Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

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Online publication date: 15 September 1999

To cite this Article Badruk, Mebrure , Kabay, Nalan , Demircioglu, Mustafa , Mordogan, Hasan and Ipekoglu, Uner(1999) 'Removal of Boron from Wastewater of Geothermal Power Plant by Selective Ion-Exchange Resins. I. Batch Sorption-Elution Studies', Separation Science and Technology, 34: 13, 2553 — 2569
To link to this Article: DOI: 10.1081/SS-100100790
URL: http://dx.doi.org/10.1081/SS-100100790

SEPARATION SCIENCE AND TECHNOLOGY, **34**(13), pp. 2553–2569, 1999

Removal of Boron from Wastewater of Geothermal Power Plant by Selective Ion-Exchange Resins. I. Batch Sorption–Elution Studies

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ABSTRACT

Boron removal was studied using *N*-glucamine-type resins Diaion CRB 02 and Purolite S 108. The resin Diaion CRB 02 exhibited a higher sorption capacity for boron removal from 0.01 M H_3BO_3 solution than did Purolite S 108. The presence of calcium, sodium, and chloride ions did not make a large interference on boron removal by both Diaion CRB 02 and Purolite S 108 resins. The sorption behavior of these two chelating resins obeyed the Langmuir isotherm model. Kinetic tests were performed to find the mass transfer mechanism of the sorption process of boron by Diaion CRB 02 resin. Five kinetic models were applied to fit the kinetic data obtained by using glucamine type-resin Diaion CRB 02. The results showed that the rate-determining step is particle diffusion for boron removal by Diaion CRB 02. The quantitative stripping of boron from both chelating resins was obtained with either 0.05 M

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H_2SO_4 or 0.1 M HCl solutions. Boron in wastewater of the Kizildere geothermal field was effectively removed by both Diaion CRB 02 and Purolite S 108 resins. Preliminary column tests showed that Diaion CRB 02 is a potential resin for column removal of boron from wastewater of a geothermal power plant.

Key Words. Boron; Ion exchange; Boron selective chelating resins; Water pollution; Water treatment; Geothermal wastewater

INTRODUCTION

Boron is found in the earth's crust at an average concentration of about 10 mg/dm^3 (1). Continental boron deposits of the Cenozoic age supply more than 90% of the world's demand for boron compounds. Borate minerals from pre-Cenozoic marine evaporatis and metamorphic rock complexes account for the balance of world production. Borate production in the United States in 1992, all from Cenozoic deposits, was 1,009,000 metric tons of ore containing 554 metric tons of B_2O_3, worth $339 million. This represented 39% of world production. In that year Turkey accounted for 38% of the world production, while Argentine, Chile, Bolivia, and Peru together accounted for a total of 8% (2). The very large borax deposit at Kirka, Turkey is associated with at least 10 other borate minerals, which include species containing Ca, Mg, and Sr in addition to Na (3).

The main boron-bearing end products in the industry are insulation- and textile-grade fiber, borosilicate glass, fire retardants, enamels, glazes, and agricultural products (2). Boron is needed for plant growth but in relatively small amounts; however, and if present in a larger amount than is needed, it becomes toxic (4). For some crops, if 0.2 mg/dm^3 boron in water is essential, 1–2 mg/dm^3 may be toxic. Surface water rarely contains enough boron to be toxic, but well-water or springs occasionally contain toxic amounts, especially near geothermal areas and earthquake faults. Boron problems originating from water are probably more frequent than those originating in soil. Boron toxicity can affect nearly all crops but, like salinity, there is a wide range of tolerance among crops (5, 6). Boron contamination of water is a serious environmental problem which lacks a cost-effective solution. In dilute aqueous solutions, boron exists as boric acid [$B(OH)_3$] or borate ion [$B(OH)_4^-$] depending upon solution pH.

There are several methods suggested for boron removal from aqueous solutions. Among those methods, the ion-exchange process is most extensively used. It is well known that chelating resins containing functional groups in which hydroxyl groups are in the *cis* position show high selectivity for boron

removal through the formation of borate-diol complexes (7–12). It was explained in the literature that boron selective resins containing D-glucitylamino groups have been prepared by the reaction of beads of chloromethylated crosslinked copolymers of styrene–divinylbenzene with 1-amino-1-deoxy-D-glucitol; 1-deoxy-1-(methylamino)-D-glucitol, 1,1-iminobis-(1-deoxy-D-glucitol), and 1,4-piperazinedylbis(1-deoxyl-D-glucitol) (13). The chelating resin Wofatit MK 51 was found to be effective to separate boric acid from brine containing alkali and alkaline earth salts in considerable concentration (11). Removal of boron from natural gas brines was studied by commercially available chelating resins containing *N*-methyl(polyhydroxyhexyl) amino groups. The resins used were Amberlite IRA 743, Diaion CRB 02, Duolite ES 371, and Uniselec UR 3500 (8). Macroporous chelating resins containing polyol groups (RGB) were prepared by the reaction of glicidyl methacrylate beads with 2-amino-2-hydroxyl methyl-1,3-propanediol, and the behavior of the resulting RGB in the uptake of boric acid was studied by Maeda et al. (14). The RGB exhibited high affinity for boric acid from geothermal power waste solutions. Ooi et al. recently reported the screening results of various adsorbents for boron removal from brine (15). Among the adsorbents, some hydrous oxide of tetravalent metals ($CeO_2 \cdot nH_2O$, $ZrO_2 \cdot nH_2O$, $HfO_2 \cdot nH_2O$) or pentavalent metals ($Ta_2O_5 \cdot nH_2O$) showed a good adsorption property for boron. A boron sorption study was also performed with a glucamine-type resin (Diaion CRB 02) by using the residual brine after salt production from seawater (15).

A process combining adsorption and solvent extraction was also reported in the literature for the treatment of large quantities of water-containing boron (16). Recently, an extraction system for the recovery of boric acid from wastewater using 2-butyl-2-ethyl-1,3-propanediol (BEDP) as an extractant was studied by Matsumoto et al. (17).

The Denizli-Kizildere geothermal field is the first geothermal field suitable for electricity production in Turkey. Water is disposed of from the power plant at a flow rate of 1500 tons/hour and contains boron of approximately 30 mg/dm^3. The level of boron is very high if this water is to be used for irrigation in agricultural areas. According to the literature, the average level of boron permitted in irrigation water is generally 1 mg/dm^3. Recepoglu and Peker investigated boron removal from Kizildere geothermal wastewater using Amberlite IRA 743 (18).

In this study, batch sorption and preliminary column tests were performed using *N*-glucamine-type resins Diaion CRB 02 and Purolite S 108 for boron removal from wastewater of the Kizildere-Turkey geothermal field. The optimum conditions for batch sorption of boron were obtained.

EXPERIMENTAL

Kizildere-Turkey Geothermal Power Plant Wastewater

Kizildere geothermal wastewater (pH 9.30) is disposed of from the power plant at a flow rate of 1500 tons/h and contains approximately 30 mg/dm^3 boron. The concentrations (in mg/dm^3) of other ionic species in the wastewater are: K = 145; Na = 1300; NH_4 = 3.5; Ca = 0.39; Mg = 0.08; Fe (total) = <0.05; As (total) = 0.58; Li = 4.8; Al = 0.71; SiO_2 = 415; Cl = 134; I = 4.6; F = 15; Br = 0.53; NO_2 = 0.01; NO_3 = <1; HCO_3 = 1037; CO_3 = 780; and SO_4 = 695.

Chelating Ion-Exchange Resins

N-Glucamine-type resin Diaion CRB 02 was kindly supplied by Mitsubishi Chem., Japan and the resin Purolite S 108 by Purolite International Ltd., UK. The properties of these resins are given in Tables 1 and 2, respectively.

TABLE 1
Characteristics of Diaion CRB 02

Constitutional type	Highly porous type
Ion form as shipped	OH form
Shipping density (g/dm^3) (approx.)	635
Moisture content (%)	50–60
Exchange capacity	Acid 0.6 meq/cm^3 (min.)
Screen grading	1180–300 μm (through 300 μm, max. 1%)
Effective size (mm)	0.35–0.55
Uniformity coefficient (max.)	1.6
Operating temperature (°C) (max.)	100°C max. (OH form)
Effective pH range	6–10
Uses	Boric acid selective adsorption
Specific surface area (m^2/g)	27
Structure:	

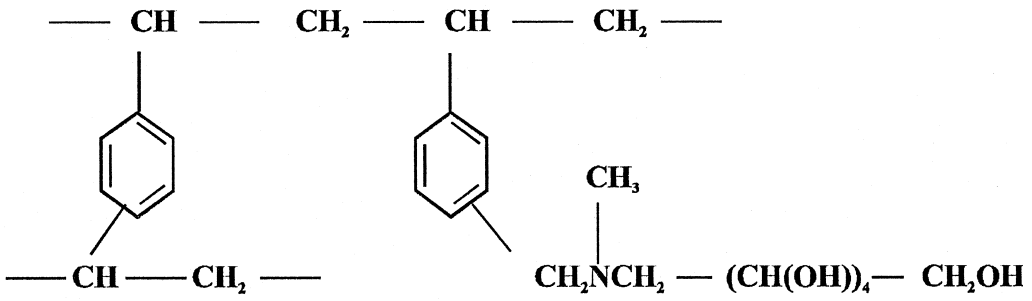

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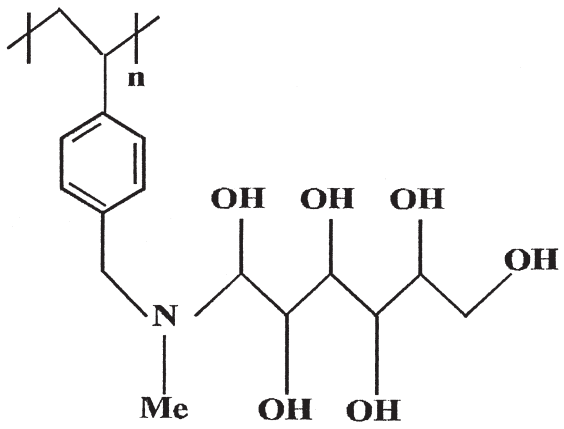
TABLE 2
Characteristics of Purolite S 108

Polymer matrix structure	Macroporous styrene–divinylbenzene
Physical form and appearance	Opaque cream spheres
Whole bead count	>95%
Ionic form (as shipped)	Cl^-
Shipping density, g/dm^3(lb/ft)	650 (41)
Screen size range:	
British Standard Screen	16–52 mesh, wet
US Standard Screen	18–50 mesh, wet
Moisture retention, Cl^- form	52–58%
Reversible swelling (Fe→Cl)	<6%
Specific gravity, moist Cl^- form	1.06
Boron-exchange capacity, Cl^- form (wet, volumetric)	0.35 eq/L
Maximum operating temperature, Cl^-/borate forms	60°C (140°F)
Operating pH range	1–13
Specific surface area (m^2/g)	17.1
Structure:	



Batch Sorption of Boron

Resin (0.25 g) was contacted with 50 cm^3 of 0.01 M H_3BO_3 solution at 30°C for 48 hours with continuous shaking. The boron concentration in the supernatant was determined spectrophotometrically using the Carmine Method. The sorption of boron from Kizildere geothermal wastewater was performed by immersing 0.25 g resin into 50 cm^3 of wastewater and shaking the mixture at 30°C for 48 hours. Kinetic tests were carried out by contacting 2.5 g resin with 500 cm^3 of 0.01 M H_3BO_3 solution at 30°C on a magnetic stirrer placed under a water bath. The concentration of boron was monitored after 5, 10, 15, 20, 30 minutes and 1, 2, 4, 6, and 24 hours by taking 2 cm^3 of supernatant from the solution. For adsorption isotherm study, 0.25 g of resin was contacted with

50 cm^3 of H_3BO_3 solution at concentrations of 50, 100, 250, 500, and 1000 mg/dm^3 at 30°C for 24 hours with continuous shaking.

Batch Elution of Boron

Resin (0.25 g) was contacted with 50 cm^3 of 0.01 M H_3BO_3 solution at 30°C for 48 hours with continuous shaking. Then the boron sorbed onto the resin was eluted using either HCl or H_2SO_4 (25 cm^3) solution at different concentrations by immersing the resin into the acid solution and shaking the mixture at 30°C for 2 hours.

Column Sorption-Elution of Boron

The resin Diaion CRB 02 in the 0.355–0.500 mm (3 cm^3) particle size range was packed into a glass column of internal diameter 0.7 cm. A solution of H_3BO_3 (600 cm^3) containing boron at a concentration of about 31.83 mg/dm^3 was delivered by downflow to the column at a flow rate (SV) of 25 bed volumes per hour using a peristaltic pump (Atto SJ 1211 H). The breakthrough curve was obtained by the analysis of successive 6 cm^3 of fractions of the effluent using a fraction collector (Advantec SF 2120 Model). The column elution was carried out using 0.25 M H_2SO_4 solution at SV 5 h^{-1}. The elution profile analysis was performed by collecting 3 cm^3 fractions of the eluate.

RESULTS AND DISCUSSION

Removal of Boron by Chelating Ion-Exchange Resins

Boric acid exists predominantly in water as the monomeric orthoboric acid, $B(OH)_3$. Boric acid chemistry is dominated by the Lewis acid character of this compound, and virtually every reaction appears to occur by a mechanism in which the first step involves a "donor atom" bonding to boron. The reaction of boric acid with strong bases in aqueous solution has been shown to produce the $B(OH)_4^-$ ion according to the following equilibrium reaction (4, 15):

$$B(OH)_3 + OH^- \rightleftharpoons B(OH)_4^- \qquad (\log Q_{1,1} = 5.28) \qquad (1)$$

In the present study boric acid selective *N*-glucamine-type resins Diaion CRB 02 and Purolite S 108 were used for batch sorption of boron from 0.01 M H_3BO_3. The following chelation mechanism (Eq. 2) was given in the literature for boron sorption by glucamine-type resins (15):

$$\begin{aligned} &P—CH_2—N(CH_3)—CH_2—[CH(OH)—]_4—CH_2OH + B(OH)_3 \rightarrow P—CH_2 \\ &\quad —N(CH_3)—CH_2—[CH(OH)—]_2—\underset{\diagdown\ \diagup}{CHO—CHO}—CH_2OH + 2H_2O \qquad (2) \\ &\qquad\qquad\qquad\qquad\qquad\qquad\qquad\quad B(OH) \end{aligned}$$

where P = polymeric backbone.

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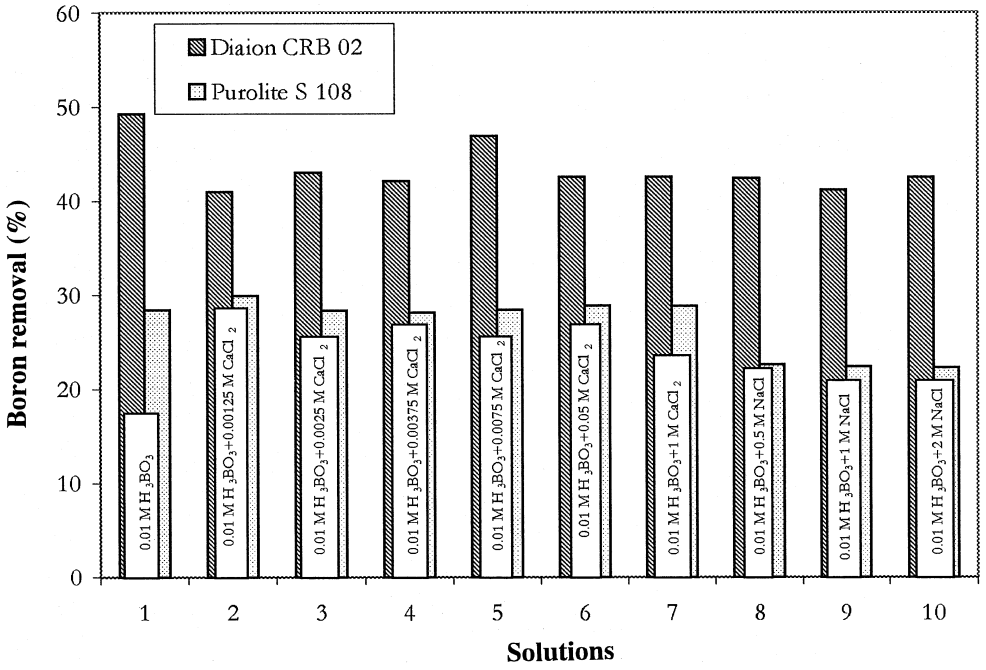

FIG. 1 Effect of Ca, Na, and Cl ions on batch sorption of boron from 0.01 M H_3BO_3 solution by chelating resins.

Boron removals by both chelating resins are shown in Fig. 1. *N*-Glucamine-type resin Diaion CRB 02 exhibited a higher sorption capacity for boron removal from 0.01 M H_3BO_3 than did Purolite S 108.

The major ionic components in wastewater of the Kizildere geothermal power plant are sodium, potassium, chloride, sulfate, bicarbonate, carbonate, and borate. Bearing in mind the existence of ionic impurities along with boron in wastewater, boron removal by chelating ion-exchange resins was examined in the presence of sodium chloride and calcium chloride. The results, given in Fig. 1, show some decrease was observed in the removal of boron by Diaion CRB 02 resin in the presence of Ca, Na, and Cl ions. However, the removal was not influenced by an increase in the concentrations of these foreign ions. In the case of Purolite S 108 resin, boron uptake remained the same in the presence of calcium chloride although a slight decrease appeared in the presence of sodium chloride (Fig. 1).

Effect of pH

In order to get some information about the relative performance of both Diaion CRB 02 and Purolite S 108 resins; the sorption of boron was investigated as a function of pH. The results are shown in Fig. 2. Boron removal by Diaion CRB 02 increased with an increase in pH. Between pH 5–6, boron uptake al-

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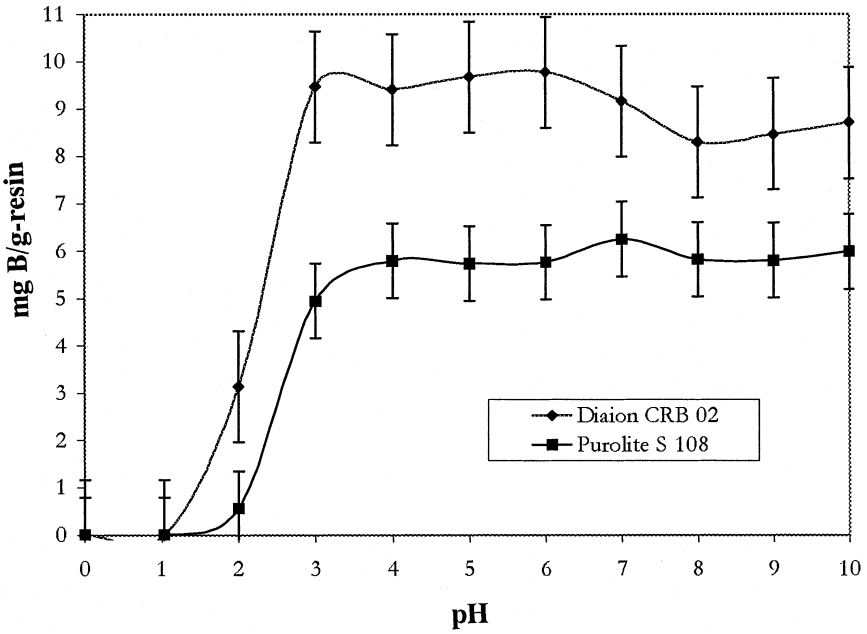

FIG. 2 Effect of pH on boron removal by Diaion CRB 02 and Purolite S 108.

most leveled off. Above pH 7, the uptake decreased slightly with increasing pH. This decrease was explained in the literature by the fact that boric acid itself exhibits acid dissociation ($pK_{a1} = 9.2$) (14). A similar behavior against pH was observed in the case of the resin Purolite S 108 although the decrease in boron removal at pH above 7 was not apparent (Fig. 2).

Adsorption Isotherms of Boron

The Langmuir equation (3) was applied for the adsorption equilibrium of both resins:

$$\frac{C_e}{Q_e} = \frac{1}{Q_0 b} + \frac{C_e}{Q_0} \tag{3}$$

where C_e is the equilibrium concentration (mg/dm^3), Q_e is the amount of boron adsorbed at equilibrium (mg/g), and Q_0 and b are Langmuir constants related to sorption capacity and energy of sorption, respectively. Linear plots of C_e/Q_e versus C_e show that sorption obeys the Langmuir isotherm model for both Diaion CRB 02 and Purolite S 108 as shown in Figs. 3 and 4. The essential characteristics of a Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor, R_L, which describes the type of the isotherm and is defined by

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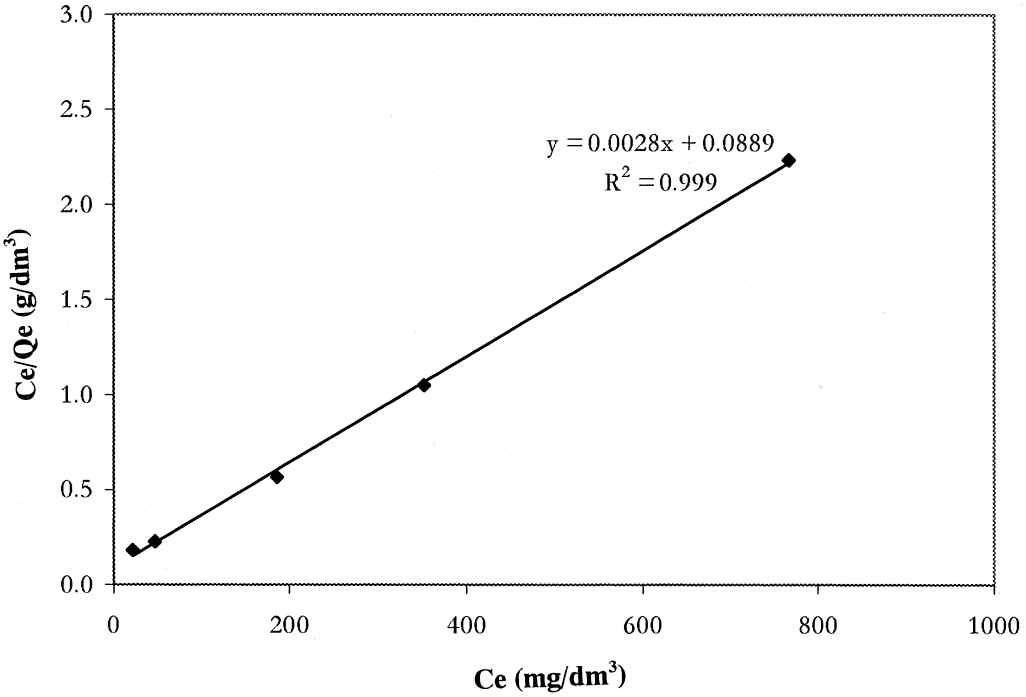

FIG. 3 Langmuir plot of boron sorption by Diaion CRB 02.

$$R_L = \frac{1}{1 + bC_0} \tag{4}$$

where b is the Langmuir constant and C_0 is the initial concentration of boron (19, 20). The values of R_L calculated were between 0 and 1, indicating favorable sorption of boron by both Diaion CRB 02 and Purolite S 108 resins.

Kinetic Study

The kinetic behavior of Diaion CRB 02 (0.710–0.500 mm) was examined in order to get a measure of the relative performance of the resin. For this, boron removal was monitored with time. The results are plotted in Fig. 5. The initial rate of boron sorption was rapid for *N*-glucamine-type resin Diaion CRB 02 as shown.

The ion exchange between the counter ion (in solution) and the exchangeable ion (on the resin) is well described by a heterogeneous process. The models for process dynamics cover both the diffusional steps (bulk solution, a film layer at the external surface of the particle, pores) and the exchange reaction on the active sites (21). Since the resistance in bulk solution is easily controlled and negligible, three resistance, such as film diffusion, particle diffu-

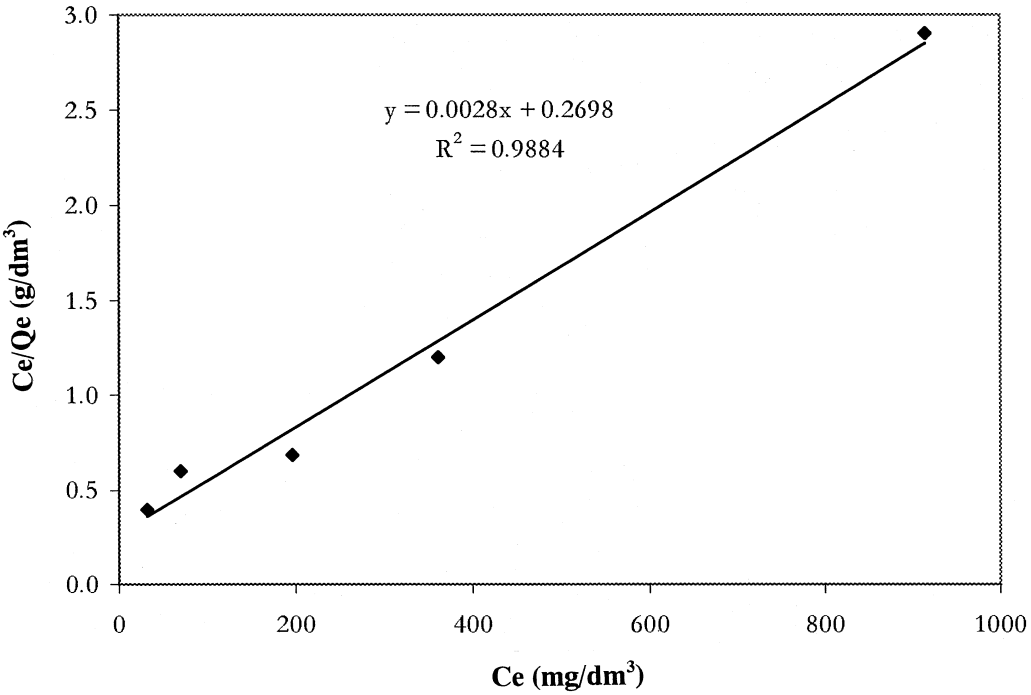

FIG. 4 Langmuir plot of boron sorption by Purolite S 108.

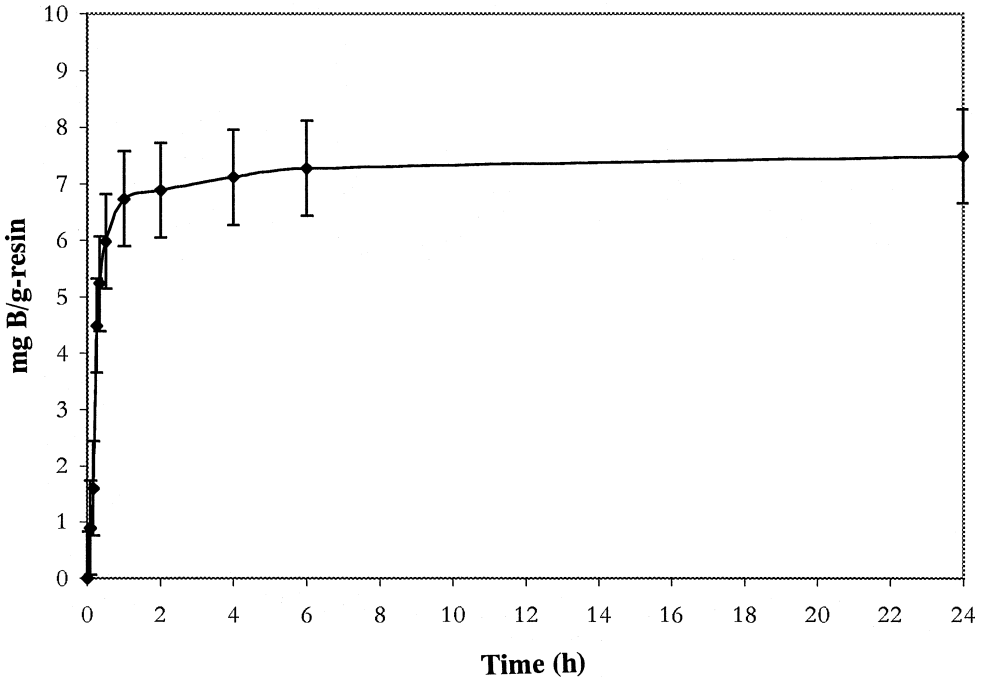

FIG. 5 Kinetic curve of boron removal by Diaion CRB 02 (0.710–0.500 mm)

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sion, and chemical reaction, usually determine the overall rate of the ion-exchange process. One approach to the kinetic study is based on Fick's first law of integration of material balance for infinite solution volume (ISV) (22), whereas the other method uses the unreacted core model (UCM) in which ion exchange is treated as a heterogeneous reaction first occurs at the outer skin of the particle, then within a zone moving into the particle through the unreacted core (23). Five kinetic models developed for spherical particles in different cases of the rate-determining steps are given in Table 3 (24, 25). These models were applied to fit the kinetic data of the resin Diaion CRB 02 (0.710–0.500 mm). Figures 6 and 7 show the functions of these kinetic models versus time. On these charts, markers are obtained by substitution of experimental values of X into model equations, whereas lines are drawn by the least-square methods on corresponding model equations. Since the resin Diaion CRB 02 reached equilibrium after about 1 hour, the data obtained at contact times larger than 1 hour were neglected in this application of kinetic models. The governing equation, i.e., the rate-controlling step, among the five kinetic models is determined statistically by least-square analysis of linearized model equations. The model having good statistics shows the rate-determining step of the heterogenous ion-exchange process. In the case of several models with acceptable statistics, the overall rate is controlled by the partial contributions of these steps. Table 4 gives the slope values and linear correlation coefficients. In order to determine the statistically meaningful set of data with seven degrees of freedom and at the 99% confidence level, the r minimum is 0.798. Hence, the particle diffusion model is the best kinetic model to describe the ion-exchange processes. As seen in Table 4, the maximum of the correlation coefficients for the linear models shows that the reaction is particle diffusion controlled.

Batch Elution of Boron

A chelating resin will be of real value if the sorbed species are removed from the resin quantitatively and the resin can be recycled many times after a

TABLE 3
Diffusional and Reaction Models

Method	Equation	Rate-determining step
ISV	$-\ln(1 - X^2) = kt$, where $k = D_r\pi^2/r_0^2$	Particle diffusion
ISV	$-\ln(1 - X) = K_{1i}t$, where $K_{1i} = 3DC/r_0\delta C_r$	Film diffusion
UCM	$X = (3C_{A0}K_{mA}/ar_0C_{s0})t$	Liquid film
UCM	$3 - 3(1 - X)^{2/3} - 2X = (6D_{eR}C_{A0}/ar_0{}^2C_{s0})t$	Reacted layer
UCM	$1 - (1 - X)^{1/3} = (k_sC_{A0}/ar_0C_{s0})t$	Chemical reaction

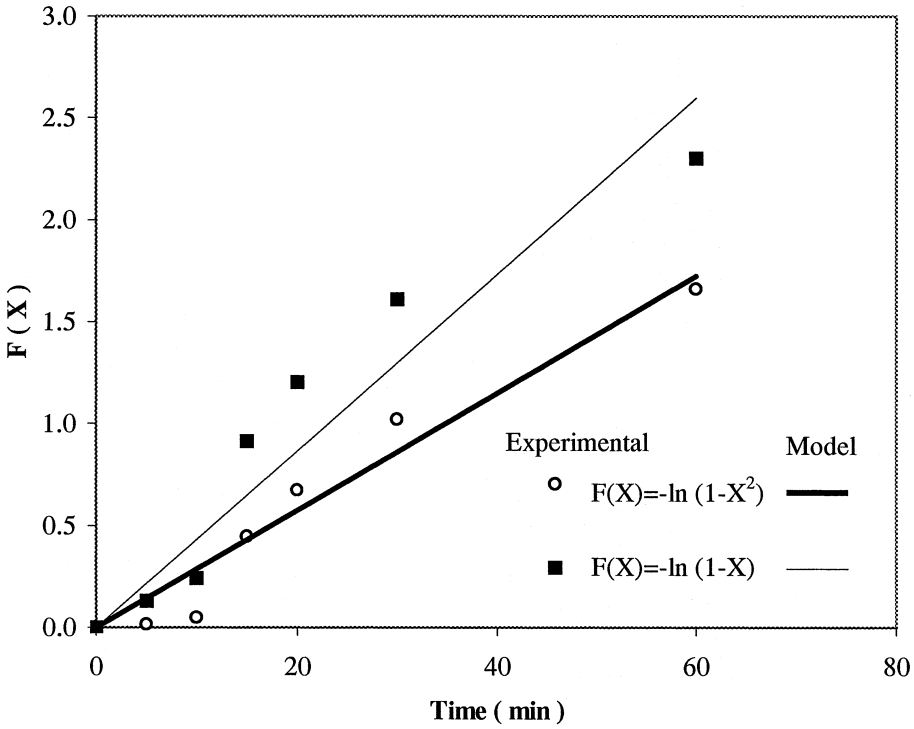

FIG. 6 Kinetic behavior of Diaion CRB 02 (0.710–0.500 mm) based on infinite solution volume models.

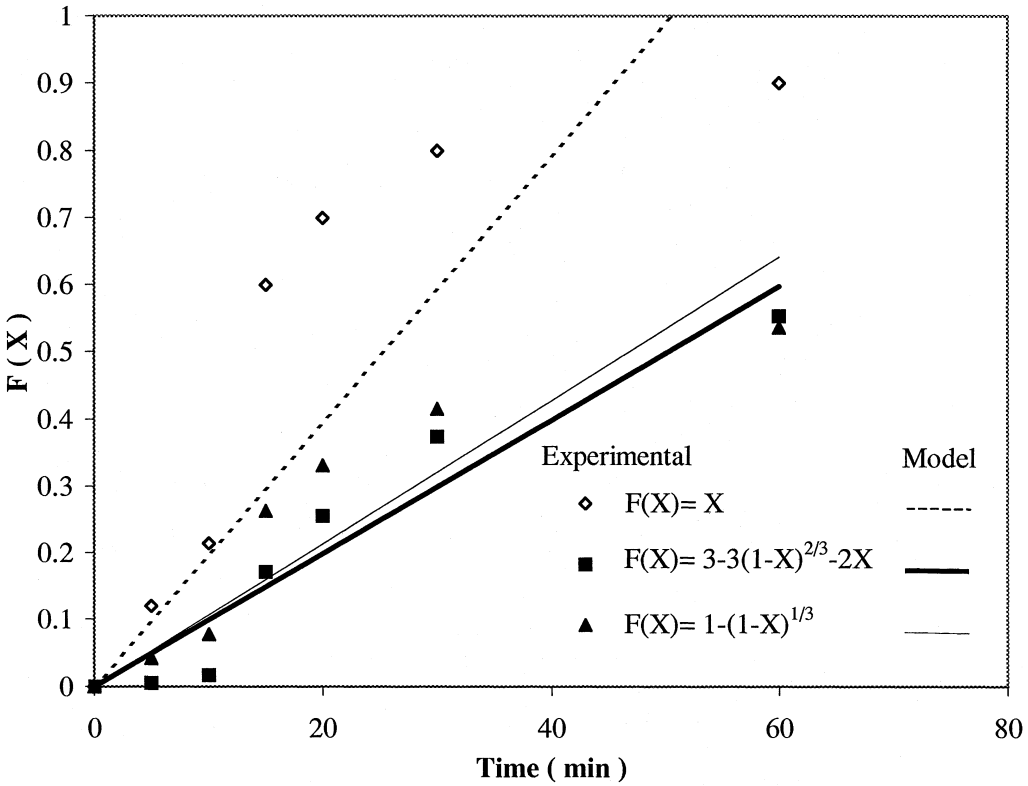

FIG. 7 Kinetic behavior of Diaion CRB 02 (0.710–0.500 mm) based on unreacted core models.

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TABLE 4
Evaluation of Kinetic Models for Diaion CRB 02
(0.710–0.500 mm)

Model	Slope	r^2
$-\ln(1 - X)$	0.0433	0.9062
$-\ln(1 - X^2)$	0.0287	0.9511
X	0.0198	0.5987
$3 - 3(1 - X)^{2/3} - 2X$	0.01	0.9260
$1 - (1 - X)^{1/3}$	0.0107	0.8194

regeneration process. In order to obtain good elution efficiency, acid eluants such as H_2SO_4 and HCl at various concentrations were used for stripping boron from both Diaion CRB 02 and Purolite S 108. The results are given in Table 5. A quantitative stripping for boron was obtained with a H_2SO_4 concentration as low as 0.05 M and with 0.1 M HCl. The column elution behavior of resins with acid eluants will be published elsewhere.

Removal of Boron from Kizildere Geothermal Field Wastewater

According to the spectrophotometric analysis results, the boron content of Kizildere geothermal field wastewater was found to be about 30 mg/dm^3. The resins Diaion CRB 02 and Purolite S 108 were used for boron removal from this geothermal wastewater. As summarized in Table 6, both resins gave quantitative batch removal of boron.

A preliminary column test was carried out to determine the column performance of the resin Diaion CRB 02 for removal of boron. An aqueous feed solution containing a boron concentration of 31.83 mg/dm^3 (pH 5.6) was deliv-

TABLE 5
Batch Stripping of Boron from Chelating Resins

Stripping agent		Stripping (%)	
		Diaion CRB 02	Purolite S 108
H_2SO_4	0.05 M	100	100
	0.25 M	100	100
	0.5 M	100	100
HCl	0.1 M	92	94
	0.5 M	95	96
	1 M	97	96

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TABLE 6
Removal of Boron from
Kizildere Geothermal Plant Wastewater

	Removal of boron	
Resin	%	mg B/g-resin
Diaion CRB 02	98.1	6.69

ered to the column at SV 25 h^{-1}. The resulting breakthrough curve is shown in Fig. 8. The boron concentration in the effluent was less than 1 mg/dm^3 up to 96 bed volumes. The resin was exhausted at 160 bed volumes. The breakthrough capacity was found to be 3.1 mg B/cm^3 of resin, and the total capacity was 4.2 mg B/cm^3 of resin. These results show that resin Diaion CRB 02 is promising for column removal of boron from Kizildere geothermal plant wastewater. The loaded column was eluted with 0.25 M H_2SO_4. The elution profile of the resin is shown in Fig. 9. The elution efficiency of boron was 80% up to 5 bed volumes of the eluate. The boron concentration of the eluate reached 2.0 g/dm^3, nearly 63 times higher than that of the feed solution.

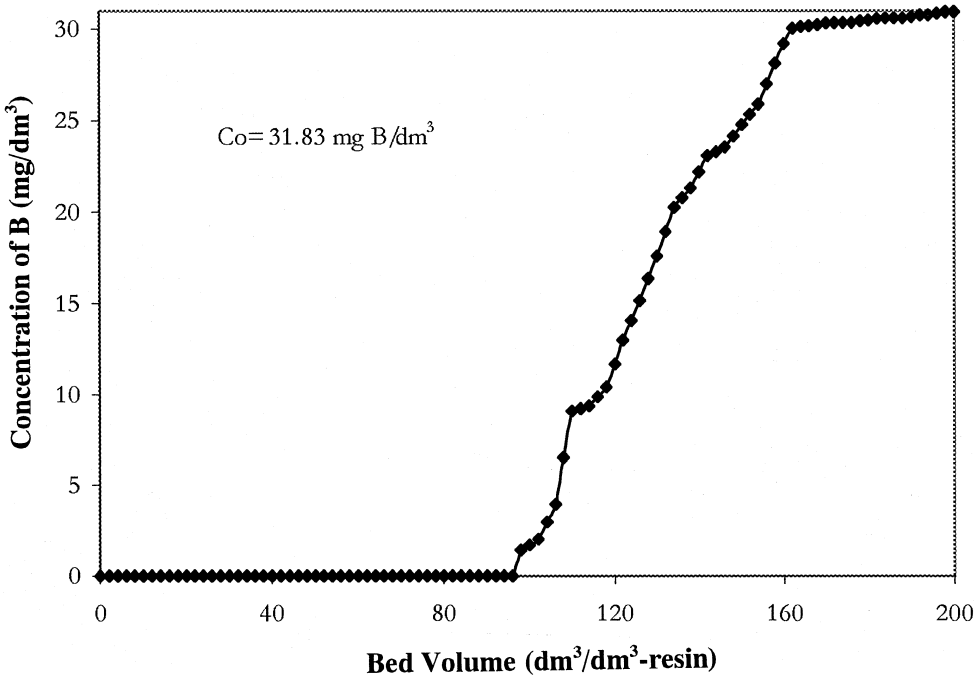

FIG. 8 Breakthrough curve of boron.

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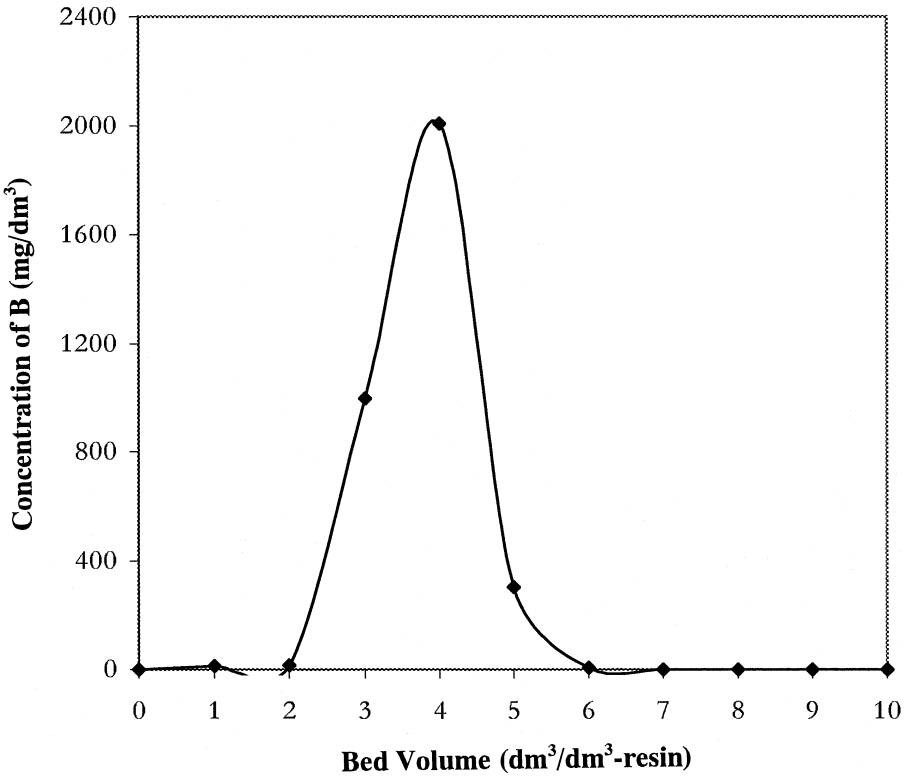

FIG. 9 Elution profile of boron.

CONCLUSIONS

The Kizildere geothermal power plant wastewater contains approximately 30 mg B/dm^3. In order to utilize this water for irrigation, the maximum content of boron should be reduced below 1 mg/dm^3. Chelating ion-exchange resins Diaion CRB 02 and Purolite S 108 performed a quantitative removal of boron from wastewater in the Kizildere geothermal field. The results of column sorption/elution studies for boron removal from Kizildere geothermal field wastewater using the same chelating ion-exchange resins and their recycle use will be published separately as a continuation of the present work.

NOTATION

$Q_{1,1}$	equilibrium quotient
X	fractional attainment of equilibrium or extent of resin conversion
k	rate constant (L/s)
t	time(s)
D_r	diffusion coefficient in solid phase (m^2/s)
r_0	average particle radius (mm)

K_{li} rate constant for film diffusion (infinite solution volume condition) (L/s)
D diffusion coefficient in solution phase (m^2/s)
δ film thickness (mm)
C total concentration of both exchanging species (M)
C_{A0} concentration of Species A in bulk solution (M)
K_{mA} mass transfer coefficient of Species A through the liquid film (m/s)
a stochiometric coefficient
C_{S0} concentration of solid reactant at the bead's unreacted core (M)
D_{eR} effective diffusion coefficient in solid phase (m^2/s)
C_{A0} concentration of Species A in bulk solution (M)
k_s reaction constant based on surface (m/s)

ACKNOWLEDGMENT

The experimental work reported here was performed at Avicenne-Project Laboratory of Chemical Engineering Department, Ege University. Thanks are due to Mitsubishi Chemical Co. (Yokohama, Japan), Purolite International Ltd. (Milan, Italy and Pontyclun, UK), and Sel Dis Ticaret (Istanbul, Turkey) for the kind supply of the resin samples.

We express our sincere gratitude to Dr. M. Yildiz, Manager of MTA-Aegean region for his kind support, and to Dr. K. Ooi, Shikoku National Industrial Research Institute, Japan, for his valuable discussion and provision of some literature on the subject research. The authors gratefully acknowledge the assistance of Mr. M. Akcay, Mr. M. E. Solak, Ms. S. Yayli, and Ms. N. Kocacik during the experimental work and calculations.

In addition, helpful comments and suggestions from the reviewers are gratefully acknowledged.

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Received by editor June 19, 1998
First revision received October 1998
Second revision received November 1998

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