

Synthesis and magnetic dispersibility of magnetite decorated reduced graphene oxide

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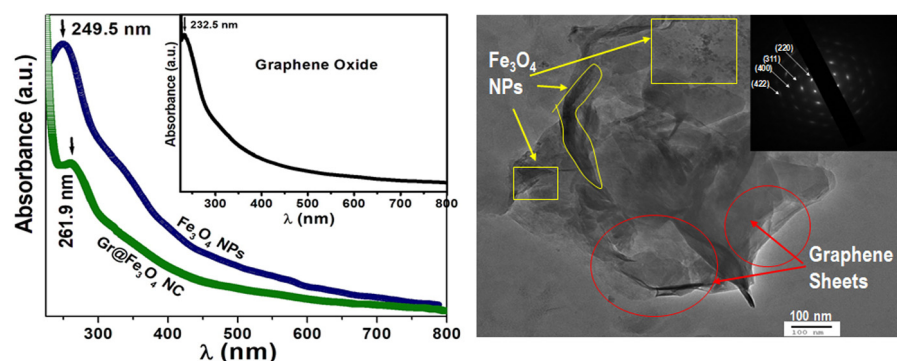
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GRAPHICAL ABSTRACT



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ABSTRACT

In the present work, we have successfully synthesized graphene oxide (GO) and reduced graphene oxide/magnetite (Gr@Fe₃O₄) nanocomposite (NC) via modified Hummers method and co-precipitation process respectively. Microstructural and physico-chemical properties were investigated through various analytical techniques. XRD and FTIR data analyses revealed highly pure nature of the samples without any other impurity phases. Electron microscopy (SEM/TEM) images of the nanocomposite clearly exhibit uniform and well disperse spherical nanoparticles (NPs) of Fe₃O₄ over the surface of the graphene sheets. UV-visible absorption spectroscopy indicates a red shift of the absorption band of Gr@Fe₃O₄ NC as compared to Fe₃O₄ NPs that suggests π-π noncovalent electrostatic interaction between the magnetic NPs and the graphene surface. The magnetic dispersibility of the NC was also explored using an external magnetic field produced by the simple bar magnets. The results affirm a higher magnetic dispersibility nature of the Gr@Fe₃O₄ NC than the graphene oxide and Fe₃O₄ NPs. These behaviour of the NC can be ascribed to synergistic structural and functional effect of Fe₃O₄ nanoparticles conjugate with graphene sheet.

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1. Introduction

Graphene has attracted tremendous attention due to its outstanding properties and potential applications. More recently, graphene based nanocomposites exhibit exceptional properties

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keeping the good characteristics of graphene viz. stiffness, electrical conductivity, optical transparency, large surface area etc. [1–3]. In particular, graphene with metal oxide nanoparticles (NPs) has shown potential applications such as cathode material for lithium-ion batteries, successful adsorbents for the removal of organic molecules and heavy metal ions from waste water, image contrast enhancing material etc. [4–9]. In this regard, H. Teymourinia et al. [10] prepared graphene quantum dots (GQDs)/TiO₂ nanocomposite that shows a significant enhancement in the photocatalytic activity for the degradation of rhodamine B organic dye. However, H. Safardoust-Hojaghan et al. prepared N-doped GQDs/TiO₂ as new nanocomposite and used for removal of methylene blue dye from water. They observed higher photodegradation efficiency of the nanocomposite to TiO₂ nanoparticles under UV light irradiation (enhanced from 40 to 85%) [11]. Now, it is well known that the nanotechnology utilizes the magnetic nanoparticles for the potential benefits of mankind due to large surface area associated with the high number of surface active sites that hope to enhance essentially disengaged by means of rate and degree of heavy metal adsorption [12]. In addition, magnetic NPs itself have rich structural, electrical and magnetic properties that can be explored for the numerous applications in hyperthermia, magnetic resonance imaging (MRI), arsenic removal, drug delivery etc. [13–16]. Among these two different types of materials, graphene/Fe₃O₄ nanocomposite (NC) has been fascinated significantly because of its enhanced magnetic properties and low toxicity. A few reports are available on the synthesis and applications of reduced graphene oxide/magnetite (Gr@Fe₃O₄) nanocomposite for specific purpose [17–19]. However, the main disadvantage of reported synthesis methods is a long reaction time and non-distribution of iron nanoparticles over the graphene sheet, that restricts the applicability of this material [20]. Therefore, many efforts have been devoted to prepare Gr@Fe₃O₄ nanocomposite using different methods. However, iron oxide nanoparticles were selected because they are eco-friendly transition metal oxide as well as being one of the most abundant and cheapest option in addition to the potential applications [21]. Moreover, Fe₃O₄ utilizes as a pseudo capacitive material because it has specific capacitance upto 165 Fg⁻¹ [22].

In the present study, we have successfully synthesized graphene oxide (GO) by modified Hummers method and monodisperse polycrystalline iron nanoparticles decorated reduced graphene oxide (Gr@Fe₃O₄) nanocomposite via co-precipitation process. The microstructure, optical properties and magnetic dispersibility of the prepared samples were investigated in detail.

2. Experimental

First of all graphene oxide was prepared by a modified Hummers [23,24] method and exfoliated using an ultrasonicator to obtain an aqueous dispersed phase of GO. Then, Gr@Fe₃O₄ nanocomposite was synthesized by co-precipitation method. At first, 100 ml aqueous solution of 4 mM FeCl₃·6H₂O and 2 mM FeCl₂·4H₂O was prepared. Thereafter, GO was added to the solution when its pH is adjusted to 4 by the addition of 1M NaOH and kept on stirring for 30 min to get more stable and homogeneous solution. In continuation, 1M NaOH was added again to make its pH value 10 and kept for 30 min with continuous stirring. The black colour precipitate thus obtained was washed several times with distilled water/ethanol and then dried in an oven at 60 °C for 48 h. The Fe₃O₄ nanoparticles were also synthesized in the similar manner without addition of GO for the comparative study. The structural characterization of the products was performed using a x-ray diffractometer (Rigaku Miniflex-II) in the 2θ range of 5–70° at a scan rate of 2°/min using CuKα radiations (λ_{Kα} = 1.5406 Å) operated at a current 15 mA and voltage of 30 kV. The thermal analysis measurement was carried out in the temperature range

of 50 to 800 °C. Morphology and microstructural properties of the prepared NC were investigated through JEOL scanning and transmission electron microscopes (SEM/TEM). Fourier transform infrared (FTIR) spectra were recorded in the wavenumber range of 400–4000 cm⁻¹. The UV-visible absorption spectra of the samples were observed by the spectrophotometer in the wavelength range of 200–800 nm.

3. Results and discussion

The thermogravimetric analysis (TGA) plot of the Gr@Fe₃O₄ nanocomposite in the temperature range of 50 to 800 °C is shown in Fig. 1(a). The weight loss between 50 to 150 °C is found to be 19.85% and that becomes 14.9% in the range of 150 to 600 °C. Further rise in temperature (i.e. 600 to 800 °C) shows only 2.63% weight loss. Thus the total percentage in weight loss for the entire temperature range is 37.38% that signifies a good thermal stability of the NC. This may be attributed to the high thermal stable structure of the Fe₃O₄ nanoparticles bonded to the graphene sheets [25]. Fig. 1(b) presents FTIR spectra of GO, Fe₃O₄ NPs and Gr@Fe₃O₄ NC. The intense absorption bands at around 461 and 542 cm⁻¹ are associated with the Fe–O bond in Gr@Fe₃O₄ NC. The band observed at ~1115 and ~1400 cm⁻¹ are due to the C–O and C–OH bending vibration respectively. The peaks at 1639 and 1732 cm⁻¹ in GO are attributed to the C=C and C=O bending vibrations respectively. The signal observed at ~2900 cm⁻¹ is accredited to the C–H vibration. Moreover, the FTIR spectrum of Gr@Fe₃O₄ nanocomposite is quite different from that of the graphene oxide in the range of 3000 to 3500 cm⁻¹. This may be due to the reduction of graphene oxide to the reduced graphene in Gr@Fe₃O₄ nanocomposite [26]. Two additional weak bands at around 1220 and 1500 cm⁻¹ are also observed due to the formation of either a monodentate or a bidentate complex between the carboxyl group and Fe ion on the surface of the magnetic particles, supporting that the Fe₃O₄ nanoparticles having π–π noncovalent electrostatic interaction with the graphene sheets [7]. The crystal structure of the GO and Gr@Fe₃O₄ nanocomposite were analysed through XRD patterns and shown in Fig. 1(c). The XRD profile of GO shows a major peak at around 2θ ~ 10°, that corresponds to the (002) plane. It has been observed that the diffraction peaks of the nanocomposite are found at diffraction angle (2θ) ~ 20.1°, 32.5°, 35.3°, 42°, 53.9°, 57.4° and 63.2° assigned to the (111), (220), (311), (400), (422), (511) and (440) crystal planes of the sample respectively. These planes are well matched with the crystal structure of the standard XRD data (JCPDS 65–3107) [27]. In addition, it is clear that the nanocomposite has crystalline structure and existence of Fe₃O₄ phase. Moreover the (002) peak present in the XRD pattern of GO is not observed for the Gr@Fe₃O₄ sample, indicating the reduction of the graphene oxide to the reduced graphene during synthesis process of the nanocomposite [14]. The average crystallite size has been estimated using the Scherrer's equation and found to be ~6 nm. Fig. 2(a) shows the SEM micrograph of Gr@Fe₃O₄ NC that clearly exhibits inhomogeneous deposition of Fe₃O₄ NPs over the graphene sheets. The chemical composition measurement of the NC was carried out through energy dispersive x-ray spectroscopy (EDS) technique. The analysis of EDS pattern indicates the presence of carbon, oxygen and iron elements in the NC that further ensures high purity of the sample and shown in Fig. 2(b). TEM image of the NC shown in Fig. 2(c) revealed a 2D graphene sheets decorated with Fe₃O₄ NPs. The average size of the Fe₃O₄ particles is found to be 7 nm, compared to that estimated by the XRD pattern [23]. It establishes a strong interaction between the reduced graphene and iron NPs that results attachment of the nanoparticles on the graphene sheets. The selected area electron diffraction (SAED) pattern of NC is presented in Fig. 2(d) that confirms the polycrystalline nature of the sample.

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