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Removal of cobalt ions from water by ion-exchange method

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ABSTRACT

In the present work, the removal of Co(II) ions from aqueous solutions using an ion-exchange resin (Lewatit MonoPlus SP 112) was investigated. For this purpose, batch adsorption studies were carried out with various parameters such as pH and contact time. The Langmuir and Freundlich isotherm models were applied to analyze the experimental data. The best interpretation for the experimental data was given by the Langmuir isotherm and the maximum adsorption capacity was found to be 21.93 mg/g. The kinetic data were tested using pseudo-first-order, pseudo-second-order, liquid film diffusion, and intraparticle diffusion kinetic models. In addition, diffusion models were applied to explore the rate-determining step in cobalt diffusion behavior. The numerical values of the different rate constants, correlation coefficients, and effective diffusion coefficients as well as activation energy were determined. Data clarified that the adsorption process followed pseudo-second order reaction-base kinetic model. The magnitudes of the effective particle diffusion coefficient indicated that cobalt adsorption on resin was not controlled by film diffusion and pore diffusion and the chemical sorption was the rate-limiting step. The activation energy E_a confirmed that the nature of the adsorption was chemical sorption. The desorption studies were carried out using various reagents. The maximum percent desorption of the adsorbed metal ions were obtained, when 2 M HCl and 2 M H_2SO_4 was used as the reagent.

Keywords: Resin; Cobalt; Ion-exchange; Diffusion coefficient; Activation energy

1. Introduction

Heavy metals are important sources of environmental pollution, even in low concentration, since they are non-degradable and thus persistent [1]. Water pollution by heavy metals remains an important environmental issue associated negatively with health and economy [2].

Cobalt is a valuable metal that has a great number of applications in different industrial and medicinal fields. Removal of cobalt ions from wastewater is extremely important not only for their economic value, but also for reducing their contamination to the water environment [3]. A number of technologies have been developed over the years to remove toxic cobalt from water, including chemical precipitation, membran separation, electroflotation, ion exchange, reverse osmosis, and adsorption on activated carbon [4].

Chemical precipitation has been conventionally used for metal ion removal, but this technique has difficulty in meeting the latest environmental standards and it produces large amounts of sludge [5]. Other methods have also been developed for the removal of

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metal ions. However, their applications have been limited due to the high energy consumption [3]. Ion exchange now is a well-established technology, particularly in water purification and removal of hazardous substances at very low concentrations in chemical process industries [6]. Recently, ion exchange has been remarkably extended by the invention and application of new organic and inorganic ion exchangers [2]. Many studies on the removal of metal ions by ion-exchange resins have been conducted. It has been reported that ion exchange is able to overcome some of the problems encountered in other techniques [7]. One of these materials, macroporous strong acid resin (Lewatit MonoPlus SP 112) is an excellent adsorbent due to its strong mechanical stability and high chemical resistibility and it contains the functional group of (–SO_3Na). Thus, it was expected that the Lewatit MonoPlus SP 112 resin had a very high adsorption capability for metal ions. Furthermore, the Lewatit MonoPlus SP 112 resin is also inexpensive and easy to regenerate. In this study, various affecting factors on the sorption process such as initial pH of the solution and the contact time were investigated. The results were analyzed on the basis of pseudo-first order and pseudo-second order kinetics using the intraparticle and liquid film mass transfer diffusion model. Moreover, in the equilibrium studies, the equilibrium isotherms were determined and the desorption of Co (II) from resin was investigated.

2. Experimental

2.1. Chemicals and instrumentations

Lewatit Monoplus SP 112 strong acid resin was provided by the Sigma Aldrich and its properties are shown in Table 1. Standard solution of Co(II) was prepared by dissolving $Co(NO_3)_2 \cdot 6H_2O$ in distilled water. The Co(II) ion concentration was determined using a flame atomic absorption spectrophotometer (Perkin Elmer model AA 400). Hanna Model pH meter was used for measuring the pH. The sample was shaken in the Memmert model constant temperature shaking water bath.

Table 1
Characteristics of strong acidic Lewatit Monoplus SP 112 cation exchange resin

Characteristics	Value
Ionic forms as shipped	Na^+
Functional group	Sulfonic acid
Matrix	Cross-linked-polystyrene
Structure	Macroporous
Particle density	1.18 g/mL
Water content	45–55%
Bead size distribution range	0.4–1.25 mm
Total exchange capacity	Minimum 1.7 eq/L
Temperature limitations	(°C) 1–120
Effective pH range	0–14

2.2. Equilibrium and kinetic studies

Batch adsorption equilibrium experiments were conducted by placing a desired amount of the treated resin in a 50 mL centrifuge tubes containing solutions with various concentrations of metal ions. The centrifuge tubes were shaken at 150 rpm in a Memmert shaking water bath at the ambient temperature to reach the equilibrium (240 min). For kinetics studies, aliquot samples were taken at appropriate time intervals until reaching the adsorption equilibrium at different temperatures. The residual concentration of the metal ions in the aqueous phases was determined by a flame atomic absorption spectrophotometer. Sorbed Co(II) concentration by the resin was calculated from the difference between initial Co(II) concentration in solution and Co(II) concentration remained in the supernatant after sorption.

2.3. Desorption studies

Batch desorption experiments were carried out using various concentrations of HCl, NaOH, HNO_3, and H_2SO_4 solutions. Desorption of metal ions was performed by mixing Co(II)-loaded resin and eluent solutions of different concentrations in a centrifuge tubes, stirring under 150 rpm at 25 °C for 240 min. The final metal ion concentrations in the aqueous phase were similarly analyzed as described above.

3. Results and discussion

3.1. Effect of initial pH of solution

The pH of the solution plays an important role in the whole sorption process and particularly on the sorption capacity as the sorbent surface creates positive or negative charge on its surface depending on the solution pH. The charge surrounding the sorbent particles is proportional to the pH of the solution [8]. In order to study the effect of this parameter on the metal sorption by the resin, the initial pH was varied within the range of 2–8. The effect of pH on the removal of metal cation is shown in Fig. 1. The percentage of Co(II) sorption increased from 21.2 to 84.3 with an increase in solution pH from 2.0 to 4.0, attains a maximum value around 4.0 and does not change

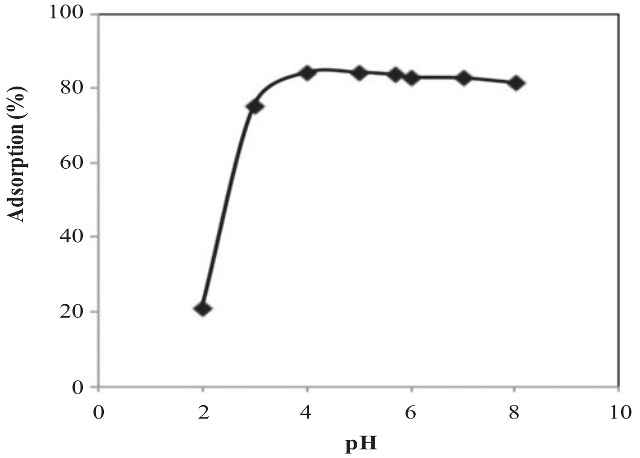

Fig. 1. Effect of pH on adsorption of Co(II). Conditions: initial metal ion concentration 100 mg/L, contact time 240 min, adsorbent dose 0.25 g.

considerably for higher pH values. Hence, in the subsequent studies, experiments were performed in the solution pH of 5.7. Similar trends for the values of pH were also observed using IRN77 and SKN1 [4] and methacrylic acid/acrylamide monomer [9] for the removal of Co(II) ions.

3.2. Effect of contact time and kinetic studies

The equipment design requires data on the amount of ions exchanged between resin and liquid in a given contact time. It is one of the important characteristics in defining the efficiency of an exchange process [7]. Fig. 2 shows the typical time profile during the exchange of Co(II) on resin. It was found that the concentrations of Co(II) in aqueous solutions decreased more rapidly in the early stage of operation (2–45 min), and the exchange was virtually completed within 240 min. That is, the decrease in the concentration of Co(II) in the solution demonstrated that higher exchange rates (dC_M/dt) of Co(II) with resin were obtained at the onset [10].

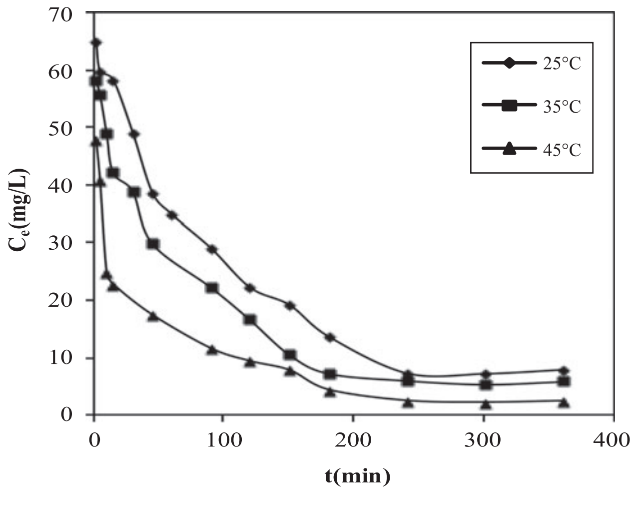

Fig. 2. Effect of contact time on adsorption of Co(II). Conditions: initial metal ion concentration 100 mg/L, adsorbent dose 0.5 g, pH 5.7.

The kinetics of the adsorption data was analyzed using different kinetic models such as pseudo-first order and pseudo-second-order models. Simple linear equation for pseudo-first order reaction kinetics is given below:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \tag{1}$$

where k_1 (1/min) is the rate constant of the pseudo-first-order adsorption, q_t is the amount of heavy metal adsorbed at time 't' (mg/g) and q_e is the amount of heavy metal adsorbed at equilibrium (mg/g). The plot of $\ln(q_e - q_t)$ versus t allows the calculation of the rate constant k_1 and q_e for cobalt removal (not showed) [8,11].

Pseudo-second-order reaction kinetics can be expressed as:

$$t/q_t = 1/k_2 q_e^2 + t/q_e \tag{2}$$

where k_2 (g/mg dk) is the pseudo-second-order rate constant. Similar to the pseudo-first-order reaction kinetics, q_e and k_2 can be determined from the slope and intercepts of the plot t/q_t versus t (not showed).

The intraparticle diffusion model proposed by Weber and Morris in 1963 was also taken into account in the experiments. They concluded that the sorption is proportional to the square root of the contact time:

$$q_t = k_{id} t^{0.5} \tag{3}$$

where k_{id} is the intraparticle diffusion rate constant (mg/(g min$^{0.5}$). When the intraparticle diffusion model controls the sorption, the graph of q_t against $t^{0.5}$ should be a straight line passing through the origin. The rate constant can be calculated from the slope of the line [12].

The liquid film diffusion equation is shown as:

$$-\ln(1 - F) = k_{fd} t \tag{4}$$

where F is the fractional attainment of equilibrium $F = (q_t/q_e)$, k_{fd} (1/min) is the rate constant. A linear plot of $-\ln(1 - F)$ vs. t with zero intercept would suggest that the adsorption process was controlled by liquid film diffusion [13,14].

The results of kinetic parameters for Co(II) are shown in Tables 2 and 3. As shown in Table 2, the

Table 2
Comparison of pseudo-first-order and pseudo-second-order rate constants and calculated and experimental q_e values

	Pseudo-first-order kinetics				Pseudo-second-order kinetics		
T(K)	q_e (exp) (mg/g)	k_1 (1/min)	q_e (cal) (mg/g)	R^2	k_2 (g/mg dk)	q_e (cal) (mg/g)	R^2
298	46.57	0.0115	28.89	0.9878	0.00097	48.78	0.9935
308	48.32	0.0145	25.48	0.9726	0.00172	50.00	0.9978
318	50.14	0.0128	16.10	0.9055	0.00128	51.02	0.9999

Table 3
Intraparticle diffusion rate constant and liquid film diffusion rate constant for the adsorption of Co(II)

	Intraparticle diffusion			Liquid film diffusion		
T (K)	k_{id} (mg/g min$^{0.5}$)	Intercepts	R^2	k_{fd} (1/min)	Intercepts	R^2
298	1.237	10.38	0.9780	0.0115	0.47	0.9880
308	1.109	12.93	0.9610	0.0180	0.52	0.9370
318	1.251	15.14	0.9670	0.0148	1.04	0.9450

correlation coefficient values of pseudo-second-order model (from 0.993 to 0.999) were higher than that of pseudo-first-order model and the calculated q_e values from pseudo second-order model were very close to experimental q_e values. Also, the rate constant of the pseudo second-order model (k_2) increased with the increasing temperature in the range of 25–45°C.

The plots were obtained to determine whether intraparticle diffusion and the liquid film diffusion models are the rate-limiting step (not showed). Although the plots were linear, they do not pass through the origin and this further indicated that the intraparticle diffusion and liquid film diffusion may not be the rate-controlling step. This suggests that the pseudo-second-order kinetic model is based on the assumption that the rate-limiting step may be chemical sorption and without mass transfer in solution. Chemical sorption mechanism for Co(II) can be expressed with the following reaction [11]:

$$\mathrm{Co}^{2+} + 2\mathrm{RNa}^{+} \longrightarrow \mathrm{R_2Co} + 2\mathrm{Na}^{+} \qquad (5)$$

In many cases, the pseudo-second-order kinetic model provided better results for the adsorption of Co (II) on waste containing boron impurity and lemon peel [15,16], for the adsorption of Ni (II) on Lewatit Monoplus SP 112 [17] and for the adsorption of Cu(II) ions on ion exchange resins 252H and 1500H [15].

3.4. Diffusion study

In ion-exchange process, the transfer of ions is controlled either by boundary layer diffusion (external mass transfer), intraparticle diffusion (mass transfer through the pores) or by the exchange reaction, or by combination. In ion-exchange systems, the following phenomena are involved, relevant for the process kinetics:

- diffusion of metal ions from the bulk of the solution to a liquid thin layer that surrounds each particle,
- diffusion of cobalt ions through this layer surrounding each resin particle to the resin surface (liquid-film diffusion),
- intraparticle diffusion of cobalt ions through resin channels (diffusion within the particle or pore diffusion),
- cationic exchange (rate of reaction) [7,14].

However, for a design of an efficient adsorption system, it is necessary to predict the diffusion coefficients using diffusion based models. If film diffusion was to be the rate-determining step in the adsorption of cobalt ions on the surface of resin, the value of film diffusion coefficient (D_f) should be in the range 10^{-6}–$10^{-8}\,\mathrm{cm^2/s}$. If the pore diffusion was to be rate x limiting, the pore diffusion coefficient (D_p) should be in the range 10^{-11}–$10^{-13}\,\mathrm{cm^2/s}$. Geometry of the adsorbent was assumed to be spherical and the overall rate constants of the process can be correlated with the pore diffusion coefficient and film diffusion coefficient independently [18,19].

Pore diffusion coefficient:

$$D_p = 0.03\frac{r_0^2}{t_{1/2}}$$

Film diffusion coefficient:

$$D_f = 0.23\frac{r_0 \partial C}{t_{1/2} C_0}$$

where r_0 represents the radius of the adsorbent which is 0.041 cm, ∂ is the thickness of the film (10^{-3} cm), $t_{1/2}$ is the time, for half adsorption of cobalt, C (mg/L) is the amount of cobalt adsorbed and C_0 is the initial cobalt concentration. Pore diffusion coefficients and film diffusion coefficients were estimated by taking all the adsorption data in consideration.

The values of D_p for resin in this study were obtained as 7.09, 9.45, and 9.45×10^{-9} cm^2/s and that for D_f were obtained as 1.22, 1.63, and 1.68×10^{-9} cm^2/s for the temperatures of 25, 35, and 45°C, respectively. These results agree with those of Dogan et al. [20].

It clearly reveals that cobalt adsorption on resin was not controlled by film diffusion and pore diffusion since coefficient values were in the range 10^{-9} cm^2/s. Moreover, the plots of intraparticle diffusion and liquid film diffusion confirmed the absence of a film diffusion effect and pore diffusion from Eqs. (3) to (4).

The magnitude of activation energy (E_a) can give an idea about whether the adsorption process is physical or chemical [13]. The activation energy of the adsorption process was calculated by Arrhenius equation.

$$\ln k = \ln A - E_a/RT \tag{6}$$

where E_a is the Arrhenius activation energy (kJ/mol), A is the Arrhenius factor, R is the ideal gas constant (8.314 J/mol K), k is the specific rate constant and T is the solution temperature (K). The k value of Arrhenius equation was obtained from the rate constant (k_2) of pseudo second order equation. To estimate the Arrhenius activation energy, $\ln k$ was calculated at three different temperatures of 25, 35 and 45 °C and $\ln k$ versus $1/T$ was plotted (Fig. 3). The plot shows a straight line with slope $-E_a/R$ and intercept of $\ln A$ [21]. The Arrhenius activation energy calculated from the slope was found to be 42.17 kJ/mol. In physical adsorption, the energy requirements are small, and the activation energy is usually not more than 4.2 kJ/mol. The value of the activation energy confirmed that the process of cobalt removal using Lewatit Monoplus SP 112 exchanger was not diffusion controlled, that is, the chemical adsorption was the rate-limiting step.

3.5. Equilibrium studies

Sorption equilibrium is usually described by an isotherm equation whose parameters express the surface properties and affinity of the sorbent, at a fixed temperature, and pH [22]. By plotting solid-phase concentration against liquid-phase concentration graphically, it is possible to depict the equilibrium adsorption isotherm [23]. The main purpose of the isotherm study is to identify the maximum capacity of each sorbent for different sorbates. The equations often used to describe the experimental isotherm data are those developed by Langmuir and Freundlich.

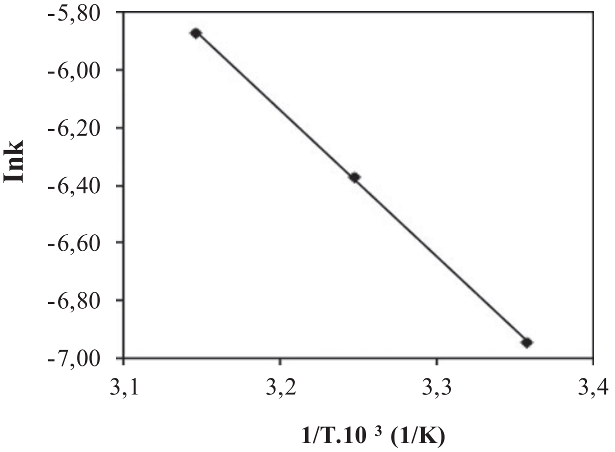

Fig. 3. Arrhenius equation plot for adsorption of Co(II) onto resin.

In 1918, Langmuir developed a theoretical equilibrium isotherm to calculate the amount of gas adsorbed onto a surface to the pressure of the gas. It is suitable for describing numerous homogeneous, constant energy solution sorption systems [5]. Langmuir model can be represented with the following equation:

$$q_e = \frac{q_m K C_e}{1 + K C_e} \tag{7}$$

where q_m is the maximum amount of sorbed material required to give a complete monolayer on the surface and K is a measurement of relative sorption affinity and is a constant related to the energy of sorption [21,24].

In 1906, Freundlich developed the earliest recognized sorption isotherm modelling equation. The empirical model can be applied to non-ideal sorption on heterogeneous surfaces as well as multilayer sorption [5]. The Freundlich equation is:

$$q_e = K_F C_e^{1/n} \tag{8}$$

In this expression, K_F and n are the Freundlich constants related to the adsorption capacity and adsorption intensity of the sorbent, respectively. If sorption is favorable, then $n > 1$.

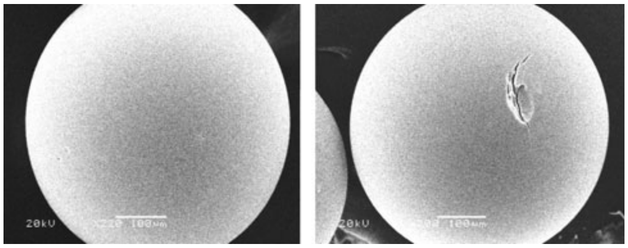

Fig. 4. SEM pictures of Lewatit Monoplus SP 112 resin.

The sorption model constants were estimated from the sorption data of Co(II) ions on resin by using a Curve Expert 1.3 biological software computer program. The Langmuir and Freundlich sorption isotherms were obtained at pH 5.7 and 0.5 g resin amount for Co(II) ions (not showed). The Langmuir and Freundlich constants and standard error of the estimate, coefficient of multiple determination (R^2) between the experimental values and the predicted values using the models are given in Table 4, and R^2 values are always greater than 0.980. However, the Langmuir model provided the best correlation for the sorption of Co(II) ions by the resin.

3.6. SEM analysis of the resin

SEM images of the cation-exchange resin before and after cobalt(II) adsorption are shown in Fig. 4. After the adsorption of as-prepared resin with cobalt (II) ion solution, its color has changed slightly. As could be seen from the figure, a visible change of the surface morphology in the cobalt(II) sorbed resin demonstrated that the sorption of cobalt(II) ions taken place onto the resin.

3.7. Desorption studies

Desorption study is useful in recycling of the adsorbent and recovery of metals. In the present study the desorption efficiency of the adsorbed metal (100 mg/L) onto resin was studied by various concentrations of 25 mL HCl, NaOH, HNO_3 and H_2SO_4 solutions. The systematic influences of these desorbing solutions on adsorbed metal onto resin are shown in Table 5. The results show that 2 M HCl and 2 M H_2SO_4 solutions can effectively desorb (100%) the Co (II) ions from adsorbent material. The 2 M HCl and 2 M H_2SO_4 solutions, respectively, indicated that the Co(II) adsorbed by the resin can easily be desorbed and thus can be used repeatedly in Co(II) adsorption.

Table 5
Desorption data of Co(II)

	Desorption (%)			
Concentration	NaOH	HCl	HNO_3	H_2SO_4
0.5 M	0.087	32.89	25.45	45.14
1 M	4.036	59.05	62.31	79.06
2 M	0.713	100.0	85.69	100.0

4. Conclusion

In this study, the removal of Co(II) ions from aqueous solutions onto a cation-exchange resin was investigated under different experimental conditions such as pH and contact time. The adsorption process was more rapid in the early stage of operation (2–45 min), and the exchange is virtually completed within 240 min. Batch equilibrium results suggested that the adsorption process followed pseudo-second-order reaction-based kinetic model and diffusion coefficient values indicated that cobalt adsorption on resin was not controlled by film diffusion and pore diffusion. The Arrhenius activation energy was found to be 42.17 kJ/ mol. The value of the activation energy confirmed that the nature of the adsorption process was chemical adsorption onto resin. The Langmuir and Freundlich adsorption models were used for the mathematical description of the sorption of Co(II) ions onto resin. The maximum cobalt(II) adsorption capacity of resin was 21.93 mg/g at 25°C according to the Langmuir isotherm. Reusability tests with 2 M HCl and 2 M H_2SO_4 showed that Lewatit MonoPlus SP 112 resin can stand acid desorption and recover binding capacity. The resin has a potential for the treatment of industrial effluents.

Table 4
Langmuir and Freundlich isotherm constants

Langmuir model				Freundlich model			
q_m (mg/g)	K (L/g)	R^2	Standard error of the estimate (σ)	K_F (L^n/mg^n)	n	R^2	Standard error of the estimate (σ)
21.93	0.0435	0.9895	0.8371	2.309	2.1	0.985	1.016

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