

The removal of toxic metals from liquid effluents by ion exchange resins. Part IX: Lead(II)/H+/Amberlite IR-120

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ABSTRACT: Lead is recognized as a highly harmful metal for humans, thus its removal from any source containing it is a primary target. In average conditions, lead is present in aqueous solutions of pH lower than 5-6 as the cation Pb^{2+} , thus in this work, the removal of such cation from aqueous solutions by the resin Amberlite IR-120 was investigated. Experimental variables that may influence to the removal of the metal were considered: stirring speed of the solution-resin system, temperature, resin dosage and resin particle size, and aqueous pH values. The metal uptake equilibrium responded well to the Freundlich isotherm, being endothermic and non-spontaneous, whereas lead uptake onto the resin responded to the pseudo-first order kinetic model; moreover, the uptake mechanism is non-dependent of the resin particle size and fits well to the aqueous diffusion model. The removal of lead(II) within the resin compared favourable to that obtained with multiwalled carbon nanotubes, and also with respect to the loading of several base metals from binary solutions loading experiments. Lead loaded onto the resin can be eluted, generally in almost quantitative form, by HCl solutions, under different experimental conditions

KEYWORDS: Amberlite IR-120; Lead(II); Liquid effluents; Multiwalled carbon nanotubes; Removal

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RESUMEN: La eliminación de metales tóxicos presentes en efluentes líquidos mediante resinas de cambio iónico. Parte IX: Plomo(II)/H+/Amberlite IR-120. El plomo está considerado como un metal altamente peligroso para los seres humano, por lo que su eliminación, de cualquier medio que lo contenga, suele ser de la máxima importancia. Comúnmente y a valores de pH inferiores a 5-6, este elemento está presente en los medios acuosos como catión Pb^{2+} . En el presente trabajo se investiga la eliminación de este catión de disoluciones acuosas empleando la tecnología de cambio iónico con la resina Amberlite IR-120. Se consideran una serie de variables que pueden afectar al proceso de carga del metal en la resina: velocidad de agitación del sistema, temperatura, dosificación de la resina y tamaño de partícula de la misma y pH del medio acuoso. La carga del metal responde a la isoterma de Freundlich, siendo el proceso endotérmico y no espontáneo, el mecanismo de carga no depende del tamaño de partícula de la resina y responde a un mecanismo por difusión en el medio acuoso. La eliminación del plomo(II) de disoluciones acuosas mediante la resina compete de forma favorable con respecto a los resultados obtenidos cuando se emplean nanotubos de carbono de pared múltiple como adsorbentes del Pb^{2+} . En ensayos llevado a cabo con disoluciones binarias, la carga del Pb^{2+} en la resina también compete favorablemente con respecto a la de otros metales base en forma catiónica. La elución del plomo(II) se puede llevar a cabo de forma casi cuantitativa con disoluciones de HCl y distintas condiciones experimentales.

PALABRAS CLAVE: Amberlite IR-120; Efluentes líquidos; Eliminación; Nanotubos de carbono de pared múltiple; Plomo(II)

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1. INTRODUCTION

Lead is another element useful for everyday life, especially in past times, but it is more than often connected with dead or murder (Emsley, 2005). Despite the above end considerations, lead is an extremely harmful metal, which ingesta, by any source, causes various diseases both in children and adults; delays in physical and mental development, high blood pressure, kidney failures, etc. (Flora *et al.*, 2012; USEPA, 2018). As example of the potential toxicity of lead, the US Environmental Protection Agency has established an action level (AL) of $0.015 \text{ mg}\cdot\text{L}^{-1}$ for lead against $1.3 \text{ mg}\cdot\text{L}^{-1}$ for copper in drinking water (USEPA, 2018).

Thus, the removal of lead from liquid effluents has an interest of the uttermost importance, and this task had been recently investigated using primarily, ion exchange or adsorption of the metal by various materials (Alexander *et al.*, 2018; Chanthapon *et al.*, 2018; Chen *et al.*, 2018; Chu *et al.*, 2018; Cunha *et al.*, 2018; El-Bahy, 2018; Elsherbiny, *et al.*, 2018; Fang *et al.*, 2018; Feng *et al.*, 2018; Georgescu *et al.*, 2018; Gupta *et al.*, 2018; Hayeeye *et al.*, 2018; He *et al.*, 2018; Huang and Wang, 2018; Kragović *et al.*, 2018; Kinnarinen *et al.*, 2018; Kulkarni *et al.*, 2018; Li *et al.*, 2018; Liu *et al.*, 2018a; Liu *et al.*, 2018b; Magri *et al.*, 2018; Quyen *et al.*, 2018; Rajamohan *et al.*, 2018; Rehman, *et al.*, 2018; Rwiza *et al.*, 2018; Sahmoune, 2018; Song *et al.*, 2018; Tran *et al.*, 2018; Wang *et al.*, 2018a; Wang *et al.*, 2018b; Yang *et al.*, 2018; Ivanenko *et al.*, 2018; Zhou *et al.*, 2018; Zhu *et al.*, 2018) liquid membranes (Ershad *et al.*, 2018; Mesli and Belkhouche, 2018), and electrochemically (Oke *et al.*, 2017).

In the present investigation, and following the series about the use of ion exchange resins in the removal of toxic metals (Alguacil *et al.*, 2002; Alguacil, 2002; Alguacil, 2003; Alguacil, 2017a; Alguacil, 2017b; Alguacil, 2018a; Alguacil 2018b; Alguacil and Escudero, 2018) the resin Amberlite IR-120 was used for the removal of lead(II) from solutions at various pH values. A series of mathematical models were employed to fit the data resulting from the experimentation, in order to a better understanding of the ion exchange process.

2. EXPERIMENTAL

Amberlite IR-120 (Fluka) is a gel type resin with the characteristics shown in Table 1; in the present investigation, and to facilitate the resin manipulation, the resin was gentle dry until constant weight. Other resins and chemical used in the investigation are of AR grade. The multiwalled carbon nanotubes (MWCNTs) has the characteristics given elsewhere (Alguacil *et al.*, 2016).

The loading and elution experiments are carried out in a glass reactor, where the mixture Pb(II)-bearing

TABLE 1. Characteristics of Amberlite IR-120

Active group	sulfonic group. H^+ form
Matrix	styrene-divinylbenzene
Particle form and particle size	Spheres, 16-45 mesh

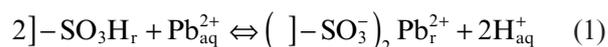
solution-resin was stirred *via* a four blades glass impeller at 850 rpm and 20°C , except when these variables are investigated.

Lead and metals in the aqueous solutions was analysed by AAS whereas metal uptake in the resin was calculated by the mass balance.

3. RESULTS AND DISCUSSION

3.1. Lead uptake onto Amberlite IR-120

Having Amberlite IR-120 resin the sulfonic group, HSO_3^- , as active group, the removal of lead(II) from aqueous solutions, if the metal is present in the solution as Pb^{2+} , such as in the present investigation, responded to the next equilibrium:



which represented to a cation exchange mechanism, in which the lead ions are loaded onto the resin and this releases two protons to the aqueous solution. In the above equation,]- refers to non-reactive matrix of the exchanger, and *r* and *aq* to the resin and aqueous phases of the system, respectively.

It is recognized, that in this type of experimentation, batch operation mode, the importance that the stirring speed of the system, has on the metal uptake; this is due that with the correct stirring speed, one can found a maximum in metal uptake onto the exchange-adsorbent or reaching this maximum at shorter times (Naushad *et al.* 2015; Aydin *et al.*, 2017). Thus, in this work the effect that this variable had in the Pb^{2+} -Amberlite IR-120 system, was investigated with aqueous solutions containing $0.01 \text{ g}\cdot\text{L}^{-1}$ Pb(II) at pH 5 and $0.5 \text{ g}\cdot\text{L}^{-1}$ of resin, and the results obtained from this investigation are shown in Table 2. Results shown, that in the present experimental conditions, the stirring speed had not effect on the metal uptake onto the resin at equilibrium but in the time in which the system reached equilibrium. Thus, an agitation speed of 850 min^{-1} was maintained in subsequent experiments. It should be noted that in these conditions, a minimum in the thickness of the aqueous layer was reached. By the use of the experimental data at 1000 min^{-1} , the kinetics of lead(II) loading onto the resin was fitted to the first and second pseudo order models. The results shown, that the experimental

TABLE 2. Percentage of lead(II) loaded onto the resin at various times and stirring speeds

Stirring speed (min ⁻¹)	30 min	1h	2h	3h
250	35.2	57.1	88.2	99.5
500	42.3	65.2	99.0	99.5
1000	45.3	68.4	99.5	99.5

Temperature: 20 °C

data well fitted the first order equation (Igberase and Osifo, 2015; An *et al.*, 2017):

$$\ln([Pb]_{r,e} - [Pb]_{r,t}) = \ln[Pb]_{r,e} - kt \quad (2)$$

with a correlation coefficient of r^2 : 0.9879, the value of k was 0.026 min^{-1} .

Studying the influence of the resin dosage on the lead uptake onto the resin it was found (Fig. 1) that, as it was somewhat expected, the metal uptake decreased as the resin doses increased but at the same time the limiting lead concentration remaining in the aqueous solution also decreased. The above set of experiments was carried out with aqueous solutions containing $0.01 \text{ g}\cdot\text{L}^{-1}$ Pb(II) at pH 5 and resin dosages of 0.065 – $0.5 \text{ g}\cdot\text{L}^{-1}$. In this case, the time to reach equilibrium was strongly dependent upon the resin dosage, i.e. for a $0.5 \text{ g}\cdot\text{L}^{-1}$ resin dose the time was 2 h against the more than the 5 h needed for the $0.065 \text{ g}\cdot\text{L}^{-1}$ resin dose. Experimental data were fit to the Freundlich and Langmuir models, the results from these fits indicated that the system responded better to the Freundlich mode (r^2 : 0.9074) (Babaei *et al.*, 2017; Mai *et al.*, 2017; Wang *et al.*, 2017):

$$\ln[Pb]_{r,e} = \ln K_f + \frac{1}{n} \ln[Pb]_{s,e} \quad (3)$$

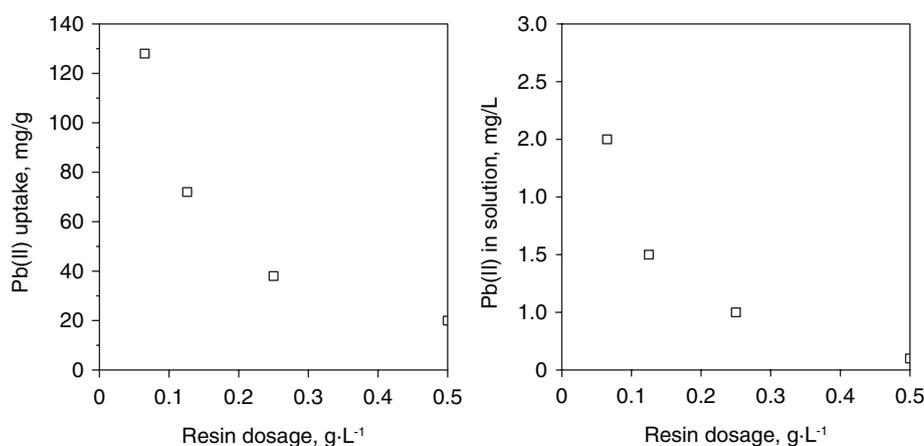


FIGURE 1. Influence of the resin dosage on the lead(II) loads onto the resin (left) and the lead remaining in the solution (right). Temperature: 20 °C. Time: 7 h. Stirring speed: 850 min⁻¹.

where $[Pb]_{r,e}$ and $[Pb]_{s,e}$ at the lead concentrations at the equilibrium in the resin and in the aqueous solution, respectively. The values of $\ln K_f$ and $1/n$ estimated from the fit were of 4.3 and 0.48, respectively. Furthermore, the influence of the resin particle size against the lead(II) uptake was also investigated by contacting aqueous solutions of $0.01 \text{ g}\cdot\text{L}^{-1}$ Pb(II) at pH 5 and resin ($0.25 \text{ g}\cdot\text{L}^{-1}$) of different particle size. The results of these series of experiments being shown in Fig. 2, plotting the percentage of lead loaded onto the resin versus time. It can be seen, that the resin particle size influences the percentage of metal loaded onto the resin, the metal uptake at the equilibrium increased as the resin particle size becomes lower (Table 3). The experimental data obtained within the three resin particle sizes were fitted to three models in order to estimate the probable mechanism responsible for the lead(II) load onto the resin. The results from the fit indicated that in the three cases, the three resin particle size, the mechanism involved in the metal uptake is that of aqueous diffusion (López Díaz-Pavón *et al.*, 2014):

$$\ln(1 - F) = -kt \quad (4)$$

The results of these fit being summarized in Table 4. In the above equation, k is the rate constant, t is the elapsed time and F is the factorial approach to the equilibrium, defined as:

$$F = \frac{[Pb]_{r,t}}{[Pb]_{r,e}} \quad (5)$$

where $[Pb]_{r,t}$ and $[Pb]_{r,e}$ at the lead(II) concentrations in the resin at an elapsed time and at the equilibrium, respectively.

Experiments about the influence of the temperature on Pb(II) removal from the aqueous solution by the resin, were carried out using aqueous solutions

of 0.01 g·L⁻¹ Pb(II) at pH 5 and a resin dose of 0.25 g·L⁻¹. The results from these experiments being shown in Table 5. The increase of the temperature produces an increase in the metal uptake onto the resin. From the experimental data, the thermodynamic ΔH° , ΔS° and ΔG° values associated with this system were estimated using the standard

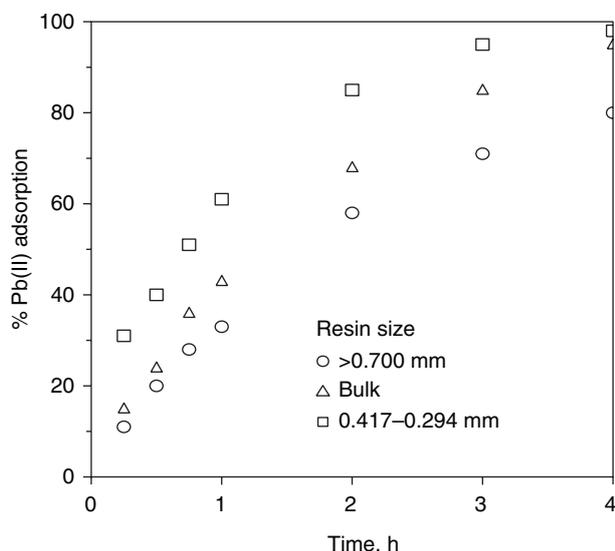


FIGURE 2. Influence of the resin particle size on lead(II) uptake onto the resin. Temperature: 20 °C. Stirring speed: 850 min⁻¹.

TABLE 3. Dependence of metal uptake at equilibrium with resin particle size

Particle size (mm)	[Pb(II)] uptake (mg·g ⁻¹)
0.417-0.294	39.8
bulk	38.0
>0.700	36.0

Temperature: 20 °C. Stirring speed: 850 min⁻¹

TABLE 4. Experimental data fit to the aqueous diffusion model

Particle size (mm)	r ²	K (min ⁻¹)
0.417-0.294	0.9943	0.0164
bulk	0.9850	0.0116
>0.700	0.9978	8.8×10 ⁻³

TABLE 5. Lead uptake at various temperatures

Temperature (°C)	[Pb(II)] (mg·L ⁻¹)	[Pb(II)] (mg·g ⁻¹)
20	0.50	38.0
40	0.22	39.0
60	0.10	40.0

Time: 5 h. Stirring speed: 850 min⁻¹

procedure of plotting log D versus 1/T for ΔH° and ΔS° estimation, and the next equation: for ΔG° :

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (6)$$

The value of D, the lead distribution coefficient between the resin and the aqueous solution, was calculated as:

$$D = \frac{[\text{Pb}]_{r,e}}{[\text{Pb}]_{s,e}} \quad (7)$$

where, $[\text{Pb}]_{r,e}$ and $[\text{Pb}]_{s,e}$ are the lead concentrations at equilibrium in the resin and in the aqueous solution, respectively. The values of ΔH° , ΔS° and ΔG° are estimated as 33 kJ·mol⁻¹, 150 J·mol⁻¹ K and 11 kJ·mol⁻¹. The system is endothermic, whereas the negative ΔG° value indicated a spontaneous system, and the positive entropy change may be an indication of an increase of randomness at the solid-liquid interface during the exchange process.

The continuous variation of the aqueous pH from 5 to 1, indicated that, the metal uptake onto the resin decreases as the solution pH decreases (Table 6). This result is in accordance with Eq. (1), since the increase in the proton concentration in the aqueous phase resulted in a shifting of the equilibrium to the left, and thus, decreasing the removal of Pb²⁺ from the solution.

In order to compare the results obtained in the load of lead(II) onto the resin against the performance of another metal smart adsorbent, as multiwalled carbon nanotubes, experiments were carried out using doses of 0.25 g·L⁻¹ of the resin or the carbon nanotubes and aqueous solutions of 0.01 g·L⁻¹ Pb(II) at pH 5, 20 °C and stirring speeds of 850 min⁻¹. After 5 hours of contact, the metal uptake is of 38.0 mg·g⁻¹ and 0.92 mg·g⁻¹ for the resin and the multiwalled carbon nanotubes, respectively, thus, indicating that the resin performance is by far better than the nanotubes one.

Furthermore, the selectivity of the Pb(II)-Amberlite IR-120 system was tested against the presence of other metals in the aqueous solution. In this

TABLE 6. Influence of the aqueous pH on lead(II) uptake onto the resin

pH	[Pb(II)] (mg·g ⁻¹)
5	19.9
4	19.7
3	19.6
2	17.6
1	15.0

Aqueous solution: 0.01 g·L⁻¹ Pb(II) at various pH values. Resin dosage: 0.5 g·L⁻¹. Time: 5 h. Temperature: 20 °C. Stirring speed: 850 min⁻¹.

case, resin doses of $0.25 \text{ g}\cdot\text{L}^{-1}$ were used throughout the experiments, which were also conducted by the use of aqueous solutions containing lead plus other element (metal-binary solutions) at pH 5 and metals equimolar concentrations of $4.8 \times 10^{-5} \text{ M}$. The results from these series of experiments showed that:

- i) lead is loaded onto the resin averaging 85.1% of the initial lead concentration, independently of the accompanying metal in the solution, this resulted in a lead uptake of $34.0 \text{ mg}\cdot\text{g}^{-1}$ ($0.16 \text{ mmol}\cdot\text{g}^{-1}$),
- ii) the accompanying metals are loaded onto the resin at different percentages, with a maximum 99.9% for indium(III) and a minimum 78.1% for cobalt(II),
- iii) the presence of these metals in the solution, and their subsequent uptake onto the resin, resulted in the decrease of the lead uptake onto the resin from $38.0 \text{ mg}\cdot\text{g}^{-1}$ ($0.18 \text{ mmol}\cdot\text{g}^{-1}$) to $34.0 \text{ mg}\cdot\text{g}^{-1}$ ($0.16 \text{ mmol}\cdot\text{g}^{-1}$) for the monoelemental lead solution and the binary solutions, respectively.
- iv) Accordingly with the results presented in Table 7, lead(II) loads onto the resin preferably to Cu, Ni, Co and Mn ($\beta > 1$), but not to Zn and In ($\beta < 1$), being β , the separation factor, calculated as:

$$\beta = \frac{D_{\text{Pb}}}{D_{\text{Metal}}} \quad (8)$$

and the metals distribution coefficients, D , as in Eq. (7).

3.2. Lead(III) elution from the Pb^{2+} -loaded resin

The reversal of Eq. (1), that is, the shifting of the equilibrium to the left, had been investigated using acid solutions as eluent for lead.

TABLE 7. Percentage of metal uptake and separation factors

System	% uptake	$\beta_{\text{Pb/Metal}}$
Pb^{2+}	85.1	0.96
Zn^{2+}	85.2	
Pb^{2+}	85.1	1.4
Cu^{2+}	80.3	
Pb^{2+}	85.1	1.2
Ni^{2+}	82.2	
Pb^{2+}	85.1	$< 6 \times 10^{-3}$
In^{3+}	99.9	
Pb^{2+}	85.1	1.5
Co^{2+}	78.2	
Pb^{2+}	85.1	1.1
Mn^{2+}	83.1	

Temperature: 20 °C. Time: 5 h. Stirring speed: 850 min^{-1} .

By using resin loaded with $11 \text{ mg}\cdot\text{g}^{-1}$ lead, it was found that with 0.5–1 M HCl solutions the recovery of lead is around 99%, whereas if the eluent is of 0.25 M HCl, this yield decreased until 64%. The conditions used to obtain the above results were 20 °C, 1 h contact time and a ratio of 250 mL solution/mg resin.

The variation in the ratio volume of the eluent solution versus resin weight was also investigated in the removal of lead from lead-loaded resin. In this case, the resin was loaded with $11 \text{ mg}\cdot\text{g}^{-1}$ lead, and the eluent solution was of 0.25 M HCl. Table 8 resummed the results obtained from these set of experiments. In the case of 0.5 M HCl solutions, the percentage of lead elution is near quantitative, resulting in solutions with, i.e. $1.7 \text{ g}\cdot\text{L}^{-1}$ lead, which represented a concentration factor of 170 with respect to the initial lead concentration in the solution treated with the resin. Using 0.25 M HCl solutions, the results are different, since there is a decrease in the recovery yield as one decrease the volume of solution versus resin weight ratio, however, it can be obtained solutions with $0.03 \text{ g}\cdot\text{L}^{-1}$, which represented a concentration factor of 3 with respect to the initial lead-bearing aqueous solution ($0.01 \text{ g}\cdot\text{L}^{-1} \text{ Pb}^{2+}$) put into contact with the resin.

The influence of the temperature on lead elution was also investigated, and the results obtained are shown in Fig. 3. It is observed that the temperature has little effect in the final lead recovery, around 99%, but has a significant influence on the time which this yield is reached, i.e. 30 min at 60 °C, more than 2 h at 20 °C.

4. CONCLUSIONS

- The cation Pb^{2+} is effectively removed from aqueous solutions by the use of ion exchange technology using the cationic exchanger resin Amberlite IR-120. However, several variables influenced the respective exchange equilibrium:

- i) the resin dosage and the resin particle size. Increasing the resin dosage, the metal uptake

TABLE 8. Percentage of lead elution at various mL solution/mg resin ratios and HCl concentrations

mL solution/mg resin	0.25 M HCl	0.5 M HCl
2000	87	
1000	72	
500	70	
250	64	99
125		99
60		99

Temperature: 20 °C. Time: 1 h

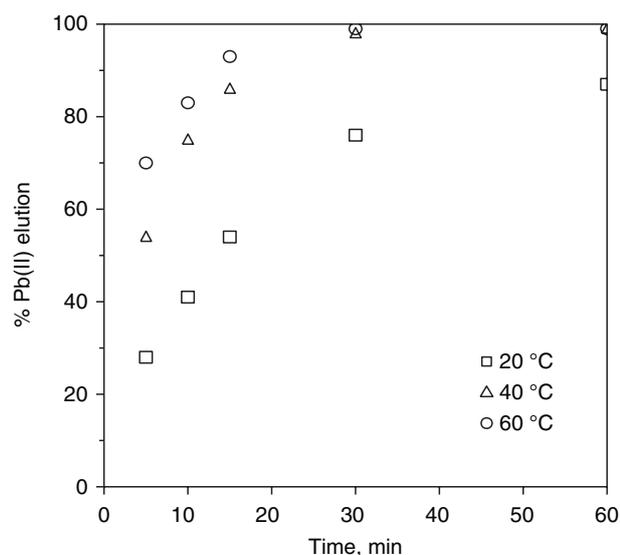


FIGURE 3. Influence of temperature on lead(II) elution. Aqueous solution: 0.25 M HCl. Resin loaded with 11 mg·g⁻¹ lead(II). Volume of the solution/ resin weight ratio: 2000. Stirring speed: 850 min⁻¹.

- decreases, whereas the metal uptake increases as the resin particle size decreases,
- ii) the temperature. Increasing the temperature from 20 °C to 60 °C, the metal uptake increases,
 - iii) the aqueous pH. A decrease of the aqueous pH value from 5 to 1 is accompanied with a decrease of the lead uptake onto the resin,
 - iv) the presence of other metals in cationic form in the aqueous solution. This presence led to a decrease in a regular lead uptake onto the resin, however, Pb²⁺ can be separate from other cations, except Zn²⁺ and In³⁺, using this resin.
- The uptake of lead onto the resin responded to the pseudo-first kinetic order model and to the Freundlich isotherm, whereas the variation in the resin particle size (0.294–0.700 mm) has not effect in the mechanism in which lead is loaded onto the resin, this uptake can be represented by the aqueous diffusion model. Lead is loaded onto the resin *via* and endothermic and non-spontaneous equilibrium.
 - The loading of Pb²⁺ onto the resin competed favourably with respect to the adsorption of this cation onto multiwalled carbon nanotubes.
 - Lead can be recovered from Pb²⁺-loaded resin by the use of acidic solutions and under various experimental conditions. Concentration factors of 170 can be reached using 0.5 M HCl solutions as eluent for Pb²⁺.

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