EFFECT OF REACTION TEMPERATURE ON PROPERTIES OF IRON(III) OXIDE NANOPARTICLES PREPARED BY SOLID-STATE ROUTE FROM IRON(II) ACETATE

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Thermally induced oxidative decomposition of iron(II) acetate was studied in air at various temperatures between 245 and 400 °C using thermogravimetry, DSC, elemental analysis, transmission electron microscopy, X-ray powder diffraction, low temperature and in-field Mössbauer spectroscopy and BET surface area measurements. Independently on reaction temperature, maghemite (γ-Fe_2O_3) was identified as the only decomposition product. In the temperature range of 320 – 400 °C, the particle size of maghemite can be controlled from 6 to 20 nm as manifested in TEM images. The increase in particle size with temperature is reflected also through decreasing surface area from 147 to 51 m^2/g, narrowing of XRD lines and gradual prevailing of the sextet fraction at the expense of superparamagnetic one in Mössbauer spectra. Sample prepared at 320 °C containing small superparamagnetic particles (see TEM in Fig. 1a) was analyzed by in-field Mössbauer spectroscopy (20K/5T) to consider the structural ordering of maghemite and degree of spin frustration (see Fig. 1b). The ratio of

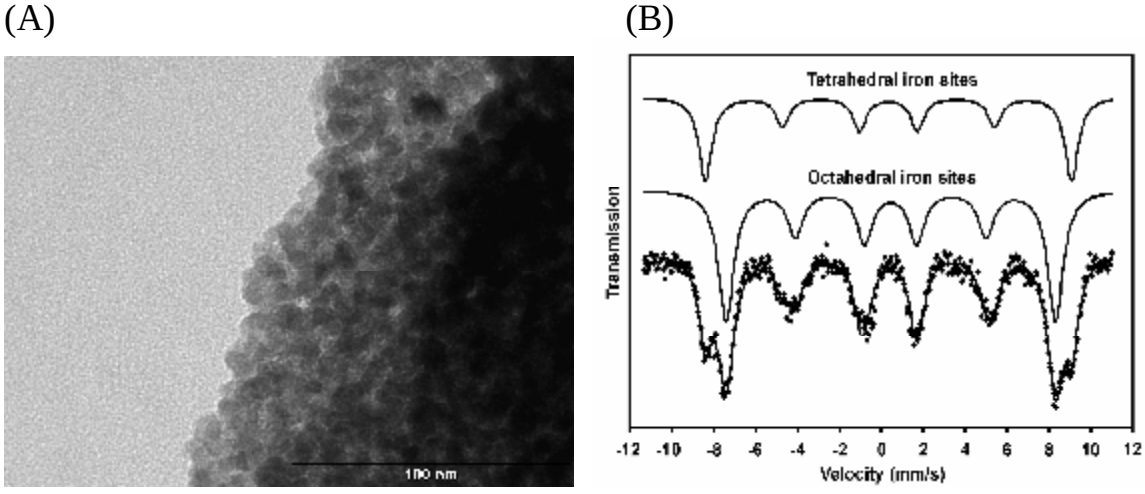

Figure 1. TEM micrograph (A) and in- field Mössbauer spectrum (B) of γ-Fe_2O_3 nanoparticles prepared from iron(II) acetate at 320 °C. Spectrum taken at 20 K in an external field of 5 T applied parallel with gamma-ray.

octahedral to tetrahedral Fe(III) positions being near to 5/3 evidences for the well ordered structure, however the increased intensities of the 2^{nd} and 5^{th} spectral lines confirm a high degree of spin frustration as expected due to the interparticle interactions and evolution of surface anisotropy [1]. All samples synthesized at 320-400 °C exhibit, after dispersion in bentonite matrix, the excellent contrast properties in magnetic resonance imaging. The anomalous decrease in surface area of samples heated at 245-300 °C is discussed with respect to the possible crystallization effect of primarily formed amorphous particles.

References:
[1] J. Tucek, R. Zboril, D. Petridis: J. Nanosci. Nanotech. 6, 926–947 (2006).

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