Analysis of Soils and Minerals Using X-ray Absorption Spectroscopy

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X-ray absorption spectroscopy (XAS) has been applied to numerous problems in soil science, mineralogy, and geochemistry. X-ray absorption spectroscopy was developed in the early 1970s (Sayers et al., 1971) and is widely used at synchrotron radiation facilities. Regardless of the complexity of the sample, the XAS signal comes from all of the atoms of a single element as selected by the X-ray energy. High-quality XAS spectra can be collected on heterogeneous mixtures of gases, liquids, and/or solids with little or no sample pretreatments, making it ideally suited for soils and many other systems. The structural information obtained from XAS is useful for identifying the chemical speciation of an element, including mineral, noncrystalline solid, or adsorbed phases. With the addition of X-ray focusing, samples can be interrogated on length scales comparable to or smaller than some level of natural heterogeneity, thus making it possible to study differences in the atomic environment of an element within or between individual particles and grain boundaries.

The acronym XAS covers both X-ray absorption near edge structure (XANES) and extended X-ray absorption fine structure (EXAFS) spectroscopies. X-ray absorption near edge structure can be used to determine the valence state and coordination geometry, while EXAFS can be used to determine the local molecular structure of a particular element within a sample. Micrometer-length scale X-ray measurements are designated μ-EXAFS and μ-XANES. Another variation to the XAS technique utilizes the natural linear polarization of synchrotron X-rays. The use of polarized X-rays allows the atomic environment of the absorber to be probed in the polarization direction and is particularly suited for minerals with layer-type structures such as phyllosilicates and manganese oxides (Manceau et al., 1999).

The nature of a complex sample is best revealed by the application of several different experimental techniques, with each individual measurement providing both unique and complementary information. The most common technique for characterizing abundant soil minerals is X-ray diffraction (XRD), which relies on long range ordering of atomic planes to probe crystalline structure at a length scale of approximately 50 Å or more. X-ray absorption spectroscopy probes the immediate environment of the selected element, within about 6 Å, and its theory and interpretation does not rely on any assumption of symmetry or periodicity. While both XRD and XAS can be used to determine distances between atoms, the information is derived from two very different X-ray interactions with the sample. For most systems the application of XRD and XAS is complementary.