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Kinetics of removal of chromium from water and electronic process wastewater by ion exchange resins: 1200H, 1500H and IRN97H

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Abstract

The removal of chromium from aqueous solution by an ion exchange resin is described. Ion exchange resins 1200H, 1500H and IRN97H show a remarkable increase in sorption capacity for chromium, compared to other adsorbents. The adsorption process, which is pH dependent show maximum removal of chromium in the pH range 2–6 for an initial chromium concentration of 10 mg/l. The metal ion adsorption obeyed linear, Langmuir and Freundlich isotherms. The adsorption of chromium on these cation exchange resins follows first-order reversible kinetics and pseudo-first-order kinetics. The intraparticle diffusion of chromium on ion exchange resins represents the rate-limiting step. The uptake of chromium by the ion exchange resins was reversible and thus have good potential for the removal/recovery of chromium from aqueous solutions. We conclude that such ion exchange resins can be used for the efficient removal of chromium from water and wastewater.

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Keywords: Chromium; Ion exchange resin; Adsorption isotherms; Adsorption kinetics; Adsorption dynamics; Intraparticle diffusion; Electronic process wastewater

1. Introduction

Electronic process waste is one of the major contributors to heavy metal pollution in surface waters [1,2]. The treatment and removal of heavy metals has received considerable attention because of their association with various health problems. It is known that

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legal standards on environment control are becoming more strict and, as a result, the discharge of heavy metals into aquatic bodies and sources of potable water is being rigorously controlled.

Industries typically use chromium in electroplating, leather tanning, metal finishing and chromate preparation. The tanning industries, due to the complexity of transforming animal hide (or skins) into leather, use large amounts of chemical agents and produce an enormous volume of residual waters and solid effluents. They are also common ground water contaminants at industrial and military installations. Some industrial effluents can contain chromium at concentrations ranging from tenths to hundreds of mg/l. Chromium is considered by the IARC to be a powerful carcinogenic agent that modifies the DNA transcription process causing important chromosome aberrations [3]. On the other hand, the presence of chromium in water causes significant environmental problems, as reported by Muir [4]. The National Institute for Occupational Safety and Health (NIOSH) recommends that the levels of chromium in water should be reduced to 10^{-3} mg/m³. Chromium has been reported to be toxic to animals and humans and is known to be carcinogenic [5].

Numerous processes are available for removing dissolved heavy metals, including precipitation, phytoextraction, ultrafiltration, reverse osmosis, and electrodialysis [6–11]. Nevertheless, many of these approaches are marginally cost-effective or difficult to implement in developing countries. Therefore, the need exists for a treatment strategy that is simple, robust, and that addresses local resources and constraints. Sorption operations, including adsorption and ion exchange, are a potential alternative water and wastewater treatment. In an adsorption process, atoms or ions (adsorbate) in a fluid phase diffuse to the surface of a solid (adsorbent), where they bond with the solid surface or are held there by weak intermolecular forces [12]. A number of investigators have studied the removal of inorganic metal ions namely cadmium, cobalt, zinc, silver, copper, mercury, chromium and lead from aqueous solution using different adsorbents [13–22]. However, the improved sorption capacity of ion exchange resins may have advantages over such non-specific adsorbents. In this regard, ion exchange resins hold great potential for the removal of heavy metals from water and industrial wastewater. Rengaraj and co-workers reported on the removal of heavy metals from water and nuclear power plant coolant water using ion exchange resins [23–26].

In the present study, 1200H, 1500H and IRN97H cation exchange resins were used for the removal of chromium from aqueous solution. Chromium compounds are present in electronic process wastewater. The main objective of this study was to investigate the equilibrium and kinetic parameters of these ion exchange resins and compare the ion exchange behaviour of various resins. In addition, parameters that influence adsorption, such as initial chromium concentration, agitation time, pH, isotherm and kinetic studies were investigated.

2. Sorption theory

The concentration of metal on a medium is calculated as the difference between the original concentration in the solution and the concentration in solution, after contact. The mass balance may be expressed as:

$$m(N_{\rm e} - N_0) = V(C_0 - C_{\rm e}) \tag{1}$$

and $N_0 = 0$; therefore,

$$N_{\rm e} = \frac{V(C_0 - C_{\rm e})}{m} \tag{2}$$

where N_e and C_e are, respectively, the adsorbent phase metal concentration (mg/l) and solution phase concentration at equilibrium (mg/l), C_0 the initial metal concentration (mg/l), V the solution volume (l), and m the mass of adsorbent (g).

3. Experimental

3.1. Materials and methods

The following cation exchange resins 1200H, 1500H and IRN97H (M/s. Rohm and Haas, France S.A.) were used in this studies. These cation exchange resins are generally used for the removal of heavy metals from water and wastewater. Their physico-chemical properties and specifications, as reported by the suppliers, are shown in Table 1. All the chemicals used were of analytical grade and were obtained from Aldrich, USA.

A stock solution of 500 mg/l of chromium was prepared by dissolving 3.8476 g of Cr(NO₃)₃·9H₂O in 1000 ml distilled water. This solution was diluted as required to obtain standard solution containing 10-30 mg/l of chromium. One hundred millilitres of a chromium solution of a desired concentration was adjusted to a desired pH and placed in reagent bottles of 300 ml capacity, known amounts of ion exchange resins were added separately and the pH was adjusted using 0.1N hydrochloric acid or dilute sodium hydroxide solutions. The solutions were agitated for a predetermined period at 25 ± 1 °C in a shaking incubator (Model-Vision Scientific Co. Ltd., KMC 8480S). The resins were separated and the filtrate was analysed by atomic absorption spectrophotometry (Perkin-Elmer model 3110) for chromium content. Adsorption isotherm and kinetic studies were carried out with different initial concentrations of chromium while maintaining the resin dosage at a constant level. For pH effects, 10 mg/l chromium and ion exchange resins 1200H, 1500H and IRN97H each at a level of 500 mg/100 ml were used. In order to correct for any adsorption of chromium on the container surface, control experiments were carried out in the absence of resins. These experiments indicated that no adsorption by the container walls was detectable. In addition, all mixing vessels were kept sealed throughout the duration of each isotherm test in order to minimise the release of gaseous species to the atmosphere.

A chromium containing synthetic electronic process wastewater sample was prepared on the basis of the analysis of chemical composition from the electronic process wastewater (http://www.cleantechindia.com/eicnew/guidelines/electronics1.htm), and its concentrations are shown in Table 2. This synthetic solution was used for the adsorption study with ion exchange resins. To study the adsorbent dosage, the sample was used at the solution pH and agitated with different dosages of ion exchange resins for a 24 h period.

Table 1
Characteristics properties of the ion exchange resins used

Styrene divinylbenzene copolymer
-SO ₃ -
Insoluble, amber beads
H^+
\geq 1.8 eq./l (H ⁺ form), \geq 2.0 eq./l (Na ⁺ form)
49–55% (H ⁺ form)
800 g/l
1.18–1.22 (H ⁺ form)
≤1.2
$630\pm50\mu\mathrm{m}$
<0.300 mm (0.1% maximum)
>0.850 mm (10% maximum)
$Na^+ \rightarrow H^+ (10\%)$
Styrene divinylbenzene copolymer
Sulphonates
Dark amber beads
H^+
\geq 2.0 eq./l (H ⁺ form)
45–51% (H ⁺ form)
820 g/l
1.28–1.32 (H ⁺ form)
≤1.2
$650\pm50\mu\mathrm{m}$
<0.425 mm (0.5% maximum)
>0.850 mm (10% maximum)
$Na^+ \rightarrow H^+ (10\%)$
Polystyrene divinylbenzene gel
-SO ₃ -
Uniform particle size spherical beads
$\rm H^+$
\geq 2.15 eq./l (H ⁺ form)
45–51% (H ⁺ form)
800 g/l
≤1.2
$525\pm50\mu\mathrm{m}$

^a Manufacturer supplied.

4. Results and discussion

4.1. Effect of pH upon adsorption

The effect of pH on the removal of chromium by the ion exchange resins is shown in Fig. 1. It can be seen that pH of the solution plays a very important role in the adsorption of chromium. The percentage of adsorbed is at a maximum in the pH range 2–6,

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Serial no.	Parameter	Maximum value (mg/l, except pH)		
1	Phosphorus	5.0		
2	Fluoride	20		
3	Ammonia	10		
4	Arsenic	0.1		
5	Chromium (hexavalent)	0.1		
6	Cadmium	0.1		
7	Copper	0.5		
8	Lead	0.1		
9	Mercury	0.01		
10	Nickel	0.5		
11	Tin	2.0		

Table 2 Composition of chromium containing electronic process wastewater^a

^a http://www.cleantechindia.com/eicnew/guidelines/electronics1.htm.

for a solution containing 10 mg/l of chromium. The percentage of adsorption decreases rapidly when the pH is increased above 6 due to the formation of a chromium precipitate at higher pH values. For comparison, the precipitation curve at different pH values in the absence of resins is also shown in the figure. For the ion exchange resins, the adsorption at pH above 6 shows a decreasing trend because of the formation of hydroxyl complexes of chromium, $Cr(OH)_3$ [27]. Hence in subsequent studies, the experiments were performed using a solution at pH 3.8. The high adsorption capacity of chromium in the lower pH range has been observed by other investigators, e.g. Sharma and Forster reported that the maximum adsorption of Cr(VI) on peat moss occurred at pH 3, but total removal is not favoured below this pH [28]. The possibility that Cr(VI) is reduced to Cr(III) is an issue,



Fig. 1. Effect of pH on the removal of chromium by ion exchange resins 1200H, 1500H and IRN97H. Conditions: resin dosage = 500 mg/100 ml; equilibration time = 24 h.



Fig. 2. Effect of resin dosage on the removal of chromium by ion exchange resins 1200H, 1500H and IRN97H. Conditions: pH 3.8; equilibration time = 24 h.

because Cr(III) is weakly adsorbed in a lower pH range. Huang and Wu also demonstrated a similar adsorption behaviour for Cr(VI) on activated carbon [29]. The maximum adsorption was observed in the pH range 3–6 with decreasing values on either side of this optimum pH.

4.2. Effect of resin dosage on adsorption

Fig. 2 shows the removal of chromium as a function of resin dosage using 1200H, 1500H and IRN97H in a solution at pH 3.8. The resin dosage was varied from 0.025 to 0.600 g and equilibrated for 24 h. It is clear that for the maximum removal of 10 mg/l of chromium in 100 ml requires a minimum resin dosage of 50, 100 and 150 mg for 1200H, 1500H and IRN97H, respectively. The data clearly shows that all the resins have a high level of performance in terms of the removal of chromium. The observed differences may be due to the high ion exchange capacity of 1200H, 1500H and IRN97H. The experimental results revealed that the chromium removal efficiency increases up to an optimum dosage beyond which the removal efficiency does not change [23–26]. It may be concluded that by increasing the adsorbent dose the removal efficiency increases but adsorption density decreases. The decrease in adsorption density can be attributed to the fact that some of the adsorption sites remain unsaturated during the adsorption process; whereas the number of available adsorption sites increases by an increase in adsorbent and this results in an increase in removal efficiency. As expected, the equilibrium concentration decreases with increasing adsorbent doses for a given initial chromium concentration, because for a fixed initial solute concentration, increasing the adsorbent doses provides a greater surface area or adsorption sites [30].

4.3. Adsorption isotherm

Adsorption data usually described by adsorption isotherms, such as the linear, Langmuir, Freundlich isotherm. These isotherms relate metal uptake per unit weight of adsorbent q_e to the equilibrium adsorbate concentration in the bulk fluid phase C_e . The linear model, which describes the accumulation of solute by sorbent as directly proportional to the solution concentration:

$$q_{\rm e} = K_{\rm D} C_{\rm e} \tag{3}$$

The constant of proportionality or distribution coefficient K_D is often referred to as the partition coefficient.

The Langmuir model represents one of the first theoretical treatments of non-linear sorption, and has been successfully applied to a wide range of systems that exhibit limiting or maximum sorption capacities. The model assumes uniform energies of adsorption onto the surface and no transmigration of the adsorbate in the plane of the surface. The Langmuir isotherm is given by:

$$q_{\rm e} = \frac{Q^{\circ}bC_{\rm e}}{l+bC_{\rm e}} \tag{4}$$

where Q° and *b* are Langmuir constants related to adsorption capacity and energy of adsorption, respectively [31]. Eq. (4) is usually linearised by inversion to obtain the following form [32]:

$$\frac{1}{q_{\rm e}} = \frac{1}{Q^{\circ}} + \frac{1}{bQ^{\circ}} \frac{1}{C_{\rm e}}$$
(5)

Eq. (5) is equally used to analyse batch equilibrium data by plotting $1/q_e$ versus $1/C_e$, which yields a linear plot if the data conform to the Langmuir isotherm.

The Freundlich isotherm is the most widely used non-linear sorption model and is given by the general form:

$$q_{\rm e} = K_{\rm F} C_{\rm e}^n \tag{6}$$

where K_F relates to sorption capacity and *n* to sorption intensity. The logarithmic form of Eq. (6) given below is usually used to fit data from batch equilibrium studies:

$$\log q_{\rm e} = \log K_{\rm F} + n \log C_{\rm e} \tag{7}$$

Eqs. (3), (5) and (7) are usually used for the analysis of equilibrium batch experiment data assuming linear, Langmuir and Freundlich isotherms, respectively [32].

Figs. 3–5 present the linear, Langmuir and Freundlich isotherm plots for chromium adsorption on the ion exchange resins. The equilibrium data was fitted very well to all three sorption isotherms. These plots were used to calculate the isotherm parameters given in Table 3 for chromium.

4.4. First-order adsorption kinetic model

Experiments were also directed at an attempt to understand the kinetics of chromium removal by the 1200H, 1500H and IRN97H resins. It is a well-established fact that the



Fig. 3. Linear isotherm plot for the adsorption of chromium on ion exchange resins 1200H, 1500H and IRN97H.



Fig. 4. Langmuir isotherm plot for the adsorption of chromium on ion exchange resins 1200H, 1500H and IRN97H.



Fig. 5. Freundlich isotherm plot for the adsorption of chromium on ion exchange resins 1200H, 1500H and IRN97H.

adsorption of ions in an aqueous system follows reversible first-order kinetics, when a single species is considered on a heterogeneous surface [33]. It must be remembered that the two important physico-chemical aspects for parameter evaluation of the sorption process as a unit operation are the kinetics and the equilibria of the system. The kinetics of sorption describing the solute uptake rate which, in turn, governs the residence time of the sorption reaction is one of the important characteristics that defines the sorption efficiency. Hence, in the present study, the kinetics of chromium removal were carried out to understand the behaviour of this adsorbent.

The sorption of chromium from liquid phase to solid phase may be expressed as:

$$\mathbf{A}_{k_2}^{k_1} \mathbf{B} \tag{8}$$

where k_1 is the forward reaction rate constant and k_2 the backward reaction rate constant. If *a* is the initial concentration of chromium and *x* the amount transferred from liquid phase

Resin	Linear	Langmuir		Freundlich	
	$K_{\rm D}$ (l/g)	$Q^{\circ} (mg/g)$	<i>B</i> (l/g)	K _F	n
1200H	3.05	84.04	0.039	3.28	1.04
1500H	3.78	188.67	0.022	4.99	0.89
IRN97H	8.63	58.14	0.16	6.31	1.31

Table 3 Summary of isotherm parameters for the adsorption of chromium on ion exchange resins

to solid phase at any time *t*, then

rate
$$\left(\frac{\mathrm{d}x}{\mathrm{d}t}\right) = \frac{-\mathrm{d}(a-x)}{\mathrm{d}t} = k(a-x)$$
 (9)

or

$$k = \left(\frac{1}{t}\right) \ln\left(\frac{a}{a-x}\right) \tag{10}$$

where k is the overall reaction rate constant. Since k_1 and k_2 are the rate constants for the forward and reverse process, the rate can be expressed as:

$$\frac{\mathrm{d}x}{\mathrm{d}t} = k_1(a-x) - k_2 x \tag{11}$$

If X_e represents the concentration of chromium adsorbed at equilibrium, then at equilibrium, $k_1(a - X_e) - k_2 X_e = 0$, because under these conditions:

$$\frac{dx}{dt} = 0$$
 or $k_{\rm C} = \frac{X_{\rm e}}{a - X_{\rm e}} = \frac{k_1}{k_2}$ (12)

where $k_{\rm C}$ is the equilibrium constant. Now under equilibrium conditions, the rate becomes:

$$\frac{\mathrm{d}x}{\mathrm{d}t} = [k_1(a-x) - k_2x] - [k_1(a-X_{\mathrm{e}}) - k_2X_{\mathrm{e}}]$$
(13)

$$\frac{dx}{dt} = (k_1 + k_2)(X_e - x)$$
(14)

The above equation is in the form dx/dt = k(a - x). Therefore,

$$k_1 + k_2 = \left(\frac{1}{t}\right) \ln\left(\frac{X_e}{X_e - x}\right) \tag{15}$$

$$\ln\left(\frac{1}{1 - (x/X_{\rm e})}\right) = (k_1 + k_2)t\tag{16}$$

$$-\ln\left(1 - \frac{x}{X_{\rm e}}\right) = (k_1 + k_2)t \tag{17}$$

The above equation can be written in the form:

$$\ln(1 - U_t) = -(k_1 + k_2)t = -kt \tag{18}$$

where $U_t = x/X_e$ and *k* the overall rate constant. Further,

$$k = k_1 + k_2 = k_1 + \frac{k_1}{k_C} = k_1 \left(1 + \frac{1}{k_C} \right)$$
(19)

where U_t can be calculated using the expression:

$$\frac{C_{A(0)} - C_{A(t)}}{C_{A(0)} - C_{A(e)}} = \frac{x}{X_e} = U_t$$
(20)

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where $C_{A(0)}$ is the initial concentration of chromium (mg/l), $C_{A(t)}$ the concentration of chromium present at any time *t* (mg/l), $C_{A(e)}$ the concentration of chromium present at equilibrium condition (mg/l). U_t is called the fractional attainment of equilibrium of chromium and is calculated by considering chromium adsorption over the resins in a given time range



Fig. 6. Kinetic fit for the adsorption of chromium on ion exchange resins: (a) 1200H; (b) 1500H; (c) IRN97H.



Fig. 6. (Continued).

1–24 h. In the present study, a concentration of chromium over the range 10–20 mg/l was examined. Using the kinetic equations, the overall rate constant, the forward and backward rate constants were calculated. For example, by plotting $\ln(1 - U_t)$ versus *t* (Fig. 6a–c), the overall rate constant *k* for a given concentration of chromium was calculated by considering the slope of straight line in Fig. 6a–c and by using Eqs. (12) and (18), the equilibrium constant k_c , forward and backward rate constants k_1 and k_2 were calculated, as shown in Table 4 [34]. From Table 4, it can be seen that the forward rate constants for the removal of chromium were much higher than the backward rate constants, namely the desorption process.

			•		
Serial no.	Resin	Concentration of chromium (mg/l)	Overall rate constant, $k = k_1 + k_2$ (h ⁻¹)	Forward rate constant, k_1 (h ⁻¹)	Backward rate constant, k_2 (h ⁻¹)
1	1200H	10	0.3914	0.3518	0.0396
2		15	0.4034	0.3991	0.0043
3		20	0.4461	0.4396	0.0065
4	1500H	10	0.6256	0.6168	0.0088
5		15	0.6022	0.5970	0.0052
6		20	0.6723	0.6676	0.0047
7	IRN97H	10	0.6006	0.5862	0.0144
8		15	0.5955	0.5872	0.0083
9		20	0.6814	0.6729	0.0085

Rate constants for the removal of chromium with ion exchange resins

Table 4

4.5. Pseudo-first-order adsorption kinetic model

The rate constant of adsorption can also be determined from the following pseudo first-order rate expression given by Lagergren and Svenska [35].



Fig. 7. Lagergren plot for the adsorption of chromium on ion exchange resins: (a) 1200H; (b) 1500H; (c) IRN97H.



Fig. 7. (Continued).

The differential equation is as follows:

$$\frac{\mathrm{d}q}{\mathrm{d}t} = k_{\mathrm{ad}}(q_{\mathrm{e}} - q) \tag{21}$$

Integrating Eq. (21) for the boundary conditions t = 0 to t = t and q = q, gives:

$$\log\left(\frac{q_{\rm e}}{q_{\rm e}-q}\right) = \left(\frac{k_1}{2.303}\right)t\tag{22}$$

which is the integrated rate law for a pseudo-first-order reaction, Eq. (22) can be rearranged to obtain a linear form:

$$\log(q_{\rm e} - q) = \log q_{\rm e} - \left(\frac{k_{\rm ad}}{2.303}\right)t$$
 (23)

Table 5

Lagergren rate constants and intraparticle diffusion values for different initial concentrations of chromium

Resin	Concentration of chromium (mg/l)	Lagergren rate constant, k_{ad} (l/h)	Intraparticle rate constant, $k_{id} (mg/(g h^{1/2}))$
1200H	10	0.3915	4.5404
	15	0.4035	7.6772
	20	0.4461	10.657
1500H	10	0.6257	5.9868
	15	0.6022	8.7401
	20	0.6725	13.459
IRN97H	10	0.6006	6.3617
	15	0.5955	9.4944
	20	0.6815	12.63

where q and q_e are amounts of chromium adsorbed (mg/g) at time t (h) and at equilibrium, respectively, and k_{ad} the rate constant for adsorption (l/h). The straight line plots of $\log(q_e - q)$ versus t for different chromium concentrations (Fig. 7a–c) indicates the applicability of the above equation. The values of k_{ad} were calculated from the slope of the linear plots and are presented in Table 5 for different concentrations for different ion exchange resins.

4.6. Intraparticle diffusion model

The rate constant for intraparticle diffusion (k_{id}) is given by Weber and Morris [36]:



Fig. 8. Intraparticle diffusion plots for the adsorption of chromium on ion exchange resins: (a) 1200H; (b) 1500H; (c) IRN97H.



Fig. 8. (Continued).

$$q = k_{\rm id} t^{1/2} \tag{24}$$

where *q* is the amount adsorbed (mg/g) at time *t* (h). Plots of *q* versus $t^{1/2}$ are shown in Fig. 8a–c for different initial concentrations. All the plots have the same general features in that the initial curved portion followed by linear portion to the intraparticle diffusion and the plateau to the equilibrium. k_{id} values were obtained from the slope of the linear portion of the curves for each concentration of metal ion (Table 5). The value for k_{id} was higher at higher concentration.

4.7. Application to synthetic electronic process wastewater

The composition of chromium containing electronic process wastewater is presented in Table 2. Since electronic process wastewater has a chromium concentration of 0.1 mg/l, it was used for the study with 1200H, 1500H and IRN97H, and then subjected to treatment. The effect of pH on chromium removal is shown in Fig. 9. The maximum removal of chromium from 100 ml of wastewater containing 0.1 mg/l chromium was adsorbed by 10 mg/100 ml of each of 1200H, 1500H and IRN97H resin at a solution pH of 5.8. The effect of resin dosage on chromium removal is shown in Fig. 10. The efficiency of these resins with respect to the removal of chromium from the synthetic chromium solution alone is higher than that for the synthetic electronic process wastewater. This can be attributed to the presence of other impurities (metal ions) present in electronic process wastewater which may interfere in the ion exchange process.



Fig. 9. Effect of pH on the removal of chromium from electronic process wastewater by ion exchange resins. Conditions: resin dosage = 500 mg/100 ml; contact time = 24 h.



Fig. 10. Effect of resin dosage on the removal of chromium from electronic process wastewater by ion exchange resins. Conditions: pH 5.8; contact time = 24 h.

5. Conclusion

Ion exchange resins have been identified as potentially and efficient materials for use in the treatment of water contaminated with selected heavy metals. The results here is indicate that ion exchange resins can efficiently remove traces of chromium present in aqueous solutions. Equilibrium removal by ion exchange resins followed Freundlich, Langmuir and linear adsorption isotherms. The values for adsorption capacity (k) and intensity of adsorption (n) indicate the greater affinity for chromium by these resins. The kinetics of chromium adsorption by these ion exchange resins follow first-order reversible kinetics and a pseudo-first-order kinetic model. The low value of k_2 (desorption process) indicates that the adsorbed chromium is relatively stable on the adsorbent. The adsorption of chromium on ion exchange resins followed intraparticle diffusion process. We conclude that ion exchange resins could be exploited for applications in the tertiary level treatment of potable water as well as industrial effluents. The kinetic data would be useful for the fabrication and design of wastewater treatment plants. The adsorption of chromium was hindered by the presence of other metals. In the case of electronic process wastewater, chromium adsorption was particularly damped by the presence of other metal ions. Detailed studies will be needed to further evaluate ion exchange resins in terms of their competitive adsorption and their reaction chemistry.

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