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Facile co-precipitation synthesis of shape-controlled magnetite nanoparticles

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Abstract

Monodispersed magnetite (Fe_3O_4) nanoparticles with high saturation magnetization, including nanospheres, nanoneedles and nanocubes, were synthesized by the co-precipitation method. The shape of magnetite nanoparticles was controlled by changing the amount of sodium dodecyl sulfate (SDS) and the particle size was adjusted by the irradiation time of visible light. The Fe_3O_4 nanospheres, nanoneedles and nanocubes were obtained with the addition of 0 g, 0.1–0.4 g and 0.5–1.0 g SDS, respectively. The particle size of nanospheres, nanoneedles and nanocubes of magnetite was ~15 nm, 100×12 nm^2 (length × width) and ~50 nm under the irradiation time of 30 min. The phase structure, particle shape and size of the samples were characterized by transmission electron microscopy (TEM), X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR). The as-prepared magnetite nanoparticles from TEM images exhibited a high level of crystallinity with narrow size distribution and good dispersion. The XRD results showed that all the magnetite nanoparticles were pure Fe_3O_4 phase with obvious diffraction peaks. The products exhibited the attractive magnetic properties with high saturation magnetization, which were examined by a vibrating sample magnetometer (VSM).

Keywords: Magnetite; Nanoparticles; Shape-controlled; Saturation magnetization; Visible light irradiation

1. Introduction

Magnetite (Fe_3O_4) exhibits the unique electric and magnetic properties based on the transfer of electrons between Fe^{2+} and Fe^{3+} in the cubic sites [1]. Due to the unique properties and advantages of magnetite, such as strong magnetism, good biocompatibility, long durability, low toxicity and low cost [2,3], it is widely used in magnetic biomedicine [4–6], heavy metal ions removal [7,8], electromagnetic wave absorption [9] and other fields [10–12]. These properties of magnetite strongly depend on their dimension, shape, saturation magnetization as well as monodispersion [13,14].

Many research groups have worked to synthesize magnetite nanospheres, nanocubes, nanoneedles and nanoporous particles by hydrothermal methods [15], co-precipitation methods [16] solvothermal methods [17,18], sonochemical methods [13] and self-assembly methods [19]. These studies provided many useful preparation technologies for the preparation of Fe_3O_4 with different shapes. However, most of Fe_3O_4 nanoparticles obtained by the co-precipitation method are spherical shaped. For instance, the magnetite nanospheres have been synthesized using the co-precipitation method, but the hexanoic acid and loeic acid were employed as the coating agents during the initial crystallization phase of the magnetite [20]. These organic acids are expensive reagents and not environment friendly, and the products obtained have wide particle size distribution of 10–40 nm and small saturation magnetization of 58.72 emu/g. The various methods have been applied to synthesize the nanocubes, nanoplates, nanoneedles or nanorods of magnetite using a solvothermal method [17], hydrothermal method [21], and template method [22] rather than the co-precipitation method. In recent years, Gao et al. have synthesized Fe_3O_4 nanocubes using the solvothermal method at 260 °C in the presence of oleic acid and oleylmine [17].

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A series of Fe_3O_4 morphologies (nanorods, nanoctahedrons and nanocubes) were synthesized via a hydrothermal process [21]. Zheng et al. have prepared different morphologies of Fe_3O_4 nanostructures, including spherical, cubic, rod-like, and dendritic nanostructure, using polyethylene glycol as a template [22].

In this study, a simple and efficient one-step co-precipitation method is reported to synthesize the shape-controlled magnetite nanoparticles using $FeSO_4$ and $Fe_2(SO_4)_3$ as reactants at room temperature. In order to improve the shape-dependent functional properties, such as electric property and magnetism, the relation between technological parameters and the shape of products were investigated in this paper. Different nanostructures and particle sizes of Fe_3O_4 nanoshperes, nanocubes and nanoneedles were successfully synthesized by carefully controlling the amount of SDS and the time of irradiation with visible light. The crystalline structure, shape and size of the as-obtained Fe_3O_4 nanoparticles were characterized by XRD and TEM technique and the magnetic properties were investigated by VSM at room temperature. The results show that the shape-controlled Fe_3O_4 nanoparticles exhibit strong monodispersity and high saturation magnetization.

2. Experimental

2.1. Characterization

A BDX-3300 model JEOL 100CX-II transmission electron microscope (TEM) was used to carry out the TEM measurements to investigate the morphology and size of magnetite nanoparticles at an accelerating voltage of 200 kV. The obtained samples were characterized on a BDX-3300 diffractometer using CuKα radiation (wavelength, $\lambda = 1.5406$ Å) with variable slits at 45 kV/40 mA to obtain X-ray powder diffraction (XRD) patterns. The structural properties of the samples were determined by using a NEXUS 870 Fourier transform infrared spectroscopy (FTIR) from 450 cm^{-1} to 2000 cm^{-1}. The magnetic measurement of the products was carried out in a USA LDJ 9600-1 vibrating sample magnetometer (VSM). Magnetic hysteresis loops were recorded at room temperature in a field of 10,000 Oe to determine the saturation magnetization (M_s) for the samples.

2.2. Preparation of magnetite nanoparticles

In the typical synthesis, 2.8 g of $FeSO_4 \cdot 7H_2O$ and 4.0 g of $Fe_2(SO_4)_3$ were dissolved in 100 mL distilled water for 5 min using a magnetic stirrer in a 250 mL beaker. Different amounts (0–1.0 g) of sodium dodecyl sulfate (SDS) were added to this solution under stirring. The pH value of the mixture was adjusted to 12 by solid NaOH with stirring for 20 min at room temperature. The visible light was generated by a lamp. The wavelength of the visible light was about 400–750 nm. The reactor was placed at a fixed distance of 10 cm from the lamp for 0–60 min. The black precipitates were formed and then were washed several times with distilled water and ethanol in turn until the pH was neutral. Finally, the resultant black precipitates were dried in air at room temperature to obtain magnetite nanoparticles. The magnetite nanoparticles obtained at different SDS concentrations and visible light time were nanoshperes, nanocubes and nanoneedles.

3. Results and discussion

3.1. TEM results

Different shaped magnetite nanoparticles were prepared by the facile co-precipitation method in the presence of SDS. The experimentations have been performed by adjusting the amount of SDS and the irradiation time of the visible light to investigate their influence on the shape and particle size of magnetite nanoparticles. The nanospheres, nanoneedles and nanocubes of magnetite were synthesized, and the TEM images of the as-prepared Fe_3O_4 are given in Fig. 1. It could be observed that all of the three synthesized Fe_3O_4 nanoparticles displayed a relatively narrow particle size distribution, good dispersion and a perfect and uniform morphology with distinct crystalline structure. Without SDS, the prepared Fe_3O_4 nanoparticles were nanospheres with monodispersion under the visible light irradiation time of 0–60 min. The particle size of magnetite nanospheres could be controlled by increasing the visible light irradiation time. As the irradiation time of the visible light increased from 30 min to 60 min, the size of the as-prepared magnetite nanospheres increased from ~15 nm to ~50 nm as shown in Fig. 1a and b. The Fe_3O_4 nanoneedles could be produced when 0.1–0.4 g SDS was added. As seen from Fig. 1c, the monodispersible Fe_3O_4 nanoneedles were obtained by adding 0.4 g SDS under the visible light irradiation time of 30 min. Under the visible light irradiation time of 60 min, the Fe_3O_4 nanoneedles were prepared with the addition of 0.3 g SDS as shown in Fig. 1d. Furthermore, the diameter of magnetite nanoneedles increased from 100×12 to 115×14 nm (length × width) with the visible light irradiation time increasing from 30 min to 60 min. With the addition of 0.5–1.0 g SDS, the shape of magnetite nanostructures changed from nanoneedles to nanocubes. As shown in Fig. 1e and f, the Fe_3O_4 nanocubes were prepared by adding 0.8 g and 1.0 g SDS, respectively. Similarly, the particle size of the as-obtained Fe_3O_4 nanocubes increased from about 50 nm to about 100 nm with the increase of the irradiation time from 30 min to 60 min. It can be inferred from Fig. 1 that the particle shape of magnetite is controlled by adjusting the SDS amount, and the particle size increases with the increase of the visible light irradiation time. In fact, the visible light irradiation will not influence the particle shape but the particle size and crystalline structure. The reason may be that the visible light irradiation is favorable to the growth of grain. Therefore, the longer the irradiation time of visible light, the larger the particle size, the better the crystallinity of Fe_3O_4 nanoparticles. The particle size is small but with poor crystalline structure without visible light irradiation.

3.2. XRD results

The phase and crystallinity of magnetite nanospheres, nanoneedles and nanocubes prepared at visible light irradiation time of 30 min were investigated using the XRD technique and the results are given in Fig. 2. The XRD patterns indicated that all of the