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Quantitative characterization of biotic iron oxides by analytical electron microscopy

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Abstract

Hydrous ferric oxides (HFO) play a tremendous role in the environment, and as a result, they have often been analyzed for their bulk chemical and mineralogical composition, but rarely at the particle level. This study used a combination of transmission electron microscopy (TEM), parallel electron energy loss spectroscopy (TEM-PEELS), and selected area electron diffraction (TEM-SAED), to determine the morphology, stoichiometry, and mineralogy of synthetic biotic HFO and assess the effect of specimen preparation (i.e., dehydration) on their overall chemical composition. A mixture of synthetic lepidocrocite (γ-FeOOH) and goethite (α-FeOOH) was used as reference material for all TEM analyses. HFO were formed by the oxidation of Fe^{2+} (pH = 8) in the presence of *Bacillus subtilis* (bacteria without extracellular polymers) and *Bacillus licheniformis* cells (with extracellular polymers). Biotic Fe-oxides occurred as small particles (5–10 nm diameter) on the bacterial cell walls and associated exopolymers and were identified as 2-line ferrihydrite by TEM-SAED. Quantitative TEM-PEELS results showed that (1) freeze-dried abiotic samples had an Fe/O atomic ratio of 0.54 ± 0.03, whereas the Fe/O ratio of the biotic ferrihydrite formed in the presence of *B. licheniformis* and *B. subtilis* was 0.48 ± 0.06 and 0.54 ± 0.08, respectively; (2) air-dried biotic ferrihydrite possessed an Fe/O ratio close to 0.30, whereas the ratio of the abiotic air-dried HFO samples remained unchanged (i.e., 0.51 ± 0.04). These results indicate that quantitative TEM-PEELS can be used for the chemical characterization of Fe-oxides, since the measured Fe/O atomic ratios of the reference HFO are in agreement with their known stoichiometry (i.e., 0.50 for FeOOH). Our results also suggest that ferrihydrite dehydrates during freeze-drying and that specimen preparation is very important when assessing chemical composition.