CHAPTER 3

A NOVEL MAGNETIC Fe$_3$O$_4$/SiO$_2$ CORE-SHELL NANORODS FOR THE REMOVAL OF ARSENIC

3.1 INTRODUCTION

The removal of arsenic in drinking water poses a major challenge for researchers. Low level exposure of arsenic causes severe problems like chronic and carcinogenic effects (Chen et al 2010). In view of the high toxicity of arsenic, world bodies like US EPA and WHO have set a permissible limit of 10 to 50 ppb for arsenic in drinking water (Gu et al 2005 and Biswas et al 2008). Several methods like ion-exchange, adsorption and reverse osmosis have been attempted for the removal of arsenic but adsorption and ion-exchange are the promising techniques for efficient removal to bring arsenic within the permissible limit (Zhao et al 2009, Rengaraj et al 2002, Ning 2002 and Akin et al 2011). Adsorption is cheap but ion-exchange is expensive compared to other techniques. In recent years nanotechnology plays a vital role for arsenic removal in water (Upadhyayula et al 2009). Magnetic nanoparticles are found to be efficient for the removal of arsenic (Yavuz et al 2006 and Tuutijarvi et al 2009). Further, easy separation and reusability of magnetic nanoparticles were achieved by Liu et al (2008) and Hakami et al (2012).

Agriculture waste and wheat straw were used for the preparation of magnetic bio-adsorbent and the same was used for the removal of arsenic (Tian et al 2011). Fe-Ce bimetal oxide nanoparticles were used as adsorbent
and they exhibited maximum adsorption capacity compared to monometal oxides (Zhang et al 2005). The mixed metal oxide viz., Al₂O₃ and TiO₂ was used both as photocatalyst to oxidize As(III) to As(V) and adsorbent. It showed maximum adsorption when compared to individual metal oxide (Yamani et al 2012). Amine functionalized magnetic materials were reported to exhibit great potential for the separation of heavy metals and bacterial pathogens in drinking water (Wang et al 2010 and Huang et al 2010). Chandra et al (2010) synthesized magnetic iron oxide nanoparticles hybrid with graphene. This hybrid material showed high arsenic removal due to enhanced adsorption sites in graphene oxide, and easy separation from the solution. Recently, glycidyl methacrylate resin immobilized on tetraethylenepentamine was used as an anion exchange resin for efficient removal of As(V) (Donia et al 2011). Tetraamine functionalized with cellulose was also reported as an efficient anion exchanger for arsenic from aqueous solution (Anirudhan & Jalajamony 2010). The advantages of magnetic nanoparticles such as efficient removal and easy separation prompted to consider iron oxide as the core, and used as ion-exchanger and adsorbent for arsenic in water.

In this chapter, a low cost functionalized magnetic Fe₃O₄ nanostructure is presented for efficient removal of arsenic. Magnetic nanoparticles were shelled with SiO₂ to obtain core-shell Fe₃O₄@SiO₂ nanoparticles. They were functionalized with (3-aminopropyl) trimethoxysilane and then the amine functionalized Fe₃O₄@SiO₂ were crosslinked using 1,2-bromochloroethane. The resultant product yielded nanorod structures. Such nanorods have not been reported so far in the literature. The removal efficiency of arsenic by adsorption and ion-exchange over nanorods was monitored using ICP-OES. The adsorption capacity, kinetics, stability and reusability of the nanorods are attempted in this work.
3.2 PHYSICO-CHEMICAL CHARACTERIZATION OF ADSORBENT

3.2.1 X-ray Diffraction (XRD)

The XRD pattern of Fe$_3$O$_4$ nanoparticles is shown in Figure 3.1a. The intensity of reflections was low and their width was high. Using full width at half maximum (FWHM) for the patterns (311) and (440), the average crystallite size was calculated using Sherrer equation. It was found to be in the range of 15-30 nm. The nanosize of Fe$_3$O$_4$ was thus confirmed from the XRD patterns. The XRD pattern of Fe$_3$O$_4$@SiO$_2$ nanoparticles is shown in Figure 3.1b. The intensity of reflections was lowered due to false scattering of silica layer. Since the characteristic reflections of silica phases viz., cristobalite, tridinite and quartz were not observed, the silica layer may be present as amorphous. The position of the reflections was slightly shifted and hence there might be covalent bonding between silica layer and iron oxide nanoparticles. The free -OH groups on the surface of iron oxide might be used to form covalent bond with silica. The presence of -OH group on the iron oxide was also evident from FT-IR spectrum. The XRD pattern of amine functionalized iron oxide silica core-shell nanoparticles is shown in Figure 3.1c. The intensity of major reflections was further reduced, but that of the peak at 44.5° (2θ) increased slightly. The lowering of intensity was due to shielding of iron oxide particles by alkyl amino groups, which were bonded to iron oxide surface by Lewis acid-base interaction. The XRD pattern of amine functionalized core-shell iron oxide nanoparticles crosslinked with 1,2-bromochloroethane (MCSNs) is shown in Figure 3.1d. The intensity of major reflections further decreased due to organics surrounding the iron oxide particles.
3.2.2 Fourier Transform- Infra Red (FT-IR) Spectroscopy

The FT-IR spectrum of Fe$_3$O$_4$ nanoparticles is shown in Figure 3.2a. The broad envelope in the high energy region was due to adsorbed water. It was also confirmed by its bending vibration close to 1620 cm$^{-1}$. In the low energy region there were two peaks at 432 and 589 cm$^{-1}$ which were due to Fe-O vibration (Li et al 2011a). The FT-IR spectrum of Fe$_3$O$_4$@SiO$_2$ nanoparticles is shown in Figure 3.2b. This also showed the characteristic vibration of -OH stretching of water in the high energy region, and confirmed by its bending vibration close to 1620 cm$^{-1}$. The broad peak at 1049 cm$^{-1}$ was due to asymmetric Si-O-Si stretching vibration of silica, and the bending vibration was observed at 589 cm$^{-1}$. The FT-IR spectra of Fe$_3$O$_4$@SiO$_2$-NH$_2$
and MCSNs are shown in Figure 3.2 (c and d). The spectra showed similar features as that of Figure 3.2b. In addition, the stretching and bending vibrations of amine were observed above 3000 and 1582 cm\(^{-1}\) respectively, and the CH\(_2\) stretching vibration around 2800 cm\(^{-1}\).

![FT-IR spectra](image)

**Figure 3.2** FT-IR spectra of (a) Fe\(_3\)O\(_4\), (b) Fe\(_3\)O\(_4@SiO\(_2\))\(_2\), (c) Fe\(_3\)O\(_4@SiO\(_2\))-NH\(_2\) and (d) MCSNs

### 3.2.3 Scanning Electron Microscopy (SEM)

The SEM images of Fe\(_3\)O\(_4\), Fe\(_3\)O\(_4@SiO\(_2\))\(_2\) and Fe\(_3\)O\(_4@SiO\(_2\)-NH\(_2\)) are shown in Figure 3.3(a, b and c). The images revealed particles of varying size and exhibited agglomeration of particles. The SEM image of amine functionalized magnetic core-shell iron oxide nanoparticles after the reaction with 1,2-bromochloroethane is shown in Figure 3.3(d and e).
Figure 3.3  SEM images of (a) Fe$_3$O$_4$, (b) Fe$_3$O$_4$@SiO$_2$, (c) Fe$_3$O$_4$@SiO$_2$-NH$_2$ and (d and e) MCSNs
Rods of different dimensions were seen due to crosslinking of amino groups. This observation precluded dense amine functionalization of each nanoparticle. Hence the defective Si-OH groups might not be dense on each particle. Amine functionalization again required close positioning of three Si-OH groups, which was less probable. If amine functionalization was dense, then the resulting material could be a bulk solid rather than a rod. Thus the appearance of rod clearly proved only a fewer functionalization. The surface of the rods was not smooth. The presence of fine particles on the surface was evident from the images, and these particles might be unfunctionalized with amine. As the particles were unevenly deposited on the rods, it indicated that the rods might have grown at different time interval. The formation of rods was important as it provided highly porous fibrous network through which water can easily pass. Such a fibrous network is therefore important for adsorption and ion-exchange of arsenic ions from aqueous solution.

3.2.4 Transmission Electron Microscopy (TEM)

The TEM images of Fe$_3$O$_4$, Fe$_3$O$_4$@SiO$_2$ and Fe$_3$O$_4$@SiO$_2$-NH$_2$ are shown in Figure 3.4(a, b and c). The particle size was in the range of 10-30 nm. The TEM images of MCSNs are shown in Figure 3.4(c and d). The formation of rods was due to crosslinking of particles through organic species. The rods were of different sizes. There were also some free particles without forming such rods.
Figure 3.4  TEM images of (a) Fe$_3$O$_4$, (b) Fe$_3$O$_4$@SiO$_2$, (c) Fe$_3$O$_4$@SiO$_2$-NH$_2$ and (d and e) MCSNs
3.2.5  Nitrogen Sorption Studies

The nitrogen sorption isotherms of Fe₃O₄, Fe₃O₄@SiO₂, Fe₃O₄@SiO₂-NH₂ and MCSNs are shown in Figure 3.5(a-d). The specific surface area, pore volume and pore diameter of the adsorbent are presented in Table 3.1.

![N₂ sorption isotherms](image)

**Figure 3.5** N₂ sorption isotherms of (a) Fe₃O₄, (b) Fe₃O₄@SiO₂, (c) Fe₃O₄@SiO₂-NH₂ and (d) MCSNs

The specific surface area of Fe₃O₄ was higher than that of Fe₃O₄@SiO₂ as a result of increase in size. Fe₃O₄@SiO₂-NH₂ showed higher surface area than the other two due to creation of pores in between particles. The formation of pore was due to hydrogen bonding of amine of different particles. When Fe₃O₄@SiO₂-NH₂ was crosslinked using 1,2-bromochloroethane, the surface area increased due to formation of different pores. The pore size was close to 10 nm. The pore volume of MCSNs was higher than all other materials. The pores of Fe₃O₄ were due to
formation of voids between particles. Thus the textural parameters proved that MCSNs could be a novel material for adsorption applications.

Table 3.1  Textural parameters of Fe\textsubscript{3}O\textsubscript{4}, Fe\textsubscript{3}O\textsubscript{4}@SiO\textsubscript{2}, Fe\textsubscript{3}O\textsubscript{4}@SiO\textsubscript{2}-NH\textsubscript{2} and MCSNs

<table>
<thead>
<tr>
<th>Material</th>
<th>Specific surface area (m\textsuperscript{2}/g)</th>
<th>Pore volume (cm\textsuperscript{3}/g)</th>
<th>Pore diameter (nm)</th>
</tr>
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<tbody>
<tr>
<td>Fe\textsubscript{3}O\textsubscript{4}</td>
<td>215.75</td>
<td>0.2922</td>
<td>5.4181</td>
</tr>
<tr>
<td>Fe\textsubscript{3}O\textsubscript{4}@SiO\textsubscript{2}</td>
<td>202.2</td>
<td>0.4534</td>
<td>8.9687</td>
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<tr>
<td>Fe\textsubscript{3}O\textsubscript{4}@SiO\textsubscript{2}-NH\textsubscript{2}</td>
<td>262.78</td>
<td>0.4454</td>
<td>6.7821</td>
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<tr>
<td>MCSNs</td>
<td>335.72</td>
<td>0.6933</td>
<td>9.1791</td>
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</table>

3.2.6  Thermogravimetric Analysis (TGA)

The TGA results of Fe\textsubscript{3}O\textsubscript{4} and Fe\textsubscript{3}O\textsubscript{4}@SiO\textsubscript{2} nanoparticles are shown in Figure 3.6(a and b). The initial weight loss up to 200 °C was due to desorption of water, and the total weight loss was 10%. There was a gradual decrease in weight from 200 to 400 °C which was presumed to loss of oxygen forming iron oxide of various phases. It was also verified from the XRD patterns discussed in section 3.2.1. Hence iron oxide was established to form low thermal stability and it released oxygen at high temperature by getting reduced to lower valent oxide. The TGA results of amine functionalized iron oxide core-shell nanoparticles are illustrated in Figure 3.6c. There was a gradual decrease in weight from 30 to 600 °C. The initial weight loss up to 200 °C was due to loss of water, and above 200 °C gave a weight loss of 19.31% due to decomposition of alkylamino groups. Elemental analysis results also revealed carbon, hydrogen and nitrogen content of Fe\textsubscript{3}O\textsubscript{4}@SiO\textsubscript{2}-NH\textsubscript{2} as 12.13, 3.32 and 5.56 wt% respectively which is in close agreement with TGA results. This confirmed the functionalization of aminopropyl
groups, which is equivalent to 3.971 mmol of amino groups functionalized per gram. The TGA results of MCSNs are illustrated in Figure 3.6d. The initial weight loss below 150 °C was due to loss of water. The total weight loss of 21.2% above 200 °C was due to decomposition of organic.

![TGA curves of (a) Fe$_3$O$_4$, (b) Fe$_3$O$_4$@SiO$_2$, (c) Fe$_3$O$_4$@SiO$_2$-NH$_2$ and (d) MCSNs](image)

**Figure 3.6** TGA curves of (a) Fe$_3$O$_4$, (b) Fe$_3$O$_4$@SiO$_2$, (c) Fe$_3$O$_4$@SiO$_2$-NH$_2$ and (d) MCSNs

### 3.2.7 Vibrating Sample Magnetometer (VSM)

The results of saturation magnetization of Fe$_3$O$_4$, Fe$_3$O$_4$@SiO$_2$, Fe$_3$O$_4$@SiO$_2$-NH$_2$ and MCSNs are illustrated in Figure 3.7(a, b, c and d). The saturation magnetization of bare Fe$_3$O$_4$ was in the order of 56.21 emu/g whereas Fe$_3$O$_4$@SiO$_2$, Fe$_3$O$_4$@SiO$_2$-NH$_2$ and MCSNs exhibited 31.20, 24.93 and 21.24 emu/g respectively. The decrease in saturation magnetization was due to diamagnetic silica layer around Fe$_3$O$_4$ and amine functionalization. Although the saturation magnetization of MCSNs was found to be 21.24 emu/g, it was found to settle the composite powder using a hand held magnet.
3.3 BATCH SORPTION EXPERIMENTS WITH MCSNs

3.3.1 Adsorption Isotherms

Adsorption of As(III) and As(V) on MCSNs at different initial concentration is shown in Figure 3.8. The adsorption data of arsenic were fitted with Langmiur and Freundlich isotherm models (Fan et al. 2011 and Shuang et al. 2012). The Langmuir isotherm is described as

$$q_e = \frac{q_{\text{max}} b C_e}{1 + b C_e} \quad (3.1)$$

and the linear expression is expressed as in Equation (3.2)

$$\frac{C_e}{q_e} = \frac{1}{q_{\text{max}} b} + \frac{C_e}{q_{\text{max}}} \quad (3.2)$$

where $q_e$ is the equilibrium adsorption capacity, $C_e$ is the equilibrium concentration of arsenic (mg/L), $q_{\text{max}}$ is the maximum adsorption capacity and $b$ is the constant related to adsorption energy. From equation (3.2), $q_{\text{max}}$ and $b$
values were determined experimentally from the slope and intercept of the linear plot of \( C_e \) versus \( C_e / q_e \) (Figure 3.9). The Freundlich isotherm is expressed by the following equation

\[
q_e = K_f C_e^{1/n} \tag{3.3}
\]

and the linear expression is given in Equation (3.4)

\[
\log q_e = \log K_f + \frac{1}{n} \log C_e \tag{3.4}
\]

where \( q_e \) is the equilibrium adsorption capacity, \( C_e \) is the equilibrium concentration of arsenic (mg/L), \( K_f \) and \( n \) are Freundlich constants, which are related to adsorption capacity of the adsorbent and adsorption intensity. From Equation (3.4), \( K_f \) and \( 1/n \) values were determined experimentally from the slope and intercept of the linear plot of \( \log q_e \) versus \( \log C_e \) (Figure 3.10). The values of various parameters of both isotherm models and correlation coefficients are given in Table 3.2.

![Figure 3.8 Effect of initial As(III) and As(V) ion concentration on MCSNs at 30 °C. Arsenic concentration (3 to 7 mg/L), adsorbent dose 0.2 g and pH 7](image-url)
Figure 3.9 Langmuir adsorption plots of (a) As(III) and (b) As(V) on MCSNs at 30 °C and pH 7
Figure 3.10  Freundlich adsorption plots of (a) As(III) and (b) As(V) on MCSNs at 30 °C and pH 7
### Table 3.2 Langmuir and Freundlich adsorption isotherm constants for As(III) and As(V) on MCSNs

<table>
<thead>
<tr>
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<th>MCSNs</th>
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<tbody>
<tr>
<td></td>
<td>As(III)</td>
<td>As(V)</td>
<td></td>
</tr>
<tr>
<td><strong>Langmuir</strong></td>
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</tr>
<tr>
<td>$q_{\text{max}}$ (mg/g)</td>
<td>16.58</td>
<td>46.01</td>
<td></td>
</tr>
<tr>
<td>$b$ (L/mg)</td>
<td>0.443</td>
<td>6.706</td>
<td></td>
</tr>
<tr>
<td>$R^2$</td>
<td>0.995</td>
<td>0.995</td>
<td></td>
</tr>
<tr>
<td><strong>Freundlich</strong></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>$K_f$</td>
<td>5.53</td>
<td>14.61</td>
<td></td>
</tr>
<tr>
<td>$n$</td>
<td>2.221</td>
<td>2.044</td>
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</tr>
<tr>
<td>$R^2$</td>
<td>0.989</td>
<td>0.970</td>
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</table>

The experimental data fitted well with Langmuir isotherm with a linear correlation coefficient of 0.9957. The maximum adsorption capacity over MCSNs at 30 °C was found to be 16.58 and 46.01 mg/g for As(III) and As(V) respectively. The value of Freundlich constant is higher than one, which indicated favorable adsorption condition for arsenic on the nanorods. Ground water containing an initial concentration of 7 mg/L of arsenic upon treatment with MCSNs (0.5 g/L) for 10 min, the residual concentration of arsenic was found to be less then 10 μg/L. This confirmed the applicability of nanorods to bring down the arsenic content in drinking water below the tolerance limit of 10 μg/L. Thus the nanorods were proved highly efficient for fast adsorption of arsenic due to high surface area.

### 3.3.2 Adsorption Kinetics

The adsorption kinetics of As(III) and As(V) on MCSNs at pH 7 is shown in Figure 3.11. It clearly revealed that adsorption and ion-exchange
were very rapid in the first 5 min and then showed a minimum increase until the attainment of equilibrium. Compared to other material it showed fast rate of adsorption. So this material could be used as a novel ion-exchanger and adsorbent for practical applications. The adsorption kinetic of arsenic fitted well with pseudo-second order kinetic model with a correlation coefficient of 0.999 as shown in Table 3.3. The linear form (Gupta et al 2008) of the model is given in equation 3.5.

$$\frac{t}{q_t} = \frac{1}{k_2q_e^2} + \frac{1}{q_e} \ t$$  \hspace{1cm} (3.5)

where $q_e$ and $q_t$ are the equilibrium adsorption capacity (mg/g) and adsorption capacity (mg/g) at time $t$ and $k_2$ is pseudo second order adsorption rate constant (g mg$^{-1}$ min$^{-1}$). The $q_e$ and $k_2$ values were determined from the slope and intercept of the kinetic plot of $t/q_t$ versus time as shown in Figure 3.12. The $q_e$ obtained from the calculated value fitted well with experimental values. This demonstrated that the adsorption process followed pseudo second order kinetics.

![Figure 3.11 Adsorption kinetics of As(III) and As(V) on MCSNs](image)
Table 3.3  Pseudo-second order kinetic parameters for As(III) and As(V) on MCSNs

<table>
<thead>
<tr>
<th>Oxidation state</th>
<th>Pseudo-second order model</th>
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<tr>
<td></td>
<td>k₂</td>
<td>qₑₑ (mg/g) (calculated)</td>
<td>R²</td>
</tr>
<tr>
<td>As(III)</td>
<td>0.08735</td>
<td>11.85</td>
<td>0.999</td>
</tr>
<tr>
<td>As(V)</td>
<td>0.30522</td>
<td>33.04</td>
<td>0.999</td>
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Figure 3.12  Pseudo-second order plots of (a) As(III) and (b) As(V) on MCSNs
3.3.3 Effect of pH

The effect of solution pH (2 to 9) on the removal of As(III) and As(V) was studied over MCSNs and the results are depicted in Figure 3.13. The \( pK_a \) values of As(III) (\( pK_{a1} = 9.23, pK_{a2} = 12.13 \) and \( pK_{a3} = 13.4 \)) and As(V) (\( pK_{a1} = 2.3, pK_{a2} = 6.97 \) and \( pK_{a3} = 11.53 \)) (Fan et al 2011) played an important role in the ion-exchange and adsorption on MCSNs. As(III) removal was found to be low and steady in the pH range 2-9 due to unionized state of As(III). The unionized form of As(III) (H\(_3\)AsO\(_3\)) might not be ion-exchanged even though the pH was less than \( pK_{a1} \) (9.23). The amount of As(III) removal was found to be 11 mg/g due to adsorption of As(III) on silanol group.

![Figure 3.13](image.png)

**Figure 3.13** Effect of pH on As(III) and As(V): concentration 7 mg/L, temperature 30 °C, adsorbent 0.2 g/L and contact time 2 h

The removal of As(V) was low close to pH 2 due to unionized H\(_3\)AsO\(_4\) present in water. The increase in the removal of As(V) was observed around pH 3 -7 due to ionization of H\(_3\)AsO\(_4\) to H\(_2\)AsO\(_4^-\). Further increase in pH did not show much change in the removal of As(V). The ion-exchange and adsorption of As(V) on MCSNs were due to protonated form of amino
group (R-NH\textsubscript{2}-R) present in MCSNs as well as electrostatic attraction between As(V) and MCSNs.

### 3.3.4 Effect of Interfering Anions

The efficiency of arsenic removal from water in the presence of other anions such as phosphate, sulphate, nitrate, chloride and fluoride was studied at pH 7, and the results are depicted in Figure 3.14. The phosphate anion showed maximum interference for arsenic adsorption on the nanocomposite compared to other anions. Phosphate anion showed maximum interference due to similarity in molecular structure and pK\textsubscript{a} values. The order of competing anions is: phosphate> sulphate>nitrate>chloride>fluoride.

![Figure 3.14 Effect of coexistence of other anions on the removal of As(III) and As(V) using MCSNs at 30 °C. Arsenic concentration (7 mg/L), other anion (30 mg/L), adsorbent dose 0.2 g/L, contact time 3h and pH 7](image)

### 3.3.5 Regeneration and Stability of Adsorbent

It was established that MCSNs could be a versatile material to remove arsenic from aqueous solution. But once the material is exhausted, it
required regeneration. Dilute acid such as hydrochloric acid was found to be a good regenerant. This required stability of the material during regeneration with acid. By stability it means resistance of the material to release Fe$^{3+}$. There was negligible release of Fe$^{3+}$ (1.1%) even with 1M HCl due to formation of shell by silica, amine functionalization and nanorod formation. MCSNs showed high stability under acidic condition, and hence it was regenerated using 0.1 M HCl. The nanorods containing arsenic was removed completely. This study was repeated three times for 24 h and even then the leachability of Fe$^{3+}$ was not significant (0.34%). Similar results were also reported by Huang et al (2009). Regenerated MCSNs was used for adsorption again and the results are shown in Figure 3.15. The adsorption capacity decreased by 6.3% in the first regeneration cycle, and in the further three cycles showed decrease in adsorption capacity less than 3%. So, the same nanorods can be used for the removal of arsenic in aqueous solution atleast for four cycles.

![Figure 3.15](image.png)

**Figure 3.15** The regeneration efficiency of MCSNs for the removal of As(III) and As(V): concentration 7 mg/L, temperature 30 °C, adsorbent 0.2 g/L and contact time 24 h
3.4 CONCLUSION

The precursors used for the preparation of MCSNs are cheap and can be prepared easily. The MCSNs was found to be good for the removal of As(III) and As(V) from aqueous solution. The porous and high surface area (335 m²/g) of MCSNs can be used for the removal of arsenic in trace quantities. Since the material was stable and carried magnetic Fe₃O₄, it was recyclable for many cycles. The ion-exchange and adsorption were fast and equilibrium reached within five minutes. The removal of arsenic was high due to protonation of amino groups and presence of more number of adsorption sites. Since it is a highly porous material, treatment of water can be carried out without any diffusion problem. It can also be applied for the removal of other hazardous anions in aqueous solution. The interference of other ions in aqueous solution for adsorption of As(III) and As(V) was not significant.