

Removal of chromium (VI) from aqueous solutions using Lewatit FO36 nano ion exchange resin

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ABSTRACT: The removal of the chromium (VI) ion from aqueous solutions with the Lewatit FO36 ion-exchange resin is described at different conditions. The effects of adsorbent dose, initial metal concentration, contact time and pH on the removal of chromium (VI) were investigated. The batch ion exchange process was relatively fast and it reached equilibrium after about 90 min of contact. The ion exchange process, which is pH dependent showed maximum removal of chromium (VI) in the pH range 5.0-8.0 for an initial chromium (VI) concentration of 0.5 mg/dm³. The equilibrium related to Lewatit FO36 ion-exchange capacity and the amounts of the ion exchange were obtained using the plots of the Langmuir adsorption isotherm. It was observed that the maximum ion exchange capacity of 0.29 mmol of chromium (VI)/g for Lewatit FO36 was achieved at optimum pH value of 6.0. The ion exchange of chromium (VI) on this cation-exchange resin followed first-order reversible kinetics.

Keywords: Adsorption isotherm; Chromium (VI); Ion exchange kinetics; Lewatit FO36; Nano resin

INTRODUCTION

Heavy metals including chromium, mercury, lead, cadmium, etc. are considered to be high risk for humans and animals. The presences of these pollutants in potable water supplies have become a major issue towards human health. Heavy metals in water originate from both, natural sources such as geological erosion, as well as man-made pollution (mining operations that contaminate ground waters)(Babel and Opiso, 2007). In recent years, there has been a growing concern with environmental protection. Removal of trace amounts of heavy metal ions from wastewater and drinking water is of great importance due to their high toxicity (Abdel-Ghani *et al.*, 2007). Companies that use chromic acid for electroplating, anodizing and other surface finishing operations represent a difficult and costly problem of waste chromic acid solution disposal and the treatment of water to wash parts. Chromium as a common contaminant is usually encountered in the environment in two stable oxidation states, Cr (III) and Cr (VI). Toxicological studies have shown that the degree of toxicity of some elements depends on the chemical form

in which the element is present. Cr (III) is considered as an essential micronutrient for human, plant and animal metabolism. However, Cr (VI) is very soluble and hazardous to health. Humans are exposed to excessive amount of Cr (VI) through food, drinking water and inhaling air that may contain Cr (VI). In natural water chromium ions mainly appear as chromate and cationic hydroxo complexes. It is necessary for risk assessment not only to determine the total chromium in different environments, but also to determine chromium in its different oxidation states (Yang *et al.*, 2003; Yalcin and Apak, 2004; Nouri *et al.*, 2008).

The main sources for contamination of natural waters with Cr (VI) are various industries such as plating, tanning, paint and pigment production and metallurgy. The recommend limits of chromium (VI) in potable water is 0.05 ppm. Thus, the elimination of Cr (VI) from drinking water is of great concern. Besides, numerous processes are available for removing dissolved heavy metals from industrial effluents, including thermal, biological and chemical methods such as; coagulation, chemical precipitation, solvent

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extraction, electrolysis, ultra filtration, membrane separation, adsorption and electro dialysis (Cortina et al., 1996; Kenwy et al., 2000; Samarghandi et al., 2007; Bazrafshan et al., 2008; Mahvi et al., 2008). Sorption operations, including adsorption and ion exchange, are potential alternatives for aqueous solutions (Soylak and Elci, 1997; Seco et al., 1999; Brown et al., 2000; Yu et al., 2000; Yu et al., 2001).

Among various techniques, adsorption is the most promising technique to remove and eliminate Cr (VI) from drinking water. Application of ion exchanger is an attractive method to remove heavy metal contaminations. Nowadays, chelating resins are increasingly used in the removal of metal ions due to their high adsorption capacities, selectivity and durability (Zhang et al., 2001; Llosa et al., 2002; Cumbal et al., 2003; Jeon and Holl, 2003; Reddy and Reddy, 2003). Very good results have been obtained in sorption and separation of trace and milligram amounts of Cr (IV) complexes using chelating ion exchangers. These resins are polymers with the functional groups able to form complexes with selected ions. In contrast to the conventional ion exchange resins, chelating resins combine with ion exchange and complex formation and hence can exhibit high selectivity for some ions or groups of ions, providing a wide range of practical use and industrial applications both in hydrometallurgy and analytical chemistry. The properties of chelating ion exchangers depend on the type of functional groups, though to a lesser extent on grain size and physical properties.

Ion-exchange resins are used in their entire range to improve water quality, depending on the types and concentrations of the impurities. Selective resins were deemed major options for treating wastewater. Traces of environmentally harmful substances accumulate on the Lewatit FO36 ion-exchange resins and are thereby removed from the wastewater. They can be used for the elimination or separation of heavy metals from aqueous solutions, accumulation and recovery of heavy metals, removal of heavy metals from water and process water. Lewatit FO36 has high exchange capacity, very good chemical, mechanical and thermal stability, good ion-exchange kinetics make it suitable for the treatment of electroplating rinse waters. Gel resins usually have higher efficiencies and cost less. A macro-pore gives better physical stability, because of its sponge like structure that gives more stress relief. Lewatit FO36 is suitable for a variety of

applications involving the treatment of the solutions containing metal salts and it has been used for removal of fluoride from water. This resin operates at any pH and requires a substantial amount of regenerant (Gode and Pehlivan, 2003; Antelo and Avena, 2005; Gode and Moral, 2008; Hubicki and Wolowicz, 2008; Boldaji et al., 2009).

The determination of traces heavy metal ions in water, food and atmosphere is very important in removal efficiency of metal contamination from these samples. Recently, several instrumental techniques including UV-Vis spectrophotometry (Gode and Moral, 2008), atomic absorption spectrometry (AAS) (Kolodynska et al., 2008), inductively coupled plasma (ICP) (Liang et al., 2003), ion exchange chromatography (Hubicki and Wolowicz, 2008), etc. have been used for analysis and determination of traces heavy metals in samples.

In this work, the feasibility of using Lewatit FO36 nano particles was investigated as adsorbent for removal Cr (VI) from aqueous solutions using batch technique under various conditions. Also, UV-Vis spectrophotometric technique was applied to determination of Cr (VI) concentration using 1, 5-diphenylcarbazine as chelating agent. This research was performed in Tehran University of Medical Science, Iran, from April until September 2008 in Tehran.

MATERIALS AND METHODS

Materials

Lewatit FO36 was used in this study. Lewatit FO36 contains a layer of iron oxide binds Cr (VI) in a specific surface complex. The typical data of Lewatit FO36 is summarized in Table 1. All other chemicals were purchased from Merck Chemical Company and were AR grade.

Stock solutions (0.01 M) of the chromium ion under study were prepared by dissolving an appropriate weight of pure $K_2Cr_2O_7$ salt in distilled water.

The batch method was used to study the adsorption of Cr (VI) on Lewatit FO36. The performance of the adsorption was carried out at room temperature and the effect of important parameters such as pH, initial concentration of Cr (VI) and resin amount on removal efficiency were studied.

Methods

Batch ion-exchange studies were conducted by stirring of solution for a period of time at a controlled

temperature to achieve equilibration. Batch experiments were carried out at 25 °C in beaker by stirring (398 rpm) a fixed mass of dry resin, with 1000 mL, 1×10^{-3} mol/L Cr (VI) solution until equilibrium was reached. The final metal concentration in the filtrates as well as in the initial solution was determined by the UV-Vis spectrophotometric method using UV-vis spectrophotometer (Hach-Long DR-5000). All experiments were performed in duplicate and the result was taken as the average value of the experiment. The concentration of metal on the resin was calculated as the difference between the original concentration in the solution and the concentration in solution after contact. The amount of metal ion sorbed at time t , q_t , was calculated from the mass balance equation:

$$q_t = \frac{[C_o - C_t]V}{m} \quad (1)$$

Where, C_o , C_t , V and m are the initial concentration of metal ion, concentration of metal ion at time t , volume of solution and mass of adsorbent, respectively.

When equilibrium was reached; $C_t = C_e$ and $q_t = q_e$, then the amount of metal ion sorbed at equilibrium, q_e , was calculated using Eq. 1 (Gode and Pehlivan, 2003). Sorption isotherms were determined with different initial concentrations varying from 0.25 to 3.0 mmol/L of metal, while holding the resin amount at constant value at room temperature. The resin amount in the batch vessel varied from 0.125 to 2.0 g for ion-exchange studies. The shaking speed was 398 rpm to maintain resin particles in suspension. The pH of solutions were changed during equilibrium between resin and solutions. Hence, filtration should be carried out after pH stabilization. After the solutions' pH was stabilized and equilibrated, the resin was filtered and Cr (VI) content in equilibrium solution was measured. Sorption isotherms were plots of the Cr (VI) uptake

and the final equilibrium concentration of the residual adsorbate remaining in the solution. The Langmuir model of ion exchanges was used to compare the single metal sorption performance at different conditions and to determine the capacity of resins for Cr (VI) ion. The ion-exchange experiments were carried out at room temperature (25 ± 1 °C). The initial pH of the sample solutions were adjusted in the range of 5.0–8.0 with 0.1N HCl or diluted NaOH. All pH measurements were performed with a Metrohm E520 pH meter. After equilibrium was reached, the pH of the solution was measured and recorded. Cr (VI) recovery factors (% R) were calculated from Eq. 2:

$$\% R = \frac{C_a}{C_o} \times 100 \quad (2)$$

Where, C_a is the amount of adsorbed metal calculated as the difference in Cr (VI) concentration in the aqueous solution before and after sorption, C_o is the initial concentration of Cr (VI). From these experiments, dependence of % R of Cr (VI) to time was investigated.

Metal ion uptake experiments using the batch method were made in order to determine the total ion exchange capacities (C_t), expressed in mmol of Cr (VI) per g of the ion exchanger. 0.5 g dry resin was placed in a beaker containing 50 cm³ of Cr (VI) ions of increasing concentration. The contents of the beaker were equilibrated on the shaker at room temperature for 90 min. The solution was filtered and Cr (VI) concentration was determined. The amount of Cr (VI) ions adsorbed on the ion exchanger was determined from the isotherms.

RESULTS AND DISCUSSION

In order to optimize the R (the ratio amount of adsorbent to adsorbate) value for maximum removal efficiency at minimum contact time, the experiments were conducted with 0.5 mg/L of Cr (VI) solution containing different amount of Lewatite FO36 cation exchange resin in the pH=7.0, typically (Fig. 1) shows plot of q_e , C_e and standard value of Cr (VI) ion, i.e. 0.05 mg/L. As have been observed, $R=1000$ show optimum result for contact time (90 min) and removal concentration of Cr (VI) ion from solution. According to these data, $R=1000$ (Fig. 1) was selected in all experiments involving pH and kinetic studies as optimum ratio of adsorbent/adsorbate.

The effect of pH of the medium on the uptake of Cr (VI) metal ion is shown in Fig. 2. Generally, as the pH

Table 1: Physical and chemical properties of Lewatit-FO36

Property	Value
Uniformity coefficient	1.1
Density	g/mL 1.25
Bulk density	g/L 0.765
pH - range stability at	4-14
Operating pH - range	4-11
Storability temperature range	Until+40 -20
Storability of the product	2 years .max
Total arsenic capacity	min.g/L > 40
Regenerate	NaOH+NaCl
	(with a mass-ratio of 1:1)



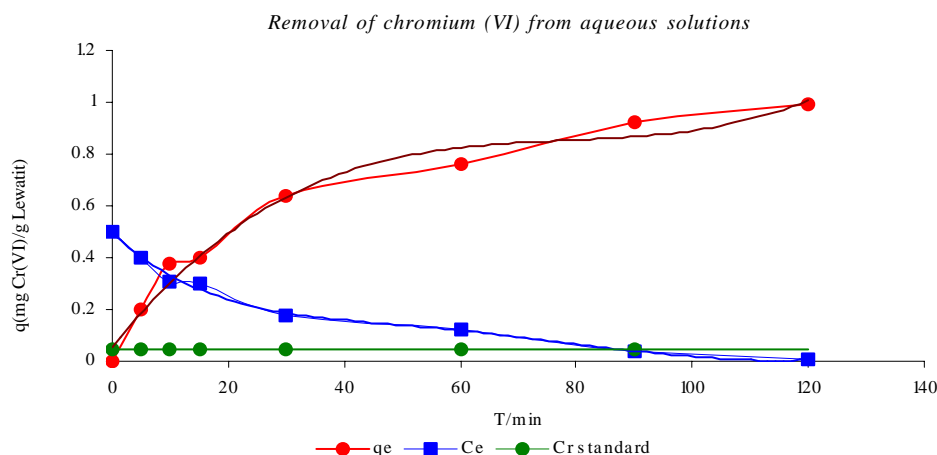


Fig. 1: Standard amount of Cr (VI) versus time for adsorption of Cr (VI) on Lewatit FO36 at different R values: R=1000 (typically)

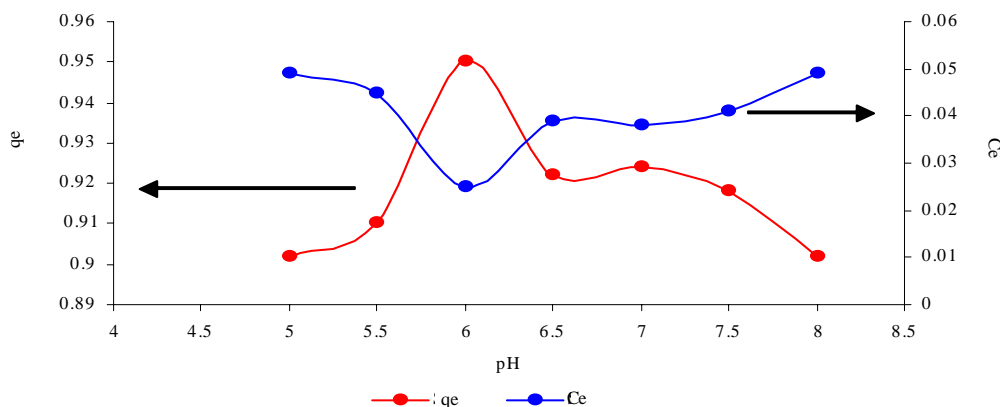


Fig. 2: Effect of pH on the ion exchange of Cr (VI) using the Lewatit FO36

increases, the uptake of metal ion increases due to the dissociation of Hydroxyl group suitable for coordination with metal ions. The experimental conditions such as pH level have strong effects on the equilibrium constant, K_c ; so it can be used as a comparative measure of the efficiencies of various exchangers. The interactions of metals with Lewatit FO36 resin are complex, probably simultaneously dominated by adsorption and ion exchange. The pH dependence of ion exchange may suggest that the metal ions are adsorbed according to the ion-exchange mechanism. The effect of pH on ion exchange of Cr (VI) was studied at room temperature by varying the pH of metal solution-ion exchanger from 5.0 to 8.0.

Fig. 3 shows the change of the uptake of Lewatit FO36 towards Cr ion as a function of time at neutral pH (7.0). It is found that the uptake capacity of the

Lewatit FO36 towards the Cr (VI) ion is comparable to that of other commercial one such as Lewatit S 100, Pmc, YK and MP 62 (Gode and Moral, 2008). On the other hand, maximum uptake was obtained through 90 min for Lewatit FO36. The Cr (VI) removal increased with time and attained equilibrium at about 90 min for Cr (VI) for 5 mg/L chromium ion concentration used (Pradhan *et al.*, 1999). The percentage of metal removal increased rapidly up to approximately 60 min for resin and thereafter, rose slowly before attaining a saturation value in 120 min. A further increase in contact time had a negligible effect on the percent removal. The percentage metal removal is higher in the beginning due to greater number of resin exchange sites available for the sorption of the metal. The ion-exchange process is the major mechanism for the removal of metal ions from solution within the pH

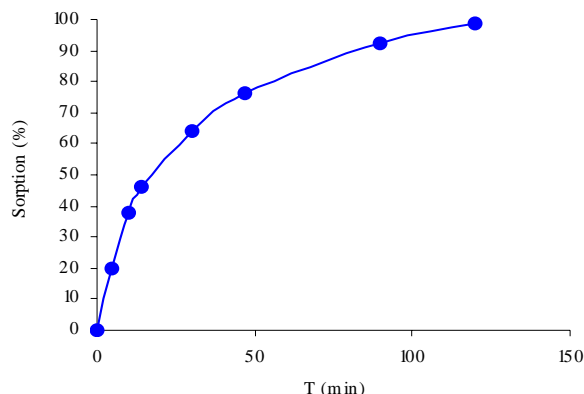


Fig. 3: Effect of contact time on the ion exchange of Cr (VI) using Lewatit FO36

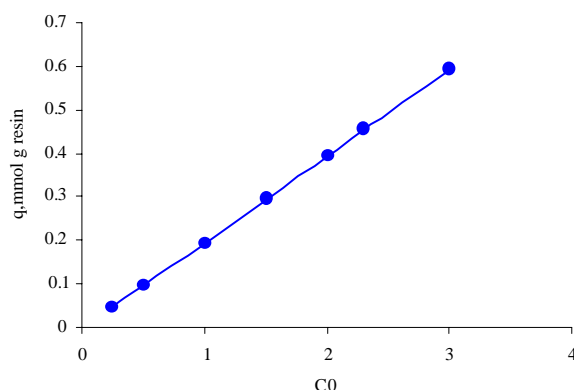


Fig. 4: Sorption isotherm of Cr (VI) on Lewatit FO36 as a function of initial metal concentration

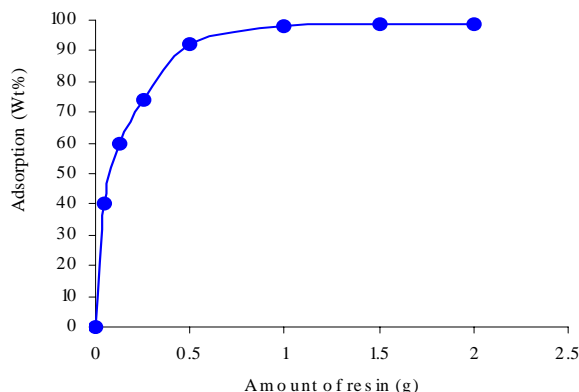


Fig. 5: Effect of amount of resin on the ion exchange of Cr (VI) using the Lewatit FO36 at R=1000, typically

range of 5.0–6.0 for resin and the basic process is essentially competitive ionic attraction for the ionic site. In all subsequent experiments, the equilibrium time was maintained at 90 min, which was considered as sufficient for the removals of Cr (VI) ion by resin (Cortina *et al.*, 1996; Namasivayam and Senthilkumar, 1999; Namasivayam and Yamuna, 1999; Bayat, 2002; Dakiky *et al.*, 2002; Yu *et al.*, 2003). 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. The maximum recoveries of Cr (VI) (about 99 % at pH = 7) were found as shown in (Fig. 3). The percentage uptake is highly dependent upon the initial concentration of the Cr (VI) ion and

the resin. The initial Cr (VI) 0.25, 0.5, 1.0, 1.5, 2.0, 2.3 and 3.0 mg/L at an amount of adsorbent of 0.5 g (Fig. 4). The data indicates that the initial metal concentration determines the equilibrium concentration and also determines the uptake rate of metal ion and the kinetic character of the process. Obtained data show that the rate of removal of Cr (VI) by Lewatit FO36 is constant with increasing of resin amount (Fig. 5). In the case of low concentrations, the ratio of the initial number of moles of Cr (VI) ion to the available surface area is larger and subsequently, it should be the fractional ion exchange becomes independent of initial concentrations, but it can be observed that at whole of initial concentration range of metal ion q varied with a constant slope.

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 thus, is critical in optimizing the use of the resins. Cr (VI) sorption isotherms of Lewatit FO36 resin is presented in Fig. 6 as a function of the equilibrium concentration of metal ion in the aqueous medium at room temperature (25 °C) for 120 min of contact time. The amount of Cr (VI) ions adsorbed per unit mass of the resin increased with the initial metal concentration as expected. To obtain maximum sorption capacities or reach the plateau values that represent saturation of the active groups which are available for interaction with Cr (VI) ions on the resin, the initial concentration was increased from 0.25 to 3.0 mmol/L of Cr (VI). The resin was saturated at relatively low concentrations indicating strong binding for Cr (VI).

Fig. 5 presents the removal of Cr (VI) as a function of resin amount using Lewatit FO36 in a solution at pH 7.0. The resin amount varied from 0.0 to 2.0 g. It is clear that the maximum removal of 5 mg/L of Cr (VI) ion in 1000 mL require a minimum resin amount of 100 mg.

It is apparent that, the amount of adsorbed metal ion per unit mass increases by increasing the resin amount and the sorption density. It was found that the retention of Cr (VI) increased with increasing amount of the Lewatit FO36 up to 0.50 g. This value was taken as the optimum amount for other trials. Since the fraction of metal removed from the aqueous phase increases as the sorbent dosage is increased in the batch vessel with a fixed initial metal concentration, the curve in Fig. 5 approach asymptotic values from 0.5 to 2.0 g resin. An increase in the sorbent amount resulted in a decrease in the contact time required to reach the equilibrium. It may be concluded that by increasing the adsorbent dose the removal efficiency increases but ion-exchange density decreases. The decrease in ion-exchange density can be attributed to the fact that some of the ion exchange

remains unsaturated during the sorption process; whereas the number of available ion-exchange sites increases by an increase in sorbent and this results in an increase in removal efficiency (Lacour *et al.*, 2001). As expected, the equilibrium concentration decreases with increasing adsorbent doses for a given initial Cr (VI) concentration due to increase of surface area or ion-exchange sites for a fixed initial solute concentration (Cortina *et al.*, 1996; Namasivayam and Yamuna, 1999; Pradhan *et al.*, 1999; Yu *et al.*, 2003).

Sorption process modeling is a topic of interest for the prediction of metal partitioning between the aqueous solution and the solid surface. In particular, the non-competitive Langmuir and Freundlich isotherms have been useful tools for the description and comparison of heavy metal sorption by different sorbents (Chong and Volesky, 1996; Yu and Kaewsarn, 1999; Mahvi *et al.*, 2007; Asgari *et al.*, 2008; Mahvi, 2008). These isotherms relate metal uptake per unit weight of adsorbent, q , to the equilibrium adsorbate concentration in the bulk fluid phase, C_e . The Langmuir isotherm is given as:

Table 2: Langmuir isotherm parameters (Eq. 3) on Lewatit FO36 resin with Cr (VI)

Ratio	A_s	b	R^2
250	0.552883	-6.28239	0.86
500	0.400769	-10.3107	0.89
1000	0.232186	-16.4889	0.80
2000	0.18499	-36.1827	0.91
3000	0.155894	-72.0742	0.95
4000	0.128319	-85.0775	0.97

Table 3: Rate constants for the removal of Cr (VI) with Lewatit FO36

Ratio	Overall rate constant, $K=k_1+k_2/\text{min}$	Forward rate constant, k_1/min	Backward rate constant, k_2/min
250	0.0202	0.013736	0.006464
500	0.0199	0.016716	0.003184
1000	0.0268	0.026532	0.000268
2000	0.0468	0.046332	0.000468
3000	0.0778	0.077022	0.000778
4000	0.0752	0.074448	0.000752

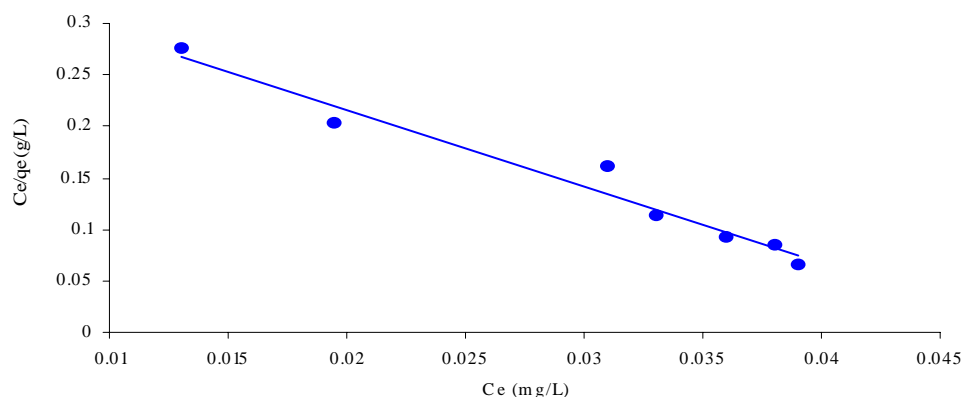


Fig. 6: Typical Langmuir plot for adsorption of Cr (VI) onto Lewatit FO36

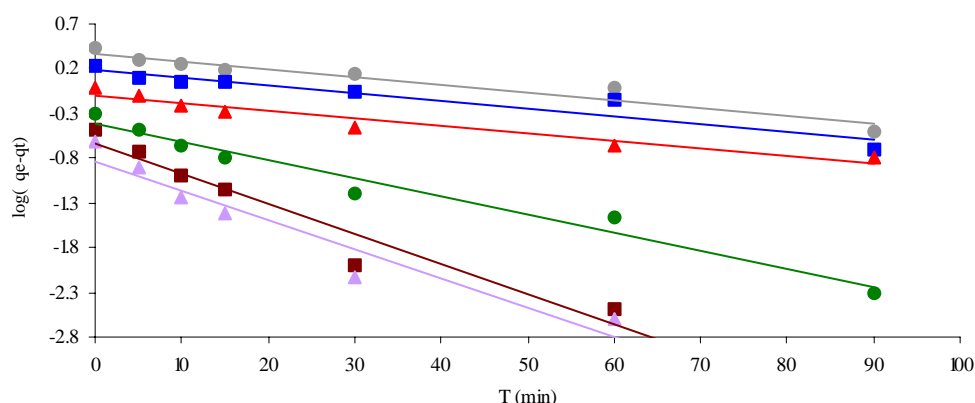


Fig. 7: Pseudo-first-order data for the adsorption of Cr (VI) on Lewatit FO36 at natural pH and 25 °C

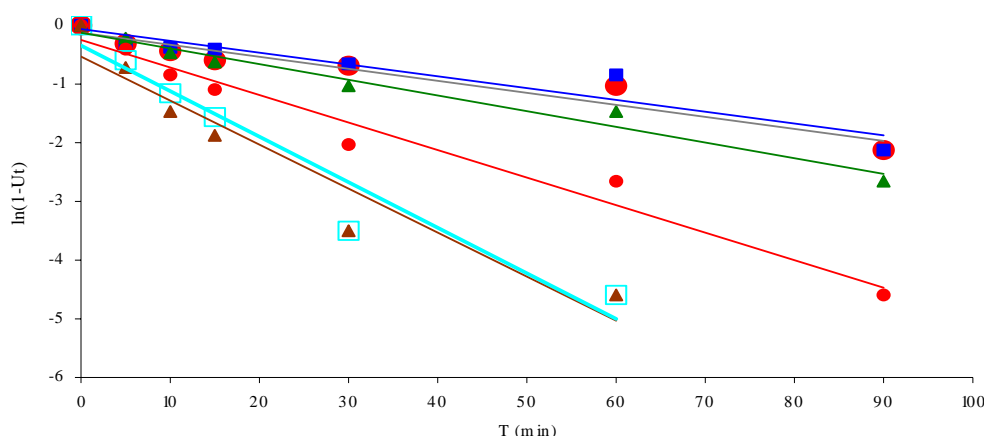


Fig. 8: Kinetic fits for the ion exchange of Cr (VI) on Lewatit FO36

$$\frac{C_e}{q} = \frac{1}{(A_s b)} + \frac{C_e}{A_s} \quad (3)$$

Where, A_s and b are Langmuir constants related to ion-exchange capacity and energy of ion exchanges, respectively. 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 ion exchange onto the surface and no transmigration of the adsorbate in the plane of the surface (Yu *et al.*, 2001). The data for the uptake of Cr (VI) ion by the resin were processed in accordance with a linear form of the Langmuir isotherm equation. The linear model, which describes the accumulation of solute by sorbent was directly proportional to the solution

concentration. The Langmuir isotherm applies to ion exchange on a completely homogenous surface with negligible interaction between adsorbed molecules.

The Langmuir treatment is based on the assumption that maximum ion exchange depends on the saturation level of a monolayer of adsorbate molecules on the adsorbent surface, that the energy of ion exchange is constant and that there is no transmigration of adsorbate molecules in the surface plane.

The Langmuir isotherm parameter was determined by least squares fit of the sorption data in Fig. 6. A_s and b were determined from the slope and intercept of the Langmuir plot (Fig. 6) and than were shown in Table 2. The Langmuir isotherm equation provided an excellent fit to the equilibrium sorption data. The results obtained showed that resin has the largest

capacity and affinity for the removal of Cr (VI) ions from solutions under the studied conditions.

The kinetics of Cr (VI) ion exchange on Lewatit FO36 followed the first-order rate expression given by Lagergren (Fig. 7):

$$\log(q_e - q) = \log q_e - k_{ad} \left(\frac{t}{2.303} \right) \quad (4)$$

Where, q and q_e are the amounts of Cr (VI) adsorbed (mg/g) at time t (min) and at equilibrium time, respectively and k_{ad} is the rate constant of ion exchange (Pradhan *et al.*, 1999). Various mechanisms and steps in ion-exchange phenomena can control the kinetics.

In the present study, the kinetics of Cr (VI) removal has been carried out to understand the behavior of Lewatit FO36 adsorbent. Using Eq06 and the equations given in Gode and Pehlivan (2003) and plotting $\ln(1-U_t)$ versus t (Fig. 8), the equilibrium constant K , forward and backward rate constants k_f and k_b were calculated and shown in Table 3. The forward rate constants for the removal of Cr (VI) were much higher than the backward rate constants namely the desorption process (Table 3). This suggests that the rate of ion exchange is clearly dominant. The rate of ion exchange is clearly dominant. Studies show that in some part of Iran, concentration of heavy metals, nitrate and fluoride are high in groundwater and therefore this process is suitable and could be recommended for treatment of such polluted waters (Mahvi *et al.*, 2005; Nouri *et al.*, 2006).

CONCLUSION

Following conclusions can be drawn from the present study of adsorption of Cr (VI) on Lewatit FO36:

- The experimental results indicated that Lewatit FO36 can be successfully used for the adsorption of Cr (VI) from aqueous solutions using batch method.
- The kinetic data would be useful for the fabrication and designing of wastewater treatment plants. Ion-exchange of the Cr (VI) is dependent on the initial concentrations of the adsorbent, adsorbate, time of contact and pH level. Experimental parameters must be optimally selected to obtain the highest possible removal of Cr (VI) from aqueous solutions using Lewatit FO36.

- The amount of Cr (VI) ions adsorbed per unit mass of Lewatit FO36 increased with the initial chromium concentration as expected. To reach the plateau values that represent saturation of the active groups which are available for interaction with Cr (VI) ions on the adsorbents. Adsorbent was saturated at relatively low concentrations indicating strong binding for Cr (VI). The equilibrium data well followed the linear Langmuir and Freundlich models.

- Adsorption of Cr (VI) ion is pH dependent. Results obtained from the effect of pH on the adsorption capacity of the Lewatit FO36 indicated that the maximum ion exchange was obtained at pH 6 for resin.

- The total ion exchange capacities for Cr (VI) ions are in the range of 0.12–0.55 mmol/g for Lewatit FO36.

- Sorption kinetics of Cr (VI) ion exchange by these ion-exchange resin followed Lagergren pseudo first-order reversible kinetics.

- Also, isothermal data of Cr (VI) ion exchange on Lewatit FO36 can be modeled with the Langmuir isotherm. The capacity of the resins for ion exchange of Cr (VI) can be calculated with this model.

- A batch contactor requires about 90–150 min contact time to achieve 80–96 % removal from aqueous solution.

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