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Removal of lithium from water by aminomethylphosphonic acid-containing resin

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Abstract: This paper gives an overview of the ability of an aminomethylphosphonic acid-containing chelating resin for the removal of lithium from water. The studies were performed under various conditions, such as resin dose, initial Li^+ concentration, solution pH and solution temperature. The results showed that the sorption of Li^+ reached equilibrium within 15 min and the experimental data were well-fitted by the pseudo-second-order kinetic model. The Li^+ sorption was highly pH dependent, and the optimum pH for Li^+ removal was ≥ 3. Isotherm sorption data displayed good correlation with the Langmuir model, and the maximum monolayer sorption capacity of the resin found to be 13.65 mg g^{-1}. Thermodynamic studies suggested that Li^+ sorption onto the chelating resin was an exothermic and spontaneous process in nature. The resin could be regenerated by 0.1 M HCl, NaCl or H_2SO_4 with > 99 % efficiency. Desorption of Li^+ with 0.1 M NaCl resulted in no changes in the uptake capacity through four sequential sorption/desorption cycles.

Keywords: chelating resin; ion exchange; lithium; Lewatit TP 260.

INTRODUCTION

Lithium is the 25th most abundant element (at 20 mg kg^{-1}) in the earth's crust.[1] The major lithium minerals with commercial value are classified into three major groups, namely, silicates, micas, and phosphates.[2] Lithium finds application in rechargeable lithium ion batteries (LIBs) because of its very high energy density by weight and high electrochemical potential (3.045 V).[1] Besides batteries, lithium compounds are used in ceramics and special glass industries, in primary aluminum production, rocket propellants, nuclear and pharmaceutical industries, in the manufacture of lubricants and greases, synthesis of vitamin A, synthesis of organic compounds, silver solders, underwater buoyancy devices, and batteries. Lithium is alloyed with aluminum and magnesium as light metals to form stronger and lightweight alloys.[3] It is recovered from mines and salt

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lakes, which contain about 17 million t of lithium in total, while seawater is also considered as a vast source of lithium (about 2.5×10^{14} kg), although the concentration of lithium is very low, *i.e.*, 0.17 mg L^{-1}.[4] Various methods have been studied for the recovery of lithium from seawater, brine, and geothermal water. These can be classified into three groups: adsorption (ion-exchange), solvent extraction and co-precipitation.[5] The efficiency and limits of such methods have been reviewed elsewhere.[3] Problems in separation or in the concentration of trace constituents are sometimes encountered. Ion exchange resins with different cation-exchange groups may possess different selectivity coefficients. Among the factors that determine the ionic selectivity of resins, the most important ones are: a) the nature of the acidic functional group and b) the density of the structure, which is largely determined by the degree of cross linkage. Regarding the first point, it is characteristic that the affinity of alkali ions toward strongly acidic groups (sulfonic acid groups, *etc*.) decreases in the order K >> Na > Li, whereas the order is precisely the opposite on resins with carboxylic, phosphonous, and phosphonic acids (especially the latter) as the functional groups.[6,7]

The purpose of this study was to evaluate an aminomethylphosphonic acid-containing chelating resin for Li^+ sorption. The sorption process was optimized by varying different parameters, such as resin dose, initial solution pH, concentration of Li^+ and temperature. Elution of Li^+ from the resin was also examined.

EXPERIMENTAL

Materials

Lewatit TP 260 resin was used in the experiments. It is a weak acidic, macroporous resin contains chelating aminomethylphosphonic acid groups. The properties of resin are listed in Table I.[8] The resin was converted into the sodium form by treatment (100 mL wet resin) with 2 M NaCl (250 mL) solution for 24 h and then washing thoroughly with water. It was dried in an oven and used in the experiments.

TABLE I. Physicochemical properties of Lewatit TP 260

Matrix	Cross-linked polystyrene
Functional Group	Aminomethylphosphonic acid
Ionic Form	Na^+
Total capacity (H-form)	2.3 eq. L^{-1}
Water Retention	59–61 %
Stable in pH range	0–14

Reagents

Analytical grade reagents were used in the experimental studies. A stock solution of Li^+ was prepared by dissolving appropriate quantities of Li_2CO_3 (Merck) salt. The weighed amount of Li_2CO_3 was transferred into a beaker containing pure water and then HCl was added for complete dissolution. The solution was heated and swirled to expel dissolved CO_2. The prepared solution was cooled to room temperature then transferred to a volumetric flask and diluted to volume with water.

The pH of the solutions used in the batch test was adjusted to its optimum value by the addition of sufficient amounts of 0.1 M HCl and 0.1 M NaOH solutions.

Lithium analysis

The concentration of Li^+ was determined using a flame photometer (Jenway PFP7). The concentration ranges of Li^+ standards for the calibration curve were in the range of 0.1 to 5 mg L^{-1}. High concentration of Li^+ (which was used in the isotherm study) was measured after appropriate dilutions.

Batch adsorption tests

Experimental conditions for Li^+ sorption were summarized in Table II.

TABLE II. Experimental parameters for Li^+ removal

Investigated parameter	Li^+ concentration mg L^{-1}	Amount of resin used, g	Volume of solution, mL	pH of solution	Temperature °C
Resin dose	5.0	0.02–0.5	25	6	25
pH of solution	5.0	0.3	25	1–6	25
Isotherm study	from 25 to 300	0.3	25	6	25
Temperature effect	5.0	0.3	25	6	30,40, 50, 55 and 60
Kinetic study	5.0	12	1000	6	25

The removal efficiency (*R*) and capacity (*q*) of the resin were calculated according to Eqs. (1) and (2), respectively:

$$R = 100\frac{c_0 - c_e}{c_0} \tag{1}$$

$$q = V\frac{c_0 - c_e}{m} \tag{2}$$

where, c_0 and c_e (mg L^{-1}) are the initial and equilibrium Li^+ concentrations, respectively, V is the volume of solution (L) and m is the mass of the resin (g).

RESULTS AND DISCUSSIONS

Effect of resin dose

The effect of resin dosage on the uptake of Li^+ was studied to understand the efficacy of the resin for Li^+ removal. The uptake of Li^+ plotted as percent removal *vs*. resin dose is illustrate in Fig. 1, from which it could be seen that on increasing the resin dose from 0.02 to 0.3 g per 25 mL, the removal efficiency improved from 53 to 98 %. This could be attributed to an increase in the availability of more sorption sites as the dose of resin was increased.[9] The optimum resin dose was found to be 0.3 g for 25 mL of solution and this amount was used in further experiments.

Effect of solution pH

The solution pH is a one of the important factor in sorption studies, because it can influence the structure of resin (for weak acidic and weak base), the structure of target molecule (especially if it has weak acidic or weak basic character) and H^+ or OH^- act as competing ions in the ion exchange process. The effect of initial solution pH values (1–6) on Li^+ ion sorption onto TP 260 resin was investigated, and the results are shown in Fig. 2. Li^+ removal was unfavorable in acidic media (pH ≤ 1) but it increased with increasing pH value. There was only 4 % removal at pH 1 that increased to 99 % at pH ≥ 3. The pK_a values of aminomethylphosphonic acid (AMPA) are 2.35 and 5.9.[10] At pH 1, the functional group of the resin is in the molecular form, thus removal of Li^+ was not possible. On the other hand, when the pH of solution was increased, the functional group of the resin ionized and sorption of Li^+ was enabled.

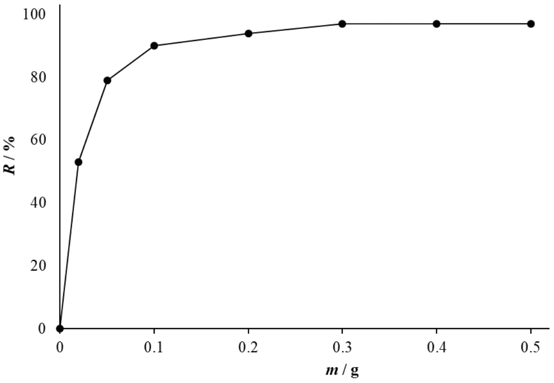

Fig. 1. Effect of resin dose on Li^+ removal.

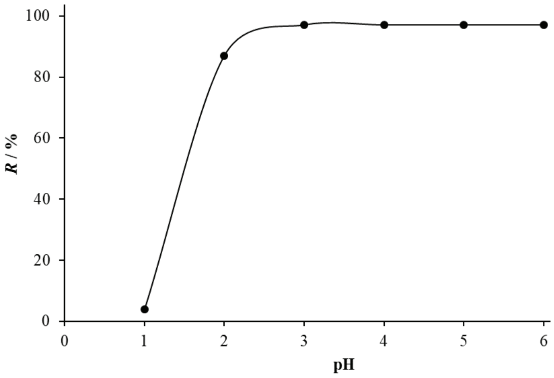

Fig. 2. Effect of initial solution pH on Li^+ removal.

Isotherm analysis of Li^+ removal

The sorption isotherm is one of the important parameters for understanding sorption behavior and mechanism. The sorption capacity of the resin for Li^+ removal was studied at different initial Li^+ concentrations and the obtained results were applied to the Langmuir and Freundlich models. The Langmuir isotherm model is used to describe a monolayer adsorption process, and the model can be described in linear form by Eq. (3):

$$c_e/q_e = 1/bq_0 + c_e/q_0 \tag{3}$$

In this equation, q_e / mg g^{-1} is the amount of Li^+ sorbed per gram of dry resin at equilibrium, q_0 / mg g^{-1}, and b / L mg^{-1} are the Langmuir constants related to the capacity and energy of sorption, respectively.[11,12] The Freundlich isotherm model shows that a multilayer of the adsorption process occurs on heterogeneous surfaces, and is expressed by Eq. (4):

$$\log q_e = \log K_F + (1/n)\log c_e \tag{4}$$

where K_F / dm^3 g^{-1} is the isotherm constant of the Freundlich model and n is the exponent of the Freundlich model. K_F and n are characteristics of the system and are indicators of the sorbent capacity (or affinity for the solute) and sorption intensity, respectively.[13]

The related parameters of the two models were calculated and are summarized in Table III. From the values of the linear correlation coefficients (R^2), the Langmuir model was found more suitable for describing the Li^+ sorption than the Freundlich model. The results suggest that monolayer sorption of Li^+ on such resin is the main mechanism. Additionally, the values of q_0 calculated from the Langmuir model was 13.65 mg g^{-1}.

TABLE III. Calculated isotherm constants of Langmuir and Freundlich models

Langmuir isotherm constants			Freundlich isotherm constants		
q_0 /mg g^{-1}	b / L g^{-1}	R^2	K_F / dm^3 g^{-1}	n	R^2
13.6553	0.0370	0.9914	2.4526	3.4710	0.9577

Dubinin–Radushkevich model

The Dubinin–Radushkevich (D–R) model is another model that is used for clarification of the mechanism of sorption (*i.e.*, physical or chemical). The linear D–R equation is given by Eq. (5):

$$\ln q_e = \ln X_m - \beta\varepsilon^2 \tag{5}$$

where β is a constant related to the sorption energy (mol^2 J^{-2}), X_m (mol g^{-1}) is the D–R monolayer capacity, ε (mol L^{-1}) is the Polanyi potential that is calculated as shown in Eq. (6):

$$\varepsilon = RT\ln (c_e+1/c_e) \tag{6}$$

The mean free energy, E (kJ mol^{-1}), of sorption can be estimated by using β values as expressed in the following equations:[14]

$$E = 1/(\beta^{1/2}) \tag{7}$$

For values of $E < 8$ kJ mol^{-1}, physical forces may have an effect on the sorption mechanism while E values between 8 and 16 kJ mol^{-1} depict sorption controlled by ion exchange and $E > 16$ kJ mol^{-1} signifies the process is chemisorption.[15]

In the present case, X_m was found to be 0.0027 mol g^{-1}, which is equal to 18.74 mg Li^+ per g-resin, β was found to be 0.0050 $mol^2\ J^{-2}$, and mean free energy, E, was 14.1 kJ mol^{-1}, indicating that removal mechanism was ion exchange. The ion-exchange reaction of the resin with Li^+ can be expressed as:[16]

$$R-CH_2PO(ONa)_2+2Li^+ \rightleftharpoons R-CH_2PO(OLi)_2+2Na^+ \tag{8}$$

Kinetics of Li^+ removal

Ion exchange time-dependent experiments were performed to evaluate the sorption kinetics. The kinetic data shown in Fig. 3 indicate that the sorption of Li^+ increased rapidly within 5 min, followed by a relatively slow process, and then the sorption equilibrium was achieved within 15 min. In addition, no remarkable changes were observed from 15 to 45 min.

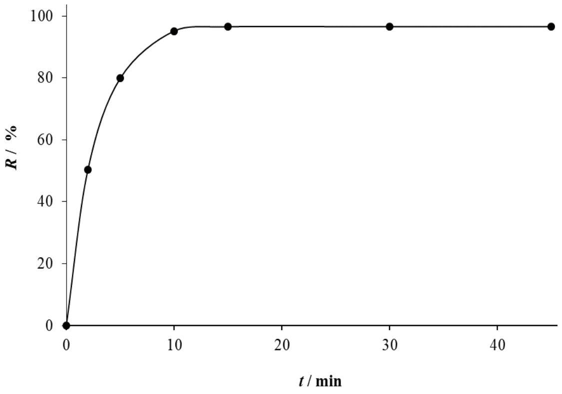

Fig. 3. Removal of Li^+ *versus* time.

Reaction-based model

The kinetic data were analyzed using pseudo-first-order, and pseudo-second-order models. The equations for these models are given as Eqs. (9) and (10):[17,18]

$$\log (q_e-q_t) = \log q_e - k_1t/2.303 \tag{9}$$

$$t/q_t = 1/k_2q_e^2 + t/q_e \tag{10}$$

Experimental results were applied to the kinetic models and the results are summarized in Table IV, from which it could be seen that the correlation coefficient (R^2) obtained from the pseudo-second-order model was larger than that from the pseudo-first-order model.

Diffusion-based model

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:[19]

$$q_t = k_{\mathrm{id}} t^{1/2} \tag{11}$$

where k_{id} is the intraparticle diffusion rate constant (mg g^{-1} $min^{-0.5}$). 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 could be calculated from the slope of the line, The liquid film diffusion equation is given as:

$$-\ln(1-F) = k_{\mathrm{fd}} t \tag{12}$$

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.[19]

The experimental data were fitted in Eqs. (9)–(12) and the obtained results are summarized in Table IV. The R^2 revealed that the retention process is the film diffusion controlled mechanism.

TABLE IV. The calculated parameters of pseudo first and pseudo second order kinetic model

Kinetic model	Parameter	Calculated value
Pseudo first order	k_1 / min^{-1}	0.4443
	q_e / mg g^{-1}	0.782
	R^2	0.2594
Pseudo second order	k_2 / g mg^{-1} min^{-1}	17.056
	q_e / mg g^{-1}	0.6085
	h / mg $g^{-1}min^{-1}$	0.6314
	R^2	0.9999
Intraparticle diffusion model	R^2	0.9756
	k_{id}	0.1998
Diffusion model	R^2	0.9890
	k_{fd}	0.4083

Thermodynamic parameters

The effect of the temperature on the removal of Li^+ from water was examined by changing the solution temperature from 30 to 60 °C under the optimized conditions of resin dose and solution pH. Changes in the free energy (ΔG), entropy (ΔS) and enthalpy (ΔH) were estimated by the usual procedure.[20] The calculated values are summarized in Table V.

TABLE V. Thermodynamic parameters for Li^+ sorption

ΔS / J mol^{-1} K^{-1}	ΔH / kJ mol^{-1}	ΔG / kJ mol^{-1} (T / K)
10	–8.16	–12.1 (303)
		–12.2 (313)
		–12.4 (323)
		–12.4 (328)
		–12.5 (333)

The negative values of ΔG shows that ion exchange reaction is spontaneous. The negative value of ΔH suggests the exothermic nature of the sorption. The positive value of ΔS suggests increased randomness at the solid/solution interface during the sorption of Li^+ onto the resin.

Regeneration of the resin

Regeneration experiments were performed as explained in the literature.[21] Recovery of Li^+ from the resin was checked with HCl, H_2SO_4 and NaCl solutions at various concentrations. The regeneration efficiency (*RE* / %) was calculated using Eq. (13):

$$RE\ (\%) = 100\frac{\text{Desorbed amount of Li}^+\text{from the resin}}{\text{Sorbed amount of Li}^+\text{ onto the resin}} \tag{13}$$

The obtained results are summarized in Table VI, from which it could be seen that 0.1 mol dm^{-3} HCl, H_2SO_4 or NaCl was enough for complete regeneration of the resin.

TABLE VI. Effect of acid concentration on the desorption of Li^+ from the resin

Regenerant	Concentration, mol dm^{-3}	Desorbed amount of Li^+, %
HCl	0.01	75
	0.05	88
	0.1	>99
	0.5	>99
H_2SO_4	0.01	85
	0.05	96
	0.1	>99
	0.5	>99
NaCl	0.01	75
	0.05	95
	0.1	>99
	0.5	>99

Multiple sorption/regeneration tests

Multiple sorption/regeneration tests were performed to determine the reusability of the ion exchange resin and the recovery of sorbed Li^+. The sorption test was realized by contacting 0.3 g resin with 25 mL of Li^+ solution (5 mg Li L^{-1}, pH 6) for 2 h. After decantation of the solution, the resin was washed with pure water until the conductivity of the eluting water reached that of pure water conductivity value. The elution step was performed by contacting 0.1 mol dm^{-3} 25 mL of regenerant solution with the washed resin for 2 h. At the end of this time, the solution was decanted and washed with pure water as explained above. This sorption/regeneration cycle was repeated 4 times and the obtained results are summarized in Table VII.

TABLE VII. Evaluation of sorption/regeneration cycle studies for Li^+ removal, %, from water by Lewatit TP 260 resin

Cycle number	HCl	NaCl
1	99	99
2	54	99
3	53	99
4	53	99

As can be seen from Table VII, the type of the regenerant influences the sorption capacity. When NaCl was used for regeneration, the sorption capacity of the resin did not change but when HCl was used, sorption capacity decreased. After regeneration with HCl, the resin was converted to the H-form and used in the next sorption cycle. In the second sorption process, Li^+ replaces H^+ and thus, the H^+ concentration in the solution increased and the pH of the solution decreased. As explained in effect of solution pH section, when the pH of solution decreased and the sorption capacity of resin decreased. Previously in the literature, various research studies were conducted for lithium removal/recovery. The results obtained in such research studies are summarized in Table VIII. The capacities of the sorbents vary from 4.07 to 62.5 mg g^{-1} sorbent. The removal performance of such materials strongly depended on the experimental conditions, particle size, and loaded metal.

TABLE VIII. Capacities of sorbents used for Li^+ removal/recovery

Resin/Sorbent	Capacity, mg g^{-1}	Reference
Manganese oxide adsorbent ($H_{1.6}Mn_{1.6}O_4$)	40	22
Magnetic lithium ion-imprinted Polymer	4.07	23
MnO_2 ion sieve	62.5	24
Nano-MnO_2	28.2	25
Ion-sieve manganese oxide, HMnO(Mg)	8.5	5
Spinel lithium manganese oxide	27.62	26
λ-MnO_2	31.55	27
Aminomethylphosphonic acid containing chelating resin	13.65	This study

CONCLUSIONS

In this study, the removal of lithium ions from aqueous solutions onto an ion-exchange resin was investigated under various experimental conditions, such as resin dose, initial solution pH, and temperature. The results clearly demonstrated that the phosphonic acid groups contributed to the sorption mechanism through electrostatic interactions between the phosphonic acid of the resin and the Li^+. The ion exchange process was quite fast and equilibrium was established within 15 min. The percentage removal of Li^+ was pH dependent, decreasing with decreasing initial pH of the solution. The equilibrium sorption behavior Li^+ onto Lewatit TP 260 resin followed the Langmuir adsorption isotherm with a maximum theoretical sorption capacity of 13.65 mg g^{-1} resin. The sorption of Li^+ onto the resin was found to be mainly based on ion-exchange interactions, and these were confirmed by the Dubinin–Radushkevich model.

Regeneration and reuse of resin work well for NaCl regenerant. Regeneration of resin with acid solution decreased the removal efficiency of resin.

Temperature variations were used to evaluate enthalpy, entropy and free energy changes. The negative value of free energy change showed the spontaneous nature of the adsorption. In the temperature range 303–333 K, enthalpy change was negative, and the ion exchange reaction was exothermic.

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ИЗВОД

УКЛАЊАЊЕ ЛИТИЈУМА ИЗ ВОДЕ СМОЛОМ КОЈА САДРЖИ АМИНОМЕТИЛФОСФОНСКУ КИСЕЛИНУ

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Ова студија даје увид у способност аминометилфосфонске киселине која садржи хелатну смолу да уклони литијум из воде. Испитивања су изведена под различитим условима као што су количина смоле, почетна концентрација Li^+, pH раствора и његова температура. Резултати су показали да сорпција литијума достиже равнотежу за 15 min, а експериментални подаци су добро корелисани кинетиком псеудо-другог реда. Сорпција Li^+ је показала велику зависност од pH, а оптимална pH за уклањање Li^+ била је изнад 3. Подаци за изотермну сорпцију показали су добру корелацију са Ленгмировим моделом, а максимални капацитет монослојне сорпције у смолу је био 13,65 mg/g. Термодинамичка испитивања су указала на егзотермност односно спонтаност процеса ове сорпције. Смола се може регенерисати из 0,1 M HCl, NaCl или H_2SO_4 са ефикасношћу преко 99 %. Десорпција Li^+ у 0,1 M NaCl није дала никакве промене у капацитету преузимања у четири узастопна сорпциона/десорпциона циклуса.

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