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A comparative study of two chelating ion-exchange resins for the removal of chromium(III) from aqueous solution

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Abstract

Macroporous resins containing iminodiacetic acid (IDA) groups (Lewatit TP 207 and Chelex-100) were investigated as a function of concentration, temperature and pH for their sorption properties towards chromium(III). The chromium(III) ions sorbed onto the resin and in the equilibrium concentration were determined by inductively coupled plasma spectrophotometer. The maximum sorption for chromium ions was observed at pH 4.5. Solution pH had a strong effect on the equilibrium constant of Cr(III). The equilibrium constants were 320 and 7 at pH value 4.5 for Lewatit TP 207 and Chelex-100 resin, respectively. The Langmuir isotherm was used to describe observed sorption phenomena. Both the sorbents had high bonding constants with Lewatit TP 207 showing stronger binding. The equilibrium related to adsorption capacity and energy of adsorption was obtained by using plots of Langmuir adsorption isotherm. It was observed that the maximum adsorption capacity of 0.288 mmol of Cr(III)/g for Chelex-100 and 0.341 mmol of Cr(III)/g for Lewatit TP 207 was achieved at pH of 4.5. The rise in temperature caused a slight increase in the value of the equilibrium constant (K_c) for the sorption of chromium(III) ion.

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1. Introduction

Ion-exchange technology is well established, yet new materials, applications and approaches are emerging at a rapid pace. Heavy metal seepage into our environment is still

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devastating. Some metals are known to have various harmful effects on many forms of life. The numerous metals that are significantly toxic to human beings and ecological environments include antimony (Sb), chromium (Cr), copper (Cu), lead (Pb), manganese (Mn), mercury (Hg), cadmium (Cd), etc. Major sources of heavy metal release to the environment include metal extraction, tannery metal fabrication and surface finishing, paints, pigment as well as the battery manufacturing industries [1–5]. To achieve zero or near-zero discharge in industrial wastewater, contaminants must be removed. The reversible nature of ion exchange and sorption processes make them particularly suited to these applications.

Many separation techniques have been proposed for the identification of metals in various environmental samples, including solvent extraction, ion exchange, co-precipitation, membrane processes and sorption [6–8]. Among these methods, ion exchange is highly popular and has been widely practiced in industrial wastewater treatment processes. Although ion exchange is widely used in the metal finishing industry, tannery etc. there still remains a need to develop, and improve ion exchange or sorption based separation techniques [9–11]. Chelating resins have ligands that can selectively bond with certain types of metal cations. The following criteria determine the affinity of chelating resins for a given metal: ionic charge, hydrated ionic radius and ligand bonding with exposed electron pairs on nitrogen, and oxygen [12]. The chelating resin employed in the ion exchange in general is selective and has affinity for heavy metal ions. In addition to the iminodiacetate and aminophosphonic resins, commonly used as well as selective resins, picolyeamine, polyethylenimine, amidoxime resins are also commercially available to removing transition metals [8,13–16].

The use of chelating resins for the pre-concentration and separation of trace elements is well understood and progress today is mainly in improving the specificity of the resin and their applications. These resins are generally used for the removal of heavy metal ions from the aqueous solutions or from the wastewater. Carboxylated resins are a special category of weak cation exchange resins that are useful in chromium metal recovery applications. Their pH range is lower for many metals and the advantage of low pH range is that the metals do not precipitate as oxides or hydroxides. Investigations on the metal sorption properties of iminodiacetic acid (IDA) resins at different temperatures and pH values are recent [3,8,13]. Utilization of Chelex-100 and Lewatit TP 207 is gaining attention as a simple, effective and economic means of treating wastewater containing heavy metals. Chelex-100 and Lewatit TP 207 are abundant, inexpensive and readily available. They also possess several characteristics that make them effective media for the removal of dissolved metal pollutants [12].

As Cr(VI) is carcinogenic, in wastewater a maximum permissible concentration for total Cr of 0.5 mg/l and for Cr(VI) of 0.05 mg/l is legally prescribed [10,17]. In our studies, chromium(III) was chosen because it is an important environmental pollutant. Therefore, the control of the chromium level in wastewater is essential. The objective of this study is to investigate the performance of the chelating exchange resins in removing Cr(III) from aqueous solution. Experiments were run in a wide range of pH, initial metal concentration and temperature values. The operation of ion exchange for the removal of heavy metals is usually carried out in batch vessels.

2. Materials and methods

The chelating resins Chelex-100 (from Merck) and Lewatit TP 207 (from Bayer) were used in this study. Chelex-100 is a chelating ion-exchange resin having functional IDA groups in a styrene-divinylbenzene matrix. Lewatit TP 207 is a chelating ion-exchange resin having functional iminodiacetic acid groups in a styrene matrix. Their physical properties and specifications as reported by the suppliers are shown in Table 1. All solutions were prepared from analytical grade chemicals and Milli-Q filtered deionized water. A stock solution of 1×10^{-3} mol/l of CrCl₃·6H₂O was prepared by dissolving 0.2664 g (from Merck). The working solutions 1×10^{-4} to 1×10^{-3} mol/l chromium were prepared by appropriate dilution of the stock solutions immediately prior to their use.

The sorption of chromium(III) on resins was studied by the batch technique. A thermostat shaker (Gallenkamp Incubator) of Orbital model was used for adsorption experiments. The general method used for this study is described as follows: 0.2 g of resin was equilibrated with 30 ml of the chromium(III) solution of known concentration in a stoppered pyrex glass flask at a fixed temperature in a thermostatic shaker bath for 2 h. Sorption experiments were performed by equilibration of the exchanger with chromium solutions at various concentrations. The concentration of the active groups on the resin was always in excess of that of the metal ions.

The resins were separated by filtration and washed in distilled water. The filtrate was analyzed by an inductively coupled plasma spectrophotometer (Varian Vista Model ICP-AES) for chromium(III) content. The concentration of chromium metal ions were calculated from the change in metal concentration in the aqueous solution before and after equilibrium sorption from the weight of the dry resin used. In all cases, mass balance was confirmed.

The initial pH's of the sample solutions were adjusted in the range of 1.5–6 by using 0.1N HCl or dilute NaOH. All pH measurements were performed with a Jenway 3010 digital pH meter. After equilibrium was reached, the pH of the solution was measured and recorded. For the constant pH studies, acetate buffer solutions were used.

Sorption isotherms were carried out with different initial concentrations varying from 0.003 to 0.03 mmol/l of metal while holding the resin amount at constant value at room temperature (25 °C). Resin amount in the batch vessel was varied from 0.2 to 1.0 g for adsorption studies. Contact time adsorption experiments were conducted at 25 °C in a well-mixed pyrex glass vessel with a cover. The sorption of Cr(III) on the resins at different temperatures ranging from 20 to 65 °C was investigated. The stirring speed was

Resins	Lewatit TP 207	Chelex-100 Iminodiacetic acid	
Functional group	Iminodiacetic acid		
Matrix	Cross linked polystyrene	Polystyrene-divinylbenzene	
Structure	Macro porous	Macro porous	
pH range	0–14	0–14	
Bead size	0.4–1.25 mm	0.3–1.0 mm	
Capacity	2.4 mmol/(g of resin)	1.6 mmol/(g of resin)	

General description and properties of resins

Table 1

180 rpm to maintain resin particles in suspension. After the solution's pH was stabilized and equilibrated, the resin was filtered and chromium(III) content in equilibrium solution was measured.

3. Results and discussion

3.1. Effect of time on the removal of chromium(III)

In the present paper, iminodiacetic acid resins were examined for their sorption properties towards chromium(III) metal ion as a function of adsorbent dose, time, equilibrium concentration, temperatures and pH value. Figs. 1 and 2, illustrate the removal of Cr(III) by Chelex-100 and Lewatit TP 207 as time functions. An initial concentration of chromium 1×10^{-3} mol/l was tried and the concentration of chromium remaining in the equilibrated



Fig. 1. Removal of Cr(III) by Chelex-100 as a function of time at pH 4.5. Amount of resin, 0.1 g; temperature, 25 °C.



Fig. 2. Removal of Cr(III) by Lewatit TP 207 as a function of time at pH 4.5. Amount of resin, 0.1 g; temperature, 25 °C.

solution was plotted as a function of the equilibration period for the resins. The concentration of chromium in the solution decreased rapidly within 45–60 min and sorption was virtually completed within 80–120 min. In each case, the decreasing concentration of chromium remaining in the solution indicates that chromium was adsorbed strongly by Chelex-100 and Lewatit TP 207. High adsorption rates of chromium for Lewatit TP 207 resin are observed at the onset; and then plateau values are gradually reached within 40 min. The removal increases with time and attains equilibrium in 60 min for Chelex-100.

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 [11]. The cation exchange capacities of Chelex-100 and Lewatit TP 207 reached in this study are 0.288 and 0.341 mmol for Cr(III) per gram of resins at pH of 4.5, respectively.

3.2. Effect of pH

Fig. 3 shows the uptake of Cr(III) ion as a function of hydrogen ion concentration in the pH range 1.5–6 for an initial metal concentration of 0.03 mmol/l. The variable that has the most significant effect on the affinity of a resin for a particular ion is pH. These chelating ion exchanger with iminodiacetic acid groups have strong complexing properties forming heterocyclic chelates with chromium(III) ions depending on the pH of the mobile phase [17,18].

The pH value of the solution is an important factor that controls the sorption of Cr(III). The sorption of Cr(III) ion on the resins is clearly very favorable at high pH values at pH 4 and 5. At lower pH, the isotherms become more linear. There is an abrupt increase in Cr(III) sorption when pH is raised from 3 to 4.5. The pH of the aqueous solution of Cr(III) affects its uptake on the resins and in general the uptake decreases at high pH values [11]. It is evident that the removal efficiency of chromium by both resins is reasonably high in the pH range 4.5–5. This sorption trend can likely be ascribed to the effect of competitive binding



Fig. 3. The equilibrium constants for Cr(III) on the Lewatit TP 207 and Chelex-100 as a function of pH. Temperature, $25 \,^{\circ}$ C; amount of resin, 0.1 g; Cr(III) solution (1 × 10⁻³ M), 30 ml.

between Cr(III) and hydrogen ions for the binding sides on the surface of the resins. At low pH, an excess of hydrogen ions can compete effectively with Cr(III) for binding sites, resulting in a lower level of Cr(III) uptake. At pH values below two, hydrogen ions are likely to compete with Cr(III) ions and at pH values above six, Cr(III) ions might precipitate. At higher pH values greater than six, the presence of OH⁻ ions forms the hydroxyl complexes of chromium Cr(OH)₃ [11,17,18]. Chromium(III) metal showed strong affinity towards the resin in the narrow pH range and it can be seen that the percentage adsorbed is at a maximum pH 4.5. The retention of Cr(III) by both resins decreased markedly from pH 5 to 6.

Solution pH had a strong effect on the equilibrium constant of Cr(III). The equilibrium constant increased from a value of 0.1 (at approximately pH 2) to 320 at pH 4.5 for Lewatit TP 207 and 0.1–7 for Chelex-100. This increases is rational; the exchanger being weakly acidic, is practically in the hydrogen form at pH 2 and is gradually converted into metal form as the pH increased. Hence, the following experiments were performed in the solution pH of 4.5.

3.3. Temperature dependence of adsorption

The isotherms for adsorption of Cr(III) ions at pH 4.5 are presented in Fig. 4 for four different temperatures (20, 35, 50 and 65 °C). The effect of temperature on the equilibrium constant (K_c) for the adsorption of metal ions onto resins was investigated in this stage. Equilibrium constants for Cr(III) were high and adsorption increased slightly with temperature for both resins. This is due to the exothermic adsorption reactions of Cr(III) ion with resins. The equilibrium constants for such reactions slightly increases with temperature. The carboxylic groups of iminodiacedic acid resin are partially protonated at all temperatures but their deprotonation degree increases at all temperature resulting in a slight increase metal capacities at high temperature. Where only ion exchange is involved, the temperature effect on retention time is relatively small.

Few studies of temperature changes have been carried out on chelating ion exchangers. Empirical studies show that temperature has a significant effect on retention in chelation



Fig. 4. The equilibrium constants for Cr(III) on the Lewatit TP 207 and Chelex-100 as a function of temperature at pH 4.5. Amount of resin, 0.1 g; Cr(III) solution $(1 \times 10^{-3} \text{ M})$, 30 ml.

exchange. The "chelate effect" concerns a large positive change in entropy on formation of a chelate complex, which means that the change of Gibbs free energy with temperature will be negative. Considering other thermodynamic factors, it is possible that the entropy change in chelation reactions may have a more important bearing on temperature effects than the enthalpy of sorption. Thus, as the temperature increases, the resulting ΔG will become more negative and so the equilibrium constant will become larger as temperature increases [9,14,19–21].

Assuming that the activity coefficients are unity at low concentrations, (the Henry's law sense), thermodynamic parameters were calculated using the following relations [18].

$$K_{\rm c} = \frac{C_{\rm Ae}}{C_{\rm e}} \tag{1}$$

$$\Delta G^{\circ} = -RT \ln K_{\rm c} \tag{2}$$

$$\log K_{\rm c} = \frac{\Delta S^{\circ}}{2.303R} - \frac{\Delta H^{\circ}}{2.303RT} \tag{3}$$

where K_c is the equilibrium constant, C_e the equilibrium concentration in solution (mg/l) and C_{Ae} the solid-phase concentration at equilibrium (mg/l). ΔG° , ΔH° , and ΔS° are changes in free energy, enthalpy, and entropy, respectively. ΔH° and ΔS° were obtained from the linear Van't Hoff plot of log K_c versus 1/T and presented in Table 2. Negative ΔG° values confirm the feasibility of the process and spontaneous nature of the adsorption with high preference of Cr(III) for the resins. The negative values of ΔH° indicate the exothermic nature of the process while the negative ΔS° corresponds to a decrease in the degree of freedom of the adsorbed species [22].

3.4. Sorption isotherm as a function of metal concentration

Equilibrium isotherm, the relation between the amount exchange (q_e) and the remaining concentration in the aqueous phase (C_e) , is important to describe how solutes interact with the resins and so is critical in optimizing the use of the resins [23]. Chromium ion adsorption isotherms of Chelex-100 and Lewatit TP 207 resins are presented in Figs. 5 and 6 as a function of the equilibrium concentration of metal ions in the aqueous medium at room temperature $(25 \,^\circ\text{C})$ for 2 h of contact time. The amount of Cr(III) ions adsorbed per unit mass of the resin increased with the initial metal concentration as expected. To reach the plateau values that represent saturation of the active groups which are available for interaction with Cr(III) ions on the resins, in other terms, to obtain the maximum sorption capacities for the

Table 2 Thermodynamic parameters for the adsorption of Cr(III) on Chelex-100 and Lewatit TP 207 resin

Resin	ΔH° (J mol ⁻¹)	$\frac{\Delta S^{\circ}}{(\mathrm{J}\mathrm{K}^{-1}\mathrm{mol}^{-1})}$	$\Delta G^{\circ} (\mathrm{J} \mathrm{mol}^{-1})$			
			$T = 298.15 \mathrm{K}$	$T = 308.15 \mathrm{K}$	$T = 323.15 \mathrm{K}$	$T = 338.15 \mathrm{K}$
Chelex-100 Lewatit TP 207	-37.36 -46.98	-111.39 -111.36	-33158.24 -33154.22	-34857.67 -34853.94	-36557.11 -36553.67	-38256.54 -38253.39



Fig. 5. Sorption isotherm of Cr(III) on Chelex-100 as a function of initial metal concentration $(1 \times 10^{-4} \text{ to } 1 \times 10^{-3} \text{ M})$ at pH 4.5. Amount of resin, 0.2 g; temperature, 25 °C.

interested metal ions, the initial concentration were increased from 0.003 to 0.03 mmol/l for Cr(III). Both resins were saturated at relatively low concentrations indicating strong binding for Cr(III). The isotherm plot in Fig. 6 clearly shows a steep slope in the initial solution phase concentration range for pH 4.5. This is a desirable feature of a sorption system as it implies efficient uptake of Cr(III) by the Lewatit TP 207 in dilute chromium solution.

3.5. Effect of resin dosage

The effects of variation of resin dosage on the removal of chromium by Chelex-100 and Lewatit TP 207 ion-exchange resins are shown in Figs. 7 and 8. Resin dosage was varied from 0.2 to 1.0 g and equilibrated for 2 h at an initial chromium concentration of 1×10^{-3} M. It is apparent that the equilibrium concentration in solution phase decreases with increasing resin doses for a given initial chromium concentration. Since the fraction of metal removed from the aqueous phase increases as the sorbent dosage is increased in the batch vessel



Fig. 6. Sorption isotherm of Cr(III) on Lewatit TP 207 as a function of initial metal concentration (1×10^{-4} to 1×10^{-3} M) at pH 4.5–5. Amount of resin, 0.2 g; temperature, 25 °C.



Fig. 7. Effect of resin dosage (0.2–1 g) on the adsorption of Cr(III) on Chelex-100 resin at pH 4.5. Cr(III) solution (1×10^{-3} M), 30 ml; temperature, 25 °C.

with a fixed initial metal concentration, the curves in Figs. 7 and 8 approach asymptotic values from 0.2 to 1 g resin. An increase in the sorbent amount resulted in a decrease in the contact time required to reach equilibrium. This result was anticipated because for a fixed initial solute concentration, increasing adsorbent doses provides greater surface area and adsorption sites of the adsorbent [11]. For the quantitative removal of 1×10^{-3} M Cr(III) in 30 ml, a minimum resin dosage of 0.8 g of Chelex-100 and 0.4 g of Lewatit TP 207 is required.

3.6. Isotherm modeling

Thermodynamic parameters were calculated and the adsorption data fit the Langmuir isotherm equation. The Langmuir isotherm applies to adsorption on a completely homogenous surface with negligible interaction between adsorbed molecules [14,21]. The Langmuir treatment is based on the assumption that maximum adsorption depends on the saturation level of a monolayer of adsorbate molecules on the adsorbent surface, that the energy of adsorption is constant, and that there is no transmigration of adsorbate molecules in the surface plane [24–26].



Fig. 8. Effect of resin dosage (0.2–1 g) on the adsorption of Cr(III) on Lewatit TP 207 resin at pH 4.5. Cr(III) solution (1×10^{-3} M), 30 ml; temperature, 25 °C.

Resin	Langmuir iosotherm					
	$Q_0^{\mathbf{a}}$	b ^b	R _L	Correlation coefficient		
Chelex-100	0.2882	0.0884	0.9999	0.8955		
Lewatit TP 207	0.3408	0.2158	0.9999	0.9508		

Parameters of Langmuir isotherm for sorption on Chelex-100 and Lewatit TP 207

^a mmol/g resin.

^b l/mmol.

Langmuir equation:

$$\frac{C_{\rm e}}{q_{\rm e}} = \frac{C_{\rm e}}{Q_0} + \frac{1}{Q_0 b} \tag{4}$$

where C_e is the equilibrium concentration (mg/l), q_e the amount of adsorbed material at equilibrium (mg/g), *b* the "affinity" parameter or Langmuir constant (l/mg), and Q_0 the "capacity" parameter (mg/g) [11]. The Langmuir isotherm parameter was determined by least-squares fit of the sorption data in Figs. 5 and 6. Q_0 and *b* were determined from the slope and intercept of the Langmuir plot and are presented in Table 3. The Langmuir isotherm equation provided an excellent fit to the equilibrium sorption data, giving correlation coefficients of 0.8955 and 0.9508 on the two sorbents, respectively.

The essential feature of the Langmuir equation can be given in terms of a dimensionless separation factor, R_L , defined as.

$$R_{\rm L} = \frac{1}{1 + bC_0}\tag{5}$$

where C_0 is the highest initial solute concentration. The value of R_L indicates the shape of the isotherm to be unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$), or irreversible ($R_L = 0$) [22]. In this work, the values of R_L obtained are 0.9999 and listed in Table 3. All ion-exchange reactions realized in the sorptions produced favorable isotherms.

3.7. Adsorption kinetics

Experiments were also performed to understand the kinetics of chromium removal by these resins. It is well established that the adsorption of ions in aqueous system follows a reversible first-order kinetics, when a single species is considered on a heterogeneous surface. Two important physicochemical aspects for parameter evaluation of the sorption process as a unit operation are the kinetics and the equilibrium. 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 defining the efficiency of sorption. Hence, in the present study, the kinetics of chromium removal has been carried out to understand the behavior of these adsorbents.

The sorption of chromium from liquid to solid phase may be expressed as [11].

$$A_{k_2}^{\underline{k_1}}B \tag{6}$$

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

Table 3

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

$$\frac{\mathrm{d}x}{\mathrm{d}t} = k_1(a-x) - k_2 x \tag{7}$$

where k_1 and k_2 are the rate constants for the forward and reverse process.

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

$$\frac{dx}{dt} = 0 \text{ or } K_{c} = \frac{X_{e}}{a - X_{e}} = \frac{k_{1}}{k_{2}}$$
(8)

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

$$-\ln\left(1-\frac{x}{X_{\rm e}}\right) = (k_1+k)t\tag{9}$$

The above equation can be written in the form of

$$\ln(1 - U_t) = -(k_1 + k_2)t = -kt \tag{10}$$

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

 U_t can be calculated using the expression

$$\frac{C_{A0} - C_{At}}{C_{A0} - C_{Ae}} = \frac{x}{X_e} = U_t$$
(11)

where C_{A0} is the initial concentration of chromium, C_{At} the concentration of chromium present at any time (*t*), C_{Ae} 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 1–2 h.

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*, the overall rate constant *k* for a given concentration of chromium was calculated by considering the gradients of the linear regressions in Fig. 9a and b and by using Eqs. (8) and (10) the equilibrium constant K_c , forward and backward rate constants k_1 and k_2 were calculated and shown in Table 4 [27]. The forward rate constants for the removal of chromium were much higher than the backward rate constants namely the desorption process (Table 4). This suggests that the rate of adsorption is clearly dominant.

Table 4 Rate constants for the removal of Cr(III) with Chelex-100 and Lewatit TP 207 system

Resin type	Cr(III) amount (mmol)	Overall rate constant $k = k_1 + k_2 (h^{-1})$	Forward rate constant k_1 (h ⁻¹)	Backward rate constant k_2 (h ⁻¹)
Chelex-100	0.0600	0.0344	0.0274	0.0070
Lewatit TP 207	0.0600	0.2159	0.2094	0.0065



Fig. 9. Kinetic fits for the adsorption of chromium on (a) Chelex-100 and (b) Lewatit TP 207.

4. Conclusion

When the sorption equilibrium is known precisely, it is possible to predict the optimum conditions for a pre-concentration or removal of chromium(III) ions from waste streams. The present work demonstrates that both resins have a large collective adsorption with Cr(III) ion, TP 207 showing stronger binding and a strong chelating ability.

The isotherm plots showed a high sorption for pH 4.5 for both of the resins. This is a desirable feature of sorption systems as it implies efficient uptake of Cr(III) ion by the resins in dilute solutions. These chelating resins are thus attractive sorbents for the treatment of wastewater containing Cr(III) ion at trace levels. For a given Cr(III)-bearing wastewater it is possible to design a batch treatment system to remove Cr(III) ion at any pH within the range 3–6. A batch contactor requires about 2 h contact time to achieve 80–95% removal percentages from aqueous solution.

The application of the both resins to leather tanning, electroplating, textile and dyeing wastewater for adsorption of toxic chromium metal is economic. According to the results of the present study, Chelex-100 and Lewatit TP 207 resins can be used effectively for the removal of Cr(III) from aqueous solutions.

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