



Removal of hexavalent chromium from aqueous solutions by D301, D314 and D354 anion-exchange resins

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ABSTRACT

Removal of hexavalent chromium from electroplating industry wastewater is obligatory in order to avoid pollution. Batch shaking experiments were carried out to evaluate the adsorption capacity of resins (D301, D314 and D354) in the removal of chromium from aqueous solutions. Varying experimental conditions were studied, including Cr^{6+} concentrations, resin amounts, initial pH, contact time and temperatures. The ion-exchange process, which is pH-dependent, indicated the maximum removal of Cr^{6+} in the pH range of 1–5 for an initial concentration 100 ppm of Cr^{6+} . It was found that more than 99.4% of the removal was achieved under optimal conditions. High adsorption rates of chromium for the three resins were observed at the onset, and then plateau values were gradually reached within 30 min. The experimental results obtained at various concentrations ($27 \pm 1^\circ\text{C}$) showed that the adsorption pattern on the resins have followed Langmuir isotherms and the calculated maximum sorption capacities of D301, D314 and D354 were 152.52, 120.48 and 156.25 mg/g, respectively. The thermodynamic parameters (free energy change ΔG , enthalpy change ΔS and entropy change ΔH) for the sorption have been evaluated. It was also found that the adsorption of chromium on these anion-exchange resins follows first-order reversible kinetics.

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1. Introduction

Wastewater containing chromium is a major source of environmental pollution. Electronic process waste is one of the major contributors to chromium pollution that generates a large amount of liquid and sludge wastes containing heavy metals. According to the relevant studies performed by the Waste's Institute of the Ministry of Environment (PR China), this category of waste represented about 4 billion tonnes in China during the year 2001 [1]. Chromium is of high toxicity, especially in the form of hexavalent chromium, even at low concentrations. Chromium is considered as a powerful carcinogenic agent that modifies the DNA transcription process causing important chromosomal aberrations [2]. The treatment and removal of chromium has received considerable attention. It is well known that legal standards on environment control are becoming stricter and stricter. The recovery of the chromium is of interest in terms of both environmental effects and economic effects [3,4].

In the chromium plating industry, about 20% of the chromic acid purchased is deposited on metal products but the other 80% is lost during the process [5]. Hence, it is necessary to remove chromium

from rinse water. Removal of chromium has been investigated by using phytoextraction, reverse osmosis, adsorption, precipitation, ion-exchange, membrane and biological processes [6–10]. The traditional techniques are based on chemical precipitation coupled to pre- or post-oxidation/reduction followed by filtration in order to concentrate the species of interest. The main disadvantage of these techniques is the production of sludges containing toxic compounds of which the final disposal is often the general landfill [1]. Therefore, the use of other alternative techniques (such as adsorption, ion exchange, membrane and biological processes) based on physical, chemical and biological mechanisms are advisable in order to protect the environment and at the same time recover valuable metal. Nevertheless, many of these approaches are marginally cost-effective or difficult to implement in developing countries. The main advantages of ion exchange are the recovery of metal value, the selectivity, the less sludge volume produced and the meeting of strict discharge specifications [11].

Many studies, which considered the Cr^{6+} removal by ion-exchange resins, have been reported in the relevant literature. Gode and Pehlivan [12] have proved that ion-exchange resins, such as Lewatit Mp 62 and Lewatit M610, can be used for the efficient removal of chromium from water and wastewater. Rengaraj et al. [13] have studied the removal of chromium from water and electroplating wastewater by ion-exchange resins: 1200H, 1500H

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Table 1
Characteristics properties of the ion-exchange resins used

Resins	D301	D314	D354
Matrix	Macroporous styrene divinylbenzene copolymer	Macroporous acrylic acid copolymer	Macroporous styrene divinylbenzene copolymer
Functional groups	–N(CH ₃) ₂	–N(CH ₃) ₂	–N(CH ₃) ₂
Physical form	Insoluble, milkiness beads	Insoluble, amber beads	Insoluble, milkiness beads
Ionic form	Cl [–]	Cl [–]	Cl [–]
Total exchange capacity (mmol/g)	≥4.8	≥7.0	≥4.8
Moisture holding capacity (%)	48–58	60–65	50–60
Shipping weight (g/ml)	0.65–0.72	0.65–0.75	0.65–0.78
Specific gravity (g/ml)	1.03–1.06	1.06–1.10	1.02–1.07
Uniformity coefficient	≤1.6	≤1.5	≤1.6
Particle size range (mm)	0.315–1.25	0.3–1.0	0.315–1.25
Operating pH range	0–9	0–10	0–9
Thermal stability (°C)	0–70	0–90	0–70

Source: Manufacturer supplied.

and IRN97H and concluded that such ion exchanges have perfect effect on removal of chromium. Sule and Ingle [14] have studied the adsorption of Cr³⁺ species by Chelex-100 chelating resin and Cr⁶⁺ by the anion-exchange resin. In another report by Zhao et al. [15], they have analyzed the removal of hexavalent chromium from wastewater, by using an ion-exchange bed with the strongly basic ion-exchange resin Mp 500 (Bayer), and found that all of expanded beds provided better adsorption than the fixed-bed.

The purpose of this work is to investigate some aspects of use of three weakly basic anion exchange: D301, D314, D354 (produce in China). The parameters that influence adsorption, initial Cr⁶⁺ concentration, agitation time, pH, resin amount, temperatures, isotherm and kinetic, were investigated at 27 °C (room temperature). In addition, the equilibrium and kinetic parameters of Cr⁶⁺ at pH 5 were analyzed at 30, 40, 50 and 60 °C, respectively.

2. Experiment

2.1. Experimental materials

The anion-exchange resins D301, D314 and D354 (Guangzhou Mingjun Chemical Co., China) were used in this study. Their physical properties and specifications as reported by the supplier are shown in Table 1. Double distilled water was used in all experiments. All the chemicals used were of AR grade and obtained from Guangzhou Chemical Co., Ltd. Solutions of 0.01 M NaOH and HCl were used for pH adjustment. Cr⁶⁺ stock solution of 200 mg/l was prepared from potassium salts of dichromate (K₂Cr₂O₇) of AR grade.

2.2. Experimental methods

UV–vis 751GD spectrophotometer (China) was used for metal analysis. A PHS-3C pH meter was used to determine pH of solutions. A shaker with thermostat (SHA-C, Leici) of Orbital model was used for adsorption experiments and temperature studies.

The resins, D301, D314 and D354, were washed several times with doubly distilled water. The resins were then conditioned by treating with certain volume of 1 M HCl and 1 M NaOH and left standing for 24 h and then washed with distilled water until the pH ranged from 8 to 9. After air dried, the resin was stored for sorption studies. The resins were treated to change the ion-exchanger to ROH form [1,12].

The sorption curves were determined by a batch procedure. The influence of the amounts of the chromium for the sorption procedure was investigated by using 40–190 ppm Cr⁶⁺ solutions. For the measurements of the exchange equilibrium, a series of samples of swollen resin (~0.02 g in the dry state) were equilibrated with a 0.031 K₂Cr₂O₇ solution of different concentrations (40–190 mg

Cr/l) at room temperature (27 ± 1 °C) in a thermostatic shaker bath for 2 h.

For initial pH effect, 100 mg/l Cr⁶⁺ solution and D301, D314 and D354 each with a dose of 0.025 g in the dry state were used and pH was adjusted using 0.01 M HCl or 0.01 M NaOH solutions. The solutions were agitated in the shaker for 2 h.

The effect of the resin dosage for adsorption was studied in the batch vessel by varying the resin from 0.01 to 0.05 g. Contact time adsorption experiments were conducted at room temperature (27 ± 1 °C) in a well-mixed pyrex glass vessel with a cover.

The sorption of Cr⁶⁺ on the resins at different temperatures ranging from 30 to 60 °C was investigated in the thermostatic shaker and after equilibrated, the resin was filtered and content of Cr⁶⁺ in equilibrium solution was measured after 2 h.

Kinetics measurements: samples of the resins in the swelling form (0.05 g in the dry state) were contacted with a 0.031 K₂Cr₂O₇ solution (100 ppm). The concentration of Cr⁶⁺ ions from the aqueous solution was determined at known time intervals.

The resins were separated. The filtrate was analyzed by UV–vis for Cr⁶⁺ content. The amounts of Cr⁶⁺ adsorbed on the precipitates at time *t*, *q_t* (mg/g), were calculated by the following mass-balance equation:

$$q_t = \frac{(C_0 - C_t)V}{m} \quad (1)$$

where *C*₀ and *C_t* are the initial and final Cr⁶⁺ concentrations at time *t* (mg/l), respectively, *V* is the total volume of the solution (l), and *m* is the mass of adsorbents used (g).

3. Results and discussion

3.1. Effect of pH

Different forms of Cr⁶⁺ were found in the solution, and the relative proportions depended on both pH and total Cr⁶⁺ concentration. The chromate may be represented in various forms such as H₂CrO₄, HCrO₄[–], CrO₄^{2–}, HCr₂O₇[–], Cr₂O₇^{2–} in the solution phase as a function of pH and concentration. Only CrO₄^{2–} ions exist in the solution throughout the experimental concentration range when pH > 6.5; in the pH range from 0 to 6.5, HCrO₄[–] and Cr₂O₇^{2–} are predominant [16]. So in acidic conditions, 1 g equivalent resin can absorb 1 g chromium, and in alkaline conditions, 2 g equivalent resin can absorb 1 g chromium only [17]. The sites responsible for the ion-exchange process are due to the –N(CH₃)₂ groups in the three resins' matrix. An anion-exchange resin will absorb chromates from aqueous solution according to the reactions shown in Eqs. (2)–(5)



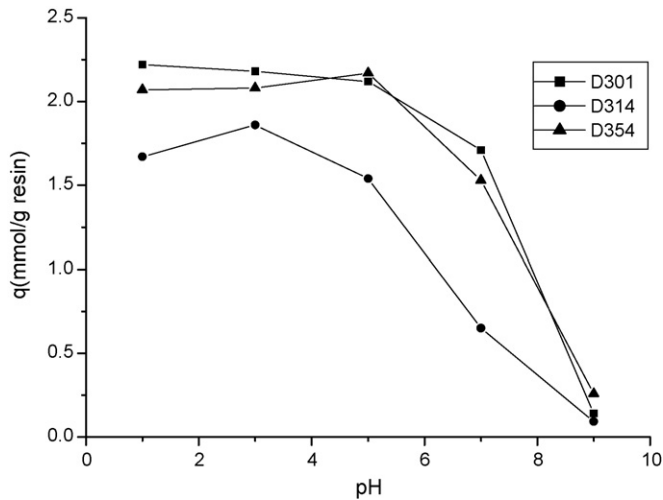


Fig. 1. The effects of pH on the sorption of Cr^{6+} by D301, D314 and D354 (adsorption conditions—initial pH, 1–9; initial concentration of Cr^{6+} , 100 ppm; amount of resin, 0.025 g; volume of adsorption medium, 30 ml; temperature, $27 \pm 1^\circ\text{C}$; stirring time, 180 rpm; adsorption time, 120 min).

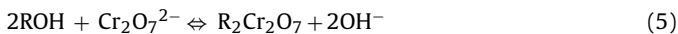
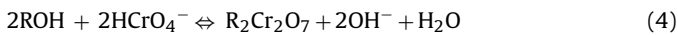
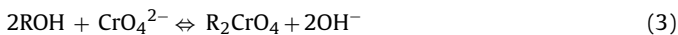


Fig. 1 shows the effect of initial pH (1–9) on the removal of chromium by D301, D314 and D354 weakly basic anion exchange. As pH changes from 1 to 5, the removal of Cr^{6+} changes slightly. When $\text{pH} > 7$, the removal of Cr^{6+} from the solution decreased sharply as the pH increased. When pH 9, the adsorption amounts of Cr^{6+} was almost 10% of the ones on acidic condition. It is known from above that the amounts of Cr can be adsorbed by one active site of the resin in acidic condition is double that in alkali condition. The process is reversible and the exchanged OH^- will react with H^+ in acid condition. So the state of equilibrium was destroyed and the forward reaction was enhanced. Gala'n et al. [18] also noticed that the useful capacity at acidic pH, (about 1.65 mol/l) is double that at neutral pH (about 0.7 mol/l), due to the presence of different ionic species in solution when there is a change in pH. At alkaline pH values, the sorption trend can likely be ascribed to the effect of competitive binding between CrO_4^{2-} and OH^- for the binding sites on the surface of the resins. At higher pH, an excess of OH^- can compete effectively with CrO_4^{2-} for bonding sites, resulting in a lower level of Cr^{6+} uptake [1]. Hence, the following pH range is suggested to be 1–5. Additionally, Gode and Pehlivan [12] has reported that the optimal pH range is 2–6.

3.2. Effect of resin amount

It is apparent from Fig. 2 that by increasing the resin amount, the adsorption efficiency increases but adsorption density decreases. It is readily understood that the number of available adsorption sites increases by increasing the resin amount and it, therefore, results in the increase of removal efficiency. 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 [19]. As expected, the equilibrium concentration decreases with increasing adsorbent doses for a given initial chromium concentration, because increasing the adsorbent doses provides a greater

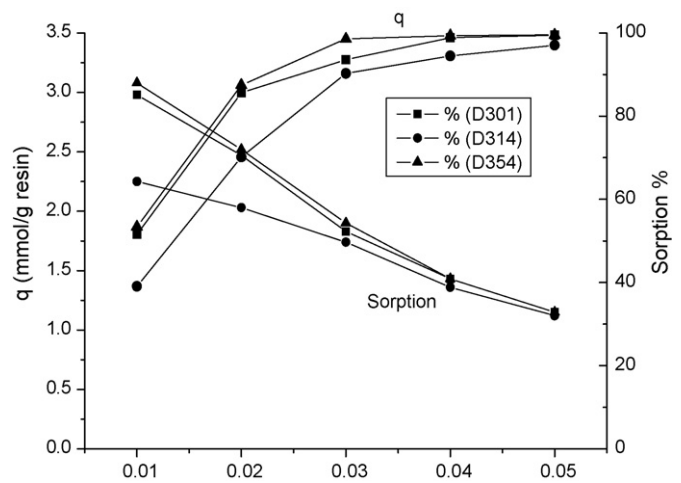


Fig. 2. The effects of the amount of resins for Cr^{6+} by D301, D314 and D354 (adsorption conditions—initial concentration of Cr^{6+} , 100 ppm; amount of resin, 0.01–0.05 g; volume of adsorption medium, 30 ml; temperature, $27 \pm 1^\circ\text{C}$; stirring time, 180 rpm; adsorption time, 120 min; initial pH, 3).

surface area or adsorption sites for a fixed initial solute concentration [20,21].

3.3. Effect of contact time

Fig. 3 shows the effect of contact time on the removal of chromium by resins. The removal increases with time and almost reaches the attains equilibrium in 30 min. High adsorption rates of chromium for the three resins are observed at the onset, and then plateau values are gradually reached within 60 min. The increasing contact time increased the Cr^{6+} adsorption and it remains constant after equilibrium reached in 30 min for initial concentration of 100 ppm. In each case, the decreasing concentration of Cr^{6+} remaining in the solution, indicates that chromium was adsorbed strongly by D301, D314 and D354. The recovery of the chromium is of interest in terms of both environmental effects and economic effects.

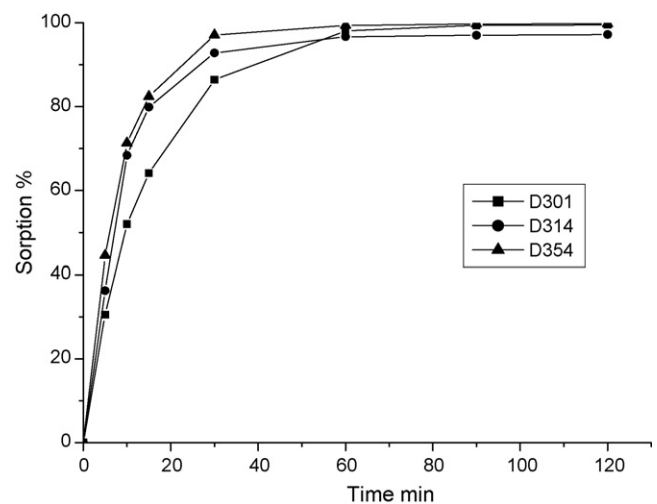


Fig. 3. Effect of contact time on the sorption of Cr^{6+} by D301, D314 and D354 (adsorption conditions—initial concentration of Cr^{6+} , 100 ppm; amount of resin, 0.05 g; volume of adsorption medium, 30 ml; temperature, $27 \pm 1^\circ\text{C}$; stirring time, 180 rpm; initial pH, 3).

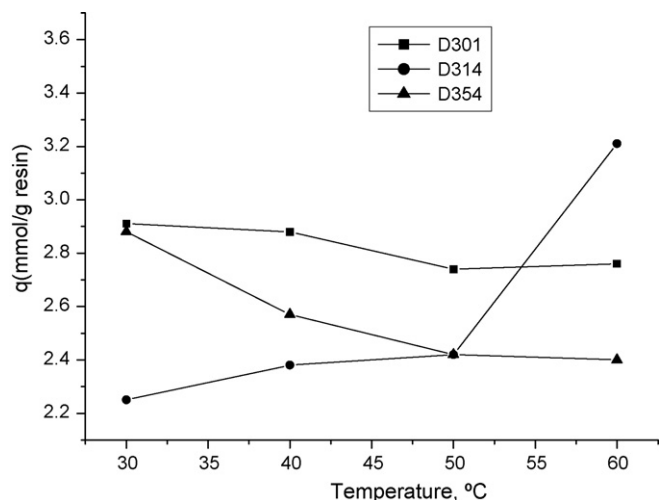


Fig. 4. Effect of temperature on the sorption of Cr⁶⁺ by D301, D314 and D354 (adsorption conditions—initial concentration of Cr⁶⁺, 100 ppm; amount of resin, 0.01 g; volume of adsorption medium, 30 ml; temperature, 30–60 °C; stirring time, 180 rpm; adsorption time, 120 min; initial pH, 3).

3.4. Effect of temperature

The effects of temperature on the sorption of Cr⁶⁺ by D301, D314 and D354 are shown in Fig. 4. The equilibrium adsorption level for D301 and D354 decreased slightly with increasing temperature, while it increased for D314 and it is especially sharp from 50 to 60 °C. The ion-exchange capacity increases with temperature with the endothermic reactions. It decreases with temperature while the reaction must be exothermic. Hence, for D301 and D354, a general temperature is suggested to be room temperature, for D314 60 °C. Thermodynamic parameters showed in Fig. 5 and Table 2 such as free energy change (ΔG), enthalpy change (ΔH) and entropy

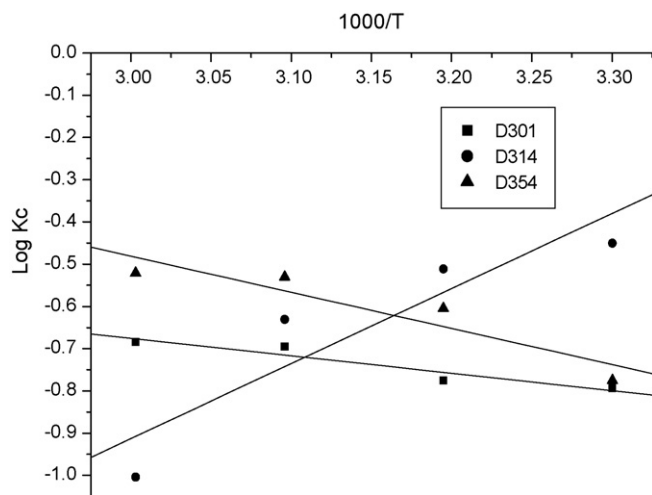


Fig. 5. Thermodynamic fits for the adsorption of Cr⁶⁺ on D301, D314 and D354 resin.

Table 2 Thermodynamic parameters for the adsorption of Cr⁶⁺ on D301, D314 and D354 resin

Resin	ΔH (J mol ⁻¹)	ΔS (J K ⁻¹ mol ⁻¹)	ΔG (J mol ⁻¹)			
			T = 303 K	T = 313 K	T = 323 K	T = 333 K
D301	7.906	10.784	-4603.17	-4646.04	-4397.26	-4360.41
D314	-34.007	119.5	-2611.04	-3064.46	-3900.41	-6400.18
D354	16.356	39.88	-4497.60	-3625.51	-3281.41	-3321.48

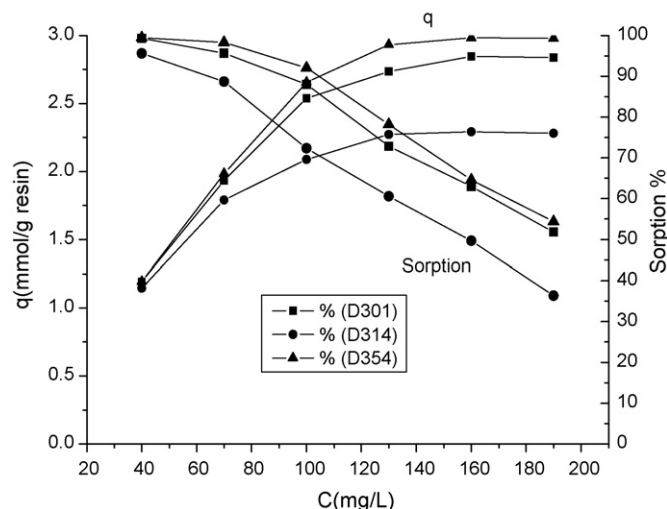


Fig. 6. Effect of initial conditions on the sorption of Cr⁶⁺ by D301, D314 and D354 (adsorption conditions—initial concentration of Cr(VI), 40–190 ppm; amount of resin, 0.05 g; volume of adsorption medium, 30 ml; temperature, 27 ± 1 °C; stirring time, 180 rpm; adsorption time, 120 min; initial pH, 3).

change (ΔS) can be calculated by Eqs. (6)–(8). The temperature of the mixture was ranged from 30 to 60 °C. The negative ΔG indicates the process for D301, D314 and D354 to be spontaneous in nature of adsorption. The positive ΔH shows that the adsorption processes for D301, D354 are exothermic reactions, while the negative ΔH for D314 proves it to be an endothermic reaction. The reason why adsorption level for D301 and D354 decreased increasing temperature while increased for D314 can be explained by the value of ΔH .

$$K_C = \frac{C_{Ae}}{C_e} \tag{6}$$

$$\Delta G = -RT \ln K_C \tag{7}$$

$$\log K_C = \frac{\Delta S}{2.303R} - \frac{\Delta H}{2.303RT} \tag{8}$$

where K_C is the equilibrium constant, C_e is the equilibrium concentration in solution (mg/l) and C_{Ae} is the solid phase concentration at equilibrium (mg/l).

3.5. Adsorption isotherms

The capacities of the three resins are different. A study of the maximum adsorption of Cr⁶⁺ from its solution by 0.05 g resin (dry state) was done at pH 4–5 by varying the concentration of Cr⁶⁺ (40–190 ppm), of which the results are given in Fig. 6. A remarkable feature of the results is that the adsorption amounts of Cr⁶⁺ increased as the initial amount of Cr⁶⁺ in solution increased, however, the percentage of sorption decreases. It can be also seen that D301 and D354 resins have higher adsorption capacity for chromium than the D314 resin.

The experimental results obtained at various concentrations shows that the adsorption pattern on the resins followed Langmuir isotherms better than Freundlich isotherms as seen in Fig. 7a and b. The Langmuir isotherm is valid for monolayer adsorption onto a surface containing a finite number of identical sites. However, Rivero et al. [22] has reported that the adsorption of chromium by resin Lewatit MP-64 was properly described by the empirical Freundlich isotherm, which may be attributed to the different characteristics of different resins. The Langmuir isotherm and Freundlich isotherm

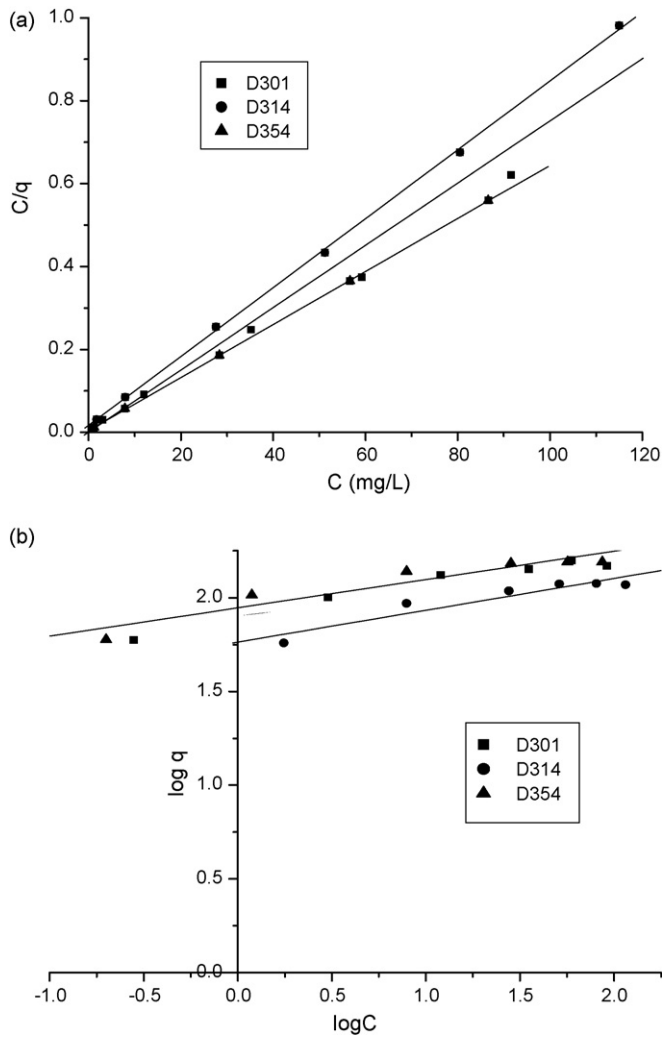


Fig. 7. Langmuir isotherm (a) and Freundlich isotherm (b) plot for the adsorption of chromium by D301, D314 and D354.

are represented by the following Eqs. (9) and (10), respectively [23].

$$\frac{C}{q} = \frac{C}{Q_M} + \frac{1}{Q_M b} \quad (9)$$

$$\log q = \log K_f + \log \left(\frac{C}{n} \right) \quad (10)$$

where C is the equilibrium concentration (mg/l), q is the amount of adsorbed material at equilibrium (mg/g), b is the “affinity” parameter or Langmuir constant (l/mg), and Q_M is the “capacity” parameter (mg/g). The values of the adsorbents are shown in Table 3.

3.6. Adsorption kinetics

Experiments were also directed at an attempt to understand the kinetics of chromium removal by the D301, D314 and D354 resins.

Table 3
Summary of isotherm parameters for the adsorption of Cr^{6+} on ion-exchange resins

Resin	Langmuir isotherm			Freundlich isotherm		
	Q_M (mg/g)	b	R^2	K_f (mg/g)	n	R^2
D301	151.52	0.9166	0.9973	79.086	6.094	0.9493
D314	120.48	0.5000	0.9995	58.036	5.903	0.892
D354	156.25	1.5614	0.9999	88.328	6.627	0.8981

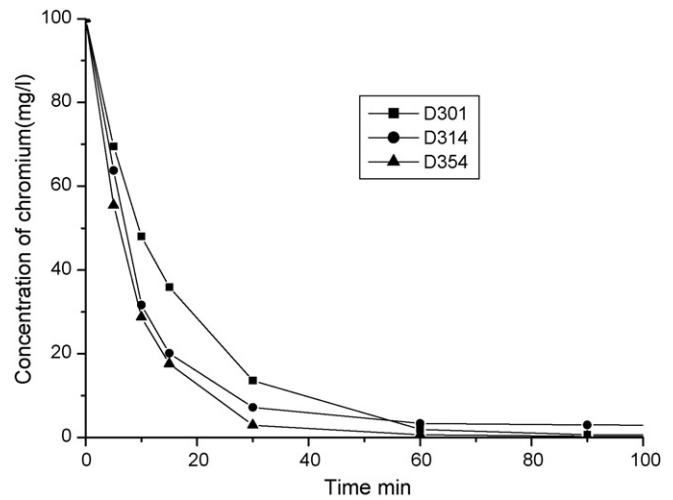


Fig. 8. Kinetics of adsorption of Cr^{6+} on D301, D314 and D354.

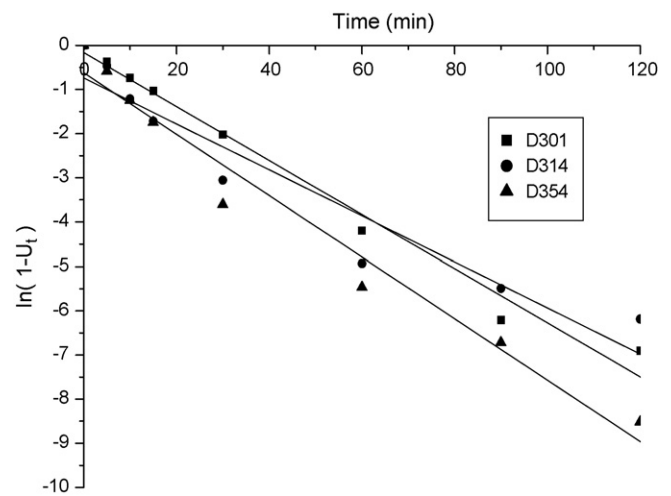


Fig. 9. Kinetic fits for the adsorption of Cr^{6+} on D301, D314 and D354.

It is a well-established fact that the adsorption of ions in aqueous system follows reversible first-order kinetics, when a single species is considered on a heterogeneous surface. 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 [24].

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



where k_1 is the forward reaction rate constant and k_2 is the backward reaction rate constant. Using the kinetic equations numbered (8)–(20) in ref. [13]. By using the data from Fig. 8, plotting $\ln(1-Ut) - t$ the overall rate constant k for given concentration of chromium was calculated by considering the slope of straight line in (Fig. 9). The equilibrium constant, K_C , forward and backward rate constants, k_1 and k_2 , were calculated and shown in Table 4. From Table 4, it is found that the forward rate constants for removal of chromium were much higher than the backward rate constants. The sorption of Cr^{6+} onto the resins followed reversible first-order

Table 4
Rate constants for the adsorption of Cr^{6+} with D301, D314 and D354 resins

Resin type	Cr^{6+} amount (mmol)	Overall rate constant ($k = k_1 + k_2$)	Forward rate constant k_1	Backward rate constant k_2
D301	0.0577	0.0604	0.06022	0.00018
D314	0.0577	0.0490	0.04773	0.00127
D354	0.0577	0.0670	0.06689	0.00011

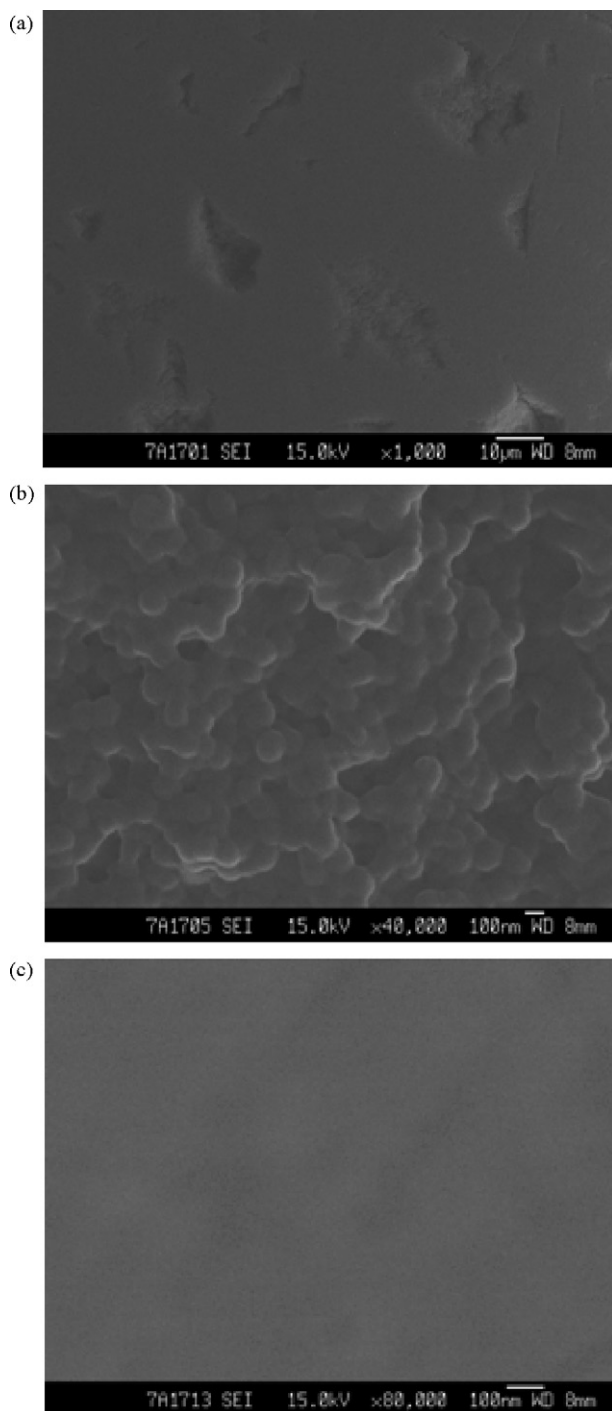


Fig. 10. Electron microscope images of D301 (a), D314 (b) and D354 (c).

Table 5
Composition of chromium containing electroplating industry wastewater

Serial number	Compound	Value (mg/l, except pH)
1	Cr^{6+}	61.6
2	Cr^{3+}	0.98
3	Fe^{3+}	0.08
4	Cu^{2+}	0.01
5	Zn^{2+}	1.12
6	Ca^{2+}	16.1
7	K^+	0.17
8	Cl^-	25.7
9	SO_4^{2-}	104.3
10	COD	38.2
11	pH	5.3

rate kinetics. This suggests that the rate of adsorption is clearly dominant.

3.7. Morphology characteristics

These differences of those resins can be seen directly from their electron microscope (JSM-6330F) photographs in Fig. 10. It showed that more physical holes were distributed on the surface of D314 (b) than D301 (a). But the surface of D354 (c) is so smooth that no physical hole can be found with 80,000 \times magnification. Obviously, the order of special surface area of these resins is D314 > D301 > D354. However, Fig. 5 showed that D301 and D354 resins have higher adsorption capacity than D314 resin.

3.8. Application to electroplating industry wastewater

The composition of chromium containing electroplating industry wastewater is presented in Table 5. The electroplating industry wastewater was supplied by Guangzhou Zengcheng Electroplating Co., Ltd., it was used for the study with D301, D314 and D354, and then subjected to treatment. Maximum removal of chromium from 100 ml of wastewater containing 61.6 mg/l chromium was adsorbed by 50 mg/100 ml each of D301, D314 and D354 resin at a solution pH of 3. The maximum sorption capacities of D301, D314 and D354 towards electroplating industry wastewater were 122.52, 97.3 and 119.6 mg/g, respectively, it is lower than that for synthetic chromium solution. This may be attributed to the presence of other impurities (ions, COD) present in electroplating industry wastewater which may interfere in the ion-exchange process. The COD of the effluent is decreased from 38.2 to 18 mg/l and the mechanism will be further studied and reported in future.

4. Conclusion

The main goal of this work was the assessment of the performance of three commercial anion-exchange resins (D301, D314 and D354) to recover the chromium from industrial effluents. The experimental results suggest that a high sorption for resins is at pH 1–5. The rate for sorption of Cr^{6+} on the three resins was rapid during the first 30 min and equilibrium was found to be attained within 60 min. D301 and D354 resins have higher adsorption capacity for chromium than the D314 resin at the room temperature, but at 60 °C D314 resin shows higher sorption capacity than the others. Equilib-

rium removal by resins follows Langmuir isotherms. The kinetics of chromium adsorption by these resins follow first-order reversible kinetics model. It was found that more than 99.4% removal was achieved under optimal conditions, and adsorption capacity can reach 152.52, 120.48 and 156.25 mg/g for D301, D314 and D354 resins, respectively. In the case of electronic process wastewater, chromium adsorption was particularly damped by the presence of other metal ions. Detailed studies are needed to further evaluate ion-exchange resins in terms of competitive adsorption and their reaction chemistry.

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