One-pot hydrothermal synthesis of Fe$_3$O$_4$/reduced graphene oxide nanocomposite for enhanced lithium storage

Xiao Zhang*, Bin Jiang, Yaping Xie & Feng Du

State Key Laboratory Base of Eco-chemical Engineering, College of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, Qingdao 266042, PR China

Email: zhx1213@126.com

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A simple one-pot hydrothermal approach is employed for the fabrication of Fe$_3$O$_4$/reduced graphene oxide (rGO) nanocomposite as the superior anode material for lithium ion batteries (LIBs). The reduction of graphene oxide sheets into rGO is achieved successfully during the hydrothermal process. The well-dispersed Fe$_3$O$_4$ nanoparticles are decorated on the surface of rGO sheets densely and homogeneously. With the introduction of rGO, the Fe$_3$O$_4$/rGO nanocomposite exhibits significant improvement in the reversible capability, cycling stability and rate performance in comparison with bare Fe$_3$O$_4$, delivering a high capacity of 884 mAh g$^{-1}$ at a current density of 200 mA g$^{-1}$ after 100 cycles. This hybrid composite is a potential candidate as anode material of high performance rechargeable lithium ion batteries.

Keywords: Composites, Nanocomposites, Hydrothermal synthesis, Iron oxides, Reduced graphene oxide, Ferromagnetism, Lithium ion battery

Magnetite (Fe$_3$O$_4$), as one of the important transition metal oxides, has been on the focus of intensive research due to its unique properties and various applications in biology$^1$, magnetic separation$^{2-3}$, magnetic response imaging$^4,5$, targeted drug delivery$^6,7$ and water treatment$^8-10$. Recently, Fe$_3$O$_4$ has aroused great research interest in its application as anode material for lithium ion batteries (LIBs)$^{11-14}$ for its natural abundance, low cost, environmental benignity, and high theoretical capacity. During the charge/discharge progress, the reaction mechanism of Li storage in Fe$_3$O$_4$ can be described as follows$^{15}$: Fe$_3$O$_4$ + 8Li$^+$ + 8e$^-$ = 3Fe + 4Li$_2$O. Based on this reaction, Fe$_3$O$_4$ achieves an attractive theoretical capacity of ~920 mAh g$^{-1}$, which is much higher than that of conventional graphite anode (~372 mAh g$^{-1}$). However, as a transition metal oxide, the commercialization of pure Fe$_3$O$_4$ anode is hampered by its intrinsic drawbacks of low electronic conductivity. Moreover, Fe$_3$O$_4$ electrode suffers from extremely high volume change during the Li insertion-extraction process, leading to pulverization of Fe$_3$O$_4$ material, causing mechanical failure and loss of electrical contact with the current collector.

Survey of the literature shows that carbon coating is an effective surface modification technique to improve the electrochemical performance of electrode materials$^{16}$. Introduction of coating carbon layer can increase the electronic conductivity of the composite electrode. Furthermore, the carbon layer can also buffer the large volume change during the discharge/charge process due to its elastic nature. As a result, the structure collapse will be avoided and the Fe$_3$O$_4$ electrode shall possess improved electrochemical performances. However, the preparation of carbon coated metal oxide composite usually requires an additional synthetic step of carbon coating, which complicates the synthesis process$^{17}$. Therefore, it is desirable to develop a one-pot facile synthesis method to prepare carbon coated metal oxide composite.

Herein, we have successfully designed a one-pot hydrothermal strategy for synthesis of Fe$_3$O$_4$/reduced graphene oxide (rGO) nanocomposite as high performance LIBs anode. Graphene has been employed as the carbon source due to its large surface area and high electronic conductivity$^{18}$. In the simple and facile one-pot hydrothermal process, Fe$_3$O$_4$ nanoparticles are deposited on the surface of graphene oxide (GO) sheets, and GO is in situ reduced into rGO simultaneously. High stability of the obtained Fe$_3$O$_4$/rGO nanocomposites has been observed under long sonication time, suggesting strong interaction between rGO sheets and Fe$_3$O$_4$ nanoparticles, which ensures good electronic transportation between the
two. The Fe₃O₄/rGO nanocomposite has been explored as the anode material for LIBs without requiring other post-processing such as heating treatment, to achieve a high reversible capacity and good cycling stability.

Materials and Methods

All reactants were of analytical grade and were used as purchased without further purification. Distilled water was used throughout. Natural graphite (Qingdao Tianhe Co., China) was oxidized following the Hummers method. In a typical synthesis route, 2 g of graphite powder and 1 g of NaNO₃ were added into 50 mL of conc. H₂SO₄ (98%) with vigorous stirring. The mixture was kept below 5 °C in an ice bath and stirred for 30 min. Then, 0.3 g KMnO₄ was added into the above mixture and stirred for 30 min, keeping the temperature below 10 °C. Subsequently, 7 g of KMnO₄ was added gradually within 1 h under stirring in order to prevent the temperature of the mixture from exceeding 20 °C. After the ice bath was removed, the mixture was stirred at 35±3 °C for 2 h when a brown dispersion solution was obtained. Then 90 mL of distilled water was poured into the above dispersion, and the temperature was kept above 90 °C for 15 min. The reaction was terminated by adding 55 mL of distilled water and 10 mL of 30% H₂O₂ solution. The resultant light yellow dispersion was filtered, washed with distilled water and 10 mL of 30% H₂O₂ solution. The obtained products were exfoliated with ultrasonication in water for more than 1 h. The resultant homogeneous brown-black GO dispersion was about 10 mg mL⁻¹ and could be conserved stably for months.

Synthesis of Fe₃O₄/rGO nanocomposite

The typical synthesis of Fe₃O₄/rGO nanocomposite is as follows: sodium dodecyl sulfate (SDS) was dissolved with magnetic stirring into the above GO dispersion and then FeSO₄ aqueous solution was added to the GO and SDS mixture. The weight ratio of Fe₂O₄ and GO was kept as 9:1, while the mole ratio of FeSO₄ and SDS was kept as 1:1. The pH value of the resulted solution was adjusted to 10 with NaOH (1 M) solution, transferred to Teflon-lined stainless-steel autoclaves, and then heated at 180 °C for 12 h. Then it was allowed to cool to room temperature naturally and the obtained precipitates were collected by centrifugation. The obtained samples were washed several times with absolute ethanol and deionized water, and then dried at 60 °C for 6 h. The bare Fe₂O₄ was also prepared following a similar hydrothermal process without the addition of GO for comparison.

Characterisation of Fe₃O₄/rGO nanocomposite

The samples were characterized by X-ray powder diffraction (XRD) on a Philips X’pert X-ray diffractometer with Cu-Kα radiation (λ = 0.154056 nm). Scanning electron micrographs (SEM) were obtained with a Jeol JSM-6700F scanning electron microscope. Transmission electron micrographs (TEM) were obtained using a FEI TECNAI F30. Raman spectra were recorded on a microprobe Raman system (Labram-1b, 632 nm as excitation source). Fourier transform infrared (FTIR) spectra were recorded on a FTIR analyzer (Nicolet Magna-IR750). X-ray photoelectron spectroscopy (XPS) measurements were made on a RBD upgraded PHI-5000c ESCA system (Perkin Elmer) with Mg-Kα radiation (hv = 1253.6 eV). The magnetic properties of Fe₃O₄ nanoparticles and Fe₃O₄/rGO nanocomposite were measured by a high sensitivity vibrating sample magnetometer (VSM) (model 7404, Lakeshore) at room temperature. The thermogravimetric analysis (TGA) were carried out on a TG2091F instrument from room temperature to 800 °C at a heating rate of 10 °C min⁻¹ in air.

Electrochemical studies

The electrochemical tests were carried out with a CR2016-type coin cell. Metallic lithium sheet was used as negative electrodes. The working electrode was fabricated by compressing a mixture of the active materials (Fe₃O₄/rGO nanocomposite or Fe₃O₄ nanoparticles), conductive material (acetylene black) and binder polyvinylidene fluoride (PVDF) in a weight ratio of 8:1:1 onto a copper foil at 10 Mpa. The typical mass load of active material was about 1–2 mg cm⁻². The electrode was dried at 120 °C for 12 h in a vacuum oven and the cell assembly was operated in a glove box filled with pure argon. The Clegard 2300 microporous film was used as separator. The electrolyte solution was 1 M LiPF₆ dissolved in a mixture of ethylene carbonate (EC)/dimethyl carbonate (DMC)/ethyl methyl carbonate (EMC) (1:1:1 in volume) Charge-discharge experiments were performed between 3 and 0.01 V on a battery cycler (Land CT2001A).

Results and Discussion

XRD studies

The crystal structures of GO, Fe₃O₄, and Fe₃O₄/rGO are characterized by XRD. As can be seen...
in Fig. 1a, all the diffraction peaks of the bare Fe$_3$O$_4$ nanoparticles can be indexed to a pure, well-crystalline, face-centered cubic spinel structure (JCPDS card no. 65-3107). There is no other impurity peaks observed, indicating the high phase purity of cubic Fe$_3$O$_4$. The good crystallization is proved by its strong and sharp reflection peaks. The XRD pattern of GO shows only one diffraction peak at around $2\theta = 9.3^\circ$, which corresponds to the (001) reflection and a $d$-spacing of 0.95 nm. The enlarged interlayer distance is attributed to the formation of hydroxyl, epoxy and carbonyl groups between GO sheets\textsuperscript{20}. This result indicates the successive oxidation of graphite into GO sheets. The diffraction peaks of Fe$_3$O$_4$/rGO composite are in good agreement with the face-centered cubic spinel structure and show no structural changes in comparison with Fe$_3$O$_4$ nanoparticles. However, the (001) peak of GO is not present in the XRD pattern of Fe$_3$O$_4$/rGO, indicating the reduction of GO during the hydrothermal process. The structural changes during the hydrothermal reduction process from GO to rGO is further confirmed by Raman spectroscopy (Fig. 1b). In the spectrum of Fe$_3$O$_4$/rGO, the peaks at about 1326 cm$^{-1}$ (D band, corresponds to $\kappa$-point phonons of $A_{1g}$ symmetry) and 1574 cm$^{-1}$ (G band, corresponds to an $E_{2g}$ phonon of $sp^2$-bonded carbon atoms) are observed. The intensity ratio of the D to G band ($I_D : I_G$) is calculated to be 1.06, higher than that of GO (0.84), indicating the reduction of GO in the hydrothermal process.

FTIR studies

FTIR techniques were employed to further confirm the formation of Fe$_3$O$_4$/rGO nanocomposite. As is shown in Fig. 1c, the characteristic FTIR peaks of GO are as follows: the strong and broad absorption bands at 3420 cm$^{-1}$ are assigned to the stretching vibrations of O-H, the peaks located at 1710 and 1060 cm$^{-1}$ are attributed to the stretching vibration of C=O and C-O of COOH groups respectively, the bands at 1385 cm$^{-1}$ are assigned to the O-H deformations of the C-OH groups and the peaks at 1625 cm$^{-1}$ are assigned to the O-H blending vibration, expoxide groups, and skeletal ring vibrations. The FTIR spectrum of Fe$_3$O$_4$/rGO shows distinct differences. The absorption peaks of 590 cm$^{-1}$ presented in the Fe$_3$O$_4$/rGO nanocomposite may be assigned to the stretching vibrations of Fe-O in Fe$_3$O$_4$ nanoparticles, which is consistent with the bare Fe$_3$O$_4$ nanoparticles\textsuperscript{21}. However, the absorption bands located at 1710 and 1060 cm$^{-1}$ corresponding to the C=O and C-O of COOH groups

![Fig. 1](image-url) — (a) XRD patterns of GO, bare Fe$_3$O$_4$ nanoparticles and Fe$_3$O$_4$/rGO nanocomposite, (b) Raman spectroscopy of GO and Fe$_3$O$_4$/rGO, and, (c) FTIR spectra of GO, bare Fe$_3$O$_4$ and Fe$_3$O$_4$/rGO. (d) TGA curve of Fe$_3$O$_4$/rGO.
and the peak at 1385 cm$^{-1}$ assigned to the O-H of C-OH groups are absent, indicating that GO has been reduced to rGO via the hydrothermal process. This result is in good agreement with the results of XRD and Raman spectra. The weight content of rGO in the Fe$_3$O$_4$/rGO nanocomposite was measured by TGA. It is clearly shown in Fig. 1d that the TGA curve shows a weight loss of 15% in the temperature range of 300–700 °C, which may be attributed to the oxidation of rGO.

XPS spectra

The XPS spectra of the Fe$_3$O$_4$/rGO nanocomposite are presented in Fig. 2. As shown in Fig. 2a, two characteristic peaks (710.6 and 724.1 eV) assigned to Fe 2$\text{p}_{3/2}$ and Fe 2$\text{p}_{1/2}$ of Fe$_3$O$_4$, respectively, are observed in XPS spectra of Fe 2p, while the typical peaks of 719 eV for Fe$_2$O$_3$ is absent, indicating the formation of Fe$_3$O$_4$.$^{22,23}$ The O 1s spectra (Fig. 2b) shows a peak located at 533 eV, which is consistent with the values reported for Fe$_3$O$_4$ in the literature,$^{24,25}$ further confirming the successful formation of Fe$_3$O$_4$. The C 1s XPS spectra of Fe$_3$O$_4$/rGO nanocomposite are shown in Fig. 2c. The peak centered at 284.6 eV is assigned to C=C bond, and its intensity is directly related with the ratio of $sp^2$ carbon. Another tiny peak of C 1s located at 287.8 eV is associated with $sp^3$ carbon, which is derived from hydroxyl (-OH), epoxide and carboxyl (-COO-) groups.$^{26}$ The high ratio of $sp^2$ to $sp^3$ carbon reveals that the hydroxyl (-OH), epoxide and carboxyl (-COO-) groups are removed almost completely, indicating the depth reduction of GO into rGO. These results are consistent with the results obtained from XRD, Raman, and the FTIR spectra.

Morphology

The morphology of the resultant Fe$_3$O$_4$ nanoparticles and Fe$_3$O$_4$/rGO nanocomposite was examined with SEM and TEM measurements. The SEM and TEM images of Fe$_3$O$_4$ (Fig. 3a and 3b respectively) show that the product comprises nanoparticles with diameter ranging from several tens to 150 nm. The SEM image of Fe$_3$O$_4$/rGO nanocomposite (Fig. 3b) shows that the surface of GO sheets is covered with Fe$_3$O$_4$ nanoparticles. As shown in Fig. 3d, the hybrid nanocomposite consists of two-dimensional rGO sheets decorated with Fe$_3$O$_4$ nanoparticles onto their surface homogeneously; the outline of rGO and Fe$_3$O$_4$ nanoparticles can be clearly observed. The average particle size of Fe$_3$O$_4$ in the Fe$_3$O$_4$/rGO nanocomposite is smaller than that of pure Fe$_3$O$_4$ nanoparticles, indicating that the addition of rGO has suppressed the crystal growth of Fe$_3$O$_4$ to some extent. It is evident that there exist several void spaces between the Fe$_3$O$_4$ nanoparticles. The porous structure will enable the infiltration of electrolyte, shorten the lithium ion transport path, and buffer the volume change of electrode during the discharge/charge cycles, resulting in improved reversible capacity, high rate capability, and good cyclic stability. It is worth mentioning that there are no large areas of rGO sheets without Fe$_3$O$_4$ decoration, no
apparent aggregation of $\text{Fe}_3\text{O}_4$ nanoparticles on rGO surface, and no individual $\text{Fe}_3\text{O}_4$ nanoparticles outside of rGO. Furthermore, the TEM image also shows the tight contact between $\text{Fe}_3\text{O}_4$ and rGO, suggesting the strong interaction between the two substances, which is also shown by the stability after a long time period of sonication. We believe that such tight interaction enables fast electron transport from $\text{Fe}_3\text{O}_4$ nanoparticles to current collector through rGO sheets, inducing the enhanced electrochemical performance. Additionally, the curled rGO sheets with good mechanical property and flexibility can prevent the aggregation of $\text{Fe}_3\text{O}_4$ nanoparticles and buffer the volume change of electrode during the discharge/charge cycles, which will be greatly beneficial to improve the cycling stability.

**Electrochemical studies**

To highlight the superior electrochemical performance of the $\text{Fe}_3\text{O}_4$/rGO nanocomposite for anode materials of LIBs, the electrochemical properties with respect to lithium insertion/extraction were tested with galvanostatic charge-discharge techniques. The electrochemical performance of bare $\text{Fe}_3\text{O}_4$ nanoparticles is also investigated for comparison. Figure 4(a & b) presents the typical galvanostatic charge and discharge profiles for the 1st and 2nd cycles of $\text{Fe}_3\text{O}_4$ nanoparticles and $\text{Fe}_3\text{O}_4$/rGO.
nanocomposite respectively at current density of 200 mA g\(^{-1}\). As is shown in Fig. 4, an obvious extended potential plateau from -0.8 V to cutoff voltage of 0.01 V is observed for both Fe\(_2\)O\(_4\)/rGO nanocomposite and Fe\(_2\)O\(_4\) nanoparticles, which is the typical characteristic of voltage trends for the Fe\(_2\)O\(_4\) electrode\(^{27,28}\). The first specific discharge capacity of Fe\(_2\)O\(_4\)/rGO nanocomposite is as high as 1422 mAh g\(^{-1}\), which exceeds the theoretical capacity of 920 mAh g\(^{-1}\) for Fe\(_2\)O\(_4\) (based on the conversion mechanism: \(8\text{Li}^+ + \text{Fe}_2\text{O}_4 \rightarrow 3\text{Fe} + 4\text{Li}_2\text{O}\)). This may be attributed to the decomposition of electrolyte and lithium salt in the formation progress of solid electrolyte interface (SEI) layer during the first discharge cycle\(^{29,30}\). In contrast, Fe\(_2\)O\(_4\) nanoparticles exhibit obviously depressed discharge capacities of 789 mAh g\(^{-1}\). The significantly improved discharge capacity of Fe\(_2\)O\(_4\)/rGO nanocomposite may be attributed to the introduction of rGO with good electronic conductivity. Fe\(_2\)O\(_4\)/rGO nanocomposite shows enhanced Li storage capacities as compared to bare Fe\(_2\)O\(_4\) nanoparticles (Supplementary Data, Fig. S1). For example, bare Fe\(_2\)O\(_4\) nanoparticles could only retain a capacity of 222 mAh g\(^{-1}\) after 10 discharge/charge cycles and 104.8 mAh g\(^{-1}\) after 100 cycles. However, when Fe\(_2\)O\(_4\) nanoparticles are deposited on the surface of rGO sheets, the resultant Fe\(_2\)O\(_4\)/rGO nanocomposites show remarkably higher capabilities. The discharge capacities are observed to be 653 and 884 mAh g\(^{-1}\) in the 10\(^{th}\) and 100\(^{th}\) discharge/charge cycles, which are 2.94 and 8.44 times the corresponding values of bare Fe\(_2\)O\(_4\) nanoparticles. The enhanced Li storage capability of Fe\(_2\)O\(_4\)/rGO nanocomposites can be attributed to the good electronic conductivity and high surface areas of rGO, since the presence of rGO guarantees the fast lithium ion and electron diffusion in the oxides anode materials.

Figure 5 shows the cycling performance of bare Fe\(_2\)O\(_4\) nanoparticles and Fe\(_2\)O\(_4\)/rGO nanocomposites at current density of 200 mA g\(^{-1}\). In the first cycle, the discharge capacity of bare Fe\(_2\)O\(_4\) nanoparticles is 789 mAh g\(^{-1}\). However, its capacity decreases dramatically and reaches 127.5 mAh g\(^{-1}\) after 20 cycles, indicating poor capacity retention for bare Fe\(_2\)O\(_4\) nanoparticles. This is because the continuous Li insertion/extraction reaction will result in large volume change, which will induce the cracking and crumbling of Fe\(_2\)O\(_4\) nanoparticles during the electrochemical cycling. As for Fe\(_2\)O\(_4\)/rGO nanocomposites, its electrochemical cycling performance shows a slight decrease in its initial 13 cycles and reaches 641.6 mAh g\(^{-1}\). Subsequently, the capacity increases significantly and reaches 884 mAh g\(^{-1}\) after 100 cycles, which is almost close to its theoretical capacity. It is interesting to note that all the capacities mentioned above for Fe\(_2\)O\(_4\)/rGO nanocomposites are much higher than the theoretical specific capacity of the commonly used graphite anode material\(^{31}\) (372 mAh g\(^{-1}\)). This phenomenon of initial drop followed by gradual rise of the capacity has been reported for other transition metal oxide electrodes and may be assigned to the possible activation processes\(^{32,33}\). Briefly, with increase in the number of Li insertion/extraction cycles, the structure and configuration of the Fe\(_2\)O\(_4\)/rGO electrode is rearranged and the connection between Fe\(_2\)O\(_4\) particles and rGO sheets becomes tight. Hence, the Li and electron transition among oxides and rGO as well as the transition between the anode materials and current collector is enhanced. Thus, it leads to the obvious increase in reversible capacity and the cycling stability. It is worth mentioning that the initial coulombic efficiency of Fe\(_2\)O\(_4\)/rGO nanocomposite

![Fig. 5 — Reversible discharge/charge capacities of (a) bare Fe\(_2\)O\(_4\) nanoparticles, and, (b) Fe\(_2\)O\(_4\)/rGO nanocomposite, at current density of 200 mA g\(^{-1}\) in a voltage window of 0.01–3 V.](image-url)
electron is about 60%, which is remarkably higher than the initial coulombic efficiency of 43.2% for bare Fe$_3$O$_4$ electrode, indicating that rGO with high electronic conductivity can reduce the initial irreversible capacity loss. The coulombic efficiency rises rapidly to 96.1% in the 10th cycle, and then remains above 97% in the following cycles, demonstrating an excellent reversible Li insertion/extraction performance.

Another surprising enhancement of the Fe$_3$O$_4$/rGO electrode is their remarkable rate performance in comparison with bare Fe$_3$O$_4$ nanoparticles electrode operated at various rates between 100 and 800 mA g$^{-1}$ (Fig. 6). Figure 6 shows the poor rate performance of bare Fe$_3$O$_4$ nanoparticles due to their inherently low electronic and ionic conductivity, as well as large volume expansion during Li insertion/extraction cycling. In sharp contrast, the Fe$_3$O$_4$/rGO electrode exhibits obviously improved specific capacities at all discharge rates. The discharge capacities of Fe$_3$O$_4$/rGO electrode are as high as about 1020, 900 and 760 mAh g$^{-1}$ at current density of 100, 200 and 400 mA g$^{-1}$, respectively. Even at the high specific current of 800 mA g$^{-1}$, the Fe$_3$O$_4$/rGO nanocomposite can still exhibit high reversible capacities of 610 mAh g$^{-1}$. All these values are obviously higher than the theoretical specific capacity of 372 mAh g$^{-1}$ for conventional graphite anode. On the other hand, discharge capacities of the bare Fe$_3$O$_4$ nanoparticles decrease dramatically with the increasing discharge rates, revealing a poor rate performance. For example, the discharge capacity is lower than 200 mAh g$^{-1}$ at a discharge rate of 200 mA g$^{-1}$. When the discharge rate increases to 800 mA g$^{-1}$, the discharge capacity is negligible. The enhanced rate capability of Fe$_3$O$_4$/rGO nanocomposite can be related to its unique structure.

The Fe$_3$O$_4$/rGO nanocomposite is composed of well-dispersed Fe$_3$O$_4$ nanoparticles anchored on the surface of rGO sheets evenly, and the contact property between the two is good. The highly conducting 2D-rGO electronic conductive network plays a key role in the discharge/charge cycle by increasing the electronic conductivity of the nanocomposite, enhancing and maintaining the electronic conductivity between the anode and the current collector, and, facilitating the electrolyte diffusion into the oxide materials due to the high surface area and existed void spaces$^{34,35}$. In addition, the nano-sized Fe$_3$O$_4$ particles can shorten the path length of electronic and lithium transport, which also favors the high rate capability$^{36}$.

**Magnetic properties**

The magnetic properties of bare Fe$_3$O$_4$ nanoparticles and Fe$_3$O$_4$/rGO nanocomposite were measured using a vibrating sample magnetometer. Figure 7a presents the hysteresis loop of Fe$_3$O$_4$ nanoparticles and Fe$_3$O$_4$/rGO nanocomposite obtained at room temperature. The specific saturation magnetizations ($M_s$) of Fe$_3$O$_4$ nanoparticles and Fe$_3$O$_4$/rGO nanocomposite are 13.32 and 9.79 emu g$^{-1}$, respectively, which are much lower than that of the bulk magnetite. The enlarged hysteresis loops (Fig. 7b)

![Fig. 6 — Rate performance of bare Fe$_3$O$_4$ nanoparticles and Fe$_3$O$_4$/rGO nanocomposite in a voltage window of 0.01–3 V.](image)

![Fig. 7 — (a) Magnetic hysteresis loop of Fe$_3$O$_4$ nanoparticles and Fe$_3$O$_4$/rGO nanocomposite measured at room temperature, and, (b) The enlargement of hysteresis loop at low magnetic field.](image)
show the coercive forces \((H_c)\) to be 197 Oe for Fe\(_3\)O\(_4\) nanoparticles and 151 Oe for Fe\(_3\)O\(_4\)/rGO nanocomposite. The remanent magnetizations \((M_r)\) are 2.98 emu g\(^{-1}\) for Fe\(_3\)O\(_4\) and 2.65 emu g\(^{-1}\) for Fe\(_3\)O\(_4\)/rGO. These results demonstrate that the Fe\(_3\)O\(_4\) and Fe\(_3\)O\(_4\)/rGO exhibit weak ferromagnetic behaviour. The Fe\(_3\)O\(_4\)/rGO nanocomposite with good magnetic performance may find applications in the fields of bionanotechnology and drug delivery.

**Conclusions**

In summary, a simple and facile one-pot hydrothermal method has been reported for the preparation of Fe\(_3\)O\(_4\)/rGO nanocomposite. In this synthesis strategy, graphene sheets are employed as the substrates for their high electronic conductivity and unique mechanical characters. The as-prepared hybrid nanocomposite may be used as anode material for LIBs without any further treatment. The hybrid Fe\(_3\)O\(_4\)/rGO nanocomposite shows enhanced electrochemical performances such as higher reversible capacities, better rate performance, improved cycling stability and higher initial coulombic efficiency as compared with the pure Fe\(_3\)O\(_4\) nanoparticles, which may be ascribed to the introduction of rGO due to which the efficient electron transport through the excellent conductive rGO from each active Fe\(_3\)O\(_4\) nanoparticles to the current collector is maintained. The nanostructure of Fe\(_3\)O\(_4\) nanoparticles is also preserved during the lithium ion insertion/extraction cycles. Magnetic measurements show that Fe\(_3\)O\(_4\)/rGO nanocomposite demonstrates good ferromagnetic behaviour with a specific \(M_r\) of 9.79 emu g\(^{-1}\). Considering the simple synthesis, improved lithium ion storage property, and good magnetic property, we expect that the Fe\(_3\)O\(_4\)/rGO nanocomposite will provide valuable insights for applications of LIBs as well as for drug delivery on a large scale.

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**Supplementary Data**

Supplementary data associated with this article i.e., Fig. S1, is available in the electronic form at http://www.niscair.res.in/jinfo/iijca/IJCA_53A(03)265-273_SupplData.pdf.