

# Laboratory-scale NEXAFS spectroscopy: the challenge of sample preparation and first spectra of Fe oxides and natural organic matter

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For NEXAFS spectroscopy, synchrotron radiation is up to date almost exclusively the method of choice. The advent of laser-driven plasma sources for X-radiation is beginning to change this. Recently, a table-top NEXAFS setup based on such a source has been developed at the Institut für Nanophotonik (former Laser-Laboratorium) Göttingen. In the X-ray energy range <1 keV it allows for NEXAFS spectroscopy such as C1s, N1s, O1s, Ca2p, Mn2p, and Fe2p. These instruments will be a way out of limited measurement options using synchrotron radiation when investigating complex environmental samples including organic matter, Fe-organic complexes, carbonates, and the often poorly crystalline Mn and Fe minerals.

A first challenge for such measurements is to produce suitable samples on thin transparent membranes without altering the sensitive environmental materials. A suitable sample consists of a chemically homogeneous, uniformly thin (<1 µm), continuous layer. So far, we focused on sample preparation by drop deposition under different conditions. The behavior of solutes and particles within a sessile drop is controlled by interactions between the solid-water, water-air, and solid-air interfaces. Concentration gradients within the drying droplet can lead to hydrodynamic flow and strongly influence both aggregation and location of the deposits. In unfavorable, but not uncommon cases, the material is deposited either at the edge of the droplet (coffee rings) or in its center due to Marangoni flow phenomena. Nevertheless, preparation trials with pure iron (oxyhydr)oxides and natural dissolved organic matter resulted samples that sufficiently meet measurement requirements, when a certain droplet size and particle concentration was chosen. Sedimentation and spin-coating are promising preparation techniques to be tested in the future.

NEXAFS spectra of a variety of iron (oxyhydr)oxides at the O K-edge and the Fe L-edge and from an organic water extract of a forest floor layer at the C K-edge allow to distinguish all features known from synchrotron spectra. Peak positions of synchrotron and table-top spectra deviate by only 0.1%.