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Mineral quantification in mixtures of synthetic goethite, hematite and ferrihydrite by table-top NEXAFS at the O K-edge

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Fe oxides offer large and reactive surfaces and are of special importance for the adsorption of e.g. organic matter, phosphate and arsenate in soils and sediments. Because of their often poorly crystalline nature, these oxides are difficult to identify and quantify. Here, we tested whether quantification of mineral phases is possible by NEXAFS at the O K-edge in binary mixtures of synthetic goethite, hematite and ferrihydrite. NEXAFS spectra were collected in transmission mode with a laser-driven table-top setup based on the ignition of a Kr gas jet yielding an x-ray radiating plasma in the energy range of 250-1000 eV. Sample preparation was challenging as chemically homogeneous, uniformly thin (<300 nm), continuous layers of sample material had to be deposited on fragile silicon nitride membranes to detect undistorted spectra. Such specimens could be prepared by dropping sample suspensions onto membranes previously exposed to an air plasma. Additionally, the hydrodynamic flows that form in sessile, drying droplets were manipulated by an air stream to prevent the formation of so-called coffee rings.

NEXAFS spectra of pure Fe oxides were found to be very similar to synchrotron measurements. Spectra of hematite-goethite mixtures could be reconstructed by linear combinations of reference spectra with an accuracy of at least 10 %. Work on other mixtures and mixtures including Fe-organic complexes or clay minerals is in progress.