Quantitative Analysis of Poorly Crystalline Fe_2O_3 Specimens

Tomas Grygar[a], Petr Bezdicka[a], Veronika Simova[a], Oldrich Schneeweiss[b], Radek Zboril[c], [a]*Institute of Inorganic Chemistry AS CR, Rez, Czech Republic.* [b]*Institute of Physics of Materials AS CR, Brno, Czech Republic.* [c]*Palacky University, Olomouc, Czech Republic.* E-mail: grygar@iic.cas.cz

We analyzed amorphous and nanocrystalline natural and synthetic ferric oxides by X-ray diffraction (XRD), Mossbauer and UV-Vis spectroscopies, voltammetry of microparticles, and thermoanalytical methods. The quantitative analytical methods were tested with intermediates of the thermal conversion of ferrihydrite to nanocrystalline hematite.

The XRD measurement with the addition of a known amount of reference material (Si) is not applicable when the mean coherence is only a few nm. Moessbauer spectroscopy and voltammetry of microparticles of such poorly ordered solids should be supported by the results of other methods, but under optimal conditions they are expected to "see" both amorphous and crystalline components with a comparable sensitivity permitting their determination. However, the minimal particle size of the target compounds is not known for Mossbauer spectroscopy and voltammetry of microparticles, and it seems to be equal or even larger than mean coherence length enabling XRD measurement. Diffuse reflectance spectroscopy can only be used to qualitative analysis, because the spectra of ferrihydrite and almost amorphous Fe_2O_3 are hardly interpretable.

The results indicate a lack of the knowledge on a local structure of 2-line ferrihydrite and XRD amorphous ferric oxides.

Keywords: amorphous materials, nanocrystalline materials, iron oxides