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ARTICLE

Fabrication, Characterization and Properties of Superparamagnetic Reduced Graphene Oxide/Fe_3O_4 Hollow Sphere Nanocomposites

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Abstract: The reduced graphene oxide wrapped Fe_3O_4 hollow nanospheres have been synthesized by a simple self-assembly process driven by electrostatic interaction. The structure, the chemical component and the morphology were characterized by XRD, SEM, TEM, FTIR, and Raman spectroscopy. Result show that the fabricated nanocomposites exhibit superparamagnetic property with the saturation magnetization of 70.2 $A\cdot m^2\cdot kg^{-1}$ at room temperature and allow rapid separation in water solution under an external magnetic field. The high magnetism and excellent water dispersibility make the nanocomposites ideal candidates for various important applications such as magnetic resonance imaging, biosensors, communication, and microwave absorption.

Key words: Fe_3O_4; graphene; nanocomposites; superparamagnetic

Owing to its superior electronic, thermal and mechanical properties as well as chemical stability, graphene as a well defined two dimensional honeycomb structure of carbon materials has been attracting more and more attention from both experimental and theoretical scientific communities[1-4]. Graphene and graphene oxide (GO) are potential nanoscale building blocks for new hybrid materials because of their layered structure and special surface properties [5,6]. It is believed that the composites of metal oxides and graphene would have better functionalities and performances in their applications[7]. Among those hybride materials, the ones with the magnetic nanoparticles (e.g. Fe_3O_4, Fe_2O_3) are an important class of materials due to their numerous applications in various technological fields[8,9].

Magnetite (Fe_3O_4) nanoparticle (NPs) have attracted world-wide research attention not only because of their unique size- and morphology-dependent physical and chemical properties, but also for their potential applications in many fields, including magnetic storage, biosensors, communication materials, magnetic resonance imaging and as materials for microwave absorbing and shielding research [10-12]. The controlled synthesis processes of Fe_3O_4 NPs to deliver a desired structure, composition, and shape control made them be used in various promising applications. There are many reports of the GO/Fe_3O_4 composites that are synthesized by the chemical methods including the in-situ reduction of iron precursors into GO sheets[13-16]. However, these strategies lack the control of morphology, loading ratio and the size of the Fe_3O_4 NPs over GO, which restricts their practical use. In this regard, the synthesis of GO/Fe_3O_4 nanocomposites via a method involving the ex-situ controlled synthesis of Fe_3O_4 NPs and their controlled assembly on GO sheets is a promising way to develop highly efficient materials for technological applications[17,18].

In the present paper, a simple self-assembly process driven by electrostatic interaction was used to produce reduced graphene oxide/magnetite (r-GO/Fe_3O_4) nanocomposites. A typical procedure is present in Fig.1. The process is driven by the mutual electrostatic interaction between negatively charged GO and positively charged 3-aminopropyltriethoxysilane (KH540) modified Fe_3O_4 hollow nanospheres, resulting in a flexible and ultrathin r-GO coating enwrapping

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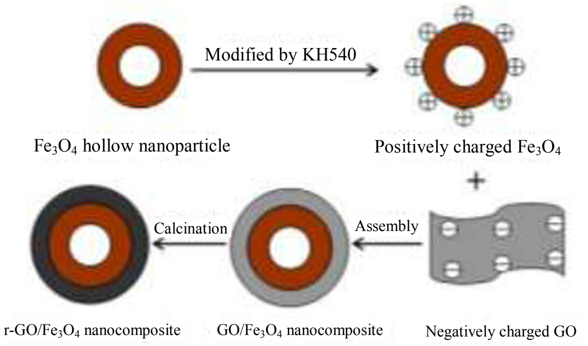

Fig.1 Synthetic route to r-GO/Fe_3O_4 nanocomposites

the Fe_3O_4 NPs. The method demonstrated in this paper is a viable and facile approach and can be extended to fabricate other graphene-based nanocomposites.

1 Experiment

1.1 Synthesis of r-GO/Fe_3O_4 nanocomposites

The preparation of GO was carried out by a modified Hummers method[19]. Fe_3O_4 hollow nanospheres were fabricated according to the literature with minor modification[20]. The as-prepared Fe_3O_4 hollow nanospheres were first surface modified with amino groups as follows: 2 mL of KH540 was added dropwise to the Fe_3O_4 NPs solution (1 g dispersed in 45 mL ethanol and 5 mL distilled water). The solution was adjusted to pH=10 by ammonia and stirred for 24 h. The resulting aminated Fe_3O_4 and GO (200 mg) were separately dispersed in hydrochloric acid aqueous solution (600 mL, pH=3.0) by sonication. The zeta potentials of the Fe_3O_4 nanoparticles and GO solutions at pH=3.0 were about +42 mV and −25 mV, respectively. The above two solutions were then mixed under mild stirring. GO/Fe_3O_4 nanocomposites were formed by the electrostatic interaction between the two oppositely charged nanomaterials. Finally the resulting production was recovered by centrifugation and rinsed with ethanol and H_2O several times, and then dried under vacuum at 60 °C to obtain GO/Fe_3O_4 composite. After calcination at 450 °C for 3 h under nitrogen, black r-GO/Fe_3O_4 nanocomposites were obtained.

1.2 Characterization

The FTIR spectra were recorded on a MAGNA-IR 750 FTIR spectrometer. The crystal structures and microstructures of the nanocomposites were characterized by XRD (Ri-gaku/ Max-3A, Cu Kα, λ=0.15406 nm), SEM (JEOL JSM- 6330F) and TEM (JEOL 2010, 200 kV). Raman spectra were recorded on a LabRAM HR UVNIR multichannel confocal microspectrometer (HORIBA Jobin Yvon, France) with 635 nm laser excitation. Magnetic properties of Fe_3O_4 and r-GO/Fe_3O_4 were investigated with a MPMS-XL-7 superconducting quantum interference device (SQUID) magnetometer (Quan-tum Design, USA) at room temperature.

2 Results and Discussion

In the present work, Fe_3O_4 NPs were firstly modified by surface grafting of KH540 to render the oxide surface to be charged positively. Fig.2 shows FTIR spectra of Fe_3O_4 NPs, KH540 and the KH540-Fe_3O_4. Compared with Fe_3O_4 NPs, KH540-Fe_3O_4 presents absorption bands at 2975 and 2858 cm^{-1} assigned to stretching vibration of C-H bond of the propyl amine group. The silica network adheres to the particle surface via Fe-O-Si bond. The corresponding adsorption bands appearing at around 550 and 635 cm^{-1} overlap the Fe-O vibration of Fe_3O_4. However, the silane polymer over the surface of KH540-Fe_3O_4 can be confirmed with the bands at around 1110, 1045 and 980 cm^{-1} from the SiO-H and Si-O-Si groups. The two broad bands at 3410 and 1630 cm^{-1} are attributed to the N-H stretching vibrations and NH_2 bending mode of free NH_2 group, respectively. Furthermore, hydrogen-bonded silanols also absorb at around 3410 cm^{-1}[21,22]. Then the modified Fe_3O_4 NPs are assembled with negative GO by electro-static interactions. Finally, the precipitations are reduced to r-GO/Fe_3O_4 nanocomposites by calcination.

The crystal structures of as-synthesized GO, Fe_3O_4 NPs and r-GO/Fe_3O_4 nanocomposites were studied by XRD (Fig.3). All the diffraction peaks and positions of Fe_3O_4 NPs and r-GO/Fe_3O_4 nanocomposites can be indexed to the cubic

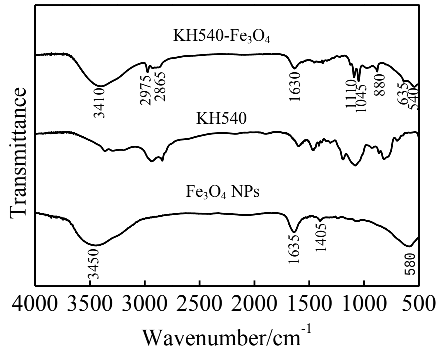

Fig.2 FTIR spectra of Fe_3O_4 NPs, KH540 and KH540-Fe_3O_4

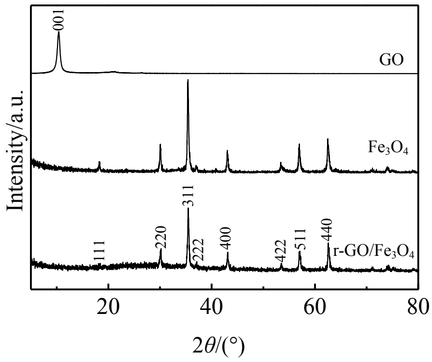

Fig.3 XRD patterns of GO, Fe_3O_4 hollow spheres and r-GO/ Fe_3O_4 nanocomposites

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