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Synthesis and magnetic properties of Fe_3O_4 nanoparticles

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Abstract

Ferromagnetic Fe_3O_4 nanoparticles with diameter of ~27 nm were prepared by a hydrothermal route in the presence of a surfactant, sodium bis(2-ethylhexyl)sulfosuccinate (AOT). The as-synthesized product was characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HRTEM). The hysteresis loops of the iron oxide nanoparticles were measured using a physical property measuring system (PPMS), and the results showed a superparamagnetic behavior at room temperature.
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1. Introduction

In the past decade, various nanomaterials have been extensively pursued for their catalytic, optical, electrical, optoelectronic, mechanical, thermodynamic and magnetic properties, which are quite different from those of their bulk counterparts, and thus, wide range of potential applications in nanodevices [1,2]. Among all the above-mentioned properties, the magnetism is the one dramatically dependent on the size of nanophase. Ferromagnetic materials are made up of domains that are groups of spins all pointing in the same direction, separated by domains walls. As the particle size decreases toward a critical value, the formation of domains walls becomes energetically unfavorable, and the particles consist of single magnetic domain, thus, results in the superparamagnetic phenomenon and quantum tunneling of magnetization occurred [3]. Superparamagnetic nanoparticles can offer a great potential applications in different areas such as ferrofluids, color imaging, magnetic refrigeration, detoxification of biological fluids, magnetically controlled transport of anti-cancer drugs, magnetic resonance imaging (MRI) and magnetic cell separation [4–7].

To date, many approaches including reverse micelles method [8] and thermal decomposition route [9–14] have been developed for the preparation of iron oxide nanoparticles. They usually lead to complicated process or require relatively high temperatures. Herein, we presented a simple sodium bis(2-ethylhexyl)sulfosuccinate (AOT) assisted hydrothermal method to synthesize the superparamagnetic Fe_3O_4 nanoparticles with relative high yield.

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2. Experimental

In a typical hydrothermal synthesis procedure, 0.404 g (1 mmol) $Fe(NO_3)_3 \cdot 9H_2O$ and 0.222 g (0.5 mmol) sodium bis(2-ethylhexyl)sulfosuccinate were dissolved in 10 ml de-ionized water followed by adding 2 ml of 50% hydrazine hydrate with agitating. The above mixture was then turned into a Teflon-lined stainless steel autoclave of 20 ml capacity. The sealed tank was heated at a rate of 5 °C/min to 160 °C and maintained at this temperature for 10 h in an oven, and then cooled down to room temperature naturally. The black precipitates were collected by filtration and washed with de-ionized water and ethanol for several times, and finally dried in the air.

The X-ray diffraction (XRD) pattern of the product was recorded by step scan in the RIGAKU-DMAX2500 X-ray diffractometer with Cu Kα radiation (λ = 0.1541 nm) at 40 kV and 100 mA. The scan range (2θ) was from 5° to 85° with the step of 0.05° and the resolution of 0.01°. The morphologies and microstructures of the as-synthesized sample were characterized by JEOL-2010 transmission electron microscope equipped with energy dispersive spectroscope (EDS) and operated at 200 kV. All the above procedures were conducted at room temperature. The hysteresis loops of the as-synthesized nanoparticles were measured using a physical property measuring system (PPMS) at 300 and 10 K, respectively.

3. Results and discussion

The XRD pattern of the as-synthesized sample shown in Fig. 1 can be indexed to the cubic Fe_3O_4 phase (ICSD 20-596) and FeO(OH) phase (ICSD 71-810). The ratio of the integral intensity from all the diffraction peaks of FeO(OH) to that of Fe_3O_4 is about 0.05, revealing that Fe_3O_4 is the major product and the proportion of FeO(OH) is less than 5% in the whole sample [16]. The calculated lattice constant of Fe_3O_4 extracted from the XRD data is $a = 0.840$ nm, in good agreement with the literature data. IR spectrum shown in Fig. 2 demonstrated the hybrid chemical nature of the nanocrystals. The absorption bands at 580 cm^{-1} were related to the vibrations of Fe–O functional group, and the other adsorption peaks at ~1621, 1398, and 1042, 886, 790 cm^{-1} were attributed to C=O and $(CH_2)_n$ vibrations, respectively, which indicated that AOT molecules were chemically bonded to the surface of Fe_3O_4 nanoparticles.

The mean particle size calculated from Fe_3O_4 diffraction peaks according to the Debye–Scherrer equation was 27 nm, in good agreement with the result observed by transmission electron microscopy (TEM) shown in Fig. 3A. TEM micrograph of the figure also revealed the quasi-spherical morphology of the synthesized Fe_3O_4 nanoparticles. The selected area electron diffraction (SAED) pattern (inset in Fig. 3A) taken from the area consisting of many particles presented Fe_3O_4 polycrystalline diffraction rings, in accordance with the XRD result. The high-resolution transmission electron microscopy (HRTEM) image presented in Fig. 3B provided an indication of the morphology of the sample in detail and exhibited that Fe_3O_4 nanoparticles were single-crystalline.

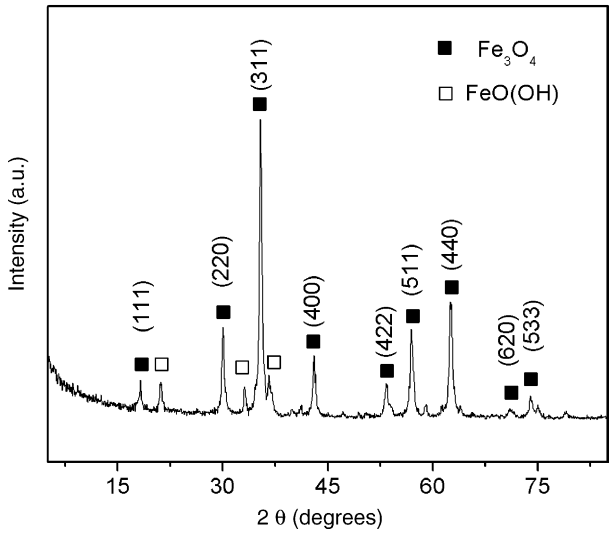

Fig. 1. XRD pattern of the sample prepared at 160 °C for 10 h.

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