Performance Evaluation of the Fe-IR-120(Na)-DEHPA Impregnated Resin in the Removal Process of As(V) from Aqueous Solution

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Abstract: In the present paper the performance of a new functionalized material Fe-IR-120(Na)-DEHPA in the removal process of As(V) from aqueous solutions, has been investigated. The new material was obtained through impregnation of Amberlite IR-120(Na) with di(2-ethylhexyl) phosphoric acid (DEHPA) through the dry method. Through impregnation of the Amberlite IR-120(Na) was obtained a resin with two functional groups which was further loaded with Fe(III) ions because of the high affinity of arsenic towards iron. The physicochemical methods of analysis (FTIR spectroscopy, EDX and SEM) approved that the impregnation with DEHPA and the iron loading, respectively, occurred. By applying the kinetic model to the experimental data it was found that the removal of As(V) ions by Fe-IR-120(Na)-DEHPA follows the pseudo-second-order rate kinetics. The linear Langmuir and Freundlich isotherm models were used to represent the experimental data and these could be well interpreted by the Langmuir isotherm. R_L values between 0 and 1.0 further indicate a favorable adsorption of As(V) ions onto Fe-IR-120(Na)-DEHPA. The maximum As(V) ions uptake calculated from Langmuir model was 21.8 $\mu\text{g/g}$.

Key words: Amberlite IR-120(Na), impregnation with DEHPA, Fe(III) ions loading, As(V) removal, kinetic studies.

1. Introduction

The contamination of water resources with arsenic is a serious worldwide environmental problem, due to its high toxic effect on plants, animals and humans [1, 2]. The symptoms of chronic poisoning on human beings are numerous: skin cancer, liver, lung, kidney, and bladder cancer as well as conjunctivitis, hyperkeratosis, and in severe cases gangrene in the limbs and malignant neoplasm [3-5]. Therefore the maximum permissible limit in drinking water according to World Health Organization (WHO) is 10 $\mu\text{g/L}$ [6-9].

Many separation techniques have been proposed for the removal of arsenic from aqueous solutions, which

include oxidation-reduction, precipitation, co-precipitation, adsorption, electrolysis and cementation, solvent extraction, ion-exchange, ion flotation, biological processing and sorption [7-20]. Among these methods, adsorption and ion exchange are highly popular and have been widely practiced for toxic element removal [1-10]. Sorbents of different types like biomaterials, metal oxide/hydroxide, zeolite, activated carbon, laterite etc. have been used by workers for arsenic removal [1-10]. Organic ion exchange resins were found more suitable for the removal of toxic elements from dilute solution, due to their faster kinetics, ease of regeneration and high adsorption capacity [21-26]. While a number of studies have been reported for the removal of the toxic elements onto Amberlite XAD series impregnated with

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different extractants, none is reported about the impregnation of Amberlite IR-120(Na) [26-32]. Through the impregnation of the IR-120(Na) it's obtained a resin with two functional groups. It was recently studied [30-38] that solutions containing Cd^{2+} , Cu^{2+} , Cr^{3+} ions were treated with the strong cation exchanger Amberlite IR-120 and the obtained results showed that the resin has a strong affinity for these ions, but very little is reported about arsenic removal onto Amberlite IR-120(Na).

The present study, thus, reports a detailed investigation of As(V) removal from aqueous solution using as adsorbent the strong cation exchange resin Amberlite IR-120(Na) impregnated with di(2-ethylhexyl) phosphoric acid (DEHPA) through the dry method. Because the iron compounds in general were found to be very efficient adsorbents for arsenic removal from water due to the high affinity of arsenic towards iron [1, 6-10, 39], the obtained IR-120(Na)-DEHPA resin was loaded with Fe(III) ions.

In order to determine the adsorption performance of Fe-IR-120(Na)-DEHPA resins, in the removal process of As(V) from aqueous solution, the equilibrium and kinetic studies were performed.

2. Experiment

2.1 Material Preparation

The Amberlite IR-120(Na) is a gel type strongly acidic cation exchange resin of the sulphonated polystyrene type. It was supplied by Rohm and Hass Comp. U.S.A. The resin has as functional groups the sulphonic acid. To obtain a resin with two functional groups the Amberlite IR-120(Na) was impregnated with di(2-ethylhexyl)phosphoric acid (DEHPA) through the dry method. The DEHPA ~ 98.5% used as extractant, was supplied by BHD Chemicals Ltd Poole England and used as received. 5 grams of dry Amberlite IR-120(Na) has been placed in 25 mL of ethylic alcohol containing different concentration of DEHPA (0-0.2 g/mL) for 24 h. The polymeric beads have been separated through a porous filter using a

vacuum pump, washed with water and dried at 50 °C for 24 h [38]. The IR-120(Na)-DEHPA resin was loaded with Fe(III) ions ($\text{Fe}(\text{NO}_3)_3$ in 0.5 mol/L HNO_3 solution (Merck Standard Solution), because of the high affinity of arsenic towards iron [1, 6-10, 39]. To study the influence of the DEHPA impregnation on the iron loading, 0.1 g of each IR-120(Na)-DEHPA obtained at different concentration of DEHPA (0-0.2 g/mL) was introduced in 25 mL of Fe(III) ion solutions of different concentrations (10-200 mg/L). The solutions pH were adjusted around the pH value of 3, in order to avoid the Fe(III) ions precipitation in the solutions. The samples were kept in contact for 24 h, and after that were filtrated and the residual concentration of the iron ions was determined through atomic adsorption spectrophotometry using a Varian SpectrAA 280FS Spectrophotometer. The dependence of the Fe(III) ions uptake versus the initial concentration of Fe(III) ions from solution for each IR-120(Na)-DEHPA obtained at different concentration of DEHPA and also the dependence of the Fe(III) ions uptake versus the concentration of the DEHPA used for impregnation, was established. The sample of IR-120(Na) and the samples of Fe-IR-120(Na)-DEHPA respectively, were submitted to physicochemical methods of analysis (FTIR spectroscopy, EDX and SEM) in order to establish if the impregnation with DEHPA and the iron loading occurred. The FTIR spectra were recorded using a Shimadzu FTIR spectrophotometer in the range 4,000-400 cm^{-1} with 2 cm^{-1} resolution and 40 scans using KBr discs. SEM micrographs and EDX studies were performed using a FEI Inspect S scanning electron microscope.

2.2 Arsenic Removal

The influence of the contact time and initial concentration of arsenic upon the arsenic removal process from aqueous solution was investigated.

In the first instance the effect of contact time was studied for all the obtained materials. In order to

establish the arsenic removal performance the experiments were performed with 0.1 g of each obtained material in 0.025 L of 100 µg/L As(V) solution. The samples were kept in contact for different times (range: 1-24 h) at the room temperature 25 ± 1 °C. After contact time elapsed, the suspensions were filtered and the residual concentration of As(V) ions in the filtrates was determined by means of atomic absorption spectrometry using a Varian SpectrAA 110 atomic absorption spectrometer with a Varian VGA 77 hydride generation system. To determine the best materials used in the removal process of As(V) from aqueous solution the dependence of the As(V) uptake versus the contact time for each studied materials was illustrated. In order to express the kinetics of arsenic removal onto Fe-IR-120(Na)-DEHPA and to find out the mechanism of arsenic removal the results were analyzed using the following models:

The pseudo-first-order kinetic model proposed by Lagergren, based on the solid adsorption capacity can be used to determine the rate constant for the removal process and the integrated form is expressed by the Eq. (1) [3, 7, 12, 23, 25, 26, 30, 38, 39]:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (1)$$

where: q_t and q_e represent the amounts of the arsenic uptake onto the studied material at time “ t ” and at equilibrium time, respectively, µg/g; k_1 is the specific adsorption rate constant (min^{-1}).

The linear form of the pseudo-second-order rate expression of Ho and McKay, based on the solid phase sorption, is given by Refs [3, 7, 12, 23, 25, 26, 30, 38, 39]:

$$\frac{t}{q_t} = \frac{1}{h} + \frac{t}{q_e} \quad (2)$$

where: $h = k_2 \cdot q_e^2$; k_2 is the pseudo-second-order constant, $\text{min}^{-1}(\mu\text{g/g})^{-1}$.

To study the effect of the initial concentration of As(V), in each experiments 0.1 g of studied material was suspended in 25 mL of As(V) solution of different concentration (range: 10-400 µg/L) for 16 h at the room temperature. After contact time elapsed the filtrate was

collected for As(V) analysis.

In order to determine the As(V) maximum uptake of the studied material two important isotherm models were chosen to fit the experimental data in this study: Langmuir and Freundlich [3-5, 7, 23, 25, 26, 30, 38, 39].

The linear form of the Langmuir equation is expressed by:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \quad (3)$$

The linear form of the Freundlich isotherm equation can be written as:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (4)$$

where: q_e is the amount of arsenic uptake per gram of sorbent (µg/g); C_e is the equilibrium concentration of arsenic (µg/L); K_F and $1/n$ are characteristic constants that can be related to the relative adsorption capacity of the adsorbent and the intensity of adsorption, respectively; q_m is a measure of monolayer adsorption capacity (µg/g) and K_L is a constant related to the free energy of adsorption.

The various chemicals employed in the experiments were of A.R. grade and used without further purification. Distilled water was used throughout.

The solutions of As(V) were prepared by appropriate dilution from a stock solution which was prepared by diluting an appropriate amount of H_3AsO_4 in 0.5 M HNO_3 solution (Merck Standard Solutions).

All the experiments for arsenic removal process were performed at a initial pH of As(V) solution of 9, because the predominant anionic species of As(V) [H_2AsO_4^- or HAsO_4^{2-}] are found in the environment samples at this pH value [3, 7, 8].

The Fe(III) and As(V) uptake, respectively, q_e (mg/g and µg/g, respectively), is determined according to the next corresponding mass balance expression [3-5, 7, 12, 23, 25, 26, 30, 38, 39]:

$$q_e = (C_0 - C_e) \frac{V}{m} \quad (5)$$

where: C_0 and C_e are the concentrations of Fe(III) and As(V) ions, respectively, (mg/L and µg/L, respectively)

in the solution initially ($t = 0$) and after a time t (min), respectively, V is the volume of the solution (L) and m is the mass of material employed (g).

3. Results and Discussion

3.1 Evaluation of the Material Preparation Process

The experimental data regarding the dependence of the Fe(III) ions uptake versus the initial concentration of Fe(III) ions from solution, for each IR-120(Na)-DEHPA, obtained at different concentration of DEHPA, are presented in Fig. 1.

The experimental data show the increase of the Fe(III) ions uptake as the initial concentration of Fe(III) ions from solution increases, for each studied material. After an initial concentration of Fe(III) ions of 100 mg/L the increasing of the Fe(III) ions is not so significant. In conclusion the optimum concentration of the Fe(III) ions in the process of the iron loading onto IR-120(Na)-DEHPA is 100 mg/L for a S:L ratio of 0.1:0.025.

The experimental data regarding the dependence of the Fe(III) ions uptake versus the concentration of the DEHPA used for impregnation in case of an initial

concentration of Fe(III) ions of 100 mg/L are presented in Fig. 2.

It can be noticed that the Fe(III) ions uptake increase with the increasing of the DEHPA concentration, used in the impregnation process of the IR-120(Na). The results allow us to suppose that the Fe(III) ion loading is realised: on one hand, because of the cation exchange process between the Na ions from the studied resin and Fe(III) ions from the aqueous solution and on the other hand, because of the Fe(III) ions loading onto the second functional group from the studied resin resulted after the impregnation of the Amberlite IR-120(Na) with DEHPA.

If our supposition is true, these results also demonstrate that with the increasing of the DEHPA concentration, used for impregnation of the studied material, increase the acid quantity sorbed by the resin, which is expected to be confirm by the EDX analysis.

Fig. 3 displays the FTIR spectra of the Amberlite IR-120(Na) and of the material obtained after resin impregnation with DEHPA ($C_i = 0.2$ g DEHPA/mL solvent) and Fe(III) loading ($C_i = 100$ mg/L).

From the IR spectrum of the both sample we observe

Fig. 1 Dependence of the Fe(III) ions uptake versus the initial concentration of Fe(III) ions from solution for each IR-120(Na)-DEHPA (pH = 3, $t = 24$ h, $m = 0.1$ g, $v = 0.025$ L).

Fig. 2 Dependence of the Fe(III) ions uptake versus the concentration of the DEHPA used for impregnation (pH=3, t=24 h, T = 25 ± 1 °C, m = 0.1 g, v = 0.025 L, $C_{\text{Fe}} = 100$ mg/L).

Fig. 3 IR spectra of IR-120(Na)-sample A and Fe-IR-120(Na)-DEHPA-sample B.

the common bands of the Amberlite IR-120(Na): 2,975 cm^{-1} -C-H stretching of CH_3 ; 2,890 cm^{-1} -aliphatic C-H stretching; 1,650 cm^{-1} C=C; 1,000-1,030 cm^{-1} -S-O; 600-700 cm^{-1} C-S. Besides these common bands the spectrum of Fe-IR-120(Na)-DEHPA exhibits a clear band at 2,340 cm^{-1} specific for P-OH stretching; and at 2,890 cm^{-1} for Fe-OH. Also can be seen a small band at 1,230

cm^{-1} for P=O stretching. The other specific bands for Fe-OH (at 1,037 cm^{-1}) and for P-O-C stretching (at 1,029 cm^{-1}) can not be clear identified because of their interaction between them and the specific band for the -S-O stretching (1,000-1,030 cm^{-1}). This proves the adsorption of DEHPA and the loading of Fe(III) ions, respectively, onto the Amberlite IR-120(Na) resin.

A scanning electron micrograph of the IR-120(Na) and Fe-IR-120(Na)-DEHPA are presented in Fig. 4.

The Amberlite IR-120(Na) (Fig. 4a) consisted of

spherical granules which have a smooth surface under the highly magnified SEM images (3000 $\times$). In case of Fe-IR-120(Na)-DEHPA ($C_{iDEHPA} = 0.02$ g/mL solvent;

Fig. 4 Scanning electron micrograph of (a) IR-120(Na); (b) Fe-IR-120(Na)-DEHPA ($C_{iDEHPA} = 0.02$ g/mL solvent; $C_{iFe} = 100$ mg/L); (c) Fe-IR-120(Na)-DEHPA ($C_{iDEHPA} = 0.2$ g/mL solvent; $C_{iFe} = 100$ mg/L).

$C_{iFe} = 100$ mg/L) the spherical granules have small fissures, but the surface is still smooth (Fig. 4b). The increasing of the DEHPA initial concentration in the impregnation process of the Amberlite IR-120(Na) resin leads to granules smashes, and the surface consisted of conglomerates with open porosity (Fig. 4c), which is expected to develop a higher adsorption capacity of As(V).

The EDX spectrums of the studied materials are presented in Fig. 5. One may notice the presence of the characteristic peak of phosphorous and iron ions. This also proves the adsorption of DEHPA and the loading of Fe(III) ions on the IR-120(Na) support. Also, it can be observed that, both the characteristic peak of phosphorous, and also, of the iron ions, increase with the DEHPA concentration increases in the impregnation process of the resin. These results demonstrate that with the increasing of the DEHPA concentration, used for impregnation of the studied material, increase the acid quantity sorbed by the resin and consequently increase the quantity of iron ions loaded onto the studied materials.

3.2 Evaluation of the Arsenic Removal Performance of the Studied Material

3.2.1 Effect of Contact Time

The experimental data regarding the dependence of the As(V) uptake versus contact time for all the studied materials, obtained at different concentration of DEHPA, used in the impregnation process, and at an initial concentration of Fe(III) ions of 100 mg/L, used in the iron loading process, are presented in Fig. 6.

It can be observed that the equilibrium time is achieved in 16 h for all the studied materials. For subsequent experiments, an equilibrium time of 16 h was chosen for the sake of convenience. One may notice that the As(V) uptake increase with the increasing of the DEHPA concentration used in the impregnation process of the resin. This effect is not so significant for a concentration of DEHPA higher than 0.08 g/mL of solvent. From this reason the optimum

concentration of the DEHPA used in the impregnation process is that of 0.08 g/mL of solvent. These results confirm the expectation concerning the As(V) removal performance of the sample after morphology of surface area analysis.

To elucidate the removal mechanism different rate law were tested to unfold the best fitting kinetic model to the experimental results in case of use as adsorbent material the Fe-IR-120(Na)-DEHPA obtained in case of an initial concentration of DEHPA of 0.08 g/mL of solvent used in the impregnation process and at an initial concentration of Fe(III) of 100 mg/L. The pseudo-first-order rate constant may be determined from the linear plot of $\ln(q_e - q_t)$ versus t (Fig. 7), while the pseudo-second-order rate constant can be estimated from the linear plot of t/q_t versus t (Fig. 8). The values of these constants determined as indicated, together with the corresponding regression coefficients (R^2), are listed in Table 1.

Data presented in Table 1 show that the correlation coefficient for the pseudo-first-order kinetic model is lower than that obtained for the pseudo-second-order model, where the correlation coefficient is very close to 1. Furthermore, the value of the equilibrium sorption capacity calculated ($q_{e, \text{kinetic plot}}$) using the first-order model, is not close to the experimental value ($q_{e, \text{exp}}$). In the case of the pseudo-second-order model the theoretically predicted equilibrium sorption capacity is close to the value experimentally determined. This shows that the kinetics of As(V) removal by sorption on the Fe-IR-120(Na)-DEHPA is described by a pseudo-second-order expression.

3.2.2 Effect of As(V) Initial Concentration

To determine the maximum As(V) uptake by the Fe-IR-120(Na)-DEHPA experimentally obtained, the dependence of the As(V) uptake versus the equilibrium concentration is presented in Fig. 9. The maximum As(V) uptake experimentally determined is 18.6 $\mu\text{g/g}$.

The equilibrium data have been correlated with the Freundlich and Langmuir isotherms. The curves and

(a)

(b)

(c)

Fig. 5 EDX spectrum of (a) IR-120(Na); (b) Fe-IR-120(Na)-DEHPA ($C_{\text{IDEHPA}} = 0.02$ g/mL solvent; $C_{\text{iFe}} = 100$ mg/L); (c) Fe-IR-120(Na)-DEHPA ($C_{\text{IDEHPA}} = 0.2$ g/mL solvent; $C_{\text{iFe}} = 100$ mg/L).

Fig. 6 Effect of contact time on As(V) uptake (pH = 9, $T = 25 \pm 1^\circ\text{C}$, $C_{\text{As}} = 100 \mu\text{g/L}$, $m = 0.1 \text{ g}$, $v = 0.025 \text{ L}$).

Fig. 7 Pseudo-first-order kinetic plot for As(V) removal process.

Fig. 8 Pseudo-second-order kinetic plot for As(V) removal process.

Table 1 Kinetic model parameters for As(V) ions removal by Fe-IR-120(Na)-DEHPA.

$q_{e, \text{exp}}$ $\mu\text{g/g}$	Pseudo-first-order model			Pseudo-second-order model		
	q_e , kinetic plot ($\mu\text{g/g}$)	k_1 (min^{-1})	R^2	q_e , kinetic plot ($\mu\text{g/g}$)	k_2 [$\text{min}^{-1}(\mu\text{g/g})^{-1}$]	R^2
13	6.45	$2.9 \cdot 10^{-3}$	0.933	13.8	$7.21 \cdot 10^{-4}$	0.9986

Fig. 9 The dependence of the As(V) metal uptake versus the equilibrium concentration (pH = 9, $t = 16 \text{ h}$, $T = 25 \pm 1^\circ\text{C}$, $m = 0.1 \text{ g}$, $v = 0.025 \text{ L}$).

parameters, as well as the correlation coefficients (R^2) for As(V) removal by Fe-IR-120(Na)-DEHPA are presented in Figs. 10 and 11 and Table 2.

The Freundlich plot has a correlation coefficient very low; this suggests a restriction on the use of Freundlich isotherms. The numerical value of $1/n < 1$, which provides information about surface heterogeneity and surface affinity for the solute, indicates a favorable sorption of As(V) and a very high affinity of the Fe-IR-120(Na)-DEHPA for As(V).

The Langmuir model effectively describes the sorption data with a correlation coefficient of 0.9938.

Fig. 10 Freundlich plot of As(V) adsorption on Fe-IR-120(Na)-DEHPA.

Fig. 11 Langmuir plot of As(V) adsorption on Fe-IR-120(Na)-DEHPA.

Table 2 Parameters of different isotherm for the As(V) adsorption on Fe-IR-120(Na)-DEHPA.

Freundlich isotherm			Langmuir isotherm		
K_F (µg/g)	$1/n$	R^2	K_L (L/µg)	$q_{m \text{ calc}}$ (µg/g)	R^2
1.008	0.5641	0.9067	0.022	21.8	0.9938

Thus the isotherm follows the sorption process in the entire concentration range studied. Furthermore, the maximum As(V) uptake obtained from the Langmuir plot is almost the same with the maximum As(V) uptake experimentally obtained.

The dimensional constant, called separation factor (R_L), was used to describe the essential characteristics of Langmuir isotherm ($R_L > 1$, unfavorable; $R_L = 1$, linear; $0 < R_L < 1$, favorable; and $R_L = 0$, irreversible),

$$R_L = \frac{1}{1 + K_L \cdot C_0} \quad (6)$$

In fact, the separation factor is a measure of the adsorbent capacity used. R_L values were calculated for the entire concentration range studied and found to be greater than 0 and less than 1, showing favorable adsorption.

4. Conclusions

In the present paper the performance of the Fe-IR-120(Na)-DEHPA resin, in the removal process of As(V) from aqueous solution, has been investigated. The studied materials has been obtained through impregnation of Amberlite IR-120(Na) resin with DEHPA and because of the high affinity of arsenic towards iron the Fe(III) ions were loaded onto the

functionalized resin. Through impregnation of the studied resin with DEHPA is obtained a resin with two functional groups. With the increasing of the DEHPA concentration in the impregnation process of the resin increased the quantity of the adsorbed acid by the resin. This led to a higher quantity of iron loaded, which changed the surface morphology of the resin and developed a higher removal capacity of As(V) from aqueous solution. The FTIR and the SEM and EDX analysis of the materials proved the adsorption of DEHPA and the loading of Fe(III) ions on the IR-120(Na) support. The optimum condition for the Fe-IR-120(Na)-DEHPA obtaining is the initial concentration of DEHPA used in the impregnation process of 0.08 g/mL of solvent and an initial concentration of Fe(III) ions of 100 mg/L. This materials showed good performance in the removal process of As(V) from aqueous solutions. By applying the kinetic model to the experimental data it was found that the removal of As(V) ions by Fe-IR-120(Na)-DEHPA follows the pseudo-second-order rate kinetics. The linear Langmuir and Freundlich isotherm models were used to represent the experimental data and these could be relatively well interpreted by the Langmuir isotherm.

R_L values between 0 and 1.0 further indicate a favorable adsorption of As(V) ions onto Fe-IR-120(Na)-DEHPA. The maximum As(V) ions uptake calculated from Langmuir model was 21.8 $\mu\text{g/g}$.

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