

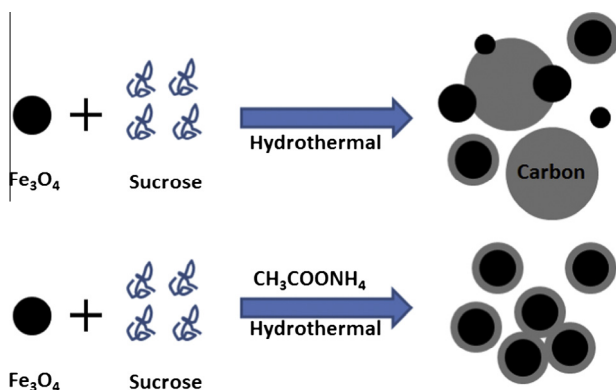
Short Communication

A general and facile method for improving carbon coat on magnetic nanoparticles with a thickness control

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GRAPHICAL ABSTRACT

A facile hydrothermal method was developed to improve carbon coating formation on the surface of magnetic nanoparticles from sucrose by using $\text{CH}_3\text{COONH}_4$ as a structure guiding agent.



ARTICLE INFO

Article history:

Received 8 July 2015

Revised 9 September 2015

Accepted 10 September 2015

Available online 11 September 2015

Keywords:

Carbon coating

Magnetic nanoparticles

Sucrose

Hydrothermal

ABSTRACT

A general and facile hydrothermal method was developed to improve carbon coating formation on the surface of magnetic nanoparticles from sucrose and hindering growth of pure carbon spheres by using ammonium acetate ($\text{CH}_3\text{COONH}_4$) as a structure guiding agent. The thickness of the carbon coating could be easily realized by adjusting the reaction time.

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1. Introduction

In recent years, magnetic nanoparticles (MNPs) with narrow size range have attracted extreme focus owing to their applications in drug delivery, biodiagnostics, catalysis, environmental remediation, and other fields [1–7]. The success of these promising

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functions highly depends on their stability against erosion and oxidation. Thus, various methods, such as surfactant/polymer-coating, silica-coating and carbon-coating, have been described to protect and stabilize the MNPs for the last decades [8–11]. Among these methods, carbon-coatings have been widely employed due to their excellent thermostability, biocompatibility and chemical stability against ambient environments [12,13]. Moreover, a plenty of functional groups on the surface of carbon shell could be easily modified and functionalized, which is beneficial to make them applicable under various conditions and improve their specific performance in applications, such as catalysis and lithium battery [14–21]. Hence there has been considerable interest in the development of new strategies to fabricate carbon coating on MNPs.

Up to now, hydrothermal method and Stöber method are two promising processes to fabricate carbon-coated MNPs with uniform and tunable size [14,22,18,19,23]. Compared to Stöber process, hydrothermal process is more facile, lower energy consumption, and more environment-friendly. However, the undesirable formation of giant pure carbon nano/microspheres, which has a crucial influence on the morphology and structure of the target products, was the most difficult and pressing obstacle to be overcome in the hydrothermal system. To restrain the formation of pure carbon spheres under hydrothermal conditions, oleic acid, urea, and polyethylene glycol have been attempted as connecting agents or surfactants to prepare carbon coated Fe_3O_4 [24,22,16]. However, the multiple steps or rigorous reaction conditions are necessary for these methods to form uniform carbon coated Fe_3O_4 nano/microspheres. Therefore, developing a facile method to fabricate uniform carbon-coated MNPs with tunable size and homogenous structure is still an urgent challenge.

Herein, we present a straightforward and general strategy to fabricate carbon coating on MNPs with a controlled thickness of carbon shells under hydrothermal conditions. The narrow size distribution of $\text{Fe}_3\text{O}_4/\text{C}$ microspheres with a controlled carbon thickness was realized by hydrothermal treatment of Fe_3O_4 microspheres, sucrose, and ammonium acetate ($\text{CH}_3\text{COONH}_4$) mixture solutions. The addition of $\text{CH}_3\text{COONH}_4$ could promote carbon layers exclusively grow on the surface of Fe_3O_4 microspheres and hinder the formation of isolated pure carbon spheres. The thickness of carbon shells ranging from 5 to ~25 nm could be easily obtained by adjusting reaction time.

2. Methods

2.1. Synthesis of Fe_3O_4 microspheres

The Fe_3O_4 microspheres were prepared via a solvothermal method. Typically, 1.35 g of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, 3.6 g of CH_3COONa , and 1 g of polyethylene glycol were added to 40 mL ethylene glycol and stirred to form a homogenous mixture liquid. Then the mixture was transferred to 75 mL Teflon-sealed autoclave and heated at 200 °C for 20 h. The diameters of obtained Fe_3O_4 microspheres range from 400 nm to 700 nm.

2.2. Hydrothermal synthesis of carbon coated Fe_3O_4 microspheres

In a typical procedure, stoichiometric amounts of Fe_3O_4 microspheres, sucrose and $\text{CH}_3\text{COONH}_4$ were added to 60 mL distilled water. Then the mixture solution was transferred to 75-mL Teflon-sealed autoclave and heated at 190 °C for 4–48 h. The products were collected from the solution with an external magnet and washed with water and ethanol for several times. Finally, the black products were dried in a vacuum oven at 60 °C for 24 h.

2.3. Characterizations

Scanning electron microscopy (SEM) images were obtained with a Hitachi S-4800 emission scanning electron microscope. High-resolution electron microscopy (HRTEM) was performed on sample suspensions dried on a carbon coated grid (200 mesh, 3.05 mm in diameter) with a JEOL JEM 2010 FEF electron microscope operated at 200 kV. Raman spectra were recorded at room temperature on a Jobin-Yvon Lab Raman spectrometer equipped with a CCD camera using a HeNe laser at an excitation wavelength of 632.8 nm and a power of 20 mW. An Olympus optical microscope with a 100× objective lens was used to focus the laser beam on the sample and collect the scattered radiation. Hysteresis loops were collected with a Quantum Design superconducting quantum interference device (SQUID) magnetometer (Lake Shore 7307) at 300 K. The SQUID measurements for all the samples were done on the pure and dried powders. All FT-IR spectra were obtained with a Thermo Scientific Nicolet iS10 spectrophotometer using pellets with KBr powder. Samples were scanned 15 times between 4000 and 400 cm^{-1} at a resolution of 4 cm^{-1} . The zeta potential of the particles was performed with a ZetaPALS at 25 °C.

3. Results and discussion

Fig. 1 shows the SEM and TEM images of the Fe_3O_4 microspheres before and after carbon coating. From the SEM images, it can be seen that the Fe_3O_4 microspheres have diameters of 400–800 nm (Fig. 1a and b), and there is no obvious change in morphologies of the microspheres after carbon coating (Fig. 1c and d). Furthermore, no giant isolated pure carbon microspheres were found in the hydrothermal products (Figs. 1c and S2d), indicating that the newly formed carbon all coated on the Fe_3O_4 microspheres. The high magnification SEM image shows that the surface of the carbon coated Fe_3O_4 microspheres is smoother than that of initial Fe_3O_4 samples (Fig. 1b and d), which further confirm that the successful carbon coating formation on the surface of the Fe_3O_4 microspheres. The TEM image shows that the thickness of the carbon shell is about 10 nm (Fig. 1e). Moreover, the carbon coating with a thickness from 5 to 25 nm could be easily realized by adjusting the reaction time from 4 to 48 h (Fig. 2).

In the present simple hydrothermal system, the $\text{CH}_3\text{COONH}_4$ additive was found to play a critical role in developing the formation of carbon coating and decreasing the formation of giant pure carbon microspheres, and many giant pure carbon microspheres were formed in the absence of $\text{CH}_3\text{COONH}_4$ (Fig. S1). To further reveal the function of the $\text{CH}_3\text{COONH}_4$, the supernatants and solids products of the hydrothermal treatment with (Experimental group) and without (Control group) $\text{CH}_3\text{COONH}_4$ were compared. After hydrothermal treatments, the supernatant of the experimental group was darker than that of the control group (Fig. S2a), indicating that the presence of $\text{CH}_3\text{COONH}_4$ improved the transformation from sucrose into carbon and shortened the stay time for the intermediates under hydrothermal conditions. The more intermediates in control group are favorable to self-aggregate to form massive pure carbon spheres, resulting in difficulty of magnetic separation for the products (Figs. S2b and S2c). On the contrary, the products of experimental group could be easily collected using a magnet within 31 s and almost no pure carbon was residual in the solution (Figs. S2c and S2d). Moreover, the dose of sucrose and $\text{CH}_3\text{COONH}_4$ also influenced the carbon coating formation on the surface of the Fe_3O_4 microspheres. It was found that Fe_3O_4 microspheres could not be fully coated by carbon with limited dosage of sucrose or $\text{CH}_3\text{COONH}_4$ (Figs. S3 and S4).

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