
Evaluation of Crystallinity in Hydrated Ferric Oxides*

Edward R. Landa and Robert G. Gast[†]

University of Minnesota, St. Paul, Minnesota 55101, U.S.A.

* Paper N. 8140 Scientific Journal Series, Minnesota Agricultural Experiment Station, University of Minnesota, St. Paul, Minnesota 55101. This work was supported by the Graduate School, University of Minnesota and by a National Science Foundation Traineeship to the senior author.

[†] Research Assistant and Professor of Soil Science, respectively, University of Minnesota, St. Paul, Minnesota 55101, U.S.A.

Abstract: The nature of freshly-precipitated and aged hydrated ferric oxides prepared by the addition of ferric chloride to KOH was investigated by the use of scanning and transmission electron microscopy, X-ray diffraction, i.r. absorption, and pH 3 · 0 ammonium oxalate extraction. The results show the fresh material to be essentially non-crystalline hydrated ferric oxide, which when aged at 60° C C and high pH rapidly crystallizes as goethite, without any indication of coexisting hematite. The various methods were evaluated as indices of crystallinity for aging materials. The acid ammonium oxalate method was shown to extract selectively only the non-crystalline portion of such mixtures. The use of X-ray diffraction analysis for estimating aging stage requires elimination of the preferred orientation of the goethite crystals. While both the oxalate and X-ray methods can detect as little as 2 per cent crystallinity, the oxalate method is probably superior for quantitative determinations as it depends directly on an inherent difference in the solubility of the crystalline and non-crystalline materials, rather than on a technique dependent intensity measurement. The use of the intensity of the O-H bending vibrations of the infrared absorption spectra can also potentially detect as little as 2 per cent crystallinity, but the procedure is probably less useful for quantitative determinations than the oxalate or X-ray methods because of the problem of evaluating the area under the peaks.

Clays and Clay Minerals; April 1973 v. 21; no. 2; p. 121-130; DOI: [10.1346/CCMN.1973.0210208](https://doi.org/10.1346/CCMN.1973.0210208)

© 1973, The Clay Minerals Society

Clay Minerals Society (www.clays.org)
