Removal of chromium from water and wastewater by ion exchange resins

S. Rengaraj, Kyeong-Ho Yeon, Seung-Hyeon Moon*

*Department of Environmental Science and Engineering, Kwangju Institute of Science and Technology,
1 Oryong dong, Puk-gu, Kwangju 500-712, South Korea*

Abstract

Removal of chromium from water and wastewater is obligatory in order to avoid water pollution. Batch shaking adsorption experiments were carried out to evaluate the performance of IRN77 and SKN1 cation exchange resins in the removal of chromium from aqueous solutions. The percentage removal of chromium was examined by varying experimental conditions viz., dosage of adsorbent, pH of the solution and contact time. It was found that more than 95% removal was achieved under optimal conditions. The adsorption capacity (k) for chromium calculated from the Freundlich adsorption isotherm was found to be 35.38 and 46.34 mg/g for IRN77 and SKN1 resins, respectively. The adsorption of chromium on these cation exchange resins follows the first-order reversible kinetics. The ion exchange resins investigated in this study showed reversible uptake of chromium and, thus, have good application potential for the removal/recovery of chromium from aqueous solutions. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Chromium; Ion exchange resin; Adsorption isotherms; Adsorption kinetics

1. Introduction

Industrial wastewaters often contain considerable amounts of heavy metals that would endanger public health and the environment if discharged without adequate treatment. Heavy metals such as Pb, Hg, Cr, Ni, Cd, Cu, and Zn which are usually associated with toxicity [1]. Major anthropogenic sources of heavy metals in the environment include metal extraction, metal fabrication and surface finishing, paints and pigments, as well as the manufacture of batteries. Pollution by chromium is of considerable concern as the metal has found widespread use in electroplating, leather tanning, metal finishing, nuclear power plant, textile industries and chromate preparation.

* Corresponding author. Tel.: +82-62-970-2432; fax: +82-62-970-2434/2588.
E-mail address: shmoon@kjist.ac.kr (S.-H. Moon).

The effluent from the industries may contain chromium at concentrations ranging from tenths to hundreds of mg/l. Chromium is considered by the IARC [2] as a powerful carcinogenic agent that modifies the DNA transcription process causing important chromosomic aberrations [3–5]. On the other hand, the presence of chromium in water causes serious environmental problems as mentioned by Muir [6]. The National Institute for Occupational Safety and Health (NIOSH) recommends that the levels of chromium in water should be reduced to 10^{-3} mg/m^3. The metal is reported to be bio-accumulated into flora and fauna, creating ecological problems [7,8]. Chromium toxicity to mammals and aquatic organisms appears to be lower compared to other heavy metals, due to the general low solubility of Cr(III) compounds, low mobility in the environmental compartments and limited availability to living organisms. However, the possibility that organic ligands and/or acidic conditions in the environment increase Cr(III) mobility, that MnO_2 oxides, present in soils, favor the oxidation of Cr(III) to the more toxic and mobile Cr(VI) forms cannot be excluded [9]. The Minimal National Standards (MINAS) upper limit of chromium in industrial wastewater is 0.1 mg/l [10]. For compliance with this limit, it is essential for industries to treat their effluents to reduce the chromium to acceptable levels. Due to more stringent environmental regulations, most of the mineral processing plants are facing nowadays the difficult problem of tailings management and disposal, which certainly are produced in huge quantities.

Previous researchers reported reasonable methods, from the economical and technological point of view, to remove heavy metals in general from superficial waters, with the aim of the preservation of the entire ecosystem from the damages due to their accumulation [11]. Among the various treatment techniques available, the most commonly used ones are:

- reduction and precipitation;
- ion exchange; and
- adsorption.

These methods suffer from some drawback such as high capital and operational costs or the treatment and disposal of the residual metal sludge. The main advantages of ion exchange over chemical precipitation are recovery of metal value, selectivity, less sludge volume produced and the meeting of strict discharge specifications. In ion exchange systems, polymeric resins are usually employed [12]. Ion exchange using synthetic resins is the method of choice in many water treatment processes for removing inorganic contaminants in water and wastewater [13]. Tiravanti et al. [9] studied the pretreatment of tannery wastewater by an ion exchange process for Cr(III) removal and recovery. They found that a considerable decrease in the use of chemical compounds, a sludge production reduced by 80% with respect to the traditional treatment and lower costs for sludge treatment and disposal.

Vagliasindi and Benjamin [14] studied the arsenic removal in fresh and NOM-preloaded ion exchange packed bed adsorption reactors. But NOM preloading did not affect the systems, but sulfate adsorbed during the preloading and the subsequent adsorption steps caused chromatographic displacement of the adsorbed arsenic. Chiarle et al. [11] studied the mercury removal from water by ion exchange resins adsorption. They reported resin has a high adsorption efficiency, reaching 30–40 wt.% and the efficiency decreases, decreasing pH, due to competition between ions H^+ and Hg^{2+}. Jorgensen [15] examined the applicability of cellulose ion exchangers for water and wastewater treatment. Three cellulose ion exchangers were examined and it was found that they are able to remove proteins, azodyes,

DBS, humic acid, chromate and heavy metal ions. The selectivity of the ion exchangers for removal of these compounds is very high, which explains their ability to remove the examined compounds with high efficiency and capacity, although the three cellulose ion exchangers have only a small capacity. Ion exchange for the removal of humic acids in water treatment was studied by Brattebo et al. [16]. Consequently, the removal of chromium from industrial wastewater is a research topic of great interest.

The objective of this study is to investigate the performance of the cation exchange resins IRN77 and SKN1 in removing chromium from water and compare its ion exchange behavior. Tests were conducted to determine the adsorption and ion exchange parameters in the uptake of chromium from synthetic coolant water. The parameters that influence adsorption, such as initial chromium concentration, agitation time and pH were investigated.

2. Experimental

2.1. Materials and methods

The cation exchange resins IRN77 (M/s. Rohm and Haas, France, SA) and SKN1 (M/s. Mitsubhishi Chemical Corporation, Japan) were used in this study. These two cation exchange resins are generally used for the removal of radionuclides from the water and wastewater. Their physical properties and specifications as reported by the suppliers are shown in Tables 1 and 2. All the chemicals used were of analytical grade and obtained from Aldrich Chemical Company, USA.

A stock solution of $500\,mg/dm^3$ of chromium was prepared by dissolving 2.4705 g of $Cr(NO_3)_3$ in 1000 ml distilled water. This solution was diluted as required to obtain standard solution containing 10–150 mg/l of chromium. About 100 ml of chromium solution

Table 1
Characteristics of IRN77 cation exchange resins

Sl. no.	Characteristics	Values
Physical characteristics		
1	Physical form	Uniform particle size spherical beads
2	Shipping weight	800 g/l
3	Harmonic mean size	$650 \pm 50\,\mu m$
4	Uniformity coefficient	<1.2
5	Particle size	<0.300 mm:0.2% maximum, >1.180 mm:2.0% maximum
6	Whole beads	95% minimum
7	Breaking weight (average)	>350 g per bead
Chemical characteristics		
8	Matrix	Polystyrene DVB gel
9	Functional groups	Sulfonic acid
10	Ionic form as shipped	H^+
11	Total exchange capacity	>1.9 eq./l (H^+ form)
12	Moisture holding capacity	49–55% (H^+ form)
13	Ionic conversion	99% minimum H^+

Table 2
Characteristics of SKN1 cation exchange resins

Sl. no.	Characteristics	Values
1	Particle size	425–1180 μm (>95%)
2	Ion exchange capacity (meq./ml)	<1.7
3	Functional groups	Sulfonic acid
4	Na (eq.%)	<0.1
5	Ca (mg/l)	<50
6	Pb (mg/l)	<10
7	Fe (mg/l)	<50
8	Cu (mg/)	<10

of a desired concentration adjusted to a desired pH were taken in reagent bottles of 300 ml capacity, known amounts of IRN77 and SKN1 were added separately and pH was adjusted using 0.1N hydrochloric acid or dilute sodium hydroxide solutions. The solutions were agitated for a predetermined period at $25 \pm 1°C$ in a shaking incubator (Model Jeio Tech SI-900R). The resins were separated and the filtrate was analyzed by a inductively coupled plasma spectrophotometry (Model Thermo Jarrel ash IRIS/AP) 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, 100 mg/l chromium and IRN77and SKN1 dose 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 without resins. It was found that there was no adsorption by the container walls. In addition, all mixing vessels were kept sealed throughout the duration of each isotherm test to minimize dissolution of gaseous species in the atmosphere.

The chromium containing synthetic primary coolant water was prepared on the basis of the analysis of chemical composition from the primary coolant in a nuclear power plant [17], and its concentrations were presented in Table 5. This synthetic solution was used for the adsorption study with IRN77 and SKN1. For the study of adsorbent dosage, the sample was used at pH 2.7 and agitated with different dosages of IRN77 or SKN1 for 24 h.

3. Results and discussion

3.1. Effect of pH

Fig. 1 presents the effect of initial pH on the removal of chromium by IRN77 and SKN1 cation exchange resins. In order to find out the optimum pH for maximum removal efficiency, experiments were conducted with 100 ml of 100 mg/l chromium solution. About 500 mg of IRN77 and SKN1 were added separately in the pH range 2–8 and the results are shown in Fig. 1. It is evident that the removal efficiency of chromium by both resins is reasonably high in the pH range 2–8. The pH of the aqueous solution of chromium affects its uptake on resins and in general the uptake decreases at higher pH values. At higher pH values, the presence of OH^- ions forms the hydroxy complexes of chromium, $Cr(OH)_3$ [18]. Hence, the following experiments were performed in the solution pH of 3.5.
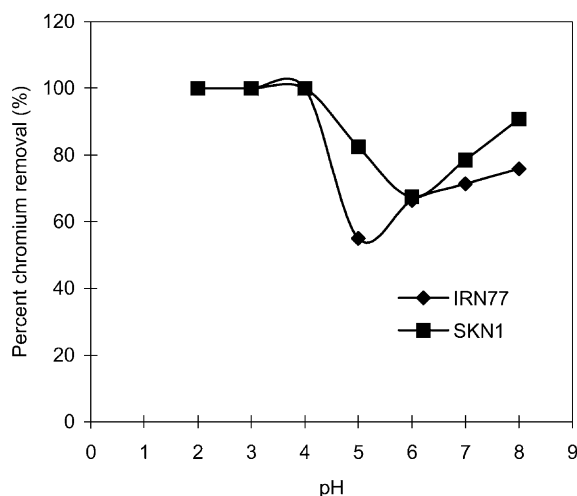
Fig. 1. Effect of pH on the removal of chromium by IRN77 and SKN1. Initial concentration of chromium = 100 mg/l; resin dosage = 500 mg/100 ml; equilibration time = 24 h.

3.2. Effect of resin dose on adsorption

The effect of variation of resin dosage on the removal of chromium by IRN77 and SKN1 ion exchange resins are shown in Fig. 2. Resin dosage was varied from 0.050 to 0.1 g and equilibrated for 24 h at an initial chromium concentration of 100 mg/l. It is apparent that the equilibrium concentration decreases with increasing resin doses for a given initial chromium concentration. This result was anticipated because for a fixed initial solute concentration, increasing adsorbent doses provides greater surface area (or adsorption sites) [19,20]. From Fig. 2, it is evident that for the quantitative removal of 100 mg/l chromium in 100 ml, a minimum resin dosage of 300 mg of IRN77 and 300 mg of SKN1 is required.

3.3. Effect of agitation time

Fig. 3 shows the effect of agitation time on the removal of chromium by IRN77 and SKN1. The removal increases with time and attains equilibrium in 150 min for IRN77 and 120 min for SKN1 for initial chromium concentrations of 100 mg/l. This indicates that SKN1 would require less residence time for the complete removal of chromium compared to IRN77. The metal uptake versus time curves are single, smooth and continuously leading to saturation, suggesting the possible monolayer coverage of metal ions on the surface of the adsorbent [21].

3.4. Adsorption isotherms

The experimental results obtained for the adsorption of chromium on IRN77 and SKN1 ion exchange resins at room temperature ($25 \pm 1°C$) at optimum conditions of contact
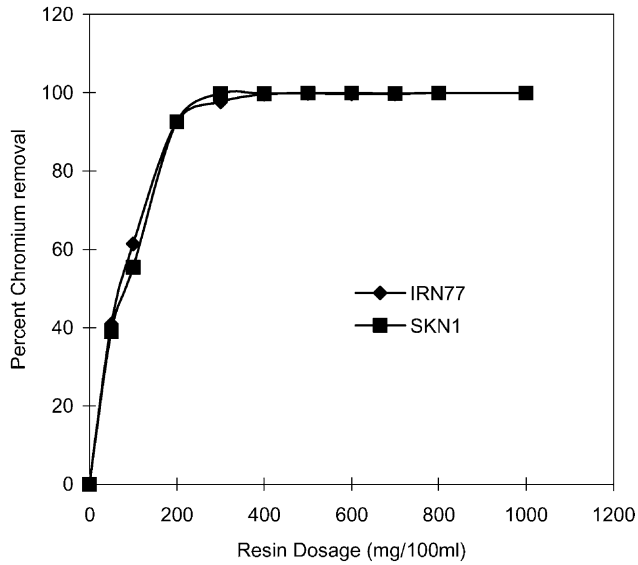
Fig. 2. Effect of resin dosage on the adsorption of chromium on IRN77 and SKN1 resins. Initial chromium concentration $= 100$ mg/l; pH $= 3.5$; equilibration time $= 24$ h.
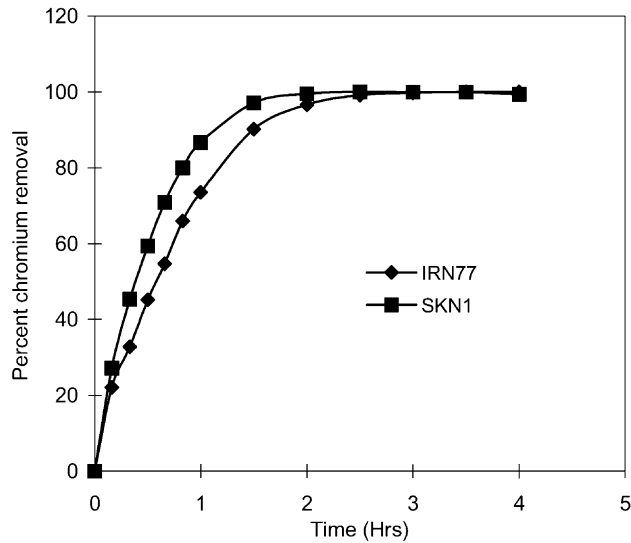

Fig. 3. Effect of contact time on the removal of chromium by IRN77 and SKN1 resins. Initial chromium concentration $= 100$ mg/l; resin dosage $= 300$ mg/100 ml; pH $= 3.5$.

time and dose of adsorbent were found to obey the Freundlich adsorption isotherm [22].

$$\left(\frac{x}{m}\right) = kC_e^{1/n}$$

where x is the amount of chromium removed (mg), m the amount of resin (g), C_e the equilibrium concentration and k and n are Freundlich constants.

The logarithmic form of the equation becomes

$$\log\left(\frac{x}{m}\right) = \log k + \frac{1}{n}\log C_e$$

Freundlich adsorption isotherm is the relationship between the amounts of chromium adsorbed per unit mass of the adsorbent (x/m) and the concentration of the chromium at equilibrium (C_e). Here k and n are constants representing the adsorption capacity and intensity of adsorption, respectively. The data obtained in this study agree with the Freundlich adsorption isotherm (Fig. 4).

The plot of $\log(x/m)$ versus $\log C_e$ for various initial concentrations is found to be linear, indicating the applicability of the classical adsorption isotherm to this adsorbate–adsorbent system. The adsorption capacities (k) were found to be 35.38 and 46.34 mg/g and adsorption intensities (n) 6.6 and 1.83 for IRN77 and SKN1, respectively. The higher value for k indicates the higher affinity for chromium and the values of n lie between 1 and 10 indicating favorable adsorption [23].
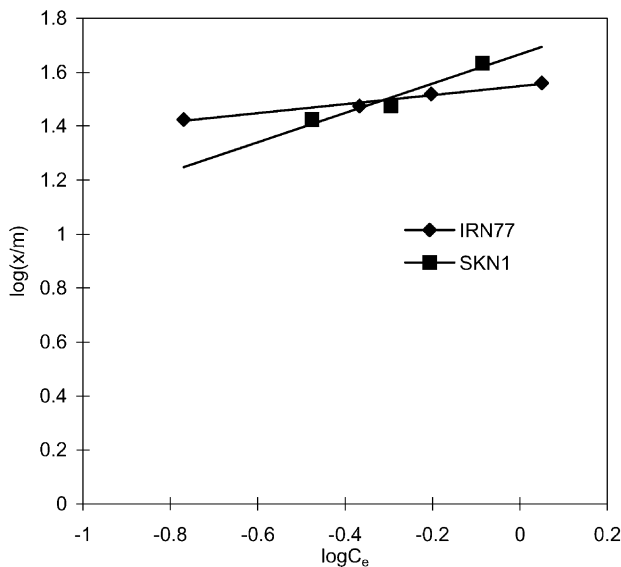

Fig. 4. Freundlich adsorption isotherm for chromium on IRN77 and SKN1.

3.5. Adsorption kinetics

Experiments were also performed in order to understand the kinetics of chromium removal by these resins. It is a well-established fact that the adsorption of ions in aqueous system follows a reversible first-order kinetics, when a single species is considered on a heterogeneous surface [24]. It must be remembered that the two important physicochemical aspects for parameter evaluation of the sorption process as a unit operation are the kinetics and the equilibria. Kinetics of sorption describing the solute uptake rate which in turn governs the residence time of sorption reaction is one of the important characteristics defining the efficiency of sorption. Hence, in the present study, the kinetics of chromium removal has been carried out to understand the behavior of this adsorbent.

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

$$A \underset{k_2}{\overset{k_1}{\rightleftharpoons}} B \tag{1}$$

where k_1 is the forward reaction rate constant, k_2 the backward reaction rate constant.

If a is the initial concentration of chromium and x is the amount transferred from liquid phase to solid phase at any time t, then the rate is

$$\frac{dx}{dt} = \frac{-d(a-x)}{dt} = k(a-x) \tag{2}$$

or

$$k = \frac{1}{t} \ln \frac{a}{a-x} \tag{3}$$

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

$$\frac{dx}{dt} = k_1(a-x) - k_2 x \tag{4}$$

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

$$\frac{dx}{dt} = 0 \quad \text{or} \quad K_C = \frac{X_e}{a - X_e} = \frac{k_1}{k_2} \tag{5}$$

where K_C is the equilibrium constant. Now under equilibrium conditions, the rate becomes

$$\frac{dx}{dt} = [k_1(a-x) - k_2 x] - [k_1(a - X_e) - k_2 X_e] \tag{6}$$

$$\frac{dx}{dt} = (k_1 + k_2)(X_e - x) \tag{7}$$

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

Therefore,

$$k_1 + k_2 = \frac{1}{t} \ln \frac{X_e}{X_e - x} \tag{8}$$

$$\ln \frac{1}{1 - (x/X_e)} = (k_1 + k_2)t \tag{9}$$

$$-\ln \left(1 - \frac{x}{X_e} \right) = (k_1 + k_2)t \tag{10}$$

The above equation can be written in the form of

$$\ln(1 - U_t) = -(k_1 + k_2)t = -kt \tag{11}$$

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

Further,

$$k = k_1 + k_2 = k_1 + \frac{k_1}{K_C} = k_1 \left(1 + \frac{1}{K_C} \right) \tag{12}$$

U_t can be calculated using the expression

$$\frac{C_{A(0)} - C_{A(t)}}{C_{A(0)} - C_{A(e)}} = \frac{x}{X_e} = U_t \tag{13}$$

where $C_{A(0)}$ is the initial concentration of chromium, $C_{A(t)}$ the concentration of chromium present at any time (t), $C_{A(e)}$ the concentration of chromium present at equilibrium condition. U_t is called fractional attainment of equilibrium of chromium and this was calculated by considering chromium adsorption over the resins in a given time range 1–24 h.

In the present study, a concentration of chromium over the range 50–150 mg/l were tried and the concentration of chromium remaining in the equilibrated solution was plotted as a function of the equilibration period for the resins and are shown in Fig. 5a and b. Examination of these figures show that the concentration of chromium under study decrease rapidly within 1–2 h and the sorption is virtually complete within 2–4 h. In each case, the decreasing concentration of chromium remaining in the solution indicates that chromium were adsorbed by IRN77 and SKN1. The figures also indicate a high initial rate of removal of chromium with much slower subsequent removal rates that gradually approach an equilibrium condition. Using the kinetic equations, the overall rate constant, the forward and backward rate constants were calculated. For instance, by plotting $\ln(1 - U_t)$ versus t (Fig. 6a and b) the overall rate constant k for a given concentration of chromium was calculated by considering the slope of straight line in Fig. 6a and b and by using Eqs. (5) and (11) the equilibrium constant K_C, forward and backward rate constants k_1 and k_2 were calculated and shown in Table 3 [25]. From Table 3, it is seen that the forward rate constants for the removal of chromium were much higher than the backward rate constants namely the desorption process. This suggests that the rate of adsorption is clearly dominant.

In order to assess the nature of the diffusion process responsible for adsorption of chromium on IRN77 and SKN1, attempts were made to calculate the coefficients of the process. If film diffusion is to be the rate determining step in the adsorption of chromium on IRN77 and SKN1 surface, the value of film diffusion coefficient (D_f) should be in the range 10^{-6} to 10^{-8} cm^2/s. If pore diffusion is to be the rate limiting, the pore diffusion
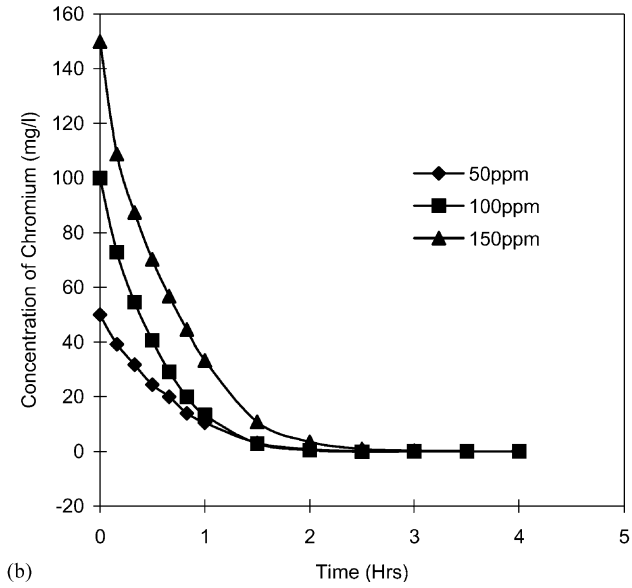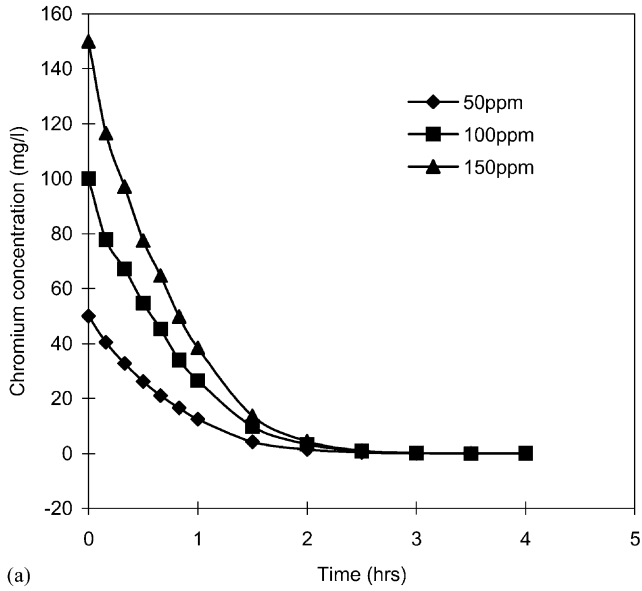
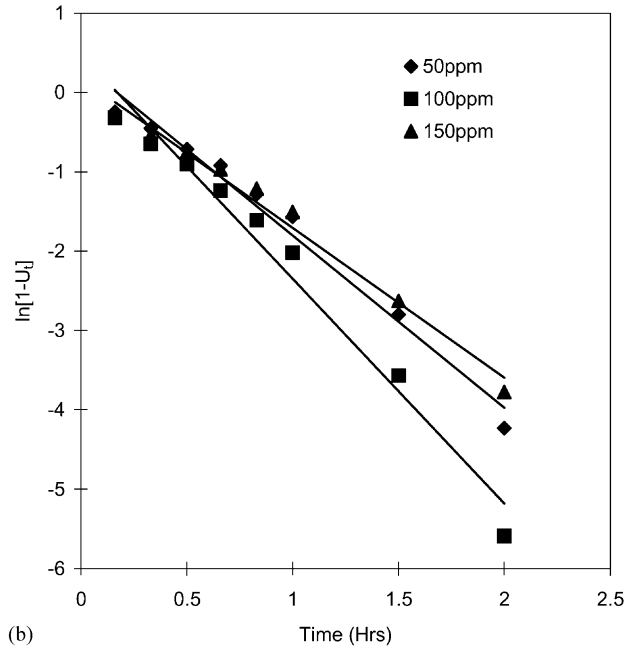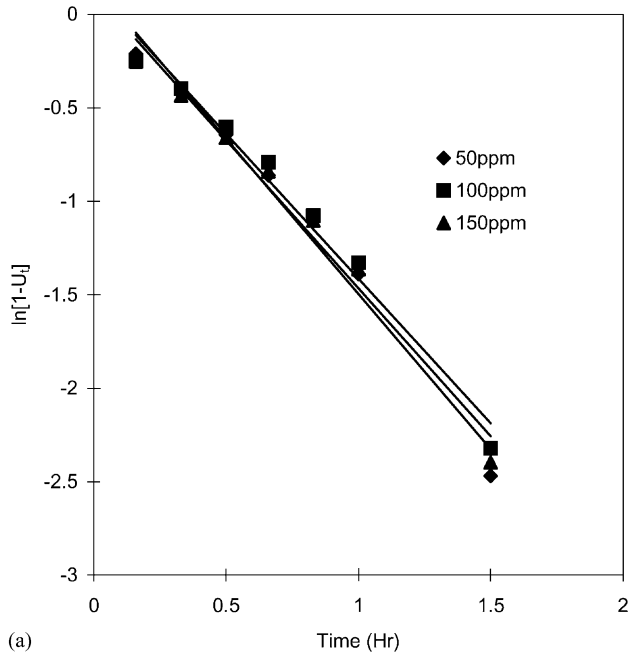Fig. 5. Kinetics of adsorption of chromium on (a) IRN77; and (b) SKN1.

Fig. 6. Kinetic fits for the adsorption of chromium on (a) IRN77; and (b) SKN1.

Table 3
Rate constants for the removal of Cr with IRN77 and SKN1 system

Sl. no.	Name of the resin	Cr(III) concentration (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	IRN77	50	1.0605	1.0602	0.0003
2		100	0.4862	0.4857	0.0005
3		150	0.4987	0.4919	0.0068
4	SKN1	50	0.5315	0.5313	0.0002
5		100	0.5587	0.5575	0.0012
6		150	0.7628	0.6439	0.1189

Table 4
Diffusion coefficient for the removal of chromium by IRN77 and SKN1 system

Sl. no.	Concentration of the chromium (mg/l)	Diffusion coefficient values (cm^2/s)	
		IRN77	SKN1
1	50	0.1428×10^{-6}	7.0670×10^{-6}
2	100	5.2424×10^{-6}	7.4351×10^{-6}
3	150	5.3770×10^{-6}	0.1015×10^{-6}

coefficient (D_p) should be in the range 10^{-11} to 10^{-13} cm^2/s. Assuming spherical geometry for the sorbent, the overall rate constant of the process can be correlated to the pore diffusion coefficient and film diffusion coefficient independently in accordance with the expressions [26].

$$\text{Pore diffusion coefficient } D_p = 0.03 \times \frac{r_o^2}{t_{1/2}}$$

$$\text{Film diffusion coefficient } D_f = 0.23 \times \frac{r_o \partial}{t_{1/2}} \times \frac{\bar{c}}{c}$$

Table 5
Composition of chromium containing synthetic nuclear power plant coolant water

Sl. no.	Compound	Concentration (mg/l)
1	Sb_2O_5	5
2	$Co(NO_3)_2 \cdot 6H_2O$	1
3	$Fe(NO_3)_3 \cdot 9H_2O$	30
4	$Ni(NO_3)_2 \cdot 6H_2O$	15
5	$AgNO_3$	5
6	H_3BO_3	20
7	$Cr(NO_3)_3 \cdot 9H_2O$	4
8	$LiOH \cdot H_2O$	0.5
9	$CsNO_3$	0.5

where r_o is the radius of the adsorbent (cm), ∂ the film thickness, $\bar{c}$ the amount adsorbed and c is the initial concentration. Employing the appropriate data and the respective overall rate constants, pore and film diffusion coefficients for various concentrations of chromium were calculated for IRN77 and SKN1 cation exchange resins. The results are presented in Table 4. It is evident that the removal of chromium follows film diffusion process since the coefficient values are in the range 10^{-6} to 10^{-8} cm^2/s for IRN77 and SKN1 resins.

3.6. Application to nuclear power plant coolant water

The composition of chromium containing synthetic nuclear power plant coolant water is presented in Table 5 [17]. As the coolant water has high concentration of chromium 4 mg/l, it was used for the study with IRN77 and SKN1, and then subjected to treatment. Effect of resin dosage on chromium removal is shown in Fig. 7. Maximum removal of chromium from 100 ml of wastewater containing 4 mg/l chromium was adsorbed by 200 mg/100 ml each of IRN77 and SKN1 resin at a solution pH of 2.75. Effect of contact time on chromium removal is shown in Fig. 8. The efficiency of these resins towards the removal of chromium from synthetic chromium solution alone is higher than that of synthetic coolant water. This may be attributed to the presence of other impurities (metal ions) present in nuclear power plant coolant water which may interfere in the ion exchange process.
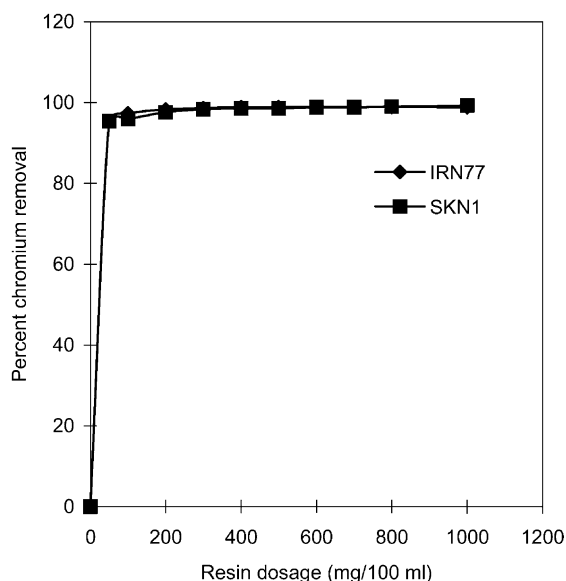

Fig. 7. Effect of resin dosage on the removal of chromium from nuclear power plant coolant water by IRN77 and SKN1. Contact time = 24 h.
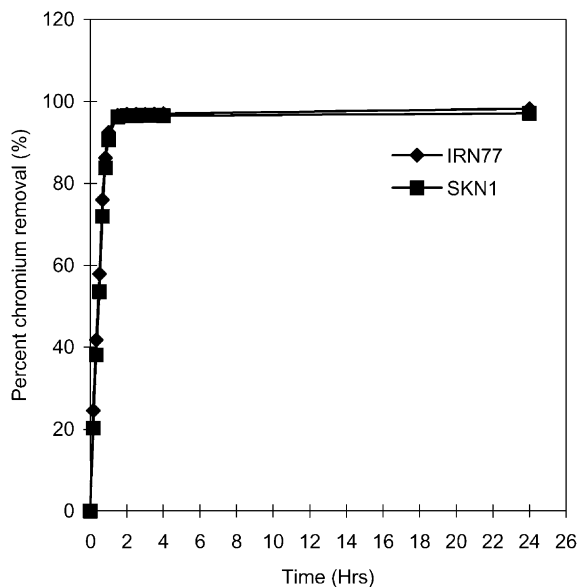
Fig. 8. Effect of contact time on the removal of chromium from nuclear power plant synthetic coolant water by IRN77 and SKN1. Resin dosage = 200 mg/100 ml.

4. Conclusions

The following conclusions were drawn based on investigation of chromium removal by IRN77 and SKN1 adsorption. IRN77 and SKN1 cation exchange resins are capable of removing 100 mg/l chromium from aqueous solution to the extent of 98% at the solution pH. The adsorption process obeys the Freundlich adsorption isotherm. The adsorption capacities (k) were found to be 35.38 and 46.34 mg/g and adsorption intensities (n) 6.6 and 1.83 for IRN77 and SKN1, respectively. The values of adsorption capacity (k) and intensity of adsorption (n) indicate the greater affinity for chromium by these resins. The kinetics of adsorption of chromium by these ion exchange resins follow the first-order reversible kinetics. The low value of k_2 (desorption process) indicates that the adsorbed chromium remains almost stable on the resins. The kinetic data would be useful for the fabrication and designing of wastewater treatment systems. The results also demonstrate that an intraparticle diffusion mechanism plays a significant role in the adsorption and it is apparent that the adsorption rate is controlled by the film diffusion process.

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