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# Separation of Cu(II) and Mo(VI) from Mine Waters Using Two Microporous Membrane Extraction Systems

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## ABSTRACT

This is a report on the separation and recovery of Cu(II) and Mo(VI) ions from two Chilean mine waters using two hollow fiber-type microporous liquid membrane extraction systems. LIX-860 (a salicylaldoxime) and Alamine 336 (a long-chain tertiary amine) were used as carrier extractants for copper and molybdenum, respectively. The measurements of the permeation of these metallic ions through the liquid membrane indicate that the selective transport of Cu(II) produced in respect to iron, arsenic, and aluminum in the experiments resulted from a sulfuric acid leach residual solution. The copper permeation is enhanced at pH 2.8 and when the concentration of LIX-860 in the organic phase was increased. Molybdenum and rhenium were efficiently separated from copper and iron metals, which were obtained from a nitric acid leach residual solution, by a liquid membrane prepared using Alamine 336 as the carrier compound. The membrane extractor consisted of two coupled reactors (one for extraction and one for stripping). Circulation of the organic solution between the modules gave a quite good performance for the extraction step and a deficient behavior in the stripping stage. This problem was resolved by using an extraction system which operates with only one module and by impregnating the pores of the hollow fibers used as the solid support with the organic solution. The feed and strip solutions are circulated on opposite sides of the fibers. The experimental results indicated the feasibility of separation and recovery of metals from diluted mine waters by using this membrane technology.

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## INTRODUCTION

The employment of liquid membranes technology for the separation and enrichment of metallic ions must be considered as one of the most interesting technological advances produced in the field of hydrometallurgical processes (1–3). One of its characteristics is the ability to transfer and concentrate small contents of metals present in aqueous solutions into another acceptor solution by means of an organic solvent containing small amounts of selective extractants used as metal carriers (4, 5).

Two types of liquid membranes are widely known: supported liquid membranes and liquid surfactant membranes. Both of them present potential practical applications including gas separation, nuclear waste processing, removal of heavy metals from industrial wastewaters, and hydrometallurgical recovery of metals (6, 7).

The application of a membrane extractor made of hollow fibers as a microporous solid support has been particularly successfully when used for the recovery of metals from dilute aqueous solutions, making it possible to predict that this technology will be competitive with other separation and recovery processes such as ion exchange and conventional solvent extraction using mixer-settler extractors (8, 9).

It has been well documented that this hollow fibers-type solid supported liquid membrane presents the best performance. Because of its higher fiber packing density, it gives a higher metal transport area and better flow ability due to its improved hydrodynamics conditions. Comparing this type of extractor with a current solvent extraction mixer-settler, it is evident that this membrane reactor presents important advantages. Among them are huge savings in the inventory of expensive solvents, smaller reactor size, no direct mixing between aqueous and organic phases, the avoidance of crud formation, and the elimination phase entrainment difficulties. In recent years substantial progress has been made and reported by many investigators in the recovery of metals by this kind of membrane extractor (10–13). Special interest has been dedicated to explain the selective transport of each species through the liquid membrane porous wall and the effect of the flow velocity on the mass-transfer coefficients (14, 15). However, most of these studies are concerned with metal extraction from synthetic solutions prepared in the laboratory.

We have attempted different approaches in order to clarify the metal transfer mechanism, including a heterogeneous interfacial reaction model which considers interfacial adsorption of an extractant at the interface between the phases (16–18). In another study we have explained the experimental results of copper extraction by a diffusion model which predicts that the extraction chemical reaction would occur at the interface of the liquid membrane (19). The right choices of extractants and stripping agents were found to be the key factor in determing an effective system for the recovery of metals (20, 21).

In this communication we report the selective separation and recovery of copper(II) and molybdenum(VI) ions from two different solutions of Chilean mining activities, using two liquid membrane extractor systems which use hollow fibers as the solid support.

Cu(II) ions are recovered from a solution that originates in an acid leach step of Chilean porphyry oxide-type copper ores with $H_2SO_4$. This solution contains variable but significant concentrations of copper and low contents of iron, aluminum, arsenic, and other metals. Mo(VI) ions were selectively separated from an acid leach residual solution of Chilean molybdenite concentrate with nitric acid produced "in situ" by the reaction of sodium nitrate and sulfuric acid. This waste solution contains molybdenum and copper—the main impurity of $MoS_2$ concentrates—and minor contents of rhenium and other nonvaluable metals.

One of the membrane extractor systems consisted of two coupled reactor modules (one for extraction and one for stripping) which works by recycling an inventory of organic phases between reactors. The other was composed of only one module that works by impregnating the porous structure of fibers with the organic solvent containing selective carrier extractants from valuable metals. The aim of the present study is to selectively extract most of both metals present in these solutions by means of a hollow fiber-type liquid membrane extractor. The final objective of this research is to develop a practical membrane-based solvent extraction process capable of achieving selective separation and enrichment of metals from dilute mining solutions. The information obtained during this study will contribute to the design of a definitive extractor module.

## EXPERIMENTAL

### Materials

The organic solutions were prepared by dissolving 5-dodecylsalicylaldoxime (LIX-860) and tri-($C_8$–$C_{10}$) amine (Alamine 336), a tertiary amine from Chile Harting Chemical Co., in *n*-hexane of commercial GR Grade. Both compounds were used as the organic carriers of the liquid membrane without further purification from the standpoint of industrial application. A solution generated in the acid leach step of Chilean oxidized copper ores was used as feed to the copper-donor phase. It is an acidic solution whose pH averages 2.5–3.0 with a density of 1.06 g/mL at 30°C. The initial concentrations of metals in the leach solution used in this study were 1.35 g/L Cu(II), 0.4 g/L Fe, 0.32 g/L As, 0.25 g/L Al(III), and minor quantities of other metals.

An acid leach residual solution of $MoS_2$ concentrate with nitric acid, provided by El Salvador Division of Copper Corporation of Chile (CODELCO), was utilized as the feed molybdenum-donor solution. The residual solution

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employed in these experiments included 1.0 g/L of molybdenum, 0.4 g/L of copper, 0.4 g/L of iron, 0.1 g/L of rhenium, and minor concentrations of other nonvaluable metals. It is a strong acidic solution whose pH averages 0.0–1.0 with a density of 1.05 g/mL at 20°C. The high ionic strength of this solution was kept constant during all the experiments.

Sulfuric acid and ammonia solutions were used as stripping agents. All other chemicals were of an analytical grade.

Both membrane extractor systems were built with reactors comprised of a glass shell and three hollow fibers made of poly(tetrafluoroethylene). The fibers were supplied by Japan Goretex Co. The inner and outer diameters of the hollow fibers used were 1.00 and 1.80 mm, respectively. The effective length of the fiber was 0.12 m; the maximum membrane pore size was 2.0 μm; and the porosity factor was 0.60. The inner diameter of the extractor glass tube was 15 mm.

## Procedure

In the extraction system that operates with two reactors modules, the experimental procedure was as follows: The aqueous feed solutions were fed into the extraction module through the inner side of the fibers and the organic solution was fed through the outer side cocurrently. In the backextraction module the stripping solution was circulated through the inner side of the fibers and the loaded organic solvent came from the extraction module by the outer side in a cocurrent way as was described in a previous paper (22).

When the one-extractor-module system was utilized, the following procedure was carried out: Using Masterflex microtube pump, the organic solution was circulated through the fibers in order to impregnate the porous structure of the solid support. After impregnation of the fibers, the feed solution and strip aqueous phases were fed cocurrently along the outer and inner sides of the hollow fibers, respectively, during 2–3 hours.

Membrane modules were equipped with a water jacket for circulation of water to carry out the experiments at a constant temperature (30°C). Due to the hydrophobic character of the polymeric fibers, the porous structure of the membranes is rapidly filled with the solvent containing the “carrier” extractants. Appropriate pressures at both side of membrane (fibers) were maintained in order to avoid entrainment of organic solvent toward the aqueous solutions. The volumetric flow rates of the solutions were about $1.5 \times 10^{-8}$ $m^3/s$.

Most metallic ions concentrations in the feed and effluent solutions were determined by atomic absorption spectrophotometry in a GBC-902 apparatus. The molybdenum content was measured by a UNICAM UV3 model UV/VIS spectrometer using a 477.0 nm wavelength with a detection limit of 1.0

µg/mL. The pH was measured by the standard procedure using a glass electrode (Corning-320).

## RESULTS AND DISCUSSION

Experimental results obtained in liquid–liquid extraction studies for the same kind of solutions constitute the basis of the analysis of membrane transport and separation. Consequently, LIX-860 and Alamine 336 were chosen as the selective carriers to extract copper and molybdenum ions from sulfuric acid and nitric acid residual leach solutions, respectively. Both organic reagents have been effectively used for the separation and recovery of these metals by conventional solvent extraction (23, 24).

By taking into account such results of the liquid–liquid distribution of Cu(II) and Mo(VI), the corresponding membrane phases were implemented. However, it is possible that a different behavior could be observed in a liquid membrane system due to the dynamic chacaracteristics of the membrane transport under different permeation conditions. As described in previous studies, the process of metal permeation through a liquid membrane can occur by a coupled transport of metal species by a selective carrier extractant from a feed solution (leach residual solution) toward a product solution constituted by an appropriate metal-receiving solution.

In solid support liquid membranes the membranes are made by dispersing or impregnating the extractant within an inert solid support. In this study the extractants were absorbed into the matrix of the hollow fiber used as the support. The process proceeds by diffusion of metal ions from the bulk feed solution to the wall of fiber, the phase boundary. There, the metal reacts chemically with the extractant to form a compound. Then this species diffuses through the liquid membrane to the opposite wall of the fiber, and from there to the organic bulk solution in the two modules-extraction system or directly to the strip liquor if the only one module-system is being employed, where the reverse reaction takes place.

Several experimental runs were made in order to study the selective transport of metals through the hollow fiber-type membrane using LIX-860 and Alamine 336 as carriers. These experiments were carried out using the extraction system composed of two modules with the recycling of organic solvent between them. Figure 1 illustrates the results of the selective transport of metals to the organic phase with time in experiments carried out with the sulfuric acid leach solutions. Figure 2 shows the transport of different metallic ions existing in the nitric acid leach solution toward the organic solvent when Alamine 336 is used as the carrier. This figure presents the change of metal concentration (%) measured at raffinates with time. These experiments were carried out under a recycling mode, allowing the feed and the strip liquor to

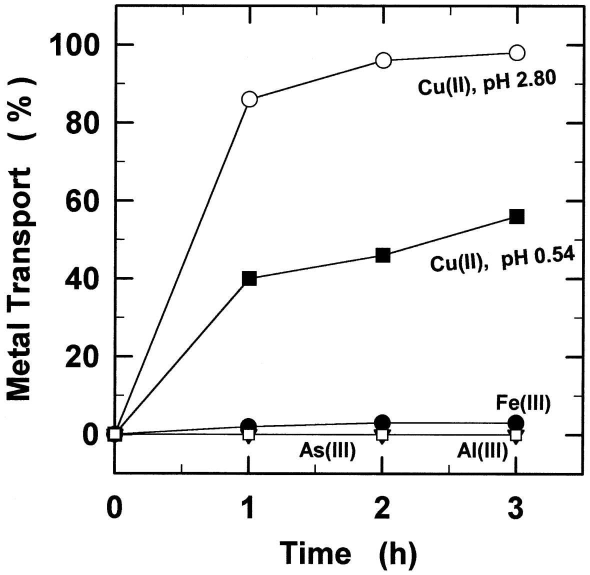

FIG. 1 Change of metals transport extent with time. Feed solution: 1.35 g/L Cu(II), 0.40 g/LFe(III), 0.32 g/L As(III), 0.25 g/L Al(III). Organic solvent: 4% (v/v) LIX-860 in *n*-hexane.

flow continuously through the extractor system in order to determine the extent of metal extraction.

The average transport percentage of ions, %*T*, is given by

$$\%T = \{1 - ([\mathrm{Me}]_{\mathrm{out}}/[\mathrm{Me}]_{\mathrm{in}})\} \qquad (1)$$

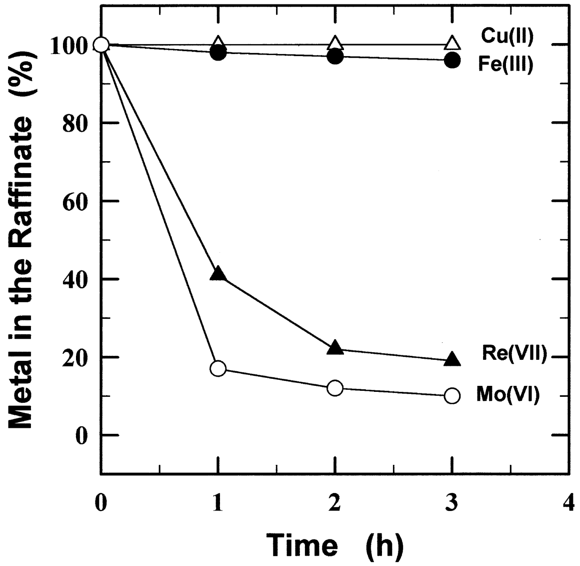

FIG. 2 Variation of metals concentration (%) in the raffinates with time. Feed solution: 1.00 g/L Mo(VI), 0.40 g/L Cu(II), 0.40 g/L Fe(III), 0.10 g/L Re(VII), pH 1.90. Organic solution: 10% (v/v) Alamine 336 in *n*-hexane.

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where $[\mathrm{Me}]_{\mathrm{out}}$ and $[\mathrm{Me}]_{\mathrm{in}}$ are the metal concentration of the donor feed solution at the outlet (raffinate) and the inlet (feed) of the extractor, respectively.

In Figure 1, transport (%*T*) enhancement of Cu(II) with time is higher for experiments carried out at pH 2.80, probably due to the larger extractant capability of salicylaldoxime at this acidity. These experiments were conducted using an organic solvent prepared with a 4% v/v LIX-860/*n*-hexane concentration. Iron, aluminum, and arsenic were not transported through the membrane during these experiments at pH 2.8, which are consistent with extraction equilibrium studies. In Fig. 2 it is clearly observed that molybdenum and rhenium were transported from the feed toward the organic liquid membranes while copper and iron were not.

The effect of the extractant concentration used for preparing the liquid membrane on the extent of transfer to the organic phase of metals present in the sulfuric acid residual leach solution is shown in Fig. 3. The transport extent of Cu(II) was affected by increasing the extractant content in the organic solvent. Little cotransport of iron was detected; however, the other metals were not extracted at all in the range of carrier concentration examined.

The influence of the initial pH of the feed solution on the transport of metals from the nitric acid waste solution to the organic solvent is shown in Fig. 4.

It was observed that molybdenum permeation through a liquid membrane is enhanced as the pH increases. Rhenium transport reach a maximum around a

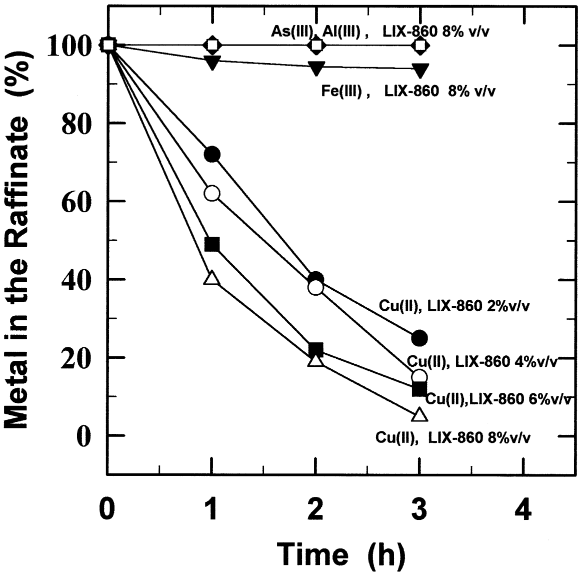

FIG. 3 Change of metals content in the raffinates with time and dependence of copper transport to organic phase on concentration of extractant LIX-860. Feed solution: 1.35 g/L Cu(II), 0.40 g/L Fe(III), 0.32 g/L As(III), 0.25 g/L Al(III), pH 2.54.

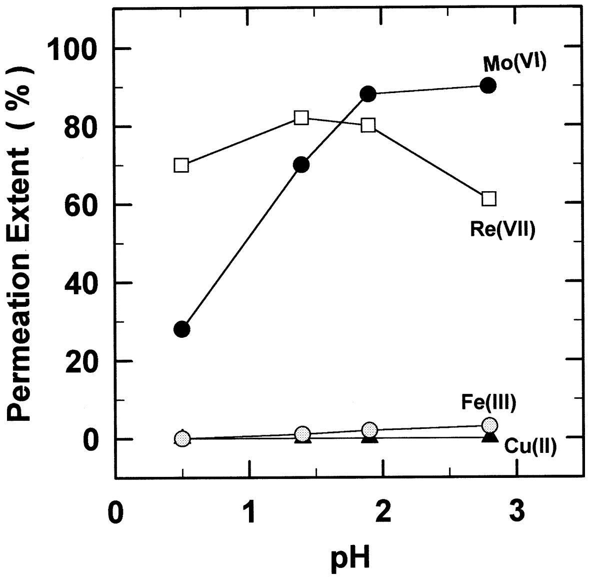

FIG. 4 Dependence of metals permeation extent on pH of feed solution. Feed solution: 1.00 g/L Mo(VI), 0.40 g/L Cu(II), 0.40 g/L Fe(III), 0.10 g/L Re(VII). Organic solution: 10% (v/v) Alamine 336 in *n*-hexane.

pH value of 1.5, while most of the copper and iron remained in the raffinate phase. These results are explained by the different chemical equilibrium-type behaviors of these metallic ions when this amine-type carrier extractant is used. Since iron precipitation start to occur around pH 3, this pH value constitutes the limit for these extraction experiments.

In spite of a fairly good performance at the extraction step for the efficient transfer of copper and molybdenum toward their respective organic liquid membrane phase, low contents of these metallic ions were measured in the respective strip liquors in these experiments. A maximum of 100 mg/L copper and 20 mg/L Mo(VI) were detected in the strip liquors under appropriate conditions taken from liquid–liquid extraction studies. This means that only a very small amount of permeated metals were backextracted to the acceptor solution employed in this stripping stage. Most of them remained unloaded from the organic solution. It would not be possible to improve the stripping stage results in this coupled-modules extractor system which works by recycling a large inventory of organic solvent between extraction and stripping reactors. A much larger membrane area in the backextraction step compared to the extraction steps is probably required.

This fact was resolved by using an extraction system which operates with only one reactor-module by impregnating the pores of hollow fibers with the organic solution in which feed and strip solution are circulated on opposite sides of the fibers.

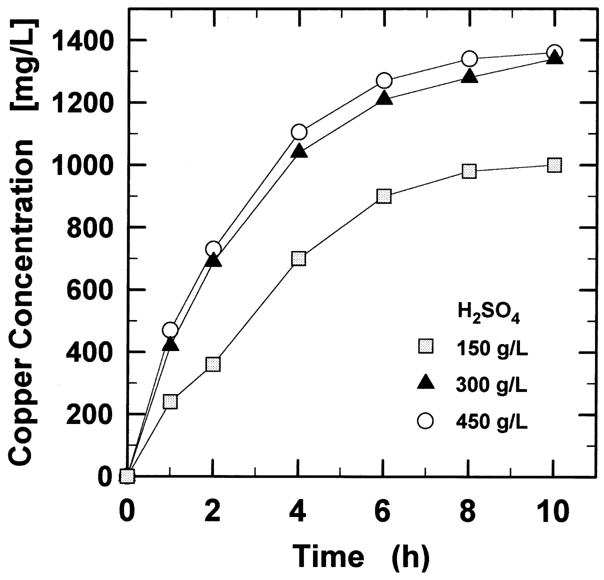

FIG. 5 Copper enrichment of strip liquor with time. Feed solution: 1.35 g/L Cu(II), pH 2.54. Organic solution: 6% (v/v) LIX-860 in *n*-hexane. (Feed volume)/(strip liquor volume) = 1.43.

Figures 5 and 6 show the results obtained when this extraction system was employed. Each extractor consisted of three fibers inside the glass-shell, with samples of feed and stripping solutions circulating in a recycling mode in order to verify the feasibility of concentrating the metals. The pH was continu-

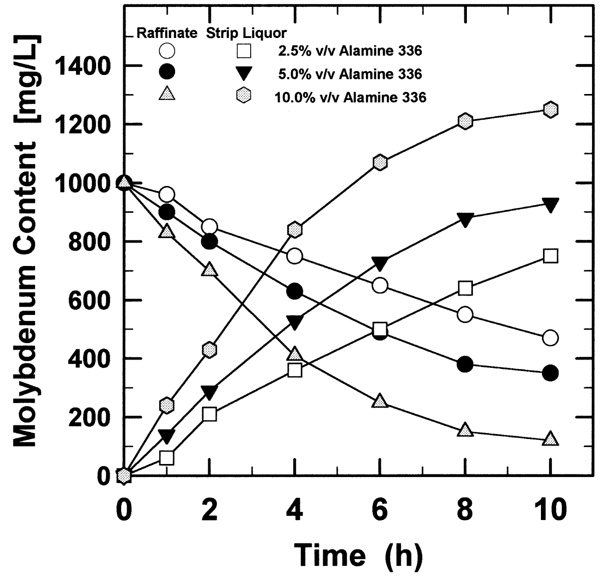

FIG. 6 The effect of concentration of extractant Alamine 336 on molybdenum transport from feed to strip liquor. Feed solution: 1.00 g/L Mo(VI), pH 1.60. Stripping solution: 2.0 mol·g/L $NH_4OH/NH_4NO_3$. (Feed volume)/(strip liquor volume) = 1.43.

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ously adjusted to a suitable value for metal recovery and especially to prevent iron precipitation when the nitric acid leach residual solution was employed. In Fig. 5 it is observed that copper enrichment of strip liquor with time is accomplished using samples of the residual solution of leaching of copper ores with sulfuric acid under different concentrations of $H_2SO_4$ in the stripping solution. It is observed that the transport of this metal to strip liquors is enhanced when the concentrations of this acid in strip solution are higher than 150 g/L. Figure 6 shows the effect of Alamine 336 in the organic solution on molybdenum transport from nitric acid leach residual solution to the strip liquor. It is observed in Fig. 6 that metal permeation to the strip liquor is favored as the content of extractant in the liquid membrane is increased.

These results are much better, under any experimental conditions, than those obtained using the extractor system made of two coupled modules which work by recycling the organic solvent between modules. An effective transport of metals toward strip liquors was achieved in this extractor which works by impregnating the porous structure of the solid support with the organic solvent. Other advantages of this extraction system are that just a small quantity of extractant is required in the organic film and that liquid–liquid separation is enhanced by combining the extraction and stripping processes in one stage.

The next steps of this study were carried out using the single reactor-system since the results clearly show that it is much more advantageous than the other one comprised of two modules.

It is clear that the cotransportation of metals depends on the right selection of a carrier extractant for each desired metal. For example, the higher extractability observed for Cu(II) with respect to iron, arsenic, and aluminum when LIX-860 was utilized as the carrier in experiments carried out with the sulfuric acid leach residual solution can be explained by the fact that only Cu(II) is able to form a chelate complex with the oximic extractant and release a hydrogen ion into the aqueous solution from which the metal was extracted. Under the pH conditions used in these experiments, only Fe(III) could be slightly extracted; arsenic and aluminum were not. From Figs. 1 and 3 it is obvious that Cu(II) is the only metallic species present in the sulfuric solution which is strongly extracted, even for higher concentrations of LIX-860 in the organic phase. These data are in agreement with the behavior observed in liquid–liquid extraction studies. LIX-860, a salicylaldoxime, is a strong copper extractant which acts by cation exchange in a chelate-type complex formation reaction but can not extract such complex metal anions as $AsO_3^{3-}$ and $AsO_4^{3-}$ (25).

Chelating extractants such as LIX-860 act by a cation-exchange mechanism in which the hydrogens of the extractants are exchanged for metal ions:

$$M_{aq}^{n+} + nHA_{org} \Leftrightarrow MA_{n\ org} + nH^{+} \tag{2}$$

where HA and $MA_n$ denote the extractant and the chelate metal complex, respectively. The results observed in Figs. 1 and 3 indicate that it is feasible to separate copper by solvent extraction on a liquid membrane from mine water by using LIX-860.

The same kind of analysis can be done on the metal transportation results obtained using the nitric residual leach solution. In Figs. 2 and 4 it is observed that molybdenum and rhenium transport is enhanced when Alamine 336 is employed as a carrier extractant. Both metals permeate through the liquid membrane and are absorbed in the polymeric matrix of hollow fibers; copper and iron are not. Alamine 336 is an ion-pair-type of extractant which acts by following a mechanism based on the principle of ion association whereby a large and positively charged organic species produces the extraction of a large and anionic metal complex toward the organic phase. The unlikely behavior in the transportation of these metallic ions can be ascribed to the fact that under the pH conditions used in this study, Mo(VI) and Re(VII) are capable of forming very stable anionic complexes ($MoO_4^{2-}$ and $ReO_4^-$) while Cu(II) and Fe(III) are not. The extraction mechanism of these metals by the tertiary amine in a $HNO_3$ aqueous solution may be explained by the following equations:

$$R_3N_{(org)} + H^+_{(aq)} + NO^-_{3(aq)} \Leftrightarrow R_3NH^+ \cdot NO^-_{3(org)} \tag{3}$$

$$2R_3NH^+NO^-_{3(org)} + MoO^{2-}_{4(aq)} \Leftrightarrow (R_3NH^+)_2MoO^{2-}_{4(org)} + 2NO^-_{3(aq)} \tag{4}$$

$$R_3NH^+NO^-_{3(org)} + ReO^-_{4(aq)} \Leftrightarrow R_3NH^+ReO^-_{4(org)} + NO^-_{3(aq)} \tag{5}$$

Equation (3) represents simple amine salt formation while Eqs. (4) and (5) represent true ion exchange. Because copper and iron ions do have not form such anionic nitro complexes, they can not undergo the anion-exchange reactions. These results support the feasibility of separating these metals with Alamine 336 by using this liquid membrane system.

Even at low pH values the Mo(VI) permeation results were quite important. Normally, cationic species of Mo(VI) as $MoO_2^{2+}$ are predominant when the pH of the solution is under 1.8 (25). However, a large extraction of this metal occurred under this highly acidic condition, which implies that a kind of cation–anion exchange equilibrium must occur since only polyanions of this metal are extractable by Alamine-type reagents. Cotransport of molybdenum and rhenium can be easily solved during the later stripping step as it has been solved in liquid–liquid extraction studies according to some authors (26).

The results of this study confirm that this technology is an interesting alternative to current conventional solvent extraction and ion-exchange processes for the recovery of valuable metals or for the removal of undesired ions, especially from dilute solutions. The application of hollow fiber-type solid supported liquid membrane technology for resolving industrial problems is highly promising.

However, further studies must be considered in order to achieve the best performance of this technology for its industrial application, including the use of carrier reagents with a higher solubility for liquid membrane preparation. The higher the solubility, the faster and larger the permeability of metals in regard to the lipophilicity of an organic extractant. It is important to adequately estimate the requirement of membrane area for every separation condition including flow rates and extent of transport. Improvements in membrane preparation and formulation are required. For instance, if a thinner film is used as the membrane, a higher flux value of metal would be obtained.

In this study we did not note the extractant loss and flux reduction during the experiments. If the feed solution contains particulates, it is absolutely necessary to clarify it by filtering in order to separate any particles which could obstruct the porous membrane.

A solid supported liquid membrane combines the high selectivity and capability to concentrate ions of solvent extraction with improved throughputs and the continuous processing ability of membrane technology. Among other advantages, the reduction of an expensive solvent inventory and the elimination of difficulties associated with phase separation such as crud formation and entrainment should be mentioned.

## CONCLUSIONS

The selective separation and recovery of copper(II) and molybdenum(VI) ions from two Chilean mine waters using a hollow fiber-type solid supported liquid membrane system has been studied. Copper(II) ions were selectively transported with respect to iron, arsenic, and aluminum through a liquid membrane prepared by using LIX-860 as the extractant carrier in experiments carried out with a sulfuric acid residual solution of a leaching process of porphyry oxidized ores. Copper transport was enhanced at pH 2.8 and by increasing the concentration of carrier extractant in the organic phase.

Molybdenum and rhenium were efficiently separated from copper and iron from a nitric acid leach residual solution using a microporous liquid membrane prepared by using Alamine 336 as the carrier extractant. Alamine 336 is an ion-pair-type extractant which acts by an anion-exchange mechanism that would explain the extractability of anionic species like $MoO_4^{2-}$ and $ReO_4^-$; copper and iron do not exist as stable anionic species in this kind of aqueous medium.

Two membrane extractor systems were employed. One of them consisted of two coupled reactors (one for extraction and one for stripping) which worked by recycling the organic phase between the reactors. It had a quite good performance in the extraction step and a deficient yield in the stripping stage. A better extractor system which operates with only one module was tested. The results showed a good performance with a minimum inventory of expensive organic solvents.

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This study shows the feasibility of the separation and recovery of valuable metals from dilute mine solutions in an extractor system based on hollow fiber-type solid supported liquid membrane.

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