Desalination and Water Treatment
www.deswater.com

doi:10.5004/dwt.2018.21788

105 (2018) 181–188
February

Removal of boron from aqueous solution by ion exchange resin LSC–800: Batch and column studies

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Received 14 February 2017; Accepted 10 December 2017

ABSTRACT

In this work, both batch and column experiments were performed to investigate on boron removal from aqueous solution by LSC–800 ion exchange resin. The effects of contact time, initial pH, resin dosage, temperature, initial boron concentration and foreign ions on boron removal were studied, and the kinetics, equilibrium and thermodynamic data of the adsorption process were evaluated. The results showed that the contact time was 120 min to reach adsorption equilibrium, and the kinetic experimental data were best described by pseudo-second-order model. The boron removal depends on the resin dosage and solution pH, and the maximum boron removal was obtained at pH 9. The amount of boron removal increased as temperature and initial boron concentration increased. There is no essential effect of NaCl, $CaCl_2$ and $MgSO_4$ on boron removal. The Langmuir isotherm model agrees well with the equilibrium experimental data. The thermodynamic parameters indicated that the boron adsorption process was a spontaneous and endothermic. The resin can be reused frequently. The column capacity value of the resin was obtained by graphical integration as 31.04 mg/g. Both the Thomas and Yoon-Nelson models fitted well with the column studies' data.

Keywords: Boron; Ion exchange; Adsorption; Resin LSC-800; Isotherm; Kinetics

1. Introduction

Boron is not only an element that exists widely in water bodies, but also an essential element for life. However, it becomes toxic to humans, animals and plants when the amount of boron is slightly greater than required [1]. The World Health Organization (WHO) and the European Union have given a recommendation of below 2.4 mg/L and 1.0 mg/L, respectively, for boron in drinking water [2]. China recommends a guide level of 0.5 mg/L for boron in the drinking water. The safe boron concentration in irrigation water depends on the type of plant: 2–4 mg/L for tolerant plants, 1–2 mg/L for semi-tolerant plants, and 0.3 mg/L for sensitive plants [3]. Therefore, extra boron in water should be removed by an appropriate method.

There are several methods for boron removal from aqueous solution, including ion exchange, adsorption, coagulation sedimentation, solvent extraction and membrane filtration [1,4–7]. Especially, ion exchange is a very popular and promising method to remove boron from aqueous solution [8–10]. Ion exchange operation is basically discontinuous: a loading phase, called service run, is followed by regeneration of the exhausted ion-exchanger, and thus ion exchange process usually requires simple operational requirements. The technique can be applied in diluted water streams as well as seawater streams [10–12].

Recently, the use of ion exchange resins as selective adsorbent for boron removal has attracted much attention [8–13]. The resins have macroporous polystyrene matrix with active functional hydroxyl groups, and the functional group are often in the *cis* postion of the resins, which are named "*vis*-diols" [13,14]. Boron can react with *vis*-diols structure to form a variety of borate esters, then immobilize on the resins, so that the resins have a high selectivity to

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uptake of boron. N-methyl-D-glucamine (NMDG) is well known as an effective functional group, which can allow boron to form stable complex with the resin [10,15]. Some commercial resins such as Amberlite IRA 743, Diaion CRB 02, Diaion WA30, Dowex 2×8, Purolite S108, XSC–700, are modified by NMDG functional group [8–10,15–17].

In the present work, both batch and column adsorption experiments were carried out to investigate on boron removal from aqueous solution by a new ion exchange resin, LSC-800 resin. Firstly, the effects of various experimental parameters such as contact time, pH, resin dosage, initial boron concentration, foreign ions and temperature on boron removal were studied. And the adsorption kinetics, equilibrium and thermodynamic parameters were evaluated. Then the reusability of the resin was evaluated by adsorption-desorption tests. Finally, the Thomas and Yoon-Nelson models were applied to the data of column experiments.

2. Materials and methods

2.1. Materials

LSC-800 resin was provided from Xi'an LanXiao Technology Company, China. The typical physico chemical properties of the resin are listed in Table 1. Before used, the resin was first immersed in distilled water for 24 h, and then in 1 M NaOH solution for 2 h, and finally washed by distilled water several times. After the pretreatment, the resin was dried at 50°C for 24 h and cooled to room temperature. Boric acid (H_3BO_3) solutions were prepared by dissolving boric acid in distilled water, and the pH of boron solution was carefully adjusted using 0.1 M HCl and 0.1 M NaOH.

2.2. Batch experiments

A series of 100 mL polyethylene flasks were filled with 50 mL of boron-containing solutions, and a known dosage of the LSC–800 resin was added to each flask. Then, the flasks were shaken for scheduled time in the thermo-stated water bath shaker at the speed of 150 rpm. Finally, the 1-mL solution was taken out and centrifuged. The concentration of boron in the supernatant was analyzed with UV–5500 pc spectrophotometer by Carmine method [18].

The adsorption kinetics experiments were performed with the resin at different periods (5–180 min) under the following conditions: initial boron concentration of 5 mg /L; resin/solution ratio of 0.2 g/50 mL; solution pH of 9.0; reaction temperature of 30°C. The surface characteristics of the resin LSC–800 before and after the ion exchange experiment were obtained by scanning electron microscope (SEM) (S–4800, Hitachi, Japan).

Table 1
Typical physicochemical characteristics of LSC–800 resin

Particle size (mm)	0.315–1.25
Constitutional type	Microporous
Functional group	N-methyl-D-glucamine
Moisture content (%)	45
Density (g/mL)	0.68
Total exchange capacity (mg/ml resin)	5.2

The pH-dependent experiments were conducted by adjusting initial pH of the solutions to 6.0 to 12.0 at initial boron concentration of 5 mg/L, resin/solution ratio of 0.2 g/50 mL and reaction temperature of 30°C.

The effect of resin dosage on boron removal was investigated by applying different dosage of the resin (0.05–0.5 g) to 50 mL of the solution containing 5 mg/L boron at pH 9 and 30°C.

To examine the effect of initial boron concentration on boron removal, the experiments were performed at different initial boron concentrations (10, 20, 50, 100, 200 mg B/L). For each initial boron concentration the following conditions have been used: resin dosage, 0.2 g/50 mL; contact time, 2 h, pH 9 and temperature 30°C.

To investigate effect of foreign ions on boron adsorption, the concentrations of NaCl, $CaCl_2$ and $MgSO_4$ were adjusted to different values at initial boron concentration of 5 mg/L, resin/solution ratio of 0.2 g/50 mL, contact time of 2 h, pH value of 9 and reaction temperature of 30°C.

Batch equilibrium experiments were performed by varying the initial boron concentrations (10, 20, 50, 100, 200 mg B/L) and keeping the resin dosage fixed at 0.2 g. The containing-boron solutions were shaken at 20°C, 30°C and 40°C for 120 min (adsorption equilibrium time).

In order to evaluate the reusability of LSC-800 resin, adsorption-desorption cycles were repeated five times. In each cycle, 0.2 g of resin was added to 50 mL of 5 mg/L containing-boron solution and shaken at 150 rpm, 30°C for 120 min. After adsorption, the saturated resin particles with boron were rinsed in 0.5 M HCl solutions for 30 min, then washed with distilled water several times, regenerated with 0.5 M NaOH solution and repeatedly used for next adsorption experiment.

2.3. Column experiments

The column adsorption experiments were carried out in a glass column with height of 20 cm and internal diameter of 1 cm. The column was filled with 1.5 g resin. Boron solution (200 mg/L, pH 9) was delivered down-flow to the column at 2.5 mL/min flow rate that was controlled by a peristaltic pump (BT 100-2J model). The experimental set-up is shown in Fig. 1. From the outlet of the column, solution samples were collected at preset intervals of time (10 min) and analyzed for boron concentration.

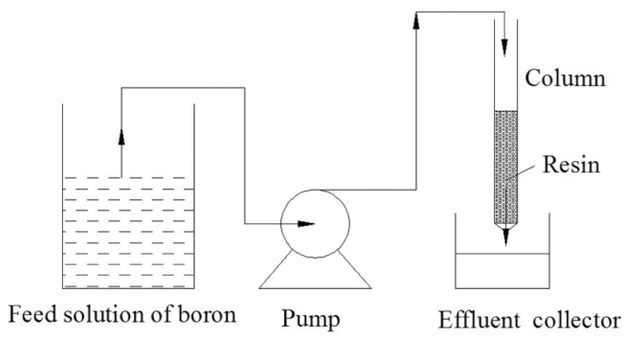

Fig. 1. Schematic diagram of experimental set-up for column adsorption.

3. Results and discussion

3.1. Effects of contact time and adsorption kinetics

Fig. 2 shows the effect of contact time on boron remval by LSC–800 resin. The boron adsorption was very fast initially, and adsorption equilibrium was achieved after 120 min.

In order to investigate the mechanisms involved in the adsorption process, the kinetic data of boron adsorption (Fig. 2) were evaluated with three typical kinetic models including the Lagergren pseudo-first-order equation (Eq. (1)) [19], pseudo-second-order equation (Eq. (2)) [20] and intra-particle diffusion kinetic equation (Eq. (3)) [21].

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303}t \quad (1)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (2)$$

$$q_t = k_p t^{0.5} \quad (3)$$

where q_t is the adsorption capacity (mg/g) at contact time t (min), q_e is the maximum adsorption capacity (mg/g) at equilibrium time t (min), k_1 is the pseudo-firs-order rate constant (min^{-1}), k_2 is the pseudo-second-order rate constant (g mg^{-1} min^{-1}), k_p is intra-particle diffusion rate constant (mg g^{-1} $min^{-1/2}$).

The plots of $log\ (q_e - q_t)$ vs. t, t/q_t vs. t and q_t vs. $t^{0.5}$ were used to obtain the values of kinetic parameters for boron adsorbed by LSC–800 resin, which are listed in Table 2. Based on the high correlation coefficient ($R^2 > 0.99$) and good agreement between the experimental value (q_e = 1.17 mg/g) and the calculated $q_{e,cal}$ value for the pseudo-second order model, it could be conclude that the pseudo-second order model is most suitable to describe the adsorption kinetic data, suggesting that the boron adsorption with the LSC–800 resin is chemisorption process [22], and is limited by the rate of boron complexation with N-methyl-D-glucamine (NMDG) functional groups of LSC–800 resin [10].

Fig. 3 indicates the SEM micrographs of LSC–800 resin before and after adsorption of boron onto it. The micrographs revealed that the LSC–800 resin was covered with a distinguishable substance, which was assumed to be boron adsorption onto the LSC–800 resin.

3.2. Effect of initial pH

The effect of the solution pH on capacity of boron removal is shown in Fig. 4. As can be seen from Fig. 4, the boron removal was apparently affected by solution pH, and the optimum solution pH for boron removal was 9. This

Table 2
Kinetic parameters for removal of boron by LSC–800 resin

Pseudo-first-order			Pseudo-second-order			Intraparticle diffusion	
$q_{e.cal}$	$k_1\times10^{-2}$	R^2	$q_{e.cal}$	$k_2\times10^{-2}$	R^2	$k_i\times10^{-2}$	R^2
1.393	4.51	0.985	1.341	3.47	0.997	8.31	0.870

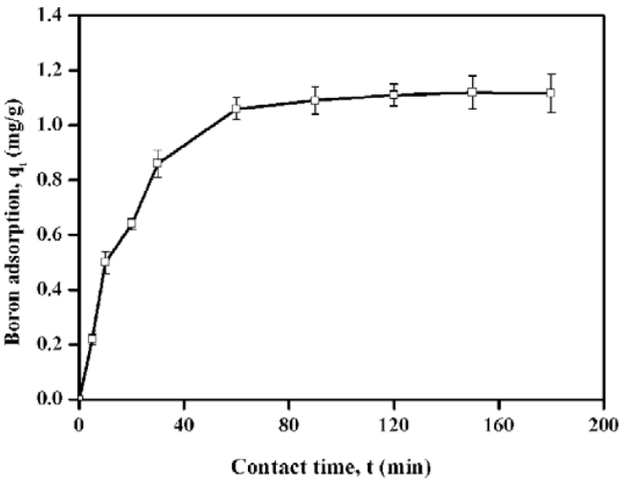

Fig. 2. Effect of contact time on boron removal by LSC–800 resin. Boron concentration = 5 mg/L, resin dosage = 4 g/L, pH = 9, T = 30°C.

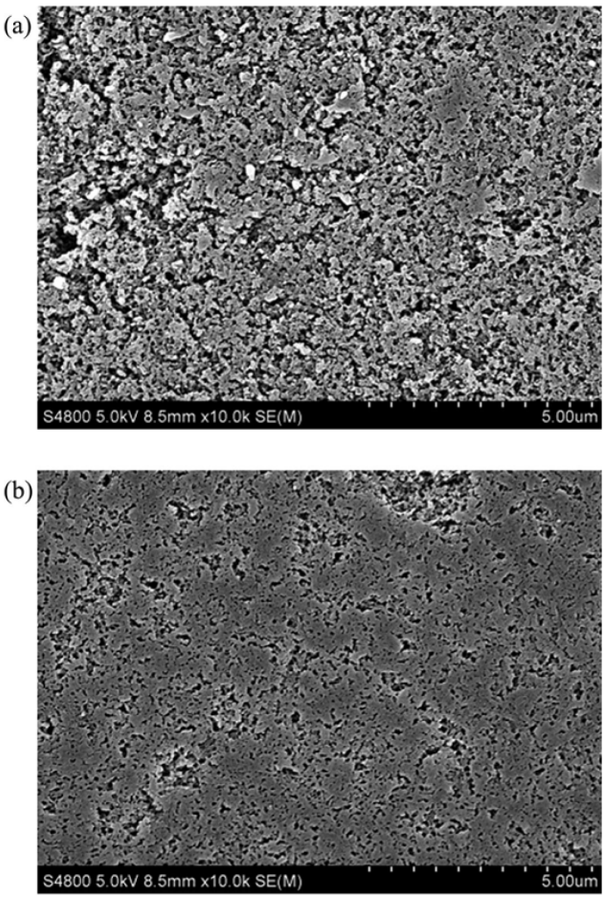

Fig. 3. SEM micrographs of LSC–800 resin (a) before and (b) after adsorption of boron.

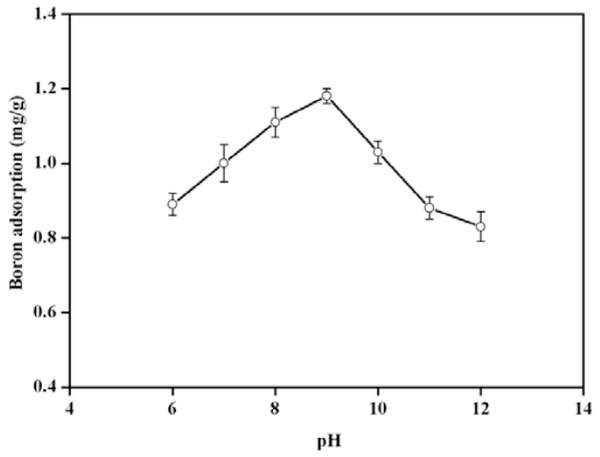

Fig. 4. Effect of initial pH on boron removal by LSC–800 resin. Boron concentration = 5 mg/L, resin dosage = 4 g/L, T = 30°C, contact time = 2 h.

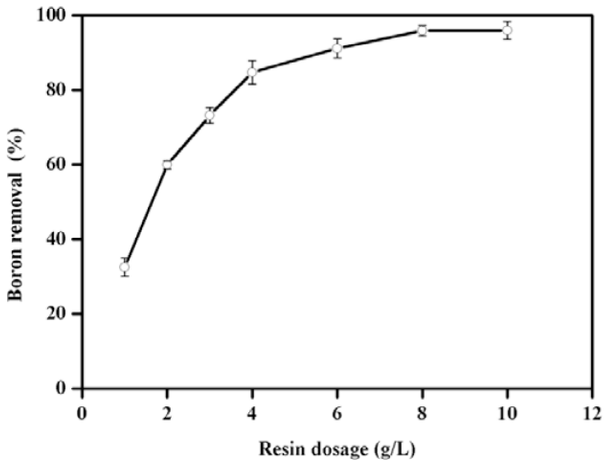

Fig. 5. Effect of resin dosage on boron removal by LSC-800 resin. Boron concentration = 5 mg/L, pH = 9, T = 30°C, contact time = 2 h..

may be explained as follows. Firstly, solution pH affects the dissociation process of $B(OH)_3$ in aqueous solution [23]. Several researchers have demonstrated that below pH 7, boron exists in the form of $B(OH)_3^0$, while at high pH (>8–9) $B(OH)_4^-$ is the primary anion [9,24]. Compared with $B(OH)_3^0$, $B(OH)_4^-$ is more easily attracted by the Resins' hydroxyl groups [10]. Secondly, the increase in solution pH was accompanied by an increase of OH^- in solution, which could result in the competition between $B(OH)_4^-$ and OH^- for adsorption sites [9]. As a result, at values of solution pH higher than 9, the boron adsorption capacity decreased.

3.3. Effect of resin dosage

The effect of the resin dosage on boron removal is showed in Fig. 5. With an increase in the resin dosage from 1 to 10 g/L, the boron removal efficiency increased from 32.53% to 96.02%, which is due to the availability of larger surface and more adsorption sites [9,10]. The optimum of boron removal was obtained for the resin dosage of 8 g/L. After this dosage, there was no obvious change in the boron removal. It may be attributed to the decrease in the boron concentration of the solution [25].

3.4. Effect of initial boron concentration

As shown in Fig. 6, the amount of boron adsorbed onto LSC–800 resin increased with increasing of initial boron concentration. This is due to increase in the driving force of the concentration gradient, as an increase in the initial boron concentration [9].

3.5. Effect of foreign ions

Natural waters can contain various ions such as sodium, calcium, magnesium,chlorine, sulphate, etc. In the present study, boron removal by LSC–800 resin was evaluated in the presence of three salts, i.e. NaCl, $CaCl_2$ and $MgSO_4$. As shown in Fig. 7, there is no essential effect of these foreign salt ions even at their high content in water on boron removal. This indicates the high specific boron adsorption by LSC–800 resin from multicomponent aqueous solutions. The similar results on high selectivity of the resin when used for boron removal from multicomponent aqueous solutions have been reported previously by Darwish et al. [10].

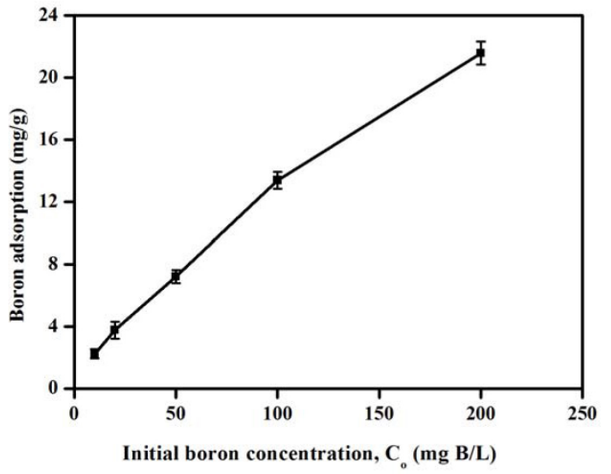

Fig. 6. Effect of initial boron concentration on boron removal by LSC–800 resin. Resin dosage = 4 g/L, pH = 9, T = 30°C, contact time = 2 h.

3.6. Adsorption isotherms and thermodynamic studies

The adsorption isotherms of boron onto LSC–800 resin at different temperatures are presented in Fig. 8. The equilibrium data were fitted using the Langmuir [26] and Freundlich [27] isotherm models. The two isotherm models can be presented as follows:

$$\frac{C_e}{q_e} = \frac{1}{bq_m} + \frac{C_e}{q_m} \quad (4)$$

$$q_e = K_f C_e^{\frac{1}{n}} \quad (5)$$

where C_e is the equilibrium boron concentration (mg/L), q_e is the amount of boron adsorbed at equilibrium (mg/g), q_m is the maximum adsorption capacity (mg/g), b is the Langmuir adsorption constant related to the free energy of adsorption (L/mg), K_f and n are the Freundlich equilibrium constants related to adsorption capacity and adsorption intensity, respectively.

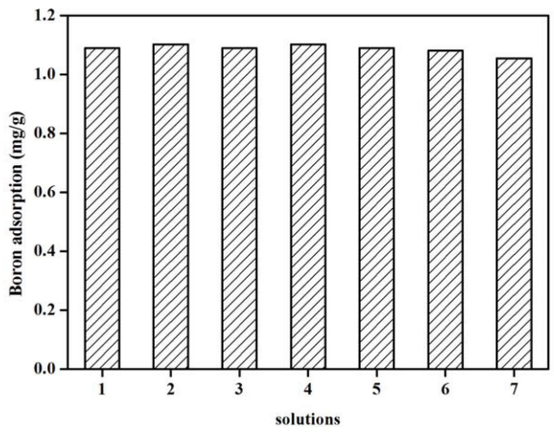

Fig. 7. Effect of foreign ions on boron removal by LSC–800 resin at pH 9 ((1) 5 mg B/L H_3BO_3; (2) 5 mg B/L H_3BO_3 + 0.01 mol/L NaCl; (3) 5 mg B/L H_3BO_3 + 0.1 mol/L NaCl; (4) 5 mg B/L H_3BO_3 + 0.01 mol/L $CaCl_2$; (5) 5 mg B/L H_3BO_3 + 0.1 mol/L $CaCl_2$; (6) 5 mg B/L H_3BO_3 + 0.01 mol/L $MgSO_4$; (7) 5 mg B/L H_3BO_3 + 0.1 mol/L $MgSO_4$).

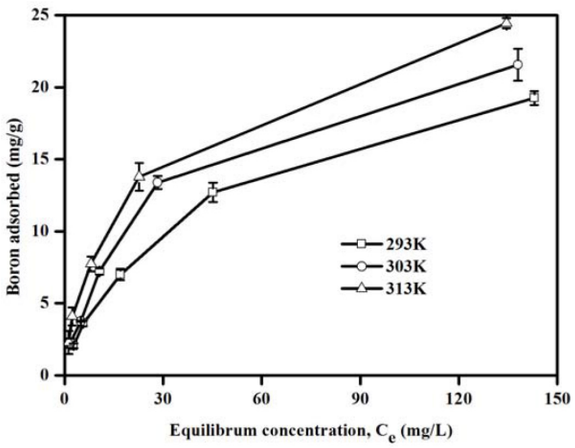

Fig. 8. Adsorption isotherms of boron onto LSC–800 resin at different temperatures. Boron concentration = 5–200 mg/L, resin dosage = 4 g/L, pH = 9, contact time = 2 h.

The parameters calculated from the two isotherm models are presented in Table 3. According to the values of correlation coefficients (R^2), the Langmuir isotherm model describes the boron onto the LSC–800 resin better than the Freundlich isotherm model, indicating that the adsorption tends to form a single mono layer instead of multi-layers. This result is similar to several reports from previous research studies [3,9,28].

Table 4 lists a comparison of the maximum adsorption capacity of boron on various ion exchange resins, which indicates that the adsorption capacity of LSC–800 resin is higher than those of other ion exchange resins. Therefore, the present LSC–800 resin could be considered a promising material for the removal of boron from aqueous solutions.

The value of Langmuir equilibrium constant b is dependent on the temperature. Therefore, this value can be used to estimate the thermodynamic parameters, such as the changes in free energy (ΔG), enthalpy (ΔH) and entropy (ΔS) for the adsorption of boron on the LSC–800 resin. The thermodynamic parameters can be calculated from the following equations [25,34]:

$$\Delta G = -RT \ln b \quad (6)$$

$$\ln b = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (7)$$

where R is the gas constant (R = 8.314 J/mol K), T is the absolute temperature (K), and b is the Langmuir constants (L/mol).

Table 4
Comparison of boron adsorption capacities for several ion exchange resins

Ion exchange resin	Capacity (mg B/g)	Reference
LSC–800 resin	25.64	This work
Dowex 2×8 resin	16.98	[9]
Amberlite IRA743	5.41	[10]
XSC-700 resin	19.81	[17]
D564 resin	16.42	[29]
Salicylic-HCHO polymeric resin	22.06	[30]
MCM-41 resin	8.64	[31]
GMA-co-TRIM resin	14.9	[32]
PS resin	4.54	[33]

Table 3
Langmuir and Freundlich isotherm parameters for boron adsorption onto LSC-800 resin at different temperatures

Temp.(K)	Langmuir isotherm				Freundlich isotherm		
	q_m (mg /g)	b (L/mg)	b (L/mol)	R^2	K_f	n	R^2
293	23.81	0.029	2.685	0.992	1.294	1.757	0.985
303	25.64	0.040	3.700	0.995	1.778	1.855	0.968
313	27.78	0.058	5.365	0.994	2.500	2.020	0.972

The values of ΔH and ΔS were determined from the slop and intercept of plot between ln *b* and 1/T (Fig. 9). The thermodynamic parameters for boron adsorption onto LSC–800 resin are listed in Table 5.

As seen in Table 5, the values of ΔG at 293 K, 303 K, 313 K for boron adsorption onto LSC–800 resin are all negative, suggesting the spontaneous nature of the adsorption process. The values of ΔG become more negative with increasing temperature, indicating the adsorption process becomes more favorable at higher temperatures. The increase in adsorption capacity of the resin at higher temperatures may be caused by the increasing mobility of adsorbate ions/molecules in the solution and the affinity to the adsorbent [24]. The positive value of the change of enthalpy (ΔH) confirms the endothermic nature of the adsorption process. The magnitude of ΔH also gives an idea about the type of adsorption. Generally, the value of ΔH for chemisorption is at the range of 20.9–418.4 kJ/mol [35]. The value of ΔH found in this study (Table 5) was 26.36 kJ/mol for boron adsorption, which implies that the chemisorption was the dominant mechanism. The result is consistent with the adsorption kinetics study in section 3.1. The positive value of ΔS (98.02 J/mol·K) suggests an increase in randomness at the solid/solution interface during the adsorption process.

3.7. Reusability of LSC–800 resin

Five cycles of adsorption/desorption of boron onto the LSC-800 resin were carried out. The obtained results (Fig. 10) indicate that the regenerated resin can be reused frequently without significant loss in its adsorption performance.

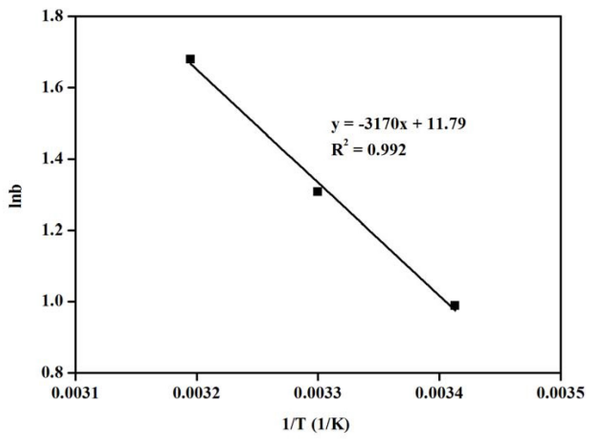

Fig. 9. Plot of ln *b* verus 1/T for estimation of thermodynamic parameters.

Table 5
Thermodynamic parameters for boron removal with LSC-800 resin

Temp. (K)	ln *b*	ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS (J/mol. K)
293	0.988	–2.41	26.36	98.02
303	1.308	–3.30		
313	1.680	–4.37		

3.8. Column studies

The performance of resin LSC–800 in the ion-exchange column can be assessed by plotting breakthrough curves. Boron adsorption capacity of the resin in the column is calculated from Eq. (8) [36]:

$$q_0 = \int_0^{V_t} \frac{(C_0 - C_t)dV}{m} \tag{8}$$

where q_0 (mg/g) is adsorption capacity of the column, m is the mass of the resin in the column, C_0 (mg/L) is inlet boron concentration, V (L) is volume of effluent, V_t (L) and C_t (mg/L) are effluent volume and effluent boron concentration at sampling time t, respectively.

The value $q_{0,exp}$ (mg/g) of LSC–800 resin was obtained by graphical integration as 31.04 mg/g (Table 6). The breakthrough curve of boron adsorption onto the resin is illustrated in Fig. 11. The column experimental data were analyzed by the Thomas model and the Yoon-Nelson model.

The Thomas model is a model that can be used to determine the maximum adsorption capacity of an adsorbent and the rate constant for an adsorption column [37]. The Thomas model is represented by the following form [12,36].

$$\frac{C_t}{C_0} = \frac{1}{1 + \exp\left[k_T \frac{(q_0 m - C_0 V)}{\theta}\right]} \tag{9}$$

where K_T is the Thomas rate constant (mL/min mg) and θ is the volumetric flow rate (L/min). The linear Thomas equation can be written as follow:

$$\ln\left(\frac{C_t}{C_0} - 1\right) = \frac{K_T q_0 m}{\theta} - \frac{K_T C_0}{\theta} V \tag{10}$$

The plot of $ln((C_0/C_t)–1)$ against sampling time t is illustrate in Fig. 12, which can be employed to determine the values

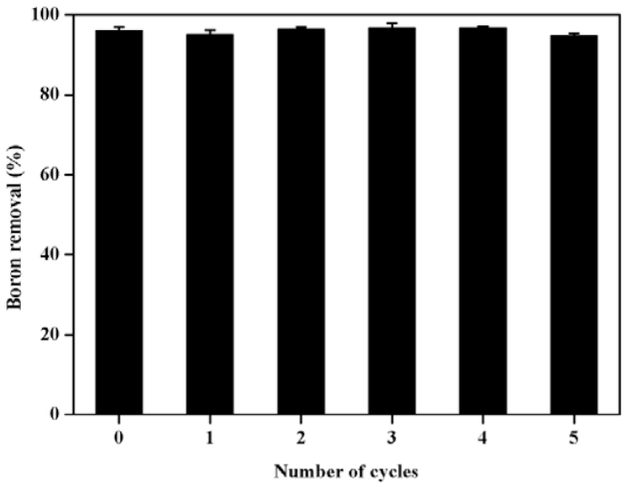

Fig. 10. Boron removal capacity of the regenerated resin. Feed boron concentration = 5 mg/L, resin dosage = 4 g/L, pH = 9, T = 30 °C.

Table 6
Thomas and the Yoon-Nelson model parameters

$q_{0,\exp}$ (mg/g)	Thomas model			Yoon-Nelson model			
	K_T [mL/(min mg)]	$q_{o,cal}$ [mg/g]	R^2	K_{YN} [1/min]	$q_{o,cal}$ [mg/g]	τ [min]	R^2
31.04	0.192	31.83	0.944	0.0407	32.17	96.52	0.944

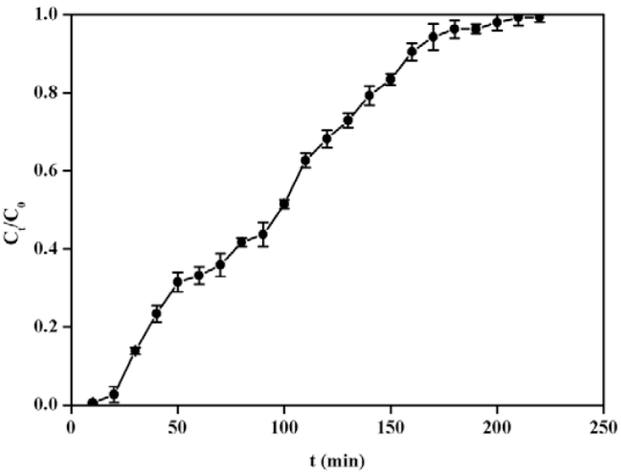

Fig. 11. Breakthrough curve of boron adsorption onto LSC–800 resin.

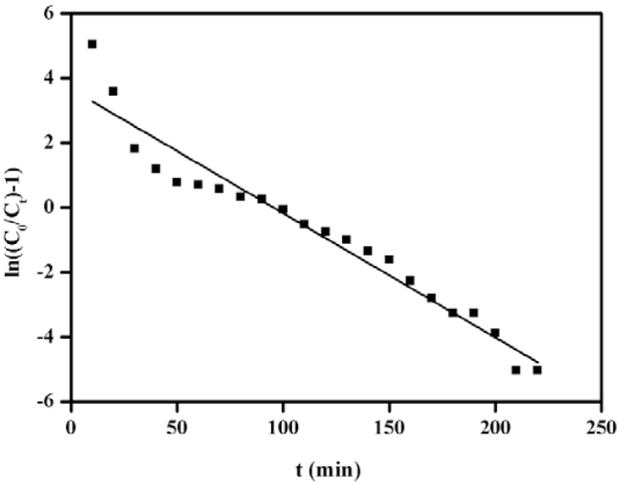

Fig. 12. Plot of $ln((C_0/C_t)–1)$ vs. t according to Thomas model.

of K_T and $q_{o,cal}$. The Thomas model parameters are given in Table 6.

The non linear Yoon–Nelson model can be described [24,38]:

$$\ln\left(\frac{C_t}{C_0} - 1\right) = \frac{K_T q_0 m}{\theta} - \frac{K_T C_0}{\theta} V \tag{11}$$

where K_{YN} is the Yoon-Nelson rate constant (L/min), τ is the time required for the column to reach 50% adsorbate breakthrough and t is the sampling time (min). The linear Yoon-Nelson equation is as follows:

$$t = \tau + \frac{1}{K_{YN}} \ln \frac{C_t}{C_0 - C_t} \tag{12}$$

A linear plot of $ln((C_t/C_0 – C_t)$ against time t was shown in Fig. 13, which was employed to determine the values of K_{YN} and τ. And adsorption capacity of the column, $q_{o,cal}$, was calculated according to the follow equation [36]:

$$q_0 = \frac{1}{2} C_0 \theta(2\tau) = C_0 \theta \tau \tag{13}$$

The Yoon-Nelson model parameters are also listed in Table 6. As seen from Table 6, the experiments adsorption of boron onto LSC–800 resin packed in column were well fitted with the Thomas and the Yoon-Nelson model (the correlation coefficient R^2 = 0.944). This result agrees with the previously studies concerning the removal of boron from aqueous solution by boron selective ion exchange resins Diaion CRB 02 and Dowex [12].

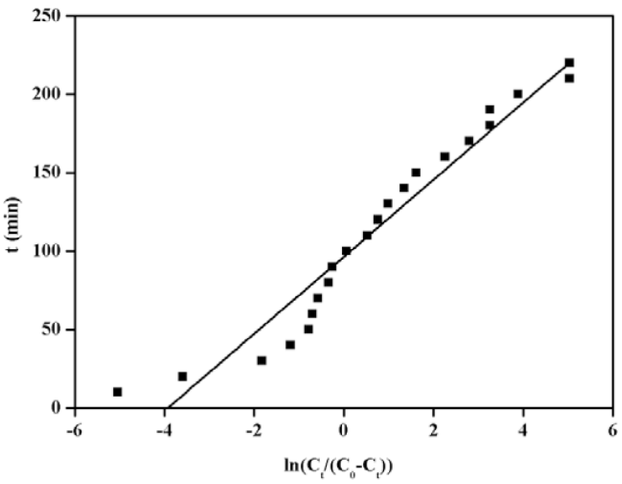

Fig. 13. Plot of $\ln(C_t/(C_0 – C_t))$ vs. t according to Yoon and Nelson model.

4. Conclusions

The present study shows that ion exchange resin LSC–800 is an effective adsorbent for the removal of boron from aqueous solution. Adsorption equilibrium was reached within 120 min, and the kinetic data were best described by pseudo-second-order model. The boron removal depends on the resin dosage and solution pH. The maximum adsorption was obtained at pH 9. The Langmuir isotherm model described the boron removal better than the Freun-

dlich isotherm model. Thermodynamic studies indicated that the boron adsorption process is mainly a chemical process of a spontaneous and endothermic nature. The boron removal increased with increasing temperature and initial boron concentration. There is no essential effect of NaCl, $CaCl_2$ and $MgSO_4$ on boron removal. LSC–800 resin can be reused frequently. The column capacity value of the resin was obtained by graphical integration as 31.04 mg/g. Both the Thomas and Yoon-Nelson models fitted well with the column studies' data. The capacity values were obtained as 31.83 mg/g and 32.17 mg/g using the Thomas and Yoon-Nelson models, respectively, for LSC-800 resin. The high capability of the present LSC–800 resin makes it potentially attractive material for the removal of boron from aqueous solutions.

Acknowledgments

This study financially was supported by the Key Laboratory of Urban Pollutant Conversion, Chinese Academy of Sciences (Grant No. KLUPC20160001), the Natural Science Foundation of Fujian Province （Grant No. 2016J01737 and the Marine high-tech industry development project of Fujian Province (Grant No. 2013013).The authors would like to thank to Xi'an Lan Xiao Technology Company.

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