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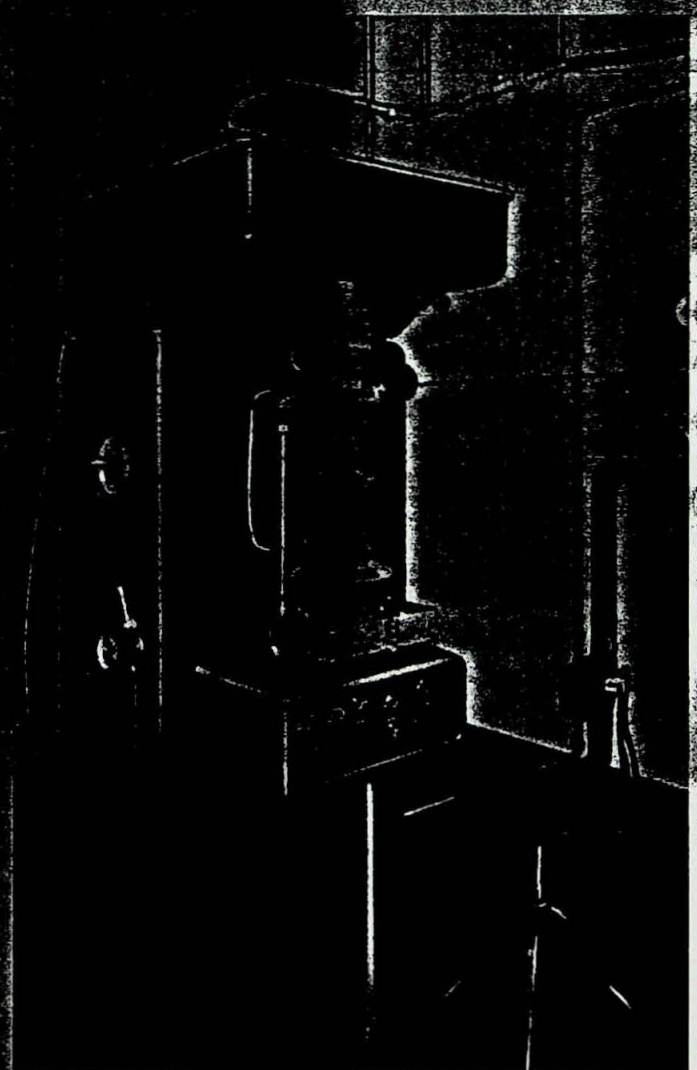

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ELECTRON MICROSCOPE OBSERVATIONS OF NATURAL AND SYNTHETIC GOETHITES

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In laboratory studies goethite (α-FeO.OH) is one of the most used iron oxide minerals, partly because it is the most widespread Fe oxide in natural environments. Partial replacement of Fe in the structure by another metal ion can modify such properties as crystal morphology, crystal size, and solubility. Hematite (α-Fe_2O_3) may occur associated with goethite in natural samples (figures 1 and 2).

The electron microscope is much more sensitive than X-ray diffraction for investigating crystal morphology. The relevant properties of the solid depend not only on the composition, but also crucially on the size and shape of the particles. The morphological properties of the iron oxide particles depend on the competition between the processes that control the development of a solid particle from a solution. These processes are nucleation, growth, aggregation and impurities adsorption.

Since the first report of Böhm in 1925 on synthesis of pure goethite, several attempts have been made to prepare material of uniform shape and size. Goethite can be synthesized in a few days by using high OH^- concentrations and temperature of 70° C. The properties of goethite have been studied in considerable detail (Schwertmann & Cornell, 1991).

Goethite may be synthesized from either Fe(III) or Fe (II) systems. The preparation from Fe (III) systems consist of pour 100 mL of solution 1 M $Fe(NO_3)_3$ into a 2 L polyethylene flask, and add rapidly and with stirring, 180 mL solution 5 M KOH. Redbrown ferrihydrite precipitates at once. Immediately dilute the suspension to 2L with distilled water and hold in a closed polyethylene flask at 70° C for 60 hours. During this period, the voluminous, red brown suspension of ferrihydrite is converted to a compact, yellow brown precipitate of goethite. Remove the reaction vessel from the oven, and centrifuge, wash and dry the precipitate. Washing the product is essential to remove OH^- and NO_3^-. This method produces 9 g pure goethite. Goethite crystals are usually acicular and elongated along the crystallographic *c* direction (figures 3 and 4).

Reference

SCHWERTMANN, U. and CORNELL, R. M. (1991) Iron oxides in the laboratory. VCH, Weinheim.

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