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THE SYNTHESIS AND CHARACTERIZATION OF SUCROSE MODIFIED MAGNETITE

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ARTICLE DETAILS

ABSTRACT

Article History:

Received 26 June 2018 Accepted 2 July 2018 Available online 1 August 2018 Monodisperse air-stable sucrose modified magnetite (SFe $_3$ O $_4$) has been prepared by precipitation via a simple and facile hydrothermal reduction route, which was characterized by scanning electron microscopy transmission (SEM), electron microscopy (TEM), energy dispersive X-ray (EDX) spectroscopy, X-ray diffraction (XRD), FT-IR spectroscopy and vibrating sample magnetometry (VSM). SEM and TEM analysis showed the formation of 60 - 80 nm particles. Hysteresis loops indicated that as prepared product could be separated from the nonmagnetic materials in a magnetic field. FT-IR spectra indicated that the carboxylate in this work might bind to the magnetite surface through two oxygen atom, and the sucrose modified magnetite (SFe $_3$ O $_4$) was rather stable.

KEYWORDS

Magnetite, sucrose modified, hydrothermal reduction, characterization.

1. INTRODUCTION

In the past decade, broad attention and research have been devoted to the design of the magnetic nanoparticles, which were consists of magnetic cores and polymeric shells. The high interests in magnetic nanoparticles are mainly related to they can be manipulated or recovered rapidly by an external magnetic field. Also due to their high surface area and excellent chemical selectivity magnetic nanoparticles are considered as potential adsorbents, which the particle shape, core size and surface functional groups can also be controlled [1,2]. By the choice and chemical modification of polymeric shells, the surface functionality of magnetic nanoparticles can be tailored for various applications in several fields (such as drug delivery, therapy, diagnosis, separation, enzyme immobilization and, etc) [3]. However, magnetic nanoparticles with $hydrophobic \, surface \, properties \, and \, aggregation \, limit \, their \, dispersion \, into \,$ aqueous solutions and matrices, which also affect the chemical and physical reactivity toward interaction with other contacted species [4]. In order to overcome such limitations along with the maintenance of the magnetic properties of such nanoparticles, various selected approached are generally used to coat the particles with biocompatible, water-soluble and nontoxic materials [5-9].

In this paper, monodisperse air-stable sucrose modified Fe $_3O_4$ (SFe $_3O_4$) was synthesized using sucrose, FeCl $_3$ and NaBH $_4$. The structure, component, morphology and magnetic property of the composites were characterized by scanning electron microscopy (SEM), transmission electron microscope (TEM), fourier-transform infrared spectrometer (FT-IR), X-ray Diffraction (XRD) and the energy dispersive X-ray (EDXA) spectrums and vibrating sample magnetometry (VSM).

2. EXPERIMENTAL SECTION

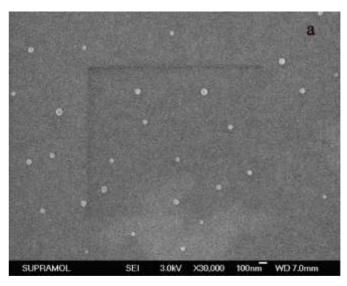
2.1 Materials

The chemical reagents used in this work are ferric chloride (FeCl $_3$), sucrose, methyl orange, and sodium borohydride (NaHB $_4$). All the chemical reagents are of analytical grade.

${\bf 2.2}\quad Synthesis\ of\ zero\ valent\ iron\ in\ Sucrose$

In a facile experimental procedure, 6 g of sucrose and 5 g of FeCl $_3$ were dissolved in 15 mL of deionized water and nitrogen (N $_2$) was introduced into the solution for 20 min, then 10 mL 0.01 mol/L NaHB $_4$ was doped with vigorous stirring. The molar ratio of NaHB $_4$ to FeCl $_3$ is 1:3. Vigorous stirring was carried on for about 30 min, and the obtained mixture was then transferred into a Teflon lined stainless steel autoclave (25 mL capacity) for crystallization at 180 °C for 48 h. After the reaction system was naturally cooled to room temperature, the product (SFe $_3$ O $_4$) was isolated from the liquid phase by magnetic and then thoroughly washed three times with deionized water to remove water-soluble organic byproducts. In order to remove un-encapsulated Fe $_3$ O $_4$, the sample was dipped in 0.1M HCl solution for 12 h, then the product was isolated from the liquid phase by magnetic and dried at 60 °C overnight and black powder was obtained.

3. RESULTS AND DISCUSSION



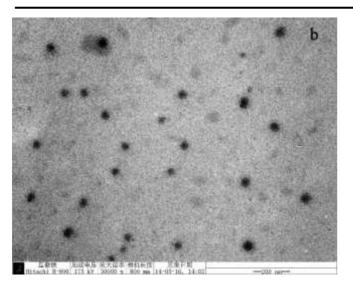


Figure 1: SEM (a) and TEM (b) image of the SFe₃O₄

The morphology of SFe $_3$ O $_4$ was characterized by SEM. As shown in Figure 1(a), the morphology of SFe $_3$ O $_4$ was the fancy totally round spheres and shown the nearly uniform distribution of particle size (about 60 - 80 nm). Figure 2(b) showed the TEM pictures of SFe $_3$ O $_4$, in which Fe $_3$ O $_4$ can be well encapsulated in the sucrose matrix, and most of the Fe $_3$ O $_4$ were located in the core of the spheres.

Furthermore, the surface composition was analyzed using an EDXA attached to the SEM. The EDXA analysis (Figure 2) shows that the surface is primarily comprised of carbon (C) and oxygen (O). Figure 2 illustrates

the EDXA spectrum of the SFe_3O_4 and the insert table shows the percentage of elements present. C was the most abundant element present at the surface, followed by O, and Fe was barely existing, which indicate that Fe_3O_4 was completely capped by sucrose.

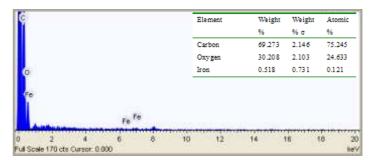


Figure 2: The energy dispersive X-ray (EDXA) spectrums for ${\sf SFe_3O_4}$ particles

To identify the iron oxide phases of all the samples, the XRD pattern was adopted as shown in Figure 3. All the peak positions at 18.2 (111), 30.1 (200), 35.4 (311), 43.0 (400), 53.7 (422), 57.2 (511), and 62.6 (440) are consistent with the standard X-ray data for the magnetite phase (JCPDS no. 19-0629). The particle size calculated by Scherrer's equation is 75 nm, which is quite close to the SEM and TEM result. It means that all the assynthesized particles are single crystals. Moreover, the relative XRD peak intensities increase with increasing sucrose concentration, as the molar ratio of sucrose to FeCl₃ was 2:1 (sample a) and 1:3 (sample b), which indicates that the relative crystallinity of the magnetite increased. Besides magnetite peaks, we did not observe any other iron oxide peaks in the diffraction pattern for any of the samples.

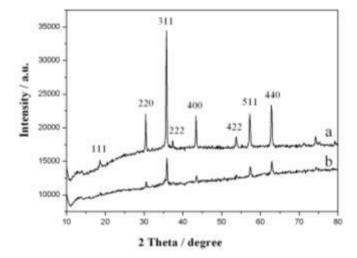


Figure 3: X-ray diffraction (XRD) of SFe₃O₄ sample a and b. The molar ratio of sucrose to FeCl₃ was 2:1 (sample a) and 1:3 (sample b)

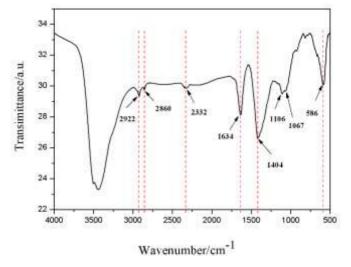


Figure 4: The FT-IR spectroscopy of SFe₃O₄ sample

The FT-IR spectroscopy was introduced to analysis the presence of the coating agents on the surface of the Fe₃O₄. FT-IR spectroscopy of the asprepared SFe₃O₄ was shown in Figure 4. The broad characteristic band from 3600 to 3100 cm⁻¹ could be assigned to O-H stretching vibration arising from hydroxyl groups on nanoparticles and adsorbed glucose, gluconic acid, and water [10]. The peaks around 2922 cm-1 and 2860 cm-1, assignable to asymmetric and symmetric vibrations of C-H in -CH₂-, can be obviously found [11]. Characteristic bands at 2922 cm-1 (asymmetric stretching) and 2850 cm⁻¹ (symmetrical deformation vibration) of C-H in -CH₃ might arise from the side reaction products of sucrose during the hydrothermal reaction. The band at 1261 cm⁻¹ of -COOH stretching, at 1102 cm⁻¹ of C-O-C stretching and at 679 cm⁻¹ of -C-H all came from the residue sucrose. In addition, the absorption band at 2332 $\mbox{cm}^{\mbox{\tiny -1}}$ on the spectrum referred to the vibration of the adsorbed CO2 in the sample. The absorption band at 1634 cm⁻¹ on the spectrum referred to the vibration of -FeOO stretching vibration, which indicated the existence of active hydroxyl group, which indicated that the carboxylate in this work might bind to the magnetite surface through two oxygen atom. The band at 586 cm-1 is related to the vibrations of the Fe-O functional group, which corroborates that the phase of as-prepared particles is magnetite [12].





Figure 5: Photography image of SFe₃O₄ solution with (a) and without an external magnetic field (b)

Magnetic property of SFe_3O_4 was also characterized. As shown in Figure 5, with the external magnetic field, the SFe_3O_4 will soon be sucked, and the solution is clear; and after removed this external magnetic field, SFe_3O_4 fall rapidly and return to a homogeneous SFe_3O_4 solution, indicated that the materials have the properties of magnetism. And magnetization curves of the samples measured at room temperature was shown in Figure 6. The saturation magnetization (Ms) of the samples 80.47 emu/g, retentivity (Mr) is 13.88 emu/g and coercivity (Hc) is 83.52 G. Attributed to the existence of organic coating agent, the saturation magnetization of the samples is smaller than the bulk magnetite (92 emu/g), which is often observed with the nanoparticles. Some references revealed that due to quenching of surface moments of the coating agents the uniformity of the samples was decreased, resulting in the reduction of magnetic moment in the samples [13].

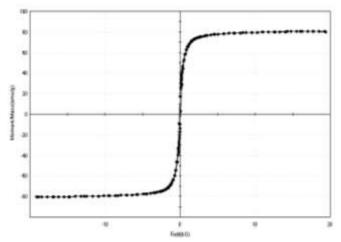


Figure 6: Magnetization curves at room temperature of magnetite samples

4. CONCLUSIONS

In this paper, monodisperse air-stable Fe_3O_4 nanocrystal encapsulated in sucrose has been successfully synthesized via a simple and facile hydrothermal reduction route. Sucrose both acted as reducing agent and

coating agent, which prevent particle growth and agglomeration by chelating with metal ions. Results indicated SFe_3O_4 could be separated with the help of external magnetic field. Therefore, with the help of an applied magnetic field as-prepared product can be used as a powerful adsorbent for different applications.

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