Synthesis and Properties of Poorly Crystalline Hydrated Aluminous Goethites

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Abstract: Al-substituted goethites were prepared by rapid oxidation of mixed $FeCl_2$-$AlCl_3$ solutions at pH 6.8 in the presence of CO_2 at 25 ° C. A combination of Al substitution and adsorption of CO_2 reduced crystal size (except for an increase at small additions of Al) and produced unusual thin, porous particles. Product goethites had surface areas up to 283 m_2/g and unit-cell expansions induced by hydration. Substitution of Al for Fe reduced the 111 spacing and increased infrared OH-bending vibrational frequencies. Al substitution split the goethite dehydroxylation endotherm during differential thermal analysis into a doublet and increased the temperature of all reactions. Both cold and hot alkali solutions dissolved Al from the goethite structure.

After drying the product in vacuo at 110 ° C. X-ray powder diffraction data indicated minimal deviation from Vegard's law for the goethite-diaspore solid solution up to about 30 mole % Al substitution. Goethite prepared in the presence of 40 mole % Al had a 111 spacing of 2.403 Å corresponding to 36 mole % structural Al if Vegard's law was obeyed. Rapid oxidation of mixed $FeCl_2$-$AlCl_3$ solutions appears to be conducive to a higher degree of Al substitution in goethite than alkaline aging of hydroxy-Fe(III)-Al coprecipitates.

Key Words: Aluminum • Diaspore • Goethite • Infrared • Synthesis • Vegard's law • X-ray powder diffraction

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