Titania-Coated Magnetite Particles for as(V) Removal from Water

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Abstract

Surface-modified magnetite (Fe_3O_4) particles are being proposed as adsorbents for arsenic (As) contaminated water due to its exploitable magnetic properties. In this study, magnetite particles were synthesised using co-precipitation method at various stirring times to obtain particles of different sizes. The particles were subsequently coated with titanium dioxide. Particle size analyser measurements indicated increase in the particles size with the increase in stirring time from 30 to 60 mins. Based on the X-ray diffractometer characterisation results, the obtained peaks were ascribed to that of magnetite. Titanium dioxide peaks were clearly evident for the titania-coated particles. The As(V) removal performance of the particles was tested using the low-cost molybdenum blue-based colorimeter method assisted by a UV-Vis spectroscopy. Prior to the testing, a calibration curve was obtained using As(V) sample solutions with different concentrations which depicted a linear relationship between the peak absorbance values and the concentration. Results indicated that all the titania-coated magnetite particles were able to remove 100% of As(V) in the tested solution for a contact time of 4 hours. The high affinity of the outer-titania shell towards As (V) ions may be beneficial to obtain an efficient adsorbent material for the removal of toxic As ions from water.

Keywords: *As(V) Adsorption; Titania-Coated Magnetite Particles; Molybdenum-Blue Colorimeter Method; Stirring Time; UV-Vis characterisation.*

1. Introduction

Water pollution is becoming increasingly difficult to curb. One of the major sources of water pollution is industrial wastewater which contains heavy metal ions such as nickel (Ni), lead (Pb), mercury (Hg), cadmium (Cd), and arsenic (As). These heavy metal ions are highly toxic and if ingested by living organisms, can lead to chronic illnesses [1]. The discharge of these heavy metal contaminated wastewater into the water sources with concentrations beyond the pollution standards will bring harmful effects towards mankind [2].

The most common source of heavy metal contamination of water is the purification process of metals, in addition to ore smelting, nuclear fuel preparations and mining. Contamination occurs via ion exchange or compound precipitation process into muds and soils. Inorganic heavy metal ions do not degrade like organic compounds, hence requiring additional removal techniques to isolate them [3]. Hence, it is extremely vital to ensure remediation of wastewater, as it has become a great concern worldwide for these past few decades.

Even at very minute concentrations, As is extremely toxic. The presence of As at high doses is not accompanied by any variation in odour, taste and visibility, thus making it hard to detect in water [4]. In 1993, the World Health Organization (WHO) lowered the maximum concentration limit (MCL) of As in drinking water from 50 μg/L to 10 μg/L [5].

Up to now, there are various methods and processes used to remediate contaminated water. Some of these techniques include membrane separation, reverse osmosis, ion exchange, evaporation, solvent extraction, and precipitation [3]. These techniques has certain disadvantages such as: 1) high operational costs for treatment and remediation of the waste metal sludge [6-8], 2) high complexity, and 3) low efficiency. Therefore, various studies are being carried out to come up with cost-efficient materials via effective remediation techniques such as adsorption. Magnetite (Fe_3O_4) particles have gained much attention especially in the field of medical care and magnetic sensing [9–11]. For the application of wastewater remediation, the magnetic particles which have adsorbed heavy metal ions can be separated from water by application of an external magnetic field. Many techniques have been developed to synthesise magnetite particles [12–15]. Thus, a non-toxic, scalable, economical, and efficient synthesis method of magnetite particles would be highly recommended for its many applications and research purposes [16–18]. Moreover, the surface of these magnetite particles can be modified by inorganic materials, polymers, or novel molecules to enhance surface adsorption [19–23].

In this study, we studied arsenate (As(V)) removal from water using titania (TiO_2)-coated Fe_3O_4 particles synthesised under different stirring times via the co-precipitation method followed by coating with TiO_2. It has been reported that the photocatalytic property of TiO_2 can potentially enhance the removal of arsenate (As(V)) from water [24]. In addition, it is an eco-friendly agent which will not cause additional harm [24]. The TiO_2-coated Fe_3O_4 particles were characterised by several tools such as X-ray diffractometer (XRD), particle size analyser (PSA) and also ultraviolet-visible spectrophotometry (UV-Vis). The arsenic removal performance of the synthesised particles was tested using the low-cost molybdenum blue-based colorimeter method. This testing method was chosen for As(V) removal as it is easy, relatively straightforward and does not require trained experts.

2. Experimental Section

The chemicals are of analytical grade and used without further purification. Ferric chloride hexahydrate ($FeCl_3.6H_2O$, 99%), ferrous chloride tetrahydrate ($FeCl_2.4H_2O$, ≥99%), ascorbic acid ($C_6H_8O_6$) and sulphuric acid (H_2SO_4, 95-97%) were obtained from Merck (USA); sodium hydroxide (NaOH, 99%), hydrochloric acid (HCL, 32%), titanium isoperoxide (TTIP) and ammonia solution ($NH_3.H_2O$, 28-30%) were obtained from Sigma-Aldrich (USA); sodium arsenate heptahydrate ($NaH_2AsO_4.7H_2O$) was obtained from Fisher (USA); ammonium molybdate ((NH_4)$_6Mo_7O_{24}.4H_2O$) and antimony potassium tartrate ($C_8H_{10}K_2O_{15}Sb_2$) were purchased from R&M Chemical (Malaysia).

2.1. Synthesis of Titania-Coated Magnetite Particles

The magnetite particles were synthesised via the co-precipitation of Fe^{3+} and Fe^{2+} ions at a molar ratio of 2:1, according to the method described by Kim et al. [25] with minor modifications. Sodium hydroxide was used as the base in this experiment. The iron source was prepared by dissolving 8.649g of ferric chloride hexahydrate (1.28M) and 3.181g of ferrous chloride tetrahydrate (0.64M) into 1m*l* of hydrochloric acid (0.4M) which was diluted to 25m*l* with deionised (DI) water. The base solution was prepared by dissolving 15g of sodium hydroxide beads into 250m*l* DI water to obtain a solution with concentration of 1.5M. The base solution in a three-necked vessel was de-gassed using nitrogen gas (N_2) for 10 minutes. This was done to avoid the oxidation of the iron source. After that, the iron source was added dropwise into the reaction vessel under varying stirring times (30, 60 and 90 minutes) at a reaction temperature of 25°C and stirring rate of 1000 rpm. At the end of the synthesis process, the obtained black precipitates was washed with DI water and centrifuged at 4000 rpm for 15 minutes. The washing and centrifuging steps was repeated twice and finally, the black precipitates were dried in an oven at 70°C overnight.To coat the magnetite particles, 3.125m*l* of TTIP was first mixed with 10m*l* of isopropanol (IPA). 1.00 gram of magnetite particles obtained as described above was then added into the prepared solution. The solution was then placed in an ultrasonication bath for 20 minutes at room temperature followed by heating (without ultrasonication) at a range of 45°C – 50°C for 1 hour to evaporate the solvent. Subsequently, the mixture was transferred into an alumina crucible and calcined in a furnace oven at 360°C for 3 hours [26].

2.2. Characterisation Tools and Procedure

To measure the average size of the synthesised particles, Malvern Zetasizer Nano S was used. Firstly, 0.1g of each sample of particles was ultrasonicated with 10m*l* of IPA for 10 minutes. After the sonication process, an aliquot of the colloidal dispersion was extracted into cuvettes for the analysis. The XRD characterisation was performed in order to identify the phase of the synthesised particles using a Bruker D8 Advance XRD with 2θ angle of 10-80° and Cu-Kα radiation (λ = 1.5406 Å). The particles were pressed into the middle of the sample holder before being submitted for XRD characterisation. The double-beam Halo DB-20 UV-Vis was utilised to test the As(V) removal performance, the procedure of which is as described in the next section.

2.3. Colorimetric As (V) Removal Testing Procedure

To test for the As(V) removal performance, the low-cost molybdenum blue-based colorimeter method was used. This method is based on the reaction between reduced molybdate in the colouring solution with As(V) to form a blue complex [27]. The colouring agent components are first prepared by dissolving the following into 10m*l* DI water respectively: 1.08g ascorbic acid, 0.3 g ammonium molybdate and 0.056g antimony potassium tartrate. 1.44 m*l*

sulfuric acid was also diluted to 10 ml with DI water. Subsequently, 5 ml of the sulphuric acid solution was mixed with 2 ml ascorbic acid, 2 ml ammonium molybdate and 1 ml antimony potassium tartrate solutions to obtain the colouring solution. 50 mg/L As(V) stock solution was then prepared by dissolving 41.6 mg of sodium arsenate heptahydrate in 200 ml DI water. Then, 10ml of As(V) solution with various concentrations (0, 2mg/L, 4mg/L, 6mg/L, 8mg/L and 10mg/L) was prepared by diluting the stock solution with stoichiometric amounts of DI water. All the As(V) sample solutions were then acidified with 0.25 ml 1% HCL [27] followed by the addition of 1.25 ml colouring agent and 1 ml DI water. A blank solution was prepared likewise, with DI water being used in place of As(V) solution. After 15 minutes, the absorbance peak for each sample solution was obtained using UV-Vis and a calibration curve of absorbance vs. concentration of the sample solutions was plotted. The line equation of the calibration curve will be used as a reference to obtain the final concentration of arsenic in the samples tested using the synthesised particles as adsorbents.

The testing was performed by diluting the As(V) stock solution with DI water to obtain 50 m*l* As(V) solution with initial concentration of 20 mg/L. 0.5g of the synthesised magnetite particles was added to the As(V) solution and the mixture was ultrasonicated for 4 hours at room temperature. After every 1 hour interval of sonication, 10 ml of the As (V) solution was extracted followed by centrifugation at 11,000 rpm for 1 hour in order to sediment the magnetite particles from the As(V) solution. Next, the addition of the colouring solution, HCl and DI water as described above was performed to the supernatant obtained after the centrifugation process. After 15 minutes, the aliquot was tested via the UV-Vis to obtain its peak absorbance value. The final concentration (mg/L) of As(V) is thus obtained from the line equation of the calibration curve, from which the final mass, M_f, of As is calculated and the adsorption capacity, q (mg/g), is obtained from the equation below:

$$q = \frac{M_i - M_f}{m} \qquad (1)$$

where M_i and M_f is the initial and final masses (in mg) of As(V), respectively and m is the mass (g) of the magnetite particles used.

3. Results and Discussion

3.1. Size and Phase Characterisation Results

The XRD patterns of the particles are as shown in Fig. 1. The diffraction peaks corresponding to (220), (311), (400), (422), (511), (440) and (533) planes (marked with inverted triangles in Fig. 1) are ascribed to magnetite for the Fe_3O_4 samples synthesised for 30 minutes (Fig. 1(a)) and 60 minutes (Fig. 1(b)). All the observed diffraction peaks could be indexed by the cubic structure of Fe_3O_4 with JPCDS no. 19-0629. The peaks are broad and this indicates the amorphous phase of the synthesised particles [28]. This is in contrast to bigger and well crystallised samples which would exhibit narrower peaks with higher intensity. Characteristic peaks of TiO_2 (marked with filled circles in Fig. 1) are evident from the XRD profiles of the titania-coated Fe_3O_4 samples synthesised for 30 minutes (Fig. 1(c)) and 60 minutes (Fig. 1(d)). For these two samples, the crystalline phase of TiO_2 is identified as anatase, as was also obtained by Li et al. for their TiO_2/Fe_3O_4 nanocomposite [29]. The rutile phase of TiO_2 was not observed for our samples as the calcination temperature was at 360°C, which is much lower than the temperature of 700-900°C needed for the rutile phase to manifest [30]. According to Raj *et al.*, calcination temperatures of less than 500°C will not show significant rutilation of TiO_2 [30]. As a summary, the XRD results show that Fe_3O_4 and titania-coated Fe_3O_4 particles were successfully synthesised using the procedure outlined in the experimental section.

The average sizes of the TiO$_2$-coated Fe$_3$O$_4$ particles are as summarised in Table 1. Their sizes were measured to be between 1251-2203 nm. The size and polydispersity index (PDI) value of the 90 minutes sample was lower than the 60 minutes sample, which may lead us to infer that the latter was more agglomerated than the former. The reduction in the size and PDI value for the 90 minutes samples may be attributed to the reduction in radius difference of the particles at prolonged stirring times [31]. The overall mid-range PDI) values indicate that particle samples are polydisperse [32].
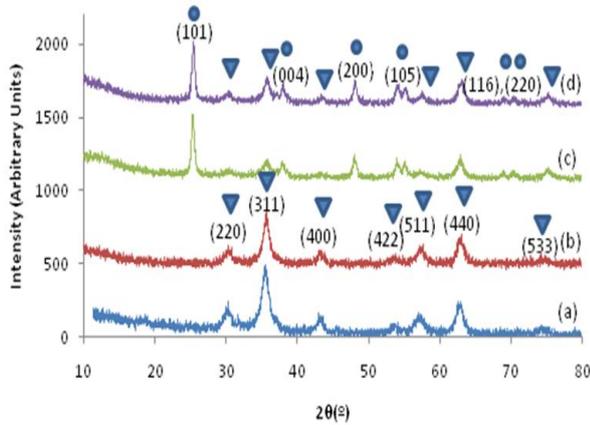

Fig. 1: XRD profiles obtained for Fe$_3$O$_4$ samples synthesised for (a) 30 minutes and (b) 60 minutes; TiO$_2$-coated Fe$_3$O$_4$ samples synthesised for (c) 30 minutes and (d) 60 minutes.

Table 1: PSA Measurement Results of the Titania-Coated Magnetite Particles

Stirring time (mins)	PSA measurement results	
	Average size (nm)	Polydispersity index
30	1251	0.330
60	2203	0.451
90	2073	0.316

3.2. Calibration Curve and As (V) Adsorption Results

Results from the UV-Vis measurements showed that all 6 of the sample solutions displayed peak absorbance values at a wavelength of λ = 900 nm. In addition, it was also observed that the peak absorbance values increased with the increase in the concentration of the sample solutions, hence depicting a linear relationship [33]. The peak absorbance values for each sample was used to plot the calibration curve as shown in Fig. 2. The linear equation (as shown in equation 2) to approximate the data points in the graph below has a coefficient of determination, R-squared (R^2) value of 0.99. This is an indication that it is a relatively good approximation of the data points. This equation will hence be used to calculate the final concentration of the As(V) solution which has been reacted with the synthesised particles. Subsequently, the adsorption capacity of each particle sample can be determined.
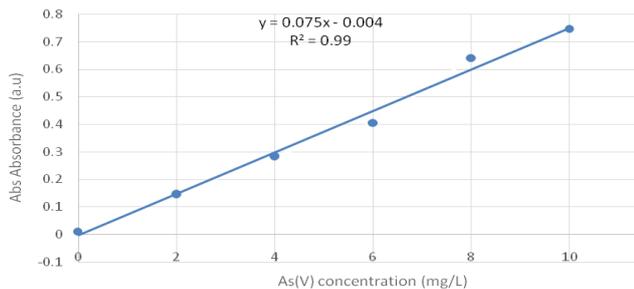

Fig. 2: Calibration curve of the As(V) sample solutions.

$$y = 0.075x - 0.004 \qquad (2)$$

Fig. 3 depicts the adsorption capacities of the titania-magnetite samples synthesised at the different stirring times of 30, 60 and 90 minutes. After 1-hour of sonication, the adsorption capacities were 1.954 mg/g, 1.933 mg/g and 1.945 mg/g respectively for the 3 samples, meaning 1 gram of the particle samples were able to remove at least 1.933 mg of As(V) from the 20 mg/L solution. The adsorption capacity continued to increase to approximately 2 mg/g for all 3 samples after 4-hours of sonication. The adsorption capabilities of the samples may be best understood by calculating the removal percentage of As(V) by finding the remaining mass of As(V) in the solution (M_i-M_f) and dividing it with M_i. The obtained removal percentages are as shown in Fig. 4 and also detailed in Table 2. Both the adsorption capacity values and removal percentages of each sample according to sonication time is as shown in Table 2. All 3 samples were able to remove 100% of the As(V) in the 20 mg/L As(V) after 4-hours of sonication. There was not much difference in the performance of the titania-coated samples synthesised at 60 and 90 minutes and this may be attributed to the similar average sizes. The removal percentage of the sample stirred for 30 minutes was only slightly better than the other 2, presumably due to its smaller average size. Conclusively, the performance of these samples was very close to each other's although smaller particles should be able to adsorb more particles due to its higher surface area [34]. Hence, the removal performance resemblance may indicate that the particles were agglomerated and perhaps similar-sized clusters were present in each sample. To draw correlation between the size and adsorption capacity of the particle samples, more different-sized particles would need to be synthesised with distinct size distribution and average sizes. This can be achieved by varying the stirring rate to obtain smaller-sized particles [31]. On the other hand, by increasing the synthesis temperature, bigger sized particles can be acquired [35].
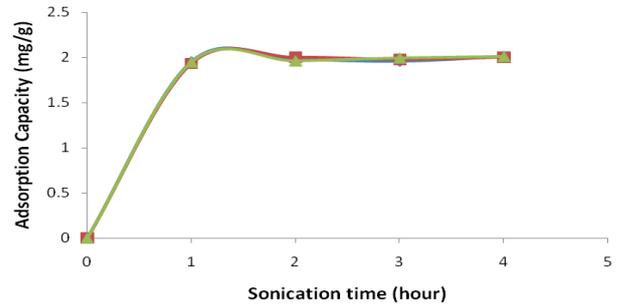

Fig. 3: Adsorption capacities for TiO$_2$-Fe$_3$O$_4$ samples stirred for 30, 60 and 90 minutes.
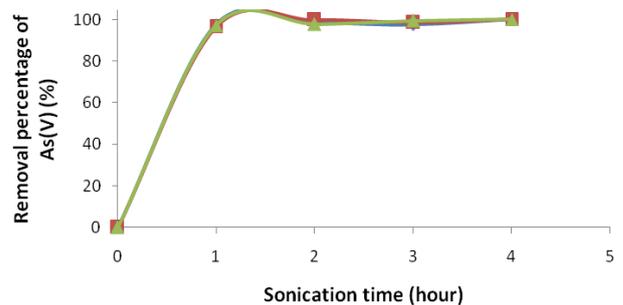

Fig. 4: Removal percentages for TiO$_2$-Fe$_3$O$_4$ samples stirred for 30, 60 and 90 minutes.

Manna et al. [36] reported the usage crystalline hydrous titania as adsorbent material for arsenite, As(III), which is more toxic and mobile than its oxidised counterpart, As(V) [37]. According to Guan et al. [38], titania removes As(III) ions by oxidation to As(V), which is easier to remove compared to the former. Hence, titania is a suitable coating material while magnetite can be the core due to its outstanding magnetic properties. Furthermore, magnetite is non-toxic and is used in biomedical field for applications such as magnetic resonance imaging (MRI), hyperthermia,

and site-specific drug delivery. By retaining magnetite as the core, the coated particles may be isolated from the water source using an external magnetic field after adsorption of the targeted heavy metal ions. Alternatively, the particles may be separated from the aqueous media through sedimentation or centrifugation. Based on the results of our experiment, the TiO_2-Fe_3O_4 particles had satisfactory removal performance for As(V) ions and may be further tested for As(III) under different experimental parameters study.

Table 2: As (V) Adsorption Capacity and Removal Percentage of the Titania-Coated Magnetite Particles

Sample stirring time (mins)	Sonication time (hours)	As(V) adsorption results	
		q (mg/g)	Removal percentage (%)
30	1	1.954	97.70
	2	1.976	98.79
	3	1.958	97.89
	4	≈2	≈100
60	1	1.933	96.64
	2	≈2	≈100
	3	1.977	98.87
	4	≈2	≈100
90	1	1.945	97.27
	2	1.958	97.99
	3	1.991	99.55
	4	≈2	≈100

4. Conclusion

Fe_3O_4 particles were synthesised via the co-precipitation method followed by coating with TiO_2. The synthesised particles were analysed using XRD and PSA while the arsenic removal capability was determined through the molybdenum-blue colorimetric technique. The characterisation results indicate that TiO_2-coated Fe_3O_4 magnetite was successfully synthesised as the characteristics peak of magnetite and titania was displayed by the coated samples. Via the PSA measurement, particles sizes were determined to be between 1251-2203 nm, possibly due to the growth at higher stirring times and also agglomeration. Through the UV-Vis measurement of absorbance of the As(V) sample solutions, a linear trend of increased absorbance values with increased sample concentration was attained. Hence, a linear equation with good linearity was used to approximate the final concentration of the As(V) solution sonicated with the synthesised particles. All the 3 tested samples were able to adsorb 100% of the As(V) ions in the 20 mg/L solution after 4 hours of sonication. Thus, titania-coated magnetite is a suitable candidate for the removal of As(V) ions through adsorption onto its surface.

Acknowledgement

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