Preparation of Anion Exchange Resin Loaded with Ferric Oxide for Arsenic (V) Removal from Aqueous Solution

Sujitra Tandorn, Orn-Anong Arqueropanyo, Wimol Naksata, and Ponlayuth Sooksamiti

Abstract—In this study, an adsorbent (FO-Dowex) was prepared by modification of commercial strong base anion exchange resin (Dowex marathon MSA) with ferric oxide (FO) and applied as adsorbent for removal of As(V) from aqueous solution. The effects of initial iron concentration and reaction contact time on adsorption capacity of As(V) were investigated for optimal conditions of adsorbent. In addition, the FO-Dowex was characterized using scanning electron microscopy and Fourier transformed infrared spectroscopy. It was shown that the optimal initial iron concentration and reaction contact time were 0.5 mol/L and 12 h, respectively. Comparison of adsorption performance between the prepared FO-Dowex and the anion resin revealed that FO-Dowex exhibited higher adsorption capacity with the value of 74.07 mg/g compared to the one from anion resin (49.75 mg/g) under the same conditions. It can be suggested that the modification of anion resin with ferric oxide can significantly improve the adsorption performance in As(V) removal.

 ${\it Index Terms} {\it --} Adsorption, \ \ arsenic, \ \ ferric \ \ oxide, \ \ anion \ \ exchange \ resin.$

I. INTRODUCTION

Arsenic pollution has become a serious concern in many countries throughout the world due to its high toxicity and adverse effect on human health and environment. Arsenic can be released into environment through the natural process and human activities [1]. Long-term uptake of high concentration of arsenic can cause several health effects such as skin hyperpigmentation, cardiovascular disease and various types of cancer. As a result, World Health Organization (WHO) has established the maximum contaminant limit (MCL) of 10 µg/L [2]. Several techniques have been applied for removal of arsenic from water including oxidation, precipitation, coagulation, adsorption, ion-exchange, and membrane filtration. Among them, adsorption is an economical and effective method for arsenic removal [3]. Ion exchange resin is an effective adsorbent for removal of arsenic from water. However, it exhibited low selectivity in the presence of other competing anions such as chloride, sulfate, and nitrate leading to the low adsorption

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performance of ion exchange resin [4]. Therefore, it is essential to develop a new selective adsorbent for arsenic removal. In recent years, polymeric-based ferric oxide adsorbents have become more attractive for the environmental remediation. These materials combined the advantages of both excellent mechanical properties of polymeric ion exchange and the specific affinity of ferric oxide toward target pollutant [5]. Previous studies have shown that fabrication of these hybrid adsorbents by dispersing or loading of iron oxide onto polymeric ion exchange can provide high efficiency for removal of toxic pollutants, including arsenic, phosphate, selenium, and antimony [6]-[9]. However, Hristovski et al. [10] suggested that the preparation condition was the important factor that significantly influenced on the formation of iron oxide nanoparticle and the adsorption capacity of adsorbent. Until now, little is known about the effect of preparation conditions on the adsorption performance of adsorbent, and thus further investigations are required.

Here, the effects of iron concentration and reaction contact time on the adsorption capacity of resulting adsorbent for As(V) were investigated and the optimal conditions for adsorbent preparation were determined. The prepared adsorbent (FO-Dowex) was also characterized by scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), and Fourier transform infrared spectroscopy (FTIR) techniques. In addition, the adsorption performance of As(V) by FO-Dowex and host anion resin were compared. The influence of iron modification on the adsorption capacity was also discussed.

II. MATERIAL AND METHOD

A. Materials

Dowex marathon MSA was purchased from Aldrich-Sigma, USA which was used as a starting material for preparation of adsorbent. It is a strong base anion exchange resin which consists of positive charge of quaternary ammonium functional group and polystyrene-divinylbenzene matrix. The diameter of resin beads varied between $560-600~\mu m$.

B. Chemicals

All reagents used in this study were analytical grade. Ferric chloride (FeCl $_3$.6H $_2$ O) was purchased from Loba Chemie Pvt. Ltd., India. Sodium arsenate (Na $_2$ HAsO $_4$.7H $_2$ O) was supplied by Aldrich-Sigma, USA. Sodium chloride (NaCl) and sodium hydroxide (NaOH) were obtained from Thermo Fisher, Australia. Stock solution of As(V) (1000 mg/L) was prepared by dissolving desired amount of Na $_2$ HAsO $_4$.7H $_2$ O

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in deionized water.

C. Preparation of Adsorbent (FO-Dowex)

The adsorbents were prepared by ion-exchange deposition technique. The preparation procedure was performed according to the method in literature [7]. In details, 5 g of Dowex anion resin was added to the mixture of FeCl₃-HCl solution containing different concentration of FeCl₃ (0.025-3M) and the mixtures were stirred at various time intervals (2-24 hours) and filtered. In this step, the optimum preparation conditions such as initial iron concentration and reaction contact time were investigated. After that, the anion resins were filtered and soaked in 1M NaOH-NaCl solution. The mixtures were stirred for 12 h at room temperature and filtered. Then, the solid particles were washed with deionized water and ethanol solution to remove unloaded iron oxide and dried at 55°C for 24 h. Finally, the obtained adsorbents were kept in vacuum desiccator for further use.

D. Characterization of Adsorbents

Various techniques have been used to determine the physical and chemical properties of Dowex anion resin and FO-Dowex. Fourier transform infrared spectrophotometer (FTIR; Tensor 27, Bruker) was used to investigate the surface functional groups in the wavenumber range of 400-4000 cm⁻¹. In addition, the specific surface and pore volumes were determined by the N₂ adsorption and desorption method at 77K using Quantachrome autosorb 1MP. Moreover, the surface morphology and elemental compositions of adsorbents before and after loading of ferric oxide were examined using scanning electron microscope (SEM; JOEL, model JSM-5910) and energy dispersive X-ray spectroscopy (EDX), respectively.

E. Adsorption Experiment

The experiments were carried out in batch system by mixing fixed amount of adsorbent with 100 ml of As(V) solution in Erlenmeyer flask. The initial pH was adjusted to desirable pH value using 0.1 M NaOH or 0.1 M HCl. The mixture was shaken in a water bath shaker (model WSB-45, Daihan scientific, Korea) at constant speed of 100 rpm. After adsorption time, the solution was filtered through whatman No.1 filter paper and the concentration of arsenic in filtrate was analyzed by inductively coupled plasma emission spectrometer (ICP-OES; Optima3000, Perkin Elmer, USA) The amount of As(V) adsorbed at equilibrium(q_e) was calculated by the following equation:

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

where C_0 and C_e are the initial and equilibrium As(V) concentration(mg/L), respectively, V is the volume of As(V) solution(L) and W is amount of adsorbent(g)

To study the effects of initial iron concentration and reaction time on As(V) adsorption, the experiments were conducted with initial As(V) concentration of 30 mg/L and 0.05 g of adsorbent prepared under various conditions. The mixtures were shaken at 30°C for 6 h and the remaining As(V) concentration was determined by ICP-OES. The optimum

preparation conditions were evaluated by comparing the adsorption performance of absorbent obtained from different conditions.

F. Adsorption Isotherm

In order to determine the adsorption capacity of adsorbent, the experiments were conducted with six different initial As(V) concentrations ranging from 10 to 60 mg/L at constant temperature of 30 °C, the contact time of 6 h, and the initial pH was controlled at pH=5. Langmuir and Freundlich isotherms were used to analyzed the experimental adsorption data of As(V) onto FO-Dowex and anion resin.

Langmuir isotherm is based on monolayer adsorption on the active sites of adsorbent with a finite number of identical binding sites [11], [12]. The linear form of Langmuir isotherm is given by

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

where q_e is the amount of As(V) adsorbed at equilibrium(mg/g), C_e is the equilibrium As(V) concentration(mg/L), q_m and b are Langmuir constants which represent the maximum adsorption capacity(mg/g) and the energy of adsorption, respectively. These parameters are determined from the slope and intercept of the linear plot of C_e/q_e and C_e .

Freundlich adsorption isotherm can be applied for multilayer adsorption on heterogeneous surface with non-uniform distribution of adsorption heat [11], [12]. The linearity of Freundlich isotherm can be expressed by the following equation

$$\log q_e = \frac{1}{n} \log C_e + \log K_f \tag{3}$$

where K_f and n are Freundlich constants associated with capacity and intensity of adsorption, respectively. The Freundlich isotherm parameters are obtained by fitting the curve of $\log q_e$ versus $\log C_e$.

III. RESULTS AND DISCUSSION

A. Characterization of Adsorbents

As shown in Fig. 1, the original anion resin was light yellow spherical beads whereas the prepared adsorbent (FO-Dowex) was brown color. However, the material kept its original spherical shape after loading with ferric oxide. The SEM image of Dowex anion resin (Fig. 2a) showed the uniform and smooth surface, while the surface of FO-Dowex (Fig. 2b) was rough due to the deposition of iron oxide particles. The EDX analysis of anion resin and FO-Dowex are illustrated in Fig. 3a and Fig. 3b, respectively. It can be revealed that Dowex anion resin contained mainly of carbon and chlorine which may result from the polymeric matrix and exchangeable ion of anion resin composition. Meanwhile, Fe element (53.24 %wt) was detected in the element compositions of FO-Dowex. This confirmed that the loading

of iron oxide onto anion resin is successful. Fig. 4 illustrated the FTIR spectra of adsorbent before and after loading of ferric oxide. The FTIR spectrum of anion resin (Fig. 4a) exhibited a broad peak at 3428 cm⁻¹, which can be assigned to O-H stretching vibration of hydroxyl group [13], [14]. Meanwhile, three peaks at about 3015, 2924 and 2824 cm⁻¹ indicate C-H stretching of aromatic polystyrene-divinylbenzne matrix in anion resin [14]. Furthermore, a significant peak at 1481 cm⁻¹ corresponds to C-H bending of quaternary ammonium functional group of anion resin [15]. As shown in Fig. 4b, the peak pattern of FO-Dowex is similar to that of anion resin. However, an additional peak observed at 581 cm⁻¹ was attributed to Fe-O stretching vibration which clearly indicated the presence of loaded ferric oxide onto anion resin [16].

The data in Table I clearly showed that the specific surface area of FO-Dowex (21.62 m2/g) was higher than that of the anion resin (17.13 m2/g), owing to the high surface to mass ratio of iron oxide particle [17]. However, the pore volume of FO-Dowex turns out to be slightly lower than that of anion resin (see Table I). This observation may be attributed by the pore blockage caused by iron oxide loading, which might reduce the porosity of adsorbent [18].

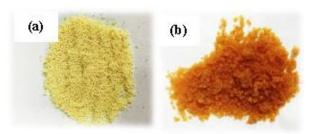


Fig. 1. Photographs of (a) Dowex anion resin (b) FO-Dowex.

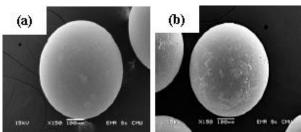


Fig. 2. SEM images of (a) Dowex anion resin (b) FO-Dowex.

TABLE I: SPECIFIC SURFACE AREA AND POROSITY OF THE ADSORBENTS IN THIS STUDY

Adsorbent	BET surface area (m ² /g)	Pore volume (cm ³ /g)
Dowex anion resin	17.13	0.0273
FO-Dowex	21.62	0.0256

B. Optimization for Adsorbent Preparation

In this part, the optimal conditions for As(V) adsorption on FO-Dowex adsorbents were evaluated by varying initial iron concentration and reaction contact time. As shown in Fig. 5, the adsorption capacity increased with increasing of $FeCl_3$ concentration from 0.025 to 0.5 mol/L. This observation may be attributed by the higher amount of adsorption sites onto anion resin surface, resulting in an enhancement of the As(V) adsorption. The maximum adsorption capacity was obtained at 0.5 mol/L of $FeCl_3$. However, a further increase of iron

concentration up to 3 mol/L resulted in a drop of the adsorption capacity. It is probably due to the pore blockage and self-aggregation of iron oxide particle at a high amount of iron. This may reduce the available active sites for adsorption and hinder the diffusion of As(V) into the adsorption sites of adsorbent, lowering the adsorption capacity [19], [20].

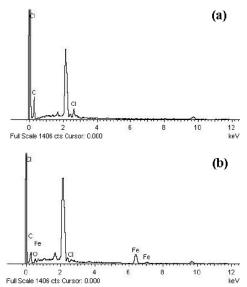


Fig. 3. EDX analysis of (a) Dowex anion resin (b) FO-Dowex.

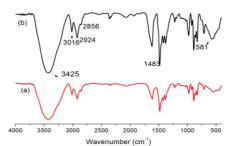


Fig. 4. FTIR spectra of (a) Dowex anion resin (b) FO-Dowex.

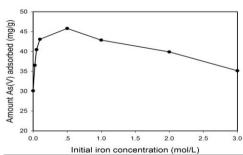


Fig. 5. Effect of initial iron(III) concentration on As(V) adsorption capacity.

To find an optimum reaction contact time, anion resins were added into iron solution and stirred at various time intervals between 2 to 24 h, as illustrated in Fig. 6. The results reveal that the adsorption capacity increased from 32.78 mg/g to 45.69 mg/g when increasing of the contact time from 2 to 12 h. For a longer reaction time, it provided more times for iron to react with quaternary ammonium functional group of anion resin through ion-exchange process. This also increases opportunity for iron oxide pre-loading onto anion resin before precipitation with alkaline solution [9]. However, more contact time (after 12 h) did not significantly affect the As(V) adsorption capacity.

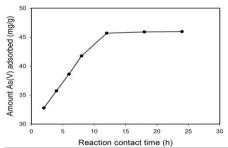


Fig. 6. Effect of reaction contact time on the adsorption capacity of As(V).

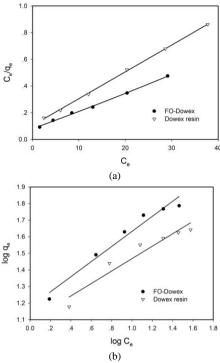


Fig. 7. (a) Langmuir and (b) Freundlich adsorption isotherm plots of As(V) adsorption onto Dowex anion resin and FO-Dowex.

From the results, the optimum conditions for adsorbent preparation were obtained with 0.5 mol/L of iron concentration and a contact time of 12 h. These conditions were further applied for further adsorption discussed below.

C. Adsorption Isotherm

The linear plots of Langmuir and Freundlich isotherm for As(V) adsorption onto FO-Dowex and Dowex anion resin are shown in Fig. 7a and 7b, respectively. The Langmuir and Freundlich isotherm parameters along with correlation coefficient value (R²) are summarized in Table II. According to the R² values, the Langmuir isotherm model fit quite well (R²>0.99) with the experimental data for both FO-Dowex and anion resin, indicating the monolayer adsorption on homogenous surface. According to Langmuir isotherm, the maximum adsorption capacities (q_m) of FO-Dowex and anion resin were 74.07 and 49.75 mg/g, respectively. The higher adsorption capacity of FO-Dowex was attributed to the increase of surface area resulting from the presence of iron oxide and this may provide more adsorption sites on the adsorbent surface for As(V) adsorption [17], [18]. Also, the formation of inner sphere complex between ferric oxide and As(V) would increase the adsorption affinity [21], [22]. Moreover, the addition of Fe may increase the positive charges on the adsorbent surface leading to the enhanced

electrostatic interaction between arsenate and iron oxide [23]. Therefore, it can be indicated that the loading of iron oxide is an important factor for enhancement of the adsorption capacity of anion resin for As(V). For Freundlich isotherm, the K_f constant calculated for FO-Dowex (15.02) was higher than that for Dowex anion resin (12.37). In addition, the values of parameter "n" obtained in this study were greater than unity for both adsorbents, indicating that adsorption of As(V) on both FO-Dowex and anion resin were favorable. Comparison of adsorption capacity of different adsorbents to the values reported in literature for As(V) removal are presented in Table III. Overall, the data clearly indicates that FO-Dowex exhibits higher adsorption capacity than other iron-coated adsorbents.

TABLE II: ISOTHERM PARAMETERS FOR AS(V) ADSORPTION ONTO DOWEX

ANION RESIN AND FO-DOWEX					
Isotherm model	Parameter	FO-Dowex	Anion resin		
Langmuir	q_{m}	74.07	49.75		
	b	0.18	0.19		
	\mathbb{R}^2	0.9972	0.9993		
Freundlich	\mathbf{K}_{f}	15.02	12.37		
	n	2.20	2.67		
	\mathbb{R}^2	0.9651	0.9250		

TABLE III: COMPARISON OF MAXIMUM ADSORPTION CAPACITIES OF VARIOUS IRON-BASED ADSORBENTS FOR AS(V) REMOVAL

Adsorbent	$\begin{array}{c} q_m \\ (mg/g) \end{array}$	References
Iron coated diatomite	8.6	[24]
Iron coated chitosan	35.7	[25]
Bead cellulose loaded with iron oxyhydroxide	33.2	[26]
Iron coated rice husk	2.5	[27]
Iron coated mesoporous carbon	5.15	[28]
FO-Dowex	74.07	This study

IV. CONCLUSION

In this study, FO-Dowex was successfully prepared by loading of iron oxide onto anion resin through ion-exchange deposition method using FeCl3 as the precursor. The presence of ferric oxide loaded on anion resin was confirmed by FTIR and EDX analysis. The results showed that both the initial iron concentration and the reaction contact time have a significant effect on As(V) adsorption capacity. The optimal conditions were obtained with the iron concentration of 0.5 mol/L and the reaction contact time of 12 h. Under the optimal conditions, the FO-Dowex exhibits a remarkably increased adsorption capacity as compared to the case of anion resin. The equilibrium adsorption data for both FO-Dowex and anion resin fitted well to the Langmuir isotherm model. Based on the results, it can be suggested that FO-Dowex would serve as a potential adsorbent for removal of arsenic from aqueous solution.

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