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Full factorial design of experiments for boron removal from Colemanite mine wastewater using Purolite S 108 resin

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Boron pollution has a vital importance in Bigadiç boron mine in Turkey because the wastewaters of the mine are stored in a soil dam that threats the underground water quality. In this study the optimization of boron removal from the boron mine wastewater using Purolite S 108 resin was investigated by means of a 2^3 full factorial experimental design. Experiments were carried out in batch mode as a function of pH, temperature and resin-to-solution ratio. The low (1) and high (2) levels of the parameters for pH, temperature and resin-to-solution ratio were 2.5 and 10, 12 ºC and 40 ºC and 1 g/50mL and 2 g/50mL respectively. Boron adsorption capacity of the resin increased with low temperature, low resin-to-solution ratio and high pH. When the probability constants ($p<0.05$) at 95% confidence level were taken into consideration, only pH was found as statistically important parameter. The optimization of the parameters to obtain optimum conditions was done by interpretation of cube plots, Pareto chart and contour plots. A time span of 48 hours was enough to reach the equilibrium. Adsorption data were analyzed with the Langmuir and Freundlich isotherms. Data fitted to the Langmuir isotherm with a coefficient of determination value of 0.988. Maximum adsorption capacity was calculated as 12.87 mg g^{-1}. The fixed bed kinetics of boron adsorption onto resin could be explained by the Thomas and Yoon-Nelson models with a coefficient of determination value of 0.938. The fixed bed capacity of the resin was calculated as 12.71 mg g^{-1}.

Keywords: Boron Removal; Ion Exchange; Purolite S 108; Full Factorial Design; Isotherm; Fixed Bed

INTRODUCTION

The borate minerals identified in nature have 230 different crystal structures and it is thought that new borates may be found in nature [1]. At nature borates found in oxide forms together with the structural metal cations such as potassium, calcium, magnesium, aluminum, etc. [1,2]. Only several borates have commercially important deposit viz., colemanite (Ca2B6O11.5H2O), ulexide (NaCaB5O9.8H2O), pandermit (Ca4B10O19.7H2O), kernit (Na2B4O7.4H2O) and tinkal (Na2O.2B2O3.10H2O) [1,2]. Boron is widely used in a variety of applications including the nuclear, fuel, military, glass, electronic and computer, energy devices, photography, medicine, cosmetic, construction, communication, paper, rubber, plastic, chemistry, surface protecting material, machinery, metallurgy, explosive, automotive, ceramic, agriculture, textile, space and aviation industries [3]. Turkey has about 61% of the World boron reserves [4]. The known borate reserves in Turkey are located in four main districts, namely Emet, Bigadiç, Kırka and Mustafa Kemal Paşa [5]. One of the richest colemanite deposits of Turkey is located in Bigadiç region. After colemanite is mined in Bigadiç deposit, it is subjected to washing to remove attached clay minerals. Eventually, colemanite is dissolved with water and washing waster is polluted with boron. Therefore, washing waters are stored in a soil wastewater dam that causes a great concern due to contamination risk of underground water with boron. Boron containing wastewaters are not appropriate for irrigation because boron accumulates very fast in soils as it adsorbs onto clays [6]. Although boron is a required trace element for plants, animals and humans, there is a narrow concentration range between its detrimental and toxic effects [7]. Boron also forms complexes with heavy metals in the soil which are more toxic than boron and heavy metals [6]. Therefore, washing waters of Bigadiç colemanite mine should be refined from boron with a suitable method.

In the last two decades, several physico-chemical methods have been reported for removal of boron viz., adsorption[5], ion exchange [6], electrocoagulation [8], reverse osmosis [9], electrodialysis [10], solvent extraction after

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complexation [11] and chemical coagulation [12]. Although boron resins are expensive, ion exchange method is still one of the effective methods for boron removal from wastewaters especially if the boron should be recovered. In the literature, several boron selective or strong base resins were reported to remove boron from solution [6]. But the Purolite S 108 resin is lack of any reported study showing its exact capacity under different experimental conditions. The cheap, easy and short-winded way of adsorption capacity determination of adsorbents is to design of experiments by the full factorial, response surface or taguchi approaches. Of these approaches, the full factorial design of experiments requires the most few experiments [13]. Therefore, in this study, the experiments were designed by the full factorial approach using Minitab 16.0 programme.

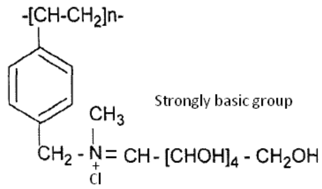
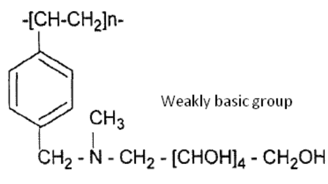
In this study, boron removal from Bigadiç mine wastewater by ion exchange method using Purolite S 108 resin was investigated by means of 23 full factorial experimental design. In the experiment the effects of pH, temperature and solid-to-solution ratio were optimized. The equilibrium data were applied the Langmuir and Freundlich models. The fixed bed kinetics of the resin were also investigated.

MATERIAL AND METHOD

Material

In this study, Purolite S 108 was used as boron resin. The characteristics of Purolite S 108 resin are given in Table 1.

Table 1: Typical chemical and physical characteristics of Purolite S 108

Property	**Description**
Polymer structure	Macroporous polystyrene cross-linked with divinylbenzene
Optical appearance	Spherical beads
Functional groups	Complex amino
Ionic form, as shipped	Cl
Total capacity (Cl_ form) (eq L^{-1})	0.6 (min)
Total boron capacity (Cl_ form) (eq L^{-1})	0.35
Selective boron capacity (Cl_ form) (eq L^{-1})	0.20 (min)
Moisture retention (Cl_ form) (%)	45–55
Reversible swelling FB→Cl (%)	10 (max)
Specific gravity (Cl_ form)	1.1
Temperature limit (Cl_ form) (ºC) 60	60
pH limits (operating)	1–13
Structure	 

The resin was in the chlorine form when purchased. The real capacity of the resin was calculated as 0.538 meq g-1 by an ion exchange reaction between OH- and exchangeable Cl- in the resin [14]. The theoretical capacity of the resin was reported as 0.545 meq g-1 [14].

Experimental Method

Batch boron removal experiments were carried out in a temperature controlled incubator shaker at 150 rpm agitation speed. The used wastewater in the experiments was supplied from Bigadiç colemanite mine and had a 382 mg L^{-1} boron concentration. The pHs of the solutions were adjusted by appropriate addition of diluted HCl and NaOH solutions. The high and low levels of the parameters used in the experimental design are given in Table 2.

Table 2: The high and low levels of the parameters used in the experimental design

Parameter	Abbreviation	Low Level (1)	High Level (2)
pH	pH	2.5	10
Temperature (ºC)	T	12	40
Resin-to-solution ratio (g/50mL)	M	1	2

Boron analysis was done by the titrimetric method in which mannitol was used as a complexing agent because boric acid is a weak acid. The procedure of the boron analysis was as follow: 5 mL boron solution was pipetted into 100 mL beaker and 50 mL distilled was added. Then solution pH was adjusted to 7.6 and 5 g mannitol was added while the solution being stirred, thereafter the solution was titrated with 0.02 N KOH up to solution pH became again 7.6. 1 mL 0.02 N KOH is equal to 0.6964 mg B_2O_3 [8]. The boron analyses were duplicated and arithmetic average of the results was put into analysis. The capacity of the resin was calculated using the following equation:

$$q_e = \frac{(C_0 - C_e) \times V}{M} \qquad (1)$$

Where C_0 (mg L^{-1}) and C_e (mg L^{-1}) are the boron concentration at initial and after equilibrium respectively. V is the volume of the solution (L) and M is the mass (g) of the resin.

The adsorption isotherm experiments were carried out by synthetic boric acid solutions of which concentrations changed from 100 to 700 mg L^{-1}(Merck Product). For this purpose, the pHs of the solutions were adjusted to 7 and 1 g resin was added to the solutions and thereafter solutions were treated with the resin during 48 hours at 30 ºC. The fixed bed experiments were carried out in a jacketed glass column reactor that had 2 cm inner diameter and 30 cm length. 10 grams of the resin were immersed in deionized water during 30 min and then filled to the reactor. The wastewater was transferred to the reactor at 2.038 mL min^{-1} speed. Temperature and pH of the wastewater was 12 ºC and 10 respectively. The optimum conditions obtained from 2^3 full factorial design were applied to fixed bed experiment. Resin capacity was calculated by the following equation.

$$q_0 = \int_0^{Vt} \frac{(C_0 - C)dV}{m} \qquad (2)$$

Where, q_0 resin capacity (mg g^{-1}), V_t solution volume passing from the fixed bed at time t, C and C_0 are the concentration of an outward solution and its initial concentration, respectively, m is resin amount in fixed bed (g).

RESULTS AND DISCUSSION

Statistical Design of Experiments

The application of statistical design to the adsorption process provides the overall process control to reach the desired response and also requires less experimental time and cost. Statistical design of experiment reduces the total number of experiments when compared with the classical single parameter experiments. The design determines separately the importance degrees of each factor and their interactions on the response [13]. In this study, the parameters such as pH, temperature and resin-to-solution ratio were optimized by 2^3 full factorial design using statistical software MINITAB (Version 16) of Minitab, Inc., USA. The low (1) and high (2) levels of the parameters were 2.5 and 10 for pH, 12 and 40 ºC for temperature and 1 and 2 g/50 mL for solid-to-solution ratio respectively. The response used in the statistical analysis was the adsorption capacity (Q_e) of the resin. The experimental matrix for boron removal from the wastewater is given in Table 3. The number of experiments in the experimental matrix was calculated by the equation of $a^k = 2^3=8$ where a is the number of levels and k is the number of factors [13]. Boron analysis was carried out in duplicate and the arithmetic average of the results was used in the statistical analysis. In the statistical analysis, the effect degrees of the parameters and their interaction effect on the response were investigated by taking into consideration the regression model coefficients. The significance of model coefficients was determined by the Student's t test. The P values (probability constants) were used as control parameter to check the reliability of the developed statistical model, individual and interaction effects of the parameters. In general, the larger the magnitude of t and the smaller the value of P, the more significant is the corresponding coefficient term [13]. Main factor, interaction effect, coefficients of the model, standard deviation of each coefficient, and probability for the full 2^3 factorial design are presented in Table 4.

Table 3: Experimental matrix for boron removal from wastewater

Trial	T	pH	M	Adsorption Capacity (Q_e, mg g^{-1}) (1)	(2)	Average
1	2	2	2	9.32171	9.32171	9.32171
2	2	2	1	12.2514	12.2514	12.2514
3	2	1	2	7.78078	7.78078	7.78078
4	2	1	1	8.14227	8.14227	8.14227
5	1	2	2	9.43586	9.37879	9.40732
6	1	2	1	13.0504	12.7080	12.8792
7	1	1	2	8.00907	7.89493	7.95200
8	1	1	1	8.14227	8.14227	8.14227

Table 4: Full factorial fit for the boron adsorption.

Term	Effect	Coefficient	t-value	p
Constant		9.4846	106.36	0.006
T	-0.2212	-0.1106	-1.24	0.432
pH	2.9606	1.4803	16.60	0.038
M	-1.7383	-0.8692	-9.75	0.065
T pH	-0.1355	-0.0678	-0.76	0.586
T M	0.0927	0.0464	0.52	0.695
pH M	-1.4625	-0.7312	-8.20	0.077
[a]pH·M·T	—	—	—	—

S.E. of coefficient = 0.251023 R^2 = 99.76%, t-value: Student's test value, p: probability.
[a]When the trial effect (pH·m·T) was added to the analysis, the programme gave error and therefore its statistical results were not shown.

The analysis of variance for the full 2^3 factorial design is presented in Table 5.

Table 5: Analysis of variance for boron adsorption.

Source	Degree of freedom (d.f.)	Sum of squares (seq. SS)	Adjusted Sum of squares (adj. SS)	Adjusted Mean square (adj. MS)	F-value	p-value
Main Effects	3	23.6716	23.6716	7.8905	124.03	0.066
2-Way Interactions	3	4.3315	4.3315	1.4438	22.70	0.153
Residual Error	1	0.0636	0.0636	0.0636		
Total	7	28.0667				

As can be seen in Table 4, only solution pH effect was found as statistically important at 95% confidence level ($p<0.05$) and the other parameters were unimportant. The developed statistical model was as follows.

Boron adsorption;

$$(Q_e) = 9.4846-0.1106T+1.4803pH-0.8692m-0.0678TpH+0.0464Tm-0.7312pHm \quad (3)$$

This function describes how the experimental variables and their interactions influence the boron adsorption (the response). As can be seen both in equation (3) and Table 4, the increasing solution temperature and resin-to-solution ratio had negative effect on the response; however, solution pH had positive effect. Furthermore, while the increasing TpH and pHm interactions had negative effect on the response, Tm interaction had positive effect on response. The reason of positive effect of Tm interaction is the swelling of resin with increasing temperature. The solution pH had the greatest effect on response and followed by resin amount (m), pH-resin-to-solution ratio interaction (pHm), temperature (T), temperature-pH interaction (TpH), temperature-resin-to-solution ratio interaction (Tm). When the trial effect (pH·m·T) was added to the analysis, the programme gave error, therefore its statistical results were not shown. We thought that this error occurred due to extremely distortion of statistical importance of p value of trial effect (pH·m·T) from 95%confidence level.

Cube Plots, Pareto Chart and Contour Plots

Figure 1 (Cube plot) illustrates the change of the resin capacity based on low and high levels of temperature, initial pH, and resin-to-solution ratio. As can be seen in Figure 1, the resin-to-solution ratio and temperature decreased the adsorption capacity with increase of the low level (1) of factors to high (2) level; however, pH increased the capacity when low (1) level of the factor increased to high (2) level. The relative importance of the main effects and their interactions was also observed on the Pareto chart (Figure 2).

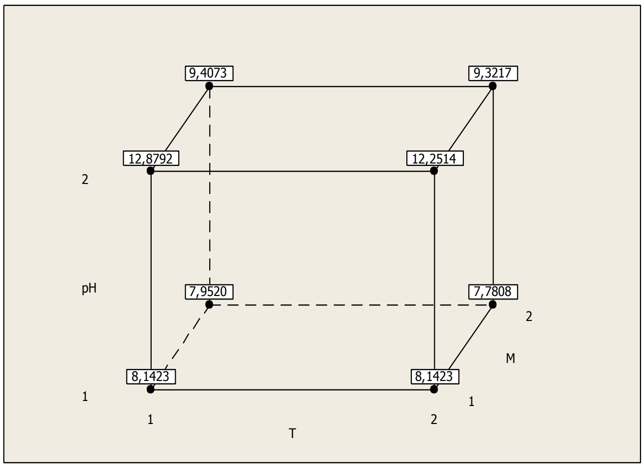


Fig. 1. Cube plots for adsorption capacity (Qe).

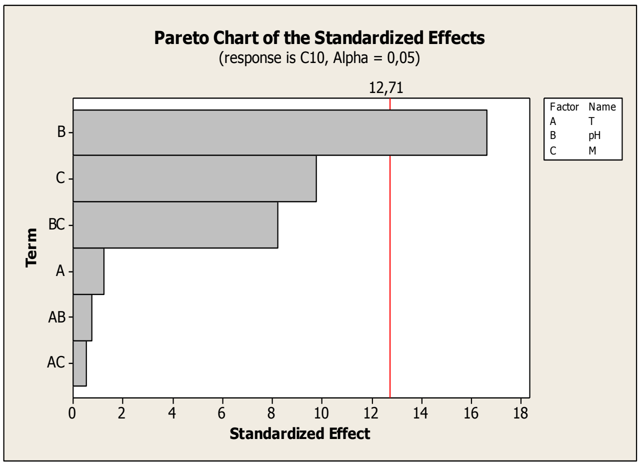


Fig. 2. Pareto chart of the standardized effects

A limit value for statistically comparison of importance of the factors was calculated by t-test as 12.71 (Pareto chart). According to Figure 2, as right side of reference line (12.71) indicates statistically importance of the factors, only pH effect was determined as statistically important and the other factors were statistically unimportant. Contours of the estimated response surface are given in Figure 3.

Contour plots enable to estimate the response Q_e values and the height of the surface represents the value of Q_e in Figure 3. In principle as the contour plots represent the interaction effect of factors, the lines are inclined shaped [15].

Effect of Parameters

In this study the effects of pH, temperature and solid-to-solution ratio on response (Q_e) were optimized using 2^3 full factorial experimental design.

Effect of temperature

Solution temperature significantly effects the boron removal by ion exchange method because boron anion type changes in liquid phase based on

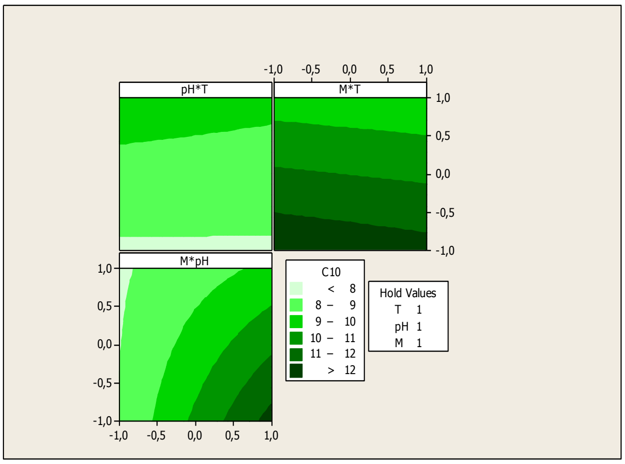


Fig. 3. Contours of the estimated response surface for Qe.

temperature. In general, lower the solution temperature and higher the concentration, the more high is the molar fraction of polyborate ions in solution [16]. According to Figure 1, the decreasing temperature increased the polyborate anion number and thus much more boron adsorption occurred on the resin [14, 16]. The increasing effect of lower temperature on the capacity showed that the process had exothermic nature.

Effect of pH

Solution pH effects boron anion type in liquid phase and resin exchangeable anion type. Purolite S 108 resin used in this study was in the chloride form at box form but it started to convert to the (OH-) form at high pHs. Korkmaz (2011) reported that when 16 grams Purolite S 108 were treated with 100 mL 2 M NaOH solution during 24 hours, the resin gave approximatelly 0.3 grams chlorine to the solution [14]. This showed the ion exchange reaction between chlorine and hydroxyl ions [14]. OH binded to the protonated amine [17]. As can be seen in Figure 1, borate anions increased at high pHs and this resulted in adsorption capacity increase [14, 16]. Furthermore complexation reaction number at the resin phase increased with conversion of the resin to OH form [14, 16]. The reaction mechanism between boric acid and resin is given in Figure 4.

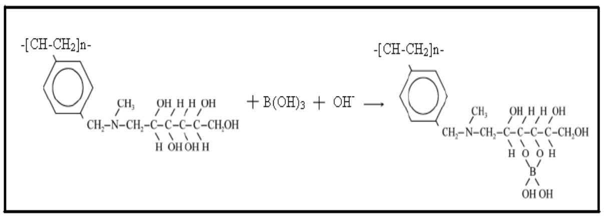


Fig. 4. The reaction mechanism between boric acid and Purolite S 108 resin

Effect of resin-to-solution ratio

Increasing resin-to-solution ratio decreased the driving force of borate anions on per unit resin particle and therefore boron adsorption capacity of the resin decreased at high resin-to-solution ratios [14, 18].

Adsorption Isotherms and Fixed Bed Kinetics

Adsorption isotherms are useful functions in design of batch adsorbers and their fitness to the equilibrium data is an important criterion. For this purposes, the most applied procedures to the isotherm data are linear regression and non-linear regression analyses. While linear-regression analysis occurs possible with the direct linearization of isotherm model, the non-linear analysis of the isotherm models occurs possible with minimization of standard normalized errors of different error functions [19]. The Langmuir and Freundlich isotherm models were applied to the isotherm data by the linear regression analysis. The Langmuir isotherm is given as follows [19].

$$q_e = q_m k_a C_e /(1 + k_a C_e) \quad (4)$$

The above equation can be rearranged to the following linear form,

$$C_e / q_e = 1/ q_m k_a + C_e / q_m \quad (5)$$

Where, Ce is the equilibrium concentration in liquid phase (mg/L). qe is the maximum amount of the boron adsorbed (mg/g). qm is qe for a complete monolayer (mg/g). ka is a sorption equilibrium constant (L/mg).

Freundlich isotherm is given as follow [19]:

$$q_e = k_F C_e^{1/n} \quad (6)$$

The equation is frequently used in the linear form by taking the logarithm of the both sides of the above equation.

$$\ln q_e = \ln k_F + \frac{1}{n} \ln C_e \quad (7)$$

Where, Ce is the equilibrium concentration in liquid phase (mg/L). qe is the maximum amount of boron adsorbed (mg/g). k_F is the Freundlich adsorption capacity (mg/g)(L/mg)$^{1/n}$. 1/n is sorption equilibrium constant (unitless).

The fitness of isotherms to the data is given in Table 6. According to Table 6, the data fitted to the Langmuir isotherm and this showed the homogeneously distribution of active sites throughout the resin particles [19]. According to Figure 5, boron capacity of the resin at high concentrations decreased.

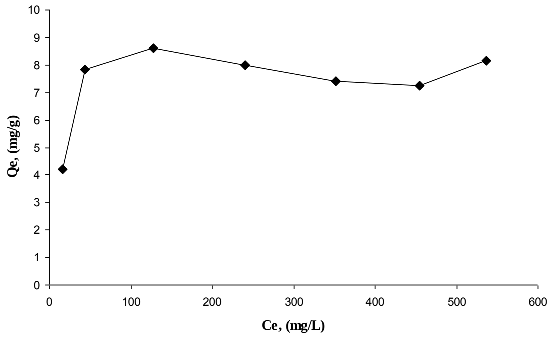


Fig. 5. Adsorption isotherm plot for boron adsorption (pH 7, temperature 30 oC, solid-to-solution ratio 1g/50 mL, agitation speed 150 rpm)

This attributed to product film outer surface of the resin [14]. The resin performance in a fixed bed is given in Figure 6.

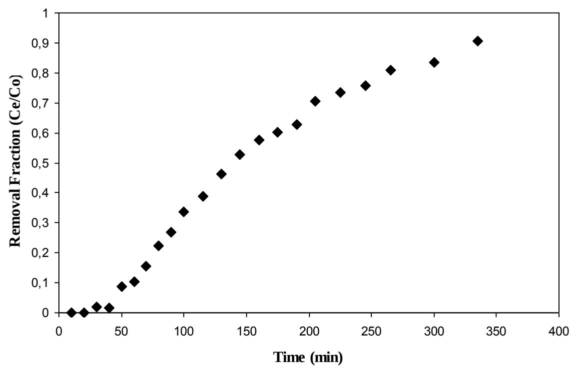


Fig. 6. Boron removal in the fixed bed reactor

The fixed bed kinetics of the resin were analyzed with the Thomas and Yoon-Nelson models. The linear model equation for Thomas model is given as follows [20].

$$\ln\left(\frac{C_0}{C} - 1\right) = \frac{K_T q_0 m}{Q} - \frac{K_T C_0}{Q} V \quad (8)$$

Table 6: The coefficient of determination values and isotherm parameters

Isotherm		Value
Langmuir Isotherm	R^2	0.991
	k_a (L/mg)	3.302
	q_m (mg/g)	7.776
Freundlich Isotherm	R^2	0.498
	k_F (mg/g)(L/mg)$^{1/n}$	21.712
	n (unitless)	7.898

Table 7: The coefficient of determination values and model constants for kinetic models.

Model		Value
Thomas	R^2	0.938
	K_T (mL/(min mg))	-39×10^{-6}
	q_0 (mg/g)	12.71
Yoon-Nelson	R^2	0.938
	K_{YN} (min^{-1})	0.015
	τ (min)	163.41

Where K_T is the Thomas rate constant (mL min^{-1} mg^{-1}) and Q is the volumetric flow rate (mL min^{-1}). C and C_0 are the concentration of an outward solution and its initial concentration (mg L^{-1}), respectively. m is the weight of ion-exchange resin (g), q_0 is the maximum concentration of boron ion-exchanged, and V is the volume of solution (L). The main advantages of this model are its simplicity and reasonable accuracy in predicting the breakthrough curves under various operating conditions [21].

The linear model equation for Yoon-Nelson model is given as follows [20].

$$\ln\left(\frac{C}{C_0 - C}\right) = K_{YN}t - \tau K_{YN} \quad (9)$$

Where K_{YN} is the rate constant (min^{-1}); τ, the time required for 50% adsorbate breakthrough (min). C and C_0 are the concentration of an outward solution and its initial concentration (mg L^{-1}), respectively. t is time (min). The Yoon-Nelson model is not only less complicated than other models, but also requires no detailed data concerning the characteristics of the sorbate, the type of the sorbent, and the physical properties of the sorption bed [20]. The coefficient of determination values and model constant for Thomas and Yoon-Nelson models are given in Table 7. The coefficient of determination values for both the models are the same (0.938). The fitness of the kinetic models to data was given in Figure 7 and 8.

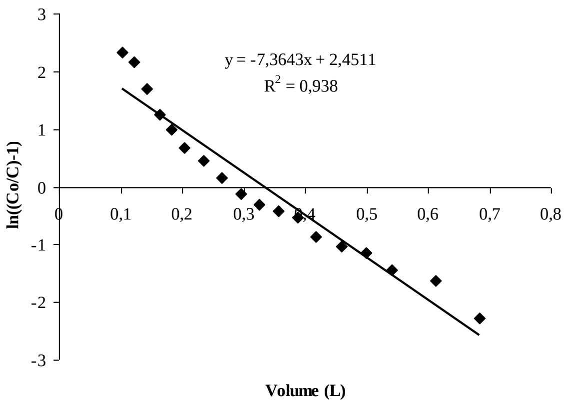


Fig. 7. The fitness of fixed bed kinetic data to the Thomas model

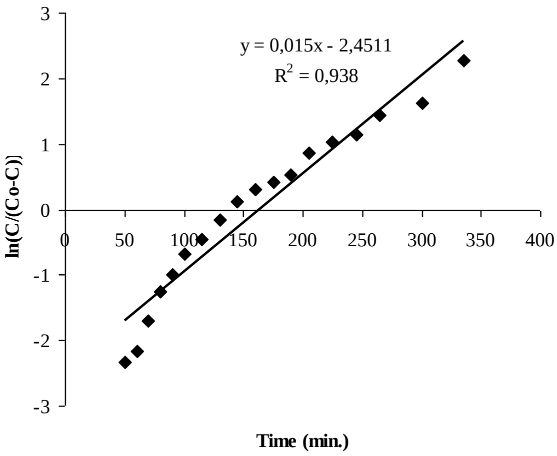


Fig. 8. The fitness of fixed bed kinetic data to the Yoon-Nelson model

CONCLUSION

The optimization of boron removal from colemanite mine wastewater using Purolite S 108 resin was performed by means of 2^3 full factorial experimental design. For this purpose, the optimization of the factors to obtain optimum conditions was done by interpretation of cube plots, Pareto chart and contour plots. Results showed that the resin-to-solution ratio and temperature decreased the adsorption capacity with increase of the low level (1) of factors to high (2) level; however, pH increased the capacity when low (1) level of the factor increased to high (2) level (Cube Plots Figure 1). The solution pH had the greatest effect on response and followed by resin amount (m), pH-resin-to-solution ratio interaction (pHm), temperature (T), temperature-pH interaction (TpH), temperature-resin-to-solution ratio interaction (Tm) (Pareto Chart Figure 2). Solution pH was found as statistically important based on the probability parameter ($p<0.05$) at 95% confidence level. The isotherm data fitted to the Langmuir model. Maximum capacity of the resin in batch mode was calculated as 12.87 mg g^{-1}. Boron removal kinetic of the resin was fitted both to Thomas and Yoon-Nelson models. The fixed bed capacity of the resin was calculated as 12.71 mg g^{-1}. Due to high boron capacity Purolite S 108 resin is an effective resin for boron removal from waters.

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ПЪЛЕН ФАКТОРЕН ЕКСПЕРИМЕНТ ЗА ОТСТРАНЯВАНЕТО НА БОР ОТ ОТПАДЪЧНИТЕ ВОДИ ОТ МИНАТА КОЛЕМАНИТ С ЙОНООБМЕННАТА СМОЛА PUROLITE S 108

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Замърсяването с бор има жизнено важно значение в мината Бигадич в Турция, тъй като отпадъчните води от мината се съхраняват в бент с пръстено дъно, което застрашава чистотата на подпочвените води. В настоящата работа се оптимизира отстраняването на бор от бородобивната мина с помощта на йонообменна смола чрез 2^3 пълен факторен експеримент. Изследванията са по периодичен способ при различни pH, температура и съотношения смола/разтвор. Ниските (1) и високите (2) нива и параметри за pH, температурата и съотношенията смола/разтвор са съответно 2.5 и 10, 12 ºC и 40 ºC и 1 g/50mL и 2 g/50mL. Адсорбционният капацитет на смолата по бор нараства при ниска температура, ниско съотношение смола/разтвор и високо pH. Когато се отчита вероятността $p<0.05$ при доверителни граници 95% се оказва, че само pH е статистически значим параметър. Оптимизацията на параметрите за постигане на оптимални условия е извършена чрез интерпретацията на кубични и контурни диаграми и таблици на Pareto. Времето от 48 часа е достатъчно за постигане на равновесие. Данните за адсорбция са анализирани по изотермите на Langmuir и Freundlich. Данните се описват по-доре с изотермата на Langmuir с коефициент на корелация 0.988. Максималният адсорбционен капацитет е определен на 12.87 mg g^{-1}. Кинетиката на адсорбция на бор в неподвижен слой може да се обясни с моделите на Thomas и Yoon-Nelson с коефициент на корелация 0.938. Капацитетът в този случай бе изчислен на 12.71 mg g^{-1}.