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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To cite this Article Kim, Jong Sung and Yi, Jongheop(1999) 'The Removal of Copper Ions from Aqueous Solutions Using Silica Supports Immobilized with 2-Hydroxy-5-nonylacetophenoneoxime', Separation Science and Technology, 34: 15, 2957 — 2971

To link to this Article: DOI: 10.1081/SS-100100815

URL: http://dx.doi.org/10.1081/SS-100100815

SEPARATION SCIENCE AND TECHNOLOGY, **34**(15), pp. 2957–2971, 1999

The Removal of Copper Ions from Aqueous Solutions Using Silica Supports Immobilized with 2-Hydroxy-5-nonylacetophenoneoxime

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ABSTRACT

Silica supports immobilized with 2-hydroxy-5-nonylacetophenoneoxime (LIX 84) were prepared after surface modification by γ-aminopropyltriethoxysilane. Three types of silica beads and a silica powder were used as supports. Batch and packed-column tests were conducted to investigate the capabilities of the prepared adsorbents to remove copper ions. A comparison of the different silica supports was performed with respect to copper adsorption capacity, copper adsorption rate, and nitrogen content. In addition, studies were carried out for the modified silica beads that showed highest copper ion removal capability among the three silica beads (SB2-L). The extraction rate of the SB2-L increases with solution pH in the range between pH 2 and 4. Stability tests show that the SB2-L is stable under acidic conditions. From regeneration experiments it was found that copper ions which are adsorbed at the surface of the SB2-L were recovered by washing with 0.1 M HNO_3 and that the regenerated beads are reversible. The recovery ratios were between 80 and 90%. A breakthrough curve was obtained using modified silica powder (SP-L). The copper recovery ratio for SP-L was 74%. The results show that the SP-L prepared is feasible for the extraction of copper ions from aqueous solutions using a fixed-bed reactor.

INTRODUCTION

As environmental regulations become restrictive, the removal of hazardous heavy metals from aqueous streams promises to be a significant industrial waste problem. Waste streams from electronics, electroplating, and photo-

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graphic industries contain metal ions, such as copper, nickel, zinc, chromium (+6), cadmium, aluminum, silver, and gold in various aqueous solutions. The most common treatment method involves the precipitation of metal ions in the form of hydroxides or carbonates. Other promising recovery methods which have been recently considered include evaporation, electrolytic metal recovery, reverse osmosis, and ion exchange (1).

Solvent impregnated resins (2–4) and inorganic supports immobilized with ion-chelating agents (5–14) have received considerable attention for separation processes of heavy metals from aqueous solutions. The technique is the result of advances in the development of commercial extractants for metal ion separation in the hydrometallurgical industries (15–17).

The advantage of using inorganic supports compared to polymeric supports can be summarized as follows: good selectivity, no swelling, rapid sorption of metal ions, and good mechanical stability (5, 6).

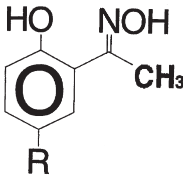
The direct attachment of organic molecules to inorganic surfaces is difficult due to the relative inertness of the surfaces. Organic extractants can be attached to an inorganic surface by using surface modification procedures which are commonly used for enzyme immobilization and chromatographic separation (18, 19).

In this study the silane coupling method was applied in order to attach 2-hydroxy-5-nonylacetophenoneoxime (LIX 84) to the surface of the silica for the removal of metal ions from dilute aqueous streams. Using three different types of silica beads and a silica powder as supports, the effects of pore structure and particle size on the efficiency of copper ion removal were investigated. In addition, the regeneration characteristics of the adsorbents prepared were investigated.

EXPERIMENTAL

Materials and Apparatus

Three different types of silica beads (Fuji Silysia Chemical Co.) and a silica powder (Aldrich Co.) were used as supports. The characteristics of the supports are listed in Table. 1. In Table 1, SB*i* (*i* = 1, 2, 3) denotes silica beads and SP represents silica powder. 2-Hydroxy-5-nonylacetophenoneoxime (3.2 mol/L) diluted in kerosene (LIX 84, Henkel Co.) was used as a chelating agent without further purification (see Scheme 1). For chemical treatment of



SCHEME 1 The structure of 2-hydroxy-5-nonylacetophenoneoxime. R = C_9H_{19}.

TABLE 1
Characteristics of Silica Supports Used in the Study[a]

Properties	SB1	SB2	SB3	SP
Surface area (m^2/g)	292	259	112	300
Diameter (mm)	0.5–1.0	0.5–0.85	0.5–1.0	0.07–0.15
Average pore diameter (Å)	148[b]	175	307	150
Pore volume (mL/g)	1.16	1.13	1.17	1.15
Bulk density (g/mL)	0.4	0.41	0.38	0.38

[a] Supplied by the manufacturer: SB*i* (*i* = 1, 2, 3) (Fuji Silysia Chemical Co.), SP (Aldrich Co.).
[b] From BET analysis.

the silica support, 10% (w/w) γ-aminopropyltriethoxysilane in toluene (Aldrich Chemical Co.) was used. $Cu(NO_3)_2 \cdot 3H_2O$ (Junsei Chemical Co.) was used to prepare copper ion solutions. All reagents were analytical grade.

Buffer solutions were used to maintain a constant solution pH. 0.2 M KCl and 0.2 M HCl (pH 2), and 0.1 M $C_8H_5KO_4$ and 0.1 M HCl (pH 2.5, 4) were used to prepare the buffer solutions.

The metal ion concentrations were measured using an Atomic Absorption Spectrometer (Perkin-Elmer, Model 3110). A CHN Corder (Leco, Model CHNS-932) was used for elemental analysis to determine the amount of organic molecules in the substrates. FT-IR (Perkin-Elmer) was used to investigate the degree of immobilization of LIX 84 on the surface of the silica support.

Preparation of Silica Beads Immobilized with LIX 84

Silica beads immobilized with LIX 84 (SB*i*-L, i = 1, 2, 3) were prepared as follows. In this case, SB*i*-L represents adsorbents immobilized with LIX 84 using SB*i* (*i* = 1, 2, 3). Silica beads (10 g) were dried at 100°C in an oven for 3 hours. The dried beads were refluxed for 16 hours with 100 mL of 10% (w/w) γ-aminopropyltriethoxysilane mixture solution with toluene. After washing three times with 100 mL of toluene followed by two washings with 100 mL of acetone, the treated silica beads were dried in an oven at 100°C for 13 hours. For impregnation with LIX 84, vacuum was applied using a vacuum evaporator for 10 minutes before introducing the extractant. After the silica beads were fully immersed into the LIX 84, vacuum was applied for 1 hour. The impregnated beads were removed, quickly washed with acetone to remove unadsorbed hydroxyoxime, and then dried under vacuum (30 mmHg) at 130°C for 16 hours to completely evaporate the kerosene. The dried beads were treated with 0.1 M HNO_3 on a rotating shaker for 2 hours and washed with deionized water. The beads were then air dried at room temperature for 24 hours and then further dried at 110°C for 5 hours in an oven to remove residual water from the interior of the pores. The silica beads thus prepared were kept in an oven at 100°C until needed.

Silica powder immobilized with LIX 84 (SP-L) was prepared using the same procedure as described above, but without the acetone washing after impregnation with LIX 84.

Adsorption and Elution of Copper Ions

The copper adsorption capacities of the adsorbents prepared as SB*i*-L ($i = 1, 2, 3$) and SP-L were investigated by batch operations. Adsorbent (0.2 g) was placed in a flask containing 25 mL of 5 mmol/L copper solutions (pH 4), and the flask was then placed on a shaker and rotated gently for 48 hours.

Transient adsorption experiments were carried out using a packed-column reactor. A Pyrex tube (ϕ 11 mm) was packed with 2.5 g of the adsorbent. The apparatus is composed of a circulation pump and flowmeter. Feed solution (125 mL) containing of 2 mmol/L copper ion (pH 4) was fed to the reactor and recycled to the solution bath at a rate of 20 mL/min. The solution in the bath was constantly mixed by a magnetic stirrer, and 0.5 mL aliquots were removed for analysis.

The stabilities of the functionalized silica surface under acidic and alkaline conditions were studied. The SB2-L was treated with two acidic solutions (0.1 M HCl and 1 M HCl) and two alkaline solutions (0.1 M NaOH and 0.01 M NaOH), respectively, for 27 hours prior to being exposed to the copper solution. The equilibrium copper concentration was measured after exposure for a 48-hour period.

Successive adsorption and elution rate data of the SB2-L were obtained using the packed-column reactor. The transient copper adsorption experiment was conducted using the method described above, and then 125 mL of 0.1 M HNO_3 solution was fed to the reactor at a rate of 20 mL/min in order to desorb the copper ions from the adsorbents. This operation was repeated for a total of three adsorption cycles.

The breakthrough experiment was performed for the SP-L using a packed-column reactor. A copper solution (1 mmol/L) was passed through a packed column (ϕ 16 mm) loaded with 20 g of the adsorbents at a flow rate of 5 mL/min. In this case the copper solution was prepared from deionized water without a buffer solution. The pH of the copper solution was 3.7. The elution experiment was conducted after the SP-L was saturated with the copper ions. A 0.1 M HNO_3 solution was fed to the reactor at a rate of 5 mL/min in order to elute the copper ions from the SP-L.

RESULTS AND DISCUSSION

Functionalization and Adsorption Capacity

The surfaces of the silica beads prepared were functionalized as follows (18, 19):

$$3(\equiv\text{Si—OH}) + (C_2H_5O)_3Si(CH_2)_3NH_2 \rightarrow (\equiv\text{Si—O})_3Si(CH_2)_3NH_2 \quad (1)$$

LIX 84 was immobilized on the silanized surface. It is well known that the organofunctional group of the silane forms chemically covalent bonds with the reactive species in the organic material. The functional groups of the LIX 84 are C_9H_{19}, CH_3, and OH. However, the OH group takes part in metal binding and the other two groups are not expected to combine with amine groups at the end of the silanized surface by covalent bonding in this study.

Thus, attachment of LIX 84 onto a silica surface by hydrophobic attraction is through surface modification. The attraction forces between the solute and the solid surface are generally van der Waals, hydrogen bonding, hydrophobic association, acid–base, and complexation interactions (19). Deorkar and Tavlarides (6) suggested that the solid phase extractant bis(2,4,4-trimethyl) monothiophosphonic acid might adhere to the silanized silica surface strictly due to van der Waals interactions. Similarly, the LIX 84 can be expected to bind with hydrocarbon chains on the silanized surface by noncovalent bonding such as van der Waals attraction.

The silanization method appears to involve the coupling of a monolayer of silane across the silica surface (18). Pores were coated by the impregnation process. Relatively smaller pores deep in beads would be expected to be blocked by LIX 84.

Figure 1 shows the FT-IR spectra of the SB2, SB2-S, and SB2-L. Here, SB*i*-S ($i = 1, 2, 3$) represents the SB*i* sample silanized with γ-aminopropyltriethoxysilane. Absorption bands at 1385 and 2967 cm^{-1} were observed for the SB2-L. These two peaks are believed to be the characteristic peaks which are associated with the immobilization of LIX 84 on the silica surface.

An elemental analysis was performed to examine the concentration of LIX 84 in the adsorbents (Table 2). The nitrogen contents of SB1-S, SB2-S, SB3-S, and SP-L were 1.44, 1.32, 0.85, and 1.38% (w/w), respectively. These results indicate that the concentration of silane immobilized on the silica surface is proportional to the surface area of the support. The concentrations of LIX 84 in the adsorbents were also calculated from the results of nitrogen analysis. For example, in the case of SB2-L, the nitrogen content of the SB2-L was 2.11% (w/w) and that of the SB2-S was 1.32% (w/w). The bulk density of the SB2-S was 0.467 g/mL and that of the SB2-L was 0.556 g/mL. Considering the change in bead density after impregnation, the net concentration of LIX 84 in the SB2-L is calculated to be 0.719 mmol/g. By the same analogy, the net

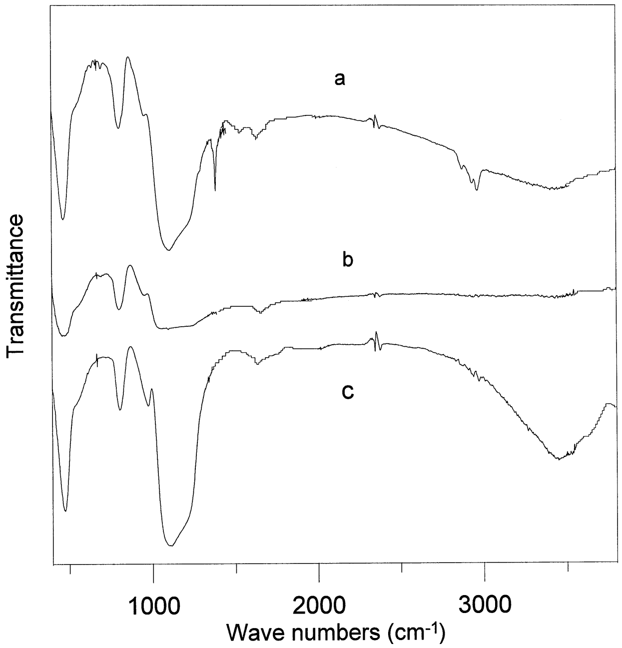


FIG. 1 FT-IR spectra of the (a) SB2-L, (b) SB2-S, and (c) SB2.

concentration of LIX 84 in the SB1-L is calculated to be 0.574 mmol/g and that in the SB3-L is 0.284 mmol/g. Duplicate adsorbents were prepared by the same procedure to investigate the deviation. The average concentration of LIX 84 in the SB1-L, SB2-L, and SB3-L samples are 0.674, 0.614, and 0.297 mmol/g, respectively. The deviations are within approximately 4–7% based on the average values. The results showed that the amount of LIX 84 adsorbed on the modified silica depends on the surface area when the pore volumes are close to each other. The amounts of LIX 84 of the SB1 and of SB2 are similar while that of SB3-L is much lower than the other adsorbents because of relatively small surface area, as listed in Table 2.

Copper adsorption capacity of the adsorbents was obtained by equilibrating 0.2 g of the adsorbents with 25 mL of 5 mmol/L copper solutions at pH 4. Cop-

TABLE 2
Copper Adsorption Capacity and LIX 84 Concentration

Properties	SB1-L	SB2-L	SB3-L	SP-L
Copper adsorption capacity[a](mmol/g)	0.162	0.240	0.111	0.370
LIX 84 concentration[b](mmol/g)	0.614	0.674	0.297	0.939

[a] Copper solution: 25 mL of 5 mmol/L. Adsorbents: 0.2 g.
[b] Measured by CHN Corder.

per ion adsorption of untreated silica was reported to be negligible at solution acidities of less than pH 5 (7). In this study the solution pH was controlled below 5 in order to examine the net adsorption capacity of the LIX 84 immobilized on the silica supports. The determined copper adsorption capacities are shown in Table 2. As two molecules of LIX 84 react with one copper ion, the theoretical maximum copper adsorption capacity is half the LIX 84 concentration.

Figure 2 shows the adsorption rate of the adsorbents for treating copper solutions. The adsorption rate order is SP-L > SB2-L > SB1-L > SB3-L. The results agree well with the capacity order as given in Table 2. For the SB1-L, 75% extraction was achieved after 6 hours, while an 86% extraction was found for the SB2-L. For the case of SB3-L, only 15% of the copper ions were extracted after 6 hours. The SP-L showed a very rapid adsorption rate with more than 99% of copper ions being removed within 1 hour. The high adsorption rate of the SP-L results from the relatively small particle size. Considering the copper adsorption capacity of the SP-L (0.370 mmol/g) and the SB2-L (0.240 mmol/g), the copper adsorption rate of SP-L is much higher than that of the SB2-L. These data suggest that adsorption rate is dependent on particle size.

Adsorption Rate Control Step

The overall metal ion exchange in the adsorption process can be expressed by a three-step processes; liquid film mass transfer, diffusion step into the in-

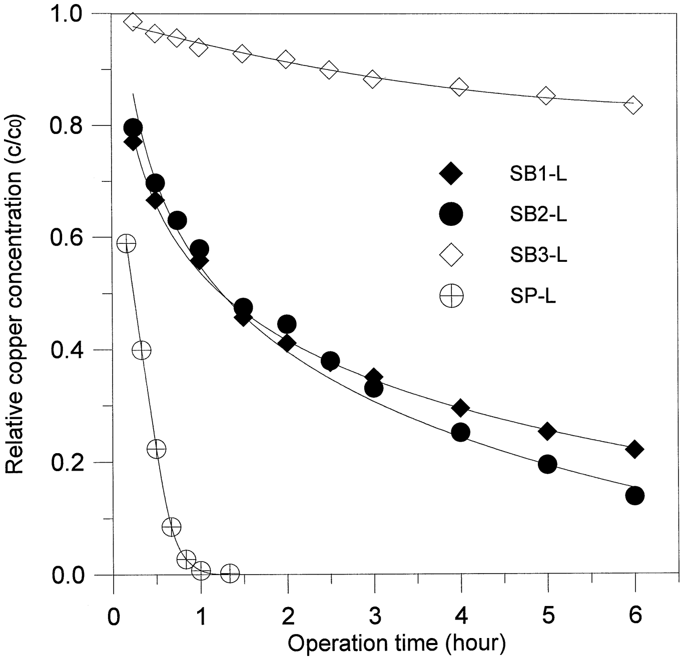


FIG. 2 Transient copper concentration change using the adsorbents: copper solution 2 mmol/L; 125 mL; pH 4.

terior of the pores, and the intrinsic reaction of metal ions with surface molecules. The rate of removal of metal in solvent impregnated resins is generally controlled by the mass transfer rate in the liquid film or inside the bead (2). The rate of removal of metal by the adsorbents prepared in this study would be expected to be controlled by film diffusion or diffusion inside the bead based on the relatively slow metal removal rate.

The possibility of film diffusion as the rate-controlling step was examined by measuring copper adsorption at different flow rates. A 2 mmol/L copper solution (125 mL) was fed to the reactor packed with 2.5 g of the SB2-L at a rate of 5, 20, and 100 mL/min. The corresponding space velocity values are 5.26, 21.0, and 105 cm/min, respectively. As shown in Fig 3, no significant effect of solution velocity on the copper adsorption rate is evident. This result suggests that film diffusion may not be the rate-controlling step (20, 21).

The interruption test (20, 21) was carried out to investigate the possibility that pore diffusion might be the rate-controlling step. A 1 mmol/L copper solution (100 mL) at pH 4 was fed to the reactor packed with 5 g of the SB2-L at a rate of 20 mL/min. The SB2-L was isolated for 15 minutes after being in contact with the copper solution for 1.5 hours and the solution was then fed again to the reactor. The pause provides a time interval in which the concen-

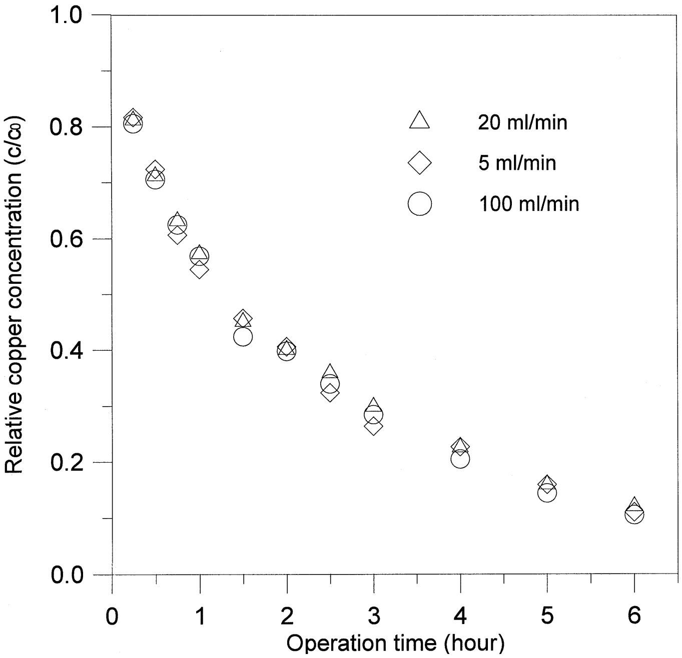


FIG. 3 Transient copper concentration change using the SB2-L with various flow rates: copper solution 2 mmol/L; 125 mL; pH 4.

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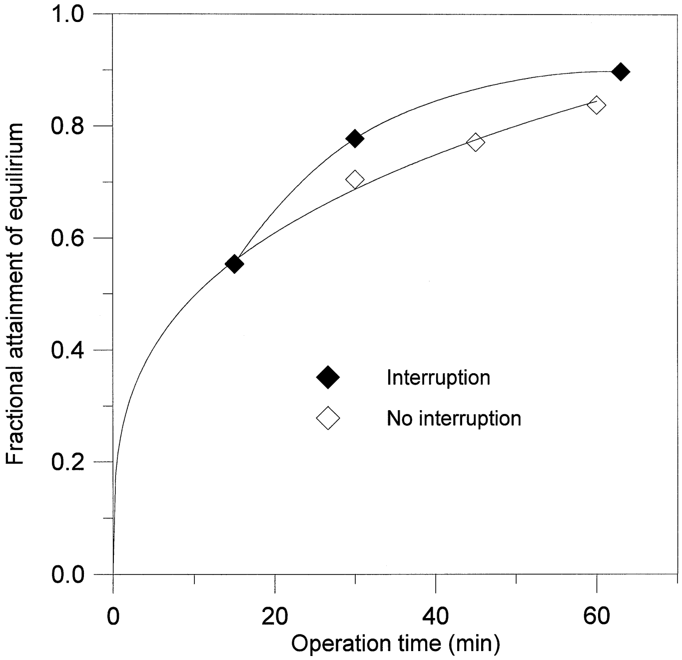

FIG. 4 Interruption test to determine the effect of particle diffusion on adsorption rate using the SB2-L: copper solution 1 mmol/L; 100 mL; pH 4, SB2-L 5 g, interrupted at 15 minutes for 1 hour and 30 minutes.

tration gradient inside the pore is decreased. Figure 4 illustrates the results of the interruption test. The fractional attainment of equilibrium is defined as the ratio of the amount adsorbed onto the adsorbent to that adsorbed at equilibrium. When the interruption was made 15 minutes from the start, the rate of adsorption after interruption was greater than that prior to the interruption. This suggests that particle diffusion is the rate-determining step for this process (20, 21).

pH Dependence of Adsorption Capacity and Rate

The reaction scheme for LIX 84 with divalent metal ions, M^{2+}, can be represented as (22)

$$M^{2+} + 2HR \rightleftharpoons MR_2 + 2H^+ \tag{2}$$

Here, HR represents the LIX 84 and MR_2 is the metal–LIX 84 complex. The above reaction is a reversible reaction wherein a cation and hydrogen ions are exchanged. Thus, the degree of extraction of metal ions depends on the pH of the aqueous solution.

The effect of solution pH on the adsorption rate of the SB2-L is shown in Fig. 5. Experiments were carried out using a packed-column reactor. The results show the transient relative copper concentration (c/c_0) change of the solution with the operation time for solution pH values of 2, 2.5, and 4, respectively. The rate of relative concentration decreased as the solution pH increased. This result suggests that the SB2-L has the characteristics of LIX 84 although the extractant is impregnated in the pores of silica beads, the surface of which was subjected to the chemical modification steps described in the Experimental section.

Stability

Stability of the SB2-L under acidic and alkaline conditions was investigated. Table 3 shows the percent of original capacity and nitrogen concentration of the SB2-L sample after treatment with acidic solutions and alkaline solutions, respectively. Contact with each of the acidic solutions (1 M HCl, 0.1 M HCl) resulted in a slight reduction in capacity. However, contacting with alkaline solutions (0.1 M NaOH, 0.01 M NaOH) caused a 13–21% loss of capacity as listed in Table 3. The results would be applied to generate the developed adsorbents by aqueous acidic solutions.

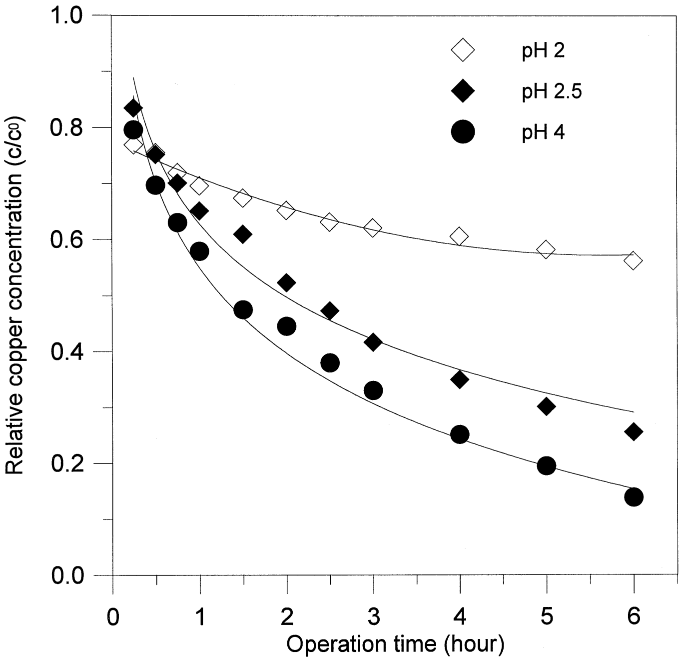

FIG. 5 Transient copper concentration change using the SB2-L at various solution pH values: copper solution 2 mmol/L; 125 mL.

TABLE 3
Stability of SB2-L

Treatment solution	Percent of original capacity[a]	Percent of original N concentration[b]
0.1 M HCl	96	96
1 M HCl	98	99
0.01 M NaOH	87	86
0.1 M NaOH	79	81

[a] Treatment time: 27 hours.
[b] Measured by CHN Corder.

Regeneration

The LIX 84–copper complex compound formed at the surface of the adsorbents can be regenerated into LIX 84 and free copper ions at low pH conditions. This is achieved by suppressing the forward reaction rate and accelerating the backward reaction rate by increasing the H^+ concentration. The results of successive copper adsorption–elution experiments for the SB2-L are illustrated in Fig. 6. The elution was very rapid for the initial 15 minutes after op-

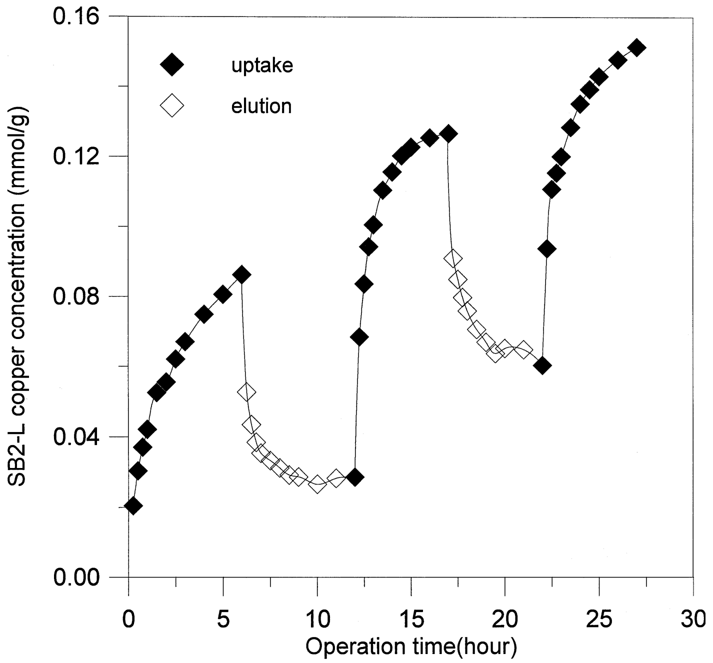

FIG. 6 Successive adsorption–elution of the SB2-L: copper solution 2 mmol/L; 125 mL; pH 4, elution solution 0.1 M HNO_3; 125 mL.

eration, after which it became slower. Desorption equilibrium was apparently reached within 4 hours under the given experimental conditions. The elution ratio is defined as the ratio of the amount eluted into the solution to that adsorbed initially in the SB2-L. The elution ratio was between 65 and 70%. For a 24-hour elution operation, 80–90% of recovery ratio was obtained in a separate experiment. Surprisingly, the rate of copper adsorption increased after successive regeneration, as shown in Fig. 6. This appears to be the result of an increase in the active surface area accessible to copper ions per unit time as the result of acid washing. Washing of the adsorbents with acid solution, however, results in a reduction in LIX 84 content in the adsorbents. Further study will be required to investigate the characteristics and morphological change in the silica support after treatment with the acid solution.

Breakthrough Curve

The breakthrough experiment was carried out for the SP-L using a packed-column reactor (ϕ 16 mm). Figure 7 illustrates the breakthrough curve for copper ions using the SP-L. As shown in the figure, about 150 bed volumes of feed solution can be passed through at a rate of 5 mL/min without leakage of copper ions into the effluent. When the copper concentration in the effluent was increased up to the saturation point (after about 340 bed volumes), a ca-

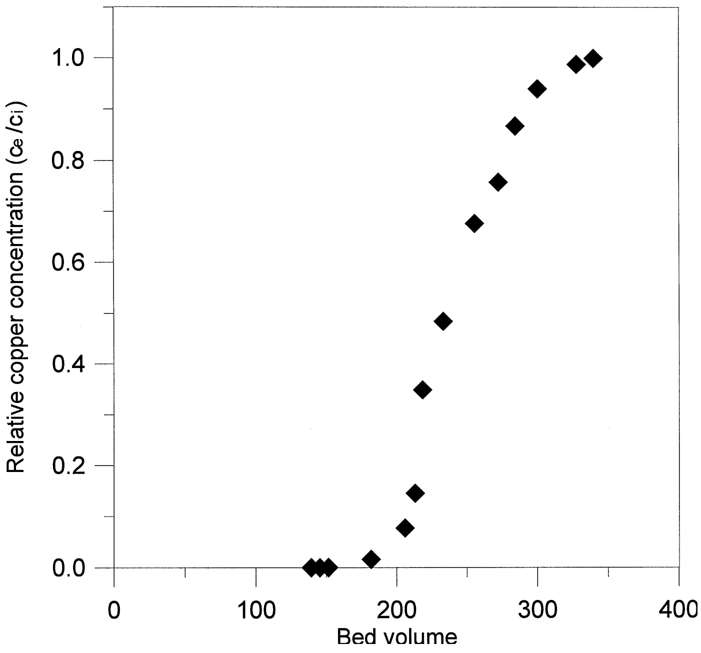

FIG. 7 Breakthrough curve for copper ions using the SP-L: copper solution 1 mmol/L; pH 3.7, flow rate 5 mL/min, SP-L 20 g, bed 1.6 × 16.4 cm.

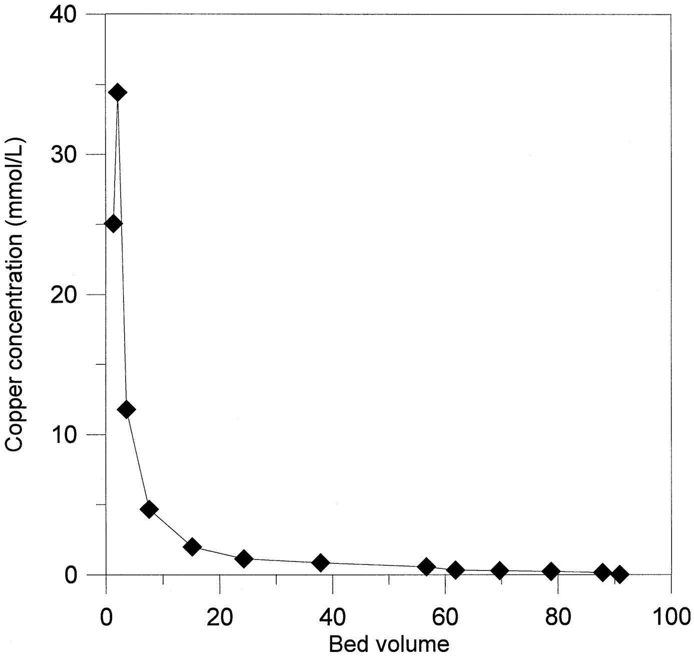

FIG. 8 Elution for copper ions using the SP-L: elution solution 0.1 M HNO_3, flow rate 5 mL/min, SP-L 20 g, initial copper loading 0.401 mmol/g, bed 1.6 × 16.4 cm.

pacity of 0.401 mmol/g was achieved. This value is 85% of the theoretical capacity based on the amount of LIX 84 impregnated in the SP-L adsorbent.

The elution of copper ions from the SP-L sample after using the breakthrough curve experiment was carried out. A solution of 0.1 M HNO_3 was passed through the bed at a rate of 5 mL/min. As shown in Fig. 8, the copper concentration of the effluent increased and then decreased dramatically within about 20 bed volumes. This result indicates that the elution rate is faster than that of adsorption. Seventy-four percent of the copper ions were eluted in up to 92 bed volumes.

The breakthrough and elution studies show that the SP-L can be used for the removal and recovery of copper ions using a once-through fixed-bed operation.

CONCLUSIONS

LIX 84 was immobilized on the surface of silica supports after surface modification. The resulting adsorbents have the potential to extract copper ions from aqueous solutions.

The copper adsorption capacity depends on the pore structure of the support. The use of a support with a defined pore size is necessary in order to maximize the capacity for copper adsorption. Copper removal capacity strongly depends on the solution pH. Adsorbed copper ions can be recovered by treatment with an acidic solution. This also allows for cyclic use after regeneration.

The adsorption rate of the developed adsorbent proved to be controlled by the pore diffusion process, among others, since particle size affects the adsorption rate. Experimental results showed that the powder-type adsorbent (SP-L) has a high adsorption rate due to small particle size.

A combination of silica and the other organic extractants by proper attachment techniques would be expected to be useful and practical in separating specific metal ions. This technique has potential as an alternative technology over such conventional heavy metal separation applications as wastewater treatment in the electronics and electroplating industries.

SYMBOLS

c	copper ion concentration in the aqueous phase (mol/L)
c_0	initial copper ion concentration of the aqueous phase (mol/L)
c_e	copper ion concentration in the effluent (mol/L)
c_i	copper ion concentration in the feed (mol/L)
HR	LIX 84
LIX 84	2-hydroxy-5-nonylacetophenoneoxime
M	metal
MR_2	metal-LIX 84 complex

ACKNOWLEDGMENT

This work was supported by Grant KOSEF 961-0100-001-2 from the Korea Science and Engineering Foundation.

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Received by editor July 8, 1998
First revision received December 1998
Second revision received February 1999

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