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# SYNTHESIS OF MAGNETITE NANOPARTICLES AND EVALUATION OF ITS EFFICIENCY FOR ARSENIC REMOVAL FROM SIMULATED INDUSTRIAL WASTEWATER

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## ABSTRACT

In this study the efficiency of magnetic nanoparticles for removal of trivalent arsenic from synthetic industrial wastewater was evaluated. The nanoparticles was prepared by sol-gel method and characterized by X-ray methods including XRD, XRF, and SEM, and vibrating sample magnetometer (VSM). The results showed that synthesized nanoparticles were in the size range of 40-300 nm, purity of about 90%, and magnetization of nanoparticles was 36.5emu/g. In initial conditions including: pH=7, As(III) concentration of 10 mg/L, nanomagnetite concentration of 1g/L, shaking speed of 250 rpm and 20 minute retention time, 82% of As (III) was removed. Competition from common coexisting ions such as $Na^+$, $Ni^{2+}$, $Cu^{2+}$, $SO_4^{2-}$, and $Cl^-$ was ignorable but for $NO_3^-$ was significant. The adsorption data of magnetite nanoparticles fit well with Freundlich isotherm equations. The adsorption capacity of the $Fe_3O_4$ for As (III) at pH=7 was obtained as 23.8 mg/g. It was concluded that magnetite nanoparticles have considerable potential in removal of As(III) from synthetic industrial wastewaters.

**Key word:** Adsorption; Arsenic; Industrial wastewater; Magnetite nanoparticles

## INTRODUCTION

Advances in nanoscale science and engineering suggest that many of the current problems involving water quality could be resolved or greatly diminished by using nonmaterials. They could be classified to absorbent, nanocatalysts, bioactive nanoparticles, nanostructured catalytic membranes, submicron, nanopowder, nanotubes, magnetic nanoparticles, granules, flake, high surface area metal particle supramolecular assemblies. They have characteristic length scales of 9-10 nm including clusters, micromolecules, nanoparticles and colloids (Mayo *et al.*, 2007). Nanotechnology, now considered as one of the most important advancements in science and technology of the past decade, is related to the manipulation of materials and systems at the nanometer scale. At this length scale materials exhibit new properties for novel applications (Uheida *et al.*, 2006).

Heavy metals have been excessively released into the environment due to rapid industrialization and have created a major global concern (Wan Ngah and Hanafiah, 2008). Arsenic contamination of ground waters concerns several countries around the world and has been recorded by the World Health Organization as a first priority (Katsoyiannis and Zouboulis, 2002). Arsenic is classified

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as one of the most toxic and carcinogenic chemical elements. European Commission has revised the maximum concentration limit (MCL) for arsenic in drinking water, which has been reduced to 10 mg/L and by 2002 all drinking water supplies should be complied with the new limit; that has been adopted by USEPA (Katsoyiannis and Zouboulis, 2002).

According to Iran drinking water standard, MCL of arsenic is 50 ppb (µg/L). In Iran, arsenic contamination has been reported in groundwater sources in vast areas of western and north-western provinces especially in Kurdistan province in which serious problems have raised (Akbari *et al.*, 2010).

Arsenic occurs in both inorganic and organic forms in natural waters. Inorganic arsenic is the result of dissolution from the respecting mineral phase, such as arsenolite ($As_2O_3$), arsenic oxide ($As_2O_5$) or realgar ($As_2S_2$); it may be present in two oxidation states, as arsenate As(V) or arsenite As(III). Dominant arsenic species are a function of pH and redox potential.

The International Agency for Research on Cancer (IARC) has classified arsenic in drinking water as an established cause of cancer (Smith and Smith, 2004). The trivalent arsenic [arsenite, As(III)] is approximately ten times more toxic than pentavalent arsenic [arsenate, As(V), (Rahaman *et al.*, 2008). There are several industrial sources that elevate naturally occurring levels. One source is smelting of ores that contain arsenic trioxide, which is the raw material for industrial arsenic materials. The oxidized forms are converted back to sulphides by anaerobic processes on land and in water. Arsenic and its compounds have also been used extensively as herbicides and insecticides, hence runoff from treated fields contribute to higher arsenic levels (Streat *et al.*, 2008).

The development of nanotechnology at the end of 20th century has widened the variety of adsorbents. The removal of arsenic by nanoparticles has shown promising results with nanocrystalline titanium dioxide, nanoscale zero valent iron (nZVI) and modified zero valent iron particles. However, the same disadvantages as in conventional adsorption apply in the use of nanoadsorbents. Novel invention in adsorption technology is the use of magnetic nanoparticles as an adsorbent. Recently, it has been discovered in USA that removal of arsenic with magnetite ($Fe_3O_4$) nanoparticles is possible and the adsorption capacity for both As(III) and As(V) is at its highest level with small-diameter nanoparticles (10 nm), (Tuutijärvi *et al.*, 2009). Currently many methods exist to remove arsenic, such as coagulation, ion exchange, membranes and sorption with iron oxides. However, these methods can produce large amounts of waste, can be expensive, have poor removal efficiencies, and create iron residuals (Shipley, 2007). A variety of methods has been developed for removal of arsenic compounds from industrial wastewater. The use of magnetic nanoparticles for separation and preconcentration in analytical chemistry is opening a new methodology that is faster, simpler and more precise than those used traditionally. The greatest advantage of this method is that desired materials are separated from solution by a simple and compact process while fewer secondary wastes are produced. Other advantages are: large active surface area for given mass of particles and the ability to process solution that contains suspended solids (Khajeh and Khajeh, 2009).

Removal of arsenic from contaminated water to satisfy the drinking water standard has been a challenge for water authorities. Iron oxides have been reported to be effective for metal ion removal. Iron oxide-coated sand was used in many studies for arsenic removal (Boddu *et al.*, 2008).

Iron compounds such as hematite, goethite iron oxide-coated materials and granular ferric hydroxide (GFH) are the preferred group of substances for arsenic adsorption because they lead to low leaching of adsorbed arsenic from exhausted adsorbent (Ghanizadeh *et al.*, 2010). Iron oxides in various forms have been also used for treatment of radioactive and heavy metals from water and wastewater solutions. The study of Hsing-Lung Lien (2005) showed that zero-valent iron can efficiently remove arsenite from water (Lien and Wilkin, 2005).

The objective of the present study was to investigate the role and the effectiveness of new synthesized magnetic nano particles ($Fe_3O_4$) in the removal of As (III) from synthetic industrial wastewater. Magnetic nanoparticles were first synthesized by sol-gel method and then different experimental conditions e.g., amount of As (III) loaded on the nanoparticles, pH, adsorption time, shaking rate, concentration of magnetic nanoparticles, adsorption isotherm equation and competition of common coexisting ions have been studied.

## MATERIALS AND METHODS

### *Chemicals and reagents*

The chemicals used in this study were mostly reagent or higher grades and used as received (Merck Co.) without further treatment. The chemicals used were: $NaAsO_2$ (>99.9%), $FeCl_3.6H_2O$, $FeCl_2.\ 4H_2O$ (>99 %), ammonia solution (25%) and NaOH. All chemical solutions were prepared with deaerated deionized water (1) (DDW) by deoxygenating 18 MΩcm deionized water with 99.9% nitrogen for 2 h.

### *Synthesis and characterizations of adsorbent*

To prepare nanoscale magnetite, it is important to conduct the following reaction by combining a molar ratio of Fe(II)/Fe(III)=0.5 in basic solution:

$$2FeCl_3 + FeCl_2 + 8NH_3 + 4H_2O \rightarrow Fe_3O_4 + 8NH_4Cl$$

In the laboratory, magnetite nanoparticles were prepared by developing the existing sol–gel method (Amin *et al.*, 2010). First, 200mL of purified, deoxygenated water (resistivity of 18MΏ) was bubbled by nitrogen gas for 30 min. Then 5.2 g $FeCl_3$ and 2.0 g $FeCl_2$ were dissolved in the above mixture with mechanical stirring. Under the protection of nitrogen gas, 1.5 mol/L $NH_4OH$ solution was added dropwise into the above mixture under vigorous stirring. After an initial brown precipitate, a black precipitate was formed. When pH reached 8.0, the stirrer was turned off and magnetite settled gradually. The black precipitate was isolated by an external magnetic field with the supernatant decanted. To obtain the pure and neutral products, synthesized materials were washed with ultrapure water several times. Finally, the wet nanogel was freeze-dried (Hu *et al.*, 2004).

Scanning Electron Microscopy (SEM) equipped with an energy dispersive X-ray analyzer (EDX), (Seron, AIS-2100, South Korea) was used in this study. EDX analysis is an analytical technique used for the elemental analysis or chemical characterization of a sample (Wang and Lo, 2009). The composition of the materials was identified by X-ray fluorescence (XRF) (Bruker, Pioneer-S4, Germany) and X-ray diffraction (XRD) (Bruker, D8ADVANCE, Germany). The instrument was equipped with a copper anode generating (Cu -Kα) radiation (λ= 1.5406 Å).

The particle size and zeta potential of magnetite nanoparticles were measured with a particle sizer and a zeta potential meter (Malvern, England). Magnetization measurement was performed at room temperature using a vibrating sample magnetometer (VSM) device, in the Development Center of Kashan University (Kashan, Iran).

### *Magnetic separations*

Magnetic separations were performed with a magnetic field column separator consisting of a stainless steel column. The stainless steel column was packed with stainless steel wool (about 80µm wire diameter), with a packing volume of (50g stainless-steel wool). A magnetic field was applied, and then a sample was passed through the column. The nanoparticles were retained in the column as the solvent passed through (Mayo *et al.*, 2007).

### *Batch tests*

Batch adsorption studies were performed by mixing 0.1g magnetite nanoparticles with 20mL $NaAsO_2$ solution of varying concentration in a 100 mL erlenmeyer flask containing 0.1g of sorbent, and were agitated. Standard acid (0.1M $HNO_3$) and base (0.1M NaOH) solutions were used for pH adjustment. All the adsorption experiments were carried out at room temperature of 22.5 °C, and pH of 7 except stated otherwise. Adsorption isotherm studies were conducted by varying the initial As (III) concentration from 10 to 200 mg/L at different pH of 3, 5, 7, and 9. To explore the competitive effects of various coexisting ions, e.g., $Na^+$, $Cu^{2+}$, $Ni^{2+}$, $NO_3^-$, $SO_4^{2-}$ and $Cl^-$, on the removal of As(III), 20 mL of 50 mg/L As(III) solutions containing each of these components in two concentrations (50 and 100 mg/L) and 0.1 g magnetite nanoparticles were shaken at pH=7. After adsorption reached equilibrium, the adsorbent was separated via an external magnetic

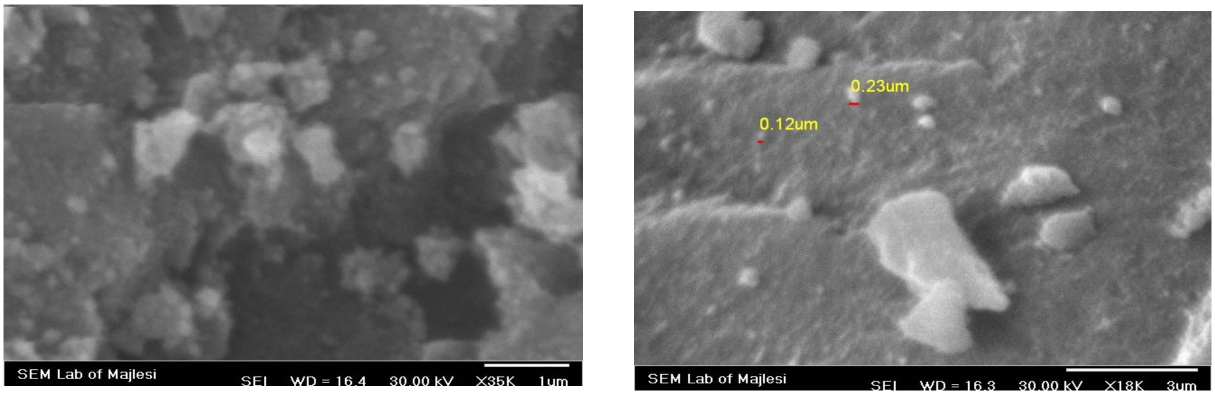

Fig. 1. SEM image of magnetic nanoparticles

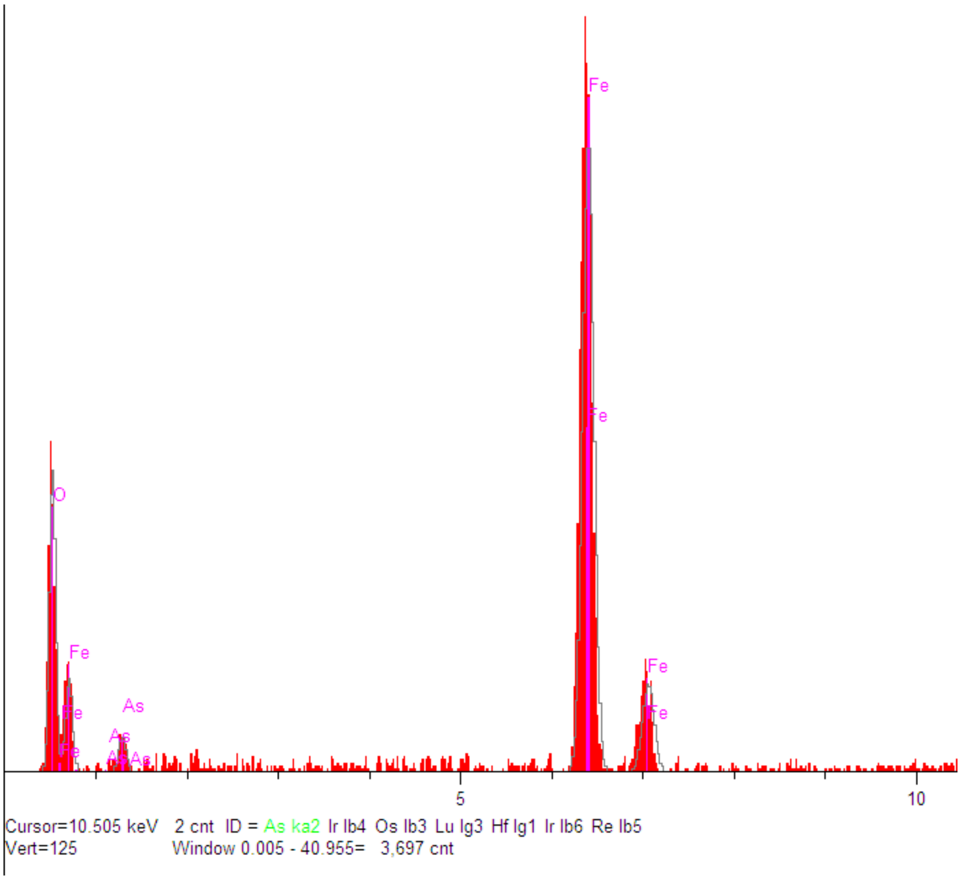

Fig 2: The SEM-Edx image spectra of nanoparticles magnetite after adsorption of arsenic

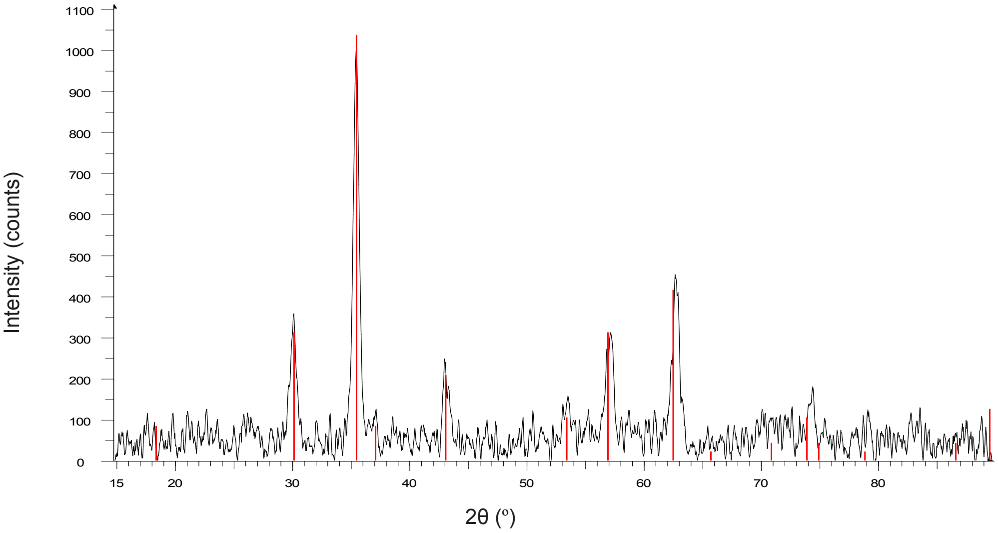

Fig 3: XRD patterns of magnetite particles

Table 1. XRF analysis of nanoparticles produced in this study

| Compound | Concentration (%w/w) |
|---|---|
| $Fe_3O_4$ | 89.61 |
| Cl | 4.09 |
| CuO | 0.048 |
| ZnO | 0.024 |
| LOI* | 7.02 |
| Total | 100.79 |

*Loss on Ignition (including compounds that affect the heat loss such as moisture and volatile matter).

field and the supernatant was collected for metal concentration measurements. All experiments were performed in duplicate and the averaged values were taken and reported here. The concentration of chromium was measured by an inductively coupled plasma optical emission spectrometer (ICP-OES) model Ultima-2 (France) (Hu *et al.*, 2005b), (Tuutijärvi *et al.*, 2009).

## RESULTS

### *Characterization of adsorbent*

The SEM image of the particles in Fig. 1, reveals that the synthesized nanoparticles in this study were multidispersed in the size range of 40-300 nm, with two specified particles of 120 and 230 nm (0.12 and 0.23 μm). The SEM image spectra of the nanoparticles after adsorption are shown in Fig. 2. The characteristics and purity of the magnetite nanoparticles were verified by XRD peaks of the nanocrystallite, which was matching well with standard $Fe_3O_4$ and was detected without other crystalline phases (Fig. 3). Results of X-ray fluorescence (XRF) of the nanoparticles, are shown in Table 1, which indicates, the purity of produced nanoparticles as about 90%.

The paramagnetic properties of the magnetic particles were verified by the magnetization curve measured by VSM (Fig. 4). The saturation moment of synthesized particles from the hysteresis loop measured from VSM was found to be 36.5 emu/g. The zeta potential of the magnetite nanoparticles was around - 6.85 mv.

## DISCUSSION

### *Effect of pH*

The results showed that pH clearly affects the removal efficiency when it was changed from

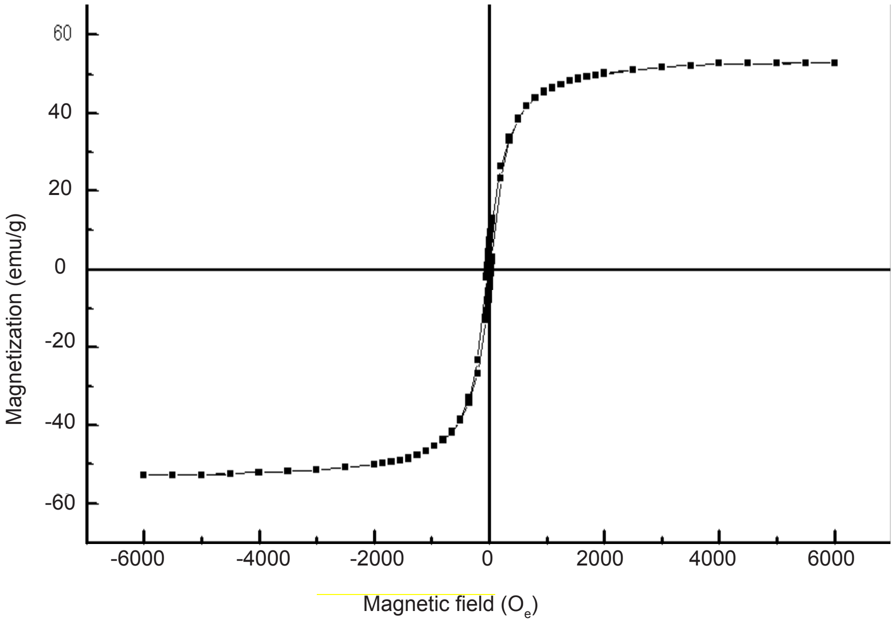

Fig 4: Magnetization curves of synthesized nanoparticles

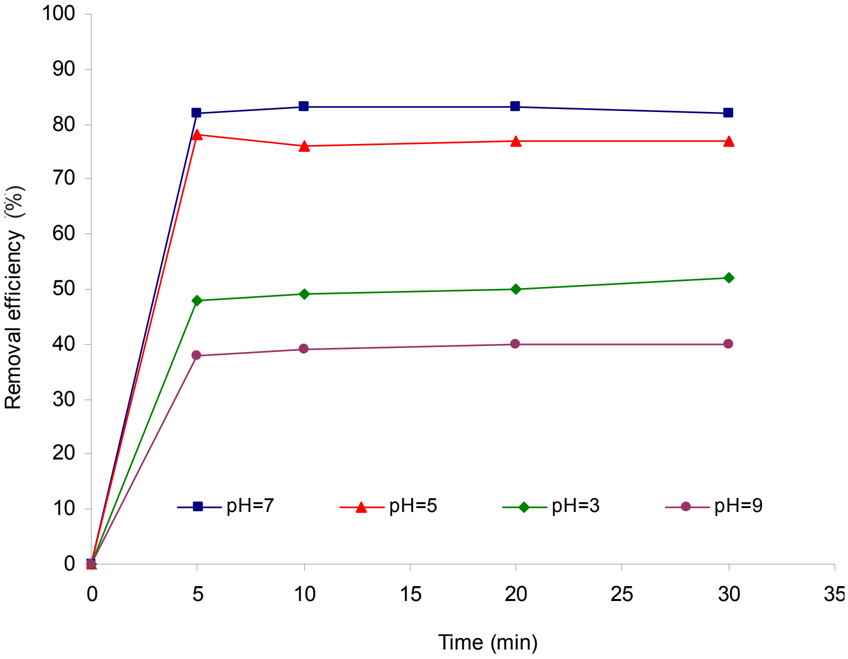

Fig 5: Effect of pH on the removal of As(III) by magnetite nanoparticles

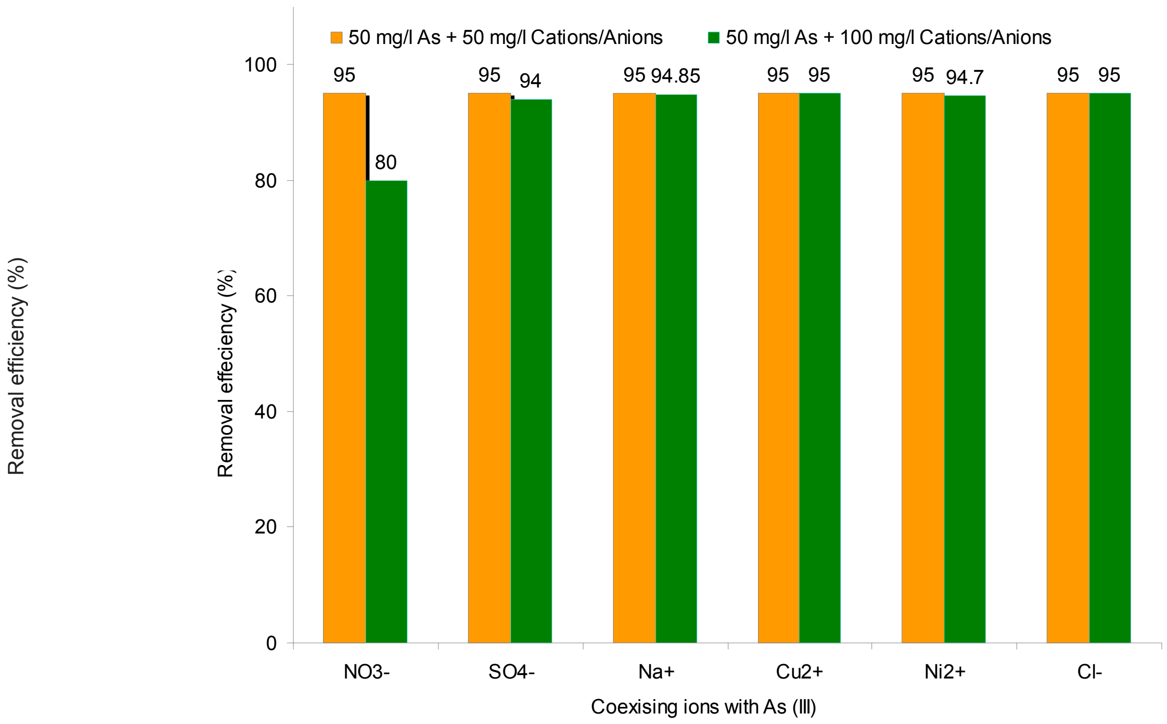

Fig 6: Effect of common ions on the removal of As(III) by magnetite nanoparticles

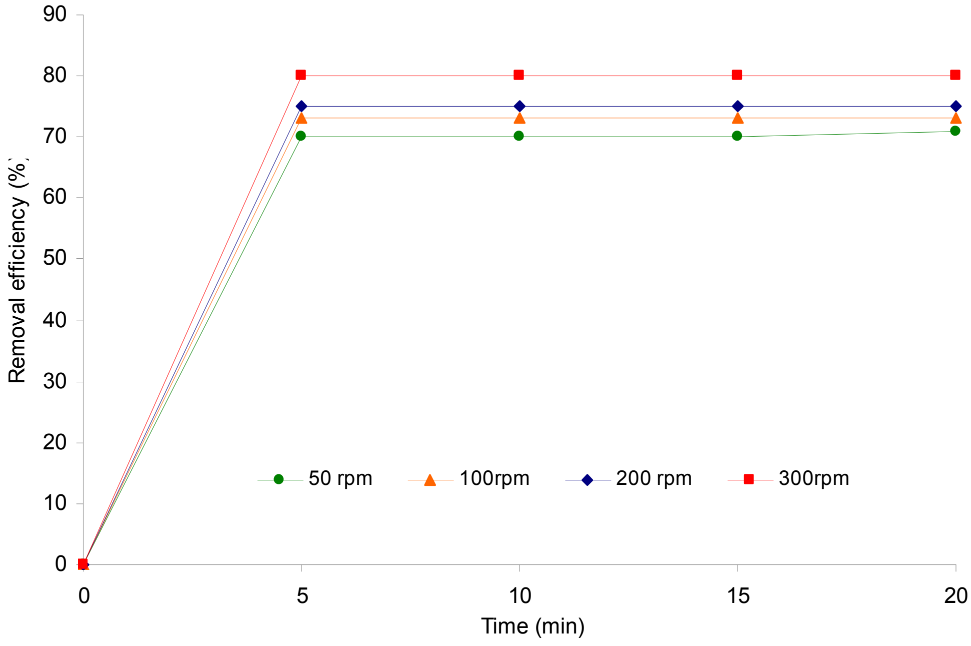

Fig 7: Effect of shaking speed on the removal of As (III) (pH=7; $Fe_3O_4$ nanoparticles dose of 0.1 g; 100 mg As/L)

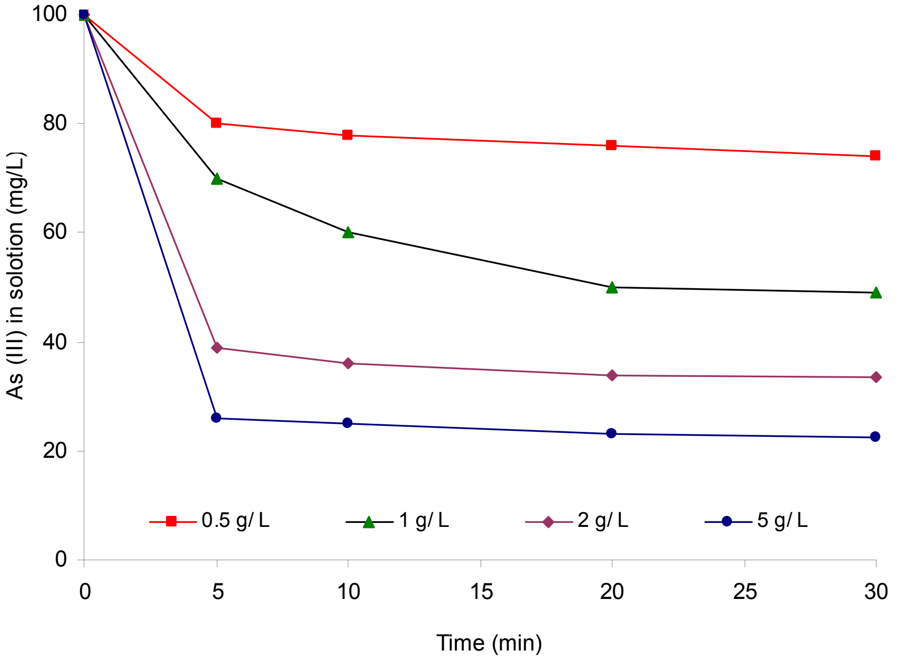

Fig 8: Effect of magnetite nanoparticles concentration on the removal of As(III) (pH=7; $Fe_3O_4$ nanoparticles dose of (0.5, 1, 2, 5 g/L)

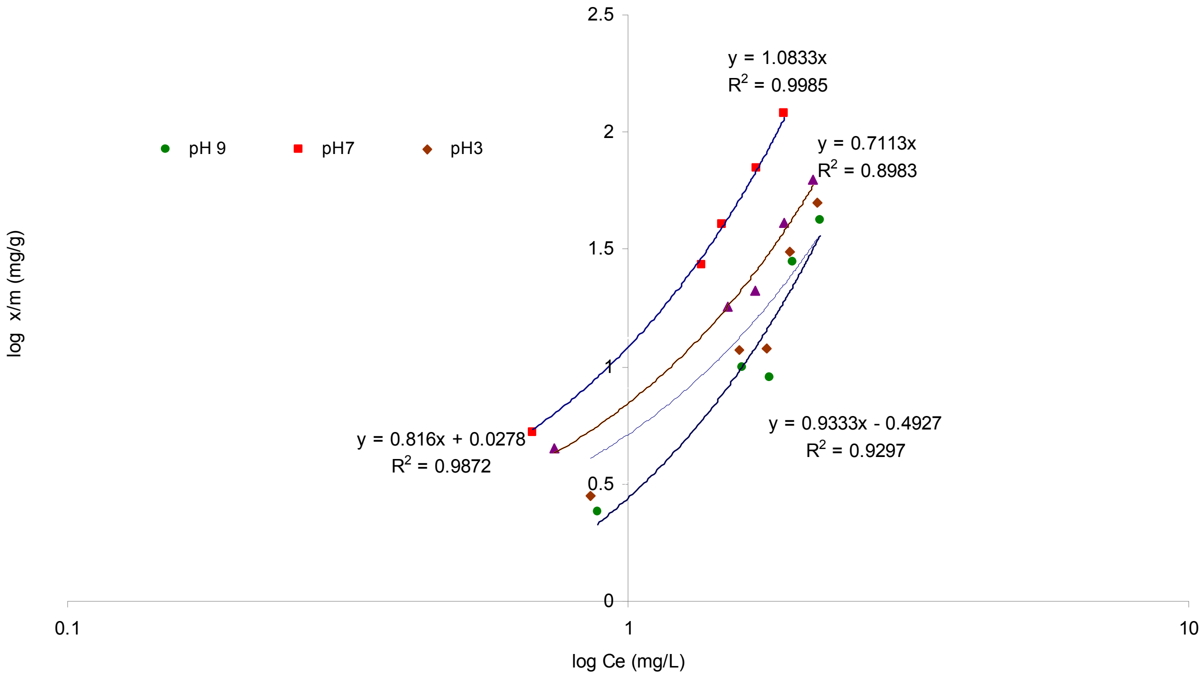

Fig 9: Freundlich isotherms at different pH [initial As(III) concentrations: 10, 50, 70, 120, and 200 mg/L; sorbent concentration=1 g/L; agitation rate=250 rpm

Table 2: The Freundlich constants at different pH

| | Freundlich constants | | |
|---|---|---|---|
| | log K | 1/n | $R^2$ |
| pH | | | |
| 3 | 0.000 | 0.711 | 0.89 |
| 5 | 0.027 | 0.816 | 0.98 |
| 7 | 0.000 | 1.083 | 0.998 |
| 9 | 0.492 | 0.933 | 0.92 |

3 to 9. The optimum pH value was 7 (Fig. 5). However, the removal efficiency of As (III) at pH of 3, 5, 7 and 9 was found to be 59%, 77%, 82, and 39 %, respectively.

The reaction occurred in a short time and at a wide range of pH. Around 70% of As (III) was adsorbed in the first five minutes, perhaps because of the adsorption characteristics of magnetite external surface. About 90% of As (III) was removed during the first minute of the reaction, while only a very small part of the additional removal occurred during the following 14 min of contact. Rapid adsorption of As (III) by magnetite nanoparticles is perhaps attributed to the external surface adsorption, which is different from the micro-porous adsorption process. Since nearly all the adsorption sites of magnetite nanoparticles exist on the exterior surface of the adsorbent (compared to porous adsorbents), it is easy for the adsorbate to access these active sites, thus resulting in a rapid approach to equilibrium (Hu, 2006). Other studies have shown that the removal efficiency of trivalent arsenic enhances when pH value increases above 7 (Katsoyiannis and Zouboulis, 2002). As pH increases, there are more $OH^-$ groups on the surface of the iron oxide. Therefore, there is increased electrostatic repulsion causing to adsorb due to its negative charge, but arsenite is neutrally charged, so it is not impacted by electrostatic repulsion. When pH< pHzpc, the surface of the iron oxide is positively charged and prefers the adsorption of a charged molecule (As V) compared to an uncharged molecule (As III). Hence at a lower pH, (such as 4) arsenate has usually higher adsorption rate compared to the arsenite (Shipley, 2007).

*Effect of initial concentration*

In the initial concentrations of 10, 50, 100, and 200 mg As/L, pH 7, shaking speed of 250 rpm, and nanoparticles dose of 5 g/L, removal efficiency of arsenic were 79%, 58%, 27%, and 16%, respectively. Therefore, As (III) removal is related inversely to initial arsenic concentrations. This was expected due to the fact that for a fixed adsorbent dosage, the total available adsorption sites are limited thus leading to a decrease in percentage removal of the adsorbate corresponding to an increased initial adsorbate concentration (Shipley, 2007).

The reason for the decrease in As(III) adsorption efficiency at higher initial concentrations may be that the adsorbent sites eventually become saturated with adsorbed arsenic and at this point further addition of arsenic to the solution would not be expected to increase the amount of adsorbed significantly coated sand (Gupta *et al.*, 2005). Also, studies by Niu showed that the arsenic removal efficiency decrease with increase of initial arsenic concentration (Shao-feng *et al.*, 2005).

*Effect of common ions*

The concentration of other ions in solution and the affinity they have for iron oxide surfaces may greatly affect the adsorption of arsenite. The simultaneous presence of common ions with As (III) invariably implies competition for available adsorption sites. Although some adsorption sites can only adsorb certain solutes and not all solutes compete for precisely the same sites, so the presence of other solutes will reduce the adsorption of any given solute to some degree. Thus, it is very necessary to investigate the competitive influence of commonly simultaneous anions and cations with As (III). In chrome-plating wastewater, the major cations are $Na^+$, $Cu^{2+}$, and $Ni^{2+}$, and the major anions are $NO_3^{-,}$ $SO_4^{2-,}$ and $Cl^-$. As shown in Fig 6, the influence of

$Na^+$ on $A_S$ (III) was rather insignificant since it did not compete for the active surface with arsenite anions at this pH. The competitive influence of $SO_4^{2-}$ on arsenite adsorption can be very small but the competition of $NO_3^-$ is significant. Therefore nitrate could play a significant role in arsenic adsorption. The nitrate ion could compete for sites on iron oxide surface which could either enhance or reduce adsorption. Literature shows that sulfate has no effect on arsenite removal with magnetite (Shipley, 2007). In addition, there was no competitive influence from these ions with As(III) with further increase ion concentration.

*Effect of shaking speed*

In the conditions including pH 7, $Fe_3O_4$ nanoparticles dose of 0.1g of 100 mg As/L solution concentration, removal of arsenic at

The results indicated that the rate of As (III) removal was controlled by the degree of shaking. The effect of increasing the shaking speed is to decrease the boundary layer and the film resistance to mass transfer surrounding the adsorbent particles (Hu, 2006).

Consequently, a reduction in the boundary layer surrounding the particle with increasing shaking speed from 50 to 300 rpm will occur. Since the system was well-mixed under higher shaking speed, the mass transfer effect became insignificant. On the other hand, the percentage removal of As (III) increased with the rising of shaking speed from 50 to 300 rpm. It can be explained by the fact that, for a relatively lower mixing rate, the system was incompletely agitated and hence the poor dispersion of nanoparticles in the solution resulted in part of surface area of adsorbent being exposed and adsorbing As (III) ions.

*Effect of magnetite nanoparticles concentration*

The effect of the magnetite nanoparticles concentration on arsenite adsorption was examined (Fig 8). The amount of arsenite adsorbed to the magnetite nanoparticles increased with time and the adsorbent dose. As the magnetite concentration increased the amount of arsenic adsorption increased as well; this result is due to the increase in surface sites for adsorption. The result of a study shows arsenite adsorption efficiency increases very rapidly with an increase in adsorbent dose. The increase in the efficiency of removal may be attributed to the fact that with an increase in the adsorbent dose, more adsorbent surface is available for the solute to be adsorbed (Gupta *et al.*, 2005).

*Adsorption isotherms*

In order to model the adsorption behavior and calculate the adsorption capacity magnetite, adsorption isotherms were investigated at different pHs of 3, 5, 7, and 9, and at 20 °C by varying the initial concentration of As(III) from 10 to 200 mg/L.

As revealed in Fig. 9, As(III) adsorption clearly correlated well with the Freundlich equation:

$$\text{Log } q_e = \log K + 1/n \times \log C_e \qquad (1)$$

Where $q_e$ is the amount of arsenite species adsorbed at equilibrium in mg/g, $C_e$ is the solute equilibrium concentration in mg/L, and $K$ and $1/n$ are Freundlich constants related to adsorption capacity and intensity of adsorption, respectively. The values of $K$ and $n$ at different pHs are shown in Table 2. For given values of initial concentration and pH, the adsorption capacity decreased with the increases in pH, indicating that lower pH was favored by the adsorption. In addition, it was established that pH and the Freundlich parameter, log $K$, have a good linear relationship ($R^2 = 0.99$). The dependence of $K$ and $n$ can be explained from the perspective of surface chemistry in aqueous phase. The surfaces of metal oxides are usually covered with hydroxyl groups that vary in forms at different pH. The removal of As (III) at lower pH is mostly due to the adsorption of $HCrO_4^-$, which is predictable to be adsorbed in larger quantities than $CrO_4^-$ under the same adsorption attraction. When $CrO^{2-}{}_4$ concentration is much higher than $HCrO_4^-$ at higher pH, the adsorption free energy of $CrO_4^{2-}$ is lower, and only under such a situation $CrO_4^{2-}$ adsorption can be better than $HCrO_4^-$(Hu *et al.*, 2005a).

The result of this study indicated that, the magnetite nanoparticles could be applied effectively in the removal of trivalent arsenic from simulated electroplating wastewater due to higher surface area and reactive hydroxyl surface sites. The removal efficiency of As(III) depends on pH and the optimal adsorption occurred at pH 7. The adsorption data fitted well with the Freundlich isotherm equation. The competitive studies showed that the effect of coexisting ions ($Na^+$, $Cu^{2+}$, $Ni^{2+}$, $SO_4^{2-,}$ $NO^{3-}$, and $Cl^-$) were insignificant except nitrate ion. It was observed that the increasing of magnetite nanoparticles concentration could be have positive effect on As(III) removal. Therefore, it could be concluded that the magnetic particles presents a good potential for treatment of arsenic wastewater. However, further research should improve the sorption capacity of magnetic particles to be applied for continuous removal of heavy metal in large-scale.

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