PAPER

Photocatalytic upgrading $\alpha$-Fe$_2$O$_3$ nanoparticles by incorporating MoS$_2$/rGO nanosheets

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Abstract
$\alpha$-Fe$_2$O$_3$/MoS$_2$/rGO nanocomposites was prepared by a two-step hydrothermal method and characterized by XRD, FESEM, EDS, FTIR, and UV–vis absorption spectroscopy. The results confirmed the formation of $\alpha$-Fe$_2$O$_3$/MoS$_2$-rGO (10 wt%) nanocomposites were composed of hematite nanoparticles with particle size of 30 nm and MoS$_2$/rGO composite nanosheets with maximum sheet thickness of $\sim$ 10 nm. Upon addition of MoS$_2$-rGO (8.0 wt%) nanosheets, the band gap of $\alpha$-Fe$_2$O$_3$ nanoparticles decreased from 2.3 to 1.7 eV that was accompanied by light absorption enhancement. Owing to synergetic effect between rGO and MoS$_2$ nanosheets leading to suppression of charge carrier recombination, prolongation of charge carrier lifetime, improvement of the interfacial charge transfer and increase in the number of active sites in $\alpha$-Fe$_2$O$_3$ nanoparticles, as-synthesized $\alpha$-Fe$_2$O$_3$/MoS$_2$-rGO (10 wt%) nanocomposites nanocomposite showed highly enhanced photocatalytic performance for Rh B degradation under light irradiation so that complete degradation of Rh B organic dye was achieved within 30 min.

1. Introduction
Among the various types of metal oxide semiconductors, hematite has shown to be an ideal candidate for many optical applications like heterogeneous photocatalytic degradation of organic pollutants and chemicals owing to its chemical inertness, cost-effectiveness, eco-friendliness, and stable nature [1–4]. As a well-known phenomenon, heterogeneous photocatalysis is light-induced generation of electron-hole pairs in a semiconductor material that is recognized as a promising strategy to easy, fast and cheap destruction-deactivation of organic pollutants [3, 5]. Effective use of electron-hole pairs is a key factor in rapid degradation of organic pollutants but the determinant step in the process is recombination of electrons and holes causing the weakening of the photocatalytic activity of many common semiconducting metal oxides [2–4]. Thus, in recent years, various approaches have been proposed to strengthen the photocatalytic activity of semiconductors, including the increase of external surface area, surface/morphology modification or creating structural defects [1, 2, 6–8]. Among them, modifying the surface of metal oxide semiconductors using noble metals such as platinum and palladium is one of the most successful methods to boost the photocatalytic activity. However, the rare nature of the noble metals imposing large costs has been restricted their widespread application [9–11]. Therefore, another contrivance must be made for improving photocatalytic activity of semiconductors.

Recently, co-catalysts based on nonprecious metals and non-metallic materials have been seriously pursued for photocatalytic improvement. Because of the very high surface area and excellent electron mobility, co-catalysts based on graphene, a single layer of graphite, have proven to be an excellent candidate to strengthen the photocatalytic activity of metal oxides. However, the photocatalysts based on metal oxide-graphene still need to be further strengthened for practical applications [12–15].

Recent study has been disclosed the synergistic effect between graphene and MoS$_2$ (a layered metal chalcogenide with direct band gap 1.9 eV at nano-scale) nanosheets as auxiliary catalysts for TiO$_2$ nanoparticles.
that can effectively prevent the electron-hole recombination and at the same time, strengthen the surface charge transmission and provide the active adsorption sites for photocatalytic reaction [16–20].

As a first effort of its kind, the present research has been allocated to synthesis, characterization and photocatalytic examination of ternary α-Fe2O3/MoS2-rGO (10 wt%) nanocomposites. To this end, the composite nanoparticles have been synthesized using a two-step hydrothermal process and characterized in terms of crystal structure, particulate properties, morphology, and elemental purity and optical properties by XRD, FESEM, EDS, FTIR, and UV–vis absorption spectrophotometry. The photocatalytic activity of the synthesized nanoparticles in degradation of Rhodamine blue (RhB) organic dye has been analyzed by UV–vis absorption spectrophotometry.

2. Experimental

2.1. Materials

The chemicals used in the experiments are listed in table 1.

2.2. Synthesis of MoS2-rGO (8.0 wt%) nanosheets

For the synthesis of MoS2-graphene nanosheets, firstly, graphene oxide (GO) was synthesized via modified Hummer method using graphite as carbon source. Briefly, 3 g graphite was added to a mixture of 53 ml H2SO4 (98%) and 27 ml HNO3 (65%) stirring on a cooling pad. 33 g KClO3 was then added slowly to the mixture kept under stirring for 90 h. The resulting slurry was poured into deionized water and filtered to obtain graphite oxide, which then exfoliated with ultrasonic treatment. The black powder was isolated by filtration, rinsed with water for five times, and then dried in a vacuum oven at 45 °C.

In a typical synthesis method of MoS2-graphene nanosheets, 1 mmol of Na2MoO4.2H2O was dissolved in 60 ml of deionized water. Then, 5 mmol of thiourea as sulfur source was added to it. After that, 0.0135 g GO was added to the solution. The resulted suspension was sonicated in an ultrasonic bath until complete dispersion of graphene. The stable suspension was then transferred into a 100 ml Teflon-lined autoclave that was kept in an oven at 220 °C for 18 h. After the desired time, the as-formed black precipitates were washed several times with deionized water and ethanol and finally dried at 70 °C for 12 h.

2.3. Synthesis of α-Fe2O3/MoS2-rGO (10 wt%) nanocomposites

For synthesis of α-Fe2O3/MoS2-rGO (10 wt%) nanocomposites via hydrothermal method, 2.3 g of FeCl3.6H2O were dissolved in 60 ml of deionized water. Next, 0.0135 g of as-synthesized MoS2-rGO (8.0 wt%) were added to the solution and put into an ultrasonic bath to form a well dispersed suspension. The resulted suspension was sonicated in ultrasonic bath until complete dispersion of graphene. The stable suspension was then transferred into a 100 ml Teflon-lined autoclave that was kept in an oven at 220 °C for 18 h. After the desired time, the as-formed black precipitates were washed several times with deionized water and ethanol and finally dried at 70 °C for 12 h.

2.4. Photocatalytic activity test

The photocatalytic degradation of Rhodamine B (RhB) was carried out in a two–walled Pyrex reactor under irradiation of a 150 W Xe lamp (MAX-150, Asahi Spectra, USA) with a maximum emission of 420 nm. In a typical test, 50 mg of the as-synthesized nanocomposites were loaded into the glass reactor containing 100 ml of 30 ppm RhB aqueous solution. The resulted suspension kept under stirring and was irradiated by the lamp. In various time intervals, 2 ml of the supernatant liquid was extracted by syringe and centrifuged at 9000 rpm for 10 min to separate the solid phase. Finally, RhB concentration at different time intervals was examined at the wavelength of 554 nm by UV–vis spectrophotometer.

### Table 1. The specification of chemicals used in the experiments.

| Chemicals         | CAT. no. | Manufacturer |
|-------------------|----------|--------------|
| C (Graphite)      | 104206   | Merck        |
| KClO3             | 104944   | Merck        |
| H2SO4, 98%        | 112080   | Merck        |
| HNO3, 65%         | 100456   | Merck        |
| Na2MoO4.2H2O      | 106521   | Merck        |
| H2NCSNH2          | 107979   | Merck        |
| FeCl3.6H2O        | 103943   | Merck        |
| C2H5OH            | 100983   | Merck        |
2.5. Materials characterization

The crystal structure of synthesized materials was determined using x-ray diffraction (XRD) measurements carried on a Siemens X-30 diffractometer with Cu Ka radiation ($\lambda = 1.5418$ Å) radiation. The morphology and elemental composition of materials were analyzed by field-emission scanning electron microscope (FESEM) performed on a Tescan Mira 3 LMU electron microscope integrated with energy dispersive x-ray spectroscope (EDX). The surface functional groups were characterized by Fourier transform infrared (FTIR) spectroscopy conducted on a Perkin Elmer Spectrum 400 spectrophotometer. The optical properties were investigated by UV–vis spectrophotometry obtained on a LAMBDA spectrophotometer.

3. Results and discussion

The XRD patterns of as-synthesized $\alpha$-$\text{Fe}_2\text{O}_3$, MoS$_2$-rGO (8.0 wt%), and $\alpha$-$\text{Fe}_2\text{O}_3$/MoS$_2$-rGO (10 wt%) nanoparticles are shown in figure 1. From figure, all diffraction peaks of $\text{Fe}_2\text{O}_3$ nanoparticles are well matched with diffraction pattern of hematite crystal structure according to JCPDS # 33-0664 [4, 19, 20]. The characteristic diffractions are (012), (104), (110), (113), (024), (116), (214) and (300) emerged at $2\theta = 24.12^\circ$, 33.14°, 35.64°, 40.74°, 49.38°, 54.08°, 56.94°, 62.44°, 64.08° and 71.76°.

Compared to XRD patterns of MoS$_2$, GO, and rGO shown in figure 2, the diffraction pattern related to MoS$_2$/rGO only shows the characteristics peaks of hexagonal MoS$_2$ (JCPDS 01-075-1539) suggesting that MoS$_2$ is the dominant phase [21, 22]. The MoS$_2$ in MoS$_2$/rGO has much better crystallinity than that of pure MoS$_2$, showing the beneficial effect of rGO on crystallization of MoS$_2$. The emerged broad reflections (002), (100), (103) and (110) at $2\theta = 14.12^\circ$, 33.24°, 39.26° and 58.76° reveal the formation of stacked layers of MoS$_2$ with small crystallite size [21, 23]. The absence of the characteristic peak of GO, (002), around 11° in MoS$_2$/rGO confirms the reduction of GO to rGO during the hydrothermal process. XRD pattern of $\alpha$-$\text{Fe}_2\text{O}_3$/MoS$_2$-rGO (10 wt%) nanocomposites presented in figure 1(c) shows the diffraction planes of both MoS$_2$/rGO and $\alpha$-$\text{Fe}_2\text{O}_3$ revealing the successful formation of nanocomposites. It seems that in this case the incorporation of MoS$_2$/rGO...
into $\alpha$-Fe$_2$O$_3$ nanoparticles leads to a decrease in the crystallinity of the as-formed nanocomposites in comparison with its main component i.e., iron oxide. This is attributed to the inhibitory effect of MoS$_2$-rGO (8.0 wt%) nanosheets on growth of hematite nanoparticles during the hydrothermal process [22, 23]. In addition, the incorporation of MoS$_2$/rGO results in slight shifting in the position of main diffraction of $\alpha$-Fe$_2$O$_3$ (104) from $2\theta = 33.14^\circ$ to $33.33^\circ$.

The calculation of mean crystal diameter of the as-synthesized nanomaterials using Scherrer equation give the values of 30, 15, and 20 nm for synthesized $\alpha$-Fe$_2$O$_3$, MoS$_2$-rGO (8.0 wt%) and $\alpha$-Fe$_2$O$_3$/MoS$_2$-rGO (10 wt%), respectively [4]. $\alpha$-Fe$_2$O$_3$ nanoparticles shows larger crystal size than $\alpha$-Fe$_2$O$_3$/MoS$_2$-rGO nanocomposites which can be attributed to the decreased crystallization and subsequently lower crystal growth of $\alpha$-Fe$_2$O$_3$ upon incorporation of MoS$_2$/rGO [22].

During the hydrothermal process, GO is reduced to graphene simultaneously with the dispersion of graphene-like MoS$_2$ nanosheets on graphene sheets. Subsequent hydrothermal treatment of FeCl$_3$.6H$_2$O and MoS$_2$-rGO hybrid led to crystallization of Fe$_3$O$_4$ and formation of $\alpha$-Fe$_2$O$_3$/MoS$_2$-rGO. Herein, the layered MoS$_2$-rGO hybrid serves as a support which is uniformly decorated with Fe$_3$O$_4$ nanoparticles.

Figure 3 shows the microstructures of as-synthesized nanomaterials investigated by FESEM. The FESEM images reveal that the $\alpha$-Fe$_2$O$_3$/MoS$_2$-rGO (10 wt%) nanocomposites are made up of hexagonal-shaped $\alpha$-Fe$_2$O$_3$ nanoparticles (as confirmed by XRD) with average diameter of 30 nm assembled by densely flower-like
MoS$_2$/rGO nanosheets with a thickness of less than 10 nm. This was estimated from FESEM micrographs using the SemAfore software (version 5.21). For this purpose, around 30 measurements were carried out on each micrograph. The figure also shows the FESEM micrographs of the components of $\alpha$-Fe$_2$O$_3$/MoS$_2$-rGO (10 wt%) nanocomposites consist of MoS$_2$ nanosheets, rGO nanosheets and spherical $\alpha$–Fe$_2$O$_3$ nanoparticles. Here, a close neighborhood of Fe$_2$O$_3$, MoS$_2$, and rGO components is achieved by the hydrothermal processing, which also has been reported by Xiang et al [16].

The EDS patterns shown in figure 4 clearly shows the elemental composition of the as–synthesized materials. From the patterns, all materials are of high elemental purity and well matched with the related stoichiometric composition. It should be noted that the whole FESEM image area was selected for the elemental characterization of as-synthesized materials. In addition, three spots in each sample were analyzed to study the homogeneity of the samples. The results did not show significant difference between EDS patterns of the selected spots indicating the homogeneity of the materials.

Figure 5 shows the 2D elemental mapping of $\alpha$–Fe$_2$O$_3$/MoS$_2$-rGO (10 wt%) nanocomposites. The figure obviously shows that: (i) MoS$_2$ sheets are uniformly decorated with rGO sheets and (ii) MoS$_2$/rGO nanosheets are well distributed between $\alpha$–Fe$_2$O$_3$ nanoparticles as dominant phase. Altogether, the results show the proper distribution of MoS$_2$-rGO (8.0 wt%) nanosheets among $\alpha$–Fe$_2$O$_3$ nanoparticles.

Figure 6 shows FTIR spectra of as-synthesized $\alpha$–Fe$_2$O$_3$, MoS$_2$/rGO (8.0 wt%) and $\alpha$–Fe$_2$O$_3$/MOS$_2$/rGO nanoparticles. In case of $\alpha$–Fe$_2$O$_3$ nanoparticle, the absorption bands located at 3443 cm$^{-1}$ and 1667 cm$^{-1}$, and 478 and 554 cm$^{-1}$ are assigned to the O–H vibrations in adsorbed water molecules, and Fe–O vibrations in $\alpha$–Fe$_2$O$_3$, respectively [24]. The FTIR spectrum of MOS$_2$/rGO includes the main peak due to Mo–S bond at 479 cm$^{-1}$. In addition, the characteristic functional groups of GO including C=O stretching at 1724 cm$^{-1}$, skeletal vibration of unoxidized graphitic domains at 1624 cm$^{-1}$, carboxyl O–H deformation at 1402 cm$^{-1}$, C–OH stretching at 1224 cm$^{-1}$ and C–O stretching at 1057 cm$^{-1}$ cannot be found in the spectrum of MoS$_2$/rGO nanosheets further confirming the reduction of GO sheets to rGO [22, 25]. The FTIR spectrum related to $\alpha$–Fe$_2$O$_3$/MoS$_2$-rGO (10 wt%) nanocomposite includes the peaks associated to both $\alpha$–Fe$_2$O$_3$ nanoparticles and MOS$_2$/rGO nanosheets. Since $\alpha$–Fe$_2$O$_3$ is the dominant phase in the nanocomposites the intensity of absorption bands related to $\alpha$–Fe$_2$O$_3$ is higher than those of MOS$_2$/rGO [22, 23].

Tauc’s plots of as–synthesized nanoparticles are presented in figure 7. Based on Tauc’s relation, the optical band gap ($E_g$) for as–synthesized nanoparticles is determined by extrapolating the straight line as portion of

![Figure 4. EDS patterns of (a) $\alpha$–Fe$_2$O$_3$, (b) MoS$_2$-rGO (8.0 wt%), and (c) $\alpha$–Fe$_2$O$_3$/MoS$_2$-rGO (10 wt%).](image-url)
The results show a decrease in the band gap of $\alpha$-Fe$_2$O$_3$ nanoparticle from 2.3 eV to 1.7 eV upon loading MoS$_2$/rGO nanosheets as deduced from absorption spectra. Upon addition of MoS$_2$/rGO the band gap of $\alpha$-Fe$_2$O$_3$ nanoparticles shows a significant red-shift that is due to the chemical bonding of $\alpha$-Fe$_2$O$_3$ to MoS$_2$/rGO to form $\alpha$-Fe$_2$O$_3$/MoS$_2$/rGO (10 wt%) nanocomposite with engineered absorption edge [26–28]. The close neighborhood of Fe$_2$O$_3$, MoS$_2$, and rGO components as shown in FESEM micrographs favors narrowing the band gap and vectorial transfer of electrons from Fe$_2$O$_3$ to MoS$_2$ and/ or graphene sheets, thus enhancing the charge separation required for photocatalytic activity [16]. The results are in agreement with our published work on $\alpha$-Fe$_2$O$_3$-decorated MoS$_2$ nanosheets [29].

The plot (ln($C_0/C$)) versus time t shown in figure 8 represents the progress in photocatalytic degradation of Rh B on as–synthesized nanomaterials. Based on obtained data, the nanomaterials could be arranged in terms of photocatalytic activity according to the following order:

$\alpha$ = Fe$_2$O$_3$ /MoS$_2$–rGO(10 wt%) >MoS$_2$–rGO(8.0 wt%) > $\alpha$ = Fe$_2$O$_3$

The obtained linear plots indicate that the Rh B degradation reaction follows the pseudo-first-order-kinetics [18, 30]:

Figure 5. Two–dimensional elemental map of as–synthesized $\alpha$–Fe$_2$O$_3$/MoS$_2$–rGO (10 wt%) nanocomposites.

Figure 6. FTIR spectra of as–synthesized nanomaterials; (a) $\alpha$–Fe$_2$O$_3$, (b) MoS$_2$–rGO (8.0 wt%), and (c) $\alpha$–Fe$_2$O$_3$/MoS$_2$–rGO (10 wt%).
where \( C_0 \) is the initial concentration, \( C \) the concentration at time \( t \) and \( k \) is the reaction rate constant. Based on the slope of the plots, the rate constant of Rh B degradation catalyzed by \( \alpha - \text{Fe}_2\text{O}_3 \), MoS\(_2\)-rGO (8.0 wt%) and \( \alpha - \text{Fe}_2\text{O}_3/\text{MoS}_2\)-rGO (10 wt%) is found to be 0.011, 0.125 and 0.149 min\(^{-1}\), respectively. The results the higher efficiency of \( \alpha - \text{Fe}_2\text{O}_3/\text{MoS}_2\)-rGO nanocomposites in degradation of Rh B compared to \( \alpha - \text{Fe}_2\text{O}_3 \) nanoparticles and MoS\(_2\)-rGO nanosheets. The higher photocatalytic activity of as-synthesized \( \text{Fe}_2\text{O}_3/\text{MoS}_2\)-rGO (10 wt%) nanocomposites is likely resulted from the synergetic effect between rGO and MoS\(_2\) nanosheets which causes the suppression of charge carrier recombination, prolongation of charge carrier lifetime, improvement of the interfacial charge-transfer and increase in the number of active sites. Here, due to the high electronic conductivity and high specific surface area of rGO, the photo-generated electrons of \( \alpha - \text{Fe}_2\text{O}_3 \) firstly transfer to rGO, and then transfer to the MoS\(_2\) for Rh B photocatalytic degradation \([22, 26, 27]\). Sun et al, confirmed that \( \text{Fe}_2\text{O}_3/\text{MoS}_2\)-graphene hybrids show enhanced photocatalytic activity which was 1.5 times higher than that of pure \( \text{Fe}_2\text{O}_3 \). The results indicated that the positive synergetic effect of the MoS\(_2\) and graphene in \( \text{Fe}_2\text{O}_3/\text{MoS}_2\)-graphene hybrids, which extended the light response and acted as a charges transfer medium, respectively, is the key to improve photocatalytic activity of \( \text{Fe}_2\text{O}_3 \) \([31]\).

\[
\ln \left( \frac{C_0}{C} \right) = k \times t
\]
4. Conclusions

In summary, a two-step hydrothermal method was employed for synthesis of α-Fe2O3/MoS2-rGO (10 wt%) nanocomposites. Crystal, particulate, elemental, and optical characterization of as-synthesized nanocomposites by XRD, FESEM, EDS, FTIR, and UV–vis absorption spectroscopy revealed the formation of α-Fe2O3/MoS2-rGO (10 wt%) nanocomposites composed of hematite nanoparticles with particle size of 30 nm and MoS2/rGO composite nanosheets with maximum sheet thickness of ~ 10 nm. The nanocomposite showed highly enhanced photocatalytic performance for Rh B degradation under light irradiation. Due to synergetic effect between rGO and MoS2 nanosheets which causes the suppression of charge carrier recombination, prolongation of charge carrier lifetime, improvement of the interfacial charge-transfer and increase in the number of active sites in α-Fe2O3 nanoparticles, α-Fe2O3/MoS2-rGO (10 wt%) nanocomposites showed higher photocatalytic activity than its constituents, α-Fe2O3, and MoS2-rGO (8.0 wt%). This work demonstrates the effectiveness and high potential of α-Fe2O3/MoS2-rGO (10 wt%) nanocomposites for photocatalysis applications.

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