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Sono-sorption as a new method for the removal of lead ion from aqueous solution

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Abstract

Sorption of Pb(II) as a toxic heavy metal ion, onto styrene-divinylbenzene co-polymer resin in the presence of ultrasound (sono-sorption) and in its absence (classical method) is presented in this work. Batch experiments were conducted to study the effect of the main parameters such as the amount of sorbent, concentration of sorbate, contact time, intensity of ultrasound and temperature on this sorption process. The equilibrium between liquid and solid phase was described by Langmuir model better than the Freundlich model. The time to reach equilibrium especially in the presence of ultrasound was very fast. This indicates that the resin mentioned has a high potential for the sorption of this pollutant ion. The isotherm constants were obtained under several different conditions. These constants were used in the calculation of the thermodynamic parameters of sorption. According to the results, the kinetics of sorption was in agreement with both pseudo-first-order and first-order reversible models. © 2006 Elsevier B.V. All rights reserved.

Keywords: Ultrasound; Lead ion; Resin; Sorbate; Sorbent; Langmuir model

1. Introduction

Lead, one of the high priority toxic pollutants, can reach the environment from various anthropogenic sources as well as from natural geochemical processes. It can accumulate in the environment and it is not amenable to biological degradation [1,2]. This can lead to the transport of lead ion to the body and therefore, threaten human life health owing due to its well-known toxicity. As well as in point of size and charge similarities, lead ion can substitute for of calcium ion in the bones of body [3]. As children require high calcium ion levels for development of their skeletal systems, they are more susceptible to lead ion accumulation. Stored lead ion in the bones is not harmful but, if high levels of calcium ion are ingested later, the lead ions in the bone maybe replaced by calcium ions and mobilized. Once it is free in the system, lead may cause nephrotoxicity, neurotoxicity, and hypertension [3]. Unlike organic compounds, lead ion is not biodegradable and the EC and WHO limit for lead in drinking water is 50 μ g dm⁻³ [4,5], therefore it may need to be removed from water.

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The aim of this study was focused on the removal of lead ion from aqueous solution by the supplied resin in the presence and absence of ultrasound. There are several methods for removing lead contaminant from effluents such as ion exchange, chemical precipitation, oxidation and reduction, reverse osmosis and sorption methods [6–12]. In the last method, different sorbents have been used such as fly ash [13], activated carbon [14], natural surface coating [15], sea nodule [16], chitosan [17–19], zeolites [20–22], clay [23–25], natural oxide [26,27], industrial waste [28], iron-coated sand [29], and some biosorbents [30–32].

The styrene-divinylbenzene co-polymer (Fig. 1) has been widely used as molecular sieve, cation-exchanger, and sorbent [33]. Generally in the preparation process, styrene and divinylbenzene as monomers, which are insoluble in water, are mixed in a suspension system by an impeller at a speed that breaks the matrix into small spheres. These spheres eventually harden, forming spherical plastic beads. The beads are then treated with sulfuric acid so the cation resin is produced in its hydrogen form. This form of resin neutralized to the sodium form by passing caustic solution though the resin. The final step involves cleaning by washing with hot and steaming the resin. This process reduces the amount of organic compounds left in the resin from the manufacturing process.

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Fig. 1. Styrene-divinylbenzene co-polymer, X = ionogenic/functional group.

2. Experimental

2.1. Materials

Lead nitrate was obtained from Merck in analytical grade. Stock solutions were prepared using lead nitrate in de-ionized water. The resin used was a co-polymer of styrene and divinyl-benzene with functional groups of SO₃–Na produced by LEWATIT, Bayer AG, Leverkusen (bulk density = 1.28 (approximately) g/ml, bulk weight (820 g/l), bead size = 0.6μ m) [34].

2.2. Apparatus

The ultrasonic irradiations were carried out by equipment operating at 20 kHz with standard horn (XL-2020, Misonix). Ultrasonic energy dissipated in the reactor was 55.9 and 55.7 W through the calorimetric method for the solution with and without resin, respectively. Conventional experiments were carried out with a Teflon-coated magnetic bar rotating at 300 rpm by Griffin (Made in England). A Varian atomic absorption spectrophotometer (spectra-110-220/880 Australia Pty. Ltd.) equipped with a Zeeman atomizer was used to determine the concentration of lead ions in the solution.

2.3. Procedure

The ultrasonic horn was inserted to the Rosett cell containing 50 ml solution of pollutant with concentration of 100 mg l^{-1} (C_0) to a depth of 10 mm. A circulating bath was used to maintain the temperature constant during the sonication. Experiments without ultrasound (referred to as 'conventional' experiments) were performed in a batch reactor with continuous stirring fixed at 300 rpm. The concentration of the lead was determined at intervals by atomic absorption spectrophotometer.



Fig. 2. Effect of contact time on the sorption of lead at different temperatures (stirring speed, 300 rpm; C_0 , 100 mg l⁻¹; dose of sorbent, 0.3 g; acoustic power at position 25%, 55.9 W).

3. Results and discussion

3.1. Effect of contact time

The lead ion concentration of solution versus contact time for a given concentration of sorbent (0.3 g) was shown in (Fig. 2) at different temperatures. The initial concentration was $100 \text{ mg } l^{-1}$ and the removal of lead ion was faster in the presence of ultrasound than its absence. As shown in this figure, the amount of sorption of Pb(II) onto the resin increased rapidly at the beginning and then reached equilibrium. The sorption capacity of resin increased with increasing the temperature. This indicates that the sorption process is endothermic in nature and that is the reason for higher sorption at higher temperatures. In the presence of ultrasound, the lead is almost completely removed in 3 min at 50 °C. This short time indicates a high interaction of resin with pollutant especially in the presence of ultrasound. It is assume that the shear forces generated in the medium due to the cavitation process may be the primary pathway for the morphology and the surface area changes of sorbent, which lead to higher removal rate.

3.2. Effect of sorbent dose

The effect of sorbent dose on the removal of lead ion was shown in (Fig. 3). The sorption increased with increasing the amount of sorbent from 0.1 to 0.3 g in both methods. It is clear that with increasing the amount of sorbent, the available sites for the sorbtion are increased. Therefore, at constant concentration of pollutant the rate of sorption was greater at higher sorbent dose. As it is shown, the presence of ultrasound for both amount of sorbent was more effective than without it. The initial slope of curves for the case of sonication was higher than conventional method. This is related to the higher mass transfer in the presence of ultrasound due to the cavitation process. Ultrasonic waves



Fig. 3. Effect of sorbent dose on the sorption of lead (stirring speed, 300 rpm; C_0 , 100 mg l⁻¹; t, 35 °C; dose of sorbent, 0.3 g; acoustic power at position 25%, 55.9 W).

removed completely the pollutant in ten minutes with higher dose of sorbent.

3.3. Effect of intensity of ultrasound irradiation

The effect of intensity on the removal of lead ion was shown in (Fig. 4). The sorption increased with increasing the intensity from 20% to 25%. The results indicated that at two selected intensities, the higher one was more effective and after 10 min the pollutant has removed completely from the solution. This could be explained that at higher intensities, there are more bubbles and also the bubbles collapse more violently. This behavior can lead to higher effects on the sorption process.

3.4. Sorption isotherm

The Langmuir isotherm has been used by various workers for the sorption of variety of compounds. The linear form of



Fig. 4. Effect of ultrasound intensity on the sorption of lead (C_0 , 100 mg l⁻¹; t, 35 °C; acoustic power at position 25%, 55.9 W; at position 20%, 40.9 W).



Fig. 5. Langmuir isotherms in the presence and absence of ultrasound.

Langmuir isotherm is given by the following equation:

$$\frac{1}{q_{\rm e}} = \frac{1}{q_{\rm m}} + \frac{1}{bq_{\rm m}c_{\rm e}}\tag{1}$$

where q_e is the amount of sorbed species at equilibrium $(mg g^{-1}), c_e$ the equilibrium concentration of the sorbate in solution (mg l^{-1}), q_m and b are the Langmuir constants related to the maximum capacity of sorption and energy of sorption, respectively. When $1/q_e$ was plotted against $1/c_e$, straight lines with the slope of $1/bq_m$ were obtained (Fig. 5), which showed that the sorption of lead ion is consistent with the Langmuir isotherm. The Langmuir constants, b and q_m , were derived from Fig. 5 and the values are given in Table 1. As shown in this table, the maximum capacity was higher in the presence of ultrasound than conventional method. This indicated that under sonication, new sites of sorption were appeared by disruption of sorbent particles. The sorption data for lead ion were also analyzed by Freundlich model, but the data better fit the Langmuir model. Due to the high sorption of the mentioned lead at low concentrations, the sorption isotherms were determined at different temperatures with concentrations in the range of $500-1500 \text{ mg } \text{l}^{-1}$. In all experiments, the amount of resin was fixed at 0.05 g of resin per liter of solution. Under this condition, there was a remaining concentration of lead in the solution at equilibrium.

3.5. Thermodynamic parameters of sorption

According to the following equation, it is possible to calculate Gibbs energy changes for the sorption process at

 Table 1

 Langmuir constants for the uptake of lead ion

<i>T</i> (°C)	Ultras	sound		Classic			
	$\overline{R^2}$	$q_{\rm m} ({\rm mg}{\rm g}^{-1})$	$b (l mg^{-1})$	R^2	$q_{\rm m} ({\rm mg}{\rm g}^{-1})$	$b (lmg^{-1})$	
32	0.98	436.68	6.80e-3	0.99	431.03	6.30e-3	
44 54	0.98 0.99	471.70 502.51	9.50e-3 9.60e-3	0.99 0.99	446.42 474.60	6.60e-3 7.30e-3	

<i>T</i> (°C)	Ultrasound			Classic			
	$-\Delta G^{\circ} (\mathrm{kJ}\mathrm{mol}^{-1})$	$\Delta H^{\circ} (\mathrm{kJ}\mathrm{mol}^{-1})$	$\Delta S^{\circ} (\mathrm{J} \mathrm{mol}^{-1})$	$-\Delta G^{\circ} (\text{kJ mol}^{-1})$	$\Delta H^{\circ} (\mathrm{kJ}\mathrm{mol}^{-1})$	$\Delta S^{\circ} (\text{J mol}^{-1})$	
32	18.41			18.21			
44	20.00	13.15	103.71	19.02	5.18	76.53	
54	20.66			19.90			

 Table 2

 Thermodynamic parameters for the uptake of lead ion

different temperatures:

$$\Delta G^{\circ} = -RT\ln b \tag{2}$$

The enthalpy and entropy changes of sorption can be determined through the slope and intercept of the line produced by drawing ln b versus 1/T (Van't Hoff equation). Table 2 summarizes these parameters in the presence and absence of ultrasound. According to Table 2, ΔG is negative for all cases which mean that the sorption of lead by the resin is spontaneous. The entropy of sorption was increased in the presence of ultrasound. It is well known that ultrasound can disrupt the sorbent particles and changed the morphology of the surface which can lead to the higher entropy of sorption. The enthalpy of sorption in the presence of ultrasound was also higher than in the conventional experiments. This could be explained by the emerging of new sites in the case of sonication. In other words, ultrasound increased the entropy changes of sorption more than conventional one and in approximate constant Gibbs energy changes, this can leads to higher enthalpy changes in the presence of ultrasound.

3.6. Sorption kinetics

3.6.1. Pseudo-first-order model

In order to determine the sorption kinetics of Pb(II), the pseudo-first-order kinetic model was checked according to the Lagergren equation [35]:

$$\frac{\mathrm{d}q_t}{\mathrm{d}t} = k_{1,\mathrm{ad}}(q_\mathrm{e} - q_t) \tag{3}$$

where q_e and q_t are the amounts of metal ions sorbed onto the resin (mg g⁻¹) at equilibrium and time *t*, respectively, and $k_{1,ad}$ is the rate constant of pseudo-first-order (min⁻¹). After integration under the boundary conditions, Eq. (3) changes to

$$\log(q_{\rm e} - q_t) = \log q_{\rm e} - \frac{k_{1,\rm ad}}{2.303}t$$
(4)

The plot of $\log(q_e - q_i)$ versus time in Fig. 6 showed a linear function. The slopes and intercepts of these lines were used to determine the pseudo-first-order rate constant $k_{1,ad}$ and the capacity of sorption at equilibrium (q_e) . The values of these parameters and the deviation (%) from the experimental values of q_e are presented in Table 3. The rate constant was higher in the presence of ultrasound than conventional method. This mostly related to the higher mass transfer and more contact of pollutant with sorbent sites which lead to the faster removal of lead ion from solution.



Fig. 6. Pseudo-first-order kinetic model at different temperatures.

3.6.2. First-order reversible model

It is well established that the sorption of ions from aqueous solution when a single species is contacted with a heterogeneous surface follows reversible first-order kinetics [36]. The sorption of lead from the liquid phase to the solid phase may be expressed as

$$A\frac{k_1}{k_2}B\tag{5}$$

Table 3

Pseudo-first-order	kinetic mo	del for the	sorption of	lead ion
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$I(^{\circ}C)$	Ultra	Ultrasound						
	R^2	$k_{1,ad} (min^{-1})$	$q_{\rm e,exp} \ ({\rm mg} \ {\rm g}^{-1})$	$q_{\rm e,cal} ({\rm mg}{\rm g}^{-1})$	Deviation (%)			
18	0.99	0.38	16.31	19.94	1.81			
35	0.99	0.51	16.31	16.69	0.19			
55	0.99	1.02	16.31	20.01	1.85			
	7) Classic							
$T(^{\circ}C)$	Class	ic						
<i>T</i> (°C)	$\frac{\text{Class}}{R^2}$	ic $K_{1,ad} \ (\min^{-1})$	$q_{\rm e,exp} ({\rm mg}{\rm g}^{-1})$	$q_{\rm e,cal} ({\rm mg}{\rm g}^{-1})$	Deviation (%)			
$\frac{T(^{\circ}\mathrm{C})}{18}$	$\frac{\text{Class}}{R^2}$ 0.88	ic $K_{1,ad} (min^{-1})$ 0.13	$q_{\rm e,exp} \ ({\rm mg \ g}^{-1})$ 15.57	$q_{\rm e,cal} ({\rm mg}{\rm g}^{-1})$ 13.09	Deviation (%) 1.24			
$\frac{T(^{\circ}C)}{18}$	$ \begin{array}{c} \text{Class}\\ \hline R^2\\ \hline 0.88\\ 0.99\\ \end{array} $	ic $K_{1,ad} (min^{-1})$ 0.13 0.19	<i>q</i> _{e,exp} (mg g ⁻¹) 15.57 16.31	$q_{\rm e,cal} ({\rm mg}{\rm g}^{-1})$ 13.09 15.25	Deviation (%) 1.24 0.53			
<i>T</i> (°C) 18 35 55	$ \begin{array}{c} \text{Class}\\ \hline R^2\\ \hline 0.88\\ 0.99\\ 0.97\\ \end{array} $	ic $K_{1,ad} (min^{-1})$ 0.13 0.19 0.60	<i>q</i> _{e,exp} (mg g ⁻¹) 15.57 16.31 16.31	<i>q</i> _{e,cal} (mg g ⁻¹) 13.09 15.25 21.91	Deviation (%) 1.24 0.53 2.80			

Table 4 First-order reversible kinetic model for the sorption of lead ion

<i>T</i> (°C)	Ultrasour	Ultrasound				Classic			
	$\overline{R^2}$	$k (\min^{-1})$	$k_1 ({\rm min}^{-1})$	$k_2 ({\rm min}^{-1})$	$\overline{R^2}$	$k (\min^{-1})$	$k_1 ({\rm min}^{-1})$	$k_2 (\min^{-1})$	
18	0.99	0.362	0.359	1.30e-4	0.94	0.151	0.105	4.50e-2	
35	0.99	0.500	0.499	1.80e-4	0.99	0.200	0.199	3.67e-4	
55	0.98	0.980	0.979	3.60e-4	0.98	0.405	0.404	1.48e - 3	

where k_1 and k_2 are the rate constants for the sorption and desorption, respectively. If the initial concentration of lead ion is *a* and the transfered amount of lead ion from liquid phase to solid phase at time *t* is *x*, then the rate can be expressed as

$$\frac{\mathrm{d}x}{\mathrm{d}t} = k_1(a-x) - k_2 x \tag{6}$$

The amount of lead ion sorbed at equilibrium, X_e , can replace x so that

$$k_1(a - X_e) - k_2 X_e = 0 \tag{7}$$

$$K_{\rm C} = \frac{X_{\rm e}}{a - X_{\rm e}} = \frac{k_1}{k_2}$$
 (8)

where $K_{\rm C}$ is the equilibrium constant. The above rate equation can be reformulate by putting (7) into (6):

$$\frac{\mathrm{d}x}{\mathrm{d}t} = [k_1(a-x) - k_2 x] - [k_1(a-X_{\mathrm{e}}) - k_2 X_{\mathrm{e}}] \tag{9}$$

$$\frac{dx}{dt} = (k_1 + k_2)(X_e - x)$$
(10)

Therefore

$$k_1 + k_2 = \frac{1}{t} \ln \frac{X_e}{X_e - x} \tag{11}$$

$$\ln \frac{1}{1 - (x/X_e)} = (k_1 + k_2)t \tag{12}$$

$$-\ln\left(1-\frac{x}{X_{\rm e}}\right) = (k_1+k_2)t\tag{13}$$

The last equation can be written in the form

$$\ln(1 - U_t) = -(k_1 + k_2)t = -kt \tag{14}$$

where $U_t = x/X_e$ and k is the overall rate constant:

$$k = k_1 + k_2 = k_1 + \frac{k_1}{K_{\rm C}} = k_1 \left(1 + \frac{1}{K_{\rm C}}\right) \tag{15}$$

 U_t can be calculated using the following expression:

$$\frac{C_{A(0)} - C_{A(t)}}{C_{A(0)} - C_{A(e)}} = \frac{x}{X_e} = U_t$$
(16)

where $C_{A(0)}$, $C_{A(t)}$ and $C_{A(e)}$ are the concentrations of lead ion at zero time, at time *t*, and at equilibrium, respectively. U_t is called fractional lead attainment of equilibrium [36]. This parameter was calculated by lead sorption over the resin in a given range of time from 30 s to 20 min.

The first-order reversible plots were presented in Fig. 7 by drawing $ln(1 - U_t)$ versus time. The overall rate constant (k)



Fig. 7. First-order reversible kinetic model at different temperatures.

was determined from the slope of the line and combined with equilibrium constant to calculate the forward and backward rate constant k_1 and k_2 . The rate constants and its correlation coefficients were given in Table 4. According to this table, k_2 is much smaller than k_1 which means that the desorption is negligible with compared to adsorption. This behavior is in agreement with pseudo-first-order model.

4. Conclusion

The present study clearly shows that the resin under study is an effective sorbent for lead ion removal from aqueous solution. This removal was enhanced in the presence of ultrasound. The shear forces generated during the cavitation process appear to be mostly responsible for this enhancement. Sorption capacity and sorption rate are strongly dependent on the applied conditions such as temperature, dose of sorbent, concentration of sorbate and ultrasonic intensity. The Langmuir model fitted the experimental data better than Freundlich model. The kinetics of sorption was obeyed the pseudo-first-order and first-order reversible models.

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