Synthesis and Characterization of α -Fe₂O₃ Nanocubes *via* Hydrothermal Method†

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Published online: 10 March 2014;

AJC-14903

In present work, α -Fe₂O₃ 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 -Fe₂O₃ 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 -Fe₂O₃ nanocubes exhibited different properties when compared with α -Fe₂O₃ nanorods obtained by calcinations method.

Keywords: α -FeOOH, α -Fe₂O₃, Nanorods, Hydrothermal method.

INTRODUCTION

α -Fe₂O₃ has drawn a great deal of interest from academic and industrial researchers due to its unique properties. It is known that α -Fe₂O₃ nanomaterials with various scales and morphologies including nanorods¹, linear² bending³, dendrimers⁴ and flower⁵ have been successfully prepared. In this paper, α -Fe₂O₃ nanocubes were prepared *via* the hydrothermal method using sodium dodecylbenzenesulfonate (SDBS) as a template to make α -FeOOH precursor convert to α -Fe₂O₃. Meanwhile, preparation of α -Fe₂O₃ 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 FeCl₃·6H₂O was dissolved with 30 mL distilled water in a beaker, a certain amount of CH₃N₂O, 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 °C for 15 h. The precipitate was centrifuged, washed by deionized water, anhydrous ethanol in turn and then vacuum dried at 60 °C. The molar ratio of $n(\text{SDBS})/n(\text{FeCl}_3)$ 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

Fig. 1. XRD patterns of (a) α -FeOOH and (b) α -Fe₂O₃ obtained by calcining the corresponding α -FeOOH

210 °C. The reflection peaks at 27.0°, 33.2°, 34.7°, 35.5°, 39.5°, 46.7°, 52.5° and 56.4° shown in Fig. 1(a) are ascribed to α -FeOOH with orthorhombic structure; while the peaks at 24.1°, 33.2°, 35.6°, 40.9°, 49.6°, 54.1°, 57.8°, 62.8°, 63.9° shown in Fig. 1(b) are assigned to α -Fe₂O₃ 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 °C.

†Presented at The 7th International Conference on Multi-functional Materials and Applications, held on 22-24 November 2013, Anhui University of Science & Technology, Huainan, Anhui Province, P.R. China

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 -Fe₂O₃ 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 -Fe₂O₃ diffraction peaks appears; with increasing N to 1.08 (Fig. 4e), pure phase α -Fe₂O₃ is obtained. The results indicate that the adding of SDBS plays an important role for α -FeOOH converts to α -Fe₂O₃.

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 -Fe₂O₃ with hexagonal structure.

Fig. 4. XRD patterns 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 -Fe₂O₃ nanorods obtained by calcination of α -FeOOH (N = 0.62) and α -Fe₂O₃ nanocubes (N = 1.08). The later shows a slight red shift compared to α -Fe₂O₃ nanorods. Fig. 8 demonstrates that nanorods have higher saturation magnetization compared to nanocubes.

Conclusion

The morphology and structure of α -Fe₂O₃ nanocubes obtained *via* hydrothermal method using sodium dodecylbenzenesulfonate as a template and α -Fe₂O₃ nanorods obtained through conventional calcinations method were investigated. The content of sodium dodecylbenzenesulfonate has important role on the morphology evolution of α -Fe₂O₃ nanoparticles. Meanwhile, the two kinds of α -Fe₂O₃ nanoparticles exhibited different optical properties and magnetic properties.

Fig. 3. SEM images of (a) α -FeOOH and (b) α -Fe₂O₃ 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 -Fe₂O₃ nanorods and (b) nanocubes

Fig. 8. Hysteresis plots of α -Fe₂O₃ nanorods and nanocubes at room temperature

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