassy network is being successively depolymerized. HRTEM images for the GBANPb-4Tb and GBANPb-6Tb glasses confirm the absence of crystals on the nano-scale. Magneto-optical measurements show that the Verdet constant (VB) increases linearly with the Tb^{3+} concentration reaching the value of -83.9 rad $T^{-1}m^{-1}$ at 650 nm for glass containing 8 mol% of Tb_4O_7. Good thermal stability ($\Delta T > 200$ °C) allowed obtaining an optical fiber of a glass containing 4 mol%Tb_4O_7.

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KEYWORDS: MAGNETO-OPTICAL GLASSES, FARADAY ROTATION AND OPTICAL FIBERS

POSTER PRESENTATION

GRAPHENE OXIDE AS A SURFACTANT IN THE NANOSTRUCTURING OF A CONDUCTION POLYMER: EFFECT ON THE ELECTRONIC STRUCTURE, CHAIN ORIENTATION, AND CHARGE TRANSFER DYNAMICS

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The search for new structures, new synthetic paths, and new processing technologies is crucial for the future of electronic devices. In this work, we present an innovative and easy method to prepare polymeric nanoparticles using graphene oxide (GO) as an alternative to traditional surfactants, using the concept of Pickering emulsions. Graphene oxide colloidal dispersion was sonicated along with toluene solutions of the organic semiconductive polymer poly[2,7-(9,9-bis(2- ethylhexyl)-dibenzosilole)-alt-4,7-bis(thiophen-2-yl) benzo-2,1,3-thiadiazole] (PSiF-DBT). The effect of PSiF-DBT concentration (4.5 and 7.5 mg mL-1) and effect of the pH on the GO dispersion were considered in thin films of GO:PSiF-DBT. The molecular structure and molecular orientation of these organic thin films were investigated by near-edge X-ray absorption fine structure (NEXAFS). Ultrafast electron dynamics in the low-femtosecond regime was evaluated by resonant Auger spectroscopy using the core-hole clock (CHC) method [1]. Infrared spectroscopy (IR), scanning and transmission electron microscopy (SEM and TEM) and atomic force microscopy (AFM) images were also performed. The results indicated that the polymer concentration and pH of the medium have a direct influence on the morphology and in the electronic properties of the materials. Infrared spectroscopy suggests the anchoring of Si atoms from the PSiF-DBT in oxygenated groups of graphene oxide, corroborating angle-resolved NEXAFS data that suggests an upright-standing molecular orientation for the thiophene units and a lying-down orientation for the benzothiadiazole units. Resonant Auger spectroscopy indicated that the charge transfer is faster for higher ratio of PSiF:GO in the film.

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KEYWORDS: GRAPHENE OXIDE, PICKERING EMULSION, NANOSTRUCTURING, CONDUCTION POLYMER

POSTER PRESENTATION

GRIPPER SYSTEM CONTROL FOR SAMPLE POSITIONING USING COMPUTER VISION

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Having recently started the operation of Sirius beamlines, sample environment control within a precise and safe way has become a key point for the kickoff experiments. One of the two endstations of the CARNAÚBA beamline, called TARUMÃ, started to develop a project to control a gripper system able to exchange, position, and align the sample holder by using Computer Vision algorithms. Through a set of optical visualizations, mainly composed of conventional and zoom lenses coupled to CCD digital cameras, an algorithm developed in Python uses the OpenCV library to do this control by carrying the holder of the sample specialty designed as small pins. Both functionalities have in common their inputs: real-time images of the optical assemblies.

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KEYWORDS: COMPUTER VISION , TARUMÃ

POSTER PRESENTATION

HIGHLY ORIENTED $NiSi_2$@Si THIN-NANOCOMPOSITE PRODUCED BY SOLID STATE DIFFUSION: MORPHOLOGICAL AND CRYSTALLOGRAPHIC CHARACTERIZATION

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This article describes a novel synthesis procedure and a morphological and crystallographic multi technique characterization of a 2D-nanocomposite consisting of highly oriented $NiSi_2$ nanoplates endotaxilly grown in a single crystalline Si(100) wafer close to its external surface. This nanostructured material is prepared using a novel procedure which promotes the diffusion of Ni atoms from a deposited Ni-dopped-SiO_2 film into the volume of a Si(001) flat wafer under controlled thermodynamic conditions. High Resolution Transmission Electron Microscopy (HRTEM) images reveal the formation of thin hexagonal nanoplates totally buried inside the Si(001) wafer and randomly oriented spherical Ni nanocrystals located within a thin layer close to external surface of the wafer. The $NiSi_2$ nanoplates formed by endotaxial growth have their large flat surfaces parallel to one of the four Si{111} crystallographic planes and exhibit coherent 8A-type interfaces with the host matrix. The quantitative analysis of GISAXS indicated that the average thickness and hexagonal sides of the $NiSi_2$ nanoplates are 3.0 nm and 28 nm, respectively. Since the described process to obtain 2D-$NiSi_2$@Si nanocomposites is reproducible, it opens new possibilities for exploiting the structural features of these materials for use in devices requiring anisotropic electrical transport properties.

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KEYWORDS: NISI2@SI, NANOPLATES, NANOPARTICLES, GISAXS, TEM

POSTER PRESENTATION

HIGH-PRESSURE STUDIES ON L,L-DILEUCINE CRYSTALS

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Peptides are some of nature's most important organic molecules of the living beings. Formed by amino acids, these molecules have several functions in the organisms. Among the peptides, dipeptides have been studied in recent years because they are simple molecules in relation to more complex peptides. In this presentation results on L-leucyl-L-leucine hydrate are shown, with particular emphasis on the vibrational and structural properties. When crystallized, L-leucyl-L-leucine hydrate forms a kind of tubular structure. Initially, we confirmed the orthorhombic structure of the crystal with X-ray diffraction measurements and Rietveld refinement and performed calculations of density functional theory to assign the normal modes of the material. After the theoretical vibrational characterization, the crystal was submitted to high pressure. Through Raman spectroscopy, up to 8 GPa, great modifications were observed in the vibrational modes of L-leucyl-L-leucine hydrate between 2.3 and 2.9 GPa. These modifications were interpreted as change in the tubular arrangement during the compression process. In addition, data up to ~ 1.0 GPa indicates that the hydrogen bonds became slightly stiffer. X-ray diffraction measurements using synchrotron radiation were done for pressures up to 26 GPa. Impressive modification is observed between 2 and 4 GPa, where the high intensity peaks associated with the (0 0 2) and (0 1 1) reflection planes had their intensities drastically reduced. This was interpreted as a phase transition and, together with other aspects of the X-ray diffractogram, it can be understood that the crystal is going into an amorphous phase. This fact was confirmed by the increase in the linewidth bands associated with the stretching modes of CH_3, C = O, and water molecules observed in the Raman spectra.

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KEYWORDS: HIGH PRESSURE, X-RAY DIFFRACTION, DIPEPTIDE

POSTER PRESENTATION

HYDROGEN EFFECT ON MECHANICAL AND STRUCTURAL PROPERTIES OF AN AISI 316L PROCESSED BY SELECTIVE LASER MELTING

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Austenitic stainless steels such as AISI 316L have been widely studied by AM-Additive Manufacturing procedures. In many solicitations, especially in the petrochemical sector, components with complex geometry can easily be made by AM processes. The use of this steel in the oil and natural gas industry stands out. However, the environment where these components are applied is often subjected to rich hydrogen surroundings which lead to an effect known as hydrogen embrittlement. Therefore, the behavior of AM obtained metals under such environments are not widely known. In order to study the characterization and mechanical behavior of a 316L austenitic stainless steel produced by Selective Laser Melting (SLM, an AM process), tensile tests with in situ X-ray diffraction measures were conducted in a Gleeble 3S50® thermomechanical system - situated in in the Brazilian Synchrotron Light Laboratory (LNLS) of Brazilian Center for Research in Energy and Materials (CNPEM). Stress-strain curves and diffractograms were obtained for the as printed 316L AM, annealed 316L AM and conventional/rolled 316L samples for comparison. The results at room temperature (without hydrogen influence) showed that both AM conditions presented only austenite, an fcc phase, and the initial phase remain the same during all the mechanical test. For the as-rolled condition, α martensite, a bcc phase, was observed initially. The percentage volume of this phase increased during the tensile test. When the samples were submitted to a hydrogen environment by cathodic charging, the hydrogen was responsible for ε martensite hcp phase formation which led to a TRIP effect during the mechanical tests. Thus, the samples that were hydrogen charged presented higher values of strain in comparison to the non-charged sample without loss of UTS.

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KEYWORDS: SELECTIVE LASER MELTING, HYDROGEN EMBRITTLEMENT, PHASE TRANSFORMATION

POSTER PRESENTATION

IN OPERANDO XAFS CHARACTERIZATION OF CATHODE MATERIALS USED IN Li-ION BATTERIES

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Li-ion batteries have an unmatchable combination of affordable cost, high energy storage capabilities and power density, making it the technology of choice for portable electronics, power tools, and hybrid/full electric vehicles1. Due to the increasing future energetic and efficiency demands some characteristics of these batteries must be improved. Li-ion batteries electrochemistry performance, life cycle and security conditions are some of these features. To overcome these challenges it is mandatory to have a better comprehension of the physicochemical processes associated to the Li intercalation during the charge/discharge process. This understanding demands most than ever in operando characterization and combination of several techniques. The task difficulty is increased by Li reactivity that suppose the need a controlled atmosphere free of $O2$ or $H2O$. In order to overcame these challenges we develop a cell that allows to characterize by XAFS in operando condition and at the same time perform a potential or current sweep as needed. In this work we present an exhaustive characterization by several conventional techniques (TEM, XPS, XRD, etc) in combination with XAFS characterization performed in operando of materials used Li ion batteries based on Ni and Mn mixed oxides doped with different amounts of Ti (LMNO doped cathodes). The In operando measurements at the Mn and Ni K-edge XANES were obtained at the DXAS beamline of Laboratorio Nacional de Luz Sincrotron (LNLS). Ex situ Ti K-edge EXAFS measurements were also performed at XAFS2 beamline of LNLS in order to correlate the increase in electrode resistance to cell cycling number with Ti incorporation.

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POSTER PRESENTATION

IN SITU S K-EDGE ANALYSIS OF CYSTEINE OXIDATION BY SILVER ATOMIC QUANTUM CLUSTERS IN PRESENCE OF REACTIVE OXYGEN SPECIES

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Cellular redox homeostasis regulation involves several catalytic processes in which are predominant the oxidation and reduction of thiols present in cysteine residues on specialized enzymes active sites [1]. These processes determine the survival of cells according to their ability to reverse oxidative stress, generated mainly by the presence of reactive oxygen species (ROS) in the environment [2]. For this reason, cells are providing with the enzymatic machinery to face the stress. Sulfur species in this machinery with an oxidation state greater than S+4 represents an irreversible stage leading frequently to cell death [3] In addition, it has been reported the capacity of atomic quantum clusters (AQCs) of low atomicity to catalyze oxidative processes due to their free electrons quantic confinement [4]. Also, recent results display Ag5-AQCs ability to catalyze the sulfur atoms oxidation for several chemical environments in aerobic conditions and room temperature [5]. In this work, we study the oxidation reaction of cysteine´s thiols by Ag5-AQCs, by measuring S K-edge XANES spectra at different experimental environments, in presence and absence of ROS at mix reaction, through the quantification of the sulfur average oxidation state in both environments. The obtained results allowed us to identify noteworthy differences in final state reaction products under studied conditions. A greater oxidizing effect is registered in ROS presence, which indicates that an oxidizing environment could be a selectivity factor for controlling the oxidation reaction. This represents a key factor due to the increase in the irreversibility of the oxidation in biological terms, considering that in many pathologies, such as cancer, the oxidative environment and ROS production are a distinguishing characteristic for the disease to advance. Finally, it can be shown the ability of XANES spectroscopy on the studied molecules, allowing to characterize faithfully the chemical changes.

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KEYWORDS: XANES, CLUSTERS, THIOLS, SULFUR

POSTER PRESENTATION

IN SITU STUDY OF AISI 304 STAINLESS STEEL USING SYNCHROTRON LIGHT

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The cryogenic straining has been extensively studied due to the possible properties improvements, such as an increase in mechanical resistance and ductility. These gains occur as a consequence of the partial suppression of dynamic recovery, which produces microstructural refining and increases microstrain, due to an increase of internal defects density. In this study, the stainless steel AISI 304 was tested under four strain-rates at room and cryogenic temperatures. The XTMS experimental station allowed tensile tests simultaneously to X-ray diffraction, providing in situ information of phenomena and microstructural changes that occur during straining. At cryogenic temperature, it was possible to verify a change in the shape of the stress-strain curve, with loss of ductility and increase of resistance, being an effect of the martensitic transformation. The onset of the austenite -> martensite phase transformation at room temperature occurs at a higher strain than for the cryogenic temperature, due to the effect of the adiabatic heating, which stabilizes austenite. The microstrain in the austenite planes decreased during the plastic deformation, indicating the presence of dynamic recovery.

KEYWORDS: CRYOGENIC STRAINING, MARTENSITIC TRANSFORMATION, X-RAY DIFFRACTION, MICROSTRAIN, AUSTENITIC STAINLESS STEEL

POSTER PRESENTATION

IN SITU STUDY OF CERIUM OXIDE NANOPARTICLES DURING REDUCTION IN A Co ATMOSPHERE

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The cerium oxide CeO$_{2-x}$ ($0 < x < 0.5$) has been strongly studied due to the high technological potential for several applications. The CeO$_{2-x}$ presents high facility to change the oxidation state between Ce(III)(x = 0.5) and Ce(IV)(x = 0)) [1]. The CeO2 (Ce(IV) oxidation state) presents the fluorite-type crystal structure and Ce$_2$O$_3$ (Ce(III) oxidation state) the hexagonal one. In a previous work [2], it was shown the possibility to control the surface area, mean diameter, and oxygen vacancies population of cerium oxide nanoparticles by means of synthesis. Moreover, the cerium oxide nanoparticles synthesized showed high reducibility (high values for the Ce(III) fraction) in comparison to similar cerium oxide nanoparticles in the literature [3]. The reduction reaction is routinely used to activate catalysts and, in some cases, it induces a phase transition with a consequent change in the catalytic properties. The aim of this study was to investigate the structural properties under reduction environment of these highly reducible nanoparticles using the in situ XRD technique. The in situ XRD measurements were performed at XPD beamline at LNLS during reduction in CO atmosphere at 900 °C. It was observed another crystal structure at 900 °C, the Cu$_2$Se-like crystal structure, whose fraction increases with the reduction time. During the cooling process, this phase turns metastable due to the high cooling rate applied, namely 15 oC/min. The metastable phase presents a negative thermal expansion coefficient (α). Using an approximation of Avrami's theory for non-isothermal processes [4], the kinetic of phase transition was studied. This analysis shows that the phase transition occurs in 1D with the crystallization mechanism controlled by volume diffusion. Therefore, the formation of the fluorite phase during the cooling process is due to the O diffusion from the Cu2Se-like to fluorite crystal structure which explain the negative α value.

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POSTER PRESENTATION

IN SITU THERMOMECHANICAL STUDY OF PURITY COMMERCIAL TITANIUM VIA SYNCHRONTON RADIATION UTILIZING X-RAY DIFFRACTION.

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Several research related to metallic materials mechanic conformation presented the possibility of achieve highest strength levels, ally to preservation of deformation capacity by means of deformation realized at low temperatures, reaching cryogenic temperatures. In the current work, it is intended to determine the deformation mechanisms that act at different temperatures and that influence in the finals properties of material and correlate properties mechanic data with the results of x-ray diffraction and optical microscopy and fracture analysis. In situ characterization of titanium samples purity commercial (grade 2) were performed by means Sincrontron radiation during the deformation process of material, utilizing the X-Ray Scattering and Thermo-Mechanical Simulation (XTMS) in the Laboratorio Nacional de Luz Sincroton (LNLS). The tests was executed at room temperature and temperatures of -5, -50, -100, -125, -150 and -180 ºC. With the following reduction of deformation temperature, the material presented gradual increase of yield point and of ultimate tension strength. Others properties as homogeneous deformation, which is the important parameter to mechanic conformation, and total deformation were measured and correlated with others results. The diffraction profile were analyzed and utilized in the ratio c/a calculation and assisted in monitoring of microstructural transformations and evolutions that the material suffered during the in situ deformation. The relation between the lattice parameters of Hexagonal Close-Packed crystalline structure (c/a) is important to identify the deformation mechanism acting during the deformation and to understand these mechanisms, the diffraction profile are necessary. The correlation of results corroborated at understanding of mechanism by which the material culminated to presented improvement of properties.

POSTER PRESENTATION

IN SITU XANES STUDIES ON $Ce_{0.9}Zr_{0.1-X}Sm_XO_{2-X/2}$ (X= 0.1, 0.08, 0.06, 0.04, 0.02, 0) SYNTHESIZED BY HYDROTHERMAL METHOD

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60%NiO/$Ce_{0.9}Zr_{0.1}O_2$ has demonstrated to be an excellent anode material for Intermediate Temperature-Solid Oxide Fuel Cells. Nevertheless, its mixed conductivity should be enhanced in reducing atmospheres [1]. The substitution of Zr^{4+} by Sm^{3+} is proposed to incorporate oxygen vacancies into the structure thus improving ionic conductivity. $Ce_{0.9}Zr_{0.1-x}Sm_xO_{2-x/2}$ (x= 0.1, 0.08, 0.06, 0.04, 0.02, 0) mixed oxides were synthesized via the hydrothermal method and calcined at 550 °C. X-ray diffraction with synchrotron radiation shows that all samples are monophasic with the fluorite structure, typical of CeO_2. Average crystallite sizes are nanometric (< 11 nm) and lattice parameters increases linearly with Sm^{3+} content. The effect of Sm^{3+} incorporation into the lattice on redox properties was studied by in situ Dispersive X-ray absorption spectroscopy (DXAS) experiments, performed in diluted H_2 gas-flow (5 vol.% in 50 $mL.min^{-1}$, He balance), from room temperature to 800 ºC with a heating rate of 10 $ºC.min^{-1}$. DXAS measurements were performed at the Ce LIII absorption edge, being this spectroscopy especially suited for tracking time-dependent evolution of chemical reactions. Three reduction steps were observed during the process, two of which can be related to the reduction of surface Ce^{4+} species (< 600 °C) and the third one to the reduction of bulk species. The incorporation of Sm^{3+} increased the reduction kinetics of bulk species. The exit gas is analyzed by online mass spectroscopy. Water production was observed in accordance with each reduction step.

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KEYWORDS: CE-ZR-SM, NANOMATERIAL, DXAS, XPD

POSTER PRESENTATION

INFLUENCE OF BA ADDITION ON THERMAL STABILITY AND CATALYTIC ACTIVITY OF $La_{2-X}Ba_{X}CuO_4$ CATALYSTS

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The replacement of noble metals by transition metals in TWC is highly advisable for both environmental and economic reasons. In this work the catalytic reduction of NO by CO was studied using mixed oxides $La_{2-x}Ba_{x}CuO_4$ (x = 0.0 or 0.4) as catalysts, prepared by the coprecipitation method and characterized by X-ray diffraction (XRD), O_2 temperature programmed desorption (O_2-TPD), H_2 temperature programmed reduction (H_2-TPR) and X-ray absorption near-edge structure (XANES). The NO reduction by CO was studied in laboratory microreactor as well as at the D06A-DXAS beamline of the Brazilian Synchrotron Light Laboratory (LNLS) for obtaining information about the evolution of the oxidation state of the catalysts active phases under reaction conditions. LaCu and LaBaCu fresh catalysts showed good activity in the NO reduction by CO reaction. The Ba addition caused structural changes creating oxygen vacancies on the surface and in the bulk of the catalyst that promoted the reducibility and activity of the fresh catalysts mainly at low temperatures. Another important role of barium was the improvement of the catalyst stability, maintaining their reducibility even after thermal aging. Based on in-situ XANES results, LaBaCu900 presented the higher reducibility with Cu^{+} and Cu° formation during the reaction. The use of a reducing pretreatment provided active sites formation, justifying the significant improvement in catalytic activity. The N_2O generation was also observed in all catalysts (fresh and aged) but in lower yields when compared with noble metal-based catalysts. The decrease in the activation energy of the pretreated catalysts at 500 °C under H_2 flow indicates a change in the active sites (presence of reduced copper species since the beginning of the reaction).

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POSTER PRESENTATION

INFLUENCE OF DOPAMINE ON THE MAGNETIC PROPERTIES OF SUPERPARAMAGNETIC IRON OXIDE NANOPARTICLES

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We report on the effect of different molecular coating as carboxylate and catecholate ligands on the magnetic properties of small (average 4.4 nm) iron oxide nanoparticles (NPs) prepared by the thermal decomposition method using oleic acid as stabilizer followed by dopamine ligand exchange step [1]. We investigated the role of the surface chemistry of ligand stabilized NPs and their overall resulting magnetic properties. To isolate the contribution of the NPs from the coating, the ligand percentage in contact with iron oxide surface is evaluated by thermogravimetric measurements. Our M(H) measurements at room temperature show a superparamagnetic behavior for all samples and ZFC-FC magnetization curves and AC magnetic susceptibility measurements indicate a decrease of magnetic interaction after ligand exchange. The insight about the surface effect on NPs magnetic properties is obtained through X-ray absorption spectroscopy (XAS) and X-ray circular magnetic dichroism (XMCD) measurement [2]. The modification of the Fe $L_{2,3}$ XAS and XMCD line shape upon ligand substitution extent show the change of Fe cations distribution between octahedral and tetrahedral sites with the ligand exchange.

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KEYWORDS: NANOPARTICLES, SURFACE, DOPAMINE, MAGNETIC PROPERTIES

POSTER PRESENTATION

INFLUENCE OF STRUCTURAL DEFECTS ON THE ELECTRONIC AND ELECTROCHEMICAL PROPERTIES OF ON-CHIP BILAYER GRAPHENE

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Pristine monolayer graphene has a slow vertical heterogeneous electron transfer (HET) rate which limits its application in electrochemical devices [1-2]. Introducing structural defects in graphene increases the rate of vertical HET, but affects its ballistic conduction [3]. We present here a device composed of bilayer graphene supported on Si/SiO_2 substrate. Defects were introduced into the top layer graphene sheet through electrochemical oxidation. Plasmonic activity is of particular interest in the studies of structural modifications, because its response depends on the electrons delocalized in the sp2 carbon network. Defects by sp3 carbons can dramatically alter the behavior of the graphene plasmons [4]. To monitor plasmonic activity on these electrodes, s-SNOM experiments were conducted at the LNLS. It was observed that plasmonic coupling of the two graphene sheets with the SiO_2 substrate phonons is greater than that of a single sheet. The insertion of defects in both cases decreases the effectiveness of the coupling. However, it is still possible to observe high plasmon activity on the oxidized bilayer graphene electrode. Electrical impedance spectroscopy experiments showed that the number of defects produced does not drastically influence the resistivity of graphene, corroborating with what was observed in the s-SNOM experiments, indicating that introducing defects to the bilayer graphene does not affect significantly changes the electronic conduction properties. Cyclic voltammetric experiments showed an improved electrochemical activity towards the external redox probe in solution for oxidized bilayer graphene as compared to pristine electrode. Electrochemical impedance spectroscopy showed a decrease in the Rct value by more than 90% after electrode was electrochemically oxidized. These results indicate that it is possible to produce an on-chip graphene electrode with high vertical HET properties without drastically affecting its electronic properties.

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KEYWORDS: BILAYER GRAPHENE, ELECTROCHEMICAL OXIDATION, ELECTRON TRANSFER, S-SNOM

POSTER PRESENTATION

INFLUENCE OF THE SUBSTRATE ON THE GROWTH OF Co_3O_4 FILMS DEPOSITED BY REACTIVE DC MAGNETRON SPUTTERING

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The Co_3O_4 films were grown on amorphous silica (a-SiO_2), $LaAlO_3$ (100) and, c-Al_2O_3 (0001) in order to investigate the substrate influence on the film preferred orientation. The optimized growth parameters of pure Co_3O_4 films by reactive DC magnetron sputtering is already reported [1]. Here, the Co_3O_4 films were characterized by Raman spectroscopy and also X-ray diffraction using synchrotron radiation at the LNLS-XRD2 beamline. The θ-2θ XRD scans confirmed the presence of only Co_3O_4 phase with a clear dependence of its texture with the substrate in use, such as both (111) and (100) orientations on the $LaAlO_3$, and (111) on both a-SiO_2 and c-Al_2O_3 substrates. Furthermore, rocking curves of the (220)Co_3O_4/$LaAlO_3$ and (111)Co_3O_4/Al_2O_3 exhibits FWHM of 2.7° and 0.84º, respectively, which indicates that the sapphire favors a better (111)Co_3O_4 film orientation (smaller mosaic spread). In this sense, {333} Co_3O_4 reflections analysis revealed a minute lattice strain on the out-of-plane and in-plane directions of 0.1903 % (tensile) and 0.122 % (compressive), respectively. Azimuthal scans with respect of the (0001) Al_2O_3 probing the {311} Co_3O_4 reflections shown a 6-fold symmetry, which indicates the presence of an in-plane twinning with 30º rotation, as suggested before [2,3]. High-resolution 3-D Reciprocal Space Map analysis covering both (0006) Al_2O_3 and the (222) Co_3O_4 reflections have shown similar out-of-plane strain (0.1905 %) and also presented an in-plane isotropic intensity distribution of the film reciprocal lattice point, indicating no preferred mosaic direction. Raman spectrums, which have only shown peaks corresponding to modes of Co_3O_4, also exhibited a compressive biaxial strain based on its blue shift of the order of 2 cm^{-1}. Particularly for the Co_3O_4/Al_2O_3, the polarized Raman spectra results revealed a clear agreement of its intensity with the selection rules for an oriented (111) Co_3O_4. Therefore, the results truly highlight the reactive DC magnetron.

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POSTER PRESENTATION

INVESTIGATION OF THE OXIDATION STATE AND ORBITAL RECONSTRUCTION AT THE INTERFACES $La_{0.67}Sr_{0.33}MnO_3$ ULTRATHIN FILMS

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Complex oxides have received great attention in the current technological and scientific scenario for exhibiting promising multifunctional and emergent properties. These arise from the strong electron-electron correlation and the interplay between lattice, orbital, charge and spin ordering. Specifically, the $La_{0.67}Sr_{0.33}MnO_3$ (LSMO) is a ferromagnetic half metal, with perovskite structure, presenting almost 100% spin polarization at room temperature, what makes it very interesting for applications in spintronics and oxide-based devices [1]. However, the formation of magnetic dead layers at thin films interfaces represents an obstacle for the development of new devices. Although previous studies have linked the formation of dead layer to strain, oxygen vacancies and cation non-stoichiometry, its microscopic origin is still not entirely clear [1,2,3]. In this work, we grow, by pulsed laser deposition, epitaxial ultrathin films of LSMO (thicknesses between 1.2 and 8.4 nm) on $SrTiO_3$ (001) single crystals in order to verify whether the oxidation state of manganese (Mn) and/or an orbital reconstruction at the LSMO interfaces could be related to the formation of the magnetic dead layer. The samples' electronic structure was investigated by Mn L2,3-edge and O K-edge X-ray absorption spectroscopy (XAS) and linear dichroism (XLD), and their magnetic properties by Mn L2,3-edge magnetic circular dichroism (XMCD). All measurements were acquired by total electron yield mode. Our results suggest a reduction in the Mn valence and a consequent decrease in the magnetic moment as the LSMO thickness decreases, i.e. at the interfaces. We also observe a breakdown in the eg orbitals degeneracy at the LSMO surface, leading to an orbital reconstruction which may be related to the loss of magnetism.

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POSTER PRESENTATION

Li-BATTERIES ELECTRODES CHARACTERIZATION: FROM CONVENTIONAL TO PHASE CONTRAST AND SPATIAL RESOLUTION

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Lithium-ion batteries (LIBs) studies look to optimize interface processes of the reversible ion uptake and limit the material deterioration during the cycles. In the pursuit of higher-energy-density LIBs, one major challenge is the stability of high-capacity and high-voltage cathodes with electrolytes. Cathode materials based on Mn spinels LMNO are promising candidates because they are safer, cheaper and possess higher energy densities compared with the state-of-the art LiCoO2(LCO) [1]. Using DXAS beamline at the recently closed LNLS, we were able to follow changes in the electronic structure in operando conditions by X-ray absorption Near Edge Structure of LMNO modified with Ni and Ti at Ni and Mn K edges. Those characterizations gave relevant information that could be improved by increasing the spatial resolution scale or taking advantage of the phase contrast capability that will be available in the new 4th generation machine (Sirius). As an example spatially correlated X-ray pictographic tomography, known as X-ray ptychography [2,3], is a lensless imaging technique that provides quantitative electron density tomograms of the extended system at levels of spatial resolution hardly achievable by common X-ray microscopic techniques. Through this phase contrast based techniques and, for example, scanning X-ray diffraction microscopy [4], it is possible to directly visualize morphological and crystalline changes. This information would enable the stresses in crystals, within individual secondary particles, that is only possible with a few nm resolutions. These tools also allow the study with chemical selectivity and in hierarchical structural levels, with spatial resolution and under real operating conditions of the batteries. In this work we describe the state of the art of these novel experimental methodologies, that could be implemented in Sirius, with the possibility of the characterization of electrodes used in Li+ ion batteries in operando conditions.

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KEYWORDS: LITHIUM-ION BATTERIES-ENERGY-SPINELS LMNO-X-RAY PICTOGRAPHIC

POSTER PRESENTATION

LITERATURE REVIEW OF LECTINS FROM MIMOSOIDEAE SUBFAMILY (LEGUMINOSEAE): PURIFICATION STRATEGIES, STRUCTURAL DATA AND BIOLOGICAL ACTIVITIES

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Lectins are a group of proteins of nonimmune origin widely distributed in nature. These proteins recognize and bind specific carbohydrates found on the surfaces of cells and play a role in interaction and communication between cells, as well as interaction with glycoconjugates. Legume lectins are an extensive group with homogeneous physicochemical and structural properties, but very different biological activities. Inside the legume lectin group, most of the studies focus on the Papilionoideae subfamily, with the other two, Caesalpinioideae and Mimosoideae being significantly less explored in the literature. The focus of the present research is the Mimosoideae subfamily, the second largest in number of taxa, comprising four tribes (Mimoseae, Mimozygantheae, Acacieae and Ingeae) with about 3275 species and 79 genera, but with only about 14 lectins purified lectins, which depicts the lack of studies for this group. To date, researchers have reported on the purification, characterization and some biological activities of the genera Parkia, Leucaena, Caliandra, Archidendron, Anadenanthera, Albizia and Acacia. The applied methodology was based on search and filtering of papers in the literature with posterior data extraction and compilation. Based on their purification protocols, as well as physicochemical properties, Mimosoideae lectins are very heterogeneous lectins. These differences become even more evident when these proteins are analyzed from a structural point of view. Although we have a few purified lectins from this subfamily, the structural studies to this date only include lectins of the Acacia and Parkia genera, and these are shown to be remarkably distinct from one another. Despite the few studies, a wide variety of biological activities have been tested, e.g. inflammatory, anticancer, antibacterial, and antifungal. In this context, considering the number of taxa belonging to the subfamily Mimosoideae and the importance of its lectins, this study aimed to present an overview of the available literature reporting the purification, structure, and biological activities of these proteins. In this context, synchrotron beamlines can help in the definition of the structure of each lectin by allowing structural solving by X-ray crystallography and other techniques such as SAXS. Several lectins already had their crystal structure solved, and the combined data of several crystal structures assisted researchers in the definition of several structural and functional properties of these proteins, but some groups of proteins still need more studies in this line with Mimosoideae lectins being a prime example.

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KEYWORDS: LECTIN, MIMOSOIDEAE SUBFAMILY, PROPERTIES, STRUCTURE

POSTER PRESENTATION

LITHIUM AND CERIA INDUCED LIFETIME IMPROVEMENT OF SMART ANTI-CORROSION HYBRID COATINGS

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Organic-inorganic coatings based on polymethyl methacrylate (PMMA)-silica and PMMA-CeO_2 provide an excellent barrier against corrosion of steel and aluminum alloys, thus representing an eco-friendly and economical alternative for conventional protection systems. In addition to their effectiveness as a physical barrier, the addition of lithium and ceria can turn them into smart coatings showing recovery after corrosion. The key factor to develop smart high-performance coatings is an in-depth knowledge of the structure, accessed by a set of surface and bulk characterization techniques. Transmission electron microscopy and small-angle X-ray scattering results showed that covalent bonds between silica or ceria nanodomains with the PMMA matrix through coupling molecule are crucial for a cross-linked structure that acts as an efficient diffusion barrier. The addition of lithium carbonate yields coatings with increased connectivity of the silica domains, uniformly distributed in the PMMA matrix. Concerning the PMMA-CeO_2 coatings, a bimodal set of CeO_2 nanoparticles with size of ~0.4 and 1.9 nm is formed, homogeneously dispersed in the organic phase. Results of electrochemical impedance spectroscopy of coated alloys, immersed in 3.5% NaCl, showed that the addition of Li and Ce increases the impedance modulus (up to 100 GΩ cm2) and significantly improves the coating lifetime by the self-healing ability (up to 240 days). Time-of-flight secondary ion mass spectrometry and X-ray photoelectron spectroscopy suggest that the regeneration occurs by leaching of Li or Ce from the surroundings of the corrosion spot, which is restored by a protective layer of precipitated species. An analog mechanism was found for artificially scratched coatings showing an impedance modulus increase after immersion compared to the Ce/Li free reference. These results evidence the dual role of lithium and ceria in improving the structure and extending the coating lifetime by the self-healing ability.

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KEYWORDS: PMMA-SILICA, PMMA-CEO2 HYBRID NANOCOMPOSITE, ANTICORROSIVE COATING, SELF-HEALING, ALUMINUM ALLOY, CARBON STEEL

POSTER PRESENTATION

MAGNETIC PROXIMITY EFFECT IN Pt/Co/Pt MULTILAYERS ESTIMATED BY Co L-EDGE X-RAY MAGNETIC CIRCULAR DICHROISM AND CONVENTIONAL MAGNETOMETRY

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Magnetic multilayers with heavy metal/ferromagnetic interfaces play a crucial role in data storage and spintronics as they can present perpendicular magnetic anisotropy, spin-transfer torque phenomenon and host magnetic skyrmions. While studying their macroscopic magnetic properties by conventional magnetometry, a relevant issue is to determine a sample's saturation magnetization because one cannot know the amount of Pt which gets polarized due to magnetic proximity effect (MPE). In this work, we show that the element specific characterization provided by X-ray magnetic circular dichroism can help to solve this problem even if measurements are acquired only around one absorption edge. We estimate the volume of Pt that contribute to the magnetization for different Co thicknesses (0.5-1.5 nm) in Pt/Co/Pt multilayers. We measured the samples total magnetic moment by vibrating sample magnetometer (VSM) and only Co contribution with X-ray magnetic circular dichroism (XMCD) at the Co L2,3-edge. Combining these two techniques we estimated the Pt magnetic moment. We observed that the thinner the Co layer, the higher the Pt total magnetic moment. Our hypothesis is that thinner samples present a larger interface area due to the increasing of the roughness. Our results show the importance of the proximity effect to determine the real magnetic parameters of ultrathin films.

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POSTER PRESENTATION

MANGANESE SYSTEMIC DISTRIBUTION IS MODULATED IN VIVO DURING TUMOR PROGRESSION AND AFFECTS TUMOR CELL BEHAVIOR IN VITRO

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Metastasis remains the leading cause of death in cancer patients and understanding the involved mechanisms continues to be a challenge. This work aims to investigate the role of manganese (Mn) in tumor progression. Our main goals include (1) using SR-XRF to evaluate if tumors affect physiological Mn distribution and content in vivo and; (2) using in vitro models to evaluate if tumor cells are affected by changes in available Mn. SRXRF elemental maps were generated from tissue sections from mice submitted to an in vivo model of tumor growth (UFRJ-CEUA approved protocol: 015/18). Maps revealed that Mn accumulates within primary tumors and secondary organs, creating Mn-rich niches, without alterations in Mn systemic levels. Consequences of such phenomenon were investigated in vitro, and we verified that short-term changes in Mn concentration cause long term effects on tumor cells, altering the cell surface molecules syndecan-1 and β1-integrin distribution, enhancing invasive behavior. Besides, tumor cells were cultured under modified conditions, whereas cations were removed from cell medium using dowex ion exchange resin and reintroduced accordingly. Cells cultured in complete medium (all cations replenished) and Mn-reduced conditions maintain proliferation, however, cells in Mn-reduced culture condition present altered behavior regarding migration pattern. Nevertheless, when exposed to a short-term increase in Mn concentration, cells cultured in Mn-reduced conditions migrate similarly as control cells. In conclusion, our analyses point to Mn as a relevant element in tumor progression due to the observation of Mn-rich niches in vivo that may promote tumor cell short-term exposure to this element, enhancing migration without necessarily affecting cell viability. Manganese accumulation in specific regions of the organism may not be a common ground for all cancers, nevertheless, it represents a new aspect of tumor progression that deserves special attention.

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KEYWORDS: MANGANESE, CANCER, X-RAY FLUORESCENCE, CELL MIGRATION

POSTER PRESENTATION

MECHANOCHEMISTRY ON TRANSITION METAL CHALCOGENIDE NANOPHASES SYNTHESIS, RIETVELD AND DEBYE FUNCTION ANALYSIS FOR MICROSTRUCTURAL CHARACTERIZATION FROM POWDER DIFFRACTION

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Nanocrystalline materials synthesized by our group have shown several technological applications [1-3] in the last decades, all of them made through the mechanochemistry route. This work aims to present a new approach in performing the microstructural analysis of the nanocrystalline samples. For that, a critical comparison is showed of two methods: 1) the Rietveld method included in TOPAS [4] and GSAS II [5] for X-Ray Diffraction (XRD) and 2) the Debussy [6] (Debye User's System) suite presenting the Debye Function Analysis (DFA) for Wide Angle X-Ray Total Scattering (WAXTS). Here, the first one uses the well-known Rietveld refinement procedure [7] that gives several useful structural and microstructural information, allowing the anisotropic modeling of size, deformation, and thermal parameters, verifying its plausibility by the Hamilton test [8]. The last tool, Debussy, makes use of a new approach where the Debye's equation takes part to calculate a theoretical pattern from the modeling of virtual sets of nanoclusters of different sizes and shapes. The XRD experiments take place at the LDRX-UFSC diffractometer (8keV radiation) and WAXTS at LNLS-XRD1 beamline (14keV), and at SLS-X04SA-MS beamline (22KeV). It is also the plan here to discuss the procedures to obtain diffraction data free some of the experimental contributions and taking into account absorption and packing (density) effects from the real sample, which is needed for DFA. For illustration, the microstructural analyses of the mechanical alloyed $Ni_{33}Te_{67}$, which contains a NiTe2 nanophase of space group P-3m, are presented. Rietveld analysis considering spherical crystallites shows the average domain size of 20 nm and isotropic microstrain of 0,633 with a reasonable data fitting, wR of 7.5% and GoF 14.30. DFA presented a size distribution of spherical crystallites centered at 15 nm with 7 nm of variance and a poor pattern description with wR of 15% and GoF of 28. The comparison between the results of the two methods cannot be directly done because the size distribution was not implemented in Rietveld as well as microstrain was not in Debussy. These two points are the next goals of our research in the field.

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KEYWORDS: XRD, TOTAL SCATTERING, DEBUSSY, RIETVELD, MECHANOCHEMISTRY

POSTER PRESENTATION

MINERALOGICAL TRANSFORMATIONS INDUCED BY MICROWAVE HEATING TREATMENTS IN PORPHYRY COPPER ORE

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Electromagnetic waves in microwave (MW) frequency is gaining notable attention in last years as a source of rapid and volumetric heating of many materials, such as polymers, ceramics, rocks and mineral ores, and so on. In general, metal-oxides and sulfides heat rapidly (which are constituents of a wide variety of minerals) while rock-forming minerals responds differently, not heating quickly, which depends on the microwave absorption characteristics of each [1]. The copper ore studied here was jaw-crushed and dry sieved in 14, 28, 50 and 100 mesh sieves until approximately 100 g samples of ore in each particle size range were obtained. For microwave treatment of the sieved ore samples, a 2.45 GHz multimode cavity microwave oven with 900 W of forward power was used, with 10, 15 and 20 minutes of treatment for each particle size distribution. Runs of 10g copper ore mixed with small amounts (less than 1g) of commercial carbon black (which is a high microwave absorbing material) were conducted with 1.8 kW of forward power during 20 minutes, leading to the melting of copper ore. The mineralogical composition before and after 20 minutes of microwave treatment with 1.8 kW were characterized by X-ray diffraction by synchrotron radiation source at XRD1 beamline (Brazilian Synchrotron Light Laboratory – LNLS/CNPEM) in a Debye-Scherrer geometry. This diffractometer configuration ensures a rapid but accurate measurement [2]. The chemical composition of samples was investigated in a FEI Inspect S50 scanning electron microscopy coupled with EDS detectors (LCE – UFSCar). Based on the analysis of the mineral phases present in the non-treated samples and treated samples, the major mineralogical transformation induced by microwave treatments (due to thermal and non-thermal effects) was magnetite to hematite and chalcopyrite to bornite transformations, the latter leading to increase in copper content.

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POSTER PRESENTATION

MORPHOLOGY OF SILICA-BASED MATERIALS MODIFIED WITH ZIRCONIUM OR TITANIUM AND PMODS FOR SOLID PHASE EXTRACTION BY SCANNING ELECTRON MICROSCOPY

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In the study of complex aqueous environmental samples for the determination of organic pollutants, as pesticides, the step of eliminating interferents that may compromise the quality and reliability of the analytical results due to the matrix effect is very important. Among the several techniques for solving problems related to the matrix effect in the extraction of complex samples, solid phase extraction (SPE) is the most used technique due to its lower consumption of organic solvents complying with the principles of Green Chemistry and the possibility to enhancing the concentrations of analytes to posterior instrumental analysis by gas or liquid chromatography (GC or LC) [1,2]. SPE uses similar solid sorbent materials as in the stationary phases of LC which have the function of extracting the analytes of interest from the matrices, being those based on silica modified with alkyl groups like C-18 or C-8 [3]. In this work, labmade sorbent materials were prepared modifying commercial silica structure (100 μm) with Zr and Ti alkoxides and poly(methyloctadecylsiloxane) (PMODS) (C-18). The morphology of the materials were characterized using a scanning electron microscope FEI Inspect F50 at different magnifications. The scanning electron microscopy (SEM) images indicated that the materials had irregular particle morphology with the mean size of approximated 95 μm. The presence of PMODS, coating the materials surface structure also could be observed in both Silica-Zr(PMODS) and Silica-Ti(PMODS) sorbents, showing a suitable hydrophobic layer coating for extracting pollutants, as the pesticides, from aqueous environmental samples, using the reversed phase separation mechanism [4], and the results were compared with those obtained by Fourier transform infrared (FTIR) spectroscopy .

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KEYWORDS: SOLID PHASE EXTRACTION, SCANNING ELECTRON MICROSCOPY, SILICA BASED MATERIALS

POSTER PRESENTATION

MÖSSBAUER SPECTROSCOPY AT SIRIUS: IS IT POSSIBLE?

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Mössbauer spectroscopy (MS) is a powerful tool with broad application in many areas of science and technology, including nuclear physics, condensed-matter physics, chemical physics, materials science, life science, medical science, earth science, planetary science and environmental science. The use of synchrotron radiation (SR) sources to assess Mössbauer resonances [1] provides a combination of high energy resolution and isotopic sensitivity of the Mössbauer effect, together with the SR properties (high brilliance, tunability in energy and defined time structure) [2]. This affords a wide variety of possibilities, such as, analyzing: very small areas, nuclei or interesting isotopes that don't have a natural radioactive decay, very fast measurements, etc. However, the use of SR for performing MS is not straightforward. While MS is an absorption spectroscopy, and relays on the absorption of one gama-quantum by one nucleus, the use of SR is based on nuclear resonant scattering (NRS), where one gama-quantum is scattered by an ensemble of nuclei. In this work, a brief overview of NRS and some own particularities are discussed. Based on a detailed presentation of the technical specifications of the facilities that perform NRS, an initial discussion about the possibility of carrying out these experiments at Sirius facilities is made, pointing out the main differences and, mainly, the inherent difficulties. Thus, we start the discussion about the technological challenges for the possibility of NRS implantation on SIRIUS.

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KEYWORDS: MÖSSBAUER, SYNCHROTRON RADIATION, NUCLEAR RESONANT SCATTERING

POSTER PRESENTATION

NEW EUIII PYROMELLITIC METAL-ORGANIC FRAMEWORK OF INTENSE RED ORANGE LUMINESCENCE AND HIGH THERMAL STABILITY FOR MARKING IN GUNSHOT RESIDUES

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The preparation, general characterization, and experimental and theoretical photoluminescence investigations of a new europiumIII metal–organic framework [EuHbtec]n 1 supported by 1,2,4,5-benzenetetracarboxylic acid, H4btec (pyromellitic acid), are discussed in this work. The crystal structure was obtained from X-ray powder diffraction (XRPD), with the data collected at the MCX beamline of ELETTRA synchrotron. In this structure, the monoprotonated Hbtec3– ligands act in three different coordination modes to EuIII centers: μ2-η1:η2 chelating/bridging, η2 chelating, and η1 monodentate modes. The three-dimensional (3D) network is constructed by secondary building units (SBU) chains, connected through carboxylate groups of the Hbtec3– ligands. Using the Sparkle/RM1 model, the ground-state geometry of [EuHbtec]n 1 was calculated in the solid phase and then used to obtain the several investigative spectroscopic parameters. This study demonstrates that intense luminescence occurs through ligand sensitization. The exceptional thermal stability is proven by thermogravimetric analysis, with the network collapsing at 489 °C, the largest among the congener compounds. Thus, by combining its intense emission and high thermal stability, [EuHbtec]n 1 was utilized for marking of gunshot residues (GSRs), with its preliminary tests showing very promising results for applications in forensic contexts and public safety.

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POSTER PRESENTATION

Ni SOLUBILITY IN $Ce_{0.9}Zr_{0.1}O_2$: EXAFS EVIDENCE OF THE FORMATION OF AN AMORPHOUS PHASE OF Ni

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Cerium dioxide (CeO_2, ceria) is one of the materials with widest range of applications. When doping with Zr, resulting materials could be used as active support for anodes of solid oxide fuel cells (SOFC), or as active catalysts for methane oxidation [1]. Materials used as anodes in a SOFC should have a good level of electronic conductivity, and for that reason many researchers have studied ceria-based anodes with transition metals. Inclusion of transition metals into the crystal lattice of ceria is not a trivial issue. It strongly depends on preparation method and thermal treatment, and there are some disagreements in the literature about solubility limits. In this work we analyzed the system $(Ce_{0.9}Zr_{0.1})_{1-x}Ni_xO_{2-d}$ (x = 0, 0.03, 0.05 y 0.1), with the aim to determine whether Ni is included or not into the ceria-zirconia lattice. The crystal structure of the samples was analyzed by X-Ray diffraction and by means of HR-TEM (images and elemental maps of the samples were obtained). The local order of Ni was studied by extended X-ray absorption fine structure (EXAFS) around the Ni K-edge, in the D08B-XAFS2 beamline of the Brazilian Synchrotron Light Source (Proposal 20180396). The results of this study give some insight of the existence of an amorphous phase of Ni in samples with low content of Ni and low temperature thermal treatment. Results of XRD show that the lattice parameter remains constant in all the range of Ni contents analyzed, and there is no evidence of phase segregation for samples calcined at 600ºC and $x \leq 0.05$. This is supported by TEM observations, and it was concluded by EXAFS that there is no long range order. Absorption as a function of wavenumber and Fourier transformed plots exhibit the typical behaviour of an amorphous phase. Samples heat treated at T □ 800°C or with Ni contents higher than 5%mol, exhibit a crystalline NiO phase detected by XRD, HR-TEM and EXAFS.

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POSTER PRESENTATION

PARTIAL CATIONIC ORDER AT THE B SITE OF THE N = 3 RUDDLESDEN-POPPER PHASES $LaSr_3(Fe,Co,Ga)_3O_{10-d}$ STUDIED BY NEUTRON POWDER DIFFRACTION AND X-RAY ABSORPTION SPECTROSCOPY

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We have studied the crystal chemistry of the Ruddlesden-Popper (RP) phases $LaSr_3(Fe,Co,Ga)_3O_{10-d}$ with tetragonal symmetry by Neutron Powder Diffraction (NPD) and X-ray Absorption Spectroscopy (XAS) with bdifferent contents of Fe, Co and Ga. NPD data shows the oxygen vacancies are located at the oxygen crystal sites at the central octahedra of the perovskite block in the RP structure. Fe, Co and Ga cations are not homogeneously distributed, showing a sort of preference for particular crystal sites in the perovskite block. For instance, Fe cation tends to accommodate rather at sites with a lower concentration of oxygen vacancies, i.e. at the crystal sites of the perovskite layers next the rock salt layer, whereas Co and more markedly Ga prefer the sites at the central perovskite layer. This cationic distribution was confirmed by Extended X-ray Absorption Fine Structure (EXAFS) analysis, which shows the average coordination number of Fe is always higher than the coordination number obtained for Co and Ga. X-rays Absorption Near Edge Structure (XANES) analysis suggests that the average valence of Fe is always higher than Co and both are higher than 3+ of Ga cations. The results of both EXAFS and XANES analyses are in agreement with the preferential location of the oxygen vacancies at the central perovskite layer where Co and Ga cations are majorly located according to NPD data. The combination of NPD and XAS has been proven effective to assess the crystal chemistry and defect structure of the $LaSr_3Fe_xCo_yGa_{3-x-y}O_{10-d}$ system.

KEYWORDS: RUDDLESDEN POPPER PHASES, CRYSTAL STRUCTURE, NEUTRON POWDER DIFFRACTION, XAFS

POSTER PRESENTATION

PERFORMANCE EVALUATION OF SEGMENTATION METHODS OF T. MILIARIS TADPOLE IMAGES USING SYNCHROTRON MICRO-CT

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Investigation of the biological structures by X-ray microtomography has been growing over the past decades due to the improvement of imaging techniques, in particular, those based on the phase-contrast mechanism [1, 2]. Such techniques allow access to internal structures of specimens without causing damage to them, which is of great importance when working with biological samples. For the purposes of biomedical imaging of small specimens, the analysis of image datasets requires computational tools to produce volume renderings, which can be segmented to provide, for example, qualitative visual information and quantitative relevant individual structures [3, 4]. Among the computational tools, Deep Learning can be highlighted as a promising tool for segmentation imaging. In this work, micro-CT experiments were carried out on the IMX beamline at the Brazilian Synchrotron Light Source Facility (UVX-LNLS), Brazil. The specimen scanned was Thoropa miliaris tadpole positioned at 200 mm away from the detector with 4.11 μm effective pixel size and field of view of about 8.42 x 8.42 mm², 1000 projections were obtained in 0.18 degree steps. The whitebeam was filtered with 200 μm Silicon resulting in 11 keV mean energy and the projections were acquired in the phase-contrast regime using propagation-based imaging. Data scans were reconstructed by Filtered Backprojection (FBP). T. miliaris tadpole lens was the region of interest (ROI) chosen to be segmented using two types of semiautomatic segmentation, Interpolation and Watershed, and an automatic segmentation algorithm by using U-Net architecture. The performance of these segmentation methods was assessed using the Dice Similarity and volume quantification of the ROI. The results showed high accuracy with a significant reduction of segmentation time using the automatic method.

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KEYWORDS: SYNCHROTRON RADIATION, MICROTOMOGRAPHY, BIOLOGICAL IMAGING, DEEPLEARNING, IMAGE SEGMENTATION

POSTER PRESENTATION

POLYELECTROLYTE COMPLEXATION: A PROMISE WAY TO IMPROVE MECHANICAL PROPERTIES AND WATER RESISTANCE OF NANOCELLULOSE FOAMS

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Nanocellulose porous materials present a wide range of applications based on their low-density and high surface area. However, its fragility and solubility can be a drawback in many systems. Here, we present a simple and scalable method to prepare robust all-cellulose foams by electrostatic complexation between oppositely charged cellulose nanofibers (CNF). CNF foams exhibited maximum stiffness (ca 7 MPa) at the mass composition of 1:1, which indicates that meaningful attractive interactions happen at this point and act as stiffening structure in the dry material. Besides the electrostatic attraction, an increase in the density of hydrogen bonds and hydrophobic contacts may also occur within the fibers clustering, improving the water stability of foams. Supramolecular assemby between negative and positive CNFs in aqueous medium prove to be an elegant way to modify viscosity and structure of the suspensions, as demonstrated by viscosimetric and SAXs experiments, leading to different foam's architectures. These results may provide a basis for the development of new approaches to prepare insoluble and mechanically resistant CNF foams.

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KEYWORDS: ELECTROSTATIC COMPLEXATION, CATIONIC CELLULOSE NANOFIBERS, ANIONIC CELLULOSE NANOFIBERS, FOAMS

POSTER PRESENTATION

POLYMERIC BASED FAST SYNTHESIS OF SIMPLE BINARY COPPER (I) AND (II) OXIDES POROUS SUPERSTRUCTURES

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Due their bandgap, both copper (I) and (II) oxides are found in several as gas sensing, next-generation rechargeable lithium-ion batteries and fabrication of solar cell, since it exhibits high solar absorbance [1]. Moreover, this material can be applied as H2 solar photoelectrochemical electrode. Porous structure can enhance the H2 production, due their large surface area. In this work, binary oxide nanoparticles containing copper I and II oxides were easily synthesized by fast and non-expensive polymeric solution method [2], where copper forms chelates with the soluble polymer. They were stabilized with HNO3 addition to avoid precipitation and fast dried under microwave radiation. The dried resin was analyzed by TGA/DTA. The resin was decomposed at expressive temperatures. The synthesized powders were grounded and sived (325 MESH) and characterized by X-ray diffraction at XRD1 beamline, under radiation wavelength of 1.033 nm. Both Cu2O and CuO were found above 200 °C, but only CuO remains above 400 °C. in all temperatures. After a Rietveld refinement, the lattice parameters of Cu_2O changed due the defect formation, which enhances the transformation to CuO at higher temperatures. On the other side, due the changes in oxygen partial pressure during the calcination, the lattice parameters of CuO changes drastically. The crystallite size of Cu_2O increases from 250 (14 nm) to 300 °C (30 nm), but CuO crystallites remains practically constant until 350 °C (around 15 nm), growing drastically above this temperature, reaching around 50 nm. At 300 °C, the .

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POSTER PRESENTATION

PRELIMINARY RESULTS OF SYNCHROTRON RADIATION DOSIMETRY AND PUTATIVE IMPACT ON BIOLOGICAL SAMPLES IN MOGNO (MICRO/NANO TOMOGRAPHY) BEAMLINE

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Longitudinal experiments are crucial to study biological dynamics in living samples. Among several techniques, bioimaging is a paramount for cellular biology up to systemic events. On the other hand, these samples are highly sensitive to ionizing radiation. Therefore, synchrotron radiation damage is a concern for biological tissue [1]. It's important to understand the effects from cellular scale to the whole organism as well as to distinguish significances between whole body irradiation and partial exposure (tissues or cells). The Mogno micro and nano tomography beamline will have a multilayer KB mirror with a conical and quasi-monochromatic beam with three energy setups: 22, 39 and 67.5 keV; with FOV from 120 nm x 120 nm to 85 mm x 85 mm. The high brightness of synchrotron light at Mogno beamline makes it possible to obtain high spatial and temporal resolution and to improve the image contrast. However, it also increases the amount of energy deposited in the sample. The in-depth knowledge of biological effects passes through computational dose deposition simulations in different scenarios as well as experimental measurements. The radiation dosimetry calculations are being conducted in FLUKA [2] and PENELOPE [3] Monte Carlo (MC) codes to help planning a setup for experimental study with thermoluminescence (TL) and optically stimulated luminescence (OSL) dosimeters. Preliminary results show optimistic scenarios for bioimaging with dose-rate from 10^{-4} to 10^{3} Gy/s/350mA. The dose rate at samples can be correlated with the damage in cells and explanted tissues. Results obtained in Geant4-DNA simulations will be compared to the experiments with yeast, high-dose resistant bacteria and eukaryotic cells intended to quantify the impact in cellular metabolism, respiration, DNA and chromosomal damage. Ultimately, all these steps will support the forthcoming experimental setup with in vivo small rodents.

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KEYWORDS: BIOIMAGING, IN VIVO TOMOGRAPHY, SYNCHROTRON RADIATION DAMAGE, MONTE CARLO SIMULATION, FLUKA, PENELOPE, GEANT4-DNA

POSTER PRESENTATION

PRESSURE-DEPENDENT STUDY OF $PbMn_7O_{12}$

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$PbMn_7O_{12}$ (PMO) belongs to the family of AMn_7O_{12} quadruple perovskites, with several these members exhibiting interesting multiferroic properties. The PMO was synthesized under high pressure (7.5 GPa) and high temperature (1373 K) conditions [1] and, similarly to $CdMn_7O_{12}$, $SrMn_7O_{12}$ and $CaMn_7O_{12}$ [2], crystallizes in the R-3 space group at room temperature. Temperature dependent analysis show that PMO undergo structural phase transition around 397K and a second phase transition related to orbital ordering at 298 K. Also, there are some magnetic transitions at 83, 77 K and 43 K. In addition, its spontaneous electric polarization below 77 K classifies PMO as a spin-driven multiferroic compound [1,3]. In this work, we present a pressure dependent behavior analysis of PMO. The sample used was synthetized through high pressure and high temperature method. The lattice parameters of PMO were monitred from 0GPa unti 22GPa and two possible phase transitions were observed at 8.5 GPa and 19 GPa. These structural changes are similar to those observed in another quadruple perovskite CaMn7O12 [4].

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POSTER PRESENTATION

PRESSURE-INDUCED STRUCTURAL PHASE TRANSITION IN MULTIFERROIC KBiFe$_2$O$_5$

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The research about renewable energy sources has attracted attention since there is a high demand for clean energy around the world. In the case of solar energy, a prominent field is the investigation of multiferroic materials for applications in photovoltaic cells. In this scenario, KBiFe$_2$O$_5$ (KBFO) emerged as a strong candidate for such applications given mostly by its low bandgap energy [1]. At room temperature, KBFO crystallizes in an orthorhombic structure and possesses interesting electronic properties making it suitable also for photovoltaic applications. Besides, it is reported that such structure transforms into a monoclinic one in very high temperatures and pressures. In this work, we obtained the monoclinic structure of KBFO at room temperatures and performed an analysis of the structural and vibrational properties of KBFO using Raman spectroscopy and Synchrotron X-ray powder diffraction (SXRPD) techniques under hydrostatic pressure variation. The study of Raman spectra obtained at high pressures revealed a first order phase transition in the material, characterized by a pressure hysteresis. Synchrotron X-ray powder diffraction (SXRPD) under varying pressure was employed to confirm such structural pressure-induced phase transition. An integrated evaluation of the results obtained by Raman and SXRPD techniques indicates that, possibly, the symmetry for the high-pressure crystalline phase belongs to the space group Pmmm [2].

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KEYWORDS: KBIFE2O5, BROWNMILLERITE, SYNCHROTRON, RAMAN SPECTROSCOPY, PHASE TRANSITION, MULTIFERROIC, PRESSURE

POSTER PRESENTATION

PRESSURE-INDUCED STRUCTURAL PHASE TRANSITION OVER THE LOW-DIMENSIONAL HALIDE PEROVSKITE-RELATED $CsPb_2Cl_5$ COMPOUND

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The perovskites-related are ideal materials for photodetectors and solar cell applications due to their low-cost raw-element constitution and high-quality optoelectronic properties. The band-gap binding energy, charge transport, and low-carrier mobility are characteristics that are directly influenced by crystal dimensionality [1,2]. Actually, novel investigations on low-dimensional materials are being described, exploring the physical-property modification over extreme conditions, in this context, we present the $CsPb_2Cl_5$ single-crystal; the compound was successfully prepared by the slow evaporation method. The sample crystallizes in a tetragonal system, with the space group I4/mcm and lattice parameters at room conditions. The X-Ray Powder Diffraction measurements over pressure until 8.7 GPa were performed in the XDS beamline at the Brazilian Synchrotron Light Source Laboratory (LNLS) with monochromatic synchrotron radiation of λ=0.61999 Å. Our results suggest an isostructural phase transition at 2.1 GPa, followed by a structural phase transition at 3.5 GPa to an orthorhombic crystal system. The crystal structure at each pressure was refined by the Rietveld method using the software GSAS-II.

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KEYWORDS: 2D PEROVSKITE, RIETVELD REFINEMENT, SYNCHROTRON X-RAY DIFFRACTION

POSTER PRESENTATION

PRESSURE-INDUCED STRUCTURAL PHASE TRANSITIONS IN THE MULTIFERROIC AURIVILLIUS $Bi_5FeTi_3O_{15}$

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Multiferroic materials are well known as scientifically promising materials because of their intriguing ferroic properties. In certain compounds, these ferroic properties are directly related to their structures and present different coupling effects[1]. The versatility of such properties allows these materials to have a range of technological applications and, therefore, a better understanding of the structural behavior of these compounds become essential. In this study, the structural stability of the multiferroic four-layer Aurivillius ceramic $Bi_5FeTi_3O_{15}$ (BFTO)[2] under hydrostatic pressure was investigated in situ by synchrotron X-ray powder diffraction (SXRPD) and Raman spectroscopy. Measurements were performed up to 15 GPa allowing the identification of two well-defined structural phase transitions. The ambient orthorhombic phase transforms into a tetragonal one around 3.2 GPa and a new orthorhombic phase rises beyond 7.5 GPa. Both phase transitions are evidenced by changes in pressure dependence of the lattice parameters and vibrational mode frequencies. Further analysis of the induced strain indicates that the transitions are, respectively, of second-order and first-order. Subtle changes in the Raman spectra and powder patterns suggest the onset of a new phase above 10.5 GPa[3].

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KEYWORDS: MULTIFERROIC, HIGH-PRESSURE, RAMAN SPECTROSCOPY, X-RAY POWDER DIFFRACTION

POSTER PRESENTATION

PRESSURE-INDUCED STRUCTURAL PHASE TRANSITIONS ON MULTIFERROIC $CaMn_7O_{12}$

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$CaMn_7O_{12}$ (CMO) recently emerged as a unique multiferroic material exhibiting the largest recorded magnetically-induced electric polarization. The origin of the electric polarization in this compound is due to two effects contributing equally: the exchange striction interaction, which determines the polarization magnitude, and the Dzyaloshinskii-Moriya (DM) interaction, driving the polarization direction. At low temperatures, CMO undergoes an unusual phase transition, exhibiting an unconventional incommensurate orbital ordering at 250 K. Such orbital ordering stabilizes the CMO chiral magnetic structure. Two antiferromagnetic (AFM) transitions were recorded at TN1 = 90 K, where ferroelectricity appears, and TN2 = 45 K, associated with multiple propagation vectors, but no additional structural phase transitions were observed below 250 K. Despite the thorough discussion on the CMO ferroelectric and magnetic properties, few investigations were focused on the structural phase transitions (SPT) undergone by this compound. At high temperatures, CMO crystallizes exhibits in a cubic structure belonging to the Im3 space group. Around 440 K it undergoes a simultaneous charge-ordering and SPT transforming into a rhombohedral structure with R3 symmetry. Recently, the effect of the Sr-substitution on CMO, which generates a ”negative” chemical pressure in the structure since Sr ion is bigger than Ca, was investigated. The authors observed a decreasing in the R3 → Im3 SPT temperature, but any change on in the electric properties was observed. These results suggested that high-pressures could induce structural changes in CMO. Thus in this work, we showed that CMO undergoes two structural phase transitions under hydrostatic pressure. CMO transforms into a monoclinic structure around 9.9 GPa and undergoes a shear of monoclinic phase for pressures higher than 13.3 GPa.

POSTER PRESENTATION

QUATI: THE QUICK-EXAFS BEAMLINE AT SIRIUS

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QUATI (QUick X-Ray Absorption Spectroscopy for TIme-Resolved experiments) is a beamline dedicated to high quality X-ray absorption spectroscopy experimentsand in-situ/in operando studies, in XANES (X-Ray Absorption Near Edge Structure) and EXAFS (Extended X-ray Absorption Fine Structure) modes, allowing measurements in the time scale of milliseconds. A multitechnique approach will be performed: XRD, Raman, and IR coupled with XAFS experiments. X-ray Emission Spectroscopy (XES) and related techniques will be accessible by a Von Hamos spectrometer, this choice is to enable fast scan acquisition.

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Thanks to LNLS staff and groups that collaborate in this project. Authors give a special thanks to Frederico Lima for the initial XES Implementation.

KEYWORDS: BEAMLINES, XAFS, XES, X-RAY ABSORPTION

POSTER PRESENTATION

ROCK WOOL FIBERS DISPERSION IN NITRILE RUBBER ASSESSED BY X-RAY COMPUTED TOMOGRAPHY

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Acrylonitrile-butadiene elastomers (NBR) are widely used on the oil and automotive industries in hoses, sealing rings, gaskets, fuel pipes, and many other rubber products due to its good resistance to oil, apolar solvents and temperature. However, a NBR artifact needs to be vulcanized and reinforced with fillers in order to present those good properties. Carbon black is the most well-known and used filler for rubber, however other types of filler such as short length fibers are subject of recent studies. This study assesses different types of rock wool fibers (RWF) with different lengths; 100 µm and 300 µm, with and without surface chemical modification on NBR composites using X-ray computed tomography. The scans were carried out in a Bruker SkyScan 2211 multiscale Nano-CT with a 0.9 µm pixel size. Nine formulations with 10, 25 and 40 parts per hundred rubber (phr) of 03 different RWF types were prepared in a standard two-roll mixer based on the parameters described on ASTM D-3187. After the mixing procedures, rubber compounds were left 24 hours in a room with 23 ± 2 °C and moisture not higher than 50% to rest. Afterwards the compounds were vulcanized in a hydraulic press at 160 °C, 180 kgf/cm^2, using the optimum cure time ($t90$) obtained through their rheometric characteristics as time parameter (ASTM D-2084). The X-ray computed tomography analysis allowed us to evaluate three major parameters. The arrangement of the fibers in the rubber matrix, exposing whether a preferential orientation was achieved, the fiber volume fraction actually dispersed in the rubber matrix, and the aspect ratio (length/diameter) of the fibers, which is important to determine crosslink density using a fast method on a Rubber Process Analyzer instead of a high time consuming swelling test. The Mogno beamline at SIRIUS synchrotron facility will provide faster and more precise measurement with submicrometric resolution, which is essential for the future characterization of our samples.

KEYWORDS: NITRILE RUBBER, ROCK WOOL FIBERS, X-RAX COMPUTED TOMOGRAPHY

POSTER PRESENTATION

Ru(II) POLYPIRYDYL COMPLEXES: A THEORETICAL AND SPECTROSCOPIC STUDY

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Ruthenium(II) polypirydyl complexes have been widely studied in the last decades because of their applicability on a large number of research areas, such as the development of dye-sensitized solar cells or the design of drugs for photodynamic therapy [1]. This work uses DFT calculations to generate theoretical structures for EXAFS data fitting of a series of 12 Ruthenium(II) polypirydyl complexes, with [Ru(bpy)2L]2+ formula, where bpy is the 2,2'-bipyridine and L is an extended conjugated ligand derivate from 1,10-phenantroline. Tris procedure allowed the elucidation of their first coordination sphere of Ru(II) in these compounds. To accomplish this goal, DFT geometry calculations at RI-PBE/def2-SVP level, with acetonitrile solvent effects included with COSMO method were done for all compounds. Then, the optimized geometry coordinates obtained were used as a FEFF input. The first coordination sphere considered the nitrogen atoms of the L ligand, the axial bipyridine nitrogen atoms and the equatorial bipyridine nitrogen atoms as showing different chemical environment, and the outputs were fitted to the experimental data obtained on XAFS1 line of LNLS. The fitting results showed an excellent correlation between experimental data and the theoretical models for all complexes. For the complexes already described on literature, the results were compared with x-ray diffraction available data, and also showed a good correlation [2,3,4]. The theoretical methodology to generate geometries for EXAFS fitting was able to describe adequately the first coordination shell for all complexes, which is a very good strategy to study reaction mechanisms involving Ru–N bond lengths changes, as for example, photochemical reactions involved in DSSC solar cells.

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KEYWORDS: RUTHENIUM POLYPIRYDYL DFT EXAFS

POSTER PRESENTATION

SAXS IN-SITU TEMPERATURE DEPENDENT STUDY OF GOLD NANOSTRUCTURES SYNTHESIS

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Ultrathin gold nanowires (AuNWs) have attracted great interest due to their unique conductivity properties making them suitable candidates for a myriad of applications. Many relatively simple syntheses protocols have been reported using gold salts and surfactants in organic media, in general $HAuCl4 \cdot 3H2O$ and oleylamine (OAm) in hexane, resulting in micrometer long wires with sub 5 nm diameters. Comparing similar reported procedures, the OAm/Au molar ratio appears to be of crucial importance to lead the synthesis toward thin wires with high efficiency or spherical nanoparticles, although the effect of the synthesis temperature is still a factor to address systematically [1,2,3]. Moreover, temperature stability studies of AuNWs are fundamental for future developments and applications. Here we present a small angle x-ray scattering (SAXS) in situ temperature dependent study of an already reported AuNWs synthesis [4]. We also studied the stability of the final nanostructures in solution in the range of 20 – 90ºC. SAXS measurements were complemented with transmission electron microscopy (TEM) images. OAm/Au molar ratio was kept at 40, within the nanowires high yield range, and the synthesis temperatures varied from 20 to 80ºC without stirring. As a result, it was found that the synthesis evolves toward the formation of nanowires when the temperature is kept below 40°C, and 10nm diameter nanoparticles started to be produced above this temperature. In addition, the AuNW were found to be stable up to 70ºC, when the correlation between AuNWs began to decrease and above 80°C the nanowires were disassembled to generate correlated nanoparticles.

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KEYWORDS: SAXS - TEM - ULTRATHIN - GOLD - NANOWIRES - TEMPERATURE - STABILITY

POSTER PRESENTATION

SEQUENCE SLIDER: A CRYSTALLOGRAPHIC METHOD OF SIDE-CHAIN EXPANSION FOR PHASING FROM FRAGMENTS AND SEQUENCE ASSIGNMENT OF NATURAL COMPOUNDS

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The protein crystallographic method SEQUENCE SLIDER generates multiple hypotheses of sequences using known information and validates one with available statistics. Applied to phasing from fragments, SLIDER was able to push the resolution and asymmetric unit content boundaries of the ab initio method ARCIMBOLDO to 2.7 Å and 682 residues, respectively [1]. By modelling most probable side chains into polyalanine fragments using a remote homologue alignment or secondary structure prediction, SLIDER scores sequence hypotheses using PHASER log-likelihood gain [2] and, if one is correct, the improvement in the number of atoms and their phases allow SHELXE [3] to reveal the rest of the structure and distinguish correct with a correlation coefficient calculation. As a side-chain evaluator, SLIDER is immediately applicable to elucidate sequence uncertainty in crystals obtained from samples purified from natural sources, such as snake venom, since multiple isoforms may be present. It uses phenix.polder real-space correlation coefficient [4] to score most probable side chains in each residue position and generate a theoretical sequence database that is used against bottom-up mass spectrometry data using PatternLab [5]. In the case of uncertainty, side-chain possibilities are narrowed down using phylogenetic conservation and residue frequency calculated with ConSurf [6]. By such integration of methods, SLIDER was able to determine the sequence of a crotoxin, a metalloproteinase, a homologue phospholipase A2 from snake venoms and a lysozyme from chicken. Its integration to cryo-electron microscopy maps to validate sequence is underway. Therefore, SLIDER is particularly useful to the scientific community that collects protein X-ray diffraction data in beamlines by offering an ab initio approach to solve the phase problem when no homology model or experimental phasing data is available and to assign the most probable sequence when this is not fully known.

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KEYWORDS: PHASING, SEQUENCE ASSIGNMENT, METHOD DEVELOPMENT, SEQUENCE VALIDATION

POSTER PRESENTATION

SPUTTERING OF N_2O ICE IRRADIATED BY GALACTIC COSMIC RAYS ANALOGS

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Nitrous oxide (N_2O) was already detected in the interstellar medium (ISM), in star forming regions [1,2]. In the Solar System, N_2O was not observed yet, however laboratory experiments on mixtures of ices of astrophysical interest show that it may be present in ices covering some minor bodies in the outer Solar System [3,4]. As all materials in space, ices are continuously exposed to ion bombardment by solar wind ions and cosmic rays. In particular, cosmic rays trigger secondary ion emission from these surfaces, contributing to the possibility to form an exosphere [5,6]. Laboratory studies demonstrated the effects of energetic processing of ices at low temperature, showing the production of new molecular species and free radicals of astrophysical interest. In the current work, N_2O ice at 10 K was irradiated by energetic (MeV/u) multicharged heavy ions (e.g., 105Rh and 140Ba); the sputtered species were detected and analyzed by the TOF-PDMS technique (Time-of-Flight Plasma Desorption Mass Spectrometry). Secondary positive and negative ions are identified like N^+, $N2^+$, NO^+, O^+, O^-. Also ejection of ion cluster series are observed: $(N_2)_nRm^+$, $(NO)_nRm^+$, $(N_2O)_nRm^+$, where $Rm^+ = N^+$, $N2^+$, NO^+, $N2O^+$, O^{m+} ($n \sim 10$, m=1-3). Their yield distributions follow a two decreasing exponentials, one fast -F and another slow -S, suggesting a two-regime formation. Most of the yield distributions have the same pair of exponential decay constants, around $kF = \sim 1.4\ u^{-1}$ and $kS = \sim 0.15\ u^{-1}$. This behavior supports an emission model for aggregates useful to understand the processes by which neutral and ionized molecular species are delivery to the gas phase in space. In addition, photon irradiation also induces desorption and photochemistry; thus, more experimental simulations with N2O under those astrophysical conditions may be useful to better understand its astrochemistry.

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Poster Presentation

$SrTiO_3$:Pr,Al PHOSPHOR MESOCRYSTALS: PHOTOLUMINESCENCE AND SHORT-RANGE STRUCTURE

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In this study, local and electronic structure of $Sr_{0.998}Pr_{0.002}Ti_{1-y}Al_yO_3$ ($SrTiO_3$:Pr,Al) mesocrystals prepared via hydrothermal route were correlated with photoluminescence spectra. Scanning electron microscopy (SEM) images confirm the microcube-shaped morphology and X-ray diffraction measurements revealed pristine structure with Pm3m space group for all $SrTiO_3$:Pr,Al samples without spurious phases. Extended X-ray absorption fine strucutre (EXAFS) results at Ti K-edge indicate the formation of O vacancies and an increase of the local disorder as a function of Al content. X-ray absoprtion near edge strucutre (XANES) at Ti K-, LII,III- and O K-edges, as well as our calculated projected density of states, show Ti off-center displacement, a small deviation from the cubic structure and the local symmetry breaking associated with the hydrothermal method and with dependence of Al incorporation. Photoluminescence spectra for $SrTiO_3$:Pr,Al samples show the expected emissions for the Pr^{3+} ions incorporated into $SrTiO_3$ lattice. The intensity of some emissions increases as a function of Al content up to 3 at. %. This increasing is associated with the disorder caused by Al, resulting in lower symmetry around Pr^{3+} sites, which can increase the probabilities of the transitions for Pr^{3+} ions due to the mixing of the opposite parity in 4f configurational levels. For higher Al concentrations, Pr emissions show a decrease, which is attributed to the quenching of the photoluminescence due to O vacancies. Furthermore, a broad emission is also observed, which is associated with the fact that the intrinsic defects, which are created as a result of hydrothermal route and Al incorporation, can originate intermediary electronic levels in the the band gap.

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Keywords: photoluminescence, SrTiO3, mesocrystal, hydrothermal synthesis, rare earth, Pr, praseodymium, XANES, EXAFS

POSTER PRESENTATION

STRUCTURAL BASIS FOR SUBSTARTE SPECIFICITY AMONG SNAKE VENOM PHOSPHOLIPASES B

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Snake venom phospholipases B (SVPLBs) are the least studied enzymes. They constitute about 1% of Bothrops crude venoms, however, in other snake venoms, it is present in less than 1%. These enzymes are considered the most potent hemolytic agent in the venom. Currently, no structural information is available about these enzymes from snake venom. To better understand its three-dimensional structure and mechanisms of envenomation, the current work describes the first model-based structure report of this enzyme from *Bothrops moojeni* venom named as *B. moojeni* phospholipase B (PLB_Bm). The structure model of PLB_Bm was generated using model building software like I-TESSER, MODELLER 9v19, and Swiss-Model. The build PLB_Bm model was validated using validation tools (PROCHECK, ERRAT, and Verif3D). The analysis of the PLB_Bm modeled structure indicates that it contains 491 amino acid residues that form a well-defined four-layer αββα sandwich core and has a typical fold of the N-terminal nucleophile aminohydrolase (Ntn-hydrolase). The overall structure of PLB_Bm contains 18 β-strands and 17 α-helices with many connecting loops. The structure divides into two chains (A and B) after maturation. The A chain is smaller and contains 207 amino acid residues, whereas the B chain is larger and contains 266 amino acid residues. The sequence and structural comparison among homologous snake venom, bacterial, and mammals PLBs indicate that differences in the length and sequence composition may confer variable substrate specificity to these enzymes. Moreover, the surface charge distribution, average volume, and depth of the active site cavity also vary in these enzymes. The present work will provide more information about the structure–function relationship and mechanism of action of these enzymes in snakebite envenomation.

KEYWORDS: SNAKE VENOM PLBS, STRUCTURAL CHARACTERIZATION, SUBSTRATE SPECIFICITY, GLYCOSYLATION

POSTER PRESENTATION

STRUCTURAL BEHAVIOR OF $BiCrO_3$ INVESTIGATED THROUGH RAMAN SPECTROSCOPY AND SYNCHROTRON X-RAY DIFFRACTION

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The science of materials is nowadays one of the main factors driving technological development and growth in modern society. The work carried out in this area of solid state physics has allowed the emergence of electronic, computational, communications and mainly storage and sending data. With the growth of interest in multiferroic materials in the last years, many techniques have been used in its base study with the purpose of providing more knowledge about the operation of its multferroic characteristics. In the present work, the X-ray diffraction synchrotron and Raman spectroscopy techniques was applied, in perovskite $BiCrO_3$ subjected to high pressures in order to analyze the possible structural phase transitions present in this compound due to the great importance of obtaining knowledge about its crystalline structure. Chromates with perovskite structure $ACrO_3$ have been investigated intensively in the last few years due to their interesting properties and applications. While rare-earth-based compounds exhibit intriguing magnetic properties and multiferroic behavior at low temperatures, Bi-based chromites are interesting because they are lead-free ferroelectric and multiferroic materials. Both the X-ray diphrogram and the Raman spectra obtained as a result in this study point to a phase coexistence in the interval between approximately 2.0 GPa and 2.6 GPa, after that interval, Raman spectroscopy indicates another phase transition. However, a more detailed analysis must be carried out in order to obtain more information around such transitions

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POSTER PRESENTATION

STRUCTURAL DETERMINATION OF CANDIDATES FOR PROTOTYPES OF NEW DRUGS *N*-ACYLHYDRAZONIC DERIVATIVES

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In the development of candidates for new drugs *N*-acylhydrazonic the crystal structure determination gives an access to a large range of information, including connectivity, conformation, accurate bond lengths and angles which provides information for a better understanding of the pharmacodynamic and physicochemical properties of them. In this work, we determined the crystal structure of the compounds LASSBio-1834 and LASSBio-1835 with X-ray powder diffraction data (DRXP). These compounds were planned and synthesized at the Laboratory of Evaluation and Synthesis of Bioactive Substances - LASSBio® of the Federal University of Rio de Janeiro (UFRJ). LASSBio-1834 and LASSBio-1835 are *N*-acylhydrazonic derivatives phosphodiesterase type 4 (PDE4) inhibitors and adenosine A2A receptor ligands, in vitro, designed as candidates for vasodilator prototypes [1]. *Topas Academic* v.5.0 and DASH softwares were applied to determine the crystal structure, later the Rietveld method was used to refine them [1,2]. LASSBio-1834 and LASSBio-1835 crystallized in monoclinic system with space group *P*21/*c* and the results of determination of the crystalline structure showed structural aspects of the solid phase of the compounds allowing the characterization of the relative configuration *E* of the double bond imine for all the compounds. We identified for the first time 1,5-N ··· S σ-hole intramolecular interactions in the candidates for prototypes of new drug developed on LASSBio®. The data used in this work was collected in a conventional diffractometer and took around 12 hours using a variable count time strategy. These data collection could be greatly benefited if we had used a synchrotron source like Paineira beamline (Sirius). It could deliver superior data quality by providing high-brilliance in terms of photon flux and angular resolution and also would greatly reduce the acquisition time.

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KEYWORDS: STRUCTURE DETERMINATION, N-ACYLHYDRAZONIC, PHOSPHODIESTERASE-4 INHIBITOR

POSTER PRESENTATION

STRUCTURAL EFFECTS OF MYOSIN VA GLOBULAR TAIL DOMAIN PHOSPHORYLATION

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Myosins constitute a protein superfamily of actin-based molecular motors involved in several important biological functions, such as muscle contraction and intracellular transport. Within this group, the class V myosin (MyoV) represents a well-conserved class known for interacting with cargoes through its C-terminal globular tail domain (GTD) promoting the intracellular transport of vesicles, organelles, proteins, mRNA and modulating cell signaling pathways [1]. The MyoV activity regulation is a key for the modulation of many cellular processes. It has been shown that the phosphorylation of the Ser1652 residue, located in a flexible loop (phospho-loop) of human GTD-MyoVa, plays a crucial role on cell cycle control and is possibly involved in the regulation of MyoV's inhibited state. Thus, the determination of the phosphorylated GTD-MyoV structure would represent a milestone in MyoV functional and molecular studies [2,3]. Nevertheless, there are some barriers to overcome, mainly regarding the GTD-MyoVa crystallization with the phospho-loop intact. The production of large amounts of a homogenous phosphorylated protein sample could be very challenge, therefore to mimic phosphorylation effects we can replace the phosphorylation target residue by an acid residue. The goal of this project is shed light on MyoV regulatory mechanisms through structural analysis at high-resolution using X-ray crystallography. We have performed expression tests for all constructions (wild-type, S1652E and S1652A) and established the best condition for protein production. Next steps include the purification and crystallization of all constructs and then the further structure solution at Sirius MANACA beamline using ab initio phasing with ARCIMBOLDO [4]. We also intent to collect data at room temperature to verify the conformation/flexibility of phospho-loop's flexibility reliably, as well as, other regions potentially involved in MyoV regulation.

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KEYWORDS: MYOSIN V, PHOSPHORYLATION, FLEXIBILITY

POSTER PRESENTATION

STUDY OF REDUCIBILITY OF Cu AND Al CATALYSTS UNDER DIFFERENT TREATMENTS

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Copper and aluminum catalysts are widely used in studies in the field of environmental catalysis [1, 2] and have been shown to have high catalytic activity when prepared from hydrotalcite precursors [3]. The preparation methods of these catalysts directly influence their catalytic performance, due to changes that may occur in their physicochemical properties. It is generally not considered that the material can undergo significant changes, such as thermal reduction, during the pretreatment stage under inert atmosphere and moderate temperatures. However, some studies in the literature comment on the reducibility of copper even in inert environments, with this self-reduction in zeolites being more common [4, 5, 6]. In this study, we used four preparation methods: the solid-state reaction route (CuAl-s), the physical mixing of the oxides (CuAl-o), individual oxide precipitation followed by physical mixing (CuAl-p) and co-precipitation of precursor followed by calcination (CuAl-HT-c). The precursor was obtained with the hydrotalcite phase of high purity and high surface area, observed through chemical analysis, XRD and textural analysis of the CuAlHT-c precursor. This method also provided the achievement of a structurally and texturally more stable mixed oxide, as observed through the FWHM evolution curve with temperature. The formation of Cu_2O and metallic Cu was visualized through XANES and XRD in situ, under an inert atmosphere of He from 440°C. These measurements were carried-out in the LNLS (Campinas, Brazil). Comparing the pretreatment reducing gas, the reduction with CO occurred more slowly than with H_2, and with greater Cu^+ formation. Thus, the method of preparing Cu and Al catalysts with the formation of the precursor with hydrotalcite structure showed more promising textural, structural and reducibility properties compared to the other preparation methods investigated in this study.

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KEYWORDS: COPPER, CATALYSTS, PREPARATION METHODS AND REDUCIBILITY

POSTER PRESENTATION

STUDY OF THE INTERACTION BETWEEN SHORT PEPTIDES AND DPPC MEMBRANE BY SAXS AND AFM

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It is known that the incorporation of proteins and peptides into lipid bilayers affect their mechanical properties as well as their three-dimensional organization [1,2]. Also, peptides at low concentrations are adsorbed at the interface with little disturbing effects on membrane elasticity [3]. However, the sequence [RF]n (R=arginine; F=fenilanalanine; n=repeat number, from 1 to 5) does not seem to follow this rule, since the increasing of n leads, even at low peptide concentration, to an increase of the system structure crystallinity obtained by XRD and the toxicity probably consequence of modifications in the membrane [4]. In this work we propose to better understand the interaction between RF peptide and DPPC membranes in the interface via Langmuir (L) and Langmuir-Blodgett (LB) films, both methodologies widely applied in the studies of lipids biological systems models [5]. The lipid membrane was composed by phosphatidylcholine (DPPC), which comprises mostly the cell membrane, mixed with RF at different peptide/lipid molar ratios (P/L). Results from small angle X-ray scattering (SAXS) experiments showed changes in the structural and thermodynamics parameters of the membrane with the increase in peptide concentration. Using atomic force microscopy (AFM), we showed the appearance of pores through the bilayer membranes and peptide aggregation at different interfaces, suggesting that the hydrophobic residues might have an effect on both pore size and layer structure, facilitating the membrane disruption and leading to different cytotoxicity effects.

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KEYWORDS: LIPID MONOLAYERS, LANGMUIR-BLODGETT FILMS, PEPTIDE, AFM, SAXS, AMYLOID

POSTER PRESENTATION

STUDY OF THE STRUCTURAL DISORDER EFFECT ON THE SPIN LIQUID STATE OF $Sr_2Cu(W_{0,5}Te_{0,5})O_6$ UNDER DOPPING WITH Fe AND Sb ATOMS

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The properties of frustrated and low-dimensional magnetic systems are of great interest in the current physics of condensed matter. These materials can provide new and exotic states of matter, particularly the so-called “quantum spin liquids” (QSL). These materials can also help improve our understanding of unconventional superconductivity and may, in the future, be applicable to quantum information technology and computing. Experimental examples of QSL materials have been found only very recently and are still rare. Furthermore, until now, strong experimental evidence of QSL states has only been found in materials with a triangular arrangement of atoms and in them small doping introduces structural disorder and takes the system out of the QSL state, this effect is called "Order by disorder". The study published recently [1] shows that the double perovskite $Sr_2Cu(W_{0,5}Te_{0,5})O_6$ presents the QSL state. This compound is differentiated from other systems characterized as QSL both because it is formed by square networks and because it presents a high structural disorder introduced by the random occupation of non-magnetic ions W and Te in the same crystallographic site. The objective of our study is to have a better knowledge of the effect of structural disorder in the QSL state already found in double perovskites $Sr_2Cu(W_{0,5}Te_{0,5})O_6$, since usually disorder removes the QSL state. We introduce more disorder on the system by dopping with Fe and Sb in different percentage from 0.5% to 5%. Mössbauer spectroscopy and magnetic measurements indicates that QSL state is preserved. Specific heat measurements have a peak near room temperature that indicate a structural or electronic transition. We also observed that Fe atoms have mainly Fe^{3+} oxidation state, however a small fraction of Fe^{2+} state seems to increase at lower temperatures. We plan to further extent this study with low temperature x-ray experiments, along with a study of the oxidation state of Cu atoms with XAFs.

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FAPERJ

KEYWORDS: MAGNETIC FRUSTRATION, QUANTUM SPIN LIQUIDS, STRUCTURAL DISORDER

POSTER PRESENTATION

STXM PHASE-CONTRAST TOMOGRAPHY

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Phase-contrast is a powerful tool for x-ray imaging to examine a variety of samples that typically has a low absorption contrast. Within this context, we implemented a phase-contrast imaging data analysis in the case of a STXM (Scanning Transmission X-Ray Microscopy) measurement. The acquired data consists of a scattering pattern detected by an area detector positioned after the sample. From those patterns, the sample's phase information can be retrieved by comparing the scattering pattern with and without the sample [1]. In this project, we performed a series of simulations and reconstructions to study the necessary setup and coding for future implementation at the CARNAUBA beam-line [2]. Also, the simulation included multiple acquisitions for several angles of rotation, simulating a tomography acquisition, and allowing the reconstruction of a 3-D map; in other words, a phase-contrast tomography [3].

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POSTER PRESENTATION

SUPERELASTIC NITI SHAPE MEMORY ALLOY IN-SITU THERMOMECHANICAL SIMULATION BY GLEEBLE® SYNCHROTRON SYSTEM

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NiTi alloys have attractive functional properties as shape memory effect and the superelasticity appropriated to be used in different segments in engineering and biomedical areas, in addition to exhibit excellent corrosion resistance and biocompatibility. These functional properties are obtained through the thermomechanical process. Thermomechanical processes aim to obtain the appropriate shape of the material, to control phase transformation temperatures, to improve the alloy characteristics, and improve shape memory effect or superelasticity. To obtain the desired microstructure the hot work should be performed to assure the mechanical and functional properties adequate. In this study the physical simulation as to the evaluation of the structural modifications in the remelted and annealed alloy was performed at Gleeble® at two different deformation rates (of the order of 10^{-1} and 10^{-2} s^{-1}) at 850 °C. The X-ray scattering and Thermo-Mechanical simulation (XTMS) experiment were carried out the XRD1 beamline, LNLS - CNPEM, Campinas - Brazil. The uniaxial compression tests were carried out on an advanced thermomechanical simulator, the Gleeble® Synchrotron system. The optical and electron microscopy were carried out the IME, Rio de Janeiro – Brazil.

KEYWORDS: NI-RICH NITI SHAPE MEMORY ALLOY, SYNCHROTRON RADIATION X-RAY DIFFRACTION (SR-XRD), GLEEBLE®

POSTER PRESENTATION

SURFACE PROPENSITY OF SMALL ORGANIC BIOMOLECULES IN VAPOR-WATER INTERFACE BY XPS

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Biochemical reaction in living systems takes place in aqueous solutions containing small organic molecules, known as osmolytes. These molecules affect the stability of different cellular structures [1]. For example, denaturant osmolytes (e.g., urea and salts) favor the unfolded state of proteins whereas stabilizing osmolytes (e.g., TMAO and betaine) favor the folded state [2, 3]. Effects of the latter can be compared with the ions on the left side of the Hofmeister series, which help stabilize the native, folded structures of proteins. Regulation of osmolytes by living organisms allows them to survive extreme conditions that, otherwise, would account for unstable cellular structures. Accordingly, TMAO levels in muscles of deep-sea organisms were found to increase with the depth in which they are capture, possibly to counteract the destabilizing effect of pressure on molecular structures. Despite intensive studies, the atomic mechanisms of osmolytes are not well understood. In addition of providing basic knowledge of how life unfolds, unraveling these mechanisms will enable rational designs of new and more efficient osmolytes to control molecular structures. The aim of this study is to provide estimates of the relative concentration as well as orientation of different osmolytes and mixture of osmolytes at the vapour-water interface using X Ray Photoelectron Spectroscopy (XPS) technique in aqueous solutions. This will allow us to compare mechanisms of action of different protecting osmolytes; TMAO, betaine and glycine as well as denaturant osmolyte; urea. Based on recent measurements of the vapour-water surface tension, the stabilizing mechanism of TMAO may be different from the one of betaine and glycine. Moreover, our study will be useful to test computational models of osmolytes.

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KEYWORDS: DENATURANT OSMOLYTE, STABILISING OSMOLYTE XPS, SURFACE PROPENSITY, TMAO, UREA

POSTER PRESENTATION

SURVIVAL OF BAKER'S YEASTS UNDER IONIZING RADIATION

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The aim of this study is to comprehend the effects of ionizing radiation on the resistance mechanisms of microorganisms that survive and even thrive in extreme environments on Earth and space, subjected to above-the-background radiation levels. Some examples are disaster nuclear plants, radioactive waste and even the International Space Station. Radiation exposure affects cellular components including nucleic acids, proteins and lipids. Understanding the molecular processes of these interactions could lead to a better understanding of the adaptive evolution and response of eukaryotes under ionizing radiation. In addition, these studies are necessary to delineate the optimal experimental conditions for *in vivo* studies at the beamlines of Sirius. *Saccharomyces cerevisiae* is a well-know model organism to study radiobiology and it was exposed to gamma radiation on Gammacell irradiator at IPEN-CNEN/SP and X-ray radiation at the Mogno beamline of Sirius - CNPEM. The radiotolerant bacterium *Deinococcus radiodurans* was also tested, as a benchmark. Additionally, one indigenous yeast isolated from *Salicornia ambigua* plant and two different strains of the common baker's yeast Saccharomyces cerevisiae were also tested due to the abundance of molecular data on this molecular biology model organism, its biotechnological potential, and also because of promising preliminary results that we already obtained. Our results showed that most yeasts' strains have survived under ionizing radiation up to 5 kGy, not far from the model organism *Deinococcus radiodurans* has survived for more than 10 kGy as expected. This reinforces the importance of yeasts as model organisms for studies of extreme environments, including for space colonization and particle accelerator applications, such as in synchrotron science.

POSTER PRESENTATION

SYNCHROTRON BASED INVESTIGATION OF CELL/DENDRITE STRUCTURES IN SOLAR GRADE SILICON

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The growing demand for silicon in photovoltaic applications triggered the development of alternative more economic routes to produce solar grade silicon [1]. The metallurgical route is a sequence of processes including unidirectional solidification, which can be used to remove most of metallic impurities as these segregate to the top of the silicon ingots. The pile-up of impurities in front of solidification interface can lead to the formation of cells and dendrites at the solid-liquid interface, which hinders the impurity segregation and ingot purification [2]. Therefore, understanding the morphology of the solid-liquid interface is essential to develop more efficient purification processes and better grain control of multicrystalline silicon.The solubility of most metallic impurities is very small in solid silicon, making the variations in their impurity concentrations too small to be detected by chemical etching or by backscattered electron contrast observed in the electron microscope. Consequently, dendrite structures that existed during solidification require more sensitive techniques to be detected. X-ray fluorescence microscopy (μ-XRF) has been frequently used to detect iron and others metallic impurities in a silicon matrix [3]-[4] to investigate their effects on the electrical performance of solar cells. In the present work, the distribution and morphology of impurities were measured by X-ray microtomography using a pixel size of 22 μm. Some interesting cell/dendrite structures could be observed as well as the intermetallic morphology change as the impurity concentration pilled up in front of the solidification interface. Future experiments using Sirius beamlines are of paramount importance, since they would give more details about the morphology of the intermetallic precipitates due to its smaller beam size and also estimate the concentration and distribution of the impurities into the silicon matrix.

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KEYWORDS: SILICON, REFINING, PURIFICATION

POSTER PRESENTATION

SYNCHROTRON LIGHT ILLUMINATING TEACHER TRAINING

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This work presents the first two versions of an in service teacher training course for high school physics teachers, in which they visit all spaces in the largest complex of Brazilian scientific laboratories and the newest Brazilian particle accelerator, Sirius, in addition to having theoretical classes. This course was carried out through a partnership between the National Center for Research in Energy and Materials with the Brazilian Physics Society, lasted for a week, in the months of January 2019 and 2020, and was called Synchrotron / Sirius School for Teachers from high-school. During the School, the teachers visited the facilities of the National Research Center for Energy and Materials and its four major laboratories: National Synchrotron Light Laboratory, the National Biosciences Laboratory, the National Bio-renewable Laboratory and the National Nanotechnology Laboratory. Over a week, the courses were taught by researchers from CNPEM, whose objective was to show the concepts involved and the applications of the various laboratories, with the goal of taking the ideas of the application of modern physics to the classroom. The teachers who participated in the courses, after being immersed in the whole research atmosphere of cutting-edge science, leave with the mission of disseminating the advances of Brazilian science and technology to students.

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POSTER PRESENTATION

SYNCHROTRON MICRO-CT APPLIED TO MORPHOMETRIC ANALYSIS OF THOROPA MILIARIS TADPOLES

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Amphibians are used extensively in physiological studies aimed at generating new insights in evolutionary biology, especially in the investigation of the evolution of air-breathing and terrestriality [1]. The Brazilian species Thoropa miliaris present morphological conditions that differentiate it from other species because live in the film of water on rock surfaces at the wet borders of waterfalls [2]. Detailed knowledge of the interior of biological structures and organisms is crucial for a better understanding of their development. For this, phase-contrast microtomography was the best non-invasive technique available and this herpetologic study and this application is quite recent [3,4]. The first work of our group was to show the virtual dissection of this species [5], where we present the first results of the high resolution non-invasive morphometric analysis of the soft tissues of Thoropa miliaris tadpoles. The microtomographic images were obtained in the IMX beamline of the LNLS. In a second step, new tomographic acquisitions were performed using the synchrotron Elettra (Italy), in the SYRMEP beamline, allowing the volumetric quantification of internal structures with greater morphological changes along the tadpole development. Both acquisitions were by phase-contrast regime and without staining. We intend to carry out the analysis of this amphibian using the Mogno beamline of the Sirius synchrotron (LNLS), with the justification of tomographic acquisitions on a nanoscale, mainly of the animal's bone region, where there is the particularity of transforming an endolymphatic calcium deposit (acellular) in bone tissue of an adult frog. In this work, we show the entire trajectory accomplished using the microtomography technique in the tadpole of the Thoropa miliaris species, from the beginning on the IMX line to future perspectives on the Mogno beamline.

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POSTER PRESENTATION

SYNCHROTRON RADIATION MICROTOMOGRAPHY AND STAINING METHODS FOR BIOLOGICAL APPLICATIONS

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Microtomography using synchrotron sources has become a useful tool in biological imaging research since synchrotron radiation properties enable the reconstruction of highly resolved 3D images. Several applications using microtomography and staining methods have been made by our group (some are still in progress) at IMX beamline-LNLS and SYRMEP beamline-ELETTRA, in order to improve the visualization of less dense tissues, among which we can highlight applications in insects, tadpoles, ovaries. In this work we will present some results involving synchrotron radiation microtomography and staining methods applied to biological samples. Among the most recent paper, we can highlight the use of synchrotron radiation microtomography to study the ovaries using an osmium staining [1], showing mainly the follicles and the vessels, at SYRMEP beamline. Another recent work deals with the insect *Rhodnius prolixus*, the most important vector of Chagas' disease [2-3]. In the first work [2] we investigated the molting period of the *Rhodnius prolixus* cuticle, using iodine staining, at SYRMEP beamline. The other work [3] tested different staining methods in order to find the best protocol to study the nervous system of *Rhodnius prolixus*, at IMX beamline. Another project is underway using synchrotron radiation microtomography: study of the internal anatomy of tadpoles, describing external and internal morphological features like cartilage, muscles, sacs containing calcium, crystalline and skin at a range of developmental stages, using iodine staining; this project was performed at IMX and SYRMEP beamlines. With the new 4th generation light source (SIRIUS) we expect a wide range of new possibilities, allowing for much more sensitive analyses, grounding the collaborations between physics, biology, environmental and materials sciences.

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KEYWORDS: SYNCHROTRON MICROTOMOGRAPHY, BIOLOGICAL APPLICATIONS, STAINING METHODS

POSTER PRESENTATION

SYNTHESIS AND CHARACTERIZATION OF PHOTOCATALYTIC Ce AND Ti DOPED NIOBIUM OXIDE SEMICONDUCTORS FOR DEGRADATION OF POLLUTANTS

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The damage caused by organic pollutants has increased the search for high efficiency and low-cost processes, that can treat large volumes of effluents. Advanced oxidative processes stand out as an excellent alternative, being extremely useful in the case of substances resistant to conventional technologies [1]. The main characteristic of the processes is to transform organic pollutants into compounds that are not harmful to the environment through the use of semiconductors as TiO2 under ultraviolet radiation. In this work, oxidation processes involving semiconductors and UV radiation will be discussed. Heterogeneous photocatalysis and the behavior of several semiconductors are of great interest, therefore study solar energy-absorbing materials is of great interest for technological development and maintenance of life [2]. Niobia has been studied as a semiconductor for photocatalysis due to its high thermal stability and because it presents a bandgap in the UV region. When doped with other elements, changes in its optical, structural and morphological properties are observed. This addition of dopants to the structure can cause an increase in photocatalytic activity, causing the material to absorb energies close to the visible, enabling solar absorption [3]. Herein, the dopants used were titanium, as it is known in the literature as an excellent photocatalyst, and cerium that has a high oxidation and reduction capacity [4]. The materials were synthesized by the hydrothermal method, for 20 h at 150 °C, and doped proportion were 0.5%, 0.8% and 1% mol. The final solids were evaluated concerning their structural (FTIR, Raman, XRD, TGA), morphological (SEM), optical (DRS in UV-Vis) properties. Doped niobia compounds are ready to be submitted to the photocatalytic study for pollutants degradation under solar radiation. The SIRIUS synchrotron facility will be essential to high-quality characterization of the compounds by means of EXAFS, XANES techniques to study the local structure of Ti species accommodated in Nb_2O_5 and XRD technique to characterize the structural changes caused by de dopant and the purity of the samples [5].

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KEYWORDS: NIOBIUM, CERIUM, TITANIUM AND PHOTOCATALYSIS

POSTER PRESENTATION

TAPHONOMY OF ICHNOFOSSILS IN EOPALEOZOIC SANDSTONES, PACUJÁ, CE, BRAZIL

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In the medium to coarse sandstones near the city of Pacujá, Ceará State, an ichnofossiliferous assembly occurs with the presence of ichnogenera, such as Circulichnis, Palaeophycus, Planolites, Furnasichnus and Arenicolites. According to the literature, the age of the records is controversial to the Pacujá Formation (Cambrian) of Jaibaras Basin, and the Ipu Formation (Silurian) of Parnaíba Basin. The petrographic description and analysis in Scanning Electron Microscopy with Energy Dispersive Spectroscopy (SEM/EDS) show cementation is composed of iron oxide/hydroxide and clay intensifies towards the top and under the ichnofossils, as well as the presence of zircon, rutile, and muscovite that occur in the middle of cement. To supplement the taphonomic studies, we used the micro X-ray fluorescence analysis, performed at the Brazilian Synchrotron Light Laboratory (LNLS) at Brazilian Center for Research in Energy and Materials (CNPEM). Elemental maps of Si, Fe, S, V, Ca, Ti, Mn and K were elaborated in two samples. We observed the increase of Fe and Mn at the top, also occurring associated S. These ichnofossils may have been preserved by occurring in microbial mats corresponding to the death masks model. The metabolic activity of microbial communities produces extracellular polymeric substance (EPS), responsible for trapping and agglutinating sediments resulting in the stabilization of the substrate. After burial and with the decomposition of organic matter, a reducing environment was generated, which caused the reduction of Fe (III) and S, allowing the deposition of pyrite (FeS).The places observed with Fe located with S in the images are the result of a posterior oxidation process, resulting in cementation observed in the rock. Formation of clay minerals is associated with the decomposition processof the mat, just as it is common for agglutination of heavy minerals and muscovite in microbial mats that support the fossildiagnetic model.

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KEYWORDS: **ICHNOFOSSILS**, **FOSSILDIAGENESIS**, **MICROBIAL MATS**

POSTER PRESENTATION

TEACHING AN OLD DOG NEW TRICKS: NEW STRATEGIES FOR SAXS DATA ANALYSIS OF PROTEINS IN SOLUTION

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Small Angle X-Ray Scattering (SAXS) is a popular and well established experimental technique with an extensive range of applications. Such fact arises from its flexible sample preparation, allowing researchers to study proteins in the most diverse environments and experimental conditions. SAXS is of special interest to protein studies, and hybrid data analysis methods with inputs from other techniques, such as NMR spectroscopy and X-Ray crystallography, yield complimentary results, allowing better understanding of protein structure and dynamics in solution. In recent years, new SAXS data analysis methods for proteins are emerging with new ways to address model validation, structural parameters calculation and integration with databases. Some of these tools do not require an user input, allowing one to obtain immediately relevant information on the protein conformation in a way that it could be implemented for automated data analysis pipelines in beamlines. The aim of this study is to highlight recent advances in this field and illustrate its applicability and performance to data obtained in the SAXS1 beamline at LNLS. To do so, we measured chaperone proteins related to the Hsp70 and Hsp90 families, which are of special biological relevance due to its role in protein misfolding and aggregation issue. We have shown that the use of these new tools for SAXS data analysis can, undoubtedly, improve our understanding on molecular chaperone structure and dynamics.

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KEYWORDS: SAXS, PROTEINS, MOLECULAR CHAPERONES

POSTER PRESENTATION

THE EFFECT OF UNDERLYING METALLIC SUBSURFACE ON THE ELECTRON TRANSFER PROPERTIES OF MONOLAYER GRAPHENE

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The use of monolayer graphene in the designing of the silicon microchips supported graphene-based devices is limited by the sp2 carbon basal surface and strongly delocalized π electrons that present low chemical reactivity and hence sluggish electron transfer kinetics [1]. Here, we propose to use a system based on the development of a surface, comprising the monolayer graphene supported on lithographically designed gold subsurface on a silicon wafer, Gr/Au/SiO_2/Si. Controlled surface modification was performed using a covalent functionalization procedure, to evaluate the impact of the underlying Au subsurface on the graphene properties. Micro-Raman spectroscopy confirmed the monolayer graphene on both SiO_2/Si and Au surface as well as their covalent functionalization. We reported a 10 times higher value of the surface concentration of the organic functional groups attached to the Gr/Au/SiO_2/Si electrode and three times higher heterogeneous electron transfer (HET) rate constant value for this electrode as compared to Gr/SiO_2/Si electrode. For the determination of the impact of covalent functionalization on the electronic properties of the graphene and distribution of charges along the functionalized surface, scattering scanning near-field optical microscopy (s-SNOM) technique was employed. The results showed that the polaritonic coupling between the pristine graphene and SiO_2/Si substrate is lost when graphene is covalently functionalized. This effect is more pronounced at the border regions between the Au and SiO_2/Si or between the SiO_2/Si and graphene, revealing that these regions are more susceptible to functionalization due to the presence of dangling bonds [2]. The combining theoretical investigations showed that effective charge transfer occurs from the underlying Au due to the shift in the Fermi energy level of the graphene, which was further manifested by detecting ds-DNA adsorption and hybridization on the surface of the electrode with enhanced sensitivity.

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POSTER PRESENTATION

THE IMPORTANCE OF CHARACTERIZING THE SUBMICROMETRICAL REGION OF THE LAYER FORMED ON NITRIDED AUSTENITIC STAINLESS STEEL

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The application of plasma nitriding on austenitic stainless steels can produce a nitrided layer of some micrometers, improving hardness, wear and corrosion resistances, optimizing their use as biomaterials or for industrial applications. Depending on the parameters used in the process, the nitrided layer is formed by the nitrogen diffusion layer called expanded austenite γ_N, which spreads into inner region. On nitrided layer surface, if besides γ_N, some iron and chromium nitrides are formed, this region is known as composite layer. There is no consensus about the composite layer formation, mainly because of the characterization limitation, which may mask the presence of such nitrides. The study about some samples, nitrided at 673 K, for 4 h, in 80% H2- 20% N_2 gas mixture, at different pressures: 4; 6 and 10 Torr, can illustrate this important question. A recent paper [1] correlated some magnetic properties of their nitrided layer. The results pointed to the formation of a submicrometer layer occurring on the outermost surface of them, which indicated the occurrence of nitrides. In order to do a more accurate characterization, the surface of the non-cross-sectional samples was analyzed by x-ray diffraction (XRD) using a synchrotron source (XRD2-LNLS) with a 7,5 keV beam energy in a grazing-incidence geometry, for different superficial depths, on the top of the layer.The results can identify reliably the presence of the non-stoichiometric Fe_{2-3} N for all the samples and Fe_4N for 10 Torr sample and show that these nitrides, decrease rapidly with depth. This contributes to better elucidate this important question in the nitriding process, showing that a carefully characterization, particularly from the top of the nitrided layer, as shallow as possible, is mandatory to understand this system and the use of a synchrotron source, for performing the XRD is essential.

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KEYWORDS: PLASMA NITRIDING, AUSTENITIC STAINLESS STEEL, XRD

POSTER PRESENTATION

THE NEW He-3 CRYOSTAT OF EMA BEAMLINE

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The study of materials at low temperatures (T<5K) is very important for the discovery of new phenomena. Although the study of macroscopic properties of materials, such as resistivity and magnetization, in the mK range is usual in many laboratories around the world, the possibility of reach the low temperature regime (T<2K) at Synchrotron facilities remains extremely challenge, mainly due the high heat power of the x-ray beam. On top of that, here we present the design of a new adsorption cryostat to operate under unique conditions at EMA beamline of Sirius. The instrument was designed to be able to suffer the incidence of up to 1mW of x-ray power having as main objective reach temperatures down to 1K for X-ray absorption, diffraction and scattering experiments. This cryostat will use He-3 for the cooling process. The He-3 reservoir will be directly connect to an activated coconut charcoal pump in order to control the He-3 pressure. The design of the cryostat was carried out using the Inventor software, with its dimensions and the heat exchange being previously simulated using appropriated softwares. The commissioning should be carried out in the first semester of 2021 and we believe that successful of this new instrumentation might open several new opportunities for study of materials using Synchrotron techniques at low temperatures for the Brazilian community.

KEYWORDS: HELIUM 3 CRYOSTAT, ADSORPTION CRYOSTAT, SYNCHROTRON SOURCES, SIRIUS

POSTER PRESENTATION

THE OLIGOMERIC STATE YBBN/CNOX PROTEINS IS LINKED TO ITS BLEACH-INDUCED HOLDASE ACTIVITY

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YbbN/CnoX is a monomeric protein that present a thioredoxin (Trx) domain linked to a tetratricopeptide (TPR) domain [1]. EcYbbN contains a degenerated CXXC motif (SQHC) along its Trx domain and did not display a disulfide reductase activity. However, its redox propriety relies in a Cys residue (Cys63) apart from SQHF motif that can form mixed disulfides with its clients, preventing irreversible oxidation [2]. TPR domain are involved in protein-protein interactions and protein folding processes. For EcYbbN, HOCl treatment induces a potent holdase activity through post translational modifications (chlorination) at TPR domain. We previously compared EcYbbN with XfYbbN (*Xylella fastidiosa*) and PaYbbN (*Pseudomonas aeruginosa*) as representatives of three conserved YbbN subfamilies [3]. In contrast to EcYbbN, both XfYbbN and PaYbbN presented a thioredoxin-like activity sustained by a conserved CXXC motif. Furthermore, XfYbbN, but not PaYbbN, also displayed a bleach-induced holdase activity. However, the molecular mechanisms underlying such holdase activity is not understood. Therefore, we aimed to investigate the structural changes of these YbbNs in response to HOCl. As chlorination increased the general hydrophobicity of EcYbbN, we compared the nile red fluorescence intensity of HOCl-treated YbbNs with untreated ones. We observed that HOCl-treated XfYbbN presented higher nile red fluorescence but the same behavior was not observed for PaYbbN. The investigation of modified residues by HOCl at TPR domain of XfYbbN and PaYbbN is under investigation. We also addressed if the oligomeric state of EcYbbN, XfYbbN and PaYbbN change upon HOCl treatment. Using both dynamic light scattering (DLS) and size-exclusion chromatography, we observed that while EcYbbN and XfYbbN presented changes in its oligomeric state upon oxidation PaYbbN remained unchanged. Therefore, our results suggest that oligomerization induced by HOCl treatment may be linked to the holdase function of YbbNs.

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KEYWORDS: THIOREDOXIN, TETRATRICOPEPTIDE-REPEAT DOMAIN, OXIDOREDUCTASE ACTIVITY, CHAPERONE/HOLDASE ACTIVITY

POSTER PRESENTATION

THERMAL AND STRUCTURAL MODIFICATION IN TRANSPARENT AND MAGNETIC GERMANOBORATE GLASSES INDUCED BY Gd_2O_3

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A series of new transparent and magnetic germanoborate glasses in the system (100-x)[$60GeO_2$–$25B_2O_3$–$10Na_2O$–$4Al_2O_3$–1PbO] – (x) Gd_2O_3, with x = 0, 1, 2, 5, 10, 15 and 20 mol%, was prepared and studied with respect to their thermal and structural changes in the presence of Gd_2O_3. Based on Differential Scanning Calorimetre (DSC) analysis, a glass with 5% of Gd_2O_3 showed a high thermal stability, which progressively decreases for samples with higher content of Gd_2O_3. By the analysis of Raman and Fourier Transform Infrared (FTIR) spectra, it was possible to identify that by increasing the amount of Gd2O3, a progressive depolymerization of 6-membered Ge[IV] rings is promoted, concomitant with an increase of Ge[IV] tetrahedra units with non-briding oxygens. The structural analysis through the local-sensitive techniques EXAFS (Extended X-ray Absorption Fine Structure) and XANES (X-ray Absorption Near Edge Structure) showed that the short-range structural modification around the elements Ge and Gd^{3+} does not change with the addition of Gd_2O_3 and the presence of germanium four-fold coordination [GeIV] and Gd^{3+} states, respectively. A simulation of the coordination number (N), the interatomic distance (R) of Ge–O and Gd–O bonds and the Debye-Waller factor was also carried out.

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KEYWORDS: MAGNETO-OPTICAL MATERIALS, GERMANOBORATE GLASSES, VIBRATIONAL SPECTROSCOPY, X-RAY ABSORPTION SPECTROSCOPY, MAGNETIC SUSCEPTIBILITY

POSTER PRESENTATION

THERMAL STABILITY OF $PdCu_3$ NANOPARTICLES AS CATHODIC ELECTRODES FOR HYDROGEN PRODUCTION

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The modern world is consuming more and more energy, and it is necessary to diversify the energy matrix, prioritizing a clean and renewable energy sources. Hydrogen fuel cells are extremely promising, but they demand hydrogen gas (H_2) with high purity in large scale. H_2 production through water electrolysis is highly efficient, but it is necessary to develop efficient and inexpensive electrocatalysts for the hydrogen evolution reaction (HER). We applied bimetallic $PdCu_3$ nanoparticles supported on Vulcan carbon as electrocatalysts in HER, seeking to understand how a thermal treatment in the air (2 h, 120 °C) affects their electrocatalytic activity and their structural and electronic properties. Electrodes were produced with the mentioned electrocatalyst, which were thermal treated and applied as cathodes in the water electrolysis in 0.5 mol L^{-1} H_2SO_4 medium. EDX and XPS analysis were performed on the electrodes before and after thermal treatment and electrochemical measurements, in order to determine changes in their structural and electronic properties.The thermal treatment favoured the oxidation of Pd and Cu. The thermally treated electrode showed a higher exchange current density for HER than the no treated electrodes, suggesting an improved electrocatalytic activity. After the electrochemical experiments, the EDX measurements indicated the loss of Cu from the electrodes, which was corroborated by the XPS analysis that showed the decrease of CuO in the nanoparticles. In this presentation we will discuss the possible mechanisms involved in the enhanced catalytic activity on the HER. We foresee the use of in situ XAS/XANES during HER on these electrodes to better understand what structural and electronic changes taking place during the reaction, and how it influences electrocatalysis.

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KEYWORDS: HYDDROGEN EVOLUTION REACTION, PDCU, ELECTROCATALYSTS, WATER ELECTROLYSIS

POSTER PRESENTATION

THREE DIFFERENT MATHEMATICAL MODELS TO DETERMINE MAGNETIC COLLOIDS STRUCTURING

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A correct determination of magnetic nanoparticle (MNP) properties is important for determining their best field of application [1]. Interactions among particles could produce MNP structuring in the supporting matrix. In this way MNP properties, influenced by structuring, are modified, and it must be taken into account especially if the MNP will be used for biomedical applications [2-4]. The presence of MNP structures in liquid and solid matrices can be estimated using scattering techniques, where the scattering pattern can be modelled using a form factor that gives information about particle size and morphology, and a structure factor, that gives information about how particles are spatially assembled [5]. In this work, Small Angle X-ray Scattering (SAXS) patterns, obtained from two different aqueous colloidal suspensions of magnetite nanoparticles electrostatically stabilized with citric acid, were fitted using three different mathematical models in order to describe the particle size distribution and aggregation state. The colloidal suspensions differ in the mean particle size (4.5±1.0 nm and 5.5±1.1 nm) and in stabilization capability in aqueous milieu (well-stabilized sample and poorly-stabilized sample), allowing control of the interaction strength between particles. The models used for SAXS analysis, reveal that the particles are almost spherical with a broad size distribution, and that particles in each suspension are aggregated and subject to attractive interaction potential, typical for magnetic nanoparticles. For the well-stabilized sample, ramified chain-like aggregates were found, and for the poorly-stabilized sample, a more compact structure was determined. The size distribution obtained by applying SAXS mathematical models are in agreement with the size distribution determined using Transmission Electronic Microscopy (TEM).

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KEYWORDS: SAXS, NANOPARTICLES, STRUCTURING

POSTER PRESENTATION

THREE DIFFERENT ROUTES TO SYNTHESIZE $La_{0.7}Sr_{0.3}MnO_3$ NANOPARTICLES WITH POTENTIAL BIOMEDICAL APPLICATIONS

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It is well known that particle size, morphology and crystalline homogeneity influence the properties of materials at nanometric scale. In the case of manganites such as $La_{1-x}Sr_xMnO_3$ (LSMO), these properties can be modified following different synthesis techniques. Particularly, the particle size can be controlled using both different synthesis methods and processing conditions. LSMO properties that depend on the particle size are critical for biomedical applications, such as saturation magnetization (Ms), for example, and it has been demonstrated that coupled with the particle size range is the order of magnetic response. By this way, Specific Absorption Rate (SAR), the merit parameter for magnetic hyperthermia applications of nanoparticles is directly proportional to Ms2 [1]. Three different pathways to synthesize $La_{0.7}Sr_{0.3}MnO_3$ nanoparticles have been tested involving the sol-gel process, the ceramic method, and the Pechini's method. The structure, morphology, and magnetic behavior of the resulting nanoparticles were studied. The nanoparticles obtained via the Pechini's method exhibited the best crystalline structure, but smallest size and reduced magnetic properties as compared to the nanoparticles prepared by the other two methods, whereas the best magnetic properties, such as magnetic moment, magnetic saturation and blocking temperature, were found in the particles prepared via the ceramic method. These results made it possible to identify the ceramic method as the synthesis route that produces improved nanoparticles with the greatest probability of utility in biomedical applications. The last was proved by numerical calculations of the specific absorption rate (SAR). Results shown that SAR of nanoparticles synthesized by ceramic method is most than 30 times bigger that SAR for samples synthesized by the other two methods at equal field amplitude and frequency conditions.

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KEYWORDS: SYNTHESIS METHODS, LSMO NANOPARTICLES, MANGANITES, MAGNETIC PROPERTIES, BIOMEDICAL APPLICATIONS

POSTER PRESENTATION

TIME RESOLVED IN-SITU X-RAY ABSORPTION SPECTROSCOPY: NEW PERSPECTIVES IN THE STUDY OF SEMICONDUCTORS WITH PROMISING PHYSICO-CHEMICAL PROPERTIES

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XANES studies, performed at XAFS2 beamline, reveal that $Cr_{(2-x)}Ti_xO_3$ (x=0.2 - CTO) particles present Ti ions in the +3 & +4 oxidation state. Even more, XPS showed that the Ti ions located at the surface of the CTO are in the +4 state. The resistance (R) of CTO is modeled by small polarons hopping (SPH). R-curve presents a change in the activation energy associated with a variation in the conduction mechanism (750-790 K). This change is not observed in the magnetic and structural characterization. The intensity of selected peaks of the Ti K-edge XANES spectra vs. temperature (T) exhibits a break as occurred in the R-curve. This change is associated with variations in the electron-phonon interactions, but there is no theory that explains this intriguing phenomenon at the present. The Ti ions in the particle lead to SPH between the transition metal sites [1]. In addition, the CTO was evaluated as acetone vapor sensor. In-situ XANES experiments shown that the CTO changes its spectra with the incorporation of the vapor [2]. The configuration of the new QUATI beamline will allow us to improve the previous experiment, performing EXAFS with better time resolution (<1 sec). Fitting the spectra we expect to obtain the Debye-Waller factor vs. T. This will let us to better understand the transition observed in the R-curve and perform theoretical calculations to explain the observed results on the EXAFS + XANES vs. T, and be able to explain this new electron-phonon interaction in this kind of system. In addition, the simultaneous measurement of complementary techniques such as Raman spectroscopy (RS) will allow the study the material in one single experiment increasing the reliability and quality of the data. It is important to have tools, like RS, that let monitoring of the surface and volume processes in-situ or in-operando conditions [3]. The measurement of EXAFS and RS simultaneously bring a unique opportunity to understand the sensing mechanism and design new material.

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POSTER PRESENTATION

TUNING OF NI OXIDATION STATE IN Ni/SrTiO$_3$ NANOPARTICLES AND APPLICATION FOR THE METHYLENE BLUE PHOTODEGRADATION

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Nowadays, around 7x10^8 kg of dyes are produced by the industries but approximately 15% are thrown into effluents [1,2]. Methylene blue is a dye that is widely used and there is a strong need to efficiently remove this pollutant from the aquatic medium. An interesting idea is using the Sun's energy that arrives at Earth's surface to induce the photodegradation of methylene blue by using photocatalysts. Furthermore, the use of two semiconductors decreases the electron-hole recombination rate, theoretically increasing the photoreaction efficiency [3]. In this work, commercial SrTiO$_3$ and Ni nanoparticles (NPs) synthesized in accordance to previous work [4] were characterized by X-Ray Diffraction (XRD), Small Angles X-Ray Scattering (SAXS) and Transmission Electron Microscopy (TEM), obtaining a mean size of 35 nm (SrTiO3) and 3 nm (Ni). Then the Ni NPs were exposed to a thermal treatment in ambient atmosphere at 100 ºC, 300ºC and 500ºC. In situ time-resolved X-Ray Absorption Near Edge Structure (XANES) and in situ Extended X-Ray Absorption Fine Structure (EXAFS) techniques were measured at the Ni K edge during the heating treatments at the DXAS and XAFS2 beamlines, respectively, at LNLS. The analysis shows the thermal treatments allows tuning the chemical components of Ni NPs from Ni0 to NiO. Afterwards, 5 wt.% of the Ni NPS was supported on SrTiO3 and used on the photodegradation reaction of methylene blue with an UV light lamp. Samples heated at 300 ºC and 500 ºC showed an improved activity compared to the sample without treatment. X-Ray Photoelectron Spectroscopy (XPS) measurements were performed at the SXS beamline at LNLS for the samples before and after reaction. The Ni 2p$_{3/2}$ region shows the major component at the surface is Ni(OH)$_2$ for samples heated up to 300ºC but NiO appears as the main chemical component for the sample heated at 500ºC. Also, TEM measurements after thermal treatment shows the agglomeration of NPs, mainly at 500ºC, impairs the activity.

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KEYWORDS: PHOTODEGRADATION, METHYLENE BLUE, NANOPARTICLES, PHOTOCATALYSTS, TIME-RESOLVED XANES, EXAFS, XPS

POSTER PRESENTATION

UNDERSTANDING THE SPECIAL CATALYTIC ACTIVITY OF SMALL COPPER CLUSTERS SUPPORTED ON TiO_2

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Sub-nanometer metal structures, usually known as Atomic Quantum Clusters (AQCs) exhibit unique electronic and chemical properties, different from bulk material and even nanoparticles. Because of this, AQCs have attracted much interest recently in the field of catalysis.[1] In particular, Cu clusters have shown important catalytic properties e.g. for the oxidation of CO [2,3] and the selective hydrogenation of olefin and carbonyl groups. [4] Moreover, it has been observed in many cases that AQCs are able to catalyze reactions at lower temperatures and pressures compared to bulk and conventional nanosized materials.[5] We have also shown that the electronic properties of Cu atoms in the AQCs can be tuned when changing the material on which AQCs are supported [6] what makes them suitable for specific-designed catalysts. It is then expected that Cu AQCs can be used for reactions in which cooper is not active. In particular, we have tested the CO_2 hydrogenation, for which bulk metallic copper is inactive, and our catalytic studies have shown that TiO_2 supported Cu_5-AQCs can catalyze the reduction of CO_2 by molecular hydrogen to produce methane. In this work, we perform an in-situ XAFS study of Cu_5-AQCs/TiO_2 catalysts for the CO_2 reduction reaction at the ISS beamline of the National Synchrotron Light Source II (NSLS-II), Upton, New York. We analyze changes in the electronic structure of the AQCs through XANES studies at the Cu K-edge in CO_2, H_2, He atmosphere, and also under reaction conditions (stoichiometric ratios of CO_2 and H_2 for methane synthesis; 1:4) at different temperatures (300-700 K) in order to elucidate the interaction of Cu atoms with the different reactants and to understand the reaction pathway.

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KEYWORDS: XAFS, AQCS, CATALYSIS

POSTER PRESENTATION

UNRAVELING THE GENESIS OF LARGE SUPRAMOLECULAR STRUCTURES OBTAINED FROM SINGLE ARYL AMINO ACID-BASED MOLECULES

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Molecular self-assembly refers to the spontaneous assembly of precursor molecules to form structures though noncovalent interactions.[1] In order to control the architecture of the resulting system, it is necessary to design building blocks assuring the presence of the functional groups which conduct to the development of stable intermolecular interactions with the desired directionality. A large self-assembled supramolecular structure can be obtained using small amino acid-based molecules as building blocks by changing the physicochemical variables of the environment, namely ionic strength, the nature of the solvent, thermal history, an others [2]. These well-organized supramolecular assemblies are employed for several technological applications and they are related to several degenerative disorders, including Alzheimer’s disease, Parkinson’s diseases, and type II diabetes [3]. In this work, a lamellar-like self-assembly model in organic media has been proposed based on single aryl amino acid, L-Phe or L-Tyr, derivatized with the aldehyde piperonal. The main core of the assembly was constructed by ionic intermolecular interactions assisted by H-bonds and, also, with the contribution of contacts involving the aromatic systems. This model was built using X-ray diffraction, density functional theory, small angle X-ray scattering and nuclear magnetic resonance results. The physicochemical entails described at molecular and nanometric level establish the seeds for the formation of crystalline and non-crystalline mesostructures which are triggered by the synthesis conditions.

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KEYWORDS: AMINO ACIDS, ORGANOGEL, SELF-ASSEMBLY, MESOCRYSTALS

POSTER PRESENTATION

UNVEILING THE OCCURRENCE OF Co(III) IN NiCo LAYERED ELECTROACTIVE HYDROXIDES: THE ROLE OF DISTORTED ENVIRONMENTSSIGHT INTO THE ELECTRONIC STRUCTURE BEHAVIOR OF Ni(II)-Co(II) LAYERED HYDROXIDES

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The current requirements of reliable and environmentally friendly energy resources, electrochemical systems have gathered great interest. Two dimensional (2D) materials, as the layered double hydroxides (LDHs), have received tremendous attention in the last years, especially due to their upstanding physical and chemical properties, including in the field of energy storage and conversion.[1-4] The structure of the LDHs consists of stacks of positively charged metal hydroxide layers with charge-balancing anions situated between the layers. They exhibit a formula M_(1-x)^II M_x^III 〖(OH)〗_2^(x+) A_(x/n)^(n-)•m〖 H〗_2 O, where M^II and M^III are divalent and trivalent metals, respectively. In particular, Co- and Ni-based layered hydroxides constitute a unique class of 2D inorganic materials with exceptional physicochemical properties and outstanding performance as supercapacitors and for water splitting catalysts.[5-6] Recently, the occurrence of Co(III) in these phases has been proposed as a key factor that enhance their electrochemical performance. However, the origin of this centers and the control over its contents, remains as an open question. In this work, we employed the Epoxide Route to synthesize a full set of α-NiCo layered hydroxides. The structural characterization (PXRD and XAS), alerts about the distortion over the Co environment mediated by the increment the Ni(II) in the solid phase. The lattice compression promote a shortening over the Co-O distance triggering the oxidation of Co(II) to Co(III) as a function of Ni content. These experimental results are contrasted by DFT+U calculations, confirming the oxidation by the double degeneration of the 3d Co orbital in a distorted environment. This study combines a multiscale crystallochemical analysis supported by a microscopic interpretation, regarding the so called synergistic redox behavior of Co and Ni, offering fundamental tools for the controllable design of highly efficient electroactive materials.

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POSTER PRESENTATION

VUV-UV LUMINESCENCE OF Ce^{3+} AND Bi^{3+} DOPED $LiLaP_4O_{12}$

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Control of light emission in luminescent materials is of great interest due to advantages in improving the applications. The co-doping in inorganic insulators materials is commonly applied in order to enhance the light emission of the activators ions. The present study aimed to observe cooperative effects of Ce^{3+} and Bi^{3+} in light emission and investigate the potential enhancement of the emission intensity caused by the interaction between Ce^{3+} and Bi^{3+}. The synthesis method utilized was the sol-gel assisted by polyvinyl alcohol. Pure and doped samples of $LiLaP_4O_{12}:Ce_x,Bi_y$ (x = 0, 0.5, 1, 2; y = 0, 2) powders were produced and single crystalline phase was confirmed by X-ray powder diffraction. The luminescent investigation was carried out from photoluminescence emission (PL) and excitation (PLE) measurements at room temperature with synchrotron radiation at the TGM beamline installed in the UVX accelerator in the LNLS. Ionizing radiation conversion to light was investigated from radioluminescence (RL) measurements. PL for Ce^{3+}-doped sample showed the emission with a maximum at around 315 nm related to the transitions from $5d^1$ to 4f ground state. For Bi^{3+} ion doping, PL results shows an emission band centered around 440 nm related to transitions from conduction band to $1^S{}_0$ ground state. In co-doped samples, the emission of each ion, Ce^{3+} and Bi^{3+}, was observed for excitation at 4.9 eV. PLE results from Ce^{3+}-doped sample showed all the five excited states due to the 5d states. For Bi^{3+}-doped sample, PLE spectra revealed two main bands, the first one with a maximum at around 5.3 eV and the other at around 6.5 eV. For co-doped samples, monitoring the emission region for Ce^{3+}, the PLE revealed that only three of the five original Ce^{3+} related bands are present, when Bi^{3+} ions are present. X-ray excited emission spectra shows an valence change in Bi-doped sample.

POSTER PRESENTATION

X-RAY ABSORPTION FINE STRUCTURE SPECTROSCOPY AND PHOTOLUMINESCENCE STUDY OF MULTIFUNCTIONAL EUROPIUM (III)-DOPED HYDROXYAPATITE

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Europium-doped hydroxyapatite has attracted considerable scientific attention to smart drug delivery scaffold applications because of its biocompatible and traceable luminescence features. In this sense, to investigated the europium incorporation mechanism and photoluminescence properties in the presence of surfactant organic molecules the europium doped-hydroxyapatite samples were synthesized via co-precipitation with hexadecyl trimethylammonium bromide (CTAB) micelles mediated approach. Results of the X-ray absorption spectroscopy techniques (XANES and EXAFS) showed that Eu^{3+} ions were incorporated preferentially at the Ca (1) site with local charge compensating via oxygen interstitial ion for samples calcined under air at 450 °C/4 h °C. Europium(III) luminescence analysis suggested that Ca site symmetry became distorted in CTAB medium.

KEYWORDS: HYDROXYAPATITE, EUROPIUM DOPING, X-RAY ABSORPTION SPECTROSCOPY, LUMINESCENCE

POSTER PRESENTATION

X-RAYS INDUCED BIOLOGICAL DIRECT AND INDIRECT DAMAGE SIMULATIONS USING GEANT4-DNA

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This present study aims on the simulation of biological direct and indirect damage done to model microorganisms, which would be caused by X-ray ionizing radiation in the Mogno beam line at the Sirius Light Source Laboratory, using the Geant4 Monte Carlo simulation toolkit, and its DNA extension [1-3]. Understanding the effects of ionizing radiations on living beings, in their cells and/or tissues, which can be either direct or indirect ones, is fundamental for radiobiology, radiodiagnosis and radio imaging, but also in the developing Astrobiology scientific field as well, where these effects have a main role in the planning of future space missions and in the knowledge about the conditions for planetary Habitability. Microorganisms simulated here were the Deinococcus radiodurans bacteria, a well-know ionizing radioresistant model organism used in many studies on the biological effects of ionizing radiations and their resistance mechanisms; and two strains of Saccharomyces cerevisiae, commonly known as baker's yeast, which are also extensively used as model organisms in many survival experimental assays. Simulations results obtained here will then be compared with the experimental ones obtained at Mogno beam line, where the models organisms will be exposed to increasing X-rays doses from the Sirius Light Source, and then checked for their viability after certain Lethal Doses thresholds. This comparative will be used for validation and for further development of guidelines for both future simulations and the experimental setups and protocols that will be developed for the new Sirius Light Source beam lines.

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KEYWORDS: DNA DIRECT DAMAGE, SYNCHROTRON RADIATION DAMAGE, GEANT4-DNA

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