Synchrotron State Of Art

The Light Fantastic

By: NEAMA IMAM
I am interested in Materials science and applications, so
By using the on-line techniques that based on the synchrotron radiation,
I can find out what I can not determine it using the off-line conventional
or laboratory techniques.

OR,
I can see (by SR) what can be seen using Lab. techniques but with more
details, more resolution, whatever the concentration, …… thanks to the
characteristic features of Synchrotron radiation …………………

First of all, what is synchrotron, properties of SR? …..etc
What is a Synchrotron light

Synchrotron light

Synchrotron can be seen as a huge (Ultra-intense) Light Bulb (Source)

Ultra-intense electromagnetic radiation arising from the relativistic motion of electron under the effect of magnetic field which forces the electron to move along curved trajectory followed by emission of radiation with exceptional characteristic called synchrotron radiation in a wide spectral range from IR (fraction of eV) to hard X-ray (KeV) [ T tunability]
Electrons are generated and accelerated in a linac, further accelerated to the required energy in a booster and injected and stored in the storage ring.

The circulating electrons emit an intense beam of synchrotron radiation which is sent down the beamline.
**Why Synchrotron Radiation**

- The unique and superior characteristics of SR like the energy tunability and the extremely high brilliance or brightness, have provided a lot of new characterization techniques for academic, technological and industrial purposes.

X-rays – especially from synchrotrons – are useful in many scientific disciplines: biology, chemistry, geology, physics, archeology, material science, energy sciences, environmental science.

<table>
<thead>
<tr>
<th>Property</th>
<th>Behavior</th>
<th>Use</th>
</tr>
</thead>
<tbody>
<tr>
<td>Interact weakly with electrons</td>
<td>penetrate deeply into matter, with strong contrast in atomic number $Z$.</td>
<td>Imaging, Tomography</td>
</tr>
<tr>
<td>Energy</td>
<td>comparable to binding energies of core electrons in atoms.</td>
<td>Spectroscopy</td>
</tr>
<tr>
<td>Wavelength</td>
<td>comparable to distances between atoms in solids and liquids.</td>
<td>Scattering, Diffraction</td>
</tr>
</tbody>
</table>
APPLICATIONS OF XAFS IN MATERIALS/NANO-SCIENCE & ENVIRONMENTAL SCIENCE—“SELECTED EXAMPLES”
WHAT You should know from this School:

- What is XAFS?
- When you use XAFS?
- How to perform the XAFS experiment?
- How to write good proposal?
- How to understand the XAFS technique?
- How to optimize your experimental parameters for good collected data? How to read the quality of your data?
- You should know if you need to collect the whole spectrum or just you need XANES or EXAFS region only (How to manage your Beamtime)?
- How to prepare the sample to XAFS data collection?
- What information you can get from XAFS data?
- How to read the XAFS spectrum from the first look?
- How to analyze the XANES data to extract the required structural information?
- How to prepare the EXAFS data for fitting and model? Understand deeply every step in the data analysis (Demeter software)
- How to play and optimize the EXAFS parameters to get the best fit?
- When you need the XANES calculations
- How to interpret your extracted parameters and then do a correlation among the different extracted parameters?
- How to do a correlation between XANES and EXAFS results, then do a correlation between XAFS and other used technique results?
- How to explain the different physical/chemical/...properties in the light of XAFS results. (Reflections of the XAFS results on different applications)

There is more and more you should know if you decide to play with XAFS.
The XAFS beamline at Elettra

XAFS at Elettra is the Italian beamline dedicated to x-ray absorption spectroscopy. It is installed on a bending magnet source and it was designed to cover a wide energy range: from 2.4 to 25 keV.

<table>
<thead>
<tr>
<th>Source</th>
<th>Bending magnet</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flux</td>
<td>$10^9 - 10^{11}$</td>
</tr>
<tr>
<td>$\Delta E/E$</td>
<td>$10^{-4}$ (Si 111), $5 \times 10^{-5}$ (Si 311)</td>
</tr>
<tr>
<td>Spot size</td>
<td>max 26 x 2 (H x V) mm²</td>
</tr>
<tr>
<td>Energy range</td>
<td>2.4 - 25 keV</td>
</tr>
</tbody>
</table>

**Acquisition modes**
- Transmission
- Fluorescence
- Total electron yield (surface sensitive)

**K edges:** S - Ag  
**L edges:** Y - Bi
X-ray Absorption Fine Structure (XAFS) is the modulation of an atom’s x-ray absorption coefficient at energies near and above an x-ray absorption edge due to the chemical and physical state of the atom. XAFS is also referred to as X-ray Absorption Spectroscopy (XAS) and is divided into two regions:

- **XANES**
- **EXAFS**

which contain related but slightly different information about the atom’s local structural environment and electronic state.

**Main XAS Characteristics:**
- local atomic coordination
- valence, oxidation state
- applies to any element ($Z > 2$)
- works at low concentrations (ppm, $\mu$M)
- minimal sample requirements.
- independent of crystal structure, isotope.

*Minimal sample requirements* (samples can be in solution, liquids, amorphous solids, soils, plant roots, surfaces, etc.)
K-Edge XANES

Mn: [Ar] 3d^5 4s^2

Excited States

Total electron energy

Ground State

Main edge

Continuum

Pre-edge

Mn^{3+}
Normalized fingerprint XANES spectra of the pristine and \(\gamma\)-irradiated Mn doped ZnO NPs at different Mn content \((x)\) along with those of the Mn reference standard compounds at Mn K-edge.
First derivatives of XANES spectra of \( \text{Li}_{0.5}\text{Fe}_{2.5}\text{O}_4 \) along with those of iron standard compounds including different Fe oxidation states.
The first derivative of the XANES spectra \( \frac{d\mu(E)}{dE} \) is necessary for providing a more accurate and distinguished absorption peak position of both the sample and the standards.
You should be sure that this Energy shift is NOT technical shift from the mono. Or/and its thermal drift,….

### To avoid this technical shift we should do :

1- Calibration the Mono. W.r.t standard Absorption energy edge

2- Measuring the metal foil simultaneously with the target sample

3- Doing calibration for the reference in the data analysis

4- Do alignment for all the reference to be all at the same energy position (without shift)

### Note that any movement/shift in the reference energy followed by the same shift in the collected sample spectra
LCF is very important fitting for anyone uses XAFS technique.
It is used in :
1- determining the different species ( chemical forms ) of the absorbing ions and their ratio %.

2- Determining the different oxidation states of the absorbing ions and their %.

3- It is used if we have multi-components such as nanocomposite, multilayers , if there is ion diffusion,… that consisting different phases containing the target element (contribution of each phase in the XAFS signal . ( It is confirmed from EXAFS fitting)).

Recently, with the help of XANES calculations, we used LCF in determining the phase purity ; if there is any minor secondary phase ( it should be confirmed from other EXAFS and XRD fitting).

Also, recently in some of our publications, we use LCF for determining the cationic distribution over different crystal sublattice…It was very close to that determined from EXAFS fitting.
Linear combination fitting (LCF) of Mn XANES spectra of agricultural soil and oxide-rich tailings performed using Mn model compounds; scacchite (MnCl₂), hausmannite (Mn₃O₄), spessartine (Mn₃Al₂(SiO₄)₃), pyrolusite (MnO₂), rhodochrosite (MnCO₃), birnessite ((Na, Ca, K)₂.6(Mn⁴⁺, Mn³⁺)₂O₄·1.5H₂O), szmikite (MnSO₄). B: Agricultural soil; SRT: Sulfide-rich mine tailings; ORT: Oxide-rich mine tailings; composite (c) and surficial (s); particle size <2 mm (*) and 0.02 mm (***)..
LCF of the sample prepared at growth time 5 sec at Se K_edge

From the Se K edge of the sample at 5 sec growth time, it is fitted well with pure CdSe (with a ratio smaller than that was detected at the main core sample (0.663)), as a core surrounded by a very thin gradient layer of Cd\textsubscript{0.99}S\textsubscript{0.275}Se\textsubscript{0.725}Zn\textsubscript{0.01} with a weighted ratio of 0.337.
The Aim:
Inspection the Existence (and %) of the UNFRIENDLY Environmental Elements such as Ni and Pb in the collected particulate samples from the most polluted/crowded regions @ Egypt (Giza and Helwan). Therefore, we can improve the knowledge about the status of heavy metals associated with PM over Greater Cairo (Egypt).

Lab. Study: NAA failed to detect Pb and Ni (they have an extremely low sensitivity/neutron cross-section for NAA).

Lab. XRF: The concentration of Pb and Ni are very tiny to be detected by Lab. XRF (It can detect concentrations from 100 per cent down to below parts per million).

The solution: Using SR-XAFS at the Fluo-mode (for extra diluted samples). # Thanks to the incredible characteristics of SR-based technique of high resolution and high detection limit.

Syndromen radiation based techniques (X-ray absorption near-edge structure (XANES) and X-ray fluorescence (XRF)) combined with inductively coupled plasma mass spectrometry (ICP-MS) were used for the assessment of heavy metal concentrations as lead (Pb) and nickel (Ni) in airborne particulate matter (PM$_{10}$) over two residential sites in Greater Cairo. Nineteen 24 h high-volume samples collected at Giza (G) Square and Helwan (H) University (Egypt) were selected for this study. Mean concentrations of heavy metals in PM$_{10}$ at both sites were found to have the same descending order of Pb > Co > Ni > Cd > Cu of which concentrations of Pb, Co, Ni and Cd in H samples were higher than those in G samples. For Pb-spectrum based XRF data obtained by ICP-MS. The K-edge was compared to provide information on the form of those elements. The ratio in both series G and H nickel was found as Ni(OH)$_{2}$, however, the content of Ni in the sample with that of the collected aching from the inhalation of both adult residents and were

By SR- YOU can discover aspects of matter which we can Not find with other means.
LCF analysis of the Pb L2-edge merged spectrum of the H samples, using Pb metal foil, PbCl2, PbSO4 and Pb3PO4 spectra as standard references.

The high brilliance provided by SR allows one to study materials where the contaminants (e.g. Pb and Ni) are highly diluted.

SR-XANES can see what Can NOT be seen by other Techniques
XANES spectra at K-edges of Fe (a) and Co (b) at different synthesis methods. Inset shows a magnified view of the pre-edge region. (c) is XAFS spectrum at La L3-edge and white line of La L3-edge (Inset) for the different three methods.
The pre-peak fit of the pristine and irradiated exampled sample at \( x = 0.03 \), along with those of the Mn reference standards.

The pre-edge peak is fitted to Gaussian function.
Fitted pre-edge peak fitting of the different samples along with the references model compounds bearing Mn with different oxidation states, spectral white line position, absorption energies set as a fraction (half) of the edge step, oxidation states of the Zn and Mn cations in the reference compounds and samples at both Zn and Mn K-edges.

<table>
<thead>
<tr>
<th>group (x)</th>
<th>Fitted Pre-edge Centroid E (eV)</th>
<th>White-line position (eV) ±0.02</th>
<th>$E_a$ (eV); selected at the half-height of the edge step ±0.03</th>
<th>Oxidation state</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Zn, K-edge (9659 eV)-tabulated value</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>&quot;x=0.03_Before irrad.&quot;</td>
<td>6534.80±1.76</td>
<td>6535.03</td>
<td>6543.52</td>
<td>Mn$^{2+}$</td>
</tr>
<tr>
<td>&quot;x=0.05_Before irrad.&quot;</td>
<td>6534.80±1.76</td>
<td>6535.25</td>
<td>6543.51</td>
<td>Mn$^{2+}$</td>
</tr>
<tr>
<td>&quot;x=0.07_Before irrad.&quot;</td>
<td>6534.80±2.66</td>
<td>6535.90</td>
<td>6543.50</td>
<td>Mn$^{2+}$</td>
</tr>
<tr>
<td>&quot;x=0.03_After irrad.&quot;</td>
<td>6534.80±2.64</td>
<td>6536.85</td>
<td>6543.33</td>
<td>Mn$^{2+}$</td>
</tr>
<tr>
<td>&quot;x=0.05_After irrad.&quot;</td>
<td>6534.80±1.83</td>
<td>6536.69</td>
<td>6543.50</td>
<td>Mn$^{2+}$</td>
</tr>
<tr>
<td>&quot;x=0.07_After irrad.&quot;</td>
<td>6534.80±1.87</td>
<td>6537.76</td>
<td>6543.45</td>
<td>Mn$^{2+}$</td>
</tr>
<tr>
<td>&quot;Mn-foil&quot;</td>
<td></td>
<td>6655.17</td>
<td>6543.47 (<em>The inflexion point of the pre-edge peak is (6538 eV)</em>)</td>
<td>Mn$^{2+}$</td>
</tr>
<tr>
<td>&quot;MnO&quot;</td>
<td>6539.36±0.79</td>
<td>6533.95</td>
<td>6543.37</td>
<td>Mn$^{2+}$</td>
</tr>
<tr>
<td>&quot;MnO$_2$&quot;</td>
<td>6541.59±1.01</td>
<td>6538.13</td>
<td>6548.66</td>
<td>Mn$^{2+}$</td>
</tr>
<tr>
<td>&quot;MnO$_3$&quot;</td>
<td>6539.79±0.60</td>
<td>6558.17</td>
<td>6546.80</td>
<td>Mn$^{2+}$ and Mn$^{3+}$</td>
</tr>
<tr>
<td>&quot;MnO$_4$&quot;</td>
<td>6542.06±0.01</td>
<td>6539.49</td>
<td>6551.62</td>
<td>Mn$^{4+}$</td>
</tr>
</tbody>
</table>
The EXAFS data analysis and EXAFS fitting are based on this EXAFS Equation:

\[ \chi(k) = \sum_j S_j^2 N_j f_j(k, \pi) e^{-2R_j / \chi(k)} e^{-2\sigma^2(k)} \sin(2kR_j + \delta_j(k)) \]

**Amplitude reduction term**

**Thermal and static mean-square disorder in \( R \)**

**Photo-electron mean-free path (including core-hole lifetime)**

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**DEMETETER**

Demeter is a comprehensive system for processing and analyzing X-ray Absorption Spectroscopy data.

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**Demeter**

X-ray Absorption Spectroscopy Using Feff + Larch or Ieffit.

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Windows download: 
Version 0.9.26

Source code: 
View On GitHub

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Fourier transform (FT) magnitude corresponding to $k^2\chi(k)$ EXAFS signal.

**EXAFS Applications:**

**EXAFS Signal Extraction and Fitting**

Comparison between the best-fit results and the collected EXAFS spectrum. Comparisons are reported for the FT magnitude and imaginary part (Left Panel), and the extracted $k^3$ signal (Right Panel).

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Theoretical Model: ICDD #89-7102 ZnO

\[ a = b = 0.3243 \text{ nm} \]
\[ C = 0.5237 \text{ nm} \]
\((90, 90, 120)\)

Best fit EXAFS signal of Mn: ZnO NPs at \(x=0.03\) @ Zn K-edge
Research articles

Synchrotron radiation X-ray absorption fine structure and magnetization improvement of A-site Ce$^{3+}$ doped LaFeO$_3$

M.M. Arman$^a$, N.G. Imam$^{b,c,d}$, René Loredo Portales$^e$, S.I. El-Dek$^{f,*}$

$^a$ Materials Science Lab (1), Physics Department, Faculty of Science, Cairo University, Giza, Egypt
$^b$ Experimental Physics Department (Solid State Lab), Nuclear Research Center, Atomic Energy Authority, 13759 Cairo, Egypt
$^c$ Italian Sincrotrone (Elettra) – Trieste S.C.p.A., 34149 Basovizza, Trieste, Italy
$^d$ Department of Chemical Physics, Chemical Center, Lund University, PO Box 124, 22100 Lund, Sweden
$^e$ CONACyT-Geology Institute, Regional Northwest Station, National Autonomous University of Mexico, Los Arcos, 83250, Hermosillo, Sonora, Mexico
$^f$ Materials Science and Nanotechnology Department, Faculty of Postgraduate Studies for Advanced Sciences (PSAS), Beni-Suef University, Beni-Suef, Egypt
XANES Investigations for following the chemical reaction of the formation of CdSe/ZnS Core @ Shell gradient layers with time

N.G. Imam, Jens Uhlig, Kaibo Zheng, Mohamed Abdellah, and Tönu Pullerits

EAEA and Chemical Physics Division, Lund University, and Dept. Chemistry, Danmarks Tekniske Universitet

The Aim of this Project:
To find out these gradient layers and what are their composition along with their contribution (weight ratio %) in the XANES spectrum.

HRXANES spectra have been collected at ESRF @ France for gradient core shell CdSe/ZnS quantum dot structures at both Se (12658 eV) K-edge and Zn (9659 eV) K-edge.

Applications:
The existence of the core shell gradient layers is very important for reducing the structure mismatch which in turns reduces the structure imperfections that act as trapping or recombination centers for the charge carriers. By this way we can increase the lifetime of the charge carriers and increase the efficiency of the CdSe/ZnS system when it is used in the optical based applications (solar cell and lighting applications).
Original Article

Thermal transport properties for unveiling the mechanism of BiSbTe alloys in thermoelectric generation: A glance from synchrotron radiation Bi L₃-XAFS

Shaimaa Elyammy a,*, N.G. Imam b, Giuliana Aquilanti c, Humberto Cabrera d, Abd El-Hady B. Kashyout a

a Electronic Materials Research Department, Advanced Technology and New Materials Research Institute, City of Scientific Research and Technological Applications (SRTA-City), New Borg El-Arab City, P.O. Box 21934, Alexandria, Egypt
b Experimental Physics Department (Solid State Lab.), Nuclear Research Center, Egyptian Atomic Energy Authority, Cairo, 13759, Egypt
c Elettra - Sincrotrone Trieste, Strada Statale 14 – Km 163,5 in AREA Science Park, 34149, Basovizza, Trieste, Italy
d Optics Lab, STI Unit, The Abdus Salam International Centre for Theoretical Physics, Trieste, 34151, Italy
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The solution: Using SR-XAIFS at the Fluo-mode (for extra diluted samples).
# Thanks to the incredible characteristics of SR-based technique of high resolution and high detection limit. down to (to fg/ m3)
Application of XAFS to trace the changes of Mn in the contaminated agricultural soils nearby the historical mine tailings

XAS oxidation state and chemical speciation

This work was performed at XAFS/XRF BL @ SESAME through the accepted pioneer proposal after commissioning in 2018. It was the first time for SESAME to cooperate with Mexican (Outside the Middle East) team through me.
Coupling between γ-irradiation and synchrotron-radiation-based XAFS techniques for studying Mn-doped ZnO nanoparticles

N. G. Imam,\textsuperscript{a} Messaoud Harfouche,\textsuperscript{b,\textdagger} A. A. Azab\textsuperscript{c} and S. Solyman\textsuperscript{d}

\textsuperscript{a}Experimental Nuclear Physics Department (Solid State Laboratory), Nuclear Research Center (NRC), Egyptian Atomic Energy Authority (EAEA), Cairo 13759, Egypt. \textsuperscript{b}Synchrotron-Light for Experimental and Scientific Applications in the Middle East (SESAME), PO Box 7, Allan 19252, Jordan. \textsuperscript{c}Solid State Physics Department, Physics Research Institute, National Research Centre, 33 El Bohouth Street, Dokki, 12622 Giza, Egypt, and \textsuperscript{d}Physics Department, Faculty of Science, Zagazig University, Zagazig, Egypt

*Correspondence e-mail: imam.eaea@gmail.com

Edited by R. W. Strange, University of Essex, United Kingdom (Received 5 February 2022; accepted 21 June 2022; online 15 July 2022)
Original Article

Thermal transport properties for unveiling the mechanism of BiSbTe alloys in thermoelectric generation: A glance from synchrotron radiation Bi L\textsubscript{3}-XAFS

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\textsuperscript{d} Optics Lab, STI Unit, The Abdus Salam International Centre for Theoretical Physics, Trieste, 34151, Italy
Original Article

Influence of polyethylene glycol on the physical properties of Co$_{0.2}$Fe$_{2.8}$O$_4$ nanoparticles used as MRI contrast agent; synchrotron radiation Fe K-edge XAFS

N.G. Imam $^{a,**}$, Mennatallah AbouHasswa $^b$, Giuliana Aquilanti $^c$, S.I. El Dek $^d$*, N. Okasha $^b$, Ahmed A.G. Al Shahawy $^d$

$a$ Experimental Nuclear Physics Department (Solid State Lab.), Nuclear Research Center, Egyptian Atomic Energy Authority, 13759, Cairo, Egypt
$b$ Physics Department, Faculty of Girls, Ain Shams University, Cairo, Egypt
$c$ Elettra - Sincrotrone Trieste, Strada Statale 14 – km 163,5 in AREA Science Park, 34149, Basovizza, Trieste, Italy
$d$ Materials Science and Nanotechnology Dept., Faculty of Postgraduate Studies for Advanced Sciences (PSAS), Beni-
Correlation between structural asymmetry and magnetization in Bi-doped LaFeO$_3$ perovskite: a combined XRD and synchrotron radiation XAS study

N. G. Imam$^1$, G. Aquilanti$^2$, A. A. Azab$^3$, and Shehab E. Ali$^{4,5,*}$

$^1$Experimental Nuclear Physics Department (Solid State Lab.), Nuclear Research Center, Egyptian Atomic Energy Authority, 13759 Cairo, Egypt
$^2$Elettra, Sincrotrone Trieste, Strada Statale 14 – km 163,5 in AREA Science Park, 34149 Basovizza, Trieste, Italy
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$^4$Physics Department, Faculty of Science, Suez Canal University, Ismailia, Egypt
$^5$ALBA Synchrotron Light Facility, Carrer de la Llum 2-26, 08290 Cerdanyola del Vallès, Spain
Materials Research Express

PAPER

Probing the local atomic structure in CoLa$_{0.15}$Fe$_{1.85}$O$_4$ as a function of the synthesis method by multi edge XAFS

N G Imam$^{1,2,3,8}$, G Aquilanti$^{2}$, S F Mansour$^{4,5}$, Zein K Heiba$^{6}$ and Carlo Meneghini$^{7}$

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6 Ain Shams University, Faculty of Science, Physics Dept., Cairo, Egypt
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8 Author to whom any correspondence should be addressed.

E-mail: neama.gomaa_imam@chemphys.lu.se and imam.eae@gmail.com

Keywords: synchrotron radiation, XAFS, XANES, EXAFS, XRD, La: CoFeO$_4$, local atomic structure
Multiferroic applications including radiation sensors, energy harvesting, solid-state refrigeration, data storage recording, and photo-voltaic technologies

BT/LFO is a good candidate for different technological applications including magnetically modulated piezoelectric, safety rechargeable batteries, and multilayer ceramic capacitors.
Thank You very Much

Acknowledgements:

I would like to thank Giuliana Aquilanti and her nice group for giving me this opportunity.

For More Information and for helping in: XAFS Data Analysis, Pre-edge fitting, XANES Calculations (FDMNES Code), EXAFS fitting (Artemis), ...Explanation the extracted data. For helping in writing the XAFS proposal, technical aspects, sample preparation, feasibility of the sample composition...etc.

please do not hesitate to contact me:

neama.imam@elettra.eu