
Infrared Vibrations of Hematite Formed From Aqueous- and Dry-Thermal Incubation of Si-Containing Ferrihydrite

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Abstract: Ferrihydrite samples having Si/Fe molar ratios ranging from 0 to 1 were synthesized by the reaction of $\text{Fe}_2(\text{SO}_4)_3$ and Na_2SiO_3 with NaOH to an equilibrium pH of 8.2. Hematite formed by incubating the ferrihydrite (Si/Fe molar ratios ≤ 0.05) at pH 12.5 and 91°C for 36 hr had a globular morphology. Hematite formed from Si-free ferrihydrite gave infrared (IR) bands at 548, 471, 397, and 337 cm^{-1} , whereas, hematite formed from Si-containing ferrihydrite having 0.001 to 0.05 Si/Fe molar ratios gave broad IR bands at about 550, 450, and 330 cm^{-1} . Ferrihydrite having Si/Fe molar ratios ≥ 0.10 did not transform to hematite following the aqueous-thermal treatment.

The ferrihydrite samples were thermally treated for 2 hr at consecutive 100°C intervals from 100° to 800°C . The Si-free ferrihydrite transformed at 300°C to poorly crystalline hematite. Transmission electron microscopic analyses indicated that the hematite consisted of aggregates of spheroidal particles of 20 – 80 Å cross sections. Broad IR bands were observed at 529 and 452 cm^{-1} ; however, after heating the sample to 800°C , the particle cross sections increased to about 150 – 600 Å, and additional IR bands were present at 378 and 325 cm^{-1} . The differences in the IR patterns of hematite formed from ferrihydrite at 300° and 800°C were probably due to increases in particle size and aggregation and improved crystallinity of the hematite particles following the higher temperature treatment. The hematite formed by the thermal transformation of the ferrihydrite having a 0.01 Si/Fe molar ratio was also spheroidal, and IR vibrations were present at about 528 and 443 cm^{-1} . An increase in the temperature of the thermal treatment, however, did not result in additional IR bands.

Differences in the IR vibrations of hematite formed during aqueous- and dry-thermal treatments of the ferrihydrite samples were probably due to differences in the particle size and morphology of the product. The Si content, due to its effect on particle size of the precursor and the prevention of sintering and particle growth of hematite, influenced the IR pattern of the product. Particle morphology and IR spectroscopy may therefore be useful indicators of the precursor of hematite and the conditions of hematite formation in soil.

Key Words: Ferrihydrite • Hematite • Infrared spectroscopy • Morphology • Silicon • Thermal treatment

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