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Synthesis and Characterization of α-Fe_2O_3 Nanocubes *via* Hydrothermal Method†

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In present work, α-Fe_2O_3 nanocubes were synthesized *via* the hydrothermal method by controlling the content of sodium dodecylbenzenesulfonate. The influence of sodium dodecylbenzenesulfonate on the morphology and structure evolution of α-Fe_2O_3 nanoparticles was carried out by X-ray diffraction, scanning electron microscopy, transmission electron microscopy, thermogravimetric analyses, UV-visible spectrophotometer and vibrating sample magnetometer. The results showed that α-Fe_2O_3 nanocubes exhibited different properties when compared with α-Fe_2O_3 nanorods obtained by calcinations method.

Keywords: α-FeOOH, α-Fe_2O_3, Nanorods, Hydrothermal method.

INTRODUCTION

α-Fe_2O_3 has drawn a great deal of interest from academic and industrial researchers due to its unique properties. It is known that α-Fe_2O_3 nanomaterials with various scales and morphologies including nanorods[1], linear[2] bending[3], dendrimers[4] and flower[5] have been successfully prepared. In this paper, α-Fe_2O_3 nanocubes were prepared *via* the hydrothermal method using sodium dodecylbenzenesulfonate (SDBS) as a template to make α-FeOOH precursor convert to α-Fe_2O_3. Meanwhile, preparation of α-Fe_2O_3 nanorods by calcining α-FeOOH nanorods precursor was also reported. Their structure, morphology, optical and magnetic properties were investigated comparatively.

EXPERIMENTAL

A certain amount of $FeCl_3 \cdot 6H_2O$ was dissolved with 30 mL distilled water in a beaker, a certain amount of CH_4N_2O, SDBS and 30 mL *n*-butyl alcohol was then added. The mixture was stirring for 15 min at room temperature, transferred into a 100 mL Teflon reactor and reacted at 150 ºC for 15 h. The precipitate was centrifuged, washed by deionized water, anhydrous ethanol in turn and then vacuum dried at 60 ºC. The molar ratio of $n(\text{SDBS})/n(FeCl_3)$ was set as N.

RESULTS AND DISCUSSION

Fig. 1 shows the XRD patterns of the obtained product reacted at N = 0.62 and its corresponding calcined product at 210 ºC. The reflection peaks at 27.0º, 33.2º, 34.7º, 35.5º, 39.5º, 46.7º, 52.5º and 56.4º shown in Fig. 1(a) are ascribed to α-FeOOH with orthorhombic structure; while the peaks at 24.1º, 33.2º, 35.6º, 40.9º, 49.6º, 54.1º, 57.8º, 62.8º, 63.9º shown in Fig. 1(b) are assigned to α-Fe_2O_3 with hexagonal structure. Fig. 2 shows the TGA trace for the as-prepared α-FeOOH. The degradation of the sample occurs in a single step and it can be found that there is almost no weight loss up 210 ºC.

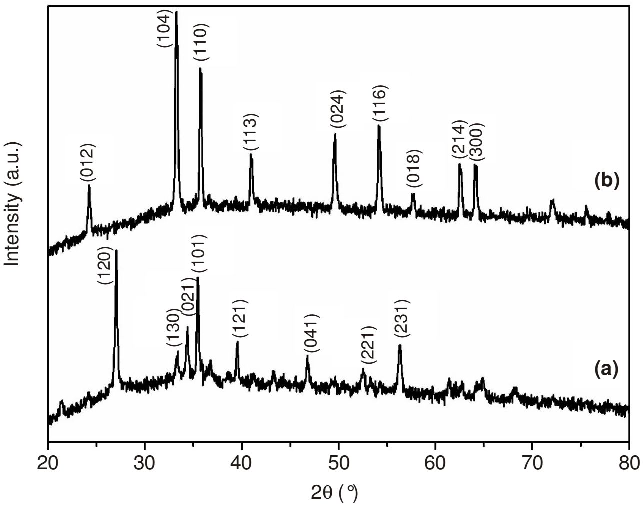

Fig. 1. XRD patterns of (a) α-FeOOH and (b) α-Fe_2O_3 obtained by calcining the corresponding α-FeOOH

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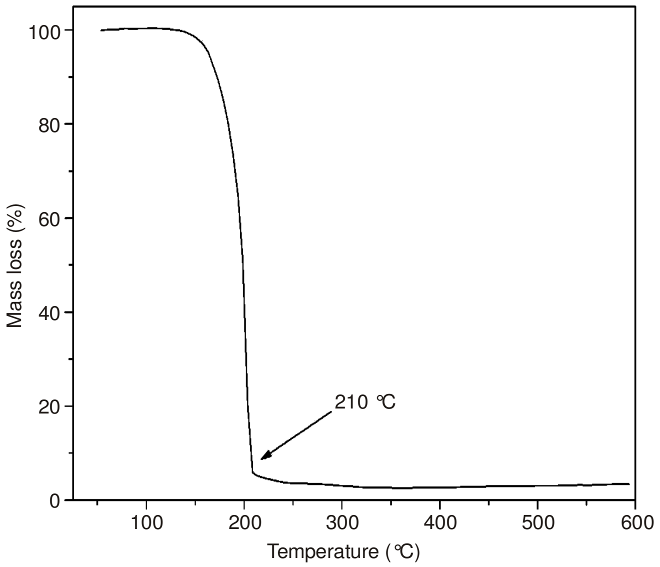

Fig. 2. TG curves of the as-prepared α-FeOOH powder

Fig. 3 gives the SEM images of the as-prepared α-FeOOH and α-Fe_2O_3 powders and both show the rendered rod-like structure.

The variations of N values from 0.46 to 1.08 on the structure and morphologies of the products are investigated. Fig. 4 shows the XRD patterns of the products at different N values. The sample shows an amorphous phase at N = 0.47 (Fig. 4a); when N increases to 0.78 (Fig. 4c), α-Fe_2O_3 diffraction peaks appears; with increasing N to 1.08 (Fig. 4e), pure phase α-Fe_2O_3 is obtained. The results indicate that the adding of SDBS plays an important role for α-FeOOH converts to α-Fe_2O_3.

Fig. 5 shows the morphology evolution. The sample firstly shows a rod-like structure with a length of 120-200 nm at N = 0.47; with increasing N to 0.78, the nanorods become shorter; further increasing N to 1.08, standard cubic nanomaterials are obtained.

Fig. 6 shows the HRTEM of the sample obtained at N = 1.08. The crystal plane spacing of 0.37 nm shown in Fig. 6c confirms the formation of α-Fe_2O_3 with hexagonal structure.

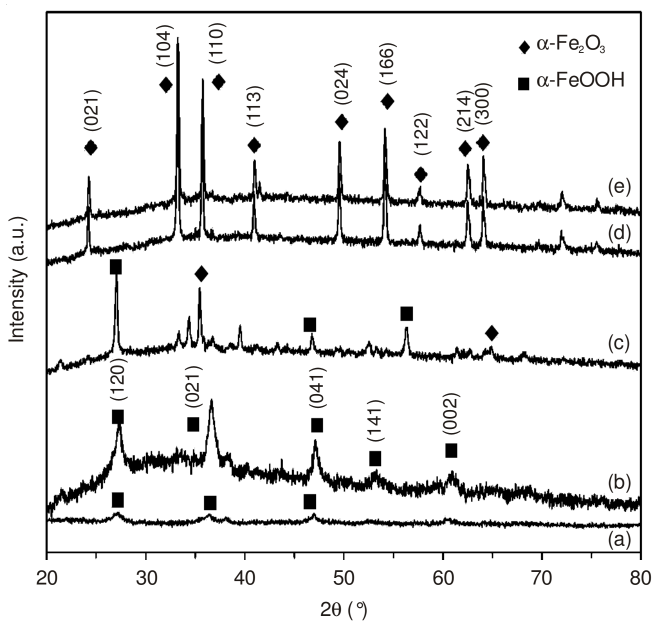

Fig. 4. XRD parterns of the products at different N values (a) 0.47, (b) 0.62, (c) 0.78, (d) 0.93 and (e) 1.08

Fig. 7 show the ultraviolet adsorption spectra of the α-Fe_2O_3 nanorods obtained by calcination of α-FeOOH (N = 0.62) and α-Fe_2O_3 nanocubes (N = 1.08). The later shows a slight red shift compared to α-Fe_2O_3 nanorods. Fig. 8 demonstrates that nanorods have higher saturation magnetization compared to nanocubes.

Conclusion

The morphology and structure of α-Fe_2O_3 nanocubes obtained *via* hydrothermal method using sodium dodecylbenzenesulfonate as a template and α-Fe_2O_3 nanorods obtained through conventional calcinations method were investigated. The content of sodium dodecylbenzenesulfonate has important role on the morphology evolution of α-Fe_2O_3 nanoparticles. Meanwhile, the two kinds of α-Fe_2O_3 nanoparticles exhibited different optical properties and magnetic properties.

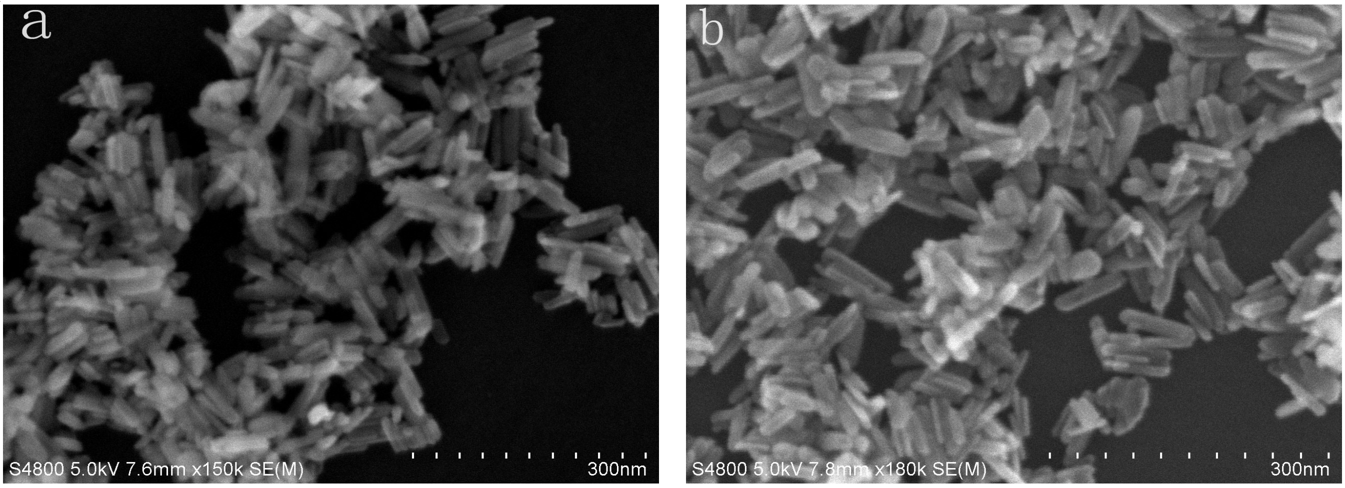

Fig. 3. SEM images of (a) α-FeOOH and (b) α-Fe_2O_3 nanorods

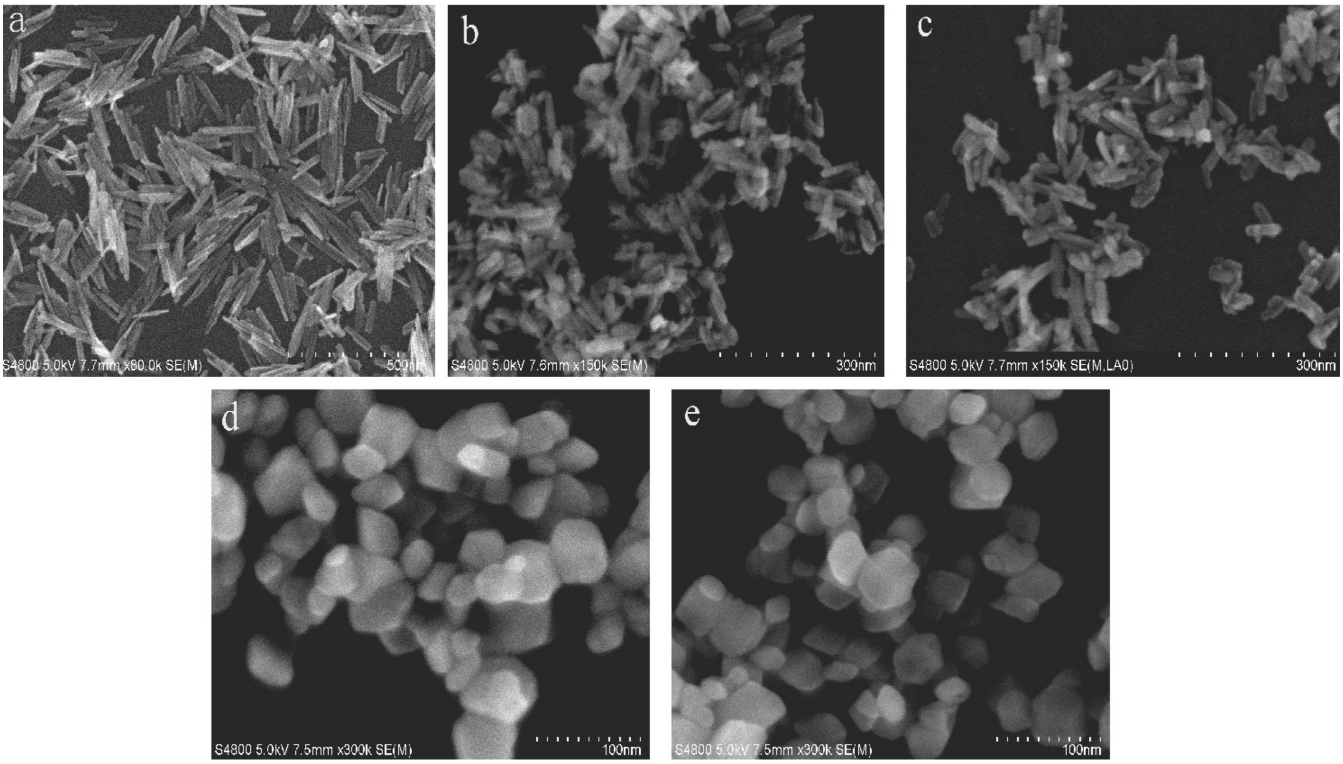

Fig. 5. SEM images of the products at different N values (a) 0.47, (b) 0.62, (c) 0.78, (d) 0.93 and (e) 1.08

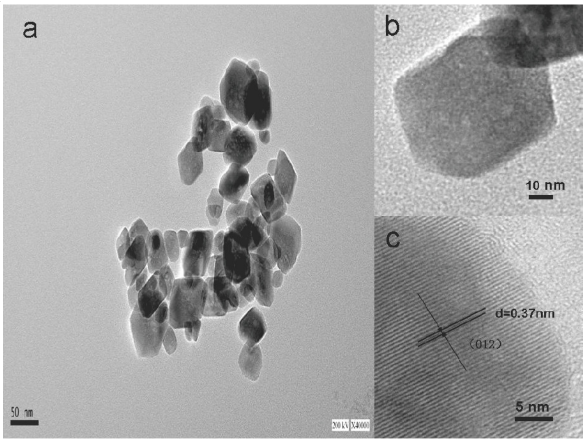

Fig. 6. TEM image of the product (N = 1.08)

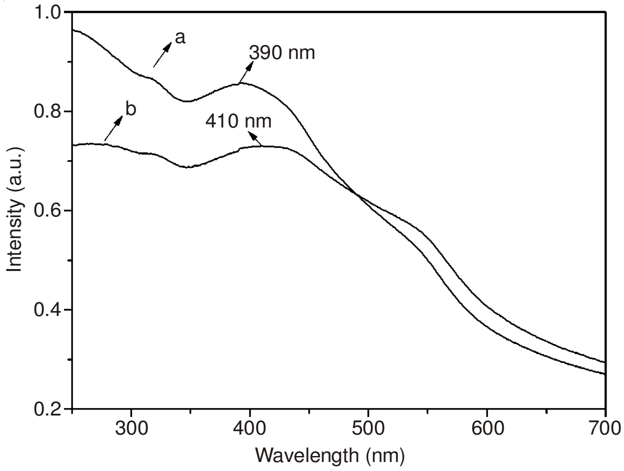

Fig. 7. UV-visible of (a) α-Fe_2O_3 nanorods and (b) nanocubes

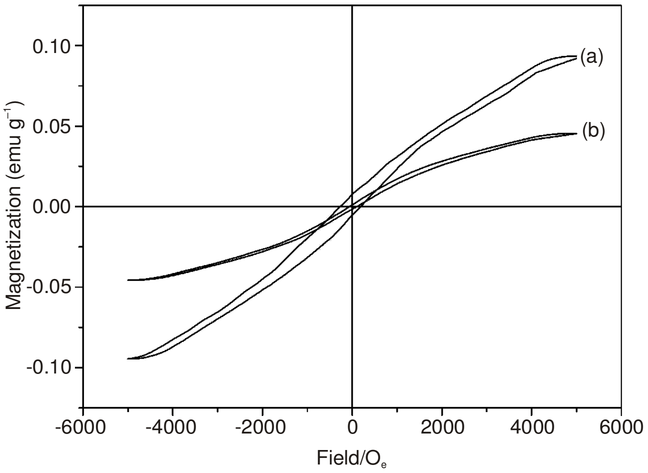

Fig. 8. Hysteresis plots of α-Fe_2O_3 nanorods and nanocubes at room temperature

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