First Mexican Synchrotron Radiation Users' Meeting

Cuernavaca, México
Mayo 4-6, 2011

Abstracts Index

Fluorescence XAS by Highly Segmented X-ray Detection –Beyond Conventional Applications

X-ray absorption spectroscopy (XAS) is a powerful local structural tool which rapidly prevailed in chemistry, biology and materials science, associated with the dedicated synchrotron radiation facilities (2nd generation storage rings) in the 1980's. Severely limited applicability of conventional XAS, partricularly, sensitivity in transmission experiments was solved by a fluorescence detection, for which high photon flux and high efficiency in x-ray detection were both essential. For the former, focusing optics (bent mirrors and sagittal focusing) and insertion devices (multipole wiggler etc.) improved excitation efficiency, while for the latter, x-ray detection with a high energy resolution and throughput was required. Combination of the two approaches successfully lowered concentration limit since the late 1980's, that was reflected in the rapid growth of applications to dilute systems (biological samples) and thin films. Since x-ray detection effiency is dependen on the two competing specifications, i.e., energy resolution and throughput, segmented fluorescence detection is realistic in recording x-ray signals over a wide solid angle. Here we focus on a germanium pixel array detector (PAD) that consists of densely arranged (10 x10 array) pure germanium pixels (5 mm x 5 mm)1. This x-ray detector not only improved the quality (statisctics) of XAS but also opend up novel application fields beyond a conventional use. As examples of new research fields available by PAD, polarized experiments on HTSC thin film single crystals (100 nm)2 and photo-induced phase transition in powder (<1 mg) specimen3 are described. In the 1990's, demands on high brilliance of synchrotron radiation aimed at imaging accelerated the costly ultra-low emittance (<10 nmrad) storage rings with a high energy (6-8 GeV). Tunable x-ray undulator radiation provided high brilliance and high flux x-ray beam in the hard x-ray region (5-40 keV), makinmg possible probing samples with a limited size or volume. However, more recently, a new class of ultra-low emittance storage rings with a medium energy range (2.7-3.5 GeV) is taking over the leadership. The successful proliferation of the 3rd generation storage rings is strongly dependent on the progress in "in-vacuum" undulator technology4 that allows a small gap distance of magnets. Indeed a comparable high brilliance with that of high energy machines is achievable by "in-vacuum" undulators inserted in a "cost-concious" medium energy storage ring. Future prospects of fluorescence XAS using such facilities will be discussed. 1. H. Oyanagi et al., Nucl. Instr. And Methods A 513, 340 (2003). 2. H. Oyanagi et al., Phys. Rev. B 75, 024511 (2007). 3. H. Oyanagi et al., J. of Luminescence 119-120, 361 (2006). 4. H. Kitamura, Rev. Sci. Instrum. 66 (2), 2007 (1995).

Primary authors : Dr. OYANAGI, Hiroyuki (National Institute of Advanced Science and Technology (AIST))

High Energy-Resolution X-rays and Applications in Materials Science: The Case with Inelastic X-ray Scattering

The advent of tunable, highly intense and bright synchrotron radiation, and technical advances in the last decades have provided an increasingly powerful source for a broad range of spectroscopic tools, including inelastic x-ray scattering (IXS), for materials research. The construction of dedicated facilities that routinely deliver x-ray beams with μm focal size, energy resolution down to ~meV, and flux on the order of 1010 photons/sec/meV was imperative for the fruitful development of various IXS techniques for the study of lattice dynamics and correlation effects of electrons in many-electron systems. The high penetration depth of hard x-rays offers furthermore unique strength in the study of materials properties under extreme thermal dynamical conditions such as high pressure and extreme temperature. In this presentation, the current status of the IXS techniques will be reviewed and illustrated with examples drawn from high-resolution experiments on a variety of systems, including low-energy charge excitations of a few archetypal materials and materials under the extremes of pressure and temperature. Some perspectives for further developments in the field, particularly in connection with the IXS capabilities planned or being developed at NSLS-II, will also be discussed.

Primary authors : Dr. CAI, Yong (Brookhaven National Laboratory)

A soft x-ray resonant inelastic x-ray scattering (RIXS) syllabus

The main ideas that make RIXS a very useful tool to study the electronic structure of solid compounds will be presented in this work. This photon-in photon-out technique allows a direct determination of symmetry and site selected density of states, both occupied and unoccupied. A basic experimental configuration and calculations using atomic multiplet models to interpret RIXS spectra will be discussed. Transition metal fluorides and oxides will be used as examples of RIXS work that provides crucial electronic structure information. For instance, a comparison of absorption and emission spectra at the fluorine K and metal L2,3 edges allows a detailed study of the evolution of the Mott-Hubbard transition metal di-fluoride insulators. A simple extrapolation of these results to the corresponding monoxides is in very good agreement with the Zaanen-Sawatzky-Allen model. For the same compounds RIXS spectra at the metal L2,3 edge give information about d-excited and charge transfer states. It will also be shown that RIXS spectra give a detailed information about the fast decay dynamics of the 2p_{3/2} hole in ionic orthovanadates and about the symmetry and nature of the gap of the lanthanum-strontium cobaltates.

Primary authors : Prof. JIMENEZ-MIER, Jose (ICN-UNAM)

Co-authors : Dr. HERRERA-PEREZ, Guillermo (CINVESTAV) ; Mr. OLALDE-VELASCO, Paul (Instituto de Ciencias Nucleares, UNAM) ; Mr. CARABALI-SANDOVAL, Giovanni (Instituto de Ciencias Nucleares, UNAM) ; Prof. MOEWES, Alexander (University of Saskatchewan, Canada) ; Dr. WILKS, Regan (University of Saskatchewan, Canada) ; Dr. YANG, Wan -li (The Advanced Light Source, Berkeley) ; Dr. DENLINGER, Jonathan (The Advanced Light Source, Berkeley) ; Prof. EDERER, David (Tulane University) ; Prof. CHAVIRA, Elizabeth (Instituto de Investigaciones en Materiales, UNAM)

Mexican Light Source Project: Outlook

Primary authors : Dr. MORENO, Matías (IFUNAM, México)

Application of XAS to study the biotransformation of potentially toxic elements and nanomaterials in plants

X-ray absorption spectroscopy (XAS) has been especially useful in determining the potential metal absorption pathways and tolerance mechanisms in plants. In tumbleweed plants germinated and grown in a media containing Cd, it was determined that this metal was bound to O and S. Further results determined that thiols, some organic acids and proteins might be involved in the tolerance mechanism in this plant species. Also, metal biotransformation in plants naturally growing on contaminated sites has been proven. As for nanofitotoxicity studies, it was recently determined that nanoparticle biotransformation depends on the nature of these materials. Thus, ZnO but not CeO2 were biotransformed by soybean seedlings. Acknowledgements: Portions of this research were carried out at the Stanford Synchrotron Radiation Laboratory, a national user facility operated by Stanford University on behalf of the U.S. Department of Energy, Office of Basic Energy Sciences. The SSRL Structural Molecular Biology Program is supported by the Department of Energy, Office of Biological and Environmental Research, and by the National Institutes of Health, National Center for Research Resources, Biomedical Technology Program.

Primary authors : Dr. DE LA ROSA, Guadalupe (universidad de Guanajuato)

Co-authors : Dr. MéNDEZ VALENCIA, Dellanira () ; Dr. MARTíNEZ AYóN, Tania () ; Dr. CRUZ JIMéNEZ, Gustavo () ; Dr. GARDEA-TORRESDEY, Jorge ()

Use of XAS for Environmental Sample Analysis

This work is focused in sample preparation, analysis and data interpretation of XAS spectra from environmental samples. Sample preparation is explained for particulate matter, soil, plants and mine tailings. The elements analyzed in the different samples include As, Pb, Cr, Fe, Br, Cu, Se, S and Au. Suggestions for XAS data acquisition will be discussed.

Primary authors : Dr. CRUZ-JIMENEZ, Gustavo (Universidad de Guanajuato) ; Dr. DE LA ROSA-ALVAREZ, Guadalupe (Universidad de Guanajuato)

Co-authors : Dr. GARDEA-TORRESDEY, Jorge L. (The University of Texas at El Paso) ; Dr. DELGADO-RIOS, Marcos (Universidad Autónoma de Ciudad Juárez) ; Dr. BERNAL-ALVARADO, Jesus (Universidad de Guanajuato) ; Dr. ROCHA-AMADOR, Diana O. (Universidad de Guanajuato)

X-ray Absorption Spectroscopy applied to the study of Systems with Lattice Instabilities

We discuss the basic principles of the process of x-ray absorption in condensed systems. It is shown how x-ray absorption spectroscopy (XAS) can be used to determine both local atomic structure and local electronic structure in materials. We discuss the use of x-ray absorption fine structure (XAFS) in crystalline materials in which the interaction between electrons and ions produces changes in the local atomic structure as a result of electronic changes. As a specific example we discuss the case of photoinduced structural changes in doped II-VI semiconductors. We also illustrate the use of X-ray Absorption Near Edge Structure (XANES) to discern local brake of crystal symmetry in materials, which exhibit correlated motions of ions and electrons, taking as an example the oxide PrNiO3.

Primary authors : Dr. JOSé, Mustre (Cinvestav-Merida)

Multiferroics Under the Synchrotron Light

Synchrotron-light investigation of multiferroics, from atomic- to meso-scales, is characterized. The information contained in diffraction peaks gives the long-range space-time average structure. It provides the general structure diagram, precise lattice parameters and average atomic positions. In synchrotron applications, diffraction is highly sensitive to symmetry breaking. Space/time deviations from mathematical periodicity decrease diffraction maxima and generate diffuse scattering. Research using ferroelectric diffuse scattering in the three-dimensional vicinity of Bragg peaks represents a current tendency aimed at clarifying the static and dynamic characteristics of ferroelectrics above the Curie temperature Tc. The final objective is to elucidate the gestation of ferroelectricity from the paraelectric state. Two models that represent extremes in the existing diversity of approaches are: a) (Solid state physics) --> Softening of the dynamic chains of correlated displacements that form the transverse optical (TO) modes. b) (Quantum chemistry) --> Static linearly ordered displacements that have short range in the paraelectric phase and show long-range order in the ferroelectric phase. Extended X-ray Absorption Fine Structure (EXAFS) investigation is briefly considered. This technique focuses on the determination of a few short-range structural characteristics invisible for the averaging eyes of diffraction techniques. X-ray Absorption Near Edge Structure (XANES) offers information on oxidation state of the absorbing element and in general on the electronic structure of the target element's bonds. Representative studies performed by the CIMAV Crystal Physics Group are presented. Considered cases include subtle symmetry break-downs in perovskite-related compounds, local-order phenomena and thin films texture analyses. Synchrotron-crystallography software development by the group is divulgued: - Software package "ANAELU" models 2D diffraction patterns produced by textured thin films, thus allowing the interpretation of complex patterns. - Program SAMZ permits the estimation of effective values for polycrystal dielectric, piezoelectric, magnetoelectric and elastic coefficients under the Voigt, Reuss and Hill approximations. Portions of this research were carried out at the Stanford Synchrotron Radiation Lightsource, a national user facility operated by Stanford University on behalf of the U.S. Department of Energy, Office of Basic Energy Sciences. Support from CONACYT- Mexico (Project 102171) is gratefully acknowledged.

Summary :

Synchrotron radiation diffraction, scattering and absorption spectroscopy are among the most powerful tools employed today for the investigation of ferroic materials' fine structure. A comparative review of the mentioned techniques, as applied to bulk and nano-structured ferroelectric and magnetoelectric samples is presented and illustrated by means of practical examples. Crystallographic texture plays a significant role on multiferroic bulk and nano- structured materials. Texture analysis by means of two-dimensional (2-D) diffraction experiments and the role of texture on multiferroic physical properties are discussed.

Primary authors : Dr. FUENTES-COBAS, Luis E. (Centro de Investigación en Materiales Avanzados) ; Dr. MONTERO-CABRERA, María E. (Centro de Investigación en Materiales Avanzados)

Co-authors : FUENTES-MONTERO, Luis (Institue Laue-Langevin) ; Mr. MUñOZ-ROMERO, Alejandro (Centro de Investigación en Materiales Avanzados) ; FUENTES-MONTERO, María E. (Universidad Autónoma de Chihuahua)

Electronic structure of MF2 (M=Cr-Zn) studied by X- ray Absorption (XAS), Normal X-ray Emission (NXES) and Resonant Inelastic X-ray Scattering (RIXS).

Electronic structure of the strongly correlated MF2 (M = Cr-Zn) is elucidated by using X-ray Absorption (XAS), Normal X-ray Emission (NXES) and Resonant Inelastic X-ray Scattering (RIXS). We have observed by aligning structures in the F Kα and TM Lα spectra the transition from Mott-Hubbard (M-H) to Charge Transfer (CT) Insulator regime. From this we concluded that the difluorides from Cr-Cu are M-H type while the only CT insulator is ZnF2. We were able to experimentally determine the value of the important parameters U and ΔCT. Our approach can be extended to other systems including oxides. LDA+U calculations confirm that the 3d TM density of states dominate over F 2p close to the Fermi level. Relative good agreement is found between the calculated F2p DOS and F Kα and F 1s XAS spectra. The bandgaps reported by theory are in good agreement with experimental results. TM L2,3 XAS spectra were measured and reproduced satisfactorily by using ligand field multiplet (LFM) calculations in D4h symmetry, the relevant parameters are reported. RIXS maps for Cr-Ni cations were taken at the L2,3 edges and the neutral d-d and CT excitations were identified. These RIXS maps were reproduced by using the LFM theory combined with Kramers-Heisengberg formalism for coherent second order processes. Since our calculations do not include CT effects we unambiguously distinguished the character of the different neutral excitations of the cations.

Primary authors : Mr. OLALDE VELASCO, Paul (Instituto de Ciencias Nucleares UNAM)

Co-authors : Prof. JIMéNEZ-MIER, José (ICN-UNAM) ; Dr. YANG, Wanli (ALS-LBNL) ; Dr. DENLINGER, Jonathan (ALS-LBNL) ; Dr. DE LA MORA, Pablo (Facultad de Ciencias-UNAM)

The ID21 beamline at the European Synchrotron Radiation Facility: an ideal tool for sub-micrometric 2D speciation analyses

The ID21 at ESRF is a beamline dedicated to X-ray and FTIR micro-spectroscopy. Localization and speciation of trace elements is primarily done using micro-X-ray fluorescence (µXRF) and micro X-ray absorption spectroscopy (µ-XANES) in the tender X-ray domain (2-9.2 keV). Typical scientific questions concern the co-localization and/or speciation of trace elements in heterogeneous matrices at the submicron scale, with applications in the fields of Environmental Sciences, Earth and Planetary Sciences, Life Sciences and Cultural Heritage. ID-21 has sensitivity in the low ppm range and allows localization with a sub-micron beam of various elements. The SXM offers a very high versatility in terms of focusing optics, detection and sample environment. The X-ray beam spot size can be tuned from macro (200 µm) to sub-micro (~500 nm), which then allows localization of trace elements at subcellular level. A large panel of complementary detectors is available and provides: high sensitivity, high throughput, or high spectral resolution, which enables the collection of µ-XRF and µ-XANES spectra on a large variety of samples. The samples can be studied in various conditions (room temperature, cryo, wet cells). Additionaly, ID21 has a SR-based FTIR end-station that can provide complementary molecular mapping, with a ~5µm lateral resolution.

Primary authors : Dr. HIRAM, Castillo-michel (European Synchrotron Radiation facility)

Co-authors : Dr. COTTE, Marine (European Synchrotron Radiation Facility) ; Dr. SALOME, Murielle (European Synchrotron Radiation Facility) ; Dr. FAYARD, Barbara (European Synchrotron Radiation Facility) ; Dr. DI CHIARO, Franck (European Synchrotron Radiation Facility) ; Dr. GAGLIARDINI, Erick (European Synchrotron Radiation Facility) ; Dr. BERRUYER, Gilles (European Synchrotron Radiation Facility) ; Dr. JEAN, Susini (European Synchrotron Radiation Facility)

Evaluation of Selenium and Cadmium on Germination and Growth of Cauliflower.

In this work we evaluate the attributed effects of some Se and Cd compounds on cauliflower (Brassica oleracea var. Botrytis). This effect has extensively been studied in animal and microorganism, however little information exists on the Se-Cd interaction in plants. The purpose of this work is to evaluate the possible antagonist or agonist toxic effect of Selenium upon cadmium. XAS analysis is used to determine the oxidation state of Se in the different parts from the plant (root, stem and leaves), in order to know if it has become by the plant. The measurements of XAS of the samples were carried out in the facilities of the SSRL (Stanford Synchrotron Radiation Lightsource). The results indicate that in the interaction of the selenate with cadmium in B. oleracea bar. Bortrytis was significant in stem and root. For the case of the selenite interaction with cadmium it was significant for root and leaves, the Se4+ reduces the poisonous effects originated in tissues of B. oleracea demonstrating therefore to a protective effect in this plant.

Primary authors : Dr. GUTIéRREZ-VáZQUEZ, Gilberto (Universidad de Guanajuato)

Co-authors : Dr. CRUZ-JIMéNEZ, Gustavo (Universidad de Guanajuato) ; Dr. DE LA ROSA, Guadalupe (Universidad de Guanajuato) ; Dr. BERNAL ALVARADO, Jesús (Universidad de Guanajuato) ; Dr. VALTIERRA-GASCA, Ariadna (Universidad de Guanajuato) ; Dr. GóMEZ-AGUILAR, Francisco (Universidad de Guanajuato)

Determination of Physicochemical Papameters of [CIPROFLOXACINATE CITRATE ZINC (II)] Complex That Could be Used in Artifitial Tears

The ciprofloxacin (1-cyclopropyl-6-fluoro-1,4-dihydro-4-oxo-7-(1-piperacinyl)-3-quinolinecarboxylic acid), is a wide spectrum antimicrobian from the second generation quinolones, due to its pharmacokinetic properties, it is considered as a good option for the treatment of multiple infections. Zinc (Zn) is an essential trace element for all lifestyles and it is the most abundant intercellular element. In addition, it is associated to multiple functions in the body such as wound healing for his role in collagen synthesis and cell proliferation. Therefore, there is an interest to synthesize zinc ciprofloxacin complexes of to study the role of such complex in both healing capacity and antimicrobial effect. The formation of zinc ciprofloxacin complexes in a rate 1:2 has been reported previously. Houever, a highly insoluble product was obtained, and its biological activity could be reduced, so in this work it is proposed the addition of a citrate group in the zinc complex to make it more soluble. In order to study the complexes synthesized, X-ray absorption spectroscopy (XAS) was used to elucidate the structure of both complexes since they were obtained as an amorphous form. The X-ray absorption spectra was obtained using the Beam line 7.3 at the Stanford Synchrotron Radiation Lightsource (SSRL). Afterwards, the XANES part of the spectra was extracted. Finally, a comparison among model compounds and sample spectra showed some difference between the chemical environment of 1 and 2 ciprofloxacine complexes. Moreover, it was observed that the spectra show more similarities to the Zn oxide model compound that the Zn nitrate spectra used as a reagent in the synthesis. Therefore, it is possible that ligands may be coordinated to the metal through deprotonated oxygens of the respective carboxylic acids from the citrate and ciprofloxacine. It is recommended the EXAFS spectra analysis to obtain the coordination number of the metal and additional structural information.

Primary authors : Dr. ANA GUADALUPE, Sánchez Vázquez (Universidad de Guanajuato)

In-situ microscopy and spectroscopy for structural and catalytic studies

Over the last decades my laboratory has developed and applied microscopy and spectroscopy techniques for in situ studies of the surface structure of catalysts in the presence of various gases. These include high pressure Scanning Tunneling Microscopy (HP-STM), in situ x-ray spectroscopies using Synchrotron radiation. These include high pressures x-ray absorption (XAS) and X-ray photoelectron spectroscopy (HP-XPS). We applied these techniques to the study of various reactions and surfaces on single crystals and nanoparticles. I will illustrate the capabilities of these techniques with several examples. These include the restructuring of Pt single crystals in the presence of CO, the structure of Rh(111) in the presence of CO and NO, the determination of the surface phase diagram of Pd oxides in equilibrium with O2, and others. In the area of nanoparticles, I will show the application of XAS and HP-XPS to study the structure and activity of Co nanoparticles for CO+H2 methanation as a function of particle diameter from 3 to 20 nm. The particles Co were found to remain metallic and covered by CO during reaction. The reactivity per Co atom was found to decrease when the particle size became smaller than 10 nm, an effect that was related to the decreased activity of the particles to dissociate H2, while the rate limiting step did not change. Using HP-XPS also we observed the structural modifications of core-shell Rh-Pd and Rh-Pt alloy nanoparticles during oxidative and reducing conditions.

Primary authors : Dr. SALMERON, M. ()

Synchrotrons Enable New Research in Genomics and Proteomics

Genome sequencing projects rapidly expand protein sequence space and allow comprehensive approaches to studies of entire cellular systems. The accumulation of sequence data has accelerated significantly and now includes studies of microbiomes and metagenomes. However, many aspects of protein function, including molecular recognition, assembly and catalysis, depend on the 3D atomic structure. Protein structural analysis contributes to an understanding of the evolutionary and functional relationships among protein families that are often not apparent from the genome sequences. In the past ten years, third generation synchrotron sources and dedicated macromolecular crystallography (MX) beamlines have expanded our competence in determining protein structures using X-ray crystallography. MAD or SAD data are collected from cryo-protected crystals and structures are determined with a semi-automatic approach at the synchrotron beamline. Protein models are auto-built and structures refined, verified and analyzed using integrated computational tools and then deposited in public databases. World-wide Structural Genomics efforts took advantage of these resources and contributed a complementary array of the rapid, highly integrated and cost effective methods in molecular biology, proteomics, structure determination and bioinformatics and created efficient structure determination pipelines. For example the semi- automated pipeline of the Midwest Center for Structural Genomics (MCSG), one of the Large-scale Production Centers of the NIH-funded Protein Structure Initiative (PSI), comprises: (1) classifying all available genomic sequences to establish a prioritized target set of proteins from human pathogens and eukaryotes, (2) cloning and expressing proteins of microbial and eukaryotic origin, (3) purifying and crystallizing native and derived proteins for X-ray crystallography, (4) collecting data and determining structures, (5) analyzing structures for fold and function assignment, and homology modeling of related proteins. The pipeline when combined with data collection MX facilities at the third-generation synchrotrons, advanced software and computing resources resulted in significant acceleration of protein structure determination and overall reduction of cost. These new approaches have allowed the MCSG to determine nearly 1300 structures and the entire PSI over 5000 structures. All structures are annotated for function and ligand binding. Homology models are generated for members of protein families and ultimately will provide good structural coverage of major protein families. Moreover the PSI has comprehensively sampled the entire prokaryotic protein sequence space more broadly than ever before. The PSI Centers have cloned nearly 150,000 genes of proteins from a significant fraction of all large protein sequence families and purified nearly 50,000 highly diverse proteins creating a unique resource. Many of these structures represent protein families of high biological and biomedical interest, and many have provided novel functional hypotheses. Results and data are made available to the scientific community. Structural Genomics efforts build on the constantly growing foundation laid by the highly successful genome and metagenome sequencing projects that are enriching and expanding our knowledge of the protein universe. This discovery-based program contributes to studies the co-evolution of protein structure and function and is highly complementary to traditional hypothesis-driven approaches. Structural Genomics provides a wealth of ideas, concepts and understanding of mechanisms for acquisition of novel biological function and the evolution of biological systems. This work was supported by NIH Grant GM094585 and by the U.S. DOE, OBER contract DE- AC02-06CH11357.

Primary authors : Dr. JOACHIMIAK, Andrzej (Argonne National Laboratory)

Synchrotron Radiation for High Quality Data in Protein Crystallography

Protein crystallography is the unsurpassable technique to obtain atomic resolution of biological entities. Those entities can have molecular masses from 6 kDa like the Immunoglobulin-binding domain B1 of streptococcal protein G (GB1) to MDa like the ribosome. In the last 15 years, a number of specific technologies like crystal freezing, SAD and MAD phasing using metals or selenium containing recombinant protein crystals and the growing amount of structures to be used as scaffold for molecular replacement makes feasible that biochemists and molecular biologists with out little or no training in protein crystallography can efficiently solve crystal structures. Today protein crystallography has two bottle necks: 1) Obtain a protein crystal 2) Obtain high quality X-ray data suitable for phasing. The problem of growth protein crystals is usually tackled by a combination of molecular biology and biochemical approaches (i.e. site directed mutagenesis, partial proteolysis, etc). The problem of high quality data is tackled by an intense beam (to increase the signal to noise ratio) with the potential of tunable wavelength (to obtain phasing information). Although the development of in house X-ray sources, the only way to have tunable wavelength is with synchrotron radiation. The typical users in protein crystallography go to the synchrotron to improve data resolution of to obtain phases. We present examples of data sets take in “house” versus synchrotron data. The main result is the improvement of the maps. For instance in our research we need a resolution better of 2.2 Å to assure the effect of specific changes in protein folding. Current crystal problems have been solved by molecular replacement methodologies; we present examples of proteins in which no homologous protein has been crystallized and will no be solved by molecular replacement. The need to use SAD or MAD approaches is critical in those cases.

Primary authors : Dr. BRIEBA, Luis (Langebio-Cinvestav)

A Personal Journey in the use of Synchrotron Radiation for Investigations in the 3D Structure of Biological Macromolecules

Durante 15 años el autor ha usado la radiación sincrotrón en diferentes centros de altas energías del mundo. En este trabajo, se presentan los resultados más relevantes y la obtención de datos de estructuras de proteínas acoplados al desarrollo de métodos de crecimiento de cristales, que han mostrado como la calidad cristalina, permite obtener datos de alta resolución y dan respuesta a muchos cuestionamientos relacionados con mecanismos químicos, reconocimiento molecular e inclusive muestran las limitaciones que pudieran tener estas técnicas de radiación sincrotrón en ciertos estudios particulares de sistemas biológicos macromoleculares. Finalmente, se muestra que el estudio de la calidad cristalina a través de técnicas de topografía de rayos-X, ha permitido evaluar, como parámetros físicos (campos eléctricos y magnéticos que suelen controlar la cinética del crecimiento de cristales), acoplados con sistemas de transporte difusivo (geles) permiten obtener un incremento substancial en la calidad cristalina y una mayor resolución en la estructura 3D de proteínas.

Summary :
Since the last 15 years, the author has used several synchrotron facilities all over the World. In this contribution, the most relevant and 3D structures of proteins are shown. It is also shown how the crystal quality research coupled to the development of crystal growth methods allows to get precise information about chemical mechanisms, chemical or molecular recognition as well as limitations that the use of synchrotron radiation may have for specific issues. Finally, the author shows that coupling physical parameters (magnetic or electric fields) with diffusing-reacting systems (gel growth) allows to get high crystalline quality for investigations in X-ray topography applied to the 3D structure of proteins.

Primary authors : Prof. MORENO-CARCAMO, Abel (UNAM)

Using X-rays as a driven force to study chemical reduction on protein crystals

Radiation damage to cryocooled protein crystals during x-ray structure determination has become an inherent part of macromolecular diffraction data collection at third-generation synchrotrons. Although it is possible to make use of this damage, for example for radiation damage-induced phasing (RIP), in the majority of cases radiation damage is an unwelcome part of data collection. X-rays interact with matter in three ways. The X-ray photons may be absorbed via the photoelectric effect, scattered inelastically (Compton scattering) or scattered elastically (Thomson scattering). Only the last of these contributes to the useful part of the observed diffraction pattern. The dominant interaction at energies typically used in macromolecular crystallography is the photoelectric effect, which accounts for more than 80% of the total interaction, with around 8% being due to Compton scattering. Therefore most of the X-rays interacting with the crystal deposit their energy into it, causing radiation damage. As a consequence of those interactions, almost every protein crystal is chemically reduced, emphasizing its effect in the vicinities of metallic atoms. For this reason the crystallographic studies focused on metallo enzymes has to be performed in a very precise and careful way, taking advantage of the apparent limitations and exploited them in order to describe the system. Such approaches would be explained, as well as the importance of synchrotron radiation in order to perform them.

Primary authors : Dr. ENRIQUE, Rudiño-piñera (Instituto de Biotecnología, UNAM)

Co-authors : Mr. DE LA MORA LUGO, Eugenio (IBT UNAM) ; Mr. SERRANO POSADA, Hugo (IBT UNAM) ; Mr. CAMPOS ACEVEDO, Adam (IBT UNAM) ; Mr. ZARATE ROMERO, Andres (IBT UNAM) ; Ms. JIMENEZ ARROYO, Nizaa (IBT UNAM) ; Mrs. ROJAS TREJO, Sonia (IBT UNAM)

X-Ray Crystallography of Betaine Aldehyde Dehydrogenases: A Tool for the Study of their Catalytic Mechanism

Aldehyde dehydrogenases (ALDHs) are a group of ubiquos and ancient enzymes that catalyze the oxidation of a broad variety aldehydes to their corresponding carboxylic acids with the concomitant reduction of NAD(P)+ to NAD(P)H. Their catalytic mechanism involves three chemical steps: (1) the nucleophilic attack of the catalytic cysteine on the aldehyde substrate resulting in formation of the hemithioacetal intermediate; (2) the transfer of the hydride from the hemithioacetal to NAD(P)+, resulting in formation of the thioester intermediate; and (3) the nucleophilic attack of a water molecule on the thioester intermediate, leading to the release of the acid product. The ALDH enzymes can be dimers, tetramers o hexamers with approximately 490-500 residues per subunit. Currently, 36 different ALDH crystal structures, in their apo forms or in complex with different ligands, are available in the Protein Data Bank. Crystallographic data, included those obtained by our research group on the betaine aldehyde dehydrogenases from Pseudomonas aeruginosa and Spinacea oleracea, contributed to the knowledge of specific details at atomic level about each of the catalytic steps and of the catalytic residues involved, but there is still a considerable amount of open questions about the mechanism of catalysis of these enzymes. All ALDH crystals determined to date exhibit big cell dimensions and a large number of residues per asymmetric unit (2000-6000), with space groups C2, P212121, P1, or P3221. The use of a source of high intensity X-ray radiation has been of great help in increasing the resolution of the diffracted crystals, and the access to CCD detectors let us work with the cell dimensions of the crystals. Financially supported by CONACYT (grants 59654 and 101986) and UNAM (PAPIIT grant IN204708).

Primary authors : Dr. GONZáLEZ-SEGURA, Lilian (Departamento de Bioquímica, Facultad de Química, Universidad Nacional Autónoma de México)

Co-authors : Mr. DíAZ-SáNCHEZ, Ángel G. (Departamento de Bioquímica, Facultad de Química, Universidad Nacional Autónoma de México) ; Dr. RUDIñO-PIñERA, Enrique (Departamento de Medicina Molecular y Bioprocesos, Instituto de Biotecnología, Universidad Nacional Autónoma de México) ; Prof. MUñOZ-CLARES, Rosario A. (Departamento de Bioquímica, Facultad de Química, Universidad Nacional Autónoma de México)

High-Resolution X-ray crystallography of nanoporous molecular materials using radiation facility

Light weight nanoporous solids of coordination polymers type, among them metal-organic frameworks, are receiving an increasing attention because of their promising properties for hydrogen and methane storage, carbon dioxide sequestering, separation capability for mixtures of gases and vapors, catalysis, etc. The porous structure of these materials can be tailored according to the application requirement. Related to the nature of their framework usually it shows a flexible behavior on temperature, pressure and chemical potential changes. In this contribution examples on such behavior are discussed from high-resolution x-ray diffraction (XRD) data recorded using XRD-10B radiation facility at LNLS (Brazil). The results herein discussed illustrate the potentiality of a high-resolution XRD station with facility to record XRD data in the 4.2 to 450 K temperature range.

Primary authors : Dr. REGUERA, Edilso (CICATA-IPN, México)

Co-authors : Dr. RODRíGUEZ-HERNáNDEZ, Joelis (University of Havana, Cuba) ; Dr. AVILA-SANTOS, Manuel (CICATA-IPN, Mexico)

Synchrotron radiation, an essential tool to understand matter at extreme conditions

The synthesis of materials can be affected when it is performed under extreme pressure and temperature. For instance, high pressures reduce the bond distances altering the electronic properties of the material, allowing the formation of new bonds or the formation of different molecular or atomic arrangements. These variations can promote changes in physical and chemical properties of both reagents and products of a reaction. Thus, the combination of extreme pressure and temperature opens a window to a new world of possibilities: new compounds, new physical properties and new applications. However, in order to achieve very high pressures the reagents under study have to be micrometric in size, limiting with this its study and further characterization. Thus, synchrotron radiation is an essential tool to understand matter at extreme conditions.

Primary authors : Dr. JUAREZ-ARELLANO, Erick (Universidad del Papaloapan)

Bio and Nano Materials Dichroic Study by X-Ray Spectromicroscopy

Scanning Transmission X-Ray Microscopy (STXM) is a technique which has been rapidly advancing in the last decades. The study of soft and bio materials and the interaction between them and other matter is crucial nowadays, for both basic science and applied science. STXM provides a means to study the chemistry (molecular, interfacial, composition) of known or new materials at the nanoscale (< 30 nm) and with high spectral resolution. In this work the linear dichroism found in bio samples such as spider and cocoon silk as well as in carbon nanotubes is presented as an example of how STXM can provide qualitative and quantitative information about the composition of polymers and biomaterials.

Primary authors : Dr. HERNANDEZ-CRUZ, Daniel (Facultad de Ingeniería - UNACH)

Co-authors : Prof. HITCHCOCK, Adam-p. (BIMR-McMaster University) ; Dr. NAJAFI, Ebrahim (BIMR-McMaster University) ; Dr. ROUSSEAU, M.-e. (Département de Chimie et CERSIM, Université Laval) ; Prof. PéZOLET, Michel (Département de Chimie et CERSIM, Université Laval)

Angular resolved photoemission spectroscopy for the characterization of the electronic properties of solids and surfaces

The photoelectric effect was observed by Heinrich Rudolph Hertz in 1887 and explained by Albert Einstein in 1905. The analysis of the electrons emitted by solids and surfaces contain information about their energy levels. Electrons are photoemitted in all directions, the basis of angle resolved photoemission spectroscopy (ARPES) is the careful interpretation of the emission angles, kinetic energy and polarization of the photoelectrons, these studies allow the experimental determination of the energy levels of solids and surfaces with many advantages over other techniques. Since the mapping of the energy relation dispersions E(k) depends on the photon energy, the use of synchrotron radiation from electron storage rings gives the opportunity of collecting a large amount of information for electronic band structure determination. Since the inelastic mean free path of the photoemitted electrons depends on their kinetic energy, the appropriate selection of photon energies and experimental set-up allows a relatively easy identification of surface vs bulk features in many materials. Fractions of an atomic monolayers can be easily distinguished. The dependence of the energy levels of atoms on their chemical environment allows the determination of the atomic bonding properties and chemical composition (core level spectroscopy). The high intensity and broad range of continuous tuning of the photon energy from synchrotron light has contributed to the development of high resolution ARPES (few meV) with high special lateral resolution.

Primary authors : Dr. HERNANDEZ CALDERON, Isaac (CINVESTAV)

Refinamiento Rietveld en una mezcla de fases usando DRX por sincrotrón en la composición (SnPb0.4In0.6)Ba3Tm2Cu7O20

La difracción de rayos-X (DRX) por sincrotrón fue utilizada para cuantificar con más precisión la mezcla de fases presentes en la composición en estudio. La caracterización estructural de la composición (SnPb0.4In0.6)Ba3Tm2Cu7O20, se realizó por medio de la técnica de difracción de rayos X, de alta resolución. Un análisis detallado del difractograma presentó los siguientes compuestos: BaTb0.5Sb0.5O3 ICSD # 33619, Yb2BaCuO5 ICSD # 80573, CuO ICSD # 87122, Ba1.99Y0.01Cu3O7 ICSD # 69611, y Yb2Cu2O5 ICSD # 79432. El ajuste Rietveld del archivo de datos de rayos X, indicó que el compuesto BaTb0.5Sb0.5O3 (PDF-01-076-0127) reportado por Wittmann, U. et al. [1] se encuentra en mayor proporción. Los cristales en la muestra con morfología cúbica observados por SEM, presentaron una mayor proporción. De acuerdo a la determinación del porcentaje atómico promedio que se realizó por EDX, se asignó la fórmula estequiométrica. Por lo que se consideró escribir la fórmula Ba(Yb0.38In0.10Sn0.42Pb0.10)O3 isoestructural al compuesto BaTb0.5Sb0.5O3. Para mantener un balance de cargas, se propuso una deficiencia en átomos de oxígeno Ba(Yb0.38In0.10Sn0.42Pb0.10)O2.66. Las posiciones atómicas para tal composición son la siguientes, dentro del grupo espacial Pm-3m: Ba2+, en la posición de Wyckoff 1a, mientras que Yb3+, In3+, Cu2+, Sn4+ y Pb2+ fueron distribuidos en la posición de Wyckoff 1b. Referencia: 1. Wittmann U., Rauser, G. y Kemmler-Sack, S., Z.Anorganische und Allgemeine Chemie, 482 (1981) 143-153. Este trabajo es parcialmente financiado por el proyecto CONACYT 80380.

Primary authors : Mr. M. ESTRADA, Joel (IIM-UNAM)

Co-authors : Dr. CHAVIRA, E. (Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México) ; Dr. ROSALES, I. (Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México) ; Prof. MARINERO, E. E. (Hitachi GST San José Research Center) ; Dr. SUCHOMEL, M. (Argonne National Laboratory)

Radiation damage at cryotempreatures - application to redox systems

Protein crystallography has been the most successful technique for determining 3D structures from protein molecules. However, the technique has several disadvantages including the need of a crystalline sample, radiation damage and a static, averaged on time and space, resulting model. Radiation damage is unavoidable and is one of the limiting steps to estimate phases by anomalous methods. Protein crystals are usually diffracted at 100 K to reduce the rate of radiation damage inflicted by the X-rays. However, despite the use of cryotechniques diffracted intensity fades with increasing dose and a dose to half intensity, D1⁄2, of 43 MGy has been experimentally established. Another consequence of radiation-matter interactions is specific damage; as the dose increases residues are damaged in a reproducible order (breakage of disulfide bonds, decarboxylation of aspartates and glutamates, OH group lost from tyrosines). At room temperature radical scavengers have been shown to mitigate both global and specific damage, presumably by intercepting radicals created in the surrounding medium [2]. In this work we test the effectiveness of radiation damage at cryotemperatures where the motion of these radicals will already be impeded and we present an approach to describe catalytic mechanisms in redox enzymes by taking advantage of radical formation during X-ray irradiation of protein crystals.

Primary authors : Dr. DE LA MORA, Eugenio (IBT UNAM)

Co-authors : Dr. CARMICHAEL, Ian () ; Dr. GARMAN, Elspeth () ; RUDIñO, Enrique ()

Infrared imaging and spectroscopy using synchrotron light sources

In this talk I will motivate why and when a synchrotron is a desirable infrared source for FTIR spectroscopy and microscopy. I will then present a series of scientific examples spanning many disciplines from Biology to Physics to Chemistry to Environmental Science. Infrared beamlines are part of every new synchrotron being built world-wide because they are cost-effective and produce high-impact science for the facility. The user community for IR beamlines continues to grow, and I will point out the new technical directions that future IR beamlines will exploit for even greater scientific impact.

Primary authors : Dr. MARTIN, Michael (Lawrence Berkeley National Laboratory)

The Biomedical Imaging and Therapy Beamlines at the Canadian Light Source

The synchrotron source provides a unique opportunity for biomedical research in both imaging and therapy research and applications. A general purpose biomedical research facility has been built at the Canadian Light Source in Saskatoon, Saskatchewan. The Biomedical Imaging and Therapy (BMIT) beamline complex at the Canadian Light Source is transitioning from construction to operations. The bend magnet beamline has been producing some results for over a year and we are now entertaining users in a Letter of Intent phase. The insertion device beam has been delivered into the POE-2 and SOE-1 experimental hutches. The BMIT facility has two beamline complexes; an insertion device source beamline at which the bulk of the imaging and therapy research on humans, animals, and plants will be carried out, and an ancillary bend magnet source beamline which will serve as a proof-of-principle facility and research tool for new methods of imaging and therapy. The bend magnet beamline is now being used for first experiments using conventional imaging and diffraction enhanced imaging). Several imaging methods (absorption-edge subtraction imaging, diffraction enhanced imaging / analyzer based imaging, phase contrast imaging, and absorption imaging) in projection and computed tomography modes as well as monochromatic beam and filtered white beam therapy methods will be available. The beamline overview and status along with examples of the types of research that are and can be carried out will be given. These examples will include a number of biomedical programs such as investigating lung function, a number of bone studies, tissue scaffold imaging, prostate cancer imaging, radiation therapy, and cardiac imaging. Some material science projects include imaging aluminum grain boundaries and visualizing aspects of fuel cell operation. Also, a number of technology development programs including new imaging methods or new approaches to imaging, detector development and high resolution dosimetry. Our plans in progressing toward large animal imaging and human research programs will be presented as well as some discussion on the challenges we face in realizing the full potential of the facility. Finally, a perspective of where the BMIT facility fits in world-wide to provide some context of the types of programs that are carried out at other synchrotron facilities. Contact Information: Prof. L. Dean Chapman Canada Research Chair in X-ray Imaging Anatomy and Cell Biology, College of Medicine University of Saskatchewan 107 Wiggins Road Saskatoon, SK S7N 5E5CANADA

Primary authors : Prof. CHAPMAN, Dean (University of Saskatchewan)

Observation and simulation of phase-effects in phantom images obtained with x-ray tubes

We have performed systematic studies of the edge enhancement observed in digital images of cylindrical plastic phantom objects obtained with x-ray tubes. Geometrical conditions are those encountered in commercial mammography units (focal spot size= 100 micrometers, total source-to-detector distance 66 cm) and in a laboratory setup (microfocus X-ray tubes, distances up to 1 m). We observe edge enhancements of the order of 1% with respect to the background signal. We interpret the observations with a diffraction-based simulation.

Primary authors : Dr. MARíA ESTER, Brandan (Instituto de Física UNAM)

Atomic and Molecular physics experiments in synchrotron sources of second and third generation

This talk will present an overview of gasphase experiments of molecular and atomic photoionization from the the standpoint of a user, at a synchrotron facility. The studies presented are specifically devoted to the study of single and double photoionization processes as well as photoelectron angular distributions. In addition to a brief overview of the physical processes studied, this presentation will have a stronger focus on the requierements and needs, in terms of flux , resolution, technical support and end station specifications that these studies pose to experimental beamline here they are carried out. A comparison of performance of experiments carried out at a second generation source and a third generation source will be presented, in order to emphasize the importance of the beamline streamlining and performance

Primary authors : Dr. JUAREZ REYES, Antonio ()

Materials Characterization of Mexican Cultural Heritage: Recent Methodologies and Strategies

Non destructive characterization of cultural heritage has been carried out in the last decade systematically in Mexico. An interdisciplinary group, the ANDREAH network (Red de Análisis No Destructivo para el Estudio del Arte, la Arquelogía y la Historia), formed mainly by scientists, conservators and archaeologists of the UNAM and INAH has been developed to study all kind of materials: Pottery, metallic artifacts, manuscripts and codex, paintings, litic materials, pigments, etc. The methodologies used involve imaging techniques (IR and UV), portable spectrometers (XRF, Raman, FTIR), ion beam accelerators techniques (PIXE-RBS, PIGE), electronic microscopes (SEM, HR-TEM), and chemical methods. Some of the most outstanding collections and artifacts of the Mexican cultural heritage has been studied by our group. In the last year, several contacts and proposals of research has been aroused to develop specific studies in the Brookhaven National Synchrotron Light Source and in the SOLEIL facility. The aim of this talk is to present the state of the art of the scientific study of the cultural heritage in Mexico and some proposals of research that may be carried out with the Mexican groups in a Synchrotron facility.

Primary authors : Dr. RUVALCABA SIL, Jose Luis (Instituto de Fisica, UNAM)

Photionization of Kr+ with synchrotron radiation.

combination of an ion-beam end station with synchrotron raditation in the energy range of 23 to 32 electron volts was used to measure a high resolution ion-yield spectrum of the ionization of single charged into double charged Kr ions. Several atomic states are resolved and idenfied. In addition, the absolute total cross section was measured. This particular system is of relevance in astrophysics and, from a fundamental point of view, it can be used as a benchmark to prove state of the art quantum mechanical calculations. If time permits, results on the photionization of a simple molecular system will be presented too.

Primary authors : Dr. HINOJOSA, Guillermo (ICF, UNAM)