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Synthesis of Monodisperse α-Fe_2O_3 Particles in Short Periods

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Abstract: Monodisperse α-Fe_2O_3 particles with different particle size (*ca.* 620±10 nm or 60±4 nm) have been successfully prepared by hydrothermal method in short periods. In this process, the crystal-growing inhibitor (DBS) favored the formation of monodisperse nanoparticles.

Keywords: Hematite, hydrothermal method, monodisperse particles.

As an important material used for magnetic, pigment, catalysis, gas-sensitive and medical imaging applications, monodisperse hematite (α-Fe_2O_3) particles have attracted more attentions in recent years[1,2]. Although various morphologic α-Fe_2O_3 particles have been synthesized by forced hydrolysis process[3,4], in which the properties of α-Fe_2O_3 particles vary considerably with a large number of synthesis parameters such as pH, the concentration of reactants, the coexistent anions *etc*., there is a lack of method for preparing monodisperse α-Fe_2O_3 particles in short periods. In this paper, a facile hydrothermal process is described to synthesize monodisperse α-Fe_2O_3 particles with different particle size in short periods. Given its simplicity, this process might be fit for the reproducibility and the scale-up preparation of these α-Fe_2O_3 particles.

Experiment and Results

Solating newly deposited $Fe(OH)_3$ gel with dilute $FeCl_3$ solution, the transparent electropositive $Fe(OH)_3$ sol formed and was named as precursor A, in which the total concentration of Fe(III) was 0.3 mol/L. Adding sodium dodecylbenzene sulfonate (DBS) solution into this sol, yellow hydrophobic inorganic-organic composite formed and the mixture was named as precursor B. Then the as-prepared precursors were respectively poured into a teflon-lined autoclave and hydrothermally heated at a designed temperature. After that autoclave was cooled to room temperature, product was collected and then dried at ambient temperature before being characterized.

XRD patterns of the particles derived respectively from precursor A and B demonstrate that both the products are pure α-Fe_2O_3 and highly crystallized (see **Figure 1)**. Calculating from the FWHM values using Scherrer's equation, it is found that the crystalline size of particles synthesized from precursor A (product A) are 28 nm, while

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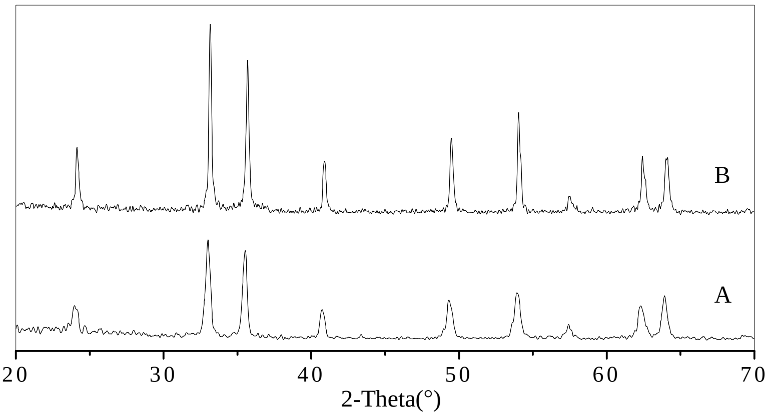
that from precursor B (product B) are 62 nm.

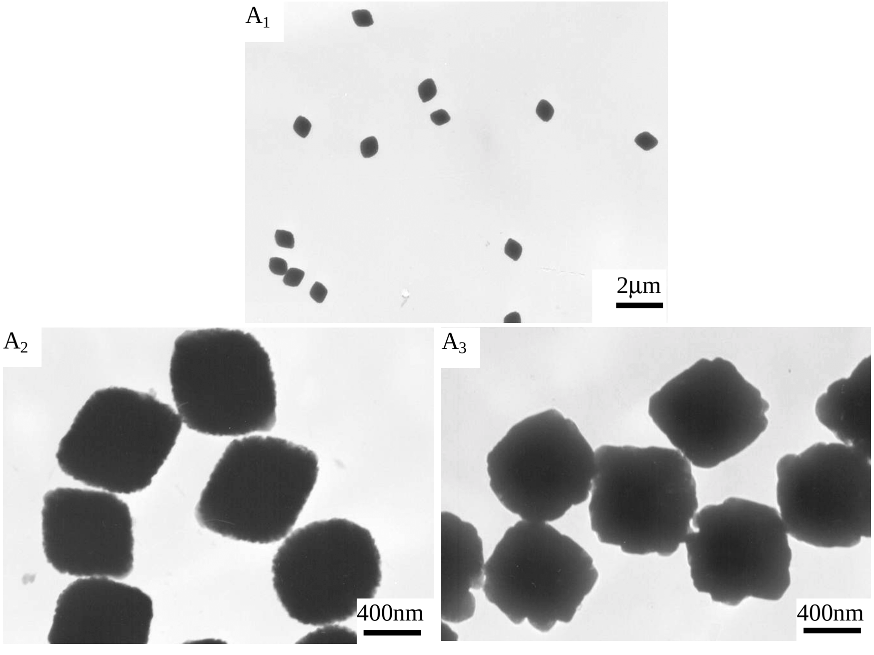
Figure 1 XRD patterns of the final particles



* A and B corresponding to sample shown in **Figure 2** A_1 and **Figure 3** B_4 respectively

The morphologies and sizes of the as-prepared particles are directly observed by TEM technique. The product A is highly dispersed while its particle size observed by TEM technique (*ca.* 620 nm) was quite different from the result obtained by XRD technique (28 nm), which demonstrated that product A formed by the aggregation of α-Fe_2O_3 nanocrystallines[5,6].

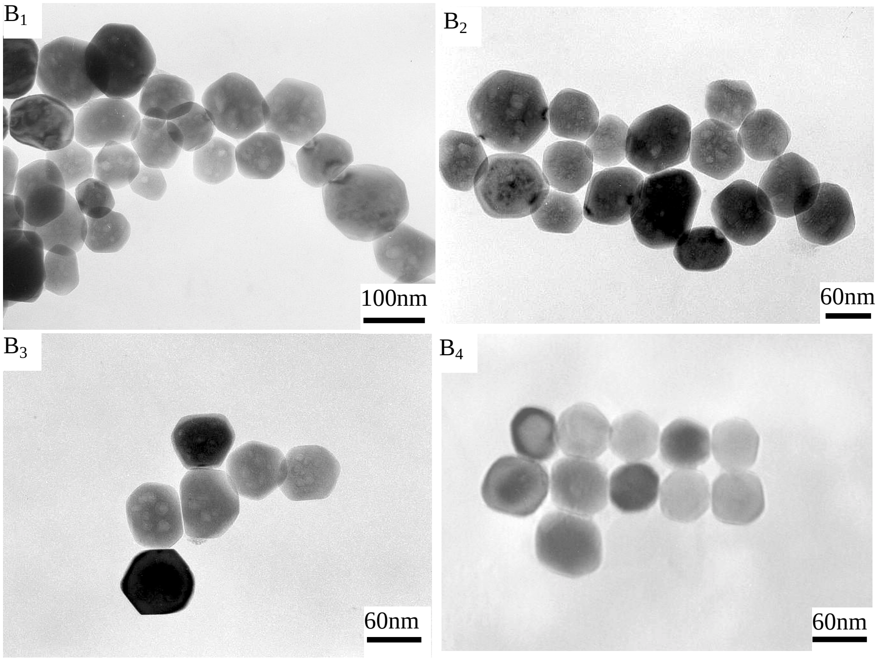
Figure 2 The TEM photographs of the product A synthesized at 200℃



*A_1, A_2 heated for 1h and A_3 6h derived from precursor A

Furthermore, the rugged surface of product A confirmed this suppose. Prolonging reaction time, the particle size of product A was invariable while surfaces of the particles become more rugged, which might be induced by the dissolution of some nanoparicles on the surface and their further growth on certain crystal plane of other nanoparticles (see **Figure 2** A_1~A_3). In contrast, the particle size of product B observed by TEM technique was in accord with that from XRD technique, which indicated that product B was single crystal. For product B, it is found that the particle size and its size distributing are greatly affected by the mol ratio of DBS/Fe(III). As shown in **Figure 3** B_1~B_3, increasing the mol ratio of DBS/Fe(III) from 0.10 to 0.25, the particle size of product B decrease from *ca.* 100±10nm to 60±4 nm, and its size distributing become more narrow. Further increasing the mol ratio of DBS/Fe(III) to 0.30 (see **Figure 3** B_4), the particle size and its distributing are hardly invariable. These results can be explained as follows: the DBS molecules might adsorb on certain crystal plane of the crystal nucleus and abstain their further growth or aggregation. So increasing the mol ratio of DBS/Fe(III), nucleation process is promoted and monodisperse nanoparticles that are quite different from product A (sub-microscale particles) formed. When the absorption of DBS molecules on crystal nucleus reaches to saturation, further increasing of DBS molecules has no effect on the final products. In this hydrothermal process, it can be concluded that DBS molecules might be a good inhibitor for the crystal growth and favored the formation of α-Fe_2O_3 nanoparticles with narrow size distribution.

Figure 3 The TEM photographs of the product B synthesized at 200℃



*B_1, B_2, B_3 and B_4 heated for 6h from precursor B with the mol ratio of DBS/Fe(III) = 0.10, 0.15, 0.25 and 0.30 respectively

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