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Short communication

Synthesis of Fe_2O_3 nanoparticles in different reaction media

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Abstract

γ-Fe_2O_3 nanocrystallites were synthesized from iron(II) acetylacetonate using the solvothermal reaction and calcined at 500–900 °C. Their structure was characterized by X-ray diffraction (XRD) and transmission electron microscopy (TEM). The crystallite size was calculated using the Scherrer equation. No influence of reaction medium was observed on the crystal growth rate during the $\gamma \rightarrow \alpha$-$Fe_2O_3$ phase transformation.

Keywords: B. Grain size; Fe_2O_3; Reaction medium

Fe_2O_3 has long been recognized in catalysis, magnetic and biomedical applications [1–4]. It can be prepared from various techniques, e.g., hydrothermal, sol–gel, pyrolysis, microwave synthesis [5–8]. Since the solvothermal reaction was introduced by Inoue et al. [9], pure single crystal with high surface area, e.g., titania [10], zirconia [11,12], and alumina [13], was obtained. Recently, the effect of reaction medium on the formation of titania and iron oxide was reported, which results in different nucleation mechanisms and amount of defects [14,15]. Therefore, it is plausible that the reaction medium may also enhance the crystallite size of the materials through the amount of defects. This research is aimed at examining the crystallite size of Fe_2O_3 through the transformation from γ- to α-Fe_2O_3. γ-Fe_2O_3 was synthesized in different reaction media by solvothermal reaction and calcined at 500–900 °C.

Iron(III) acetylacetonate was dissolved in 1,4-butanediol (or toluene) in a glass vessel placed in an autoclave filled with the same solvent. The autoclave was then sealed and nitrogen gas was purged into the system to check leaks and remove impurities. The system was heated to the desired temperature and kept at that temperature for 2 h. The obtained product was rinsed with methanol and air-dried at room temperature. An amount of product was calcined at 500–900 °C in a Carbolite RHF1600 muffle furnace with a programmable heating rate of 10 °C/min. Phase identification and crystallite size analysis were performed by the powder X-ray diffraction (XRD) technique. A Rigaku 1200 + series X-ray diffractometer equipped with a monochromator was conducted at 2θ ranges of 10–60° using a Cu Kα radiation source accelerated at 40 kV and 30 mA. Transmission electron microscope (TEM) was applied on a JEOL TEM-200CX at 100 kV.

Pure γ-Fe_2O_3 was successfully synthesized by the decomposition of iron(III) acetylacetonate in 1,4-butanediol and in toluene, as shown in Fig. 1. The crystallite size was calculated using the Scherrer equation based on the half-width of the (311) reflection and the data are summarized in Table 1. With decreasing reaction temperature, the crystallite

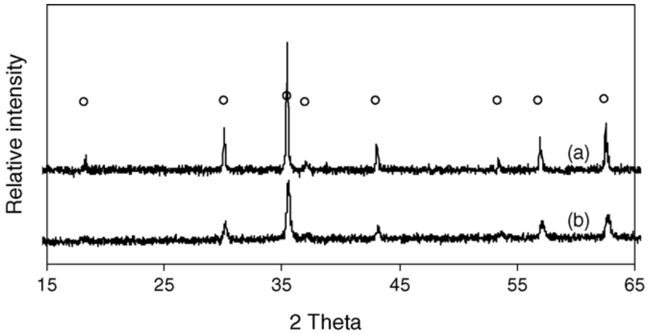

Fig. 1. XRD patterns of Fe_2O_3 products synthesized at 300 °C for 2 h; in toluene (a) and in 1,4-butanediol (b). The open circles are γ-Fe_2O_3, JCPDS no. 190629.

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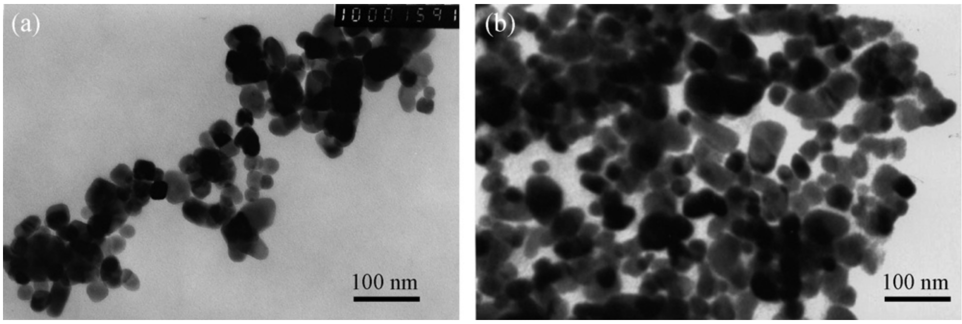

Fig. 2. TEM micorgraphs of Fe_2O_3 synthesized at 300 °C for 2 h; in 1,4-butanediol (a) and in toluene (b).

Table 1
Crystallite size of Fe_2O_3 synthesized at various reaction temperatures

Solvent	Reaction temperature (°C)	Reaction time (h)	Fe_2O_3 phase	Crystallite size (nm)
1,4-Butanediol	250	2	γ	20
	300	2	γ	32
Toluene	250	2	γ	30
	300	2	γ	73

size decreases, suggesting the influence of temperature on crystallite size of Fe_2O_3. TEM images in Fig. 2 show single crystals of γ-Fe_2O_3 with average diameters of 30 and 87 nm in 1,4-butanediol and in toluene, respectively. The results are in good agreement with the data calculated from the Scherrer equation.

γ-Fe_2O_3 with the crystallite size of 30 ± 1 nm was calcined at various temperatures and times. The results are shown in Fig. 3 as a relationship between crystallite size and calcining time. The crystallite size rapidly increases and remains constant with the increase of the calcining time. Fig. 3 evidences that time affects the crystallite size during the initial calcining period and after that the crystallite size is independent on time. In addition, at the final stage the crystallite size of Fe_2O_3 prepared in 1,4-butanediol, is the same as that prepared in toluene, indicating that the reaction medium does not influence the final crystallite size.

The increase in crystallite size at the initial period of calcination was further investigated and the data are shown in Fig. 4. The relationship between crystal growth (d/d_0) [16] and calcining time at various temperatures indicates different crystal growth rates. The higher the temperature, the faster the growth rate. At a constant time and temperature, the d/d_0 values of Fe_2O_3 prepared in different reaction media are similar.

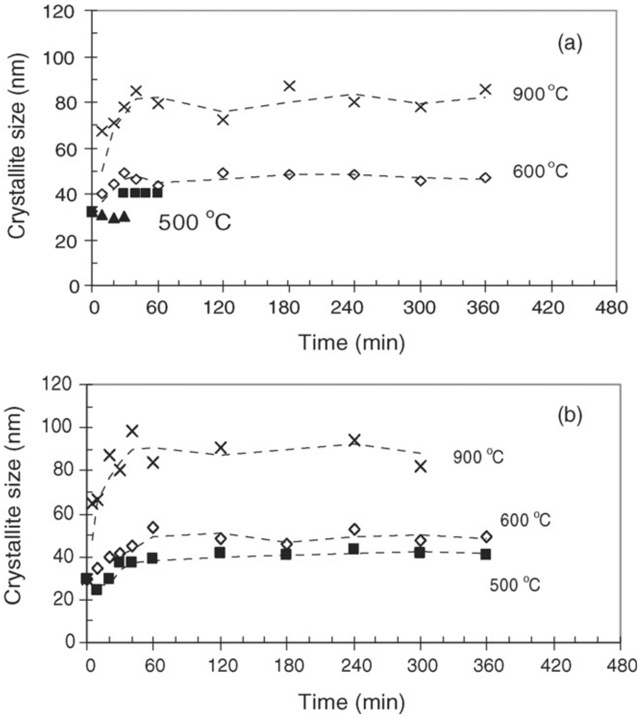

Fig. 3. Relationship between crystallite size and calcining time of Fe_2O_3 synthesized in 1,4-butanediol (a) and in toluene (b). The black triangles indicate γ-Fe_2O_3 whereas others are α-Fe_2O_3.

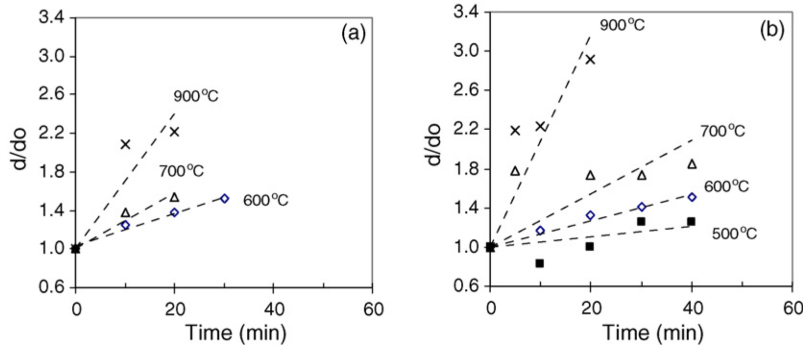

Fig. 4. Relationship between d/d_0 and calcining time of Fe_2O_3 synthesized in 1,4-butanediol (a) and in toluene (b).

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