Fabrication and adsorption properties of magnetic graphene oxide nanocomposites for removal of arsenic (V) from water

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Abstract
In this work, magnetic graphene oxide nanocomposites were synthesized by co-precipitation method and used as an adsorbent for removal of arsenic (V) ions from water. The structure and morphology of magnetic graphene oxide nanocomposites were studied by X-ray diffraction, Fourier transform infrared spectroscopy, transmission electron microscopy,
Brunauer–Emmett–Teller specific surface area, and vibrating sample magnetometry. Fourier transform infrared spectroscopy, X-ray diffraction, and transmission electron microscopy results of magnetic graphene oxide presented that the Fe$_3$O$_4$ nanoparticles in the size range of 10–25 nm were decorated on graphene oxide nanosheets. The adsorption properties of magnetic graphene oxide nanocomposites for arsenic (V) from water were investigated to study the effects of magnetic graphene oxide mass ratio, contact time, pH, and initial concentration. The suitable magnetic graphene oxide mass ratio of nanocomposites for arsenic (V) adsorption was determined to be 4:1 (FG2). The adsorption process on FG2 followed a pseudo-second-order kinetic and well fitted in to Langmuir isotherm model with the maximum adsorption capacity of 69.44 mg/g at pH 3. Accordingly, FG2 could be used as an effective adsorbent for removal of arsenic (V) from water.

**Keywords**
Magnetic, graphene oxide, nanocomposite, adsorption, arsenic

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**Introduction**

Arsenic (As) is considered as one of the most toxic chemicals and a carcinogenic element (Chandra et al., 2010). The most common forms of arsenic species in aqueous environments are arsenite As(III) (as H$_3$AsO$_3$ and H$_2$AsO$_3^-$) in ground water system and arsenate As(V) (as H$_2$AsO$_4^-$ and HAsO$_4^{2-}$) in surface water. Arsenic compounds easily accumulate in human organs and cause various health problems (Feng et al., 2012; Vaclavikova et al., 2008). Thus, the removal of As from water has been investigated.

Currently, the research and development of new adsorbent materials is being promoted. Graphene (Gr) with unique characteristics, such as large surface area, mechanical strength, and thermal conductivity, has attracted great interest of many researchers. Gr is a monolayer of carbon atoms, which is packed firmly, forming a two-dimensional structure of honeycomb lattice (Meyer et al., 2007; Zhu et al., 2010). Graphene oxide (GO), a derivation of Gr, is fabricated by exfoliation of graphite oxide, which is synthesized from graphite by oxidizing. GO contains a variety of oxygen-containing groups on the surface like hydroxyl (–OH), epoxy (–O–), carbonyl (–C=O), and carboxylic (–COOH). These groups provide GO with negative charged surface and an ability to interact with positive ions such as heavy metals, dyes, and organic compounds (Stankovich et al., 2006; Zhao et al., 2011). However, the nano size, high dispersion, and difficult separation of GO prevent the direct use of GO as adsorbents. To solve these problems, the magnetic graphene oxide (Fe$_3$O$_4$/GO) nanocomposite has been developed. The unique advantages of Fe$_3$O$_4$/GO such as high adsorption capacity and easy separation of Fe$_3$O$_4$/GO make it a potential adsorbent for the removal of pollutants from water (Fan et al., 2016).

In this work, the Fe$_3$O$_4$/GO nanocomposites were synthesized by co-precipitation method. The characterization of nanocomposites was examined by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), transmission electron microscopy (TEM), Brunauer–Emmett–Teller (BET) specific surface area, and vibrating sample magnetometry (VSM). Effects of Fe$_3$O$_4$/GO mass ratio, contact time, pH, and
initial concentration on the As(V) adsorption capacity of Fe₃O₄/GO nanocomposites were studied.

**Materials and methods**

**Materials**

Graphite powder (particle size of 20\(\mu\)m) and arsenic acid (H₃AsO₄ solution, 1000 mg/l) were purchased from Sigma-Aldrich, Germany. H₂SO₄ (98 wt%), H₃PO₄ (85 wt%), H₂O₂ (85 wt%), NaOH (99 wt%), HCl (36 wt%), FeCl₃·6H₂O (99 wt%), and FeCl₄·4H₂O (99 wt%) were purchased from Xilong, China. KMnO₄ (99 wt%), NH₄OH (30 wt%), and C₂H₅OH (95 wt%) were purchased from ChemSol, Vietnam.

**Synthesis of Fe₃O₄/GO nanocomposites**

GO was prepared by improved Hummers’ method (Marcano et al., 2010). Fe₃O₄/GO was synthesized by co-precipitation method (Fan et al., 2016). Briefly, 10 ml of FeCl₃·6H₂O and FeCl₂·4H₂O solution was slowly added into 50 ml of GO suspension (6 mg/ml). The mixture was stirred and heated to 80°C at pH 10 for 2 h. The black precipitation was collected by using a magnet, then washed with water and ethanol. After drying at 50°C for 24 h, the nanocomposites were obtained. The nanocomposites with different Fe₃O₄:GO mass ratios of 8:1, 4:1, 2:1, and 1:1 were marked as FG1, FG2, FG3, and FG4, respectively.

**Characterizations**

The crystal state was analyzed by XRD D2 Phaser machine (Bruker, Germany) with Cu Kα radiation (\(\lambda = 0.154\text{ nm}\)). FTIR spectra (Bruker FTIR Alpha-E, Germany) are used to analyze the functional groups on the surface of materials. The morphology of the materials was investigated by TEM (JEM-1400 microscope, Japan). The BET specific surface areas of materials were determined by Nova 3200e through nitrogen adsorption–desorption isotherm method (Quantachrome Instruments, USA). The elemental composition of materials was specified by using scanning electron microscope along with energy-dispersive X-ray analyzer (SEM/EDX) (Jeol JMS 6490, JEOL, Japan). Magnetic properties of the nanocomposites were performed using a MicroSense Easy VSM version 9.13 L machine (MicroSense, USA).

**Adsorption experiments**

Twenty milligrams of adsorbent was poured into 50 ml of As(V) solution. Effects of mass ratios of Fe₃O₄ to GO (FG1, FG2, FG3, and FG4), contact time (30, 60, 120, 240, 480, and 1440 min), pH (3, 5, 7, 9, and 12), and initial As(V) concentration (25, 50, 100, 150, and 200 mg/l) were studied. The residual As(V) concentration was determined by inductively coupled plasma mass spectrometry (ICP-MS 7500, Agilent, USA). All experiments were triplicated to estimate the error. The adsorption efficiency (H, %) and adsorption capacity (q, mg/g) were calculated using the following equations (1) and (2)

\[
H = \left(\frac{C_0 - C_e}{C_0}\right) \times 100\% (1)
\]

\[
q = \frac{V C_0}{m} - C_e \times \frac{V}{m} (2)
\]
\[ q = \frac{(C_0 - C_e)V}{m} \]  
(2)

where \( C_0 \) and \( C_e \) (mg/l) are the initial and equilibrium concentration of As(V), respectively; \( V \) (ml) is the volume of As(V) solution; and \( m \) (mg) is the mass of adsorbent.

The kinetic of adsorption process was investigated by pseudo-first-order and pseudo-second-order models. These models can be expressed as equations (3) and (4)

\[ \ln(q_e - q_t) = \ln q_e - k_1 t \]  
(3)

\[ \frac{1}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \]  
(4)

where \( q_e \) and \( q_t \) are the adsorption capacity at equilibrium and at time \( t \) (mg/g), respectively, and \( k_1 \) (min\(^{-1}\)) and \( k_2 \) (g/mg min) are the pseudo-first-order and pseudo-second-order rate constants (Chandra et al., 2010).

The zeta potential (pHpzc) values of materials were determined by pH drift method (Martinez-Vargas et al., 2018). Besides, to investigate adsorption capacity, the experimental data were applied to the Langmuir and Freundlich isotherm models. The linear equations for isotherm models are expressed as follows (equations (5) and (6))

\[ \frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{q_m k_1} \]  
(5)

\[ \ln q_e = \ln k_f + \frac{1}{n} \ln C_e \]  
(6)

where \( q_m \) (mg/g) is the maximum adsorption capacity, \( k_1 \) (l/mg) is the Langmuir constant, and \( n \) and \( k_f \) (l/mg) are the Freundlich constants (Hosseini et al., 2011).

**Results and discussion**

**Characterization**

**XRD patterns.** The XRD patterns of GO, Fe\(_3\)O\(_4\), and Fe\(_3\)O\(_4\)/GO are shown in Figure 1. The diffraction peak at \( 2\theta = 11.12^\circ \), ascribed to the crystallographic planes (002), indicates the distance of 0.795 nm between GO sheets. This distance was higher than the interlayer spacing of graphite (0.340 nm) since the oxygen-containing groups were inserted between GO sheets (McAllister et al., 2007). Fe\(_3\)O\(_4\)/GO had several peaks at \( 2\theta = 30.15^\circ, 36.27^\circ, 43.32^\circ, 53.89^\circ, 57.13^\circ, \) and \( 62.29^\circ \), which were respectively assigned to the crystallographic planes (220), (311), (400), (422), (511), and (440) of Fe\(_3\)O\(_4\) (Compton and Nguyen, 2010; Li et al., 2012; Liu et al., 2011). Additionally, the disappearance of GO peak indicates that Fe\(_3\)O\(_4\) nanoparticles were attached on GO surface, thus increasing the interlayer spacing between GO sheets.
The functional groups on the surface of GO and Fe₃O₄/GO were studied by FTIR spectra (Figure 2). For Fe₃O₄/GO, several peaks at 3355.43, 1600.63, 1399.45, and 1059.30 cm⁻¹ correspond to the vibration of hydroxyl (O–H), carbonyl (C=O), alkoxy (C–O), and aromatic C=C bonds, respectively (Metin et al., 2014; Yang et al., 2009). The peaks at 630.98 and
584.13 cm\(^{-1}\) can be ascribed to Fe–O bonds of Fe\(_3\)O\(_4\) (Sheng et al., 2012). This result indicates the existence of distinctive functional groups of GO and Fe\(_3\)O\(_4\).

**TEM images.** The morphologies of the materials were analyzed by TEM images as shown in Figure 3. GO shows high transparency with small wrinkles, which indicates effective exfoliation during sonication. The effect of Fe\(_3\)O\(_4\):GO mass ratio on the size and the distribution of Fe\(_3\)O\(_4\) on GO surface is shown in Figure 3(b) to (e). The Fe\(_3\)O\(_4\) nanoparticles with the average size of 10–15 nm were distributed uniformly on GO surface. However, Fe\(_3\)O\(_4\) nanoparticles in FG1 were larger with average size of 15–25 nm. The Fe\(_3\)O\(_4\) nanoparticles in FG2 were highly dispersed and showed an excellent interaction with GO sample. Besides, TEM images of FG3 and FG4 present fewer decoration of Fe\(_3\)O\(_4\) nanoparticles compared to FG2 and FG1.

The formation mechanism of Fe\(_3\)O\(_4\)/GO nanocomposite is shown as follows: (1) Fe\(^{2+}\) and Fe\(^{3+}\) ions linked with functional groups on the surface of GO and then (2) hydrolyze to form Fe\(_3\)O\(_4\) NPs.

![Figure 3. TEM images of (a) GO, (b) FG1, (c) FG2, (d) FG3, and (e) FG4. GO: graphene oxide.](image-url)
**BET specific surface areas.** Table 1 shows the BET surface areas of Fe$_3$O$_4$/GO nanocomposites and other materials. The Fe$_3$O$_4$ nanoparticles were attached on the surface of GO, decreasing the aggregation of Fe$_3$O$_4$ and stacking of GO, leading to the increase in specific surface area. Besides, the surface area of FG2 was higher than that of other nanocomposites due to a larger number of active sites on the surface. FG3 and FG4 had fewer Fe$_3$O$_4$ nanoparticles, leading to the reduction in the number of sites, which resulted in the decrease in surface area.

**SEM/EDX.** The composition of elements in Fe$_3$O$_4$/GO was determined through SEM/EDX data as shown in Figure 4 and Table 2. The Fe$_3$O$_4$ nanoparticles of FG2 were evenly dispersed on GO surface, indicating GO could reduce the agglomeration of Fe$_3$O$_4$.

| Materials   | BET specific surface area (m$^2$/g) | References          |
|-------------|-------------------------------------|---------------------|
| FG1         | 150.34                              | This work           |
| FG2         | 180.84                              | This work           |
| FG3         | 132.10                              | This work           |
| FG4         | 99.41                               | This work           |
| GO          | 84.76                               | This work           |
| Fe$_3$O$_4$ | 11.31                               | Yusuf et al. (2015) |
| CoFe$_2$O$_4$/Gr | 126.36                          | Khandanlou et al. (2015) |
| NiFe$_2$O$_4$/Gr | 57.11                           | Khandanlou et al. (2015) |

Table 1. The BET specific surface areas of Fe$_3$O$_4$/GO nanocomposites and other materials.

BET: Brunauer–Emmett–Teller; GO: graphene oxide.

Figure 4. The EDX results of FG2.
nanoparticles effectively. In EDX results, the weight percentage of Fe$_3$O$_4$ on FG2 was approximately 69.98%, which was consistent with experiment (66.67%).

**VSM.** The saturation magnetization ($M_s$) values of Fe$_3$O$_4$/GO nanocomposites were calculated to be 41.13, 36.34, 28.52, and 18.54 emu/g, respectively. The nanocomposites can be easily separated by using external magnetic field. The $M_s$ values of Fe$_3$O$_4$/GO and other materials are shown in Table 3. The $M_s$ values of nanocomposites are less than that of the

| Element | Mass (%) | Atom (%) |
|---------|----------|----------|
| C       | 16.97    | 0.08     |
| O       | 32.35    | 0.12     |
| Fe      | 50.68    | 0.25     |

**Table 3.** The $M_s$ values of Fe$_3$O$_4$/GO nanocomposites and other materials.

| Materials   | $M_s$ (emu/g) | References            |
|-------------|---------------|-----------------------|
| FG1         | 41.13         | This work             |
| FG2         | 36.34         | This work             |
| FG3         | 28.52         | This work             |
| FG4         | 18.54         | This work             |
| Fe$_3$O$_4$ | 92.00         | Van Lam et al. (2018) |
| CoFe$_2$O$_4$/Gr | 32.79       | Khandanlou et al. (2015) |
| NiFe$_2$O$_4$/Gr | 24.28       | Khandanlou et al. (2015) |

**Figure 5.** The As(V) adsorption capacities of GO, Fe$_3$O$_4$, FG1, FG2, FG3, and FG4. GO: graphene oxide.
bulk Fe₃O₄ (92 emu/g) due to the smaller size of Fe₃O₄ particles and the amount of loading of GO in the nanocomposite.

**Effects of factors on the As(V) adsorption capacity of Fe₃O₄/GO nanocomposites**

**Fe₃O₄:GO mass ratio.** The As(V) adsorption capacities of GO, Fe₃O₄, and Fe₃O₄/GO nanocomposites are shown in Figure 5. The presence of Fe₃O₄ nanoparticles on GO surface increased the adsorption capacity of Fe₃O₄/GO. Compared to GO and Fe₃O₄, the adsorption capacities of nanocomposites were also higher due to the following reasons: (1) the

![Graph showing the effect of contact time on As(V) adsorption capacity of FG2.](image)

**Figure 6.** Effect of contact time on As(V) adsorption capacity of FG2.

![Graph showing the linear pseudo-second-order model of FG2 for As(V) adsorption.](image)

**Figure 7.** The linear pseudo-second-order model of FG2 for As(V) adsorption.
electrostatic interaction between the negative charged surface of GO and As(V) ions, and (2) the complex interaction between metallic ions and oxygen-containing groups on the surface of Fe₃O₄/GO (Wang et al., 2013).

The FG2 has the highest As(V) adsorption capacity due to its largest surface area and the uniform distribution of Fe₃O₄ nanoparticles on the surface of GO. Therefore, FG2 was selected as an optimal adsorbent for the following experiments.

**Contact time.** Figure 6 shows the increase in As(V) adsorption capacity of FG2 as contact time increased. The As(V) adsorption process occurred rapidly due to a substantial amount of

![Figure 8](image)

**Figure 8.** Effect of pH on As(V) adsorption capacity of FG2.

![Figure 9](image)

**Figure 9.** Effect of initial concentration on As(V) adsorption capacity of FG2.
of active sites. After 480 min, As(V) ion concentration slightly changed due to the reduction of empty active sites on the surface of FG2. Therefore, the adsorption time of FG2 for As(V) was 480 min. The adsorption process of As(V) by FG2 well fitted to the pseudo-second-order model, with a correlation coefficient value close to 1 ($R^2 = 0.9987$) (Figure 7).

pH. The increase of pH values inhibited the As(V) adsorption capacity of FG2 (Figure 8). As(V) ions exist in different forms including $\text{H}_3\text{AsO}_4$ ($\text{pH} < 2.1$), $\text{H}_2\text{AsO}_4^-$ ($2.1 < \text{pH} < 6.9$), $\text{HAsO}_4^{2-}$ ($6.9 < \text{pH} < 11.5$), and $\text{AsO}_4^{3-}$ ($\text{pH} > 11.5$) (Zhu and Bates, 2013). Based on the experimental data, the pH of zero-point charge ($\text{pH}_{ZPC}$) of FG2 was measured to be 5.2. When the pH was lower than 5.2, the surface of FG2 was positively charged, making $-\text{OH}$ and $-\text{COOH}$ groups on FG2 surface become $-\text{OH}^+$ and $-\text{COOH}^+$ cations, which increased the number of active sites to interact with As(V) ions (Saadi et al., 2015; Zhu and Bates, 2013), leading to the increase in the adsorption capacity of FG2. As pH was increased, the surface of FG2 became less positively charged, the interactions between surface charges and As(V) declined gradually and changed into repulsive forces, decreasing the adsorption capacity. The experimental data showed that the effective removal conditions of FG2 for As(V) adsorption was achieved at pH 3.

| Table 4. Parameters of the Langmuir and Freundlich models of FG2 for As(V) adsorption. |
|---------------------------------|-----------------|-----------------|-----------------|
| Langmuir model                 |                 |                 |                 |
| $k_l$ (l/mg)                   | 0.385           | q$_m$(mg/g)     | 69.44           |
|                                |                 | $R^2$           | 0.9059          |
| Freundlich model               |                 |                 |                 |
| n                              | 4.787           | $k_f$ (mg$^{1-(1/n)}$ l$^{1/n}$/g) | 26.57 |
|                                |                 | $R^2$           | 0.8199          |

Figure 10. Langmuir (a) and Freundlich (b) isotherm model of FG2 for As(V) adsorption.
Initial concentration. Figure 9 shows the relationship between the adsorption capacity of FG2 and initial As(V) concentration. The uptake positively correlated with the initial concentration of As(V). The parameters of Langmuir, and Freundlich isotherm models are showed in Table 4 and Figure 10. The correlation coefficient of Langmuir model ($R^2 = 0.9059$) was higher than that of Freundlich model ($R^2 = 0.8199$).

The adsorption of FG2 followed the Langmuir isotherm model with $q_m = 69.44$ mg/g. Besides, the As(V) adsorption capacity of FG2 was in comparison with other materials (Table 5). The result could be explained as the linkage of Fe$_3$O$_4$ nanoparticles on the GO surface, leading to an increase in active sites and surface area, resulting in the increase in adsorption capacity.

Besides, the As(V) adsorption capacity of FG2 was compared with other materials as shown in Table 5. The result can be elaborated that Fe$_3$O$_4$ nanoparticles linked on the GO surface, leading to an increase in active sites and surface area, resulting in the adsorption capacity being increased.

Adsorption mechanism. The As(V) adsorption mechanism of Fe$_3$O$_4$/GO was interpreted via the surface complexation between As(V) ions and functional groups on nanocomposite surface, consisting of two types: outer-sphere and inner-sphere. For outer-sphere complexation, the interaction between As(V) ions and adsorption sites on nanocomposite surface depended on the electrostatic interaction. The in-sphere complexation was formed in Fe$_3$O$_4$/GO structures, the As–O–Fe bonds were formed between As–O(H) groups from H$_2$AsO$_4$/$\text{C}_0$ and HAsO$_4^{2-}$/C$_0$ as ligands and –OH, –COOH groups of Fe$_3$O$_4$/GO structure (Huong et al., 2018; Kumar et al., 2014).

Based on the results of the survey of pH effects, the adsorption capacity of Fe$_3$O$_4$/GO reduced while pH values increased. At low pH, the concentration of H$^+$ ions in the solution increases, –OH and –COOH groups become –OH$_2^+$ and –COOH$_2^+$ cations, which is advantageous for the adsorption of As(V) (Huong et al., 2018). The main mechanism of adsorption depends on the outer-sphere complex as follows

$$\text{Fe}_3\text{O}_4 - \text{OH}_2^+ + \text{H}_2\text{AsO}_4^- \rightarrow \text{Fe}_3\text{O}_4 - \text{OH}_2 \ldots \text{H}_2\text{AsO}_4$$  (7)

$$2\text{Fe}_3\text{O}_4 - \text{OH}_2^+ + \text{HAsO}_4^{2-} \rightarrow (\text{Fe}_3\text{O}_4 - \text{OH}_2)_2 \ldots \text{HAsO}_4$$  (8)

$$\text{Fe}_3\text{O}_4/\text{GO} - \text{OH}_2^+ + \text{H}_2\text{AsO}_4^- \rightarrow \text{Fe}_3\text{O}_4/\text{GO} - \text{OH}_2 \ldots \text{H}_2\text{AsO}_4^-$$  (9)

### Table 5. Maximum adsorption capacity $q_m$ (mg/g) of FG2 and other materials for As(V).

| Materials        | $q_m$ (mg/g) | References               |
|------------------|-------------|--------------------------|
| FG2              | 69.44       | This work                |
| Fe$_3$O$_4$      | 6.36        | This work                |
| GO               | 24.39       | This work                |
| Fe$_3$O$_4$/rGO  | 5.83        | Chandra et al. (2010)    |
| FeOOH/GO         | 23.78       | Wang et al. (2013)       |

GO: graphene oxide.
\[2\text{Fe}_3\text{O}_4/\text{GO} - \text{OH}_2^+ + \text{HAsO}_4^{2-} \rightarrow (\text{Fe}_3\text{O}_4/\text{GO} - \text{OH}_2)_2 \ldots \text{HAsO}_4 \] (10)

\[\text{Fe}_3\text{O}_4/\text{GO} - \text{COOH}_2^+ + \text{H}_2\text{AsO}_4^- \rightarrow \text{Fe}_3\text{O}_4/\text{GO} - \text{COOH}_2 \ldots \text{H}_2\text{AsO}_4^- \] (11)

\[2\text{Fe}_3\text{O}_4/\text{GO} - \text{COOH}_2^+ + \text{HAsO}_4^{2-} \rightarrow (\text{Fe}_3\text{O}_4/\text{GO} - \text{COOH}_2)_2 \ldots \text{HAsO}_4 \] (12)

**Conclusions**

Fe\(_3\)O\(_4\)/GO nanocomposites were successfully fabricated by co-precipitation method. XRD, FTIR, TEM, and SEM/EDX results showed that the Fe\(_3\)O\(_4\) particles were anchored on the surface of GO sheets. The Fe\(_3\)O\(_4\)/GO with mass ratio of 4:1 (FG2) showed highest adsorption capacity of As(V) compared with other nanocomposites. The adsorption process of FG2 well fitted to the pseudo-second-order and Langmuir isotherm models with the maximum adsorption capacity of 69.44 mg/g at pH 3. Therefore, FG2 could be applied as an adsorbent to remove As(V) from water.

**Declaration of Conflicting Interests**

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