28th RAU ANNUAL USERS MEETING LNLS/CNPEM

November 7th - 9th, 2018

ABSTRACT BOOK

MINISTRY OF
SCIENCE, TECHNOLOGY
INNOVATION AND COMMUNICATION

BRAZILIAN
GOVERNMENT

Organization

LNLS sirius

CNPEM

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SCIENCE, TECHNOLOGY
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Foreword

Dear LNLS Users,

On behalf of the Scientific Committee of the 28th Annual Users Meeting of the LNLS, I welcome you warmly.

When a living organism procreates, evolution is inevitable. The Brazilian (and why not Latin American) synchrotron users community, has behaved as a living organism. It was born in 1997 when UVX shone its first light and made available its first few beamlines. And the community has thrived over the years, multiplying in number, bringing new, younger and more diverse members together every year. Using different tools, with broader scientific interests and instrumentation needs, accompanying the new and better beamlines available, and pushing UVX to its maximum potential. Nowadays evolution is not only a need, it is a reality. As we will walk through the floor of SIRIUS we will witness the next step of evolution, but also the ecological niche where this community will continue growing, evolving to a world class community with state of the art tools. We will be using newer and better techniques to make the best of our science. This is the 28th RAU, the last step in UVX path, and the first step of SIRIUS unexplored land.

The excitement of this time is evident in the large number of registered participants (more than 250). The scientific program will include around 90 poster presentations, 30 oral contributions and 4 invited speakers, making this the largest RAU so far. In order to prepare for the new challenges that this new ecological niche opens, the Scientific Committee has brought a superb group of speakers in very different areas of knowledge. Dr. Brian H. Toby (APS Chief Computational Scientist, Advanced Photon Source, Argonne National Laboratory, USA) will show us how large user facilities handle the large amounts of data generated and what they offer to the users for analysis and handling, in addition to his own contributions to software development; Prof. Adam P. Hitchcock (McMaster University, Canada) will talk about soft X-ray Scanning Transmission Microcopy advances and potentialities for *in situ* materials characterization at the nanoscale; Prof. Robert Leheny (John Hopkins University, USA) will talk about X-ray photon correlation spectroscopy applied to complex fluids characterization such as nanocolloids, clay suspensions and nanocolloidal glasses; and last but not least Dr. Giuliana Tromba (SYnchrotron Radiation for MEdical Physics – SYRMEP Group, Elettra Sincrotrone, Trieste, Italy) will talk, in a web conference, about the latest developments in biomedical research using X-ray imaging techniques. The scientific program will be completed with SIRIUS status presentations and a plethora of discussion opportunities with speakers and presenters during coffee and lunch breaks, posters session and a roundtable with users just before the closure.

No part of this RAU would have been possible without the dedicated work of the Local Committee and CNPEM staff that participated in the effort to have everything ready for us. We are also indebted to the sponsors and main funding agencies FAPESP, CAPES, and CNPq which made this important event possible.

Leopoldo Suescun
Chair of the 28th RAU

CAPTION:

📍 CTBE Brazilian Bioethanol Science and Technology Laboratory

📍 LNLS Brazilian Synchrotron Light Laboratory

📍 LNNano Brazilian Nanotechnology National Laboratory

📍 LNBio Brazilian Biosciences National Laboratory

📍 sirius

Program

November 6th

08:30 – 18:00	**Satellite Events Program, see details below**

November 7th

08:30 – 10:00	**Satellite Events Program, see details below**
10:00 – 12:00	**Reception and Registration**
10:00 – 11:00	**Special Seminar: Crystallographic data analysis with GSAS-II package** Brian Toby (Argonne National Laboratory, USA)
10:00 – 12:00	**Sirius tours**
12:00 – 13:45	**Lunch**
13:45 – 14:00	**Welcome by the Scientific Committee**
14:00 – 14:30	**Sirius Status Report** Antonio José Roque da Silva (CNPEM, Director)
14:30 – 15:00	**LNLS Science Highlights** Yves Petroff (LNLS Director)
15:00 – 15:30	**Overview of Sirius Beamlines** Harry Westfahl (LNLS, Scientific Director)
15:30 – 16:00	**Official Photo and Coffee Break**
16:00 – 18:30	**Thematic Session: LNLS Beamline Highlights**
18:30 – 20:30	**Social Gathering**
20:45	**Bus Departure (Matiz Hotel/Barão Geraldo)**

November 8th

08:30 – 09:15	**Plenary 1: Addressing the Computational Needs of APS Users** Brian Toby (Argonne National Laboratory, USA)
09:15 – 10:00	**Plenary 2: Nanoscale materials analysis by soft X-ray Scanning Transmission Microscopy** Adam P. Hitchcock (McMaster University, Canada)
10:00 – 10:30	**Coffee Break**
10:30 – 12:30	**Oral Communications 1: Plenary and Sirius A rooms**
12:30 – 14:00	**Lunch**
14:00 – 15:45	**Oral Communications 2: Plenary and Sirius A rooms**
15:45 – 16:15	**Coffee Break**
16:15 – 17:15	**Poster Session (Odd Numbers)**
17:15 – 18:15	**Poster Session (Even Numbers)**
18:30	**Bus Departure (Matiz Hotel/Barão Geraldo)**

Program

November 9th

08:30 – 09:15	**Plenary 3: Microscopic dynamics underlying the nonlinear rheology of soft glassy materials** Robert L. Leheny (Johns Hopkins University)
09:15 – 10:00	**Plenary 4 (web conference): Hard X-ray biomedical imaging at Elettra: status and perspectives** Giuliana Tromba (Elettra Sincrotrone Trieste)
10:00 – 10:30	**Coffee Break**
10:30 – 12:30	**Roundtable with users**
12:30 – 12:45	**Closing**
12:45 – 14:00	**Lunch**
12:45 – 15:00	**Bus Departure (GRU/VCP Airports)**

CPU: Satellite Event Program

November 6th

13:30 – 14:00	**Reception and Registration**
14:00 – 14:15	**Welcome and Sirius Introduction** Harry Westfahl (LNLS Scientific Director)
14:15 – 14:30	**Instrumental resolution versus results resolution in 2D and 3D imaging** Marin Van Heel (LNNano)
14:55 – 15:20	**Distributed Processing of Fast Fourier transforms** Fernando Furusato (Unicamp)
15:20 – 16:00	**Scientific Computing at Diamond Light Source – Challenges and development** Dave Bond (Diamond)
16:00 – 16:20	**Coffee Break**
16:20 – 16:50	**The phase problem and future perspectives** Eduardo Miqueles (LNLS)
16:50 – 18:00	**Image Segmentation and Analysis at LNLS/Sirius: Yesterday, Today, and Tomorrow** Thiago Spina (LNLS)
18:00	**Bus departure (Matiz Hotel/Barão Geraldo)**

November 7th

08:30 – 09:15	**Volumetric data visualization in virtual reality** Edgar Gadbem (Instituto Eldorado)
09:15 – 10:00	**Mona Project: prototyping real-time HPC analysis** Brian Toby (Argonne)
10:00 – 10:15	**Closing remarks** Eduardo Miqueles (LNLS)

MIRCA: Satellite Event Program

November 6th

08:30 – 9:00	**Reception and Registration**
09:00 – 9:15	**Opening** Yves Petroff (LNLS Director)
09:15 – 10:00	**IR program at LNLS and future at SIRIUS** Raul Freitas (LNLS)
10:00 – 10:45	**Committing SINS and Other Acts of Infrared** Hans Bechtel (ALS)
10:45 – 11:05	**Coffee break**
11:05 – 11:35	**Optical characterization of 2D and layered materials** Christiano de Matos (Mackgraphe)
11:35 – 12:05	**Polaritons in Two-dimensional Materials and Heterostructures** Francisco Maia (LNLS)
12:05 – 12:15	**Sample preparation strategies for micro and nano-FTIR** Thiago Miguel (LNLS)
12:15 – 13:00	**Discrimination of materials and heterogeneities in stratigraphic samples from cultural heritage by µSR-FTIR** Victoria Beltran (SOLEIL)
13:00 – 14:00	Lunch
14:00 – 14:45	**Past, present and future of IR spectromicroscopy in SOLEIL** Ferenc Borondics (SOLEIL)
14:45 – 15:15	**Fabrication Process, Challenges and Applications in New Two-Dimensional Materials Beyond Graphene** Ingrid Barcelos (LNLS)
15:15 – 15:45	**Synchrotron radiation applied to the characterization of perovskite films: morphology, structure and composition** Ana Flavia Nogueira (Unicamp)
15:45 – 16:20	**Coffee break**
16:20 – 17:05	**Rapid 2D and 3D Infrared Imaging Applied to Biologically and Chemically Complex Systems** Carol Hirschmugl (UoM)
17:05 – 17:45	**Open Discussion**
17:45 – 18:00	**Closing remarks** Raul Freitas (LNLS)
18:00	**Bus departure (Matiz Hotel/Barão Geraldo)**

SMX: Satellite Event Program

November 6th

08:30 – 09:30	**Reception and Registration**
09:00 – 09:15	**Opening** Harry Westfahl (LNLS Scientific Director)
09:15 – 09:45	**Manaca and crystallography at Sirius** Ana Carolina Zeri (LNLS)
09:45 – 10:45	**The XChem Facility at Diamond** José Ribeiro Brandão Neto (Diamond)
10:45 – 11:05	**Coffee Break**
11:05 – 12:00	**Experience of using high-throughput fragment screening for developing new anti-Schistosoma drugs** Nicholas Furnham (LSHTM)
12:00 – 13:00	**X-ray crystallography fragment screening against the human prion protein: pitfalls and achievements** Maria Cristina Nonato (USP/RP)
13:00 – 14:00	**Lunch**
14:00 – 14:45	**Chasing a hit from a fragment library** Andre Godoy (USP/SC)
14:45 – 15:30	**Fragment based drug discovery approach to targeting Dihydrofolate reductase from Mycobacterium tuberculosis** Marcio Dias (USP/SP)
15:30 – 16:15	**Structure-based drug discovery for Chagas Disease at LNBio** Artur Cordeiro (LNBio)
16:15 – 16:45	**Coffee**
16:45 – 17:45	**NP3 platform: empowering bioactive natural product drug discovery using protein crystallography** Daniela B Trivella (LNBio)
17:45 – 18:00	**Informal Discussion**
18:00	**Bus Departure (Matiz Hotel/Barão Geraldo)**

November 7th

09:00 – 09:40	**High-Field Solution NMR Spectroscopy Approach for Fragments and Drugs Evaluation** Mauricio L. Sforça (LNBio)
09:40 – 10:15	**Structure-based Development of Chemical Probes for Understudied Protein Kinases** Rafael Counago (SGC/Unicamp)
10:15 – 10:30	**Closing remarks and Visit to the beamline site** Ana Carolina Zeri (LNLS)

Thematic Sessions Program

November 7th

16:00 – 16:25	**Science opportunities on the Carnaúba beamline** Helio Tolentino (LNLS)
16:25 – 16:50	**Science opportunities on the Cateretê beamline** Florian Meneau (LNLS)
16:50 – 17:15	**Science opportunities on the Manacá beamline** Ana Carolina Zeri (LNLS)
17:15 – 17:40	**Science opportunities on the Ipê beamline** Raul de Oliveira Freitas (LNLS)
17:40 – 18:05	**Science opportunities on the Mogno beamline** Nathaly Lopes Archilha (LNLS)
18:05 – 18:30	**Science opportunities on the Ema beamline** Narcizo Marques de Souza Neto (LNLS)

Poster Session Program

The poster session will take place on November 8th at LNLS´ Experimental Hall (Sponsors and coffee break area). During the poster session, presenters must stay by their posters for discussion and to answer questions.

November 8th	
16:15 – 17:15	**Presentation of odd-numbered posters**
17:15 – 18:15	**Presentation of even-numbered posters**

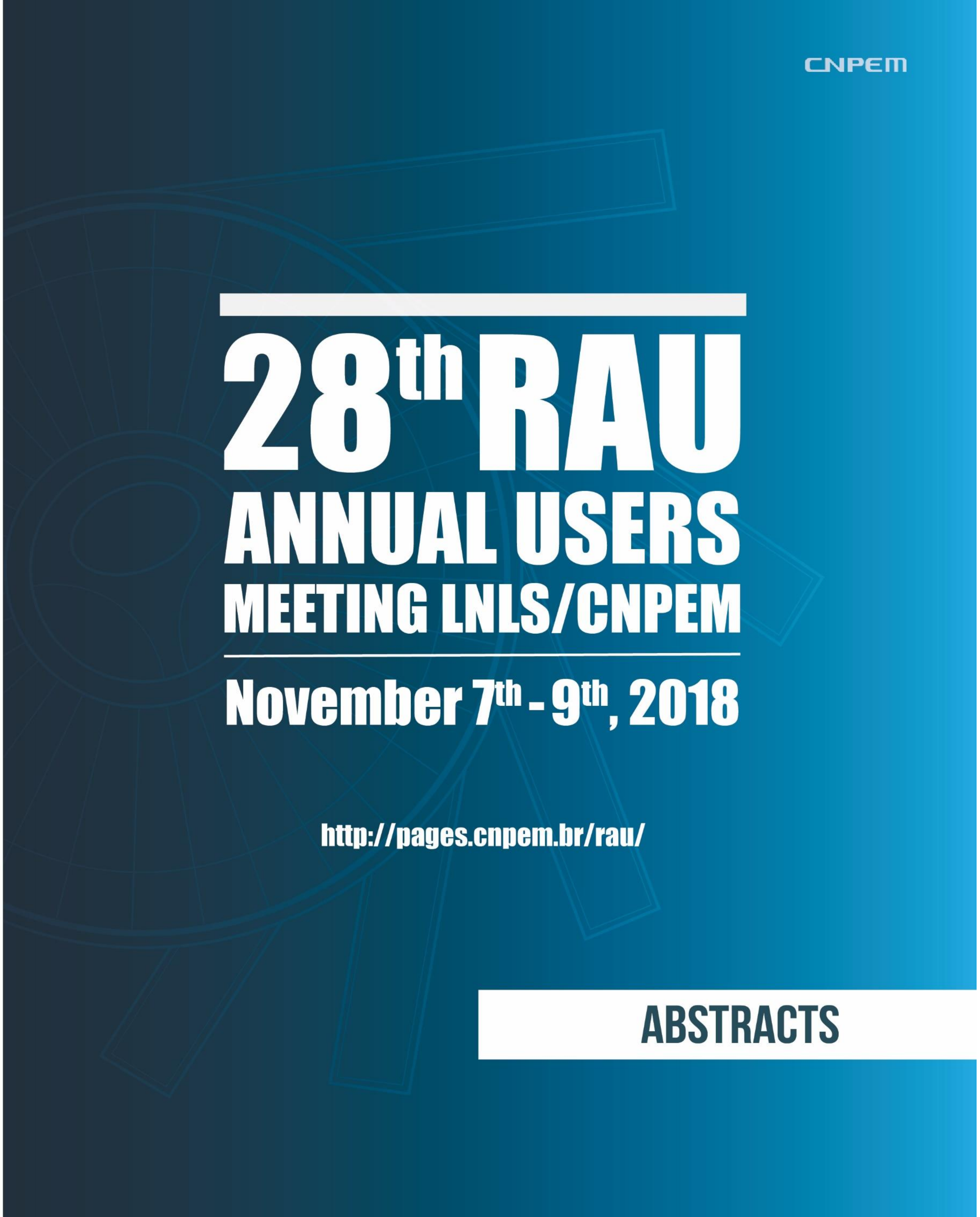
Poster presenters must check the number assigned to their posters on the website (http://pages.cnpem.br/rau/poster/) or in the venue before placement of the poster.

Oral Communications Program

November 8th	PLENARY Room
	Condensed Matter, Surface science, Instrumentation, etc. CHAIR: Flávio García & Roberto Paniago
10:30 – 10:45	**Investigation of Protein-Surfactant Complexes** Cristiano Luis Pinto de Oliveira
10:45 – 11:00	**In situ Small Angle X-ray Scattering monitoring of traditional FAU zeolite synthesis.** Paloma Vinaches Melguizo
11:00 – 11:15	**Understanding hybrid organic-inorganic perovskite formation during spin coating through the in-situ time resolved GIWAXS** Rodrigo Szostak
11:15 – 11:30	**Probing PtPd nanoparticles using ions, electrons and x-rays** Jonder Morais
11:30 – 11:45	**Unraveling the Atomic Structure of Fe Intercalated under Graphene on Ir(111)** Abner de Siervo
11:45 – 12:00	**Characterization of CdWO4-polystyrene composite films using synchrotron light** Zelia Soares Macedo
12:00 – 12:15	**Cation inversion in bimagnetic CoO/$Co_{1-x}Zn_xFe_2O_4$ core/shell nanoparticles: a XANES study** Martín Eduardo Saleta
12:15 – 12:30	**Use of soft X-ray scattering for 3D characterization of magnetic nanostructures** Samuel Flewett
	Hard condensed matter & Condensed Matter CHAIR: Eduardo Granado
14:00 – 14:15	**Magnetic Structures and Transitions of GdNiSi3 and TbNiSi3** Rodolfo Tartaglia Souza
14:15 – 14:30	**Anisotropic magnetic excitations and incipient Néel order in Mn substituted Iron Arsenides** Fernando Assis Garcia
14:30 – 14:45	**Residual Stress of Ni9Mn and Ni2MnGa thin films** Aluizio Jose Salvador
14:45 – 15:00	**$La_3Co_4Sn_{13}$ Superconducting Compound Under Pressure** Eduardo Matzenbacher Bittar
15:00 – 15:15	**X-ray induced persistent luminescence: How and why?** Lucas Carvalho Veloso Rodrigues
15:15 – 15:30	**Analysis of the surface lattice structure of ion beam nanostructured Si substrates for thin film deposition** Roosevelt Droppa Jr.
15:30 – 15:45	**Mechanochemical synthesis and characterization of nanocrystalline Chalcogenide alloys** Carlos Eduardo Maduro de Campos

Oral Communications Program

November 8th	SIRIUS A Room
	Life and Medical Sciences & Drug Discovery CHAIR: Rafael V. C. Guido & Maria do Carmo Alves
10:30 – 10:45	**NP3 platform: assisting new discoveries from natural products** Daniela Barretto Barbosa Trivella
10:45 – 11:00	**Mayaro virus infection in immunocompromised mice leads to paw inflammation** Ana Carolina de Carvalho
11:00 – 11:15	**Metals and Glycosaminoglycans in Tumor Progression** Mariana Paranhos Stelling
11:15 – 11:30	**Three-dimensional neuronal architecture revealed by high-resolution synchrotron-based X-ray microtomography** Matheus de Castro Fonseca
11:30 – 11:45	**Morphological analysis of induced pluripotent stem cells derived cerebral organoids from corpus callosum dysgenesis patients and control subjects** Livia Goto Silva
11:45 – 12:00	**Application of SR microXRF for mathematical modelling of Pb diffusion in Brassica napus leaf blade used for phytoremediation studies** Maria Fernanda Mera
12:00 – 12:15	**Unveiling early microfossils with integrative imaging techniques** Lara Maldanis
12:15 – 12:30	**Mars soil analogues: the case of Diamantina-MG red soil** Thiago Monfredini
	Environmental and Earth Science & Catalytic Materials CHAIR: Félix Requejo
14:00 – 14:15	**Bacterial mutualism as novel strategies for ROSs (Radical Oxygen Species) and copper homeostasis under extreme acidic and microaerophilic conditions** Julio Castillo
14:15 – 14:30	**µ-XRF and µ-XANES analysis of metal nanoparticles in common bean seeds: Understanding NPs transport and their role during germination** Susilaine Maira Savassa
14:30 – 14:45	**Structural and physicochemical properties of complex oxides for energy applications: XRD and XAS studies** Rodolfo Oscar Fuentes
14:45 – 15:00	**XANES study of cerium oxidation state of ceria-based catalyst in catalytic operating conditions** Lucia M. Toscani
15:00 – 15:15	**Tunning the oxygen vacancy population of CeO_{2-x} ($0 < x < 0.5$) nanoparticles** Fabiano Bernardi
15:15 – 15:30	**XANES study of Sulfur oxidation by Ag and Cu Atomic Quantum Clusters in Cysteine and Glutathione** Juan Marcelo Devida
15:30 – 15:45	**In situ characterization of CuxNi1-x/CeO2 (0 < x < 1) nanoparticles applied to the RWGS reaction** Wallace Torres de Figueiredo



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http://pages.cnpem.br/rau/

ABSTRACTS

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SUMMARY

PLENARY SESSION ABSTRACTS

ORAL AND POSTER COMMUNICATIONS ABSTRACTS

PLENARY SESSION ABSTRACTS

PLENARY TALK 1

Addressing the Computational Needs of APS Users

Brian Toby,
Advanced Photon Source, Argonne National Laboratory, USA

ABSTRACT:

Available software very much limits the ability of scientists to obtain scientific results from their beamline experiments. The need for software has grown due to revolutionary use of area detection for x-ray experiments and by how these detectors have improved in performance. Another factor increasing the need for software development has been the success for synchrotrons in acquiring users who are experts in their scientific domain and want to apply synchrotron-based measurements in their work, but are not experts in synchrotron use and do not wish to become such. These users need easy-to-master data analysis tools, since they cannot invest as much time in learning as someone dedicating his/her career to synchrotron science. In the immediate, future x-ray detectors will continue generate data at increasingly faster rates and new light sources will produce beams with vastly greater brilliance and coherence. Keeping up with these data streams requires software that runs on massively parallel computers.

This presentation will provide an overview of software development projects at the APS and some of future directions.

PLENARY TALK 2

Nanoscale materials analysis by soft X-ray Scanning Transmission Microscopy

Adam P. Hitchcock
Brockhouse Institute for Materials Research, McMaster University, Hamilton, Canada
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ABSTRACT:

Soft X-ray scanning transmission microscopy (STXM) [1-3] is a powerful tool for nanoscale materials analysis, with significant advantages over analytical X-ray microscopies for studies of radiation sensitive materials, and for *in situ* and operando studies. Over 12 facilities are operational world-wide and several more are being developed. Ptychography (scanning coherent diffraction imaging) [3,4], which can be measured using soft X-ray STXMs, provides significant improvements in spatial resolution (3-14 nm relative to 15-30 nm for conventional STXM. 4D imaging – quantitative 3D imaging with spectroscopy at each voxel – can be performed with STXM [5] and ptychography [6]. STXM and ptychography instrumentation at various facilities will be described, with emphasis on spectromicroscopy, the use of chemical mapping by imaging at multiple photon energies. A recently commissioned cryo-STXM [7] will be described and cryo-spectrotomography results presented. Applications to be described include: electrode materials for polymer electrolyte membrane fuel cell (PEM-FC) devices [4,6,8,9], and alumina aerogels coated with zinc oxide by atomic layer deposition (ALD).

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[2] H. Ade and A.P. Hitchcock, Polymer 49 (2008) 643;
[3] D.A, Shapiro et al., Nat. Photonics 8 (2014) 765;
[4] A.P. Hitchcock, J. Electron Spectrosc. Rel. Phen. 200 (2015) 49-63;
[5] G. Schmid, M. Obst, J. Wu and A.P. Hitchcock, *3D chemical imaging of nanoscale biological, environmental and synthetic materials by soft X-ray spectro-tomography* in Characterization Tools for Nanoscience & Nanotechnology, Vol. 5 (Springer, Berlin, 2015);
[6] J. Wu et al. *High-resolution imaging of polymer electrolyte membrane fuel cell cathode layers by soft X-ray spectro-ptychography*, J. Phys. Chem. C (2018) in press;
[7] A.F.G. Leontowich et al., *Cryo scanning transmission X-ray microscope optimized for spectro-tomography*, Rev. Sci Inst. (submitted);
[8] A.P. Hitchcock, et al., J. Power Sources 266 (2014) 66;
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ACKNOWLEDGEMENTS

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PLENARY TALK 3

Microscopic dynamics underlying the nonlinear rheology of soft glassy materials

Robert L. Leheny
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ABSTRACT:

Small-angle x-ray photon correlation spectroscopy (XPCS) accesses dynamics on length scales from nanometers to hundreds of nanometers and time scales from milliseconds to hundreds of seconds that figure prominently in the rheological behavior of complex fluids, and the technique has provided numerous insights into the microscopic origins of the rheology of soft materials. This talk will review recent efforts and discuss future prospects to expand the repertoire of XPCS by using it to probe structural dynamics during in situ mechanical deformation and flow. Such rheo-XPCS experiments include those incorporating steady shear, large amplitude oscillatory shear (LAOS), or tensile strain. As specific examples, the talk will describe recent experiments that reveal the microscopic structural dynamics associated with yielding and stress relaxation in a set of disordered soft solid materials including nanocolloidal gels, clay suspensions, and soft nanocolloidal glasses.

PLENARY TALK 4

Hard X-ray biomedical imaging at Elettra: status and perspectives

Giuliana Tromba, on the behalf of the SYRMEP team
Elettra – Sincrotrone Trieste, Italy

ABSTRACT:

The SYnchrotron Radiation for MEdical Physics (SYRMEP) beamline has been in operation for more than 20 years at the Elettra light source in Trieste. Biomedical research has been developed following three main directions, each corresponding to a resolution and dimension scale: clinical imaging with patients, studies on small animals, high resolution imaging (embryos, tissues, histologies, biomaterials, etc.). The beamline offers various setups allowing for imaging modalities with monochromatic and white beam, and moreover a radiological unit is used for mammographic studies on patients. Phase contrast imaging in Free Propagation modality and Analyzer Based Imaging are the most used imaging techniques. The experimental setups are very flexible, suited for studying samples with dimensions ranging from a few mm to several cm, and cover a wide resolution range, with detectors' pixel sizes varying from 0.9 micron to 100 microns. The talk will give an overview of the latest achievements, highlighting the upgrade plans and the main research perspectives in view of the Elettra 2.0 program.

ORAL AND POSTER COMMUNICATION ABSTRACTS

POSTER PRESENTATION

Alterations in mammary cells exposed to radiation doses used in screening mammography

Carla L Mota[1], Andrea Mantuano[1], Arissa Pickler[2], Camila Salata[3], Samara C. Ferreira-Machado[1], Carlos E. de Almeida[4]

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[2] COPPE, Federal University of Rio de Janeiro (UFRJ), Brazil
[3] National Commission of Nuclear Energy (CNEN), Brazil
[4] Fundação do Câncer, Brazil
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ABSTRACT:

Controversy exists regarding the biological effectiveness of low energy x-rays used for mammography breast screening [1]. Recent radiobiology studies have provided compelling evidence that these low energy x-rays may be 4.42 times more effective in causing mutational damage than higher energy x-rays [2]. Studies indicate changes in the concentration of some elements in different tumor cell lines, after different treatments, using the TXRF method [3,4]. We analyzed changes in elemental composition in both human breast tumor cells (MCF-7) and non-tumor cells (MCF-10), subjected to doses used in mammography considering low dose (LD) (17 mGy) and high radiation (HD) doses (2 Gy). The trace elements (S, K, Ca, Cr, Fe and Zn) were quantified using TXRF line of LNLS (Laboratório Nacional de Luz Síncrotron, Brazil) [5]. The results showed statistically significant differences of all the analyzed elements among control (non-irradiated) and irradiated samples for both tumor and non-tumor cell groups. In HD non-tumor cells, the concentration of S, K, Cr are lower when compared to control and Ca and Zn are lower when compared to LD group. On the other hand, S, K, Ca, Fe and Zn concentrations are higher in HD tumor cells when compared to control and LD groups. For both tumor and non-tumor groups the trace element profile changed after high radiation doses exposure, and this profile only changed for non-tumor cells after low radiation dose exposure. This can indicate that tumor mammary cells are more radioresistant than healthy mammary cells. The study of this elements can help to better understand how the ionizing radiation changes the cell metabolism, prevent the possible damage caused by radiation on healthy mammary cells, an enhance the effects caused on tumor cells.

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ORAL PRESENTATION

Analysis of the surface lattice structure of ion beam nanostructured Si substrates for thin film deposition

Roosevelt Droppa Jr.*[1], Silvia Cucatti[2], Mónica Morales[2], Vinícius Antunes[2], Fernando Alvarez[2]

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ABSTRACT:

Studies done recently have been reported on the big influence of the metallic thin films, such as TaNx [1] and TiNx [2] on both the growth and the physical properties of carbon nanotubes grown by CCVD (Catalytic Chemical Vapor Deposition) on coated silicon substrates. Such films not only act as a barrier against catalyzer diffusion in the silicon substrate, but also influence directly the distribution of the catalyzer, which in its turn modifies the diameter as well as the surface density of carbon nanotubes. Thus, to obtain an organized distribution of nanotubes, a nanostructured (001) single crystal silicon substrate was previously prepared in such a way to induce the self-organization of the catalyzer particles. The preparation consisted in bombarding almost perpendicularly the silicon crystal with a 1.0 keV xenon ion beam during 60 minutes, in the same manner and getting the same results as other groups [3]. Such a nanostructuring process induces the formation of periodic undulations on the substrate surface which acts like a template of hills and valleys. Such a relief pattern generates stress at the first surface nanometers of the initially crystalline silicon. Once the substrate surface was prepared, a TiNxOy thin layer approximately 15 nm thick was then deposited thereon. In this work we studied the strains in the surface crystal structure of the bombarded silicon substrate, relating them to the direction of its hills and valleys formed after bombardment and correlating them to the process of the islands self-organization. The bombarded Si substrate samples were analyzed by high resolution scans in the reciprocal space in in-plane grazing incidence x-ray diffraction (GIXD) mode in order to obtain the lattice strain in the surface top layers. High resolution reciprocal space maps were obtained, and the direction of the strain was compared with the direction of the undulations formed by bombarding the Si substrate.

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ACKNOWLEDGEMENTS:

FAPESP

ORAL PRESENTATION

Anisotropic magnetic excitations and incipient Néel order in Mn substituted Iron Arsenides

Fernando A. Garcia*[1], Oleh Ivashko[2], Daniel E. McNally[3], Lakshmi Das[2], Mario M. Piva[4], Pascoal G. Pagliuso[4], Johan Chang[2], Thorsten Schmitt[3], Claude Monney[5]

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ABSTRACT:

Magnetic excitations in the iron arsenides BaFe1-xMnx2As2 (x = 0.0, 0.007, 0.009, 0.08) are investigated by means of Resonant Inelastic X-rays Scattering (RIXS) at the Fe L3-edge, for momentum transfer q along the high symmetry Brillouin zone (pi,0) and (pi,pi) directions. It is shown that with increasing Mn content (x), the excitations become anisotropic both in dispersion and lineshape along the (pi,0) and (pi,pi) directions. Both effects are detected even for small values of x, evidencing a cooperative phenomenon between the Mn impurities, that we ascribe to emerging Néel order due to Mn spins. For x =0.08, in particular, the excitations for which q || (pi,0) are strongly damped and nearly non-dispersive, a finding that can be connected to the absence of superconductivity in phases of arsenides wherein local moments where added to the FeAs layers, as in Mn or Cr substituted phases.

POSTER PRESENTATION

Application of NEXAFS and resonant Auger spectroscopies to investigate electronic structure, morphology and charge transport of GO-PEDOT:PSS blends for organic photovoltaics

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ABSTRACT:

Organic photovoltaics (OPVs) present important advantages such as flexibility, thinness, and simple manufacturing process [1]. In this study the electronic structure and molecular orientation of thin films of GO-PEDOT:PSS with the composition of 1, 5, 10 and 100 v/v% of PEDOT:PSS were investigated by Near Edge X-ray Absorption Fine Structure (NEXAFS) spectroscopy [2] using two different detection modes: total electron yield (TEY) and fluorescence yield (FY), at different incident angles. Ultrafast electron dynamics in the low-femtosecond regime was assessed by resonant Auger spectroscopy using the Core-Hole Clock (CHC) method [3] at the sulfur 1s absorption edge. The experiments were performed for two series of cooled and not-cooled samples, aiming to monitor the change in electronic and structural properties caused by the thermal treatment. The experiments were performed at the SXS beamline of the LNLS, Campinas, SP. Charge transfer time was calculated from the ratios of the spectators and normal Auger signals using the sulfur core-hole lifetime 1.27 fs [4] as an internal reference clock. Charge transfer time results showed that the GO-PEDOT:PSS 5% blend has the lowest charge delocalization time in comparison with the other blends, in accordance with electrical results. This observation indicates that there is a synergetic effect for the blend containing 5% of PEDOT:PSS, which has higher electron mobility than other blends. Sulfur 1s TEY and FY NEXAFS spectra of GO-PEDOT:PSS (5%), were measured at three different incidence angles. The PEDOT transitions include the overlapping of $S1s \rightarrow \pi^*$ and $S1s \rightarrow \sigma^*$ at 2472 and 2473.2 eV, respectively. A strong angular dependence for PEDOT at both transitions, especially at the $S1s \rightarrow \pi^*$, in cooled and not-cooled samples, can be observed, which is more intense in the FY mode. At normal incidence, the intensity of the $S1s \rightarrow \pi^*$ transition is higher, indicating that the PEDOT unit is preferably oriented perpendicular to the substrate surface.

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Poster Presentation

Application of Ni/SrTiO3 nanoparticles to the photodegradation reaction of methylene blue

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ABSTRACT:

Methylene blue is a widely used dye that gives serious environmental issues, especially concerning the water's quality. There are methods to transfer dyes from solid to liquid phase but side pollution is produced [1]. An interesting idea is to use the Sun's energy that arrives at the Earth surface [2] to induce the photodegradation of methylene blue by using catalysts [1]. However, due to the typical band gap of these catalysts only photons of the UV region can be absorbed, which represents only 5% of the intensity of the solar spectrum at the Earth surface. Ni/SrTiO3 nanoparticles (NPs) are able to extend the absorption of the UV radiation to the visible region of the spectrum [3], then increasing the photocatalytic activity. In this work, Ni NPs were synthesized according to previous work [4] and exposed to thermal treatments at 100 °C, 300 °C and 500 °C during 1h in atmosphere aiming to tune the Ni size and oxidation state. After, these Ni NPs were supported on commercial SrTiO3 and applied to the methylene blue photodegradation using a homemade photoreactor, which is equipped with two 15 W lamps that simulate the solar spectrum. After the reaction, UV-Vis measurements showed a photocatalytic activity increase of up to 38% in comparison to the NPs without thermal treatment. The NPs were characterized by XRD, SAXS and XPS techniques before and after the reaction. The XRD patterns showed the presence of the SrTiO3, SrCO3 and TiO2 crystalline phases for the support and Ni and NiCl2 for the NPs. Rietveld refinement results showed a mean size of the SrTiO3 and Ni NPs of 40 nm and 2 nm, respectively. SAXS curve fitting suggests SrTiO3 NPs with a mean size in accordance to the XRD results and shows that Ni NPs present two distinct NPs population: a spherical one and a core-shell like structure formed by the Ni and NiCl2 phases. The XPS measurements allowed to identify the oxidation state of the NPs, which are correlated with the photocatalytic activity results.

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ORAL PRESENTATION

Application of SR microXRF for mathematical modelling of Pb diffusion in Brassica napus leaf blade used for phytoremediation studies

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ABSTRACT:

Hyperaccumulators plants can translocate heavy metals absorbed by roots to the shoots and certain amount of metal remains in the leaves tissue until the leaf falls. Following the pathway of Pb from soil to leaf, it is postulated that the contaminant enters from the root and then is drawn to leaves blade via the xylem of the stem, midrib and secondary veins of the leaf, to allocate it in the widened and flat part called limbus (leaf). To investigate the uptake of lead and to understand the mechanisms involved in the phytoremediation process, the aim of this work was to develop a mathematical modelling of Pb uptake in Brassica napus leaves. The measurements were carried out at the D09B-XRF Fluorescence beamline of the LNLS. The experiments were conducted in leaves of B. napus, grown in Pb soil at industrial and basal levels, in controlled environment, cultivated in greenhouses at CEPROCOR. We present a mathematical model that describes the process and coefficient of Pb diffusion from the midrib, and secondary veins towards the lamina of leaves removed from B. napus plants in different times of growth in contaminated soil. A numerical approach of the analytical solution is corroborated by Pb concentration measurements obtained from the XRF maps at the diffusion zone. This could allow the assessment of the main variables involved in Pb uptake by the plant.

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ORAL PRESENTATION

Bacterial mutualism as novel strategies for ROSs (Radical Oxygen Species) and copper homeostasis under extreme acidic and microaerophilic conditions

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ABSTRACT:

The Iberian Pyrite belt contains some of the most diverse and extreme acidic ecosystems. In order to study the biogeochemical processes supporting the biome under acidic and oxygen limiting conditions, a biofilm was extracted from two cores, sampled at an abandoned mine pithead. Analysis by SEM-EDS, XRD, XPS, µ-XRF, SXS and Py-GC/MS-CSIA revealed that the SO_x species present in the biofilm were originated from oxidative processes (High δ18O y δ34S values) and it was associated with Cu0. The copper interaction is interesting as it acts as cofactor for enzymes such as cytochrome oxidases or superoxide dismutases, because of its ability to cycle between Cu2+ and Cu+ [1]. One consideration is the accumulation of Cu+/2+ ions as it contributes to oxidative stress due its participation in Fenton-type reactions [2]. Next generation microbial diversity analysis showed the coexistence of oxidative and reductive microorganisms such as Alicyclobacillus sp. and Desulfosporosinus sp.. This special coexistence and data suggested novel strategies for ROSs (Radical Oxygen Species) and copper homeostasis. Hypothetically, the oxidation processes by Alicyclobacillus sp. generates ROSs that oxidize CuS precipitated by Desulfosporosinus sp. metabolism. This oxidation releases SOx and Cu0, which is accumulated in the biofilm. This process could regulate the ROSs in the system and could allow the accumulation of Cu0 as a neoformed mineral phase now possibly not toxic for the biofilm. This would be the first insight about a process that has been studied for decades and it had not been answered. Finally, this study proposes a new bio-metallurgic process, as well. Since, it gives a clue about the selective extraction and accumulation of native Cu from drainage of abandoned mines, which could have a large socio-economic impact.

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POSTER PRESENTATION

Canavalia bonariensis lectin: Molecular bases of glycoconjugates interaction and antiglioma potential

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ABSTRACT:

The recognition between proteins and carbohydrates is fundamental in many biological processes, such as viral, bacterial and parasitic infections, separation of cells and soluble components, fertilization, growth, differentiation and cancer metastasis. Modifications to the glycosylation patterns present in these affected cells are striking evidence of these pathologies and disease progression. Within this problematic the use of lectin can facilitate, therefore, the discovery of new biomarkers, considering that they are proteins able to bind in a specific and reversible way to carbohydrates. Among the plant lectins, the most extensively studied are the ones isolated from the leguminous family species, with emphasis in this work the lectin of Canavalia bonariensis (CaBo). Motivated by the biotechnological potential of these lectins, in the present work, we present the CaBo crystal structure determined to atomic resolution in the presence of α-methyl-d-mannoside, a specific ligand. Similar to the structural characteristics of other legume lectins, CaBo presented the jellyroll motif, a metal binding site occupied by calcium and manganese ions close to the carbohydrate-recognition domain (CRD). In vitro test of CaBo cytotoxicity against glioma cells demonstrated its ability to decrease the cellular viability and migration by induction of autophagy and cell death. Molecular docking simulations corroborate previous data indicating that the lectin's biological activities occur mostly through interactions with glycoproteins since the lectin interacted favorably with several N-glycans, especially those of the high-mannose type. Together, these results suggest that CaBo interacts with glycosylated cell targets and elicits a remarkable antiglioma activity.

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POSTER PRESENTATION

Characterization of Hydroxyapatite doped with Zn thin films by s-SNOM and FTIR/ATR spectroscopy.

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ABSTRACT:

Hydroxyapatite (HA, Ca10(PO4)6(OH)2) is the main inorganic constituent of bones and teeth. Synthetic HA has been used for bone regeneration, drug delivery system and thin coatings of metallic implants because its biocompatible and osteoconductive properties [1,2]. Zn substitution in hydroxyapatite (ZnHA) has been proposed to improve the biocompatibility and antibacterial properties of calcium phosphate bone grafts and coatings of metal implants. The characterization of the ZnHA surface properties such as molecular composition, stoichiometry and topography is the first step for the understanding of surface interactions with proteins, drugs and compounds of the biological medium. Nano-spectroscopy using infrared for scanning near-field from s-SNOM (scattering spectroscopy near-field optical microscopy) is a recent approach that begins to be used to characterize biomaterials at the nanoscale. Nano-IR combine atomic force microscopy (AFM) image with s-SNOM analysis in selected scanning regions up to 50nm. In this work, we use synchrotron infrared microscopy spectroscopy (SIMS) coupled to s-SNOM to characterize ZnHA thin films (2nm – 600nm) produced by radio frequency right angle magnetron sputtering (RAMS). The s-SNOM data was compared with those of conventional Fourier Transform infrared spectroscopy by attenuated total reflectance (FTIR/ATR). The ZnHA thin film has been deposited on 50 nm thick Au films which were previously deposited over Si(100). The s-SNOM measurements were performed in the IR-1 beam line at LNLS/Campinas. Deconvolution of nano-IR spectra did enable to characterize the transformation of amorphous calcium phosphate (ACP) phases into crystalline ZnHA. In addition, it was possible to identify non-apatite phosphate sites in the ealy stage of ZnHA film crystallization.

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POSTER PRESENTATION

Carbon supported Pd-Cu nanoalloys: in-situ XAS study under reducing and oxidizing atmospheres

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ABSTRACT:

The well-known research efforts to produce and characterize metal nanoalloys are due to their attractive size-induced properties, which are important for the development of new systems and their application fields [1]. The possibility to tailor their chemical and physical properties through the simple choice of the nanoalloy composition and atomic arrangement creates a quite exciting research field[1,2]. In this regard, compositional effects on the electronic and structural properties of Pd-based and Pt-based nanoalloys have been intensively studied [2]. In a recent study of carbon-supported Pd-Cu nanoparticles [3] we have demonstrated that alloying is able to prevent surface oxidation due to air exposure, and the higher the amount of Pd in the nanoalloy, less oxidized are both the Pd and the Cu atoms. To further deepen the understanding concerning these nanoalloys, and to provide tools to better design PdCu-based materials, this work presents new findings on the reactivity of the PdxCu1-x/C systems under both oxidizing and reducing atmospheres. Hence, three selected samples (Pd0.7Cu0.3/C, Pd0.5Cu0.5/C and Cu/C) were alternately exposed to CO and synthetic air flows under heating up to 450°C. The changes in the short-range order and in the chemical environment have been followed by in situ XAS measurements at the Cu-K edge (8979 eV). Additionally, valence band photoemission experiments shed some light on the correlation between the catalytic activities and density of occupied states near the Fermi edge. Valence band photoemission data and in situ XAS results showed that stronger oxygen-metal bonds are formed in the Cu-richer samples. A regeneration process assisted by the support was observed during the oxidation reaction, and its reduction efficiency was found to be dependent on the distribution of occupied electronic states near the Fermi level.

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POSTER PRESENTATION

Carbonates' precipitation by halophilic bacteria as a potential biosignature for the search for life on Mars

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ABSTRACT:

The mechanisms of Mg-carbonate precipitation, as dolomite, in the Earth's modern sedimentary environments has not yet been completely elucidated. However, it is known that the microbial activity is significant to facilitate or induce mineral formation. The organic EPS matrix secreted by microorganisms is very important to provide an ideal physicochemical environment to the mineral nucleation. Sediments and water samples were collected at Lagoa Vermelha, located in the municipality of Araruama, state of Rio de Janeiro, Brazil. This lagoon is characterized of being a hypersaline lake, where we can find a range of microorganisms known as halophilic and halotolerant extremophiles that have adaptation strategies to compensate for the harmful effects of high salinity, such as EPS biosynthesis, which is one of the most common protection mechanisms in bacteria, helping to maintain the integrity of their cells' membrane. These biominerals formed by microbial activity may be useful to test terrestrial and extraterrestrial habitats for life evidences; they are known as biosignatures. A biosignature is any material of biological origin that can be analyzed as evidence of past or present life. In the present project, the bacterial capability of bioprecipitation using EPS-secreting bacterial isolates from a hypersaline lagoon was investigated by combining microbiological, microscopic and geochemical analytical techniques. The isolates were evaluated for their ability to produce bioprecipitates using this multi-technique approach that includes Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray Spectroscopy (EDS), powder X-ray Diffraction (XRD) and Raman spectroscopy. It was possible to characterize the carbonates formed by the bacteria isolated from Lagoa Vermelha. Finally, these carbonates could represent a potential target for astrobiological studies of potential biosignatures for the search for life beyond Earth.

ORAL PRESENTATION

Cation inversion in bimagnetic CoO/Co1-xZnxFe2O4 core/shell nanoparticles: a XANES study

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ABSTRACT:

Aiming at controlling the exchange coupling at the AFM/FiM interface, we have fabricated a series of bimagnetic nanoparticles (NPs) by changing the shell composition. The system consists of 3 nm CoO NPs encapsulated in a 4 nm thick Co1-xZnxFe2O4 shell. A magnetic characterization shows that the coercive field decreases with Zn content while the exchange bias field presents a non-monotonous behavior, being maximum for intermediate concentrations. In order to elucidate the evolution of the magnetic properties with Zn incorporation, an investigation of the inversion degree of the spinel by XANES was performed. We have measured 5 different NPs and the bulk samples CoFe2O4 (CFO), ZnFe2O4 (ZFO) & Fe3O4 ([Fe]T[Fe]O2O4) at Fe, Co and Zn K-edges. In the region of pre-peak at Fe K-edge we have observed that their position and their shape do not vary in the NPs, the same behavior is presented by the sample CFO (partial inverted) and Fe3O4. This effect evidences some degree of inversion in the spinel structure. On the other hand, if we analyze the Co K-edge it is worth noting that the shape of the spectra of the NPs does not vary with the Zn content. This is a remarkable result because, a priori, the sample with 100% Zn only presents Co2+ from the CoO core. This is evidence that the Co of the core was partially dissolved and migrates to the shell producing a mixture of Co-Zn in the spinel with x=1. The analysis of the Fourier Transform brings information about the site occupancy of the different cations within the spinel structure (sites tetrahedral "T" or octahedral "O"). For the NPs and the bulk spinels both in Fe and Co K-edges data, peaks labeled as "O" and "T" are present. Besides, for Zn K-edge data only the "T" peaks are present in the NPs and ZFO spectra. Therefore, the absence of the "O" peak is a sufficient condition to exclude the occupancy of the octahedral site by the Zn atoms. These results reinforce the inversion between Fe and Co ions in the spinel (shell).

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POSTER PRESENTATION

Cerium oxide nanoparticles applied to the photocatalytic H_2 production

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ABSTRACT:

The photocatalytic water splitting reaction showed to be a promising process to obtain renewable and clean energy, but the efficiency reached in this process is still low. Considering this, the research on improving the efficiency of photocatalysts has attracted a strong interest on the past last years. Cerium oxide (CeO_{2-x}, $0 < x < 0.5$) is a material recently investigated as a possible photocatalyst to obtain H_2 from H_2O [1,2]. In a previous work [3], CeO_{2-x} nanoparticles were synthesized with high surface area ($96 < S_{BET} < 201$ m2/g) and pore volume ($28 < V < 132$ mm3/g) values and wide range of diameter ($2 < d < 90$ nm) and oxygen vacancies population – directly related to x ($0.05 < x < 0.46$) values. In this work, these CeO_{2-x} nanoparticles were applied to the photocatalytic H_2 production and presented activity of up to 10 times higher than the commercial CeO_2 standard. UV-Vis, Fourier Transform Infrared, X-Ray Diffraction, Ultraviolet Photoelectron Spectroscopy, X-Ray Photoelectron Spectroscopy and X-Ray Absorption Spectroscopy measurements were performed aiming to elucidate these results. It was obtained that the bandgap value depends on the nanoparticle synthesized and can be as low as 2.7 eV. The Ce 4f orbital occupation and the Debye-Waller factor of the Ce-O scattering is directly related with the bandgap values obtained. Moreover, the surface oxidation state plays a major role on the photocatalytic activity. Density Functional Theory (DFT) calculations were performed to obtain the relation between the band structure (DOS) and the x value in order to explain the dependence of the bandgap value with the Ce 4f orbital occupation. The results allowed shedding light on the improvement of the synthesis of CeO_{2-x} nanoparticles applied to the photocatalytic H_2 production by choosing the synthesis parameters.

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ORAL PRESENTATION

Characterization of CdWO4-polystyrene composite films using synchrotron light

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ABSTRACT:

Scintillator compounds are continuously investigated due to their potential applications in medical image detectors, dosimetry and industrial inspection. Besides the features of efficiency, decay time and chemical stability, it is also important to achieve low costs of production especially when large areas need to be covered [1,2]. Systems based on inorganic particles embedded in polymeric matrix receive great attention since they can combine the mechanical features of polymers with the optical properties of scintillators [3,4]. In the present work, composite films of CdWO4 (CWO) embedded in polystyrene (PS) have been prepared and characterized through microtomography and photoluminescence. CWO powder was synthesized via microwave-assisted hydrothermal method and the composite films were prepared by solvent casting procedure [5]. MicroCT images acquired at the IMX beamline were useful to investigate the particles distribution along the PS matrix. It was observed that the as-prepared scintillator is mainly deposited at the bottom of the film, whereas a uniform distribution was achieved by modification of the particle surface with stearic acid. The potential application of the CWO-PS screen was also tested at the IMX beamline, replacing the detector used in the facility and measuring a reference sample of bamboo toothpick. From the results, it can be concluded that the resolution of CWO-PS prototype is comparable to the commercial detector, so it can be used as radiation sensor screen. PL measurements were performed at the TGM beamline, showing the characteristic CWO emission in the blue region as well as PS luminescence due to organic impurities. Emission spectra were also acquired in an ISS spectrofluorimeter that uses a 300 W Xenon lamp as excitation source, in order to obtain a comparative study of the light yield of samples with different CWO/PS mass ratios. A preliminary set of results have already been published [5].

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POSTER PRESENTATION

Characterization of Compound 316L + 6% (Sr1.7Y0.3) CrReO6

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ABSTRACT:

The production of a composite that is resistant to corrosion and has a magnetic response may be of interest for use in structural devices, actuators and magnetic sensors that need to be immersed in extreme environments, such as: petrochemical, food and pharmaceutical industries, nuclear power plants, etc. it is intended to combine mechanical and chemical properties of the 316L steel with the magnetic properties of the perovskite sycrreo [1,2,3], to obtain controllable magnetic properties, in regards to the composition and working temperature range, for applications in chemically and thermally aggressive environments. the composite obtained might present chemical and mechanical resistance characteristics controlled by the percentage composition of each one. the composite formed from 316L steel with 6% double perovskite $(Sr_{1.7}Y_{0.3})CrReO_6$ (SYCRREO) was successfully sintered. Scanning electron microscope (sem) images showed that the nanoparticles of sycrreo were homogeneously deposited on the steel surface. The scattering spectra of the images confirmed the presence of the Sr and Re elements homogeneously distributed on the surface of the composite, which corroborates the images observed by sem. X-ray diffraction (XRD) measurements in the LNLS indicate the inclusion of the sycrreo particles in the composite. Microtomography (μCT) measurements indicated the dispersion of the particles in the composite, defects (cracks) and pores. Magnetization showed that the magnetic behavior of ferromagnetism of the nanostructured ceramics sycrreo remained unchanged (ferromagnetism up to 625 K). We can see that new composites can be elaborated from this idea for several other applications: biomaterials (biocompatible matrix), microheaters (oil tubes).

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POSTER PRESENTATION

Characterization of microstructures of special optical fibers used in electric field sensing by synchrotron x-ray phase-contrast microCT

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ABSTRACT:

X-ray micro computed tomography (micro-CT) can be used both as a qualitative and as a quantitative imaging technique. As a qualitative tool, it allows biologists and material scientists to section samples virtually in any direction, with submicrometer resolution, in their natural state and without elaborate. Synchrotron micro-CT (SR micro-CT) techniques offer significant advantages over micro-CT, such as a wide energy range, high flux and small source size and beam divergence [1]. Herein we carried out a 3D inspection of a microstructured optical fiber used in pulse selection systems [2], electric field sensors [3], phase modulators [4] among other utilities. These fibers, produced by ACREO, Stockholm, Sweden, have two holes that are nonsymmetrical in relation to its core. This lack of symmetry is fundamental in order to get a high nonlinear coefficient $\chi(2)$ through a technique called poling [5]. In this work, the segmentation of structures was performed, and dimensions and positions of each structure was obtained. The analysis slice per slice allowed us study distribution of fiber diameter and holes diameter, distances hole to hole and center of fiber to each role. Therefore, it was possible simulate and study the dynamic of creation of the second-order nonlinearity.

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POSTER PRESENTATION

Characterization of surface metal-organic frameworks (SURMOFS) films for applications in electrical devices

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ABSTRACT:

Surface Metal-Organic Frameworks (SURMOFs) is a new class of nanostructured materials based on the tridimensional networks of metallic clusters and organic ligands. These structures have important characteristics such as crystalline orientation, high porosity and superficial area [1]. The SURMOFs can directly be grown on a support surface functionalized with self-assembled monolayers (SAMs) [1]. The possibility of incorporating this supported film onto devices and sensor platforms leads to the studies of film's morphology, crystallinity and interface quality. Previous studies have investigated the nucleation and film growth of the first layers [2,3] and defined the preferential crystallographic orientation of SURMOF selecting the SAMs end-group [3]. However, to the best of our knowledge, no study focusing on the SAMs chain length dependence of the growth properties has been performed. In this sense, we evaluated the influence of SAMs chain length on the preferred crystalline orientation of SURMOF HKUST-1. The results show a preferential growth in [100] direction when the SAM chain length increases. The study was carried out considering a conductor and an insulator surface (Au and Al2O3, respectively), in order to evaluate their influence on the nucleation and growth of HKUST-1. These results are crucial when SURMOFs are applied to electrical devices, associating the characteristics of the films with their electrical properties, considering both solid/solid and solid/liquid interface.

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POSTER PRESENTATION

Chitosan and silica as fertilizer nanocarriers in sugarcane micropropagation: characterization and implications

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ABSTRACT:

Nanomaterials research for agriculture are motivated by the expected reduction of nutrients use and the possibility to obtain a smart delivery system.[1,2] In our current project, N, P and K have been incorporated into chitosan and silica nanoparticles as strategy to maximize their uptake in sugarcane plants. Different nutrient concentrations were added to the nanoparticles and we compare their morphological and structural differences and how their properties reflect on physiological processes of plants. The average size of chitosan nanofertilzers were 150–697 nm while the silica ones were 106–400 nm, larger than the ones without nutrients, 50 and 63 nm, respectively, Fig. 1A. K and P on nanoparticles were confirmed by EDS and FAAS. The K amount was in a range of 0.15–0.28% for silicas and 0.11–0.36% for chitosan. The diffraction peaks of KH2PO4 and Na5P3O10 were observed in diffractograms. SAXS results showed the nanofertilizers structures depended on the nature of the support and on the nutrients amount. N, K and P incorporation yeld smaller silica than chitosan-based nanofertilizers, revealed by the shoulder at Guinier region. Fig. 1B and D show the morphology and size (18-21 µm x 1.4 µm) of adaxial sugarcane stomata, which allow nutrients uptake. Currently, the nanofertilizers are being tested on micropropagation in vitro of sugarcane seedlings in order to reach higher multiplication rates and to assure phytosanitary quality in large scale. The microstructural characteristics of plants from acclimatized seedlings in vivo will be compared by IMX (20180098) with micropropagation in vitro. Fig.1D shows in vitro inoculation of sugarcane seedlings using nanomaterials into culture medium. The results showed that the amount of nutrient loaded in the nanoparticles matrix is dependent on several factors including size, surface charge, and nature of the nanoparticle, in agreement with the fractal structure and radius of the secondary particles of nanofertilzers by SAXS.

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POSTER PRESENTATION

Complex block copolymer nanostructures characterization using coherent X-ray beam

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ABSTRACT:

Complex structures mimicking biological systems are being developed in our lab mainly for separation purposes and high efficiency drug and protein encapsulation. These systems are nanoporous microparticles with internal bicontinuous nanochannel structure [1], hierarchical scaffolds formed by microcompartments interconnected by hexagonally ordered nanochannels [2], asymmetric membranes with nanoporous ordered top layer and ordered nanophase separated block copolymers with different chemistries and morphologies [3]. Their morphological characterization has been based so far on electron microscopy and small angle x-ray scattering. The new techniques available at Sirius will allow us to deliver in-depth information on the formation mechanism of the structures, some of them with continuous interconnected nanochannels, that mimic the lipid cubic membranes in cellular organelles. The developed particles are formed only in specific solvent/non-solvent solution, which favors the block copolymer self-assembly into triple periodic continuous structures with minimum surface free energy. The coherent flux at the Cateretê beamline will make possible the use of X-ray Photon Correlation Spectroscopy to measure the dynamics of polymer solutions, by means of time correlation functions. As suggested by microscopy and light scattering, a nucleation and growth mechanism with an initial fast stage, followed by slower rate kinetics lead to the particle development. The brighter beam will provide the needed temporal resolution to study in-situ the fast initial stage of the formation mechanism, while, for the slower stage of particle formation, information about the interface evolution will be obtained. We plan to use the nano-focused and coherent beam to perform Coherent X-ray Diffraction Imaging for the tomographic reconstruction of the nanostructured materials, providing a much more comprehensive characterization than achieved by electron microscopy 3D image reconstruction methods.

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POSTER PRESENTATION

Considerations on the thermoplastic starch applied in the design of delivery devices for aerial seeding

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ABSTRACT:

Cassava starch mixed with different contents of glycerol and urea was processed by extrusion and compression molding. To characterize the nanomorphology SAXS experiments were performed on the beam line at the National Synchrotron Light Laboratory. During the measurements, two different sample-detector distances were used: 3022 mm and 889.6 mm. The starch A-type crystal typical scattering peak was observed at 0.69-0.73 nm-1, indicating a long period distance of 9 nm. Furthermore, at Lorentz corrected curve, the typical reflection of lamellar structures, a second order reflection peak, was noticed for the SGU-10 formulation. For the urea contain curves, SAXS curves presented a broader peak around 3.8 nm-1 which might be ascribed to B-type crystal. However, as the peak intensity seems to be correlated to the urea content, the peak was attributed to the urea crystals. After compression, the Lorentz correction exhibit two extra peaks at 0.07 nm-1 and 0.30 nm-1, implying the distortion of the A-type crystals layers, indicating the transitions/transformation from smectic phase of amylopectin helices (plasticized phase) to a nematic phase (glassy phase). Furthermore, the SAXS data were employed to analyze the fractal structure of obtained materials, two length scale were investigated. At first, at low q values, the fractal nature of larger objects that may contain amylose molecules at both the crystalline and amorphous phase are surface fractal compact structures with smooth surfaces. The cluster radius of gyration is, approximately, 51 nm. Finally, at high q values, the fractal nature of the basic blocs, the double helices chains of amylopectin changed with material processing. Before compression, the basic blocks are mass fractal structures, nearly, a cylindrical low density object. After compression, the original double helices chains are distorted by the new cycle of temperature and pressure.

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POSTER PRESENTATION

Copper local structure in spinel ferrites determined by X-ray absorption and Mössbauer spectroscopy and their catalytic performance

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ABSTRACT:

Magnetite and copper spinel ferrites were synthetized by co-precipitation method and characterized by X-ray powder diffraction, 57Fe Mössbauer spectroscopy, XAS and scanning electron microscopy. According to M. F. F. Lelis et al [1] the incorporation of Co in the spinel lattice can significantly modify the redox properties of ferrites and influence their chemical stability. For the obtained samples, XRD data showed the formation of cubic structure with particle sizes ranging from 30 to 23 nm and according to 57Fe Mössbauer data magnetite as well as copper ferrites were partially oxidized. In non-stoichiometric or partially oxidized magnetite, which is defined as a solid solution of magnetite (Fe3O4) and maghemite (γ-Fe2O3), Fe2+/Fe3+ ratio decreases ($x < 0.50$) [2]. EXAFS data showed that for copper ferrites the increase of Cu2+ content induced a change on its location in the spinel structure from octahedral to the tetrahedral coordination sites. The occupancy of Cu2+ changes from tetrahedral to octahedral sites with the increase of copper content, as observed by the analysis of 57Fe hyperfine parameters and XAS data. Catalytic behavior of the obtained samples was investigated towards methylene blue dye degradation using photo-Fenton reactions. For copper ferrites the photocatalytic efficiency increased from 57% to 94% with the increase of copper content. The obtained results also showed that the presence of copper in the spinel ferrites led to an increase of catalytic efficiency compared to pure magnetite, and it is attributed to the formation of active species when Cu2+ is oxidized to Cu+3 by H2O2, as was observed for Fe3-xMxO4 nanoparticles, where M= Mn, Co or Ni, in the presence of H2O2 [3].

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POSTER PRESENTATION

Designing a SAXS methodology to determine the up-take of magnetic nanoparticles in cell cultures

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ABSTRACT:

The incorporation of magnetic nanoparticles (MNP) in cells is a topic of increasing interest due to its application in medical theranostics. Examples among others are tissue engineering, cell therapy and magnetic hyperthermia (MH) allowing a combination with magnetic imaging and cell tacking [1]. In-vitro experiments have demonstrated that MNP are mostly located inside cells vesicles called endosomes. Regarding MH, how MNP structuring affects heating efficiency has been studied in ferrofluids[2] and in cell cultures [3]. Nevertheless, how these MNP are arranged inside cells and whether is possible to quantify the up-take using SAXS are open questions. In this work, we show how to use SAXS to quantify the cellular up-take and to analyze the MNP structuring in cells. To this end, SAXS data was acquired in MNP aqueous suspensions and in B16 cell line labelled with MNP. In order to analyze size and coating effects in MNP structuring inside endosomes, iron oxide core MNP, and coated with two different surfactants (DMSA and citric acid), a core/shell structure composed by iron/iron oxide coated with sodium citrate, were used. Subsequently, the up-take was determined as follows. SAXS patterns from colloid were measured at dilutions from 0.01 mgFe/ml to nominal concentration. All these patterns were well fitted using the spherical form factor and a mass-fractal structure factor following the monodisperse approximation. The spherical form factor contribution was used to extrapolate the $d\Sigma/d\Omega$ value at $q = 0$ ($d\Sigma(0)/d\Omega$). A linear approximation between colloid iron concentration ([x]) and $d\Sigma(0)/d\Omega$ was determined and used to find the iron concentration in cell cultures ([x]in cell). Finally, the up-take was calculated by dividing [x]in cell with the cell volumetric concentration (N). Results showed that up-take is around 23 and 54 pgFe/cell for the used cells suspensions, these values are in agreement with previous reported values obtained using dc magnetometry [3].

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POSTER PRESENTATION

Development of new in-operando characterization instrumentation: correlation of (Cd,Gd)O_2 IT-SOFC electrolyte high-temperature properties through XRD/XAS coupled to Electrical/Electrochemical simultaneous measurements

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ABSTRACT:

The development of a device that enables the characterization of several properties simultaneously under controlled atmospheres in a wide range of temperatures represents an advantage over the traditional independent measurements since those properties could be strongly correlated. At the previous RAU, we presented the advances made in a currently ongoing Long Term Proposal carried out at XPD and XAFS2 beamlines involving the design and implementation of a new high temperature cell that allows performing in-situ or in-operando X-ray Diffraction (XRD) or X-ray Absorption Spectroscopy (XAS) simultaneously with electrochemical characterization at a wide range of temperatures and atmospheres [1]. In this work we present the first results obtained with this device from a scientifically relevant sample: we performed XAS/XRD and electrical conductivity simultaneous measurements on dense pellets of gadolinium-doped ceria (GDC), one of the most promising electrolytes for intermediate SOFC. The case for enhanced ionic conduction in nanocrystalline oxides remains unresolved due to conflicting reports and inadequate efforts to isolate the ionic from the total conductivity [2]. Although GDC materials possess high ionic conductivity over 500–700°C, they also may exhibit mixed electronic-ionic conductivity under reducing atmospheres, which may be detrimental to the fuel cell operation. Besides, direct carbon fuel cells are an attractive alternative for conventional power generation; however, there is almost no information on the stability of conventional fuel cell materials in direct carbon fuel cell environments, in particular when exposed to realistic fuels [3]. The correlation between changes in the GDC electronic and crystallographic structures by following the Ce L3-edge energy and lattice parameters under different temperature/atmospheres conditions with the macroscopic electrical conductivity properties of this material is here presented.

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POSTER PRESENTATION

Discriminating oxides from L-line resonant inelastic x-ray scattering spectra

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ABSTRACT:

The oxidation state information of a specific element in a sample has been obtained by means of Resonant Inelastic X-Ray Scattering (RIXS) (also known as Resonant Raman Scattering) using a variety of experimental conditions, as total reflection, grazing incidence and even confocal setups [1-3]. The discrimination methodology that we have proposed for this purpose has been applied only to the study of the information given by the resonance involving the K shell of the atom (mainly transitions from L to K shell). Because of this, there is a lack in knowledge about the sensitivity of the method for the discrimination of compounds by the analysis of the L lines (M to the L atomic shells). In this work, we present the first results of speciation of rare earth compounds using this tool on L-line resonance spectra. The measurements were performed at the Brazilian Synchrotron Light Source in the D09B-XRF beamline, using the typical 45°-45° geometry, and an energy dispersive system. The incident beam energies were selected just beneath the different L absorption edges of the studied elements as to achieve the resonance condition. Spectra were analyzed by two independent mathematical methods: a fine structure deconvolution and a multivariate analysis approach. The results show a clear discrimination of the different element compounds. The outcome is significant since the proposed tool successfully allowed to identify different species of the studied elements by means of the inspection of the L line resonance. Comparisons with the characterization potentiality of the K line analysis by RIXS are also discussed.

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POSTER PRESENTATION

Dual temperature meter of 0,05 K accuracy and its calibration system

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ABSTRACT:

We report the construction of a dual temperature measurement system with accuracy better than 0.05 K at 273 K composed of a hybrid circuit of a 24-bit ADC converter, current source, programmable gain instrumentation amplifier and a multiplex (ADS1248, TI – Texas Instruments), which allows us to connect two PT100 class AA sensors. The sharing of front end and processing electronics allows, after a calibration procedure, a pair of practically identical thermometers. The thermometers are specified from 273 k to 373 K, however, it can be easily extended beyond these limits by the appropriate selection of the sensors. The system communicates by SPI interface and can be read easily by a microcontroller, for example what makes up the Arduino platform. To obtain the accuracy of 0.05K, a temperature calibration system with a stability better than 0.01K over 30 seconds, time required for the measurement of the temperatures of the reference sensor and the sensor to be calibrated, was built. The system consists of a dual wall thermal chamber, a PT100 class AAA sensor (0.03 K @ 273 K accuracy, Omega) which resistance is read by an HP3457A multimeter. The thermal chamber is powered by a HP6624A high stability power source and the external temperature monitored by a PT100 and HP3478A multimeter. The entire calibration system is controlled by software written in the Python language through a GPIB-RS232 interface (NI-National Instruments), connected to the RS232 port of a computer. In the calibration of the ADS1248 system, it is connected to an Arduino board and this to the computer via USB port and temperature control through the RS232 port, both controlled by the code written in Python.

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POSTER PRESENTATION

Effect of oxidation process of asphaltenes present in oil refining wastes followed by XANES spectroscopies

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ABSTRACT:

Asphaltenes are complex mixture of heteroatom rich aromatic hydrocarbons that are present in crude oils. Sulphur is the most abundant heteroatom in the oil, present in the heavier fractions as asphaltenes. Besides sulfur is a good marker of the alteration of oils because it is very sensitive to oxidation.[1] The XANES spectroscopy is an appropriated technique for the speciation of chemical species and provide qualitative and quantitative information about the sulphur content in asphaltenes. [2] A large fraction of oil refining wastes contains high molecular weight and heteroatom rich hydrocarbons. The general aim of this project is the remediation of oil bottom tank sludge combining chemical oxidation followed by biological treatment. The knowledge of the sulphur chemistry in asphaltenes extracted from oils bottom tank sludge before and after different oxidation treatments constitutes an important information for understanding such a complex system. Three samples of asphaltenes were extracted from different oils bottom tank sludge by precipitation with n-heptane. The asphaltenes were further treated with different oxidizing agents (persulfate, hydrogen peroxide and permanganate) followed by biological treatment with previously studied fungal species. The Sulphur speciation at different stays of the sample treatment were studied with XANES spectroscopy in the SXS beamline at LNLS. The spectra of asphaltenes can be describe in terms of the contribution of four principal sulfur structures: sulfide, thiophene, sulfoxide and sulfonate. After oxidation treatments an increase in the contribution of more oxidated Sulphur form was observed, attributed to sulfate or sulfonate group. The completed oxidation of Sulphur observed with persulfate and the evaluation of total petroleum hydrocarbons in the petrochemical sludge samples after biological treatments allow select the oxidation agents to optimize the remediation treatments.

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POSTER PRESENTATION

Elemental and molecular investigation of anatase (TiO_2) at the brazilian synchrotron light laboratory – defining a possible biosignature

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ABSTRACT:

The study and the recognition of biosignatures are of extreme importance for research in Paleontology and Astrobiology. Both areas converge on finding evidence and establishing criteria for traces of life, whether on Earth and/or in other parts of the cosmos. Especially, the recognition of biosignatures in the geological record may directly influence in the creation and improvement of biogenicity parameters. In this context, the use of advanced techniques may enable the detection of such signals, allowing better comprehension of geochemistry evidences of life and/or biological processes. Here we tested the use of Synchrotron Radiation micro-X-ray Fluorescence (SR-µXRF) and micro-Raman Spectroscopy (µRS) for the investigation of biosignatures in 3D fossilized microbial mats from the Itajaí Basin (Ediacaran, Brazil). Among our SR-µXRF data, we have detected higher intensities of titanium at the fossils in contrast to the rock matrix, and Raman spectra confirmed these elemental results. Both point and mapping micro-Raman analysis revealed the presence of characteristic anatase bands in higher abundance at the fossil mats in comparison with the surrounding rock matrix. The association of anatase with microbial mats structures suggest a biological influence in the origin of this mineral for the Itajaí Basin material. If confirmed by other techniques, such as Scanning Electron Microscopy (SEM) and Ion Microprobe, the results could have great implications for establishment of anatase as one important type of biosignature for paleobiological and astrobiological research. However, further analyses are needed to refute other (abiotic) possibilities, such as sedimentary and/or diagenetic processes.

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POSTER PRESENTATION

Elucidating the cathode chemical process of lithium ion batteries using in-situ XASF electrochemical cell

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ABSTRACT:

To improve the performance and efficiency of Li based batteries is indispensable to have a clear comprehension of the processes that occurs during the use of these systems. But sometimes, these physicochemical processes cannot be detected by electrochemical measurements, and in the case of lithium ion batteries, the possibility to make electrochemical experiments in a classical three electrodes open cell joined to other chemical or physical techniques is very hard. For this reason, we develop an in-situ lithium ion battery cell system capable to work in typical current/potentials experiments for these kinds of batteries and at the same time allow performing X-Ray absorption experiments over the active materials. Mn spinels (used as cathode) were synthetized by the traditional method of solid phase calcination, starting from the stoichiometric mixture of Li and Mn carbonates and Ni oxide in atmosphere of O2 at 820 ºC for 12 hours. The samples were grounded and conditioned for carry forward the electrochemical experiments. In the recent literature In situ x-ray absorption demonstrate to be a very useful elucidating the structural and electronic changes that follows lithiation/delithiation process [1]. In this work we highlight some of our recent results using in situ x-ray absorption spectroscopy obtained at the DAXS beamline. XAS measurements were done in situ at Ni-K edge and at Mn-K edge for comparative purposes in order to intent to determine the role of the Ni inclusion in the lithiation/delithiation process. In both experiments, at Ni and Mn K edges, XANES demonstrate to be sensitive to the Li intercalation. The comparison of spectra obtained for samples with and without Ni shows a higher variation on the Mn K edge in the samples without Ni. The biggest change during the discharge is observed on the Ni K edge and is associated to the variation on the oxidation state and coordination number of the Ni atoms during Li intercalation.

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POSTER PRESENTATION

Dynamic study of crystalline structures in stainless steel TIG welded using gleeble simulator and resonant spreading

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ABSTRACT:

The properties of duplex stainless steel UNS S32304 have been investigated since 2012 as a candidate for the construction of a receptacle for the transport of radioactive material associated with the future Brazilian Multipurpose Reactor (RMB). The focus of this work is to evaluate the effect of different heat treatment temperatures on the physical properties of UNS S32304 sheet with a thickness of 1.8 mm after autogenously TIG welding process. Pulsed current was used with direct polarity, protection gas with a mixture of argon and 2% of nitrogen and an automatic drive system with digital control of the parameters. The welded and as received specimens were heat treated for 8h at 475° C, 600° C and 750° C in a programmable muffle furnace (with accuracy of ± 5 ° C) with subsequent air cooling. These samples were submitted to tensile tests in the thermo mechanical simulator Gleeble® 3800 (LNNano - CNPEM) and Gleeble® 3500 coupled to X-ray diffraction with synchronous light (LNLS - CNPEM) with a strain rate of 10-4 s-1. It was verified that only the sample treated at 600°C showed a tension-strain behavior similar to the sample as received. The treatment at 600°C in the welded sample indicated to be the most adequate, presenting tensile strength limit and elongation superior to the welded sample without heat treatment.

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POSTER PRESENTATION

Evaluation of structural alterations in hair subjected to cosmetic treatments

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ABSTRACT:

Human hair contains a helical fraction that comprises a crystalline phase (α-keratin) embedded in amorphous matrix [1]. It is well known that cosmetic treatments, bleaching, straightening and/or dyeing, cause changes in the hair structure, more specifically in the α-keratin [2,3]. The literature relates the use the small angle X-ray (SAXS), small angle neutron scattering (SANS) and differential scanning calorimetry (DSC) measurements to evaluate the changes in the hair structure [3,4,5,6]. In this context, the aim of this study was to investigate the structural alterations caused by bleaching in the human hair. To this end, we have applied SAXS, quasi-elastic neutron scattering (QENS) and DSC to get insight on possible changes in the crystalline α-keratin of the hair as well as on the hydrogen mobility. The DSC data showed the bleaching caused a decrease of the endothermic peak area, related to the melting of the crystalline fraction of hair. SAXS diffractograms taken in bleached hair showed two equatorial reflections located at about 90 Å and 45 Å stronger than virgin hair (control sample). Kreplak et al., 2002 [7] described this phenomenon as being an alteration in the crystalline lateral organization and crystallized lipids in the structure of the hair, respectively. To get further insight in differences in hydrogen mobility caused by structural changes, QENS experiments were performed. Preliminary results showed that the hydrogenous molecules (water, protein, lipids) are either protected by the amorphous matrix filaments or still bound to the virgin hair structure. For bleached hair, the hydrogen molecules are freer to move when compared to the control sample. These results might indicate that bleaching makes the hair more porous, due to the degradation of the melanin, which could explain the changes in the hydrogen mobility. Therefore, these techniques allowed evaluating modifications in the hair samples subjected to bleaching treatments.

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Poster Presentation

Evaluation of the ZnO and ZnO:Ag for the photodegradation of Rhodamine B

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ABSTRACT:

Dyes are organic pollutant molecules and their release as wastewater in ecosystems is a source of aesthetic pollution, which causes disturbance in the aquatic life. The vast majority of dyes are resistant to biodegradation and the direct photolysis; and many nitrogenous dyes such as Rhodamine B (RB) undergo a natural anaerobic degradation that produces potentially carcinogenic aromatic amines. RB can be degraded by irradiation with UV light in the presence of a semiconductor photocatalyst such as ZnO, which has a theoretical direct band gap of 3.3 eV. In this work photocatalysts based on ZnO and ZnO-Ag0 nanoparticles were evaluated for the photocatalytic decomposition of RB. The samples were characterized by X-ray diffraction (XRD), SEM (Scanning Electron Microscopy), TEM (Transmission Electron Microscopy) and XPS (X-ray Induced Photoelectron Spectroscopy). The synthesized ZnO has a hexagonal crystalline structure with a band-gap value (Egap) of 3.2 ± 0.06 eV. A large change in the absorption spectrum of ZnO was observed after the incorporation of the Ag0 nanoparticles, which caused a change in the color of the samples ranging from light yellow to dark brown as a function of the silver content. The XPS results indicated the formation of Ag0 nanoparticles on the ZnO surface. The correlation with the photocatalytic activity will be presented.

POSTER PRESENTATION

Examining Cr3+ and Cr6+ local environment and solubility in xCr2O3−(1-x)PbO−SiO2 glasses by spectroscopic techniques: Raman Scattering, UV- Vis and XANES

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ABSTRACT:

Cr addition in lead containing glasses have been investigated, among other things, in order to improve their potential as gamma radiation shielding material. However, not much is known about the valence, structure and solubility of Cr ions in these materials. Thus, in this study we use Raman scattering, UV-Vis spectroscopy and XANES in order to explore the valence state, structure and solubility of Cr ions in PbO•SiO3 (PS) glass. The Raman spectra of the Cr doped glasses show additional bands that are related to the formation of CrO4- (chromate) units due to the presence of Cr6+ ions, similarly arranged as in PbCrO4. The dependence the of the CrO4- stretching vibrations intensity with chromium concentration is used to determine the Cr6+ solubility limit, that is achieved in the sample with 1.05 Cr2O3 mol.% . UV-Vis absorption demonstrates that Cr6+ ions incorporation alters the band gap that becomes narrower as Cr concentration increases. Additionally, characteristic Cr3+ transitions are observed, indicating that not all Cr ions are present in 6+ valence state. However, from the XANES spectra no Cr+3 could be identified in the glasses bellow the solubility limit, which suggest that the amount of Cr3+ present in these samples is very small compared to the amount of Cr6+. Additionally, no dependence of the Cr6+/Cr3+ ratio with Cr concentration is observed for those samples. An attempt to increase the solubility limit, using the same synthesis condition, resulted in Cr2O3 phase segregation detected by Raman spectroscopy. Using XANES technique we estimate that the Cr6+/Cr3+ ratio in the phase segregated sample is 46% lower than in the samples containing until 1.05 mol.% of Cr2O3. Once the Cr ions are incorporated by the lead environment, we suggest that increasing the amount of lead in the glass composition the solubility limit could be increased, which has an important consequence in the use of these glasses in gamma ray protection.

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POSTER PRESENTATION

Formation of molecules in condensed chlorobenzene by X-rays interactions

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ABSTRACT:

The precursor molecule of polycyclic aromatic hydrocarbons (PAHs), benzene (C6H6), and their derivatives in which a hydrogen atom is replaced by a functional group, nitriles (-CN), benzonitrile (C6H5CN) and by a chlorine atom (-Cl), chlorobenzene (C6H5Cl), have been detected respectively in the pre-planetary nebula CRL 618, in the molecular cloud TCM-1, in meteorites samples and in Mars craters. The search for chlorinated compounds has been highly emphasized. Recently, the detection of chloromethane (CH3Cl) was reported in the coma of comet 67P/Churyumov-Gerasimenko and in the gas surrounding the low-mass protostar IRAS 16293-2422. These molecules are formed and destroyed in different astronomical environments through chemical reactions in the gas phase and on the frozen surface of interstellar dust under action of incidence of photons (UV and X-rays) and charged particles (electrons and ions). In this experimental work, we studied the chemical modification induced by X-rays photons (1-5 KeV) from the SXS beam line at the Brazilian Synchrotron Light Laboratory (LNLS), in condensed chlorobenzene on a substrate. After each exposure time an absorption spectrum at the Cl K-edge (2822 eV) was taken using near-edge X-ray absorption spectroscopy (NEXAFS). The experimental set up consists in a high vacuum chamber (P < 10-8 Torr) where the sample of chlorobenzene was kept at low temperatures (100 K), simulating the conditions of interstellar and circumstellar environments. The NEXAFS spectra showed that the photodegradation of the sample is due to the decrease of the transition from the Cl 1s to π*(C-Cl) orbital and the increase of the transition Cl 1s to σ*(C-Cl) as a function of the exposure time. These results suggest that the presence of the chlorine atom favors the reaction of association of benzene rings that may form new PAHs and chlorinated PAHs on the frozen dust grain surface.

ORAL PRESENTATION

In situ characterization of CuxNi1-x/CeO2 ($0 < x < 1$) nanoparticles applied to the RWGS reaction

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ABSTRACT:

The increasing emission of the CO2 molecule in the atmosphere, enhancing the greenhouse effect, attracts scientist's attention to study its dissociation reactions, like the Reverse Water Gas-Shift (RWGS) reaction [1]. The use of catalysts can be impaired by alterations on its properties due to atomic events, like the Strong Metal-Support Interaction (SMSI) effect and atomic arrangement induced by the exposition to high temperature gaseous treatments [2]. In this work, CuxNi1-x/CeO2 ($0 < x < 1$) nanoparticles were applied to the RWGS reaction while the atomic events were elucidated in situ. The nanoparticles were synthesized in accordance to [3] and Transmission Electron Microscopy (TEM) measurements showed a mean diameter of (4 ± 1) nm. After, the nanoparticles were heated to 500 ºC in a H2 atmosphere. At the end of this activation process, the nanoparticles were exposed to the RWGS reaction at 500 ºC. The nanoparticles were characterized in situ by measurements in Synchrotron Labs such as Near Ambient Pressure X-Ray Photoelectron Spectroscopy (NAP-XPS), X-Ray Absorption Spectroscopy (XAS) (at the Cu K edge in transmission mode) and time-resolved XAS measurements (at the Cu K edge and Ce L3 edge in transmission mode). The NAP-XPS measurements were performed with 2 different incoming photon energies, 1250 eV and 2000 eV. The results point to a Cu (Ni) enrichment at the nanoparticles surface when exposed to a H2 (H2 + CO2) atmosphere. Moreover, there is a threshold on the Cu concentration for the occurrence of the SMSI effect. The SMSI effect influences negatively on the reactivity of the nanoparticles in the RWGS reaction since the best performance was found for SMSI-free nanoparticles. Finally, the precise and detailed nature (structural and electronic characteristics) of the SMSI effect was elucidated for the first time.

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ORAL PRESENTATION

In situ Small Angle X-ray Scattering monitoring of traditional FAU zeolite synthesis.

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ABSTRACT:

Small Angle X-ray Scattering (SAXS) is a powerful technique to study in situ the formation of zeolitic materials [1, 2]. It is crucial to unravel the nucleation to optimise the synthetic methodologies, to decrease substantially the industrial synthesis costs. FAU zeolite is an aluminosilicate of high industrial interest due to its variety of applications, such as catalysis, photovoltaic solar cells and drug delivery [3]. Surprisingly, very few studies have been carried out on the nucleation and growth of this topology [4, 5]. To date only ex situ studies were performed of the traditional hydrothermal FAU formation [6], as it is difficult to replicate these conditions while performing X-ray measurements. To this end, we developed a hydrothermal cell and followed in situ the formation of the FAU zeolite in the temperature range 100-110°C and captured its nucleation, growth and crystallization processes. In the presentation the results of these measurements performed in the in situ reactor developed at LNLS/SAXS1 beamline will be shown, allowing the observation of different nanoparticles sizes and shapes during the nucleation, opening the possibility of performing in situ under hydrothermal conditions nucleation experiments at the synchrotron.

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POSTER PRESENTATION

In situ synchrotron radiation evaluation of hydrogen embrittlement and cryogenic deformation in duplex stainless steels

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ABSTRACT:

In situ synchrotron radiation measurements of axial strain were made in duplex stainless steels (DSS), in order to evaluate the hydrogen embrittlement of DSS 2205 and dynamic recovery of DSS 2507 during cryogenic straining. For DSS 2205, this provides a direct comparison between the kinetics of the main hardening mechanisms (martensitic transformation and dislocation density) as a function of strain for DSS under hydrogen effects. The hydrogen charging reduces the stress relaxation, reducing the ductility and suppressing the martensitic transformation. In addition, it also reduces the strain hardening (dislocation multiplication) in austenite. The influence of the martensitic transformation on failure process seems to be lower than that of the dislocation density. For DSS 2507, the microstructure evolution as a function of strain is reported for both cryogenic and room temperatures. Dynamic recovery suppression seems to be easier at cryogenic temperatures, leading to the enhancement of the steel strength.

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POSTER PRESENTATION

In Situ XAS study of Pr0.6Sr0.4CoO3-d for IT-SOFC cathode application

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ABSTRACT:

Cobaltites are high-temperature mixed ionic and electronic conductors that have been studied for long time as possible cathode materials for solid oxide fuel cell (SOFC) application. The high working temperature (900-1000ºC) of SOFCs requires expensive materials as current collectors and interconnectors and reduces the life span of the device. So, it is necessary to reduce the working temperature to intermediate temperatures in the range of 500-700 ºC (IT-SOFC). In this work, we present an in situ XAS study on Pr0.6Sr0.4CoO3-d (PSC) nanopowders (average crystallite size, D≈30 nm), focused on the speciation of Co atoms under different temperatures and O2(g) concentration in view of their possible application as cathode in IT-SOFCs. PSC was synthesized via the gel-combustion method. In situ XAS studies were performed at the D04B-XAFS-1 beamline of the LNLS. The Co K- Sr K- and Pr L3-edges were measured in transmission mode, from 20 to 700 ºC in controlled atmosphere with different O2(g) concentrations. In situ XPD analysis was performed at the D10B-XPD beamline of the LNLS, under identical experimental conditions. PSC exhibits the orthorhombic phase in the whole temperature range with a possible secondary phase. Pr atoms are in +3 oxidation state, independently of the temperature or O2(g) concentration. The relative amount of cobalt's +2, +3 and +4 oxidation states varied according to temperature and O2 content but cobalt's average oxidation state was invariant, close to +3.2. For comparison, PSC micropowders (D>300 nm) exhibited almost the same average oxidation state as PSC nanopowders, but the relative amounts of Co species were invariant under the different conditions and the Co2+ and Co4+ content was lower. This suggests that the electronic compensation reaction would play an important role in the electrochemical properties of PSC materials and would explain partially the enhanced electrochemical performance of PSC nanopowders when used as IT-SOFC cathode.

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POSTER PRESENTATION

In situ XRD studies of stability of crystalline structure of Ce-Zr-Sc mixed oxides

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ABSTRACT:

Solid Oxide Fuel Cells stand as a promising technology for efficient and sustainable energy production. Anode material is subjected to high temperatures during the construction process and the operation of the SOFC1. Therefore, it is important to study the impact of temperature on material stability. The aim of this work is to study the effect of temperature in composition homogeneity, secondary phase segregation and crystallite size in CeO2-ZrO2-Sc2O3 mixed oxides. The addition of Sc2O3 to CeO2-ZrO2 system is expected to improve ionic conductivity through aliovalent doping of the mixed oxide lattice and, as a consequence, improve overall anode performance.2 Samples were synthesized using two different routes, namely glycine/nitrate combustion route (GC) and citrate complexation route (CIT), preparing the following nominal compositions: Ce0,9ScxZr0,1-xOδ, with $0 \leq x \leq 0.1$. The crystal structure of the samples prepared was studied by means of in situ X-ray powder diffraction (XPD) experiments performed at the D10B-XPD beamline of the LNLS. Samples were studied using a high intensity and low resolution configuration without analyzer crystal and a Mythen 1K detector. Samples were heated from ambient temperature to 1000°C in air flux to study structural evolution. In addition they were subjected to CH4/O2 atmospheres to study structure stability in complete methane combustion reaction conditions. In situ XPD experiments allowed us to confirm the successful incorporation of Sc2O3 into the CeO2-ZrO2 mixed oxide prepared both by citrate and glycine/nitrate routes and fired at 500°C. The samples exhibited a cubic fluorite type structure characteristic of CeO2. Crystallite size growth kinetics was similar for all prepared samples, however, ternary samples exhibited lower values of crystallite size in the whole temperature range, reaching values of ca. 60 nm at 1000 °C. Catalytic experiments indicated structural stability in reaction conditions with no formation of graphitic carbon doing complete combustion experiments.

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POSTER PRESENTATION

Innovative microstructural characterization for nanocrystalline tellurides using advanced synchrotron Wide Angle X-ray Total Scattering

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ABSTRACT:

Here we present a thorough structural and microstructural characterization of Fe5Te4 nanoparticles (NPs), performed through conventional powder diffraction, advanced synchrotron Wide Angle X-ray Total Scattering (WAXTS) techniques. Synchrotron WAXTS measurements were performed at the X04SA-MS beamline of the Swiss Light Source. With these data we have presented an innovative microstructural model for Fe5Te4 NPs, aiming at integrating the effects of microstrain within the DFA modeling of a population of spherical NPs of variable size. [1] Through this approach we provided a quantitative estimate of the defectiveness (microstrain) of this nanosized material. On the methodological side, a newly developed protocol for integrating microstrain analysis within the DFA method of characterization was here presented, opening the way to new, and unexpected, outcomes of advanced total scattering experiments. A DFA-based microstrain model, integrated with structure, size and morphology information, indicates the presence of highly strained nanoparticles, with strain preferentially accumulated in the ab-plane than along the c-axis of the tetragonal structure. We've also produced series of nickel sulfides and tellurides by mechanical alloying looking for its promising application in sensing or catalytic materials, in batteries, solar cells, super-capacitors and electrochemical catalysis. We expect to use part of the time of this oral presentation to show our proposals to repeat such innovative and successful microstructural characterization for the nanocrystalline nickel tellurides and sulfides using LNLS (Proposal ID: 20180111) and Sirius Facilities.

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POSTER PRESENTATION

In-situ SAXS investigations of nucleation of palladium-gold nanoparticles

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ABSTRACT:

Nanoparticles of noble materials such as platinum, silver, and gold have been largely studied due to their unique physical and chemical properties. In particular, core-shell palladium-gold nanoparticles are investigated due to their potential application in catalysis processes. In the present study we retrieved time-resolved structural information on the time-dependent growth process of these nanoparticles in wet-chemical processes. Using SAXS in-situ measurements we were able to monitor the formation of the palladium seeds followed by the epitactic formation of the gold-shell, which is confirmed by high resolution scanning transmission electron microscopy (STEM) and energy-dispersive X-ray spectroscopy (EDX). Interestingly, cell culture experiments demonstrated the high biocompatibility of the formed nanoparticles.

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POSTER PRESENTATION

Interplay between intrinsic structure and multistate emissive properties of N and N, B-doped Carbon Dots

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ABSTRACT:

The emerging carbon-based quantum dots, carbon dots (CD), have attracted attention because of their potential in optoelectronics [1] and biomedical applications [2], due to their unique tunable photoluminescence properties, easy synthetic procedures, long-term stability, biocompatibility and ability to modify the surface functionality [3]. In general, CDs consist of sp2 hybridized aromatic domains (mainly graphitic) embedded in a sp3 hybridized molecular domain (mainly amorphous). Complex interplay between these two domains controls the overall photophysics of CDs, and the presence of surface defects and hetero atom doping (e.g. N, P, B etc.) play a crucial role to modify the photoinduced radiative/nonradiative recombination processes (photoluminescence) as well as charge separation pathways [4, 5, 6]. Herein, N doped and N, B co-doped CD were synthesized from citric acid (C-source), urea (N-source) and boric acid (B-source) by using an already reported bottom-up microwave assisted method [7]. Initial optical studies showed two broad absorption bands at 340 and 410 nm in N doped CDs, responsible for excitation dependent blue and green photoluminiscence. A single, almost excitation independent, blue emissive state was observed in N, B co-doped CDs. In order to understand the intrinsic structural features of the CDs and their direct correlation with the optical properties, XPS (with both conventional and synchrotron X-ray sources) and synchrotron N-K-edge X-ray absorption fine structure (NEXAFS) measurements were performed. These characterizations showed that N atoms in the N doped CD are directly bonded to the C atoms inside aromatic domain, which creates new lower energetic states resulting in a green emission (n-pi* transition) due to N extra electron. In presence of B, B-N bonds are formed in the aromatic domain localizing the extra electron in the bond, and probably being this rearrangement of charges the reason for the lack of green emission.

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Synthesis, optical studies and conventional XPS measurements were performed at the PhOG, Ludwig Maximilians Universität, Munich, Germany. XPS y NEXAFS measurements using synchrotron X-ray sources were performed at PGM beamline at LNLS, Campinas, Brazil (Proposal 20170633). Project partially financed by ANPCYT (PICT 2015-2285).

POSTER PRESENTATION

Investigating the support influence on the CO oxidation reaction in Cu/CeO2-x (0 < x < 0.5) nanoparticles

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ABSTRACT:

Carbon monoxide (CO) is a poisonous gas formed by the incomplete combustion of organic matter. In order to reduce the CO emission, it is typically used catalysts containing noble metals, such as Pt, Ru and Ir [1]. Cheaper catalysts with higher efficiencies would attract attention from industries. In a previous work, CeO2-x (0 < x < 0.5) nanoparticles were synthesized with high surface area values [2]. When exposed to a CO reducing atmosphere, it was observed the formation of a high oxygen vacancy population on these CeO2-x nanoparticles. Cu nanoparticles (synthesized following [3]) were supported on these CeO2-x nanoparticles, since a high oxygen vacancy population influences strongly on the catalytic activity of the CO oxidation reaction. The Cu/CeO2-x nanoparticles were characterized by Transmission Electron Microscopy (TEM), that showed Cu nanoparticles with two size populations of mean diameter around (6 ± 2) nm and (19 ± 6) nm. X-ray Diffraction (XRD) measurements allowed the identification of the crystalline phases existing in the samples. Later, the catalysts were exposed to a CO reducing atmosphere at 400 ºC aiming to tune the oxygen vacancy population. After this, the samples were cooled to 250 ºC or 150 ºC where the CO oxidation reaction occurred, while the product formation was monitored by mass spectrometry. The samples were characterized by Synchrotron Lab techniques like in situ X-Ray Absorption Spectroscopy (XAS) (at the Cu K edge in transmission mode), in situ time resolved XAS (at the Cu K edge and Ce L3 edge in transmission mode) and X-Ray Photoelectron Spectroscopy (XPS). The reactivity towards the CO oxidation reaction is up to 2 times higher for the Cu/CeO2-x nanoparticle synthesized in comparison to the commercial standard and it depends on the reduction and oxidation kinetics properties of Cu and Ce atoms, which are directly related to the interaction between Cu and CeO2-x support.

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ORAL PRESENTATION

Investigation of protein-surfactant complexes

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ABSTRACT:

Surfactant molecules, in particular Sodium dodecyl sulfate (SDS) has consistently been shown to induce secondary structure, particularly α-helices in polypeptides, and is commonly used as a model system for hydrophobic environments. In recent publications [1-3] we demonstrate this effect for three proteins, hen egg white lysozyme (HEWL), α-Lactalbumin and Casein. Combining transition points obtained from isothermal titration calorimetry (ITC), which provided principle guidance for other techniques, one can use fluorescence spectra and circular dichroism to investigate the unfolding and refolding of the proteins; obtain detailed structural information about the formed complex in the different binding stages by small angle X-ray scattering (SAXS) data and advanced modeling, among several other methods. These results permitted the proposition of a general model for the protein-surfactant complex which showed a novel, generic structural model of SDS-protein complexes. The obtained results provide molecular insights into the role of surfactant on the unfolding and refolding of protein, in general.

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POSTER PRESENTATION

Investigation of the band structure by means of ARPES technique in transitions metal substituted BaFe2-xMxAs2 (M = Co and Cu)

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ABSTRACT:

The recent discovery of superconductivity in iron-arsenide compounds has attracted much attention to this family in the last decade. Much of this interest came from this family hosts a variety of high temperature superconductors, with the advantage they are intermetallic compound unlike cuprite superconductor that are ceramics. Although the iron-arsenide family presents a wide number of compounds, all of them presents a common FeAs layers in their crystal structure, which is known by influence their physical proprieties, including the value of TC itself. In this work we present polarization dependent angle resolved photoemission spectroscopy (ARPES) measurements for the indium-flux growth BaFe1.99Co0.01As2 and BaFe1.92Cu0.08As2 single crystals below the magnetic transition temperature (TSDW) using the facilities of the LNLS at PGM beamline. Our preliminary results show an existence of a hole-like pockets at Γ point and an electron-like pockets at X point for both compounds. These results are in accordance with the literature [1,2] for the parent compound BaFe2As2 and the Co-doping compound, which shows an existence of five Fe 3d Fermi surface sheets, with three hole-like pockets at Γ point and two electron-like pockets at X point. Here we will show the fitting for the bands that form the hole pocket in Γ point and compare the size of this pocket between each compound and with the previous data for the parent compound BaFe2As2, which can be related with the physical proprieties that these compounds show in low temperature.

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ORAL PRESENTATION

$La_3Co_4Sn_{13}$ superconducting compound under pressure

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ABSTRACT:

The superconducting La3Co4Sn13 material with transition temperature at Tc = 2.70 K, also presents a superlattice structural transition at T* ~ 150 K, which doubles its lattice parameter in respect to the higher temperature phase. Questions whether these lattice distortions arise from a charge density wave (CDW) and the order parameter of the system, i.e., first or second-order, are still in debate. It has been suggested in isostructural non-magnetic intermetallic compounds that T* can be suppressed to zero temperature, by combining chemical and external pressure, and a quantum critical point is argued to be observed near these critical doping/pressure. Our electrical resistivity and x-ray diffraction experiments under pressure, on La3Co4Sn13 single and powdered crystals, reveal an enhancement of Tc and the decrease of T* as a function of pressure. We show that superlattice structural transition remains a second-order phase transition under pressure and estimate its suppression at around 5.5 GPa, though no quantum critical behavior is observed up to 2.53 GPa. Thermal hysteresis loops are seen around T* in electrical resistivity curves under pressure and we argue that this effect is due to the pinning of a partially gapped CDW phase, which sets in at T*.

POSTER PRESENTATION

Li compounds characterization of Li-enriched mushrooms for TGM

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ABSTRACT:

The aim of this project was to determine lithium (Li) compounds of Li-enriched edible mushrooms. This project was performed from 2015 to 2017 at the Brazilian Synchrotron Light Laboratory (LNLS). Initially, we performed Li quantification by atomic absorption spectrophotometry (AAS) in the mushroom of Pleurotus djamor PLO13, which was produced in the coffee husk with 0, 100, 150, 200, 300 mg Kg-1 supplementation of Li as LiCl. Other organic and inorganic compounds of Li were used as standards. After this initial evaluation, they were measured by X-ray absorption spectroscopy (XAS) and X-ray photoelectron spectroscopy (XPS). The TGM beamline was used for the XAS measurements at Li K-edge, around 54.7 eV in TEY mode. The XPS measurements were performed at the Brazilian Nanotechnology Laboratory. Inorganic and organic standards were used for comparison of the obtained spectra. The low concentration and the location of Li in the mushrooms limited its identification directly from the mushrooms' powder. In October 2017, using a sample preparation based on extraction in chloroform and acetonitrile it was possible to obtain good quality XAS spectra at the TGM beamline. This was due to a higher amount of Li in the mushrooms extracted by the above-mentioned solvents, as confirmed by AAS. In these sample fractions and solubilized standards, Li was detected in varying K-edge energies and spectral shapes, indicating the interaction of this element with fungal compounds. Therefore, we can conclude that Li is associated with the cytoplasmic membrane, in an interaction with phospholipids and fatty acids. However, a better XAS characterization of standards still poses a great challenge for the field and shows the importance of beamlines operating at the energy range of tens of electron-Volts, such as TGM.

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POSTER PRESENTATION

Low energy electronic excitations in oxides with trirutile structure - A resonant inelastic X-ray scattering study

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ABSTRACT:

Noticeable collective effects on Physics of Condensed Matter, such as superconductivity and the colossal magnetorresistance, originate in the coupling between electronic spin, charge and/or orbital degrees of freedom with the crystal lattice. The interaction between orbitals and the crystal lattice can be found in materials with spins chains. An example of quasi-1D magnetism is the Mott insulator $CuSb_2O_6$. The charge transfer gap of $CuSb_2O_6$ is 2.2 eV [1], the magnetic susceptibility curve deviates from the Curie-Weiss law below ~ 150 K, reaching a maximum at 60 K, being well modelled by a 1D Bonner and Fisher model [2, 3]. Raman measurements on $CuSb_2O_6$ revealed a well-defined excitation at 520-550 cm-1 (~50 meV) [4]. This excitation was associated to an orbital wave (orbiton). To our knowledge, such a low energy orbital excitation has not yet been observed for e_g electrons, which typically present a higher energy scale for the electronic excitations due to the Jahn-Teller energy splitting. For instance, recently found orbital excitations on Sr_2CuO_3, and $LaTiO_3$ presents energies of 2 eV and 250 meV. In $CuSb_2O_6$, the low energy orbital excitations strongly couples with phonons with similar excitation energy. This makes $CuSb_2O_6$ a unique system for studying new phenomena associated with orbital-phonon coupling. However, this orbital excitation still needs to be confirmed, which could be obtained experimentally by resonant inelastic X-ray scattering (RIXS). This RIXS study is proposed to be performed at the IPÊ beamline at the new synchrotron facility SIRIUS. This orbital collective excitation is expected to be found relatively close to the elastic line (at about 50 meV far from the elastic peak), which makes this a challenging measurement. The resolving power [E/ΔE] of the IPÊ beamline is about 50000 in the high resolution mode (~1.7 times larger than the best RIXS setup in operation today), which makes this study perfect for the new beamline.

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ORAL PRESENTATION

Magnetic structures and transitions of GdNiSi3 and TbNiSi3

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ABSTRACT:

Compounds of the intermetallic family RNiSi$_3$ (R = Gd-Yb) present antiferromagnetic phases at low temperatures (TN= 2-32 K) with fairly marked anisotropy, revealed by magnetization and magnetic susceptibility measurements. In spite of this, the magnetic structures still need to be determined. This information can help the understanding of the competition between long-range RKKY interaction and crystal electric field. In this work we will present the magnetic structures of GdNiSi$_3$ and TbNiSi$_3$ determined by means of resonant magnetic x-ray diffraction experiments. It is known that photons are also sensitive to the electronic magnetic moments, leading to the appearance of magnetic Bragg peaks in the magnetically ordered phase of the material and that the intensity of these peaks are enhanced when the energy of the X-rays coincides with some absorption edge of the magnetic element, causing a resonant scattering condition [2]. Furthermore, photons scattered by magnetic moments have a different polarization when compared with the ones scattered by charge cloud. With the help of a polarimeter on the detector, it is possible to eliminate a great portion of the undesirable photons from Bragg peaks and allow us to solve the magnetic structures. The diffraction measurements described above were carried out at the XDS (X-ray Diffraction and Spectroscopy) beamline of LNLS for GdNiSi$_3$ and TbNiSi$_3$. In our measurements on the Gd based-compound, we found that the magnetic Bragg peaks are located at the same points as structural Bragg peaks. Comparing the measured and calculated, we determine that this compound have a commensurate magnetic structure, where ferromagnetic layers of Gd magnetic moments in the ac plane are stacked in a + - + - sequence, with magnetic moments along the a direction. A preliminary analysis of the results obtained for the Tb based-compound indicates that its magnetic structure is similar to GdNiSi$_3$.

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ORAL PRESENTATION

Mars soil analogues: the case of Diamantina-MG red soil

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ABSTRACT:

The investigation of microbial life and/or inorganic biosignatures on Mars has been done mainly by the study of analogue environments on Earth. The nature of the ferriferous regions in Brazil seems to be suitable, as a substantial sources of extremophiles (Duarte et al, 2012) and potential source of inorganic biosignatures. This work is aimed to improve the knowledge needed to test the possibility of present or evidence of past life on Mars by characterizing samples of the iron-rich soil of the region of Diamantina, located at the center of Brazil. This characterization consists of examining the atomic and molecular compositions and the structural, mechanical and optical properties of the soil. This is done by applying different experimental techniques, such as thermo-gravimetry, electron microscopy and X-ray spectroscopy. The molecular and atomic structure had been measured through, both, X-ray diffraction and fluorescence at LNLS (Brazilian Synchrotron Light Laboratory - Campinas). Also, the volatile content was investigated through thermo-gravimetry in a high-temperature furnace and by H:C:N analysis. The X-ray diffractogram is dominated by crystalline silicon oxide with low contribution of an amorphous phase. Also, there is no strong evidence of phylosilicates (clay minerals). Thermo-gravimetry showed weight losses of just ~1 wt% at 600oC. For comparison, JSC Mars-1, a well-known Martian analog soil, has a large volatile content, presenting losses of 21.1 wt% in the same conditions. It also confirms the absence of phylosilicates. Viking in situ experiments of the Martian soil released ~1.0 wt% water from samples heated at 500oC, which suggest that Martian regolith is also extremely dry (Biemann et al. 1977). So, our preliminary results showed good evidence that Diamantina's soil can be a good candidate for Martian simulant, just as JCS Mars-1 and JMSS-1, commonly used by space agencies to test life supporting systems or the mitigation of transport equipment.

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ORAL PRESENTATION

Mayaro virus infection in immunocompromised mice leads to paw inflammation

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ABSTRACT:

Introduction and objectives - Mayaro virus (MAYV) is an emerging mosquito-borne virus present in Latin America, including Brazil. MAYV is the causative agent of Mayaro fever, an acute febrile illness characterized by pain, rash and polyarthralgia, which is often confused with Chikungunya. Mayaro fever patients may evolve to a chronic disease with incapacitating joint pain which may endure for months. Besides the increased risk posed by MAYV, infection is poorly understood and there is no treatment or vaccine available. Herein, our goal was to develop an animal model to study Mayaro fever development and test potentially protective compounds against the disease in vivo. Methodology and results - We established a mouse model of MAYV infection in immunocompromised mice (ABR-/-) using a MAYV strain isolated from a brazilian patient. Infected mice died after 4-5 days post infection and presented weight loss, high viral loads in target tissues and significant pathology in the inoculated paw. Histological analysis indicated paw edema and recruitment of granulocytes and mononuclear cells. In collaboration with the IMX beamline at LNLS, we developed a X-ray microtomography technique that allowed us to measure the volume of infected paws with µm3 precision. This data confirmed the edema caused by MAYV, which increases as the infection develops, and indicated that the bones may also be affected. Conclusions and perspectives – Our mouse model of MAYV infection recapitulates several features of the disease in humans, notably the viremia and inflammation in the limbs. Our new microtomography technique corroborated our previous data on paw inflammation with unprecedented precision, can be used as a marker of disease development and showed aspects of MAYV infection that were not observed by classic histological techniques. Our mouse model of MAYV, including paw microtomography, will be useful in the screening of potential treatments against MAYV in the near future.

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ORAL PRESENTATION

Mechanochemical synthesis and characterization of nanocrystalline Chalcogenide alloys

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ABSTRACT:

Ni-Te nanocrystalline alloys containing Ni3Te2, trigonal $NiTe_2$, Ni and Te nanocrystals was prepared through mechanochemical processing of pure elemental tellurium and nickel powders in argon atmosphere. Ni25Te75 and Ni75Te25 samples processed from 3 h up to 30 h milling times were characterized by X-ray powder diffraction, transmission electron microscopy, magnetization and Raman spectroscopy. Ni3Te2 was obtained with just few hours (6 h) of milling of the Ni75Te25 sample coexisting with Ni in nanocrystalline form. This sample are being characterized and promising applications in electroanalitics were found and will be published elsewhere. Trigonal NiTe2 crystals with average size of 16 nm can be obtained after only 3 h of processing time of Ni25Te75 sample [1]. For longer milling times, the trigonal NiTe2 phase becomes majority (about 70% with 30% for nanometric Te and no pure Ni was detected) and its average crystallite size slightly increases to 20 nm. Transmission electron microscopy images and electron diffraction patterns confirm the nanometric size of the crystalline domains in the agglomerated particles. The magnetic properties of the Ni25Te75 powders are dependent on synthesis time, suggesting a paramagnetic behavior mainly associated with the NiTe2nanophase. Raman spectra showed peaks that can be associated with unreacted Te and tellurium oxides modes, but it also showed several modes that can be attributed to trigonal NiTe2 nanophase. The high-pressure experiments showed no phase transitions for $NiTe_2$ up to 17 GPa and Te phase transitions from form I to forms II and III occurred simultaneously at 4.5 GPa, remaining up to 12 GPa; after that, only reflections of Te-III and the $NiTe_2$ were observed. All the phase transitions observed with pressure are reversible after decompression. The bulk modulus determined from the least-squares fit of first-order Murnaghan equation of states is 110 GPa for the $NiTe_2$ nanophase and 28 GPa for Te-I.

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POSTER PRESENTATION

Metal/bamboo bionanocomposite: a Lignocellulose-based microreactor for flow chemistry organic synthesis

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ABSTRACT:

Biomicrofluidic [1] use biomaterial and inspired design from the nature to create biomimetic microfluidic reactor useful for organic reaction and analytic platform.2 Our project is focused on the development of a bamboo microreactor whole constituted by biodegradable biopolymers. The principle aim is to internally functionalize the bamboo vascular bundles with catalytic metal nanoparticles (Me-NPs). A nanocomposite bamboo is used as a chemical platform for metal-catalyzed organic synthesis. Biomimetic microfluidic devices allow us to develop a new class of cheaper and sustainable flow reactor compared to artificially made microfluidic systems available on the market. To analyze in real-time metal nano- and microsized coating dispersion into lignocellulose microchannels, SyncMicroCT is advantageous, in this case due to the high acquisition speed, as compared to lab-based equipment, to observe the dynamic flow during the injection of colloidal solution. Synchrotron microtomography analysis (SyncMicroCT) was already used to investigate the plant hydraulics system in xylem vessels tracing in real time water/Au-NPs uptake [3]. Kim S. et al. used a SyncMicroCT to visualize the entry of water into embolized vessels in bamboo's leaves [4]. Our aim is to determine the selective region of the metal nano- and microscale deposition and quantify the volume fraction of their aggregates during the injection of different chemically modified Me-NPs. To deeply control the production of hybrid bio-engineering material, the bamboo filled system has to be fully characterized to determine the following data: metal-polymer interaction inside the biological matrix, density aggregates per volume, the volume fraction of the cluster in to the microambient and the interfacial region between the metal coating and microchannels system. The filled bamboo with the selected metal nanoparticles can pave the way for new functional materials with enhanced catalytic activities for flow chemical reaction.

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ORAL PRESENTATION

Metals and glycosaminoglycans in tumor progression

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ABSTRACT:

Metals are relevant elements in physiology as regulators of numerous cellular activities, exerting their roles as local cofactors and systemic modulators. Metals, such as manganese, are involved in cell migration, oxidative metabolism and cell proliferation and are, therefore, relevant in tumor progression. Invasive cells migrate and survive, colonizing healthy tissue. Manganese is a key element in this process, as integrins activation depends on this cofactor, as well as heparan sulfate proteoglycans – which modulate growth factor binding and may further promote tumor progression. Finally, tumor cell resistance to oxidative environments may also depend on MnSOD, a central enzyme in the antioxidant response. We hypothesize that manganese is a central element in tumor progression by enhancing migration via integrin activation, and by enhancing tumor cell survival promoting an effective antioxidant response. Manganese is possibly captured on cell surface and/or extracellular matrix heparan sulfate, therefore, we also hypothesize that exogenous heparin may compete with heparan sulfate proteoglycans and affect tumor progression. Our work has revealed that tumor cells respond to manganese by migrating faster, while heparin treatment, post manganese exposure, reestablishes cells' migration. Investigation of manganese distribution by X-ray fluorescence at LNLS in an in vivo model of tumor progression has revealed manganese accumulation in tumor-bearing mice. We verified that primary tumors gradually accumulate manganese, while livers only present high manganese spots at late stages of tumor development. In conclusion, manganese appears to be a key factor in tumor development, and heparin and its analogues are able to affect tumor cell migration. Our perspectives include further investigation of tumor cell resistance to oxidative stress by analyzing manganese extra- and intracellular distribution, especially within mitochondria, a key organelle in the oxidative metabolism.

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POSTER PRESENTATION

Microbial syntropy evidences in preserved biofilms in Meso-Neoproterozoic stromatolites from the Vazante Group, Brazil

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ABSTRACT:

The stromatolites from the genus Conophyton cylindricus is a datum for the Proterozoic (Meso to Neoproterozoic), and its typical conical shape has been considered a result of interspecific competition between photosynthesizers microorganisms for space, light and nutrients. At the “Paleontological Site of Cabeludo”, São Francisco´s Craton (Southeastern Brazil), there are expressive records of this genus and it is exceptionally well preserved. These stromatolites are composed of in situ fossilized biofilms; they are silicified, and it contains carbonaceous matter preserved. Petrographic and geochemical analysis using EPMA, SEM/EDS, Raman spectroscopy, synchrotron-based µ-XRF and XANES revealed three different laminae composing the biofilms. The laminae may record that the fossil biofilm has been a multi-laminated microbial community, and the three biofilm types show a clear tendency to organize into a specific pattern that occurs in each layer of the stromatolite. This pattern of organization denotes certain equilibrium between the three different biofilms. In addition, micrometric crystals of apatite in these biofilms suggest metabolic activity of sulfide-oxidizing bacteria, which means that these stromatolites were formed through the activity of more than one type of bacterial metabolism, not only by the photosynthetic ones. These findings mean that maybe the microorganisms that formed the Conophyton structures in Vazante Group may have maintained a syntrophic relation instead the interspecific competition, reaching an equilibrium between the three different biofilms along the vertical section of the structures, in order to benefit all the microbial community. At least for this record, the photosynthesizers may not be the only metabolic type that caused the bioconstructions of the Conophytons, showing that the conical shape was originated by a syntrophic relation instead of interspecific competition.

ORAL PRESENTATION

µ-XRF and µ-XANES analysis of metal nanoparticles in common bean seeds: Understanding NPs transport and their role during germination

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ABSTRACT:

Nanoecotoxicology is an emerging field of science driven by the increasing use of nanomaterials in commercial products. The so far found effects of nanomaterials on plant development are not consensual. The presence of nanoparticles (NP) in agricultural inputs is a fact, as can be seen in many patents involving this application [1]. Recent works in our laboratory and part of a Ph.D. sandwich at ESRF, investigated the effect of ZnO and Ag NP on the germination of seeds of Phaseolus vulgaris (common bean). In this research we propose the application of ZnO and Ag NPs as a seed coating, to improve the seedling growth and crop conditions providing micronutrients and protection to the crop. The treatment of the bean seed coat before sowing can be performed in a safer way compared to the common dispersion method in the field. Silver NP treatments and analysis were performed at the ID21 beamline in ESRF. While ZnO NP treatments were conducted at the CENA and µ-XRF and µ-XANES analysis will be done at the XRF beamline in LNLS on second half of 2018 (Proposal 20180037). The bean test presents three different layers, which are objects of this study. Common bean seed coats were exposed to Ag2S and Ag metallic (Ag°) NP. After 5 days, seed coats were recovered and embedded in O.C.T., cryo-fixed and sectioned (~20 µm thickness). µ-XRF maps and µ-XANES measurements were done under cryogenic conditions. From µ-XRF analysis we determined the localization of Ag in the different tissue layers of the seed coat. µ-XANES spectra showed changes in the chemical environment of the Ag° NP treated sample: Ag° was concentrated in the internal layer and the released ions are forming thiolate complexes. µ-XANES of Ag2S NP treatments did not show changes in speciation. Similar results, of metal transformation, are expected for samples treated with Zn that will be freeze-dried to analysis. The combination of these results will contribute to build another step in the understanding of how nanoparticles can improve agricultural inputs and benefit food crops.

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ORAL PRESENTATION

Morphological analysis of induced pluripotent stem cells derived cerebral organoids from corpus callosum dysgenesis patients and control subjects

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ABSTRACT:

Dysgenesis of the corpus callosum (DCC) is a neurodevelopmental disease in which the corpus callosum (CC), the largest commissure in the brain, is absent or reduced. Although not having part or all the CC, DCC patients do not show a typical inter-hemisphere disconnection syndrome [1]. This phenomenon has remained as a neurological paradox for decades and it is still subject of studies to which our team has intensively contributed [2,3,4]. Here, we developed an ex-vivo model to investigate molecular and cellular basis of DCC. Our model is based on cerebral organoids produced from induced pluripotent stem (iPS) cells generated from DCC patients and controls. We aimed to study morphological changes in cerebral organoids that correlates with phenotypes observed in patients, by using IMX line to perform microtomography. Cerebral organoids were stained with osmium tetroxide, dehydrated and embedded in paraffin. Transmission radiography was obtained using a polychromatic beam with peak energy of 11 keV together with an indirect detector based on a scintillator and an optical microscope. The CCD detector (PCO.2000) was composed of a 2048x2048 pixel array that would limit the field of view (FOV) in accordance with the desired resolution of 0.82 um to 1.6 mm FOV for high resolution images. An acquisition covering an angular range of 180 degrees was used to compose a complete sinogram with a total of 1024 projections and exposure time of 1 second per projection were used for composing the necessary dataset for 8-bit image reconstruction using the raft algorithm. Morphological features could be extracted from patients and controls such as total volume, ventricle volume and subventricular area in reconstructed slices. Our results are promising to the analysis of morphological features comparing DCC patients and controls.

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POSTER PRESENTATION

Morphological description of reproductive system of P. harpagos and P. anisophallos by SR-MicroCT

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ABSTRACT:

We report in this work the possibility of taxonomic identification using the reproductive system of the fishes of the species Phalloceros harpagos and Phalloceros anisophallos. Systematically speaking, Phalloceros are yet poorly studied, despite being well known in some biological standpoints. The collected fishes were stored in Ichthyology Laboratory at Science Department at the Faculdade de Formação de Professores (UERJ). Specimens had about 10 to 15 mm in length. The specimens were euthanized using benzocaine in aqueous solution, by prolonged immersion for approximately 10 minutes and then fixed in 5% formalin for 24h, then transferred to 70% ethanol. Prior to scan, specimens were dried in series of ethanol, up to 100%, and scanned in a pipette tip (sealed on the tip), immersed in ethanol 100%. A lid was placed on the top of the pipette tip and pressed to increase internal pressure and avoid the formation of bubbles. Specimens were scanned at IMX beamline, at the Brazilian Synchrotron Light Source (LNLS), using pink beam configuration, at a resolution of 1.64 µm. Reconstruction was performed with an in-house software provided by the beamline team. For 3D visualization, Avizo 8.0® was used. The images showed excellent details of the different rays of the gonopodium for adult and young males. Clear differentiation between young and adult fish was also possible using both internal and external structures of the gonopodium. Details such as the presence of the right hook and absence of the left one, as well as symmetry of the gonopodial appendix, presenting its halves similar to each other, could be visualized. Identification of fully developed gonopodium could also be performed. A fully developed gonopodium is the one whose distal appendix is completely developed and lacks external coverage of skin.

POSTER PRESENTATION

Morphometric analysis of Thoropa miliaris tadpole using phase-contrast synchrotron microtomography

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ABSTRACT:

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]. Anurans present a variety of reproductive modes, most species are direct developers and lay eggs on water and a tadpole hatches and swim in bodies of water till completion of metamorphosis, with a small froglet that abandons the aquatic life [2]. The species Thoropa miliaris present morphological conditions that differentiate it from other species. Tadpoles live in the film of water on rock surfaces at the wet borders of waterfalls in rain-forest areas, and in rock fields of mountain ranges of southeastern Brazil [2]. Although most species in the genus are common and well sampled in collections of natural history museum, little is known of the group's morphology. Application of microtomography (microCT) for the study of frogs is quite recent [3,4]. The first work of our group was to show the virtual dissection of this species [5]. Here, we present the first results of the high-resolution non-invasive morphometric analysis of soft tissues of the Thoropa miliaris tadpoles. Microtomographic images were obtained at the imaging beamline (IMX) at LNLS. A detailed knowledge of the interior of biological structures and organisms is crucial for a better understanding of their function and evolution. In this work, whole-specimens of Thoropa miliaris in three different larval stages of development were imaged. Phase retrieval algorithm was implemented together with the conventional filtered backprojection reconstruction algorithm using the PYRAFT software developed by the LNLS team. The resulting voxel data sets were visualized with Avizo 8.0. The contrast between the different tissues in the microCT images was sufficient to be able to distinguish complex internal structures, such as skeleton tissue, notochord, brain and crystallines.

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POSTER PRESENTATION

Multitechnique size determination of Fe/FeOxide@NaCit magnetic nanoparticles after magnetic separation

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ABSTRACT:

Colloids of Fe/FeOxide@NaCit were prepared by a combined route of mechanochemistry + chemical coprecipitation + electrostatic stabilization. The obtained nanomaterial presents a high magnetic response characterized by a saturation magnetization up to 130 Am2/kgFe, long stability of more than two years, and biocompatibility confirmed by MTT essays which gave viability values of 80%. These materials are excellent candidates for several biomedical applications. In this work we present size characterizations of different fractions of the colloid obtained by magnetic separation. The separation process was performed using an ad hoc multi chamber prototype holding two NdFeB permanent magnets. The size distributions of the resulting fractions were characterized by TEM, SAXS and a novel optical method based in UV-Vis spectroscopy. The results of these three techniques are compared, finding consistent concordance and complementary information.

POSTER PRESENTATION

Nano-tomographic analysis of the amelodentin junction in teeth submitted to radiotherapy in vivo

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ABSTRACT:

Radiation caries is a chronic side effect of head-and-neck radiotherapy with a high potential for tooth destruction. It has been debated whether the occurrence of radiation caries is due to a direct or indirect effect of irradiation on teeth or both. Studies addressing this issue and that analyze in depth the structural changes are rare because teeth are extracted prophylactically due to the high probability of causing future problems, such as very rapid and extensive destruction, and to avoid extraction after radiotherapy (risk of osteoradionecrosis). On the other hand, in cases where the teeth are kept, almost invariably they become damaged, with extensive tissue loss, making a complete analysis almost impossible. Better understanding of radiation's effects on tooth structure non-destructively is paramount. The development of novel imaging technologies amplifies the excellence of scientific dental research. Nano-tomography is a non-destructive method that allows for samples to be examined many times and continue to be available, after scanning, for additional biological and mechanical testing. Nano-tomography systems allows clear visualization of structures on cellular level as well as internal ultrastructure of dentin and submicron hard tissue cracks. This work was carried out in partnership with the Center for Cancer Control of the Pedro Ernesto Hospital – UERJ, where we were able to obtain teeth extracted because of severe periodontal disease and that still maintained its structure in good condition, without loss of enamel or greater destruction after radiotherapy. Due to complete periodontal bone loss, the teeth were fixed only by soft tissue and the extraction could be performed with minimal risk and will be analyzed to observe structural changes specifically at the enamel-dentin junction.

ORAL PRESENTATION

NP3 platform: assisting new discoveries from natural products

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ABSTRACT:

Natural products are excellent sources for new medicines, and about 65% of the approved drugs are natural products or derived molecules. However, it is a great challenge to obtain and characterize natural compounds, once this activity is time and resource consuming. Among other bottlenecks, it can take months or even years to isolate, identify and characterize a bioactive natural product using the standard available methods. From the medicinal chemistry perspective, the information necessary for designing new drugs from natural products are: i) the compound's chemical structure, and ii) its binding site and mechanism of binding to the protein target. The NP3 platform aims to overcome the challenges and time to get this information. The platform's inputs are i) biological data of active and inactive unpurified natural product samples; ii) X-ray protein crystallography data sets obtained from crystals of the target protein soaked with the unpurified bioactive natural product samples; and iii) mass spectrometry (MS1 and MS2) data collected from the compounds present in the unknown natural product mixture. All the experimental data are obtained from miniaturized and sample saving experimental set-ups. We are designing the computer-aided platform (NP3) so it can process these three experimental data in an integrative manner, being able to identify the bioactive chemical structure and its molecular interactions with the protein target, using routines of chemical structure proposition and validation, with outputs supported by the orthogonal experimental techniques. The implementation of NP3 would increase the throughputs of synchrotron X-ray and mass spectrometry data analysis and compound identification, benefiting the scientific community, thus resulting in a higher number of novel bioactive natural products discovered and, consequently, new drugs.

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POSTER PRESENTATION

Operando XAS investigation of homogeneous copper catalysts during electrochemical activation

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ABSTRACT:

The quest for sustainable and green energy sources is one of the fundamental challenges of humankind for the following centuries. Sunlight is the primary energy source in our world and photosynthetic organisms use it to perform chemical reactions in a controlled fashion with a very complex machinery that is yet to be fully explained. The thermodynamic bottleneck to achieve efficiency in artificial photosynthesis is the very high activation barrier for the water oxidation reaction and candidate catalysts must be stable in highly oxidative conditions. Inspired by biological structures, several studies are focused in catalysts from earth-abundant elements, but the challenges are huge for both heterogeneous and homogeneous systems. It was only in 2012 that the first copper-based homogeneous catalyst was described with the $[Cu(bpy)(H_2O)_2](OAc)_2$ formula and fast reaction with water (100 s–1). In this work we describe the first XANES study on this catalyst in different pH and applied potentials. In order to achieve an operando condition in water, we have produced 3D-printed spectroelectrochemical cells in transmission mode in order to acquire XANES spectra for copper during the electrochemical oxidation of the samples in the DXAS beamline. The cells were produced by printing with 3D Printer Model Graber I3 and the material used for printing was filament ABS (acrylonitrile butadiene styrene). The 3D cell designs were obtained by using TinkerCad and the files were sliced and prepared to be printed through use of Repetier-Host v2.0.5 software. After obtention of cells, the surface of these ones was polished and finally Kapton tape and lead tape were used to cover them and guarantee the beam passing only the slit (1 mm).

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POSTER PRESENTATION

Oxygen spillover study in the MnxOy-Na2WO4/SiO2 catalyst for the oxidative coupling of methane in operando conditions by chemical looping experiments

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ABSTRACT:

Proved reserves of natural gas have grown in the last years, emphasizing the importance of developing technologies for the conversion of methane to more valuable fuels. Current processes that convert methane to olefins require the intermediate step of reforming methane to synthesis gas. However, there is also the Oxidative Coupling of Methane (OCM) which is a direct upgrading process. Energy-producing companies have high interest in developing this route because it simplifies the overall process and reduces costs. The MnxOy-Na2WO4/SiO2 catalyst is one of the most promising materials due to its high yield values and high stability. There is a general consensus that the limiting step of the OCM process is the methane activation (275 kJ/mole). Then, different oxygen species have been proposed as responsible for that (i.e. superoxide O2-, peroxide O- or lattice oxygen O2-). Therefore, the ability of the catalyst to release these oxygen species through different oxidation states is a critical property. However, there is controversy about the role of the oxidation state for the two cationic centers (Mn and W). In fact, WO4 species could undergo a possible redox mechanism W6+/W4+ [1]. Conversely, Jian et al. [2] reported that the methane activation takes place on Mn3+ sites which are reduced towards Mn2+. However, these mechanisms have not been corroborated in operating conditions either under several REDOX cycles (chemical looping)[3]. In order to elucidate the oxygen spillover in continuous REDOX cycles, it is proposing the use of XANES for the identification of the oxidation states of Mn and W in operando conditions (i.e. 800°C and CH4 and O2 feed). Based on this information, it is expected to propose a mechanism by this catalyst is deactivated and reactivated by redox cycles. This mechanism serves as a starting point in the search for methodologies that mitigate its deactivation after several hours on stream.

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POSTER PRESENTATION

Prehispanic ceramics from the Pampa La Cruz archaeological site: investigating its composition and provenance

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ABSTRACT:

The goals of this work were to correlate the archaeological ceramic products of “Pampa La Cruz” in Trujillo, Perú with minerals from a nearby quarry in order to understand the origin and composition of the ceramics. For this purpose, we have tackled the phase composition of these materials applying X-ray diffraction at the Laboratório Nacional de Luz Síncrotron (Brazil), using the XPD beamline and linear counter. These results were confirmed by structural refinement by the Rietveld method. All phases identified in the qualitative analysis were included in the refinement to determine their percentage by weight. In the samples of ceramics phases of quartz, anorthite, albite, riebeckite, aluminum phosphate and illite were identified. The analysis of the geological samples reported the presence of these same phases, which induces to conclude that the ceramics would come from these geological materials. One of the geological samples presents additionally phases muscovite and chlorite in small proportion, which could be due to the conditions of preparation of the paste before cooking. Structural refinement reported a high percentage of anorthite in most samples, reaching more than 40%. The complex structure of the archaeological materials requires high-resolution measures such as synchrotron radiation. By the results obtained in this study, we believe it is appropriate to continue using synchrotron radiation for studies of similar complex samples of archaeological interest.

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This work was supported by Vicerrectorado de Investigación de la Universidad Nacional Mayor de San Marcos. The authors would like to thank to the Laboratorio Nacional de Luz Síncrotron by providing facilities for the measures with synchrotron light.

ORAL PRESENTATION

Probing PtPd nanoparticles using ions, electrons and x-rays

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ABSTRACT:

Regardless of all efforts devoted to the synthesis of bimetallic nanoparticles with controlled shape and structure, the underlying relationship between their atomic arrangement and reactivity are still under debate. Particularly for the NO abatement reaction, a major environmental issue, those discussions are relatively scarce. Nevertheless, there is still a constraint of proper tools to successfully probe nanoparticles' composition and atomic arrangement. In this work, bimetallic PtPd nanoparticles with approximately 5 nm mean diameter were synthesized to achieve distinct atomic distributions: nanoalloys or core@shell. The samples were probed by Medium Energy Ion Scattering (MEIS) and space-resolved elemental analysis via energy dispersive X-ray (EDX) spectroscopy in STEM (Scanning Transmission Electron Microscope) mode. The complementary association of STEM-EDX profiling with MEIS, which simultaneously surveys millions of nanoparticles, becomes a powerful tool for a statistically representative structural analysis [1]. As result, the measurements provided key details such as core size, shell thickness and composition, and even distinguished core@shell from core@alloy structures. PtPd nanoalloys and Pd-core structures were successfully obtained while the attempt to produce Pt-core NPs actually resulted in a mixture of nanoalloy and core@alloy structures (core = Pt or Pd). In order to correlate structure and reactivity, catalysts were prepared using these PtPd nanoparticles, and their performance was evaluated toward the direct decomposition of NO reduction using XAS measurements under operando conditions [2, 3]. The Pd K and Pt L3 edges EXAFS signals were collected and simultaneously, the effluent gases from the reactor were analyzed by a mass spectrometer. The results shed some light on the structural evolution that took place during the NO abatement, at the atomic level, and proved that the structural properties strongly influence the nanoparticles reactivity.

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We would like to acknowledge the Brazilian funding agencies CAPES, CNPq, PRONEX/FAPERGS, and INCT/INES for the financial support. We also thank INMETRO (Rio de Janeiro, Brazil) for allowing STEM measurements.

POSTER PRESENTATION

Probing the tetragonal-to-collapsed tetragonal transition in $SrCr_2As_2$ pnictide under high pressures with synchrotron x-ray diffraction

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ABSTRACT:

The aim of this proposal was to study the tetragonal-to-cT phase transition as function of pressure in high-quality $SrCr_2As_2$ polycrystalline sample [1]. Using synchrotron x-ray diffraction measurements up to ~35 GPa at room temperature, tetragonal and cT phases can be determined by the refined lattice parameters a and c from the diffraction profiles. X-ray diffraction were performed at XDS beamline at LNLS with wavelength at 0.6199 Å (~20 keV) and a beam size of 80 × 50 m2 due to the Kirkpatrick-Baez mirror focalization. Diamond anvil cells with 350 culet were used with Re gaskets and He and Ne gas as pressure medium. The luminescence of Chervin Ruby balls was used to monitor the pressure up to 50 GPa. For detection it was used the bidimentional MARCCD and the diffractograms were converted through the Fit2d software. Two series of diffractograms as function of pressure were taken. At 1.5 GPa, it was observed all Bragg peaks of the expected tetragonal phase (I4/mmm) from which we determined the lattice parameters a = 3.8768 Å and c = 12.9168 Å. The expected collapsed-tetragonal phase was observed around 17.5 GPa. Under further pressure, around 22.5 GPa, a surprisingly symmetry lowering reentrant phase was observed. As a partial conclusion, the experiment was well succeeded in probing structural transitions between 0 and 50 GPa.

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One of the authors (P. F. Gomes) acknowledges the financial support from LNLS for the realization of the measurements.

POSTER PRESENTATION

Prospective studies on plant science and environmental toxicology research using advanced techniques at CARNAÚBA beamline of Sirius

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ABSTRACT:

The new Brazilian synchrotron radiation source, Sirius represents nowadays one of the most modern synchrotron sources capable of studying several kinds of materials with unprecedented brilliance [1]. CARNAÚBA (Coherent X-rAy Nanoprobe BeAmline) is the longest among Sirius first phase beamlines with 145 m from source to the sample. Multiple techniques such as, X-ray fluorescence, X-ray absorption, X-ray diffraction and advanced X-ray coherent methods can be possible to use and combine to understand the local properties of heterogeneous material at a nanometer range. Recent technological advances in X-ray focusing optics and fluorescence detection have greatly improved the use of synchrotron techniques in plant science and environmental toxicology research. Soil contamination with toxic metals causes damage to both human health and the environment. Although the adverse effects of these pollutants have long been recognized, several aspects such as their behavior in soils, their toxicity to plants, and the mechanisms that some vegetable species use to tolerate high levels, have been still poorly explored SR based techniques are increasingly being used in soil–plant systems to evaluate toxic metals with important applications on environmental toxicology studies. We proposed the use of CARNAÚBA beamline at Sirius to investigate the uptake of toxic metals by plants and to understand the mechanisms involved in the process of tolerance developed by combining XRF imaging techniques and micro X-ray absorption spectroscopy, to analyze the roots in their natural undisturbed state in the rhizosphere. B napus and F arundinacea will be tested to study their behavior in a Pb contaminated soil Researchers can take advantages of these new techniques as data collection will be in a way that has not been possible previously, in this way new perspectives of research in strategic areas such as environmental toxicology, plant science, health, and many others.

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POSTER PRESENTATION

Quantitative determination of element distributions in biomembrane models at the air/water interface by grazing-incidence X-ray fluorescence

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ABSTRACT:

In the Annual User´s Meeting of 2012 we proposed to install a Grazing incidence setup for Langmuir monolayers at the air/water interface; this means in horizontal sample geometry (Oliveira, 22 RAU). Such setup evolved [1] and is currently producing the very first published works [2]. This setup will be translated to SIRIUS at the SAGUI beamline where the performance will increase significantly (Antonio Gasperini, personal communication). Moreover, the time consuming and sometimes difficult experiments will be performed much quick and easier due to the higher photon flux, allowing getting GIXOS and GIXD on second time scales. Then, performing additional measurements on the same monolayer can be affordable and desirable. In this opportunity, we propose to couple the existing grazing incidence setup of the Langmuir trough with a fluorescence detector. By slights modifications of the incident grazing angle (with the very same system to deflect the beam below the critical angle for total reflection in grazing incidence setup), the penetration length of the evanescent wave will be modified. This allows the differential excitation of X-ray fluorescence of atoms present along the vertical profile of the monolayer itself or in the counter-ion layers beneath it. This will help during the electron density modelling of the monolayer itself and/or to account for the distribution of ionic layers [3]. Of course, this setup will be useful in other biomembrane model systems like solid supported layers.

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POSTER PRESENTATION

Radiation damage effects to hybrid-pixel detectors images

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ABSTRACT:

Hybrid pixel detectors are being consolidated as a competitive approach for X-ray imaging, addressing synchrotron and medical applications. In both cases, failures of the detection processes related to the radiation damage can result in severe data loss. The high intensities of modern synchrotron beams and photon energies high enough for reaching the ASIC beneath the sensor [1] could compromise the detector's life cycle. This work aimed at quantifying the influence of the dose deposited at the detector ASIC on the image, obtaining a dose threshold for a data quality assurance. The dose deposition at the ASIC was modelled through Monte Carlo simulation, using the PENELOPE 2014 with the PenEasy 2015 code [2]. The dose deposited on the oxide layers of the transistors was simulated, since the damage in the 2 – 100 keV photons energy range correspond to effects in the oxide [3]. Low (up to 809 ± 6 Gy) and high (up to 6230 ± 20 Gy) dose experiments were performed by irradiating a Medipix3RX single chip [4] bonded to a 200 µm thick silicon sensor with the polychromatic beam from the Brazilian Synchrotron Imaging beamline. The irradiation was divided in 30 to 40 dose steps in both experiments, and images of monochromatic beam were taken between steps for gradual evaluation of the effects. It was possible to evaluate both the subtle effects, by adapting a CCD area detector relative noise modelling [5], and visible effects, by analyzing the evolution of the histogram of the pixel counts. The threshold dose for subtle damages was 347 ± 9 Gy, while visible damages were observed for doses higher than 2616 ± 13 Gy. It was also possible to obtain a quantitative estimation of the detector recovery time scale: half of the saturated pixels were recovered at 110.4 ± 1.3 minutes. These results encourage periodical maintenance procedures, for example through a new equalization matrix generation [6], which proved to be a possible tool for recovering the detector performance.

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POSTER PRESENTATION

Recommendation: existence of GISAXS in the SIRIUS SAXS station

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ABSTRACT:

The National Institute of Science and Technology of Organic Electronics (INEO) has numerous projects in the field of organic thin films, ranging from tens to hundreds of nanometers thick, and in these projects, it is of utmost importance to correlate the morphological structure of the films with their optical and electronic properties. Many of these projects manufacture the most diverse sensors and biosensors that are applied in the areas of Health, Energy, Environment, among others. Other projects are related to the fabrication and characterization of electronic and optoelectronic devices such as field-effect transistors, light emitting diodes and solar cells. These projects are underway in dozens of research groups in the state of São Paulo and in several other states. Therefore, techniques that facilitate in-situ characterization, such as SAXS in its Grazing Incidence Small Angle X-ray Scattering (GISAXS) mode, will be extremely useful for this research, and will certainly be much sought in the new LNLS synchrotron light source (SIRIUS).

POSTER PRESENTATION

Rehydration of dehydrated Portland cement by in-situ synchrotron XRD

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ABSTRACT:

Dehydrated cement usually releases large amount of heat in the first minutes of rehydration [1-3]. In Portland cement hydration, the formation of phases is due to dissolution-precipitation mechanism [4-5]. The rehydration and reformation of hydrated phases from dehydrated cement may happen by the (re)adsorption of water in the amorphous structure. Anhydrous Portland cement (CP V-A, 95% of clinker Portland and calcium sulfate) was taken and hydrated during 91 days. Then, it was milled (< 0.075 mm) and dehydrated at 500ºC during 2 hours, namely as CP V-D (dehydrated cement). Cement pastes of CP V-A and CP V-D were then prepared with the following water/cement ratios: 0.38 and 0.63, respectively. Both cement suspensions were dispersed with polycarboxylate dispersant contents (0.35 and 3.00 % of the mass of the cement, respectively) and its conditions confirmed by oscillatory rheometer. A IKA Labortechnick RW 20 mixer was used to prepare the suspension using 10,000 rpm during 60 s. Pastes were added into borosilicate capilar sampler and phases during (re)hydration were monitored. All measurements started after ~4 min after the preparation procedure. The formation of phases during the hydration of the pastes with the anhydrous and dehydrated cements were monitored using in-situ synchrotron XRD (XRD-1 line). Reformation of ettringite was observed just after 4 minutes in contact with the water and remained almost constant until 48 days. After days of hydration, ettringite was converted into carboaluminates; a layered-double hydroxides (LDH) structure. Rehydration increased the carbonation state of the cement paste. During rehydration, calcium silicates (belite-like) was consumed mostly in the first hours, increasing the presence of tobermorite and amorphous C-S-H.

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ORAL PRESENTATION

Residual Stress of Ni9Mn and Ni2MnGa thin films

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ABSTRACT:

Magnetic behaviour of NiMn alloys provides a classical example of a system with competing antiferromagnetic Mn-Mn and ferromagnetic Ni-Ni and Ni-Mn interactions. Such magnetic interactions give arises the ferrimagnetic behaviour in Ni_2MnGa alloys with rather distinct magnetic moments on the Ni and Mn sites which exhibit strong influence of antisite disorder defects and local strain. In addition, the residual stress in Ni_2MnGa alloys is an important parameter which determines the martensitic transformation temperature and its feasibility as magnetic shape memory alloy. In this work we investigate the residual stress in thin films of Ni90Mn10 and $N_{i2}MnGa$ alloys grown by molecular beam epitaxy and DC sputtering techniques on GaAs wafers and glass substrates. The determination of the residual stress by X-ray diffraction measurements was performed monitoring the intensity of Bragg reflections under various tilt angles of the samples with respect to the scattering plane. Whereas, residual stress can be relaxed by moderate thermal cycling and rapid thermal annealing procedures, the effective changes chemical disorder require much longer thermal anneals and temperatures set-points to activate diffusional mechanisms. Our results shown that residual stress in the (110)-textured thin films on GaAs substrate is greater than those on glass substrate. In addition, residual stress decreases with the increase of substrate temperatures during growth and post-annealing procedures.

POSTER PRESENTATION

Resonant X-Ray Diffraction of the spin liquid candidate BaTi0.5Mn0.5O3

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ABSTRACT:

BaTi0.5Mn0.5O3 is a transition metal oxide with a double perovskite-type structure wherein the transition metals sit in three distinct crystallographic sites termed M(1), M(2) and M(3). Based upon bond lengths and Rietveld analysis, of both Neutrons and X-ray diffraction, the relative occupation of these sites was determined [1,2]. In this work, we performed resonant X-Ray Diffraction at the Mn K edge in order to improve our knowledge of the occupancy of the transition metal sites. This is a fundamental result to contribute to our recent work concerning the spin liquid behavior observed in the system [3]. The difficult to determine the M(1), M(2) and M(3) occupancies arises from the similarity between the coherent scattering lengths of Mn and Ti. We proposed Resonant X-Ray Diffraction to use the element specific absorption effects to confirm the proposed structure. This is important because the Mn ions have magnetic moments, while Ti ions do not. Complementary measurements such as muSR, heat capacity and magnetic susceptibility in the lowest T region, along with theoretical simulations, allowed us to propose an emerging magnetic lattice [3] which is frustrated. The frustration is based on the formation of magnetic dimers and trimers, coexisting with orphan ions in the structure. The effective low temperature lattice is identified as magnetically isolated triangular lattices of Mn spins. The proper characterization of this novel state still requires further theoretical and experimental efforts, such as single crystal growth.

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POSTER PRESENTATION

Results of a 3D tomography of smectite clays

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ABSTRACT:

Smectite clays are negatively charged lamellar silicate colloids which interact readily with cations and H2O. This interaction results in notable change of the physical state, easily seen in XRD measurements. In this study, we tried to measure changes in mesoscale using the 3D tomography technique, provided by the LNLS IMX Line, by exposing several clays types, like montmorillonite saturated by Li+ and Na+ cations, to the X-ray Beam. In special, during this experiment we imaged completely dry samples, and then we added water in the bottom of the glass capillary, in order to image the change of the clay structure as function of the water content inside the sample. Our results show a severe change of the observable proprieties between the wet and cry states, mainly the mesopores size distribution. These results are compatible with our previous studies with XRD. We also will discuss the problems of noise and resolution, foreseeing the impact of the Sirius' IMX lines in this kind of study.

POSTER PRESENTATION

Size and morphology differences in Pt nanoparticles synthesized with mild and strong reducing agents

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ABSTRACT:

Noble metal nanocrystals are undoubtedly important materials. As common characteristic we can cite their high chemical stability, unique plasmonic and catalytic properties, making them excellent candidates in many applications. However, their practical applications are limited by their cost and scarcity. To overpass this issue the synthesis of these nanocrystals has been explored to obtain control of the size, composition, and shape of the materials, which are parameters that directly affect their final properties, such as optical, mechanical, catalytic, magnetic, and electrical. Among the various ways of synthesizing noble metal nanoparticles, one of the most common is the reduction of their respective salts with sodium borohydride (BH) and ascorbic acid (AA), among others. These and other synthetic techniques can be used to create different nanostructures of noble metals, a branch of nanotechnology that has gained attention among researchers [1]. We have employed two reduce agent, BH and AA to synthesize platinum (Pt) nanoparticle (np), using TTAB as stabilizing agent at different TTAB/Pt ratios. Those np and their precursors were characterized with synchrotron techniques as SAXS and XAS and other techniques as XRD, UV-Vis spectroscopy and TEM. The structure of the precursor was estimated by XAS and UV-Vis spectroscopy and found to be [PtBr4]-2. SAXS results indicated that the polydispersity and size of the Pt depend on the TTAB/Pt ratio. Furthermore, the set of results obtained by SAXS, DRX, EXAFS, and TEM guided us to withdraw some conclusion: 1) the number of Pt neighbor shown by EXAFS analyses is smaller for the Pt_AA when compared with Pt_BH; 2) the radius obtained with SAXS indicates that the size of Pt_AA are bigger than Pt_BH; and 3) the size of the crystallite are bigger for Pt_BH. Despite the bigger size of the np synthesized with AA, indicated by SAXS, this particle is formed by smaller crystallites, as confirmed by TEM.

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POSTER PRESENTATION

Size control and growth mechanisms of calcium phosphate particles produced by pulsed laser ablation

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ABSTRACT:

Hydroxyapatite (HA, $Ca10(PO4)6(OH)2$) is one of the most important ceramic for biomedical applications [1]. In the latest years, much effort is focused on the nanofabrication of enhanced HA coatings. Mainly in the most biologically active section of mammalian bones: "the trabecular region" that features a honey-comb type spatial arrangement of mineralized collagen fibrils with a porosity range of 30 - 95% [2]. In this context, the ultra-porous and micro-rough films of nanoparticles deposited by the technique of pulsed laser deposition (PLD) mimics the high porosity and micro-nano structural hierarchy of the trabecular bone region. In this work, NPs were deposited by the technique of pulsed laser deposition (PLD). The ablation of target material was accomplished by a Nd:YAG laser using its two harmonics with wavelengths of 1064 nm and 532 nm with different energies per laser pulse (between 100- 600mJ). The laser beam is focused on a HA target in the presence of argon buffer atmosphere at 1 Torr of pressure. The NPs were collected on different substrates made of silicon wafers (100) and carbon-coated copper grids, for independent analyses of physicochemical properties. Results by transmission electron microscopy show control of particle size by changing the deposition parameters and the results of XRD (line XRD2) indicate the formation of the hydroxyapatite phase.

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POSTER PRESENTATION

Spatially-Resolved Energy-Dispersive Resonant Inelastic X-ray Scattering (EDRIXS) of Cu Paintings

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ABSTRACT:

Recent works have shown that the species of an element can be determined by inspecting the fine structure of the RIXS emission peaks [1,2]. One of the most important characteristics of RIXS, using SDD systems with low-resolution in energy, is the possibility of combining this tool with all of the experimental techniques and geometries associated to XRF spectroscopy. Combinations of this RIXS tool with total reflection, depth-profiling analysis using grazing incidence geometries and confocal setups have been already reported [3-6]. In this work, we present results of RIXS-2D maps in different samples containing Cu paints. The measurements were carried out at the XRF beamline @ Brazilian Synchrotron Light Laboratory (LNLS, Campinas). The samples were irradiated with photons having a fixed monochromatic energy just below the K absorption edge of copper. After a simple data processing, involving multivariate methods, the fine structure of the RIXS spectra show oscillation patterns that depend on the different Cu chemical states. The studied samples are interesting since they present a rich contrast of compounds of the same element, being difficult to analyze using conventional/time-consuming spectroscopic techniques, as the absorption ones. Using this novel RIXS tool, we produced fast and reliable maps/images showing the different species of the element of interest, providing atomic environment information at micrometer level in a 2D regime. Examples of applications of this RIXS tool for chemical state determinations in Cu paintings are presented and discussed.

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POSTER PRESENTATION

Spectroscopic study of polymeric membranes for fuel cells use

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ABSTRACT:

Proton exchange membrane fuel cells are a very versatile tool that has arising as a field with a bright future since it can produce energy in an efficient and sustainable way. Unfortunately, the membrane used in industrial applications is not suited to some environments, which greatly diminishes the true capacity of the cell to produce electric energy. One way to circumvent this obstacle is to manufacture or modify new polymers with high thermal and mechanical resistance, with also good proton conductivity. In this work, a new polymer was produced and modified with a fluorine reinforcement on its surface. After the process, it was doped with phosphoric acid and analyzed using three spectroscopic techniques: x-ray photoelectron spectroscopy (XPS), x-ray photoelectron spectroscopy with depth profile and near edge x-ray absorption fine structure (NEXAFS). Those techniques were applied to unveil modifications in the membranes structure, orientation, composition and electronic structure after the doping process. The analysis indicates that both the membranes (the pure and the reinforced one) had the chemical environment of nitrogen and oxygen modified. The fluorine reinforced membrane had greater modifications in the carbon spectrum. In both cases, the phosphorus spectrum had only one peak related to its deprotonation. The depth profile analysis showed that the phosphorum diffused a bit more in the membrane without fluorine. The NEXAFS analysis showed that both samples had peaks related to sulfide groups, but in the membrane without fluorine those peaks were less intense and spaced. It also showed that both membranes had different molecule orientations. These results indicate that the pure membrane is less prone to degradations after the doping process and has a better defined orientation in its surface.

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The XPS

POSTER PRESENTATION

SR-TXRF analysis of trace elements in whole blood of rats: Effects of irradiation with low and high doses

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ABSTRACT:

On the last decades, studies showed that the exposure to low doses of ionizing radiation on the body could sense and activate the cell signaling pathways needed to respond to any induced cellular damage. This procedure reduces cell killing compared with a single dose of high radiation dose. Damage to the vasculature can affect the function of most body organs by restricting blood flow and oxygen to tissue. The precise relationship between long-term health effects and low-dose exposures remain poorly understood. Biological markers are powerful tools that can be used to determine dose-response relationships and to estimate risk specially, the effects of low dose exposures in humans. These markers should be specific, sensitive, as well as easy and fast to quantify. Various types of biologic specimens are potential candidates for identifying biomarkers, but blood has the advantage of being minimally invasive to obtain. In this study, we proposed to apply total reflection X-ray fluorescence (TXRF) to quantify possible chemical elemental (S, Fe, Zn, K and Ca) changes in blood of Wistar rats after total body irradiation with low (0.1 Gy) and high (2.5 Gy) doses. The TXRF measurements were carried out at the X-ray Fluorescence (XRF) beamline in the Brazilian Synchrotron Light Laboratory (LNLS). The results showed that the irradiated animals with low doses have significant alterations in blood when compared with animals that received high doses of radiation. Through the analysis of all the elements, we can observe that the radiation induced oxidative stress may be the leading cause for alteration of the elemental level in the studied samples.

POSTER PRESENTATION

Steam reforming of liquefied petroleum gas over Ni/CeSiO2, Pt/CeSiO2, Rh/CeSiO2 and Ru/CeSiO2 catalysts

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ABSTRACT:

Hydrogen production by steam reforming of liquefied petroleum gas (LGP) is investigated over nickel, platinum, rhodium and ruthenium catalysts supported on CeSiO2. In situ X-ray absorption near edge structure spectroscopy (XANES) on the Ce LIII-edge and X-ray diffraction (XRD) were carried out in order to evaluate the oxidative state of cerium and the changes in the structure of the metallic phases of the CeSiO2-supported samples during reduction at 500°C under 20% H2/He flow. The XDR patterns show broad diffraction lines correspondent to CeO2 cubic structure for all samples. The characteristics lines of SiO2 are not found in the diffractograms, indicating the presence of amorphous silica. The diffractograms of Ni/CeSiO2 and Ru/CeSiO2 samples also exhibits the lines of the respective metals. The absence of metal peaks in the XDR patterns of Pt/CeSiO2and Rh/CeSiO2 samples indicated the high metallic dispersion [1]. Ce LIII-edge XANES spectra of CeO2 and Ce(OH)CO3 samples were used as references of Ce4+ and Ce3+, respectively. The fractions of Ce3+ were determined by a linear combination of the XANES spectra at the Ce LIII-edge of the sample with the Ce3+ reference. The order of Ce3+ fraction estimated after reduction at 500 °C was: Pt/CeSiO2 (61%) > Rh/CeSiO2 (53%) > Ru/CeSiO2 (50%) > Ni/CeSiO2 (47%). Rh/CeSiO2 sample showed the best performance in the catalytic tests, probably due to the nature of the metal and the high metallic dispersion. The results also indicated a slight increase in the resistance of deactivation of the samples probably due to the presence of the reducible cerium.

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POSTER PRESENTATION

Structural and biochemical characterization of LsfA, a 1-Cys Prx related with P. aeruginosa virulence

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ABSTRACT:

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 against pathogens, phagocytic cells release oxidants in an attempt to kill pathogens. LsfA belongs to peroxiredoxins (Prx) family of enzymes that are Cys-based peroxidases that can remove peroxides with extremely high efficiency. LsfA belong to the Prx6 sub-family, which comprises mostly enzymes that possess only one catalytic cysteine (so-called 1-Cys Prx). Dr. Regina Baldini´s group (Instituto de Química – USP) previously showed that LsfA is involved with P. aeruginosa virulence. Therefore, our goal here is to biochemically and structurally characterize LsfA. Using different techniques; the second order rate constants for the reactions between reduced LsfA with H2O2 (107 M -1.s -1), tert-butylhydroperoxide (106 M -1.s -1) and peroxynitrite (107 M -1.s -1) were determined. Our group had already determined the rate constant between ascorbate and oxidized LsfA (103 M -1.s -1) and here we presented preliminary data on the reduction of this 1-Cys Prx by glutathione. Furthermore, we elucidated two crystallographic structures of LsfA in distinct oxidative states (sulfenic and sulfonic acid in the CP at 2.6 and 2.0 Å resolution, respectively). Both of them are in the dimeric state, revealing the dimerization interface. Features in the LsfA active site are also described here, such as poor exposure to the solvent. In the LsfA crystal structure where CP is hyperoxidized to sulfinic acid, we observed the presence of an electronic density compatible with a PEG molecule that might be mimicking one of the possible substrates. As perspective, we want to co-crystallize LsfA with glutathione/ascorbate in an attempt to gain insights on the enzyme –substrate interactions.

POSTER PRESENTATION

Structural and functional characterization sub I-binding protein of sulfate of ABC system of Mycobacterium tuberculosis.

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ABSTRACT:

The identification of new targets for drug development is a goal to combat tuberculosis, given the growing emergence of strains of extreme resistance to current treatments. The understanding of proteins involved in essential metabolism of the pathogen is a preponderant strategy to reach that. Among the genes identified as primordial, rv2400c encodes for the SubI protein, highly conserved in Mycobacterium sp. genus and essential in M. tuberculosis. SubI is a periplasmic sulfate-binding protein that belongs to an ATP-Binding Cassette (ABC) transporter responsible for sulfate internalization into the cell. In this work, we expressed SubI in E. coli BL21 (DE3) cells and purified it using IMAC and SEC chromatographies. Crystallization screenings were performed at LBEA (USP-SP) and Robolab in the LNBio in Campinas, using different kits. Crystals that diffracted at 2.3 Å resolution were obtained with 10 mg/ml of protein in buffer 20 mM Tris-HCL pH 7.5, 50 mM NaCl. The three-dimensional structure of SubI was solved with one molecule of sulfate in the active site, confirming its binding function. A set of biophysical experiments were performed to characterize the protein behavior, including thermal shift analysis, fluorescence and circular dichroism to evidence the structural changes in the presence of sulfate and molybdate. In addition, aiming to identify leader molecules for development of a possible SubI-inhibitor, we performed thermal shift analysis, fluorescence and NMR experiments. Thermal shift results showed 21 possible interactions with different compounds, which were tested and confirmed by NMR-STD and fluorescence. Seven molecules were confirmed to interact with SubI from which, four in the active site. Fluorescence experiments were used to obtain the dissociation constants (Kd) of SubI that is in the order of micromolar. Virtual docking and crystallization assays of SubI with the fragments are under way to determinate the exact location of their interactions.

POSTER PRESENTATION

Structural and magnetic study of the Mn2-xFexSn compounds for application in innovative thermal energy conversion devices

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ABSTRACT:

The Mn_2Sn compound is an intermetallic hexagonal crystal phase. Magnetic and structural properties of Mn2Sn phase can be tailored by Mn and Fe co-substitution. In this work, our interest is to understand structural and magnetic properties of $Mn_{2-x}Fe_xSn$ compounds from x = 0.00 up to x = 2.00, Δx = 0.25. This opens a potential application range in technological devices, such as thermomagnetic motors and magnetic refrigerators. Samples were prepared by arc melting followed by heat treatment. These compounds were investigated by: Optical Metallography, Scanning Electron Microscopy with energy dispersive spectrometer, X-Ray Diffraction, Rietveld method, and magnetic measurements. The combined results indicate that most samples are biphasic formed by the hexagonal $(MnFe)_2Sn$ and $(MnFe)_3Sn$ phases with $P6_3/mmc$ space group and Ni_2In [1] and Ni_3In-type structure [2], respectively. The $(MnFe)_2Sn$ is the majority phase, except for the sample with x = 2.00. The lattice parameters decrease with the increase of Fe concentration. The Curie temperature (Tc) or transition temperature (TT) behavior has a piece-wise linear TC,T profile. This behavior is accompanied by a large ΔTC,T = 569 K indicating the potential applications of the material in cascade thermomagnetic motors that require a wide temperature span between the hot and cold thermal sources. Although typical ferromagnetic behavior is observed in the M(H) isothermal curves for all samples at applied field between -5 T up to 5 T, an anomalous feature appears for the samples with $0.00 \le x \le 1.00$, at very low applied field (around 0 up to 0.2 T). This result suggests a magnetic ordering different from ferromagnetic. In addition, it is observed a second order magnetic transition (SOMT) for the samples with $0.00 \le x \le 1.75$ and a large temperature hysteresis in the sample.

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ORAL PRESENTATION

Structural and physicochemical properties of complex oxides for energy applications: XRD and XAS studies

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ABSTRACT:

Intermediate temperature solid oxide fuel cells (IT-SOFCs) are considered promising electrochemical devices for direct conversion of chemical energy to electricity with high efficiency, high power density, low emission, and excellent fuel flexibility. The development of a new process for fabrication of electrodes with controlled micro- or nanostructures is critical to improving both ionic and electronic conductivity and electrocatalytic activity at lower operating temperatures. During the last ten years, our group has been focused on the study of structural and physicochemical properties of complex oxides for electrodes (anode and cathode) for IT-SOFC, mainly in anodes based in nanostructured ZrO2-CeO2 or (Gd, Pr, Tb, Ni, Cu)-doped ceria solid solutions with different morphologies (nanopowders, nanostructured tubes and spheres) [1-3] and nanostructured cathodes based in perovskite mixed conductors La0.6Sr0.4CoO3−d (LSC) [4]. Recently, we have recently begun to study materials for protonic ceramic fuel cells (PCFCs) based in perovskite proton conductor BaZr0.7Ce0.2Y0.1O3-d (BZCY72) and BaPr0.9Y0.1O3-d (BPY) [5]. In order to compare the effect of the morphology on the structural and physicochemical properties, in situ SR-XRD, X-ray absorption near-edge structure (XANES) and extended X-ray absorption fine structure (EXAFS) experiments were carried out under controlled atmospheric conditions (5% H2/He; 5% CO/He and 5, 21, 40% O2/N2) in different beamlines at the LNLS. These results combined with those obtained by other techniques -such as high resolution transmission electron microscopy (HRTEM), energy dispersive X-ray spectroscopy (EDS), neutron powder diffraction (NPD), electrochemical impedance spectroscopy (EIS) and catalytic tests for different reactions- which allowed us to understand the different physico-chemical processes that can lead to a substantial improvement of fuel cell devices.

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POSTER PRESENTATION

Structural characterization by SAXS and CryoEM of P9-1, the major viroplasm component of Mal de Río Cuarto virus (MRCV)

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ABSTRACT:

Mal de Río Cuarto virus (MRCV, Fijivirus, Reoviridae) causes the most important maize disease in Argentina. The virus replicates in cytoplasmic inclusion bodies called viroplasms composed by non-structural virus proteins P9-1 and P6. P9-1 is a 45 kDa protein that has ATPase activity, which is able to bind ssRNA and to self-assemble giving rise to homomultimers [1,2]. In this work we will show preliminary structural characterization of P9-1 by using two complementary techniques: Small Angle X-ray Scattering (SAXS) and Cryo-Electron Microscopy (Cryo-EM). Additional experiments included the presence of ATP and/or RNA in order to evaluate changes in the structure. According to CryoEM assays, quaternary structures consisting of 10 and 12 monomers were observed. Experimental SAXS patterns were fitted by using the combination of ATSAS package [3] and python scripting in order to set up each protein complex using Cryo-EM volume information of the monomer as building block. Our preliminary results showed that SAXS patterns were compatible with cylindrical objects composed by the stacking of a five subunits ring. The aim of this work is to provide structural information in order to understand the function of P9-1 complexes in the presence of ATP and ssARN.

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POSTER PRESENTATION

Structural determination of the prototype drug LASSBio-1860

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ABSTRACT:

LASSBio-1860 is an inhibitor of phosphodiesterase type 4 (PDE4). This enzyme plays a major role in modulating the activity of virtually all cells involved in the inflammatory process acting in the control of cAMP levels. This compound was planned and synthesized in Laboratory of Evaluation and Synthesis of Bioactive Substances – LASSBio® as part of a research program to develop a series of compounds with anti-inflammatory activities. As important as the planning and synthesis of new drugs is its structural characterization, since its structure may be related to a biological activity. In many cases suitable single crystals cannot be prepared, and the most used technique that is the single crystal X-ray diffraction (XRD) is not a viable approach for structure determination. Under such circumstances, structure determination must be tackled instead from X-ray powder diffraction (XRPD) data and that is considerably more challenging than structure determination from single-crystal XRD data. In this work we determined the crystal structure of LASSBio-1860 compound using XRPD that has been a very important tool in the structural determination of new synthesized compounds. X-ray powder diffraction data were used for indexing with the Topas Academic v.5 software program, a simulated annealing algorithm implemented in the DASH software program were used to determine the crystal structure of LASSBio-1860, on the basis of previous procedures and a Rietveld refinement of the final crystal structure was conducted using the Topas Academic v.5 software program. Our preliminary results showed that LASSBio-1860 crystallized in a monoclinic system (P21/c) and the crystal structure consists of four formula units per unit cell (Z = 4), thus accommodating four molecules in the asymmetric unit (Z' = 1).

POSTER PRESENTATION

Structural study of the influence of the electrical field in crystals by the dynamical theory of x-ray diffraction

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ABSTRACT:

The study of crystals under the effect of an external electric field is the key to the origin of several physical properties, in addition, the study of the structural response to the field application is one of the major challenges of modern structural resolution. For such studies, X-ray diffraction is one of the main characterization techniques. In order to explain the diffraction results, normally use the kinematical theory of diffraction, but over time, studies have shown that this theory is not totally correct. Another theory was then used, the dynamical theory of diffraction, unlike kinetical theory, this new theory is more physically complete. In this work we will observe the structural behavior of KDP crystal under the effect of an external electric field using the dynamic theory of X-ray diffraction.

POSTER PRESENTATION

Structure and dynamics of fluid nanofilms: study of peptide-membrane interactions with classical and new techniques

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ABSTRACT:

Bacterial cell membranes are important target for new antimicrobial strategies based on amphipathic peptides because occurrence of resistance is slower than in protein-based targets. Knowing structural details of peptide-membrane interactions is of utmost importance for understanding the mechanism of action of antimicrobial peptides, as nonbilayer structures can arise when bilayers encounter peptides and as a result, pores or carpeting are formed. Even though antimicrobial peptides have been studied intensively in the last years, there's still lacking a molecular view of the dynamics and structural determinants of bacterial bilayer destabilization. In this regard, x ray scattering techniques provide information on the lipid phase and its tridimensional dimensions in biologically relevant aqueous dilutions. We studied membrane model systems resembling Gram positive and Gram negative bacterial cells and their interaction with antimicrobial peptides enterocin CRL35 and microcin J25 active against Listeria and Escherichia coli cells, respectively. Our methods included x ray scattering at small angle in liquid samples at different temperatures consisting in liposomes made of PG and PE at different proportions according to the model coincubated with peptides a 20:1 lipid:peptide ratio. The results indicated that control samples formed uncorrelated bilayers due to repulsive forces of the negatively charged lipids but in the presence of peptides, correlated bilayers and even non-bilayers structures are formed. By using oriented multilamellar stacks of membranes, we also studied how mechanical properties of lipid bilayers at full hydration are changed by measuring diffuse x ray scattering at grazing incidence, applying liquid crystal theory for calculating bending moduli Kc. The two techniques provide useful complementary information for understanding molecular details and dynamics of the mechanism of action of antimicrobials in membranes.

POSTER PRESENTATION

Structuring of iron oxides magnetic nanoparticles used in magnetic hyperthermia experiments: effects of size and coating

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ABSTRACT:

Iron oxide magnetic nanoparticles (MNP) have been widely used in both, diagnostic and therapy for cancer disease research. One of the most important applications is magnetic hyperthermia therapy, in which, the cells are killed by increasing temperature using MNP as electromagnetic to heat energy transductors. For this, MNP properties must be optimized in order to obtain a higher heat quantity using low MNP doses [1]. In this work, Small Angle X-Ray Scattering is used to obtain morphologic and structural information from aqueous colloidal suspension of MNP synthesized following the chemical co-precipitation method [2]. The synthesis temperature was fixed in 25, 40, 60 and 80oC in order to achieve different particle size distributions. The MNP were superficially modified using citric acid and dispersed in water. A spherical form factor and a mass-fractal structure factor [3] were needed in order to fit all the SAXS patterns. Results showed that the lower synthesis temperature the higher particle size (radius between 2.7 and 4.1 nm) and the higher heat dissipation at radiofrequency fields of 100 kHz varying amplitude. A partial aggregation of nanoparticles was also determined by fitting SAXS patterns with almost spherical aggregates (df ≈ 2.9) with sizes around 20 nm. Better heat dissipation results were achieved when the coating was changed from citric acid to sodium citrate and poly ethylene glycol without significant change in the structural parameters found with SAXS.

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POSTER PRESENTATION

Study of anti-tumorigenic actions of Omega-3 fatty acid in a murine mammary gland adenocarcinoma by micro-XRF analysis

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ABSTRACT:

A total of 50 samples of dried neoplastic tissues fixed in paraffin blocks were evaluated by micro-XRF. The neoplastic tissues were previously implanted in 50 male and female BALB/c mice randomly distributed among two dietary groups: a normal diet (control) and a diet rich in ChO. The micro-XRF was calibrated to quantify traces of Ca, Fe, Cu and Zn on paraffin blocks. Standard materials with known amount of Ca, Fe, Cu and Zn were specifically developed for the quantification process. The standard materials were homogeneous solid solutions with uniform metal distributions at micro-level scale and similar attenuation properties than dried soft biological tissues fixed in paraffin blocks. Thin slices of 30 microns of neoplastic tissues were analyzed by micro-XRF using synchrotron white beam focused at 20 microns by a KB optic. Scanned rectangular areas with edges of few millimeters were carefully selected with help of the histological image of the contiguous tissue. The results showed a significant decrease of metal concentrations in the neoplastic tissues for mice fed with ChO diet. A previous research showed that ChO inhibits growth and metastasis in a murine mammary gland adenocarcinoma model [1]. These results are correlated with the observed decrease of metal concentrations which gives us the possibility to develop an improved model of the anti-tumorigenic actions of Omega-3 fatty acid present in ChO diet [2].

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POSTER PRESENTATION

Study of films of natural clays by X-Ray Reflectometry (XRR)

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ABSTRACT:

Clay shale, particularly those predominantly composed of smectite have a high swelling potential. Often the swelling of clays shale is not desirable, and it has been recognized as one of the major causes for formation damage in hydrocarbon reservoir [1]. Natural clays are one of the main components of these reservoir rocks in Argentina unconventional deposits. Understanding their physical, structural and chemical properties is necessary for optimization of production process of hydrocarbons stored in these types of rocks [2]. The development of new analytical tools that overcome conventional analysis techniques is sought. In this context we propose using XRR technique for the study of this type of samples. In this work, clays synthetic patterns were used. A method for preparation of thin films was developed, analogous to the method of preparation of mesoporous oxides [3]. The deposit was optimized using the spin coating technique and homogenous and transparent films were obtained. This method of preparation has allowed the adaptation of natural clays to be studied by means of the X-Rays Reflectometry technique. Some of the measures were done in LNLS. From this work it was possible to estimate the density of clays quickly and easily.

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POSTER PRESENTATION

Study of metal-based catalyst using a multi-purpose catalytic cell

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ABSTRACT:

In this work, we describe the use of an in situ cell designed for XAS and XRD studies to investigate the different species formed on metal-based catalysts [1]. The cell is composed by a stainless steel body with exchangeable heads (for solid-gas, solid-liquid and electrocatalysis) aiming to cover most of the standard experiments emphasizing on the quality of the spectroscopic data. Moreover, a movable control system is available in order to allow an easy transportation to other laboratories or large-facilities. Because of that, experiments can be easily reproduced and the results compared with those obtained in synchrotron beamlines. Up to now, several metal-based catalysts have been studied by our group during samples activation or catalytic conditions [2], in experiments involving either solid-gas or solid-liquid setups. For example, our group has tested new Pd catalysts based on structured carbon materials that have shown high activity for the catalytic reduction of bromates under neutral pH, at room temperature and with low Pd content. In this particular case, the sample is mounted on the stainless steel holder that is placed inside of a cavity through which the reaction liquid is continuously flowing. Then, a close contact between the liquid and the solid is obtained, working as a real reactor. All the Pd/C catalysts show significant bromate conversion, while EXAFS data analyses of the Pd/C samples under operating conditions show that Pd remains as metal along the whole experiment regardless of the experimental conditions. Also, the activation of precursor-dependent Pd nanoparticles supported on alumina and activated carbon has been studied using the solid-gas assembly. In this latter we demonstrated by XAS that varying the metallic precursor it is possible to obtain Pd nanoparticles of different nuclearity due to the thermal stability of metal precursors and consequently, their unlike decomposition pathways [3].

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POSTER PRESENTATION

Study of phase transition in NVP-SA co-crystal

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ABSTRACT:

Nevirapine (NVP) is an antiretroviral drug used to treat HIV infection [1]. It is a class II drug [2], presenting low solubility, which leads to problems during formulation and a low bioavailability. In order to improve the drug solubility and other physicochemical properties, a co-crystal of NVP and salicylic acid (NVP-SA) was obtained by our group. This co-crystal has been described in the literature and its structure was determined only at low temperature (100 K) [3]. These data were used as reference to characterize NVP-SA. Differences were observed between our data and the reported results [3]. Thus, the goal of the present study was a minacious study in order to understand those differences and determine the crystal structure in the range of temperature from 300K to 100K. Single X-ray diffraction (SXRD) was used to determine crystal structure at 300K and 100K. Synchrotron X-Ray Powder Diffraction (SXRPD) data were collected at XRD1 beamline [4] in a continuous range of temperature from 300K to 100K. Solid-state Nuclear Magnetic Resonance (ss-NMR) was used to corroborate the crystal structure obtained by SXRD. The single crystal data at 300K and 100K were used to refine the SXRPD data collected at the temperature range. A phase transition of the NVP-SA was observed from the low-temperature structure to high temperature one. Finally, the differences between our results and the literature ones were due to the high-temperature structure of NVP-SA is different from the low temperature one.

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POSTER PRESENTATION

Study of the structural and optical properties of the BaTiO3@CaF2 system

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ABSTRACT:

Many systems based on perovskite have shown promising results for the application in solar cells with or without dye sensitization [1]. One of the main relevant challenges for non-sensitized solar cells is their low photocurrent generation [2]. In this sense many methodologies are implemented to avoid the electron-hole recombination and hence improving the photocurrent for non-sensitized systems. The reduction of electron-hole recombination can be performed by the spatially separation of them in core and shell locations by the band alignment of two semiconductors with Fermi level located at different energies. The performance of solar cells can be optimized with the insertion of the system with rare Earth ions. The presence of dopants (such as Eu, Dy, Sm, Tb) can act in the down/up conversion process, increasing the spectral response, including the UV to IR range. Along with the defects caused by doping, the method of obtaining the decorated system can influence the disorder of the matrix. In this context, a study was conducted to obtain Barium Titanate electrodes decorated with fluorides doped with rare earth ions. These systems were crystallized with short times of synthesis by means of the hydrothermalization method assisted by microwave. First, X-ray diffraction analysis was performed on samples of the composite system with and without the presence of dopants. In both systems, the XRD patterns reveal the formation of two phases, belonging to the core and the decoration. The scanning electron microscopy images show differences between the systems depending on the time of synthesis employed, not differentiating changes as the rare earth ion varies. XANES and EXAFS measurements were performed in transmission and fluorescence modes combined with XEOL detection at the edges of the matrix elements (Ba, Ti and Ca) and dopants ions (Eu, Sm and Dy). The studies provided information on the conversion intensity of luminescence down/up influenced by the presence of doping.

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POSTER PRESENTATION

Sulfur speciation in a tropical soil under long-term no-till as a function of lime and phosphogypsum application

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ABSTRACT:

Sulfur (S) is presented mostly in organic forms (> 95 %) in the top soil of well-drained soils [1]. Concentrations of S are closely related to organic carbon content, and the maintenance of adequate soil organic matter (SOM) levels can provide S gradually to the plants via mineralization. The inadequate use of soils can deplete the SOM content, and the No-Tillage system (NT) is an alternative to increase it. A field-scale study has been carried out in São Paulo State, Brazil, in area under NT for 15 years, with four treatments: control (no amendments), lime (6,300 kg ha-1), phosphogypsum (10,000 kg ha-1) and lime + phosphogypsum (6,300 kg ha-1 + 10,000 kg ha-1, respectively). Previous study conducted in the area shows a net increase of SOM content throughout the soil profile [2]. Soil samples were collected, frozen and lyophilized before finely ground in a glove box with N2 environment in order to prevent oxidation of organic forms of S[3]. In the first set of analysis conducted at SXS beamline of the LNLS, we acquired data for all treatments in the in depths of 0 – 5, 5 – 10, 40 – 60 and 90 – 120 cm. A decrease in intensity of the peak at 2472 eV is seen with increasing soil depth, which may be an indicative of the decrease of reduced organic sulfur forms (amino acids such as methionine and cysteine). This feature agrees with the sharper white line signals presented in spectrum from 90 – 120 cm of lime + phosphogypsum treatment at 2482 eV, which represent oxidized forms of sulfur. We collected samples from a native forest which had a total S content in the same range of the cultivated soil previously analyzed. At the second beamline time we intend to acquire spectra from the native forest soil and of the reference compound chondroitin sulfate, a major form of organic sulfate found in soils, to improve linear combination fitting (LCF) modeling. Our goal is to understand the dynamics of S with application of lime and phosphogypsum when compared to the native forest.

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POSTER PRESENTATION

Surface damage in cystine, an amino acid dimer, induced by keV ions

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ABSTRACT:

We have studied the interaction of an ion beam (17.6 keV F-) with cystine, a dimer formed by the binding of two cysteine residues. Cystine can be considered as an ideal prototype for the study of the relevance of the disulfide (-S-S-) chemical bond in biomolecules. For the sake of comparison, the amino acid cysteine has also been subjected to the same experimental conditions. Characterization of the samples by XPS and NEXAFS shows that both pristine cystine and pristine cysteine are found as a dipolar ion (zwitterion). Following irradiation, the dimer and the amino acid show a tendency to change from the dipole ion form to the normal uncharged form. The largest spectral modification was observed in the high resolution XPS spectra obtained at around the N 1s core level for the two biomolecules. The 2p sulfur edge spectra of cysteine and cystine were much less sensitive to radiation effects. We suggest that the disulfide bond (-S-S-) remains stable before and after irradiation, contributing to the larger radiation stability of cystine as compared to the amino acid cysteine.

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POSTER PRESENTATION

Surface propensity of atmospherically relevant amino acids studied by XPS

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ABSTRACT:

Amino acids constitute an important fraction of the water-soluble organic nitrogen compounds in both marine and continental aerosols and are involved in many processes in the atmosphere [1]. In this work, we applied XPS to study aqueous solutions of four amino acids: glycine, alanine, valine and methionine in their zwitterionic forms. These amino acids were selected as they have quite different size and side chains and may therefore have quite different propensities to the water-vapor interface. We found that amino acids with hydrophilic side chains and smaller size, GLY and ALA, tend to stay in the bulk of the liquid, while the hydrophobic and bigger amino acids, VAL and MET, are found to concentrate more on the surface. We found experimental evidences that the amino acids have preferential orientation relative to the surface, with the hydrophobic side chain being closer to the surface than the hydrophilic carboxylate group [2]. The orientation of the amino acid on the liquid surface creating a hydrophobic layer will reduce the probability of an incoming gas phase water molecule to be accommodated into the droplet. The observed amino acid surface propensity has implications in atmospheric science as the surface interaction play a central role in cloud droplet formation, and they should be considered in climate models [3].

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POSTER PRESENTATION

Surface treatments for components used in the gas and petroleum industry – a XAS study.

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ABSTRACT:

In Gas and Petroleum Industry, the effect of premature damage of the metal components due to combination of the corrosion-abrasion phenomena causes substantial economic losses. One of the most common types of damages in the ferrous materials (pipes, encasings, pistons, etc.), occurs when micro fractures are generated by the extreme thermal and pressure conditions. In order to mitigate these damage mechanisms, we have begun to evaluate a series of surface treatments involving the generation of an interface between different materials. We focus on a combination of Plasma Nitriding (PN) and Physical Vapor Deposition (PVD). [1] The main advantage of these techniques is that they generate a gradual transition of hardness, governed by the diffusion mechanism of nitrogen in the base metal, resulting in a surface hard layer of the same material, reaching a hardness of 3000 HV. Although this type of treatment has great advantages with respect to the obtained, using the thermal spray technique, it has been observed that, under certain experimental conditions of nitriding, deterioration in the resistance to the corrosion is found. The decrease in resistance to the corrosion could be explained by an excessive formation of chromium nitride (CrN). The formation of this kind of nitrides is very sensitive to the temperature, time and atmosphere of the nitriding treatment, as well as to the type of steel used. For this reason, it is extremely important to obtain an estimation of the amount of the different nitrides presents. To check our hypothesis that the presences of chromium nitrides are responsible for the premature corrosion, we perform XANES and EXAFS measurements to be correlated with electrochemical characterizations (used to determine the corrosion resistance), with the different nitriding conditions and the steel used (AISI 316L and AISI 420). [2]

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POSTER PRESENTATION

Synchrotron-based x-ray computed microtomography to evaluate lime application effects on soil aggregate properties

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ABSTRACT:

The hierarchical organization of aggregates in soil is responsible for the presence of inter and intra-aggregate pores. This research aimed to investigate effects of soil surface liming, considering lime rates of 0, 10 and 15 t ha-1, on the intra-aggregate porous system of soil aggregates with equivalent diameters of 2-4 and 1-2 mm, from 0-10 (A) and 10-20 cm (B) soil layers. These aggregates were selected by the wet sieving method carried out for determination of the mean weight diameter. Synchrotron-based computed microtomography (μCT) of aggregates were analyzed in terms of porosity, connectivity, tortuosity, and fractal dimension. Additionally, x-ray fluorescence was used to evaluate the elemental composition of the soil aggregates. All liming effects were concentrated at layer A, where calcium percentage was elevated in aggregates from 1-2 mm class as compared to those from 2-4 mm class. Accordingly, the physical parameters studied were generally more affected in the case of smaller aggregates (1-2 mm). Liming decreased total porosity and increased tortuosity of pores for 1-2 mm aggregates, but it did not affect pore connectivity under any of the circumstances. In addition, liming decreased the fractal dimension in aggregates from both 2-4 and 1-2 mm classes, which is in line with the fact that larger pores were replaced by smaller ones in 1-2 mm aggregates, as found via both quantitative and qualitative analyses.

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POSTER PRESENTATION

Synthesis and characterization of lipopeptide nanostructures: application as catalysts for aldol reactions in aqueous medium

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ABSTRACT:

Research on self-organizing properties of peptides has emerged in recent years as an active and diversified field involving either fundamental analysis or applications in biotechnology and materials science. These materials exhibit intrinsic multi-level organization, and this structural multiplicity generates several types of intra- and intermolecular interactions making them very attractive as a model for the study of self-organization phenomena occurring in living arrangements and catalytic processes [1]. Therefore, our objective in this present work is to study the self-assembling processes of amphiphilic peptides aiming their application in stereoselective catalysis. In general, new amphiphilic peptides were synthesized and characterized and evaluated for their efficiency in the catalysis of direct aldol reactions in water. The studied lipopeptides have the amino acids L-proline, L-arginine or the synthetic amino acid GCP (Guanidiniocarbonyl pyrrole) [2], L-tryptophan and L-glycine, covalently linked to one or two long aliphatic chains (16 or 18 carbon atoms), leading to molecules with surfactant characteristics. Cryo-TEM and SAXS studies showed the formation of distinct morphologies, such as spherical and cylindrical micelles, and bilayers. These compounds were then applied as supramolecular catalytic systems in the stereoselective direct aldol reaction between cyclohexanone and p-nitrobenzaldehyde in water as the solvent. The produced nanostructures acted as excellent catalytic systems in the performed tests, in which superior conversions (> 99%), high diastereoselectivities (ds= 94:6) and excellent enantioselectivities (ee= 92%) were obtained. Our results allowed a systematic study that contributed to understanding the organization of these materials and demonstrated the potential of lipopeptide arrangements in the catalysis of aldol reactions promoted in an environmentally-friendly manner.

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POSTER PRESENTATION

The effect of barium in Ni-La based catalysts on dry reforming of methane

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ABSTRACT:

Several studies on dry reforming of methane (DRM) have proposed that the nature of the support effect the mechanism of carbon species oxidation [1, 2]. However, for nickel based perovskites, few investigations have been done with barium as a promoter [3]. Therefore, the aim of this study was evaluating Ni-La based catalysts preparation method and the impact of barium on carbon deposition over DRM . In situ XRD showed the phase evolution when the catalysts were exposed under CH4/CO2 from room temperature to 800 ºC. For all catalysts, metallic nickel was oxidized to nickel oxide when exposed to reaction atmosphere. This suggests that CO2 on the feed was responsible for the nickel sites oxidation. Comparing the catalysts obtained via LaNiO3 and La0.8Ba0.2NiO3 it was verified that the nickel sites present on LaNiO3 are more susceptible to oxidation. At 289 ºC, the intensities of the peaks associated to La2O3 phase decreased in intensity and the peak (111) attributed to metallic nickel disappeared, due nickel oxidation to NiO. At 463 ºC, the peak (111) attributed to metallic nickel becomes detectable, suggesting the catalyst was being regenerated. For the catalyst obtained from La0.8Ba0.2NiO3, the oxidation takes place at higher temperatures, starting from 432 ºC to 610 ºC. Nonetheless, the nickel sites were not totally oxidized, suggesting a partial oxidation process, which was characterized only by the decreasing of the metallic nickel peak (111). This behavior considered that BaCO3, during reduction process, may cover the nickel sites, at least partially, blocking such sites and making them less susceptible to oxidation. It is possible to explain the higher resistance of barium-content catalysts to carbon deposition observed in table below. Thus, the CO2 present on the feed may promote the BaCO3 regeneration.

Catalyst	$LaNiO_3$	$La_{0.95}Ba_{0.05}NiO_3$	$La_{0.9}Ba_{0.1}NiO_3$	$La_{0.8}Ba_{0.2}NiO_3$
$mg_{carbon}/g_{catalyst}$	1.64	0.08	0.16	0.03

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POSTER PRESENTATION

The electronic structure of Ba[Fe(1-x)Mn(x)]2As2 (x = 0.0, 0.05 and 0.1) probed by XAS at the Fe and As K-edges: angular dependence

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ABSTRACT:

A very diverse set of material families is related to the field of the Iron based superconductors, each of which are represented by a parent compound, as the BaFe2As2 family. In spite of a great diversity of physical properties, there is a common thread to all these materials: the relevance of the structural and electronic properties of the FeX layers and of the FeX4 (X = As, Se, Te) tetrahedral coordination complexes. Their investigation underlines a major division in the field: should we understand the physics of this materials in terms of localized or itinerant electronic states? In this work we investigate the electronic and structural properties of the (Fe1-xMnx)As4 complexes in Ba(Fe1-xMnx)2As2 (x = 0.0, 0.05 and 0.1) by means of Fe and As K- edge X-ray absorption spectroscopy. In general, the Fe and As K-edges XAS spectra contain information concerning the orbital mixing, electron exchange and site disorder of the (Fe1-xTMx)As4 (TM = transition metal) complexes, all of which are important parameters in the context of the magnetic and superconducting properties of the iron based superconductors.

POSTER PRESENTATION

The investigation of fossil preservation by SR-XRF

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ABSTRACT:

Synchrotron Radiation (SR), particularly micro-X-ray Fluorescence (μSR-XRF), has been employed for elemental quantification and distribution, respectively by point and mapping analysis, in addition to the characterization of oxidation state (by μXANES) in several fossils from different periods of the history of the Earth. Investigators have been mainly concerned in testing whether the primary signal of several elements of interest has been kept unchanged over geological processes. In the core of this research area, Geobiology–an interdisciplinary field that integrate Geology and Paleontology–aims to unravel how the elemental and mineral compositions change over biological and geological processes of fossil formation. The aim of our research group is to employ μSR-XRF to test geobiological hypotheses concerning fossilization, mainly of Brazilian specimens. Over the last few years, we have used the XRF beamline of the LNLS to investigate the preservation of soft tissues of fossil insects and fish from the Crato Formation (Ceará, ca. 110 Ma) and, more recently, we have started to investigate the preservation of Corumbella werneri, a cnidarian from the Tamengo Formation (Mato Grosso do Sul, ca. 541 Ma). μSR-XRF yielded the spatial distribution of elements, such as Fe and Zn, at insect and fish samples, which shed light on the role of bacteria for the fossilizations of these specimens. Moreover, μXANES has been, preliminary, applied to elucidate the oxidation state of those elements identified by μSR-XRF. The continuation of the forementioned research line at the Carnaúba beamline of the Sirius synchrotron light source will enable us to benefit both from the nanoscopic beam and from the beamline wide energy range, to respectively (1) map either very small and/or very thin fossils with high spatial resolution, and (2) measure light elements, such as Al, Si, S, and P. Ultimately, these achievements would enable us to test taphonomic hypotheses.

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POSTER PRESENTATION

The use of total reflection of X-ray fluorescence to study possible heart disease induced by ionized radiation

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ABSTRACT:

The ionizing radiation can induce a variety of biological effects depending on the physical nature, duration, doses and dose-rates of exposure. However, the magnitude of health risks for low doses and dose-rates (below 100 mSv and/or 0.1 mSv min−1) is still debated due to a lack of direct human evidence. Sequential single element determination techniques are common in investigations using body fluids and tissues. More information from these samples could be obtained by simultaneously using multi-element techniques, such as total-reflection X-ray fluorescence (TXRF). There is great interest in identifying biological markers related to ionizing radiation exposure for the detection of people exposed to high or low radiation doses, who could be at risk of developing late side effects due to this exposition. To our knowledge, there has been no identified biomarker. The aim of this work was to quantify the trace elements and major elemental components (S, Fe, Zn, K and Ca) contained in heart tissue of Wistar rats after exposure to low and high doses of ionizing radiation (0.1 Gy and 2.5 Gy, respectively). The obtained results showed that the irradiated animals with low dose had a significant ($p< 0.05$) alterations for the concentrations of some key elements in cardiac tissue when compared with animals that received high doses of radiation. This can indicate that the elemental alterations observed in the LV tissues is the beginning of the late side effects, like apoptosis and necrosis, in patients submitted to HD radiations. More studies must be done to confirm this hypothesis. Therefore, those analyses are important to better understand the mechanisms involved in the ionizing radiation induced damage and enhance the possibility to predict and prevent the side effects observed in people exposed to different levels of radiation, due to treatment, diagnosis or accident.

POSTER PRESENTATION

Three dimensional structure determination of the native lectin from Platypodium elegans seeds

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ABSTRACT:

Carbohydrates, similarly to nucleic acids and proteins can code information in their structure but, differently from these molecules, form highly branched polymers, besides presenting anomeric carbons in their constituent monomers, which allows a more complex and rich information storage in the form of a glycocode in their structure. This code is widely used by cells in several processes such as cancer and inflammation. Lectins are proteins capable of deciphering this glycan code by binding in a highly specific and non-catalytic way to sugars. The crystallographic study of lectin allows a complete study of the interaction among these protein with their glycan ligands, corroborating to the decyphering of the glycocode. The native lecton nPELa, isolated from seeds of Platypodium elegans, Dalbergiae tribe, was isolated by affinity chromatography, crystallized and structurally characterized by X-ray diffraction crystallography and bioinformatics tools. The obtained crystals diffracted to 1.6 Å resolution, and nPELa structure was solved through molecular substitution. nPELa has a metal binding site and a conserved carbohydrate recognition domain (CRD) similar to other Dalbergieae tribe lectins, such as PAL (Pterocarpus angolensis) and CTL (Centrolobium tomentosum).

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ORAL PRESENTATION

Three-dimensional neuronal architecture revealed by high-resolution synchrotron-based X-ray microtomography

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ABSTRACT:

The study of brain cytoarchitecture with a cellular resolution and in 3D, persists as a great challenge. X-ray microtomography (μCT)has showed to be a valuable non-destructive tool for imaging a broad range of samples, from dense materials to soft biological specimens. Although 3D images of nervous system have been illustrated with different imaging techniques, neuronal network, morphology and organization in situ are still a challenge. Thereby, we aimed to develop a non-destructive imaging technique that allows a high-resolution visualization of neurons in the brain in 3D, combining synchrotron-based X-ray microtomography with the Golgi-Cox impregnation histology protocol. Briefly, mouse brain structures were removed, and subjected to a mercury-based impregnation of neurons using Golgi-Cox fixative. After paraffin imbibition, brains were subjected to synchrotron-based μCT. To compare a healthy brain to a pathological condition, we took hand of a protocol that leads to neuronal death based on the administration of pilocarpine (280mg/kg), what leads to a status epilepticus condition, and we compared with non-treated animals. After treatment, animals were euthanized, and groups of data were compared. $P<0.05$ was considered statistically significant. The technique shown opens a new way for studying complete 3D individual neuron morphology and distribution. X-ray imaging of mercury-impregnated neurons allowed the complete reconstruction of neurons from different regions of the brain, without the need of tissue slicing. In addition, we analyzed the damage induced by pilocarpine in frontal cortex and medium part of the hippocampus. As expected, we observed a significant decrease in cell density in frontal cortex, but specially in the hippocampus in pilocarpine-treated animals. Therefore, X-ray imaging technique allows not only the study of in situ neuromorphology with a single cell resolution, and in a non-destructive manner, but also the quantification of cell number.

POSTER PRESENTATION

Ti rich NiTi Alloy Thermomechanical Behavior Under Compression at High Temperature

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ABSTRACT:

The focus on these thermomechanical processes has been to obtain the appropriate material shape, to control transformation temperatures, to improve the alloy properties, and to preserve shape memory effect and superelasticity in NiTi alloys, which are widely used in biomedical and engineering applications. It is very important to understand the hot deformation behavior of NiTi alloy because at this stage we can promote the functional properties. The hot deformation promotes the metallurgical transformations and the microstructural changes that will be further improved by final cold deformation, influencing the materials final mechanical properties and their applications. The most important phenomena during the hot working are dynamic and subsequent static recrystallization or recovery. We aim to perform the structural characterization of the hot forging steps sample - NiTi shape memory alloys (SMAs). We simulate the thermo-mechanical treatment (hot forging) in Ti rich NiTi alloy by compression test. All the samples that were analyzed are correspondent to each step of the rotary forging process during hot deformation process. To understand the actual condition of the samples we need to study the simulated condition during the forging process. Ni-51%at.Ti alloy, were produced by melting in a vacuum arc remelting (VAR) with copper crucible and previous homogenization at 950ºC / 120 minutes. They were submitted to thermomechanical process on Gleeble machine, at 850ºC, installed in Brazilian Synchrotron Facility (LNLS). Information during loading and unloading of the samples were acquired to correlate the structural modifications (X-Ray Diffraction - XRD) during the four steps of hot forging with two distinct deformation rates. The microstructural aspects before and after tests on Gleeble at XRD beam line were observed on Scanning Electronic Microscopic. These results revealed a significant and effective homogenization.

ORAL PRESENTATION

Tunning the oxygen vacancy population of CeO2-x (0 < x < 0.5) nanoparticles

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ABSTRACT:

Oxygen vacancies are one of the most active defects existing at the surface of metal oxides. These defects play a central role on determining the catalytic properties of nanoparticles (NPs). Tuning the oxygen vacancy population of CeO2-x (0 < x < 0.5) NPs represents the possibility of controlling the catalytic activity [1]. In this work, the oxygen vacancy population of CeO2-x NPs was monitored by means of the combination of synthesis parameters and CO reducing atmosphere [2]. High surface area CeO2-x NPs were synthesized with different structural and electronic properties. The synthesized samples were characterized with Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), X-ray Diffraction (XRD), and N2 adsorption-desorption isotherms. Then, the surface properties of the NPs were probed in situ with the samples exposed to a CO reducing atmosphere at different temperatures (ranging from RT to 500 °C) and CO gas pressure of 1 mbar by means of Near Ambient Pressure X-ray Photoelectron Spectroscopy (NAP-XPS) measurements. These results are complemented by Synchrotron Lab measurements such as in situ time resolved X-ray Absorption Spectroscopy (XAS) and in situ XAS measurements at Ce L3 edge during the NPs reduction by a CO atmosphere. Compared to a commercial CeO2 standard, an improved reduction tendency is observed for the synthesized NPs. The reduction temperature (TR) and Ce(III) fraction values reached can be as low as 108 °C and as high as 0.92, respectively. The in situ measurements results also point to a control of the reduction temperature and Ce(III) fraction values as a function of the size, surface area, porous size distribution, initial Ce(III) fraction and hydrophobicity of the NPs. The results provide a valuable route to design CeO2-x NPs with controlled oxygen vacancy population, then making smart NPs to be applied in the industry.

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ORAL PRESENTATION

Understanding hybrid organic-inorganic perovskite formation during spin coating through the in situ time resolved GIWAXS

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ABSTRACT:

Organic inorganic hybrid perovskites (OIHP) is the most promising material achieving high power conversion efficiency (PCE) and low cost in solar cells. The high-quality optoelectronic properties combine with solution process using low-cost materials making this material so hopeful. The OIHP is generally labeled as an ABX_3 compound, where A is a monovalent cation, as methylammonium (MA), formamidinium (FA), or cesium (Cs+), B is a divalent metal, as lead (Pb2+) or tin (Sn2+), and X is a halide anion, bromide (Br-) or iodide (I-). The most used method to prepare perovskite is the solvent engineering method that consists to prepare a solution with perovskite precursors in a mixture of dimethyformamide (DMF) and dimethylsulfoxide (DMSO). During the spin coating process, one antisolvent (e.g. chlorobenzene) is dropped onto the substrate to remove the excess of DMF and DMSO. Here we used in situ time-resolved grazing incidence wide angle X-ray scattering (GIWAXS) during the spin coating step to monitor and get insights into the perovskite formation. For that, a spin coater was installed, for the first time, at the XRD2/LNLS beamline inside a closed assembly and the antisolvent injection was done using an automatic syringe coupled to a hose. The results revealed that relative humidity (rH) have a big influence in the solvent evaporation during the spin coating and higher amount of DMSO extend the duration of colloidal gel formed during the process. The colloidal gel is crucial to get good morphology of the perovskite films in mild condition of rH (20%) and the time to inject the antisolvent have no big influence in the morphology when the antisolvent is injected onto the colloidal gel. The results also revealed that Cs0.17FA0.83Pb(I0.83Br0.17)3 perovskite formation occurs through intermediates (polytypes 2H and 4H)2 without thermal annealing.

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ORAL PRESENTATION

Unraveling the atomic structure of Fe Intercalated under graphene on Ir(111)

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ABSTRACT:

During the last years, graphene and others 2D materials have attracted much attention from the scientific community due to a large number of possible technological applications of these materials. In particular, there is a large demand to functionalize graphene through metal deposits and intercalation and in several cases the system present large corrugations. The corrugated graphene on metal substrates is naturally a complicated system and determine its atomic structure is a challenge from the theoretical and experimental point of view [1,2]. Herein [3], the interaction of Fe deposited on graphene grown on Ir(111) was studied in detail to better understand the kinetics and thermodynamic parameters involved in the growth, intercalation, and oxidation of Fe ultrathin films on and under graphene. The study has combined a multiple technique approach that allows extracting at once the chemical, topographic and the precise atomic structure of the system submitted to different conditions of growth and atmospheric environment. For instance, scanning tunneling microscopy (STM) measurements allowed us to follow the formation of Fe nanostructures during Fe deposition and intercalation. Synchrotron-based high-resolution photoelectron spectroscopy (HR-XPS) untangled the different chemical environments for C-Fe bonds. We have also used photoelectron diffraction experiments to unravel site-specifically the atomic structure of the intercalated Fe under graphene. Oxidation experiments were also performed for samples prepared under different conditions which revealed that indeed one can set the thermodynamic parameters to selectively protect or oxidize the intercalated/supported materials which open interesting possibilities to engineer complex metal-oxide graphene-based devices.

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ORAL PRESENTATION

Unveiling early microfossils with integrative imaging techniques

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ABSTRACT:

Microfossils are morphological biosignatures of microorganisms preserved in the geological record and comprise the oldest direct record of life on Earth. However, due to the high level of geological processing over billions of years, the micrometric size and the chemical composition (highly dense and homogeneous rocks), the study of such structures has been limited, and many questions about their morphology, preservation and specially biogenicity remain unsolved. The use of nanometric resolution imaging techniques based on different physical phenomena has been proposed as a potential approach to overcome these limitations, exploring both the morphology and chemical composition of the samples, preferably in a non-destructive way. Here we present our results applying Ptychographic X-ray Computed Tomography [1] at cSAXS beam line of the Swiss Light Source to investigate some of the oldest microfossils on Earth. We obtained 3D nanometric resolution with electron density contrast, providing together the ultrastructure and geochemical composition of these fossil microbes. These results add significant contribution to the debate of the biogenicity for morphological biosignatures, representing a novel approach to resolve putative terrestrial fossilized microbes, as well as from meteorites and near-future sample return missions from Mars. We also present the perspectives of correlating 3D imaging with chemical composition in the nanoscale at Sirius, and how it may contribute for the study of the controversial records of fossilized life.

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ORAL PRESENTATION

Use of soft X-ray scattering for 3D characterization of magnetic nanostructures

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ABSTRACT:

Following on from earlier work [1], we present results from an experiment conducted in 2017 at Soft X-ray Scattering beamline of LNLS where "sandwich" thin film samples of permalloy/CoPd/permalloy were studied using resonant soft X-ray scattering in an attempt to study their 3D domain structure to nanometer resolution. Such samples are of interest because of the interaction between the isotropic magnetic behaviour exhibited by the permalloy, and the strongly anisotropic nature of the CoPd multilayers. Scattering measurements were made at the absorption edges of both Cobalt and Nickel at a range of different incidence angles so as to generate a quasi-tomographic dataset, with the differences in energy used to "zoom-in" on the domain structure in either the CoPd layer or the permalloy layers. The subsequent analysis is based upon the generation of a model via micromagnetic simulations, the simulation of X-ray propagation through such simulated magetisation patterns and the matching of the micromagnetic simulation parameters with the experimental data. As a result, we are able to observe the effect of the permalloy layer on the domain pattern in the CoPd thin film both in terms of domain size and out of plane vs. in-plane orientation. This work was substantially limited by the low brightness of the LNLS source, which restricted our work to statistical characterisation of the domain structure. However higher brightness sources such a SIRIUS will allow these experiments to be performed using a coherent microprobe which will allow the technique of ptychography to be employed to obtain full real-space characterisation of the materials to be obtained with minimal additional effort. The final part of the presentation will discuss the technical requirements for the use of soft X-ray ptychography for such studies.

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ORAL PRESENTATION

XANES study of cerium oxidation state of ceria-based catalyst in catalytic operating conditions

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ABSTRACT:

In light of developing efficient anodes for intermediate temperature solid oxide fuel cells (IT-SOFC), materials with mixed ionic-electronic conductivity and excellent redox properties are sought-after. The main objective of this work is to study the effect of temperature and fuel composition (H_2 or CH_4) in the reducibility of Ce-Sc-Zr-O mixed oxides in reducing atmospheres. Samples were synthesized using two different routes, namely glycine/nitrate combustion route (GC) and citrate complexation route (CIT). Samples were prepared with the following compositions: $Ce_{0,9}Sc_xZr_{0,1-x}O_\delta$, with x=0; 0.02; 0.04; 0.06; 0.08 y 0.1 and fired at 500°C. Furthermore, some selected samples with superior catalytic and electrocatalytic performance at conventional laboratory experiments were fired at 800°C and/or at 1000°C and impregnated with CuO-NiO. In-situ DXAS experiments were performed at the Ce LIII-edge of the D06A-DXAS dispersive beam line 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. From in-situ DXAS experiments we concluded that CeO_2-ZrO_2-Sc_2O_3 samples prepared by both CIT and GC routes and fired at 500°C exhibited a superior Ce redox behavior than CeO_2-ZrO_2 and CeO_2-Sc_2O_3 samples in the temperature range of 500°C-800°C, temperature range of interest for IT-SOFC applications. Sc-doped and non-doped samples fired 1000°C exhibited a similar redox behavior in H_2, whereas in CH_4 atmosphere, 4 at.% Sc sample exhibited higher Ce reduction values when impregnated with CuO-NiO, indicating that oxygen vacancy generation through aliovalent doping promotes Ce reduction when CuO-NiO is added to the system. This improved redox capacity is highly desirable when NiO is present in the system as it can prevent catalyst deactivation from carbon deposition by delivering oxygen from its lattice to gasify carbonaceous species.

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ORAL PRESENTATION

XANES study of sulfur oxidation by Ag and Cu atomic quantum clusters in cysteine and glutathione

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ABSTRACT:

A large group or proteins and oligopeptides has been reported as essentials for their role in the redox homeostasis regulation. This regulation involves several catalytic processes that includes the oxidation, and their reduction back, of Cysteine residues (Cys-SH), which are susceptible to oxidation at physiological pH[1]. In addition, it has been reported the capacity of Atomic Quantum Clusters (AQCs) as oxidation catalysts[2]. In this work, we have studied the oxidative capacity of Ag and Cu AQCs on Cysteine and Glutathione as a first approach in elucidating the oxidant effect in molecules with biological interest. For this purpose, the sulfur species both before and after catalytic reaction, were characterized by S-K XANES spectroscopy. Ag L3 (3351 eV) and S K-edge (2470 eV) XANES experiments were performed at de SXS beamline and Cu K-edge (8988 eV) XAFS experiments at the XAFS-2 beamline at the Laboratório Nacional de Luz Síncrotron (LNLS, Campinas, Brazil). For Cysteine and Gluthatione characterization, a fraction of solution were deposited by drop casting on carbon disks in order to have a S concentration in a detectable value. PBS buffer was used as solvent in all the reaction mixes, with the purpose maintain a pH value according to a physiological environmental. We observed that sulfur present in Cysteine and Glutathione is deeply oxidized by AQCs, from -2 to +6 . However, decreasing the AQC concentration between three and six times, a lower oxidation reaction also take place for cysteine. Disulfide species, with external groups corresponding to aliphatic-disulfide species can be also identified by XANES. This effect is not clearly observed for glutathione, due to this conformational structure and S-atoms localization. Finally, it can be shown the ability of X-ray absorption near edge structure spectroscopy on the studied molecules, allowing to characterize faithfully the chemical changes allowing the knowledge of possible reaction mechanisms.

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POSTER PRESENTATION

X-ray absorption study of Yb valence state as a function of applied pressure in the intermetallic complex $YbFe_2Zn_{20-x}Cd_x$ (x=0.0, 1.0, 1.3, 1.4) compounds.

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ABSTRACT:

The Heavy Fermion compound $YbFe_2Zn_{20}$ (Sommerfeld coefficient γ = 535 mJ.mol^{-1}. K^{-2}), which crystallizes in the $CeCr_2Al_{20}$-type structure with space group $Fd\bar{3}m$ (227), was doped with Cd atoms to study the physics of hybridized ions in crystal lattices. As one explanation to the changes in the physical properties reported by Cabrera-Baez et al they suggested a valence shift of the Yb ions due to a negative chemical pressure that the Cd substitution represents [1]. So, to investigate the valence state of Yb ions we measured the x-ray absorption near the Yb L3-edge (XANES) as a function of Cd amount and as a function of temperature. For all these measurements, within the experimental limitation, the obtained Yb valence is +2.84±0.04 and no changes were observed as a function of Cd amount and as a function of temperature. On the other hand, previous measurements of electrical transport under pressure for $YbFe_2Zn_{20}$ indicate that a quantum critical point (QCP) may be present at ~9.8GPa [2]. Inspired by this result, we propose to measure the Yb L3-edge x-ray spectrum as a function of physical pressure to probe any possible valence shift of Yb ions near the proposed QCP.

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POSTER PRESENTATION

X-ray crystallography, ancestral protein reconstruction and biophysics of enzymes from the ribokinase superfamily

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ABSTRACT:

Organisms that inhabit extreme environments such as thermophiles and halophiles have adapted their proteins to withstand extreme conditions. By using X-ray crystallography, kinetic and biophysical characterization, ancestral enzyme reconstruction and bioinformatics analysis our group study structural changes in proteins that lead to adaptations to extreme conditions like high temperature or high salt concentrations, as well as functional and regulatory changes through evolution. Recently, we have been studied the halophilic adaptation of enzymes from Methanosarcinales organism, a group from Archaea domain composed of organisms that colonize diverse environments. Using the beamline MX2 of the LNLS, we have obtained structures of the last common ancestor of ADP-dependent sugar kinases family, and by biophysics characterization, we showed that the ancestral enzyme displays an extremely high salt tolerance and thermal stability. Structure determination of the ancestral protein reveals unique traits such as an increase in the Lys and Glu content at the protein surface and yet no reduction in the volume of the hydrophobic core. Our results suggest that the halophilic character is an ancient trait in the evolution of this protein family and that proteins from Methanosarcinales have adapted to highly saline environments by a non-canonical strategy, different from that currently proposed for classic halophilic protein. These results open up new avenues for the search and development of novel salt tolerant biocatalysts.

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POSTER PRESENTATION

X-ray diffraction of the skutterudites RFe_4Sb_{12} (R=Na, K, Ca, Sr, Ba) under high pressure.

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ABSTRACT:

Pressure is known to be one of the main parameters to tune the properties of solid state systems. In the particular case of correlated electron systems, it may lead to a rich phenomenology, by triggering electronic and structural transitions. In this work, we present X-ray diffraction of the skutterudites RFe_4Sb_{12} (R=Na, K, Ca, Sr, Ba) under high pressure. RFe_4Sb_{12} (R=Na, K, Ca, Sr, Ba) are itinerant magnets for which most of the magnetic response derives from the Fe atoms with minute contribution from the Sb atoms. Our experiments were performed at the XDS beam line of the LNLS, employing a Diamond Anvil Cell (DAC) to subject our samples to extreme pressures up to 30 GPa. The Rietveld analysis of the experimental data allowed the refinement of the lattice parameters under pressure, which in turn allowed us to determine the Bulk modulus of these materials. The results are discussed in the context of the Bulk modulus of other intermetallic materials, the rigidity of the Fe_4Sb_{12} framework and the deformation of the Fe coordination structure. This last information is connected to the Fe derived magnetic properties of these itinerant magnets.

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ORAL PRESENTATION

X-ray induced persistent luminescence: How and why?

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ABSTRACT:

Persistent luminescence is nowadays a well-known phenomenon and the new generation materials have been studied for +20 years. Claimed applications of these materials can be long listed with examples like temperature sensors, emergency signage, bioimaging etc. [1]. However, the attention to these materials are focused on low energy excitation and the effect of high energy radiation is normally forgotten. X-ray induced luminescence could be used to get more information in X-ray imaging, combining the absorbance by the tissues with luminescent biomarkers [2]. Combining X-ray imaging with persistent luminescence materials could expand the horizons on bioimaging or photodynamic therapy [3]. In this work we describe several materials with green, red or near infrared persistent luminescence induced by soft and hard X-rays: $Cd_2SiO_4:Pr^{3+}$, $SrS:Eu^{2+}(Ce^{3+})$,RE3+ and $LaAlO_3:Cr^{3+},Sm^{3+}$. Total X-ray Excited Optical Luminescence (XEOL) and 3D emission-excitation XEOL spectra were studied for all materials at SXS beamline at LNLS. The excitation mechanism of each material was evaluated by analyzing the positive or negative edges in the XANES region and proved to be different depending on the emitting center and host material. The persistent luminescence was evaluated after irradiation at energies lower, equal and higher than the edge. In order to fully understand the persistent luminescence mechanisms, the luminescence features were compared to the conventional UV-excited luminescence. Different persistent emitting colors could be observed after soft and hard X-ray irradiation, proving to be good start materials for the development of new applications of this familiar phenomenon.

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POSTER PRESENTATION

X-Ray microtomography for 3D imaging of chitinized structures of Rhodnius prolixus: an evaluation of Triflumurom as potential tool for integrated vector control programs

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ABSTRACT:

In this work, we report on the particular advantages of synchrotron radiation phase-contrast microtomography, using the imaging beamline IMX at LNLS (Brazilian Synchrotron Light Laboratory), in the study of the biological effects of treatments with triflumuron (TFM) (Starycide® sc 480 Bayer) on the fifth instar nymph of Rhodnius prolixus, the most important insect vector of Chagas' disease [1]. Fifteen specimens of fifth-instar nymphs of Rhodnius prolixus were taken from the Laboratory of Biochemistry and Physiology of Insects, Oswaldo Cruz Foundation (FIOCRUZ), Brazil, and were divided into two groups. In the first group, immediately after feeding the insects with defibrinated rabbit blood, a Starycide® containing 48 g of TFM per 100 mL, provided by Bayer (Brazil), was applied directly to the ventral surface of the abdomen of these insects [2]. The second group, the control group, remained also unfed and was kept under the same conditions. They were sacrificed after 25 days. For the measurements, the images were acquired with 4× magnification optics, what gives a virtual pixel size of 1.9 µm. The advantage of using this setup was the faster acquisition time achieved and a very high photon flux. For our experiments, 1000 radiographic projections were acquired over an angular range of 180° with angular step of 0.18°. The results showed Rhodnius prolixus in the intermoulting period what had never been presented before with any type of insects using microtomography. The images obtained have also confirmed the efficacy of TFM, and that it can be used as a pesticide being excellent in vector control, disrupting chitin synthesis during larval development, and so contributing for non-proliferation of Chagas' disease. The results also highlight the capability of IMX beamline at the Brazilian Synchrotron Laboratory in terms of resolution and visibility of details with the single-distance phase contrast microtomography.

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POSTER PRESENTATION

X-ray multiple diffraction in D-alanine crystal at XRD2 - LNLS: a step towards to investigate radiation damage mechanisms

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ABSTRACT:

X-ray multiple diffraction (XRMD) is one of the techniques that can be used in the characterization of single crystal. In XRMD experiment a family of plans (called primary planes) needs to be in diffraction condition at the same time when two other families of planes. The interaction among beams involved gives rise to variation, either an increase or a decrease, in the intensity of primary diffracted beam [1-3]. Azimuthal scanning of a Bragg reflection is a systematic procedure to excite many cases of XRMD. Measuring their positions along the scan, i.e. the azimuth where each case is exited, allow determination of unit cell parameters that can reach an astonishing resolution of a few femtometer in cubic unit cells. Moreover, in single crystals of good crystallinity, the intensity profiles of each XRMD can be susceptible to dynamical diffraction effects, and thus, providing information on the phases of the structure factors, or more precisely, on invariant triplet phases. Maximum accuracy in phase measurements is achieved by choosing a suitable XRMD where the phase values fall on a particular range of good resolution, which vary to each case depending on what structural information is desired. In our recent searches we have achieved strong progress concerning procedures for XRMD as a tool for refining crystal structures determined by standard crystallographic methods [4-7]. In this work X-ray phase measurements was applied to alanine crystal, proving useful information for molecule dynamics studies of intermolecular forces. Feasibility of this experimental to investigate radiation damage mechanisms was also demonstrated.

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