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Synchrotron radiation techniques for soil analysis: a review of basics and methods

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ABSTRACT

Objective: The objective of this work is to present the basics of synchrotron radiation and the fundamentals of some synchrotron-based analytical techniques useful in soil research.
Approach: Synchrotron radiation has revolutionized all areas of human activities. The unique opportunities that synchrotron-based analytical tools offer to vast research areas in the natural sciences is relatively unknown in Mexico and Latin America, with the notable exception of Brazil. This review offers a brief introduction to the principle of operation of a synchrotron radiation facility, its main components and some spectroscopic tools which can be applied to research on soils. We aim to motivate the reader and the researcher on soils to consider the inclusion of synchrotron-based techniques in his/her research activities.
Limitations on study/implications: Synchrotron-based analytical tools offer the possibility of performing analysis at the nanometric scale with elemental sensibility, chemical state (though charge) and orbital selectivity. Other key points of these techniques are the low volume of sample required and the nondestructive nature of the probes, which allow to correlate chemical composition, structure, and physicochemical properties of soils at a very detailed and fundamental level.
Conclusions: Soils are fundamental for the sustainable development of humanity. Regardless of the specific area of soil research in question (composition, fertility, productivity, pollution, remediation), it is possible and desirable to consider the application of synchrotron based spectro-microscopic analytical tools to deepen and broaden our current understanding on any specific topic related to soil science.

Keywords: Synchrotron, X-ray Absorption, Fluorescence, Soils.

INTRODUCTION

Soil is the mineral and organic material that has been generated on the Earth's surface formed by environmental and biological factors, and that allows the growth of plants. It is a fundamental component for supporting life on Earth (Dumanski and Peiretti 2013). The integrity of terrestrial ecosystems depends on the relationships between all the processes that occur in the soil. A deep understanding of the soil requires research, for example, on the chemistry, uptake and distribution of contaminants, amount of dissolved organic matter, among others. However, spectroscopic and analytical methods conventionally used to investigate such phenomena lack the sensitivity necessary for investigation under relevant conditions (Bertsch and Hunter 2001).On the other hand, synchrotron-based techniques are currently established tools sometimes tailored to obtain specific information about several of the processes that occur in the soil, surpassing conventional methods by multiple orders of magnitude (Lehmann and Solomon 2010). In this sense, techniques that use synchrotron radiation have begun to open various opportunities for the study of the phenomena that occur on the soil. This has been possible due to the significant improvements in the spectral and spatial resolution of synchrotron-based instruments in hand with the inherent advantages of synchrotron radiation (SR). Techniques like SR-FTIR (Fourier Transform Infrared Spectroscopy), SR-XRD (X-ray Diffraction), different modalities of X-ray absorption (XAS), emission spectroscopy (XES), and tomography are almost a requirement for the development of a complete understanding of the behavior of elements in soil. Furthermore, this information is necessary to more optimally predict the processes that occur in the soil, develop strategies for soil remediation, in the case of contaminants, and provide strategies to accurately assess risks (Bertsch and Hunter 2001). In this review we present the main components and operation principle of a synchrotron radiation facility. We then discuss the key characteristics of SR and how it is produced. Then we present the fundamentals of several SR-based techniques and a few examples where these techniques found application in various fields of study related to soil science.

Synchrotron radiation basics

Synchrotron radiation (SR) is electromagnetic radiation emitted when electrons, moving at relativistic velocities (close to the speed of light) change their direction under an applied magnetic field. Its virtues are: high intensity and brightness (synchrotron light is hundreds of thousands of times more intense than that from conventional X-ray tubes), beam highly collimated (narrow cone of emission), continuous and tunable in a wide range of photon energies spanning from the infra-red (IR) to the hard X-rays regime (thousands of eV), allows control over its polarization (linear, circular, elliptical) and has time structure due to the time separation of electron bunches in the ring (ns pulse separation) and their width (ps pulse duration) (Margaritondo, 2002) (see Figure 2). It was first observed in 1947 at the General Electric research laboratories in Schenectady, New York. A theoretical description of the phenomena was made by Schwinger (Schwinger, 1949), Sokolov and Ternov (Sokolov and Ternov, 1967). At the beginning, spectroscopists used this radiation in a parasitic manner. In mid-1980s, insertion devices (IDs, wigglers (W) and undulators (U)) using permanent magnets were developed and were implemented in the straight sections of the 2nd generation machines (see Figures 1 and 2). The 3rd generation sources are characterized by being built for the optimal use of undulators. In 1994, the European Synchrotron Radiation Facility (ESRF) in Grenoble opened the era of the 3rd generation light sources. The first batch of machines followed immediately in USA and Asia in the soft (100 eV - 3 keV) and in the hard X-ray regimes (up to 50 keV). The 'Lorentz factor' (γ) is a dimensionless parameter that expresses the ratio of the electron energy Ee to the rest mass energy of the electrons $m_e c^2$ = 511 keV ($m_e = 9.109 \times 10^{-31}$ kg is the electron rest mass and $c = 2.9979 \times 10^8$ m/s is the speed of light). More conveniently can be expressed as $\gamma = 1957\, Ee$ (GeV) and it helps to characterize different synchrotron facilities. Hence, for a soft X-ray optimized synchrotron like the ALS in Berkeley with $Ee = 1.9$ GeV, $\gamma = 3718$, while for a synchrotron radiation facility like the ESRF, optimized for hard X-rays, with $Ee = 6$ GeV, $\gamma = 11800$. Other parameters like circumference, bunch length, energy spread, filling pattern, etcetera, are used to characterize different facilities (Atwood, 1999). Today, there are about 70 SR facilities in all the continents of the world except mainland Africa (http://www.lightsources.org).

Main components and operation of a synchrotron facility

The main components of a SR facility are presented in panel a) of Figure 1. Synchrotrons operate typically with electrons produced under vacuum conditions at the **electron gun** by thermionic emission from a heated tungsten cathode. The emitted electrons packed into bunches nanoseconds apart from each other (Figure 2) are then accelerated to energy in the order of tens of keV by a potential difference applied across the gun. Then they are moved into the linear accelerator. **Vacuum** is crucial in a synchrotron facility because if the electrons were to travel through air inside the storage ring, they would scatter off air molecules and be lost from the electron beam, resulting soon in no emission of SR. Therefore, the electrons circulate around the ring in a vacuum chamber with pressure in the order of 10^{-10} torr. The electrons fired from the electron gun travel through vacuum chambers and reach the **linear accelerator** or **LINAC**. This device accelerates the electron beam in the form of electron bunches to an energy of few hundred MeV by means of a series of **radio frequency** (**RF**) cavities placed over a distance of about 10 meters. At the ESRF for example they reach 200 MeV. After the first meter of acceleration in the LINAC, the electrons are already travelling at more than 99.99% of the speed of light. From the LINAC the electron bunches are transferred into the **booster ring**, which boosts the energy of the electrons from approximately 200 MeV to approximately 1.9 GeV (ALS) to 6 GeV (ESRF), depending on the facility. The booster ring increases the

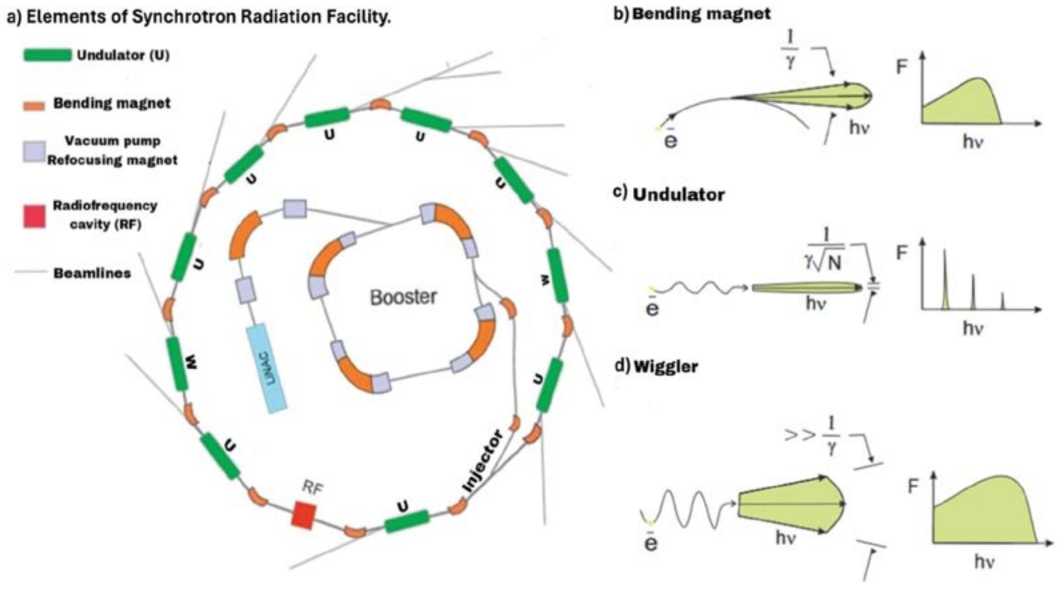

Figure 1. a) Main components of a synchrotron radiation (SR) facility. b), c) and d)are the angular aperture and energy distribution profile of emitted SR from a bending magnet, an undulator and a wiggler, respectively. (see text for details).

speed of the electrons even closer to the speed of light (approximately 99.9999985% of the speed of light) on each turn using a RF voltage source. As the electrons travel around a booster ring, they are kept into a near-circular path by dipolar bending magnets and by quadrupolar focusing magnets known as the lattice. The electrons change their trajectory because of the bending magnets and emit SR, which results in the electrons losing energy and momentum. The RF system provides a synchronous boost to the electrons every time they complete a turn within the storage ring, compensating the energy and the momentum lost through SR emission. At the ESRF, the booster synchrotron has a circumference of 300 meters and a 10 Hz cycling frequency. Once the electrons have reached the nominal energy (1.9 GeV for ALS, 6 GeV for ESRF) they are transferred into the **storage ring** by an **injection system**. Each storage ring can hold few hundred mA circulating inside. For example, the ALS typically operates with 500 mA while the ESRF with 200 mA. Depending on the facility the electrons keep circulating for few hours or even for days in the so called top-off (continuous) mode. Each time electrons pass through magnets which alter their trajectory the electrons lose energy by emitting SR. Each of these magnetic arrays (bending magnets, wigglers and undulators) display a characteristic electromagnetic emission pattern (in terms of energy, brilliance, polarization, and angular aperture, see Figures 1-2) which is directed towards the beamlines. Insertion devices (IDs) as undulators and wigglers are magnetic structures made up of a series of small magnets with alternating polarity that make electron beam to move onto a sinusoidal trajectory (See Figures 1-2). The beams of X-rays they produce are a million times more intense than those generated by the bending magnets and, particularly the undulators have the smallest angular aperture (see Figures 1c-2), the highest brightness and coherence properties that are close to lasers. In an undulator, the electron motion in the transverse direction is set to be on the order of the opening angle. In a wiggler, however, the motion is made to be larger than the opening angle and therefore a wider beam results (See Figure 1 c-d). In an undulator, radiation from the various periods interfere coherently. Sharp peaks are produced at odd harmonics of the resonant frequency (Figure 1c), which depends on the electron energy, the undulator period (λ_u), the field strength of the magnets, and the observation position (Atwood 1999) (see Figures 1-2). Again, to compensate the energy lost by emission of SR, synchronized RF cavities restore and maintain the energy of the electron bunches close to the nominal storage ring energy. After each ID or bending magnet, there is a photon port to allow the tangential extraction of SR light towards the **beamlines** and their research **endstations**. The extraction of SR is achieved by optical devices (slits, mirrors, gratings). The components of each endstation are experiment specific. The 6 GeV storage ring at ESRF has a circumference of 844 m and delivers SR to 44 highly specific beamlines. A complete set of parameters for each SR facility can be found in the machine/parameters section of each facility website.

METHODS

X-ray Absorption Spectroscopy (XAS)

Atoms absorb X-rays sharply at certain energies called absorption edges that are characteristic of that atomic species. Each of these absorption edges have different

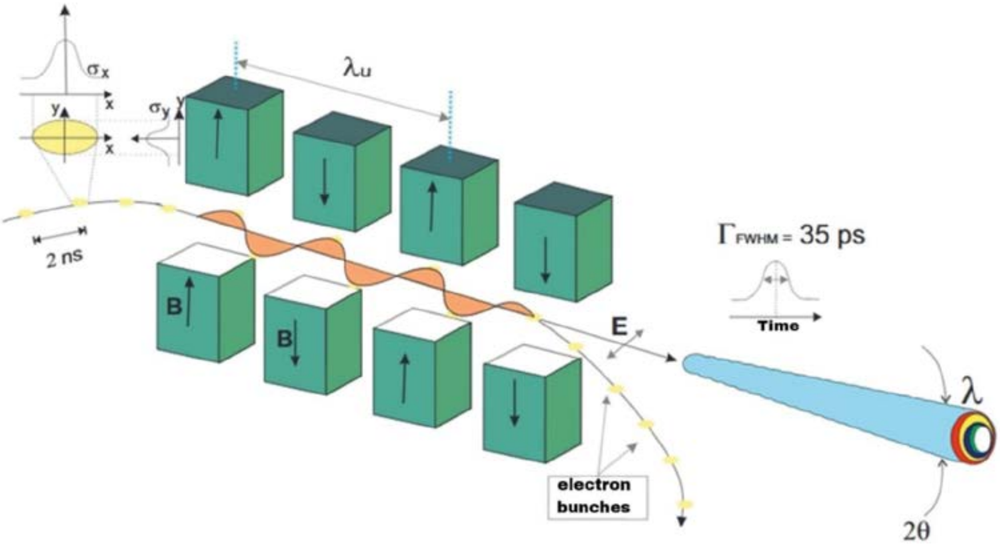

Figure 2. An undulator with its alternating periodic poles (λ_u) induces electron bunches (separated nanoseconds apart from each other) to move in a sinusoidal trajectory between the magnetic arrays, resulting in a narrow cone of SR emission.

electron occupation numbers with characteristic binding energies (see Figure 3 a-d). **XAS** can have different names depending on the energy range used. It is an element and orbital specific technique governed by dipolar radiation selection rules (Atwood, 1999; Groot and Kotani, 2008). Today, **Near Edge X-Ray Absorption Fine Structure (NEXAFS)** is typically used for soft X-ray absorption spectra (low Z elements) (Figure 3a) and **X-ray Absorption Near Edge Spectroscopy (XANES)** for hard X-rays (heavy elements, metal atoms). With more detail, XANES refers to the absorption of X-rays about 10 eV below and 20 eV above the absorption edge. This region usually shows the largest variations in the X-ray absorption coefficient and is often dominated by intense, narrow resonances (see Figures 3b and 3f). Either as NEXAFS or XANES, XAS provides information on the electronic structure of the unoccupied levels or conduction band (CB) (see Figures 3a-b) and the geometry surrounding the absorbing element. Different oxidation states of transition metals are observed in this region by relative shifts in the absorption onsets: the more oxidized the species the more it shifts towards higher photon energies. The pre-edge structures at the onset of the XANES spectrum (Figure 3f) typically corresponds to quadrupolar transitions (for example 1s to 3d orbitals) in transition metal elements (Groot and Kotani, 2008). The higher energy region in XANES spectra is typically referred to as the **Extended X-ray Absorption Fine Structure (EXAFS)**, typically starts ∼50 eV above the absorption edge. The so-called EXAFS oscillations originate from the scattering of photoelectrons as waves ejected from the absorbing atom and its neighbors interfering constructively and destructively among them (Figure 3g). Therefore, EXAFS is typically used to determine the local structure of the studied element and its surrounding atoms (Groot and Kotani, 2008).

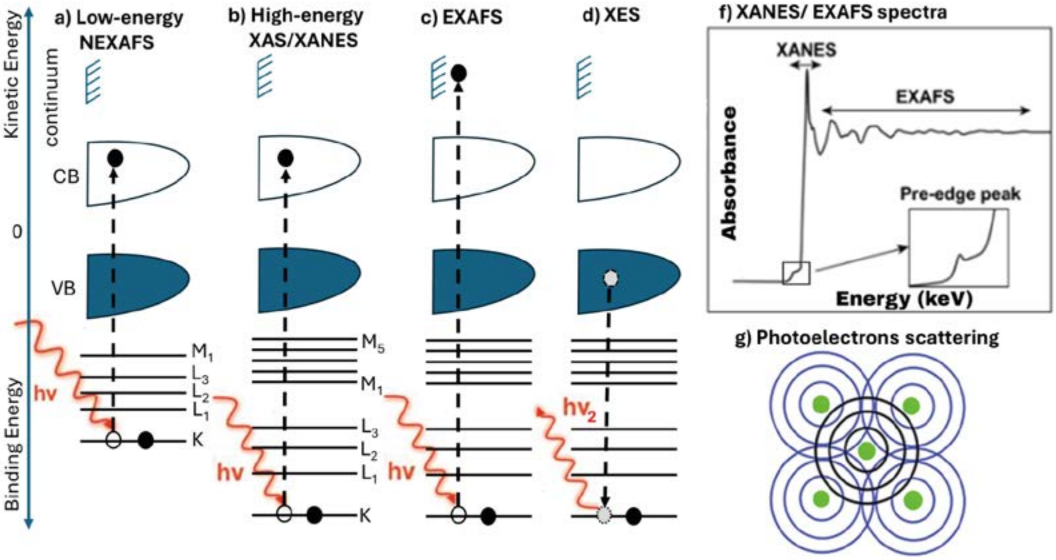

Figure 3. Basic processes in X-ray absorption spectroscopy (XAS) in its different varieties: a) NEXAFS, b) XANES, c) EXAFS, f) XANES/EXAFS spectrum and the scattering process of photoelectrons which produces EXAFS oscillations. d) X-ray emission process in XES spectroscopy. K-M are characteristic absorption edges of atoms in a solid with its valence band (VB) and conduction band (CB) $h\nu/h\nu_2$ are the incoming/outgoing X-rays. See text for details.

X-ray emission spectroscopy (XES)

XES is also known as **X-ray Fluorescence (XRF)** in the literature (Groot and Kotani, 2008). X-ray emission occurs as a core hole is relaxed after photoionization (ejection of an electron after X-ray irradiation) from a core level. The photon generated during the relaxation of the core hole has the energy corresponding to the energy difference of the atomic levels involved, in Figure 3d $h\nu_2 = E(1s) - E(VB)$. A XES spectra provides information on the elemental/orbital composition of the valence band (VB). The relaxation of the core hole can also occur through the Auger process, where instead of emitting X-rays the excited atom emits electrons with well-defined kinetic energies. Both processes compete and are Z and absorption-edge dependent: core holes in lighter atoms predominantly decay via Auger processes, while heavier elements do it via emission of X-rays (Atwood 1999). At this point is important to emphasize that both techniques XAS and XES require a SR facility. This is so because to register the XAS/XES spectra we need to scan the incoming photon energy and tune it to the absorption edges of the various elements in our sample(s). In addition, to enhance the X-ray emission signal, we need to create the largest possible number of core holes in our atom, and thus we need an undulator beamline with the highest photon flux possible. At this point, the potential that these techniques offer for agriculture research should be evident, see for examples the work of Castillo *et al.* on plants and engineered nano materials (Castillo-Michel, Larue *et al.*, 2017) and Lanziroti *et al.* on soils (Lanzirotti, Tappero *et al.*, 2010). Having reviewed the basic atomic processes involved in X-ray spectroscopies, we will proceed to discuss some other SR techniques bassed in these processes and their application to soil research.

X-ray computed μ-tomography (SR-μCT)

X-ray tomography is the construction of a three dimensional image from two dimensional projections (slices) taken at different orientations. According to Lombi and Susuni (Lombi and Susini, 2009), X-ray computed μ-tomography (SR-μCT) is a technique that provides high contrast and high resolution 3D images by combining μCT and SR that leads to detailed intern structure analysis for materials inner structure, without the need of dissecting. Therefore, X-ray computed μ-tomography (SR-μCT) is a non-destructive technique. There are three modes of contrast for imaging with X-rays: X-ray absorption, fluorescence and phase contrast. The X-ray beam used permits the imaging of non-geometrical shapes in the sample. Many applications in soils and vegetable tissue are described for this technique. Peth (Peth, 2010) highlights the use of SR-μCT for studying the biophysical and physicochemical properties of soil aggregates. In this case, the technique allowed to image natural undisturbed soil samples, showing the patterns of internal pore structures. These spaces are important because it is habitat for soil microbiome, and the physical properties of the pore may be involved in regulation of microecological relations. SR-μCT also was used for comparing topology and morphology of pores in different tilled soils.

Absorption SR-μCT

In X-ray absorption computed μ-tomography (SR-μCT), the contrast is achieved by registering the response of the sample at energies below and above the energy of the element of interest, it works better for heavy elements that strongly absorb X-rays. The use of a monochromator to select the photon energy is mandatory. This technique requires less sample to work with but needs a long distance between the SR source and the tomography facility. First, the X-rays beam needs to be attenuated with a scintillator that converts it into visible light. Then, the absorption projection is captured by a detector (usually a CCD camera). The sample is slightly rotated to receive another beam and generate the next image. The process is repeated and finishes when the sample completes an arc between 0° and 180°. The images generated are reconstructed to allow a 3D visualization. As mentioned above, it is possible to obtain a set of images above and below an elemental absorption edge and get a 3D distribution of it in the sample. The exposure of the sample to SR is only a few seconds per image with a micrometric resolution. Scheckel *et al.* (2007) used this technique to enlighten how *Iberis intermedia* absorbs and bioaccumulates thallium in a differential way within the plant structures. This study shows a promising area for phyto-mining and soil phytoremediation because thallium is a precious metal.

SR-XRF-μCT

This technique combines the quantitative approach of XRF and μCT for 3D imaging and composition information of the analyzed sample yielding the spatial distributions of the elements. Xie *et al.* (Xie, Deng *et al.*, 2020) describe XRF computed tomography (XFCT) used in the Shanghai SR facility. A beam of SR X-rays is used to create core holes in the atoms of the elements of interest within the sample. This generates a fluorescent

X-ray whose energy is unique for each element. The sample is then rotated to acquire various images that will be processed to create a 3D representation. For this technique raster scanning is needeed, therefore this technique is slow in comparison with phase contrast and X-ray absorption modes, and hence radiation damage maybe an issue for high resolution measurements. Other drawbacks to consider are 1) the energy at which the analysis is done impacts the elements to be detected and 2) low (Z) energy can only be analyzed in thin samples (remember low XES probabilty vs Auger for low Z), else XRF form within the sample does not reach the detector. This robust technique has been widely used for studying the interactions of pollutant removing plants and soil, showing that chemical speciation of contaminants varies in different plant structures (Lombi and Susini, 2009).

Phase contrast SR-μCT

This technique is based on the fact that SR is affected by both the absorption of the materials and their phase.The contrast between phases is more remarkable at the absorption edge, especially if materials at the interface have different refractive index. While producing μ-CT images, SR through the sample lowers its energy, so the attenuation is registered and used to build the 3D image. Due the relation between refractive index an electron density, the images generated show a 3D map for electron density at micrometric scale (Lombi and Susini, 2009). The procedure is similar to absorption SR-μCT (Indore, Karunakaran *et al.*, 2022) but differs in the distance from the detector. This method is more sensitive to small differences in refractive index, so it increases the contrast of edges in the sample's borders allowing to detect soft materials in it. Experimentally, this technique is fast and, as mentioned it is very sensitive. It is typical to use high energy X-rays (energies greater than 20 keV) directly form the source as white or pink beam, and hence no monochromator is needed. This technique has been used by Ma *et al.* (Ma, Cai *et al.*, 2015) for studying changes in the pore inner structure in two different soils during various cycles of wetting and drying. It showed the presence of a very complex pore network due the clay composition in soil while wetting.

Synchrotron Radiation X-Ray Diffraction (SR-XRD)

The conventional X-ray diffraction system is limited to crystalline materials and has the drawbacks of requiring large sample quantities, long time for analysis, low resolution, beam hardening, low photon energy, low photon flux, and fixed wavelength. SR-XRD provides outstanding resolution and sensitivity, even with very small samples. This enables the identification and quantification of trace phases, something not achievable with conventional X-ray sources (Surabhi, 2021). Compared with lab-based sources, SR based X-rays have a much shorter, providing much greater spatial resolution and penetration capability. They interact with the atoms of the sample, producing characteristic diffraction patterns that can be detected (Manceau, Marcus *et al.*, 2002). SR-XRD enables the non-invasive study of trace matters, amorphous minerals, hydrated and oxygen samples, thin films and solution phase. The study of areas as small as 1 mm^2 is rutinary (Surabhi, 2021) although it is also possible t perform SR-XRD-μCT.

SR-Fourier Transform Infrared Spectro-microscopy (SR-FTIR)

In general, infrared radiation interacts with the sample and is absorbed to varying degrees depending on the molecular vibrations present in the sample. The absorption of energy results in absorption spectra containing information about the characteristic vibrational bands of the chemical bonds present in the sample. SR-FTIR spectro-microscopy integrates three methodologies: microscopy, mid-infrared spectroscopy, and SR. Due to its superior spatial resolution (down to micrometers) and enhanced signal-to-noise ratio (SNR) (given the high photon flux from SR facilities in comparison to traditional FTIR methods), this approach enables a quantitative analysis, composition, structure, and distribution of chemical constituents within the sample. SR-FTIR spectro-microscopy allows for the extraction of more intricate structural details, characterization of soil mineral components, including mineral identification, structural assessment, and in situ monitoring of mineral formation (Margenot, Calderón *et al.*, 2017).

Scanning Transmission X-ray Microscopy (STXM)

STXM is a transmission microscopy technique able to perform XAS with spatial resolution of about 20-nm. STXM provides two-dimensional (2D) quantitative information about the distribution of chemical components and interactions in the soil (Obst and Schmid, 2014). The technique can be adapted for the use of hard and soft X-rays. Each regime has its own optics and instrumentation and offers different advantages. Hard X-rays, have higher energies, can penetrate deeper into samples (which is useful for studying internal structures and buried components of the soil). Therefore, they are used for the detection of heavy elements and to obtain structural information at greater depths. Typical applications include the characterization of heavy minerals, detection of metallic contaminants, and evaluation of soil microstructure. Soft X-rays, on the other hand have lower energies (typically between 100 and 2200 eV), are more sensitive to light elements and organic compounds, providing detailed information about the surface chemistry and the upper layers of the soil. Therefore are used to study organic composition, chemical interactions, investigation on nutrient cycles, and the speciation of light elements in the surface of the soil (Obst and Schmid, 2014). STXM in both X-ray regimes allows for a comprehensive and multidimensional characterization of soil, encompassing both organic and inorganic components, and providing a more integral understanding of soil chemistry and structure.

Limitations for Mexican and Latin American Researchers

Probably the four most important limitations for employing SR in soil studies for Mexican researchers are: 1) the lack of a SR facility in Mexico, 2) the necessary funding for even a small team (2 people) traveling abroad to perform experiments, even to the SR facilities in America (USA, Canada and Brazil), 3) the lack of previous experimental experience and 4) the very competitive process of beamtime allocation (which often requires previous SR experience). Fortunately, it is possible to reach for experienced users aiming to collaborate. It is also advisable to contact the beamline scientists in charge of the targeted instrument in advance to discuss the viability of our research project, and who sometimes even help with preliminary measurements to support our research proposals. It is necessary to create options, several alternatives are possible: like developing collaborative schemes at the University-SR facility or even at binational level to favor the training of graduate students. For example, the Brazilian SR facility partially supports the trip of Latin American students provided beamtime has been awarded through the regular proposal mechanism. Despite all these limitations, we must keep trying to incorporate SR techniques into our research, we must continue submitting research proposals and continue to train the next generation of Mexican researchers well versed in SR techniques, especially if we ever aspire to host a SR facility.

CONCLUSIONS

In this brief review we presented the most important SR based techniques applied to soil science. We discussed the basic operation of a SR facility as well as the main characteristics of the SR. The properties of synchrotron radiation allow for the development of unique tools with extraordinary analytic capabilities with spatial resolution at the nanometric level, with molecular, elemental, charge, and orbital resolution. The aim of this review is to promote the incorporation of SR based techniques among Mexican and Latin American researchers performing research in soils.

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