Influence of Silicon and Phosphorus on Structural and Magnetic Properties of Synthetic Goethite and Related Oxides

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Abstract: A series of synthetic goethites containing varying amounts of Si and P dopants were characterized by X-ray powder diffraction, electron diffraction, microbeam electron diffraction, and Mössbauer spectroscopy. Very low level incorporation produced materials having structural and spectral properties similar to those of poorly crystalline synthetic or natural goethite. At higher incorporation levels, mixtures of noncrystalline materials were obtained which exhibited M6ssbauer spectra typical of noncrystalline materials mixed with a superparamagnetic component. Microbeam electron diffraction indicated that these mixtures contained poorly crystalline goethite, poorly crystalline ferrihydrite, and a noncrystalline component. If the material was prepared with no aging of the alkaline Fe^{3+} solution before the addition of Na_2HPO_4 or Na_2SiO_3, materials were obtained containing little if any superparamagnetic component. If the alkaline Fe^{3+} solution was aged for 48 hr before the addition, goethite nuclei formed and apparently promoted the precipitation of a superparamagnetic phase. The M6ssbauer-effect hyperfine parameters and the saturation internal-hyperfine field obtained at 4.2 K were typical of those of goethite; however, the Mössbauer spectra indicated that the ordering temperature, as reflected in the relaxation rate and/or the blocking temperature, decreased with increasing incorporation of Si and P. The complete loss of crystallinity indicates that Si and P did not substitute for Fe, but rather adsorbed on crystal-growth sites, thereby preventing uniform crystal growth.

Key Words: Electron diffraction • Ferrihydrite • Goethite • Mössbauer spectroscopy • Phosphorus • Silicon • X-ray powder diffraction

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