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Sorption of hexavalent chromium metal onto Amberlite IRA 410 – equilibrium isotherms and kinetic studies

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ABSTRACT

The removal of chromium (VI) from aqueous solution by a strong anion exchanger Amberlite IRA 410 was studied. Batch mode experiments were conducted to study the effect of the initial concentration of Cr (VI) and the equilibrium isotherms. The sorption process of chromium (VI) was tested with Freundlich, Langmuir and Khan models, and the results showed that sorption behaviour of chromium (VI) followed a Langmuir isotherm, namely a monolayer sorption onto the resin surface. The sorption capacity was determined to be 153.8 mg/g. Elovich, Ritchie and the pseudo-second order models were tested to represent kinetic data and the equation parameter values were evaluated. It was found that the sorption kinetics followed a pseudo-second order model in the concentration range 0–100 mg/l, while above 100 mg/l the Ritchie model matched experimental kinetic data. In addition, the capacity of sorption increased for increasing initial Cr(VI) concentration. The thermodynamic parameters for the sorption process have been evaluated. The entropy change ΔS was found to be 318.4 J/K/mol, the heat of adsorption (enthalpy change) ΔH was 85.2 kJ/mol indicating the endothermic nature of the adsorption process, and a decrease of the Gibbs free energy (ΔG) for increasing temperatures indicated the spontaneous nature of the process.

Keywords: Sorption; Anion exchanger; Sorption isotherms; Batch kinetics; Kinetic models; Diffusion; Mass transfer coefficient

1. Introduction

Chromium is among the contaminants, which exists in hexavalent and trivalent forms. The hexavalent form is more toxic than the trivalent one and significant exposure to Cr(VI) causes cancer in digestive tract and Lungs and may cause epigastric, pain, nausea, vomiting, severe diarrhea and hemorrhage [1,2]. The maximum level allowed for trivalent chromium in wastewater is 5 mg/l [3]; while the permissible limit of Cr (VI) in potable water is 0.05 mg/l, but industrial and mining effluents can be discharged with 0.5 mg/l [4]. It is therefore essential to remove Cr (VI) from wastewater disposal. The main sources of chromium (VI) are leather tanning processes, electroplating, manufacturing of dye, paint and paper etc [5]. Since most of the industrial and mine effluents contain higher than the permissible limit, treatment to reduce/remove the pollutant before discharge into the environment becomes inevitable.

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Several technologies have been tested for toxic ion removal or to reduce the concentration of metal ion, such as chemical precipitation, reverse osmosis, oxidation, electrocoagulation, electrodialysis and sorption [6–9]. However, most of these technology processes have significant disadvantages, including incomplete metal and monitoring systems high reagent or energy requirements or generation of toxic sludge or other waste products that require disposal [10]. The selection of the wastewater treatment method is based on the concentration of waste and the cost of treatment. Adsorption processes have increasingly received attention in environmental treatment applications in recent years, including chromium removal [11–13]. Among this class of processes, ion exchange received a particular attention, since the method is simple, relatively low-cost and effective in removing heavy metal ions [14,15]. There are different natural and synthetic products that show ion exchange properties. Among them, anion exchange resins are used to remove Cr(VI) from synthetic wastewater. The main advantages of using these resins are due to their high chemical and mechanical stability, high ion exchange capacity and ion exchange rate. Another advantage is the possibility of selecting the fixed ligand groups and the degree of cross-linking. Ion exchange resins are suitable for metal-ion complexation because of their hydrophobicity and high selectivity. Anion exchange resins are generally prepared from synthetic polymers such as styrene–divinylbenzene copolymers, which are aminated and methylated to form strongly base anion exchangers. Removal of Cr(VI) from cooling water and groundwater by ion exchange has been investigated by some researchers [16,17]. Weak-base anion exchange resins are generally used for removing chromates from water at acidic pH values [18–20].

Amberlite IRA 410, used in this work, is a strong anion exchanger in chloride form; it was tested at various solution pH and high concentrations. The main objective of this study was to investigate the equilibrium and kinetic parameters of this ion exchange resin and compare its behaviour to those of other ion exchange resins. The Langmuir, Freundlich and Khan isotherms were used to fit the equilibrium isotherms. Kinetics parameters were also evaluated; in this aim several kinetic models were selected to determine the main mechanism predominates in chromium sorption by Amberlite IRA 410.

2. Materials and methods

2.1. Sorbent

The following anion exchange resin Amberlite IRA 410 was used in this study. Amberlite IRA 410 is a strong anion exchanger in chloride form. Its physicochemical properties and specifications are shown in Table 1. Before use, the resin was washed with distilled water to remove impurities. It was then dried at 50°C for 24 h and then sieved to obtain the desired particle size (375–825 μm).

Table 1
Characteristics of Amberlite IRA410[1]

Polymer matrix	Quaternary polystyrene ammonium
Functional group	$–N^+ (CH_3)_2C_2H_4OH$
Ionic form	$Cl^–$
Physical aspect	Pale yellow transparent ball
Exchange capacity	3 eq/kg of dry resin
Retention of moisture	45–51%
Density	1085–1115
Granulometry	0.3–0.9 mm
Coefficient of uniformity	<1.6

[1] Information provided by the manufacturer (Merck, Darmstadt, Germany).

2.2. Analyses of chromium (VI) ions

An aqueous stock solution (1000 mg/l) of Cr(VI) ions was prepared using $K_2Cr_2O_7$ salt. The concentration of residual chromium (VI) ions was read spectrophotometrically (Jenway UV-Vis spectrophotometer) by using diphenylcarbazide as a complexing agent. The absorbance of the purple colored solution was read at 540 nm after 5 min. Balasubramanian and Pugalenthi [21] compared different analytical methods and found that UV-visible spectrometry analytical technique was more efficient than more complex methods like FAAS (flame atomic absorption spectrometry) and ICP-AES (inductively coupled plasma-atomic emission spectrometry) methods, especially for solutions containing relatively high chromium concentrations. The detection limit of the UV-visible spectrometry (1,5-diphenyl carbazide) method was about 5 μg/l.

2.3. Isotherm experiments

Sorption isotherm studies were carried out in a series of 400 ml glass flasks. Each flask was filled with 200 ml of different initial Cr(VI) concentrations (in the range 50–200 mg/l), while maintaining the resin dosage at a constant level. The glass flasks were then shaken at 300 rpm to maintain resin particles in suspension. After equilibrium, the solution was separated and analysed. Preliminary tests showed that the sorption was complete after 120 min. Experiments were mainly carried out without initial adjustment of the pH. Preliminary tests showed that the solution pH did not change significantly during experiments, remaining close to its initial value (5.2). All experiments were conducted in duplicate and average values were considered.

The sorbed amount was deduced from material balance:

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

where q_e was the amount of Cr(VI) sorbed per gram of resin (mg/g) at equilibrium, V was the volume of solution (l), m was the mass of resin (g), and C_0 and C_e were respectively the initial and the equilibrium concentrations in solution (mg/l).

2.4. Kinetic experiments

Sorption rate studies were carried out in a stirred batch reactor. The geometry of the reactor was standardized according to the standards' suggested by [22]. A 1 g of resin were exactly weighed and added into 1 l of solution at the required concentration. Experiments were mainly carried out without initial adjustment of the pH. The pH of the aqueous solutions was approximately 5.2 and did not varied significantly during the sorption. The solution was stirred with a Ruston impeller driven by a motor equipped with a speed controller at 400 rpm to completely suspend the resin particles. Fixed volume samples of 2 ml were taken at different time intervals and filtered through filter paper. The filtrate was analysed for residual chromium concentration in the solution. The amount sorbed was calculated from:

$$q_t = \frac{V(C_0 - C_e)}{m} \tag{2}$$

where q_t was the amount of Cr(VI) sorbed per gram (mg/g) and C_t was the concentration in solution at a given time t (mg/l).

All experiments were conducted in duplicate and average values were considered.

3. Results and discussion

3.1. Sorption isotherms

Analysis of the isotherm data is important to develop an equation that correctly represent the results and which could be used for design purposes [23]. Freundlich, Langmuir, and Khan isotherms are often used to describe experimental isotherm data.

The Freundlich [24] isotherm model was also used to explain the observed phenomena. The Freundlich isotherm is represented by the following equation:

$$q_e = K_f C_e^{1/n} \tag{3}$$

K_f (mg/g)(l/mg)$^{1/n}$ is the Freundlich sorption constant and *1/n* is a measure of the sorption intensity [25]. The linearized form of Freundlich sorption isotherm was to evaluate the sorption data and is represented as:

$$\log q_e = \log K_f \frac{1}{n} \log C_e \tag{4}$$

The Langmuir [26] isotherm is valid for monolayer sorption onto a surface containing a finite number of identical sites. The Langmuir isotherm is represented by the following equation:

$$\frac{C_e}{q_e} = \frac{1}{q_0} C_e + \frac{1}{q_0 b} \tag{5}$$

Khan et al. [27] have compared different types of isotherms to describe their experimental data and concluded that the generalized equation was the most relevant to represent experimental data. The Khan isotherm is given by:

$$q_e = \frac{q_0 b_K C_e}{(1 + b_K C_e)^{a_k}} \tag{6}$$

q_o (mg/g) is the maximum adsorption capacity, b_K the adsorption affinity (l/mg) and a_K the heterogeneity parameter.

An error function should be defined in order to evaluate the relevance of the fit of the isotherm to the experimental equilibrium data. The experimental data were analyzed by the Chi-square test to compare the models. The statistic Chi-square test, namely the sum of the squares of the differences between experimental and calculated data divided by the corresponding calculated data, is given by the following mathematical statement:

$$\chi^2 = \sum \frac{(q_{\exp} - q_{cal})^2}{q_{cal}} \tag{7}$$

and the corresponding results were presented in Table 2.

The linear regression coefficient in Table 2 shows that, the Freundlich isotherm failed to describe experimental sorption data of chromium (VI) sorbent systems,

Table 2
Isotherm parameters for removal of hexavalent chromium by Amberlite IRA 410 resin

Isotherm type	Constants				
Langmuir isotherm	q_o (mg/g)	b (l/mg)		R^2	χ^2
	153.8	0.9		0.99	4.31
Freundlich isotherm	K_f	*1/n*		R^2	χ^2
	76.67	4.35		0.78	18.71
Khan isotherm	q_o	b_K	a_K	R^2	χ^2
	212.8	0.509	1.109	0.97	2.60

while, the Langmuir and Khan isotherms seemed to agree with experimental data of chromium (VI) considering that the linear regression coefficients were above 0.97. The error function χ^2 was also in agreement with this conclusion, since the Freundlich isotherm led to a significantly higher error (Table 2). This can be also observed at the examination of Fig. 1, since both Lagmuir and Khan models matched experimental data. The maximum adsorption capacity of Amberlite IRA 410 was compared with the literature values of others adsorbents for chromium and the corresponding values are collected in Table 3. As observed, Amberlite IRA 410 shows a remarkable capacity for chromium sorption, compared to most of the other adsorbents.

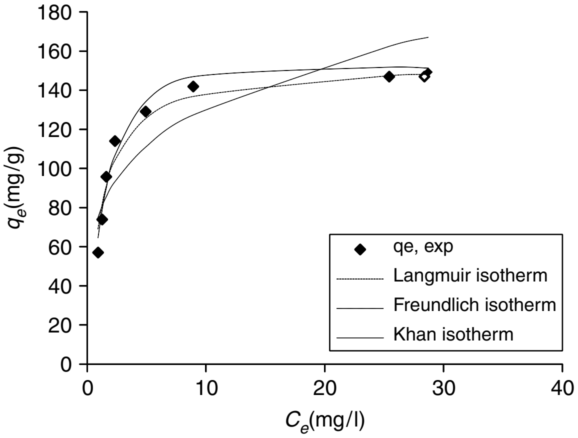

Fig. 1. Comparison of the various sorption isotherms for Cr(VI) ion removal.

Table 3
Comparison of chromium adsorption capacity of Amberlite IRA 410 with others resins

Adsorbent	pH	q_o (mg/g)	Ref.
XSD-296	3	235	[28]
Amberlite IRA 96	3	53.36	[19]
Dowex 1x8	3	63.8	[19]
Lewatit MP500	6	39.44	[18]
Lewatit MP64	5	34,8	[18]
D301	1	151.5	[20]
D314	3	120.5	[20]
D354	4.5	156.25	[20]
Amberlite 1200H	3	84.04	[29]
Amberlite IRN97	3	58.14	[29]
Lewatit M616	5	47.56	[30]
Lewatit62	5	46.4	[30]
Amberlite IRA410	5.2	153.8	This work

3.2. Sorption kinetic model

Kinetics of metal sorption governs the rate, which determines the residence time, one of the main factors characterizing the efficiency of an adsorbent. The Kinetics of sorbate uptake is required to select the optimal operating conditions for full-scale batch process. Kinetics parameters, which are helpful to predict the sorption rates, give important information for designing and modelling the processes [31]. Determination of the kinetic parameters and explanation of the mechanism in heterogeneous systems is often a complex procedure, as surface effects can be superimposed on chemical effects [32]. Three kinetic models, Ritchie, Elovich and pseudo-second order equations were tested to describe experimental sorption kinetic data.

3.2.1. Elovich equation

The Elovich equation is given as follows:

$$\frac{\mathrm{d}q_t}{\mathrm{d}t} = \alpha \cdot e^{-\beta q_t} \tag{8}$$

where α is the initial rate (mg/g.min) since $\left(\frac{\mathrm{d}q_t}{\mathrm{d}t}\right)$ approaches when approaches zero, and the parameter is related to the extent of surface coverage and activation energy for chemisorptions (g/mg). Given that $q_t = 0$ at $t = 0$, integrated form of Eq. (10) is:

$$q_t = \frac{1}{\beta}\ln(t + t_0) - \frac{1}{\beta}\ln t_0 \qquad \text{where } t_0\ \frac{1}{\alpha\beta} \tag{9}$$

If t is much larger than t_0 Eq. (11) can be simplified as:

$$q_t = \frac{1}{\beta}\ln(\alpha\beta) + \frac{1}{\alpha\beta}\ln(t) \tag{10}$$

Thus, the constant can be obtained from the slope and intercept of a straight line plot of q_t versus t.

3.2.2. Ritchie equation

Ritchie proposed a method for the Kinetic sorption of gases on solid. If metal ion sorption medium is considered to be a second order reaction, Ritchie equation is:

$$\frac{1}{q_t} = \frac{1}{K_1 q_e t} + \frac{1}{q_e} \tag{11}$$

where K_1 (g/mg/min) is the rate constant of the second order sorption, and q_e and q_t denote the amounts of sorption at equilibrium and at a given time t (mg/g), respectively.

3.2.3. Pseudo-second order model

The pseudo-second order model is in the following form:

$$\frac{\mathrm{d}q}{\mathrm{d}t} = K_2(q_e - q_t)^2 \tag{12}$$

where K_2 is the rate constant of the pseudo-second order equation (g/mg/min), q_t and q_e (mg/g) are the amounts of sorption at a given time t (min) and at equilibrium (mg/g). Integration leads to:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \tag{13}$$

Thus, metal uptake can be expressed as a function of time, for pseudo-second order model, using Eq. (16):

$$q_t = q_e \frac{q_e K_2 t}{1 + q_e K_2 t} \tag{14}$$

3.2.4. Effect of the initial concentration

The initial chromium (VI) concentration was varied in the range of 10–200 mg/l. The sorption capacity (mg/g) as a function of the sorption time (0120 min) for different initial chromium concentrations is plotted in Fig. 2. The figure shows that the amount sorbed at a given time t increased as the initial concentration in the solution increased. The sorption of Cr(VI) by IRA410 appeared rapid in the early period, while it slowed down at the late period of the sorption process. A higher initial concentration provides an important driving force to overcome all mass transfer resistances of the pollutant between the aqueous and solid phases, resulting in a higher sorption capacity. Fig. 3 also presents a simulation of the Elovich, the Ritchie and the pseudo-second order equations. The amount of sorption equilibrium q_e, the constant, α, β, K_2, q_{e,c_2}, K_1 and q_{e,c_1}, of the equations and the regression coefficient R^2 are shown in Table 4.

The correlation coefficient R^2 (Table 4) and Fig. 2 show that the Ritchie model and the Pseudo-second order model matched experimental data. However, the values of q_e obtained from the pseudo-second order model were closed to those experimentally determined; hence the sorption of Cr(VI) on ion exchange resin Amberlite IRA 410 followed a pseudo-second order model. From Table 4, it was also observed that for increasing initial Cr(VI) concentrations from 10 to 100 mg/l, the pseudo-second order rate constant (K_2) decreased, while the initial sorption rate increased.

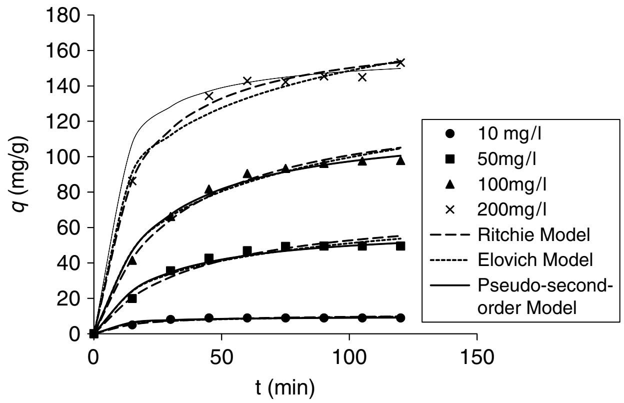

Fig. 2. Effect of the initial Cr(VI) concentration on the sorption of Cr(VI).

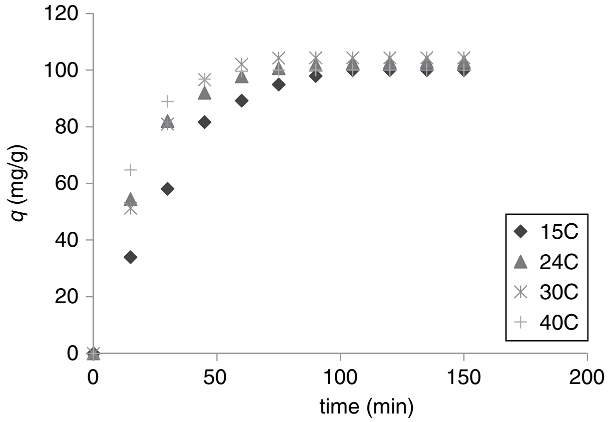

Fig. 3. Effect of the temperature on the sorption of Cr (VI).

Table 4
Parameter values recorded from fitting of sorption kinetic data with Elovich equation, Ritchie model and pseudo-second order model

C_0 (mg/l)	$q_{e,exp}$ (mg/g)	Elovich equation				Ritchie model			Pseudo-Second order model		
		α (mg/g.min)	β (g/mg)	t_0 (min)	R^2	q_{e,c_1} (mg/g)	K_1 (g/mg/min)	R^2	q_{e,c_2} (mg/g)	K_2 (g/mg/min)	R^2
10	8.96	4.52	0.6	0.368	0.71	11.04	0.060	0.89	9.68	0.0135	0.99
50	49.52	5.09	0.07	2.806	0.91	72.99	0.026	0.96	60.97	0.00072	0.98
100	97.80	10.00	0.036	2.777	0.95	133.33	0.031	0.98	120.48	0.00035	0.99
200	153.1	41.90	0.033	0.723	0.92	172.41	0.067	0.98	158.73	0.00088	0.99

Table 5
Thermodynamic parameters for the adsorption of Cr(VI) on Amberlite IRA410

ΔS (J/K/mol)	ΔH (kJ/mol)	ΔG (kJ/mol)			
		T (K)			
318.4	85.2	288	297	303	313
		– 6.58	–9.06	–11.3	–14.4

3.2.5. Effect of the temperature on chromium (VI) adsorption

The effect of the temperature (in the range 15 to 40°C) on the sorption of Cr(VI) by Amberlite IRA410 is shown in Fig. 3. As observed, adsorption rates and equilibrium levels increased for increasing temperatures. The result obviously indicated a favorable adsorption, showing the endothermic nature of the adsorption [17].

Thermodynamically, parameters such as free energy change (ΔG), enthalpy change (ΔH) and entropy change (ΔS) can be calculated using the following Eqs. (17)–(19) where K_c is the equilibrium constant.

$$\ln K_c = \frac{q_e}{C_e} \tag{15}$$

$$\Delta G = -RT \ln K_c \tag{16}$$

$$\ln K_c = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \tag{17}$$

The slopes and intercept of ln K_c versus $1/T$ were used for the determination of ΔH and ΔS. The linear equation was $y = -10{,}244x + 38{,}265$ and the correlation coefficient was 0.996. As seen from Table 5, ΔG values were negative, indicating that the adsorption process led to a decrease of the Gibb's free energy, confirming the feasibility of the process and the spontaneous nature of the adsorption under the considered experimental conditions. Positive values of ΔS refer to an increasing randomness at the solid–solution interface. The positive value of ΔH indicated that the adsorption process was an endothermic process.

4. Conclusions

The following conclusions can be drawn from the above results. Amberlite IRA 410, a strong anionic exchanger resin, appeared as a good candidate for the removal of hexavalent chromium. Adsorption rate was rapid during the first 30 min and equilibrium was found to be attained within 120 min. Amberlite IRA 410 resin had high adsorption capacity for chromium at the room temperature. The capacity increased for increasing temperatures. Equilibrium removal by the resin followed the Langmuir isotherm and maximum adsorption capacity reached 153.8 mg/g at pH 5.2. The kinetics of chromium adsorption followed a peudo-second order kinetics model. The adsorption load increased by increasing the initial Cr(VI) concentration. The process was spontaneous in nature and endothermic.

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