Synthesis and characterization of nanoscale iron oxid particles

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It is possible to synthesize different phases of iron oxide nanoparticles applying the polyol [1] and the reverse-micelle [2] methods.

α-Fe_2O_3 particles were synthesized by the polyol method. The modification of Fe_2O_3 can be controlled by varying the reaction conditions (temperature, time) during the solution phase reaction of iron (III) acetylacetonate with triethylene glycol. Thus, α-Fe_2O_3 is prepared at temperatures below 200 °C. The products are yielded as spherical, almost crystalline and monodisperse particles. The size can be adjusted by the concentration of the reaction components from 5-100 nm. The products have been investigated by transmission electron microscopy (TEM) and X-ray diffraction.

Powder material of ε-Fe_2O_3 was synthesized by thermal decomposition of iron hydroxide particles coated with SiO_2. Well-dispersed and uniform $Fe(OH)_3$ particles were obtained by a reserve-micelle method in a microemulsion. Hydrolysis of tetraethoxysilane added to the emulsion resulted in the formation of SiO_2 around the micelles. As a result, the SiO_2 shell homogeneously covered the precursor particle surface. This combined technique provided suitable conditions for producing nanometer-sized ε-Fe_2O_3. The iron oxide was separated from the silicate phase by leaching with sodium hydroxide solution. The analysis of the Fe_2O_3 crystals by powder diffraction yields an orthorhombic crystal system and space group compatible to $Pnm2_1$ (lattice parameters: a = 509.25 pm, b = 879.27 pm, c = 948.33 pm). Epsilon-Fe_2O_3 is isostructural with κ-Al_2O_3, $AlFeO_3$ and $GaFeO_3$ having an oxygen stacking sequence /ABAC/, and ¼ of the cations in tetrahedral coordination. Imaging of ε-Fe_2O_3 powder in the transmission electron microscope shows rod-like nanoparticles. The diffraction pattern shows that the long axis of the particles corresponds to the crystallographic *a* axis. The sizes range between 60-100 nm in length and 20-40 nm in width.

1. C. Feldmann, H.O. Jungk, Ang. Chem. **113** (2001) p 372.
2. J. Jin, S. Ohkoshi and K. Hashimoto, Adv. Mater. **16** (2004) p48.

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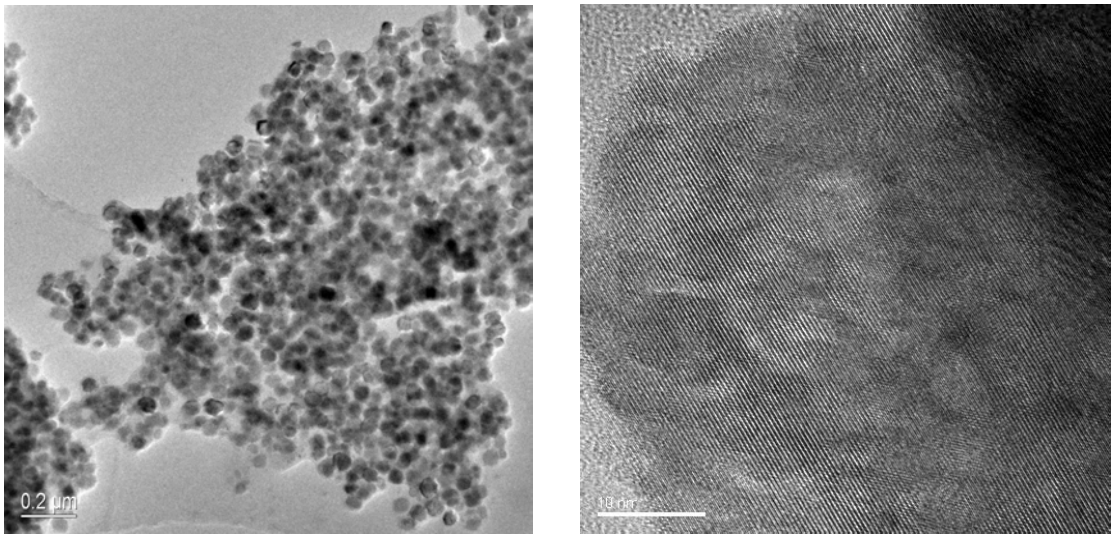

Figure 1. TEM-brightfield images of α-Fe_2O_3 nanoparticles synthesized by the polyol method. Left: Overview of the monodisperse particles, sizes 55 +/- 5nm. Right: HRTEM image of one iron oxide particle showing high crystallinity.

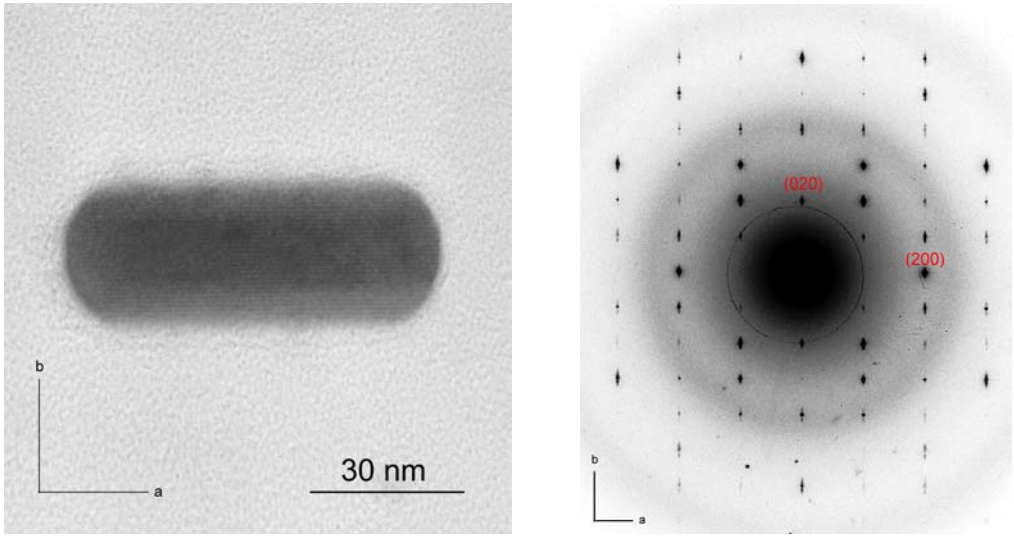

Figure 2. Left: TEM-brightfield image of one rod-like ε-Fe_2O_3 nanoparticle synthesized by the reverse-micelle method. Right: Corresponding electron diffraction pattern.

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