A method for preparing ferric activated carbon composites adsorbents to remove arsenic from drinking water

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Abstract

Iron oxide/activated carbon (FeO/AC) composite adsorbent material, which was used to modify the coal-based activated carbon (AC) 12×40, was prepared by the special ferric oxide microcrystal in this study. This composite can be used as the adsorbent to remove arsenic from drinking water, and Langmuir isotherm adsorption equation well describes the experimental adsorption isotherms. Then, the arsenic desorption can subsequently be separated from the medium by using a 1% aqueous NaOH solution. The apparent characters and physical chemistry performances of FeO/AC composite were investigated by X-ray diffraction (XRD), nitrogen adsorption, scanning electron microscopy (SEM) and X-ray photoelectron spectroscopy (XPS). Batch and column adsorption experiments were carried out to investigate and compare the arsenic removal capability of the prepared FeO/AC composite material and virgin activated carbon. It can be concluded that: (1) the main phase present in this composite are magnetite (Fe_3O_4), maghemite (γ-Fe_2O_3), hematite (α-Fe_2O_3) and goethite (α-FeO(OH)); (2) the presence of iron oxides did not significantly affect the surface area or the pore structure of the activated carbon; (3) the comparisons between the adsorption isotherms of arsenic from aqueous solution onto the composite and virgin activated carbon showed that the FeO/AC composite behave an excellent capacity of adsorption arsenic than the virgin activated carbon; (4) column adsorption experiments with FeO/AC composite adsorbent showed that the arsenic could be removed to below 0.01 mg/L within 1250 mL empty bed volume when influent concentration was 0.5 mg/L.
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1. Introduction

It is well known that arsenic (As) is severely harmful to the human and the drinking water containing arsenic is a challenging environmental pollution problem [1–3]. Arsenic is one of endocrine disruptors and is classified as one of the most toxic and carcinogenic chemical element [4–7]. The removal of arsenic from aqueous solution has been reported by some researchers. Lee and Rosehart [8,9] found that the adjustment of the pH could alone control the removal of arsenic by the activated carbon. Evdokimov et al. [10] improved the capacity to remove arsenic by impregnating ferric hydroxide or tartaric acid into

carbon and achieved desorption using a 1% aqueous KOH solution. Eguez and Cho [11] measured the removal of As(III) and As(V) at various pH values on activated charcoal. Rajakovic [12] developed that the pretreatment activated carbon with Ag^+ or Cu^+ ions could improve the removal of As(III), but reduced that of As(V). Diamadopoupos et al. [13] found that the carbon with higher content of ash was more effective in removing As(V) from the solutions. The absorption capacities of arsenic with virgin activated carbon were rather poor, but can be improved by modifications. One modification introduced zirconyl ions into this porous material, which was suggested for the enrichment of arsenic for analytical purposes and seems to be highly efficient [14]. The activated carbon was tested for the removal of arsenic, and also tried out by a modification with an iron hydroxide phase [15,16].

It has been reported that high-level arsenite can be removed from groundwater by zero-valent iron [17]. The hydrous oxides

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of rare earth elements have a high affinity for such hazardous anions as arsenate [18,19]. Iron oxides, especially amorphous iron oxides, have also been reported to be effective for the removal of arsenic [20]. Several iron(III) oxides, such as amorphous hydrous ferric oxide (FeOOH), poorly crystalline hydrous ferric oxide (ferrihydrite) and goethite α-FeO(OH), are promising adsorptive materials to remove As(V) and As(III) from aqueous solutions [21–25]. However, most iron(III) oxides are available only as fine powders or are generated in situ as gels or suspensions in aqueous solution. These forms of iron(III) oxides retain their strong affinities to As(V) and As(III), but are limited to reactor configurations incorporating large sedimentation and filtration units, which cause difficulty in solid/liquid separation [26]. Thus, they are not suitable for column adsorption in water and waste water treatment. Furthermore, iron(III) oxide alone is not suitable as a filter medium due to its low hydraulic conductivity [27]. Activated carbon and zeolites surface are treated by FeSO$_4$ solution. Two iron treatments were investigated as well as the effects of varied pH, temperature, and ionic strength increases on adsorption effectiveness [28]. Granular activated carbon (GAC) was used primarily as a supporting medium for ferric iron that was impregnated by ferrous chloride (FeCl$_2$) treatment, followed by chemical oxidation. Surface physical chemistry changes were analyzed by the BET, SEM and X-ray diffraction between untreated GAC and modified one with iron. Column studies showed that both As(V) and As(III) could be removed to below 0.01 mg/L within 6000 empty bed volume when the groundwater containing approximately 0.05 mg/L of arsenic was treated [29]. The model of As(V) removal by an iron oxide-impregnated activated carbon (FeAC) was investigated by means of the surface complexation model (SCM). The results showed that this work will be used as a foundation theories instruction in developing a dynamic model to predict As(V) adsorption in a column experiment [30].

The purpose of this study was to develop one new method to prepare the granulated FeO/AC adsorbents, which consisted mainly of amorphous hydrous ferric oxide and activated carbon, and are suitable for the column adsorption of arsenic. In this study, it was emphasized on determining appropriate process parameters for the preparation of adsorbents, including C/Fe molar ratio, process pH value, aging time and drying temperature. Also, the arsenic removal capability of the prepared FeO/AC composite material and virgin activated carbon were investigated and compared by batch and column adsorption experiments.

2. Experiments

In this study, the activated carbon (AC) (AC12 × 40, China Calgon) was used in this study. This kind of AC has moisture content of 1.2%, ash content of 10.3%, iodine values of AC adsorption of 1029 mg/g, the hardness of 96.2%, and the density of 480 g/L. Grain sizes of AC were: less than 1.7 mm in diameter (less than 12 US mesh sieve; less than 1.8% by mass) and more than 0.425 mm in diameter (more than −40 US mesh sieve; less than 1.9% by mass). All the chemicals used in this study were of analytical reagent grade. The virgin activated car-

bon was firstly rinsed with distilled water to remove dirties, and then was washed by 0.001 M HCl solution to remove all salts precipitated in its pores. Then, the AC was repeatedly washed with the distilled water to remove all traces of the acid. Subsequently, the washed AC was oven-dried at 85 °C for 24 h to volatilize the organic impurities, and then was modified by 1 N HNO$_3$ for 5 h at the room temperature.

FeO/AC adsorbent was prepared by mixing FeCl$_3$/FeSO$_4$ (molar ratio 2:1) and 5 mol NaOH and remaining for 10 min at the temperature of 70 °C and pH value of 9.5, along with the gentle stirring (60 rpm), and then was impregnated into the modified AC. The obtained materials were dried in an oven at 100 °C for 3 h. The samples were analyzed by X-ray diffraction (XRD) (Ni filtered Cu Kα, λ = 1.5418 nm), X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM) and nitrogen adsorption measurements.

Batch adsorption experiments were performed. A flow-chart of the batch experiments (adsorption–desorption) is shown in Fig. 1. First, the different weights of Na$_3$AsO$_4$·12H$_2$O were separately added into conical flasks and the volume was made up to 100 mL with tap water. After adjusting the values of pH by HCl and NaOH solution, flasks were continuously shaken for 10 min. Each flask contained a known concentration of the arsenic. A known amount of adsorbent (normally 10 g/L) was then added into each flask. Afterwards, the flasks were shaken continuously at 120 rpm and 25 °C for 10 h on a model 2500 platform shaker, which was manufactured by Suzhou Electronic Co. Inc. Then, the agitation time was set to approximately 10 h. The solution was filtered with a 0.45 μ-membrane filter for analysis. The values of pH were measured with a pH meter (model PHS-3C, Shanghai, China), and As(V) was measured by atom adsorption fluorescent photometer (model 180-80, hydride equipment appurtenance model 508-0182, Japan). The
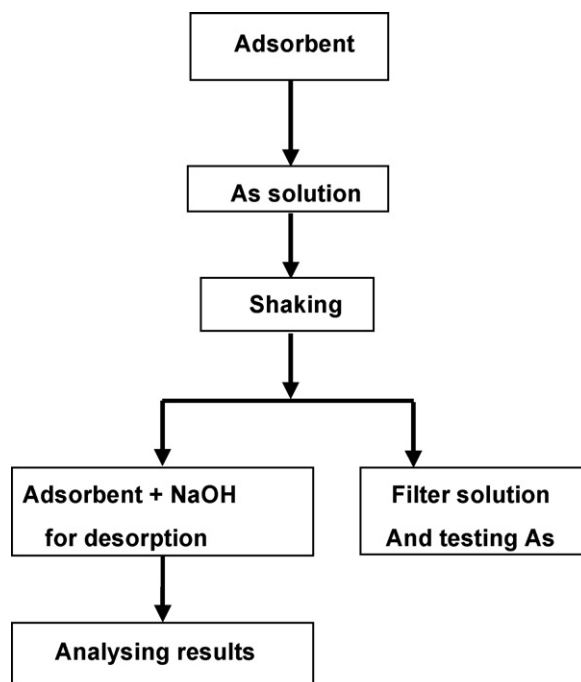

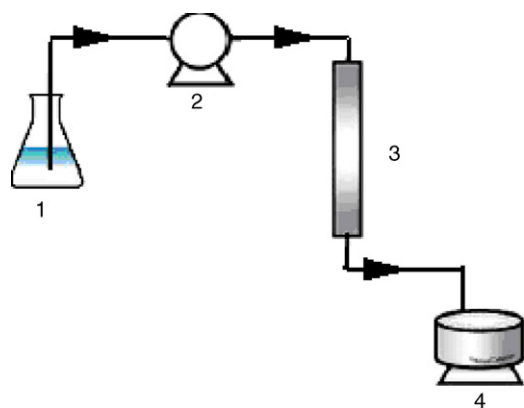Fig. 1. A flow chart of the batch experiments (adsorption–desorption).

Fig. 2. A flow chart of column experiments.

adsorption isotherms were obtained from the batch equilibrium experiments.

Experiments were carried out to investigate the dependence of desorption characteristic of As from solid adsorption residues on pH. A 200 mL solution with desired pH was combined with 20 g of adsorption residue in a 250 mL flask. The mixtures were agitated at 25 rpm for 12 h. All of the experiments were carried out at 25 °C. The values of pH of the solution used in desorption experiments were adjusted with the mixture of hydrochloric acid and sodium hydroxide.

Column adsorption experiments were carried out using laboratory-scale adsorption columns. A process flow chart is shown in Fig. 2. These columns are made by the graduated Perspex with inner diameter of 40 mm and height of 260 mm. The Perspex column was equipped with a bottom filtration device to prevent the escape of fine adsorbents during processing. The influent solution was prepared by the dissolution of $Na_3AsO_4 \cdot 12H_2O$ using tap water. The prepared influent solution was passed downward through the column at a fixed flow rate. A total of about 3.0 L of influent solution was used in each column study. The downstream effluent was collected at different fractions with each fraction measuring about 100 mL. The flow rate of the solution was kept at 500 mL/h, samples of solution were taken from each fraction of the effluent and were analyzed using atom adsorption fluorescent photometer.

3. Results and discussion

3.1. Characterization of the composites

The composite was prepared by the precipitation of iron oxide or hydroxide onto a carbon surface. Then, the content of Fe, BET surface area, iodine no., pH_{ZPC} and porous volumes of the prepared composites can be measured, as shown in Table 1. It can be observed that FeO/AC has a higher content of Fe oxide, and the impregnation increases the content of iron to 8%. The pH_{ZPC} did not change with the impregnation of iron oxide or hydroxide, while the N_2–BET surface area, pore volume, and iodine no. all decreased.

XRD analysis was carried out on FeO/AC composite adsorption and virgin activated carbon, and the analysis results were shown in Figs. 3 and 4. XRD analysis indicated that the pres-

Table 1
Physical property of FeO/AC integer scan analysis for FeO/AC composite material and activated carbon 12×40

Type	Unit	AC12 × 40	FeO/AC
Ash content	wt%	10.3	8
Iodine no.	mg/g	1000	800
Fe-content	% (w/w)	0.51	8.0
pH_{ZPC}		7.35	7.53
BET-surface area	m²/g	1026	807
Macro pore volume[a]	cc/g	0.255	0.178
Meso pore volume[b]	cc/g	0.182	0.146
Micro pore volume[c]	cc/g	0.356	0.282
Total pore volume	cc/g	0.793	0.606

[a] Pore diameter >50 nm.
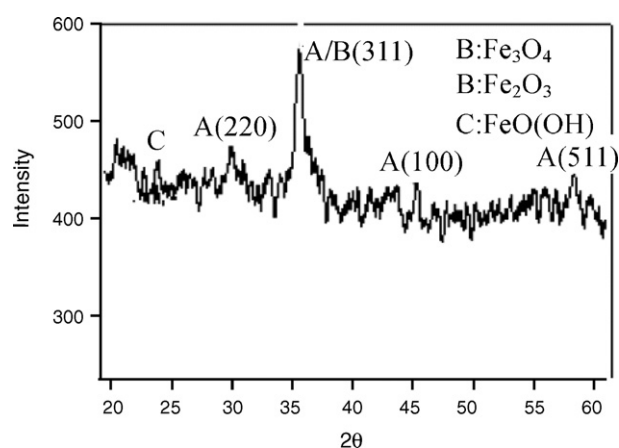[b] Pore diameter between 2 and 50 nm.
[c] Pore diameter <2 nm.

Fig. 3. Powder XRD for FeO/AC composite adsorption.

ence of a cubic iron oxide phase, which may be related to the presence of the maghemite (γ-Fe_2O_3) and magnetite (Fe_3O_4). For the broader peaks, a smaller crystallite size (5–15 nm for the composite) is determined by Scherrer's equation. Weak diffraction peaks observed may be related to the presence of small amounts of goethite (α-FeO(OH)) and hematite(α-Fe_2O_3). For the virgin activated carbon, there is no appearance of crystallites on the surface, as shown in Fig. 4.
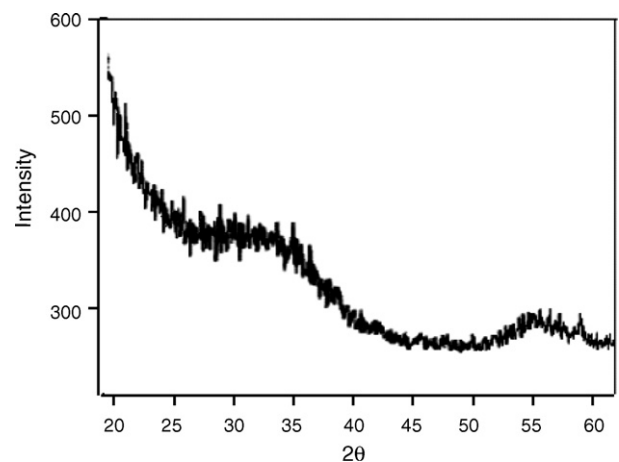

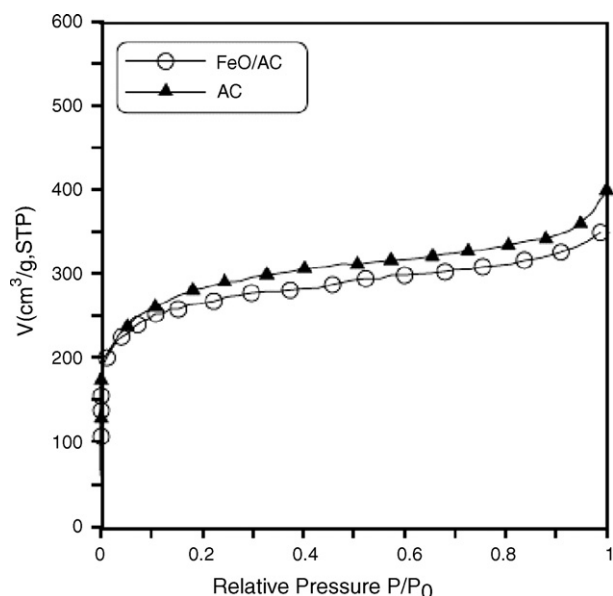Fig. 4. X-ray diffraction graph of virgin activated carbon.

Fig. 5. Nitrogen adsorption isotherms on FeO/AC and AC.

Fig. 5 shows N_2 adsorption isotherms of FeO/AC composite and virgin activated carbon. The physical properties shown in Table 1 suggest that the BET surface area and microporous volume are not significantly affected by the presence of iron oxide in the composites. For example, the surface area of virgin activated carbon is 901.1 m^2/g, and that of FeO/AC is 630.3 m^2/g; the total pore volume of virgin activated carbon is 0.793 cc/g, and that of FeO/AC is 0.606 cc/g. As iron oxide has a relatively small surface area and microporous volume, which decreases the surface area and microporous volume of the composites, compared to virgin activated carbon.

Surface images of the FeO/AC adsorbent were analyzed by scanning electron microscopy (XL Series: XL30ESEM, Holland), as shown in Fig. 6(a)–(d). Fig. 6(a) shows SEM macrographs of the rough areas of the virgin activated carbon at 1000× magnification. It can be found that there are a few macropores, and the rough surface micrographs show a distinct roughness with scraggy patterns. Fig. 6(b) shows SEM micrographs of the surface areas of FeO/AC (1000× magnification). Obviously, the activated carbon surfaces are covered with fine and uniform iron oxide particles. Fig. 6(c) shows SEM micrographs of the surface areas from another side surface of FeO/AC (1000× magnification). Fig. 6(d) shows a magnified part of Fig. 6(c) at 4000×, small crystallitic covered on activated carbon.

The XPS technique provides valuable information on the location and distribution of the active metal in the pore structure based on the carbon. Figs. 7 and 8 show XPS integer scan analysis of activated carbon AC12 × 40 and XPS analysis on C1s apices and disperse apices of activated carbon AC12 × 40, respectively. It indicates that there are five functional groups. Figs. 9 and 10 show XPS integer scan analysis of FeO/AC and XPS analysis on C1s apices and disperse apices of FeO/AC, respectively. It was found that the carbons treated by acids improved the distributions of iron oxide in porous carbon, which leads to more iron oxide diffused into the inner pores. The main Fe 2p peak indicates the goethite (α-FeO(OH)) and other Fe III species, such as Fe_2O_3.

3.2. Batch adsorption experiments

3.2.1. The adsorption equilibrium

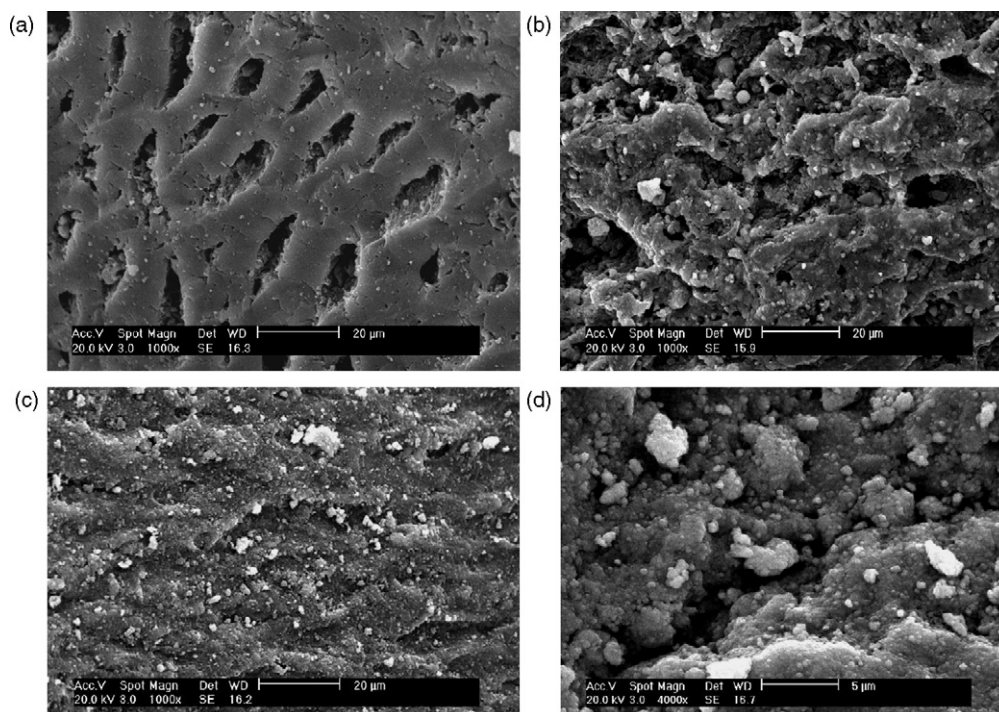The removal of arsenic from aqueous solutions by the activated carbon 12 × 40 and FeO/AC composite was studied. The

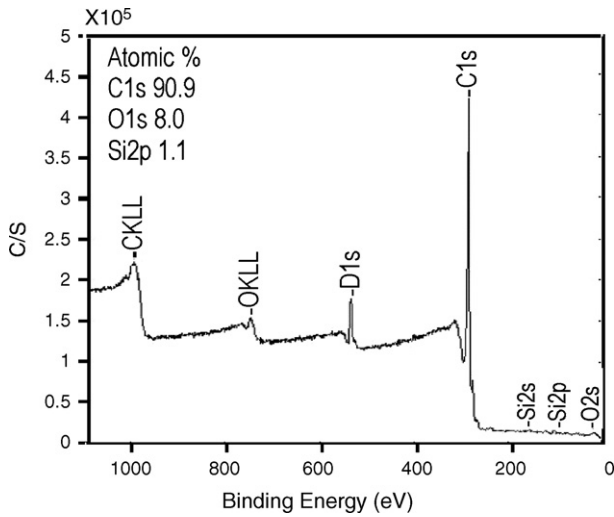Fig. 6. SEM micrographs of (a) (b) FeO/AC composite, (c) (d) virgin activated carbon12 × 40.

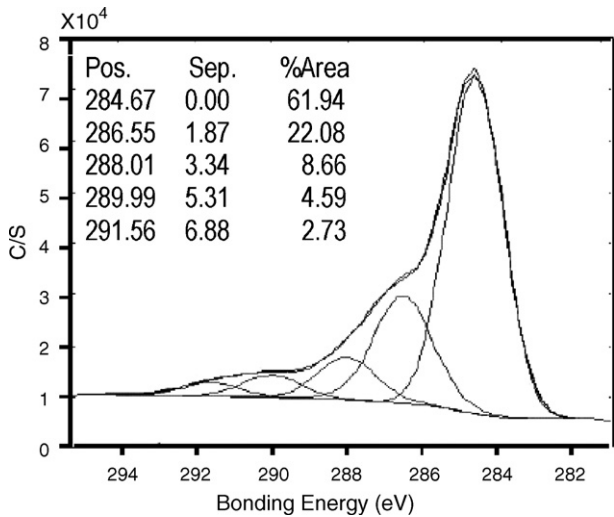Fig. 7. XPS integer scan analysis of activated carbon AC12 × 40.

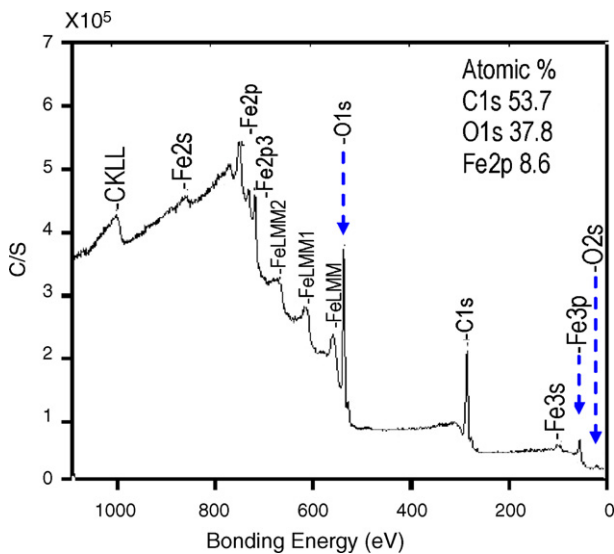Fig. 8. XPS analysis on C1s apices and disperse apices of activated carbon AC12 × 40.

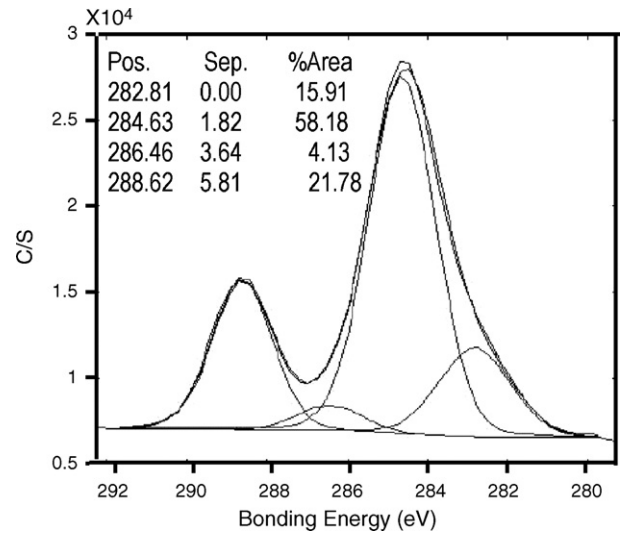Fig. 9. XPS integer scan analysis is for FeO/AC composite material.

Fig. 10. XPS analysis on C1s apices and disperse apices for FeO/AC composite.

isothermal adsorption experiments were carried out, following the same procedure, temperature, and pH value (pH = 5.0). The adsorption equilibrium concentrations were tested by atom adsorption fluorescent photometer. The adsorption capabilities can be calculated by the following equation:

$$q_e = \frac{x}{W} = \frac{V(C_0 - C_e)}{W} \tag{1}$$

where x is solute-absorbed capacity of adsorbents (mmol), V is volume of aqueous solution (l), C_0 and C_e are original and equilibrium concentration (mmol/L), respectively. W is the mass of adsorbents used (g), and q_e is the adsorption capability (solute-absorbed capacity of unit adsorbents, mmol/g).

Fig. 11 shows the relationship between the adsorption capability q_e and equilibrium concentration C_e. It is shown that the arsenic removal capability of the composite material FeO/AC
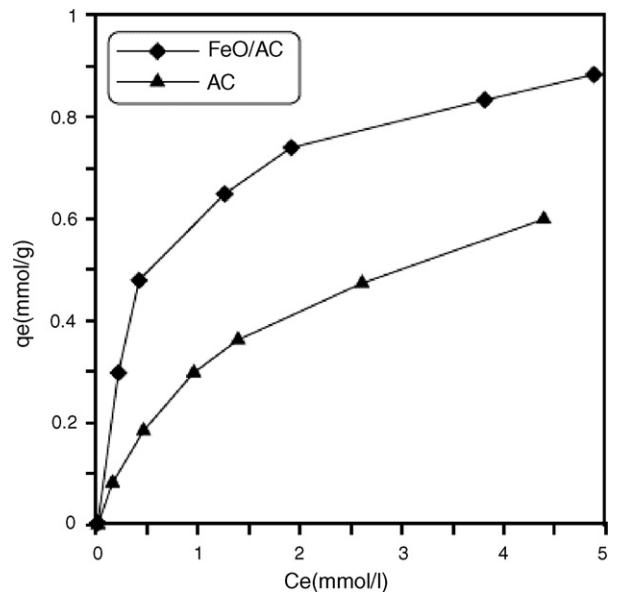

Fig. 11. Relationship between the adsorption capability (q_e) and equilibrium concentration (C_e).

is better than that of AC. All results suggest that removal rates of arsenic are obviously increased with increase of the amount of iron oxides, which were deposited on the surface of virgin activated carbon. This indicates that the better affinity between ferric and arsenic. On the other hand, it is interesting to note that the presence of the iron oxide does not significantly affect the adsorption capacity of the activated carbon. On the contrary, the FeO/AC composite, which has lower surface area, increases the arsenic-absorbed capacity. This composite can be used as adsorbent to remove arsenic from drink water.

3.2.2. The sorption isotherms

Experimental results of the sorption experiments, which take into account the effects of equilibrium concentration on the adsorption capacity, were evaluated with two popular adsorption models, Langmuir and Freundlich models. Through the analysis of the isotherm data, an equation is developed to accurately represent the experimental results and be used for design purposes [31]. The linearized forms of the equations representing the models were used.

Langmuir isotherm model assumes that adsorption occurs at specific homogeneous adsorption sites within the adsorbent and intermolecular forces decrease rapidly with the distance from the adsorption surface. Then, the linearized form of Langmuir equation is defined as

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{bq_mC_e} \tag{2}$$

where q_e is the amount of solution adsorption on the surface the adsorbent (mmol/g), C_e is the equilibrium concentration in the solution (mmol/L), q_m is the maximum surface density at monolayer coverage and b is the Langmuir adsorption constant (L/mmol). The plots of $1/q_e$ versus $1/C_e$ give straight lines. The values of q_m and b can be calculated from the intercept and slope of the plots, respectively. Langmuir constants and correlation coefficients (R^2) are shown in Table 2.

Freundlich equation is derived to model the multiplayer adsorption and the adsorption on heterogeneous surfaces. The Freundlich model is formulated as:

$$q_e = K_fC_e^{\frac{1}{n}} \tag{3}$$

where q_e is the equilibrium solution concentration on adsorbent (mmol/g), C_e is the equilibrium concentration of the solute (mmol/L), K_f is Freundlich constant (mmol/g), which indicates the adsorption capacity and represents the strength of the adsorptive bond and n is the heterogeneity factor which represents the bond distribution. A linear of the Freundlich equation can be obtained by taking logarithms of the equation. The plots of log q_e versus log C_e give straight lines. K_f and n values can be calculated from the intercept and slope of these straight lines, respectively. Freundlich constants and correlation coefficients (R^2) are shown in Table 2.

From Table 2, it can be seen, for Freundlich equation, K_f values are found to be 0.5623 and 0.2644 mmol/g for FeO/AC and AC, respectively. The values of n are found to be greater than 1.5 in two cases. These results reveal that the adsorption capacity for As(v) is increased by loading ferric on activated carbon; while for Langmuir equation, q_m and b are calculated to be 0.9932, 2.0030 and 0.8066, 0.5833 for FeO/AC and AC, respectively. These values show that the capacity of FeO/AC for As (v) is greater than that of AC. From the comparisons of the R^2 values, the adsorption isotherm for As(v) are fitted satisfactorily well to Langmuir equation with a correlation coefficient to be greater than 0.9910. That is, for the adsorption of As (v), Langmuir equation gives a better fit to the experimental results than Freundlich equation.

3.2.3. Desorption studies

Desorption studies can help to regenerate the adsorbents, and then can be reused to adsorb As(V). Desorption efficiency of FeO/AC and AC is checked with acids like HCl, H_2SO_4, HNO_3, and NaOH. Preliminary studies on the adsorption of As(V) using various acids (from 0.5 to 5 M) indicate that the desorption efficiency of As(V) from FeO/AC is approximately 2–10%. The concentrations of desorption As(V) is varied with pH and type of adsorbents. The concentration of As(V) desorbed from solid residue (adsorption process: adsorbent is FeO/AC) are higher than those from the solid residue (adsorption process: adsorbent is AC). Maximum desorption from FeO/AC is observed at pH range of 13–14.

Fig. 12 shows that the desorption efficiency of FeO/AC for As is enhanced with increase of pH values. Maximum concentrations of As (V) dissolved are found to be about 4.31 mmol/L at pH over 13. It has been determined that 97.3% of As(V) in FeO/AC is dissolved in this pH range. These results demonstrate that the adsorbed As(V) can be desorbed from the FeO/AC using 1% NaOH and successfully applied for the regeneration of the FeO/AC with a shaking time of 12 h.

3.3. Column adsorption experiments

The breakthrough curves of arsenic were obtained from the column adsorption experiments. The efficiency of arsenic removal is presented by breakthrough curves, which show the concentration ratios (C/C_0) is a function of throughput volumes (C is the instantaneous concentration of effluents and C_0 is the

Table 2
Langmuir and Freundlich isotherm constants

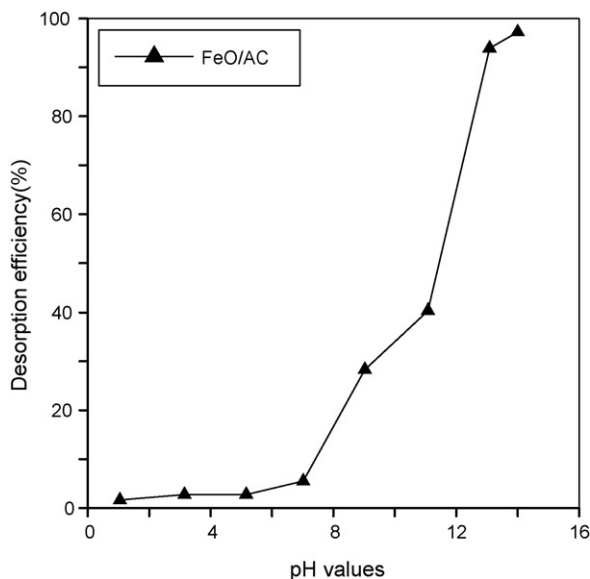| Adsorbent	Langmuir isotherm parameters			Freundlich isotherm parameters		
	q_m (mmol/g)	b (L/mmol)	R^2	K_f (mmol/g)	n	R^2
FeO/AC	0.9932	2.0030	0.9910	0.5623	2.9943	0.9404
AC	0.8066	0.5833	0.9998	0.2644	1.5779	0.9738

Fig. 12. The relationship of desorption and pH values.

initial concentration of influent). Fig. 13 shows the curves for arsenic removal by FeO/AC and virgin activated carbon. In this study, the influence concentration is 0.5 mg/L, the solution flow rate is 500 mL/h, the nominal resident time of the influents solution in the adsorbents column was 12 min. It is shown that the arsenic breaks through the virgin activated carbon column fast (at 60 mL, about 7.2 min), after feeding and the column took approximately 750 mL before being completed exhausted with arsenic. On the other hand, the FeO/AC column was broken through for a substantial period of time (150 min), not achieving breakthrough until 1250 mL, and reached exhaustion after 2200 mL of throughput volume. In each of the effluent fractions, which were collected prior to 1250 mL, the arsenic concentration was below 0.01 mg/L. That is, the FeO/AC could remove arsenic by at least 99.8%.
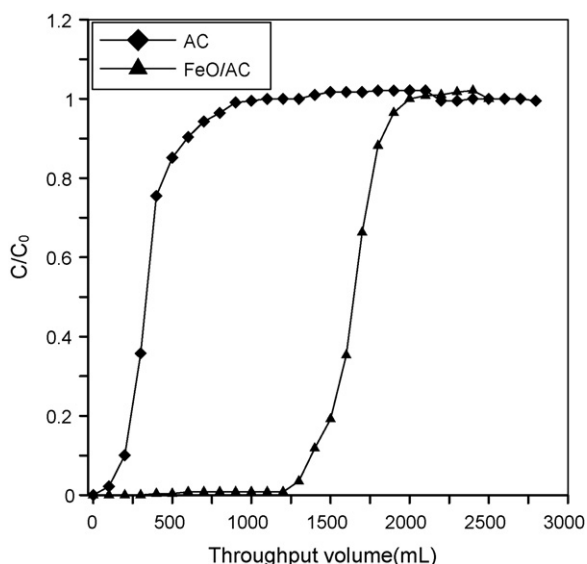

Fig. 13. Removal of arsenic onto adsorbents (influent concentration: 0.5 mg/L; solution flow rate: 500 mL/h).

4. Conclusions

The FeO/AC composite adsorbent was prepared using the available and low-cost chemicals. The characteristics of this composite material were investigated by different experimental analysis means. The arsenic removal capability of the prepared FeO/AC composite material and virgin activated carbon were investigated and compared by the batch and column adsorption experiments. Langmuir isotherm adsorption equation well describes the experimental adsorption isotherms. The desorption studies show that the concentration of As(V) desorbed from solid residue (adsorption process: adsorbent is FeO/AC) is higher at the value of pH over 13. Results suggest that the main phase formed in the composite is magnetite, goethite, maghemite and hematite; the presence of iron oxides did not significantly affect the surface area or the pore structure of the activated carbon. Batch and column adsorption studies revealed that FeO/AC composite adsorbent and virgin activated carbon were effective in removing arsenic. Furthermore, the arsenic removal capability of the prepared FeO/AC composite material is higher than 99.1%. That is, the FeO/AC composite adsorbent has excellent capacities of removing arsenic from drinking water.

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