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Highly Selective and Efficient Membrane Transport of Molybdenum Using Di(2-Ethylhexyl) Phosphoric Acid as Carrier

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ABSTRACT

Di(2-ethylhexyl) phosphoric acid (D2EHPA) was used as a highly efficient carrier for the transport of molybdenum ions as $MoO_2(D2EHPA)_4$ complex through a carbon tetrachloride bulk liquid membrane. By using H_2O_2 as a metal ion acceptor in the receiving phase at the optimum HNO_3 concentration of 0.5 M, the amount of molybdenum transported across the liquid membrane after 4 hours was $99.5 \pm 0.7\%$. The selectivity and efficiency of molybdenum transport from acidic solutions containing a competing ion as well as from a multicomponent mixture were investigated. With the exception of W, the interfering effect of the diverse ions tested was negligible. However, in the presence of 0.5 M citric acid as a suitable masking agent in the source phase, the interfering effect of W(VI) ion was completely eliminated.

Key Words. Liquid membrane; Molybdenum(VI) transport; D2EHPA carrier

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INTRODUCTION

Molybdenum is an essential trace element for plant (1) and has a large number of applications. It is used in many electric and electronic parts, in steels, in lubricants for high temperature high pressure conditions, and in catalysts in dehydrogenations of complex hydrocarbon mixtures (2). Along with tungsten, it is used in thermocouples for high temperature measurements. Also molybdenum-99 is widely used in nuclear medicine (3–6). The ^{99}Mo fission product is mainly used for the preparation of the ^{99m}Tc generators. This carrier-free molybdenum renders it possible to obtain ^{99m}Tc generators of high specific activity required in medical applications. In general, the production of ^{99}Mo can be divided into two parts: 1) thermal neutron bombardment of MoO_3 according to the reaction ^{98}Mo(n,γ)^{99}Mo, and 2) separation from the fission products of irradiated enriched or natural uranium. The former offers a simple process but low specific activity of ^{99}Mo; the latter can produce a high specific activity of ^{99}Mo, though the separation process is more important. The most widely used method for the separation of molybdenum from excess uranium and fission products is column adsorption using Al_2O_3 and ZrO_2 as the adsorbent materials (7, 8). The column method with alumina is simple, relatively fast, and efficient. Its serious drawback consists in contamination of the molybdenum fraction by iodine, tellurium, etc. Because of the similar chemical behavior of the anion forms of the latter elements and molybdenum, other feasible separation method are required (9).

The most widely used methods for the separation of molybdenum from other elements are liquid–liquid extraction by using di(2-ethylhexyl) phosphoric acid (D2EHPA) in different solvents (5, 10–13), 1,10-phenanthroline in isobutyl methyl ketone (14), dibenzo-18-crown-6 in nitrobenzene (15), tri-*n*-butyl phosphate (TBP) (16), precipitation (4, 17), anionic chelating agents loaded on anion-exchange resin (18), column chromatographic separation with poly(dibenzo-18-crown-6) (19), polyurethane foam loaded with cyclic polyether (20), as well as supported liquid membranes (21–23).

Among several conventional chemical methods for metal cation separations such as precipitation, ion exchange, solid phase extraction, and liquid–liquid extraction, separation by membranes in particular have drawn maximum attention in recent years. Polymeric membranes have the inherent disadvantages of usually low transmembrane fluxes in the condensed phase and poor selectivities. On the other hand, liquid membranes generally permit relatively higher fluxes and much improved selectivities. Moreover, a liquid membrane system incorporating a nonvolatile reversible complexing agent provides much improved enhancement of transporting solutes against their concentration gradients. Better selectivity is easily achieved through the use of specific legands dissolved in an appropriate organic liquid membrane phase. In addition, they have great potential for low cost and energy saving (24–26). This type of pertraction has been experimentally successful in hydrometallurgy for the recovery and separation of metal solutes (27). Over the past two decades there has been increasing interest in the transport of metal ions across bulk, emulsion, and supported liquid membranes (24–37). There is great potential in processing dilute metal solutions for the recovery and separation of metals (38, 39). Numerous practical application of such membranes have also been envisaged for the recovery of metals from hydrometallurgic leach solutions and the removal of uranium and americium from nitric acid waste streams (40, 41).

Emulsion membrane extraction (31–33) and supported liquid membrane extraction (34–37) are the most popular techniques. In some cases the method of bulk volume membrane extraction can be used for the separation of various metal ions (28–30).

In the present paper the results of an investigation on membrane extraction of molybdenum using the bulk liquid technique with D2EHPA are shown. Different experimental conditions, e.g., the effect of D2EHPA concentration in the membrane, type and concentrations of acids in the source and receiving phases, and the hydrogen peroxide concentration for the molybdenum ion acceptor in the receiving phase, were investigated. The method has been extended to the separation of molybdenum(VI) from a large number of elements in binary as well as in multicomponent mixtures.

EXPERIMENTAL

Reagents

15-Crown-5 (15C5), dicyclohexyl-18-crown-6(DC18C6), dibenzo-18-crown-6 (DB18C6) and D2EHPA (mixture of 40–55% 2-ethylhexylphosphoric acid and 40–60% D2EHPA) were purchased from Merck Chemical Company and used as received.

Reagent-grade carbon tetrachloride (Merck) was used as the membrane organic solvent. All other chemicals used in this study were of the highest purity available from either Merck or Aldrich chemical companies and used without further purification. Doubly distilled water was used throughout.

Procedure

All transport experiments were carried out at ambient temperature. A cylindrical glass (inside diameter of 4.0 cm) containing a glass tube (inside diameter of 2.0 cm), and thus separating the two aqueous phases, was used. The inner aqueous phase or source phase (SP) contained sodium molybdate, and nitric acid (5 mL). The outer aqueous phase (receiving phase) contained hydrogen peroxide and nitric acid (10 mL). The carbon tetrachloride solution (15 mL) containing carrier ligand lay below these aqueous phases and bridged the two phases.

The organic layer was slowly stirred (200 rpm) by a Teflon-coated magnetic bar. Determination of the metal ion concentration in both aqueous phases was carried out by ICP. A similar experiment was carried out in the absence of the carrier for reference. The detailed experimental conditions are included in the table and figures. The inductively coupled plasma (ICP) used for the measurement of metal ions concentrations was a Varian Liberty 150AX Turbo instrument. A bulk-type liquid membrane cell (24) was used.

RESULTS AND DISCUSSION

Molybdenum(VI) forms various species depending on conditions in the aqueous solutions. In high acidic solutions a mono- or divalent cation (MoO_2^{2+}) is present (21). Based on the observations of Kolarik (42), the extraction reaction of MoO_2^{2+} with D2EHPA can be represented as follows:

$$MoO_2^{2+}(aq) + 2(HR)_{2(org)} \rightleftharpoons MoO_2R_2{\cdot}2HR_{(org)} + 2H^+$$

where the subscripts (aq) and (org) represent the aqueous and organic phases, respectively, and HR represents D2EHPA. On the other hand, since hydrogen peroxide forms a very strong complex with molybdenum, $H_2MoO_2(O_2)_2$ (43), the molybdenum is removed from the organic phase as follows:

$$MoO_2R_2{\cdot}2HR_{(org)} + 2H_2O_{2(aq)} \rightleftharpoons H_2MoO_2(O_2)_{2(aq)} + 2(HR)_{2(org)}$$

In the present work a liquid bulk membrane containing D2EHPA in CCl_4 was contacted with a highly acidic solution initially containing the metal ion (SP) and a receiving phase (RP) containing H_2O_2 as a stripping agent. It should be noted that the cell design is such that the larger volume port could be selected for either the SP or the RP, depending on the purpose of the separation process. Since the aim of this work was to increase the rate of separation of molybdenum from the accompanying elements, the larger volume (i.e., 10 mL) was chosen as the receiving phase.

The effect of the concentration of different acids in the source phase on the transport of molybdenum was studied (Fig. 1). It is seen that the percentage of molybdenum ion transported increases with an increasing concentration of acids, and maximum transport occurs in the presence of 8 M $HClO_4$ or 6 M HNO_3. However, a 8.0 M $HClO_4$ solution possesses a high density, so that some mixing of SP and RP will occur; meanwhile, tungstenum interferes with Mo transport. Therefore, the 6 M HNO_3 solution was chosen for further studies. It has been reported in the liquid–liquid extraction of molybdenum by D2EHPA (5, 10, 11) that an increased concentration of acid in the source phase will increase the MoO_2^{2+}, which is expected to reach a plateau at a given acid concentration.

The transport efficiency of molybdenum was found to be critically dependent on the type of ligand used as the carrier in the organic phase. Among the different ligands used (15C5, DC18C6, DB18C6, and D2EHPA), only D2EHPA exhibited the highest transport efficiency for the transport of molybdenum as $MoO_2R_2{\cdot}2HR$.

The influence of the concentration of D2EHPA in the organic phase on the transport efficiency of molybdenum was also studied (Fig. 2). As shown,

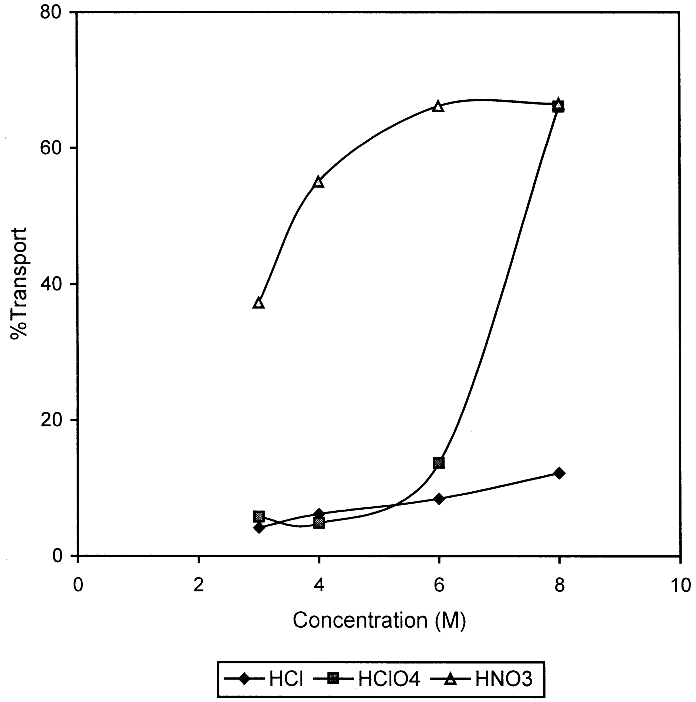

FIG. 1 Effect of acid concentration in the source phase on molybdenum ion transport into the RP. Conditions: Source phase, 5 mL of 10^{-4} mol/L Mo(IV) and varying concentrations of different acids; membrane phase, 15 mL of 0.02 mol/L of D2EHPA in CCl_4; receiving phase, 10 mL of 0.2 M H_2O_2 in 0.1 M HNO_3; time of transport, 2 hours.

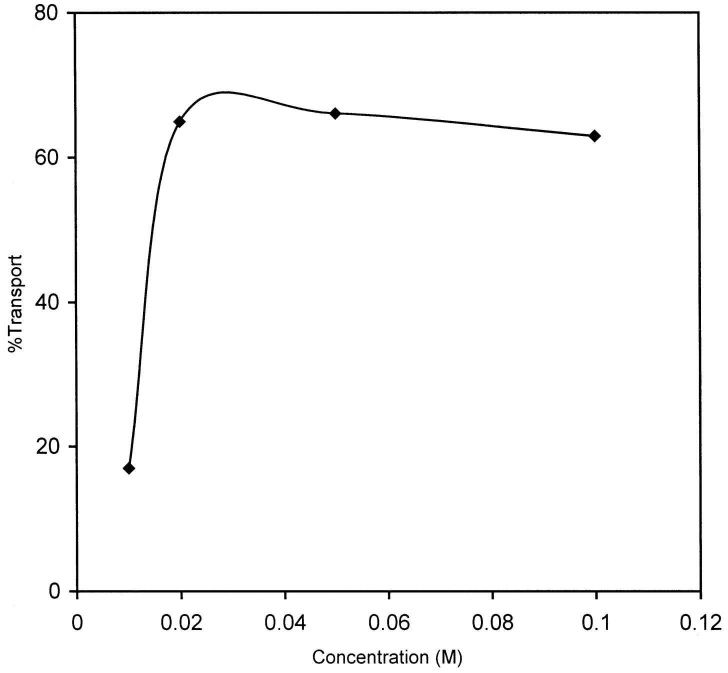

FIG. 2 Effect of D2EHPA concentration in the membrane phase on molybdenum ion transport into the RP. Conditions: Source phase, 5 mL of 10^{-4} mol/L Mo(IV) and 6 mol/L HNO_3; membrane phase, 15 mL of varying concentration of D2EHPA in CCl_4; receiving phase, 10 mL of 0.2 M H_2O_2 in 0.1 M HNO_3; time of transport, 2 hours.

the percentage transport of molybdenum increases with increasing D2EHPA concentration in CCl_4. Maximum transport occurs at a concentration above 0.02 M, and additional ligand has no significant effect on transport efficiency. Further transport studies of molybdenum were carried out with 0.05 M D2EHPA. It has been supposed that the amount of $MoO_2R_2{\cdot}2HR$ in the liquid membrane would increase by increasing the concentration of D2EHPA in the membrane until a concentration of 0.02 M was reached. However, Fig. 2 shows that addition of an excess amount of the carrier (up to 0.1 M) will not change the percent transport of molybdenum. This is most probably due to the fixed stochiometry of resultant 4:1 carrier–Mo complex ($MoR_2{\cdot}2HR$) as well as the more or less constant viscosity of the membrane phase at this concentration range.

Permeability of the membrane for $MoO_2R_2{\cdot}2HR$ was found to be strongly dependent on the nature of the stripping agent for molybdenum ion in the receiving phase. Among SCN^-, $P_2O_7^{3-}$, PO_4^{3-}, and H_2O_2, the results show that H_2O_2 is the best stripping agent for molybdenum. The optimum concentration of H_2O_2 in the receiving phase was investigated (Fig. 3) and found to be 0.2 M.

The transport efficiency of molybdenum was also found to be dependent on the concentration of HNO_3 in the RP (Fig. 4). Maximum transport occurs at

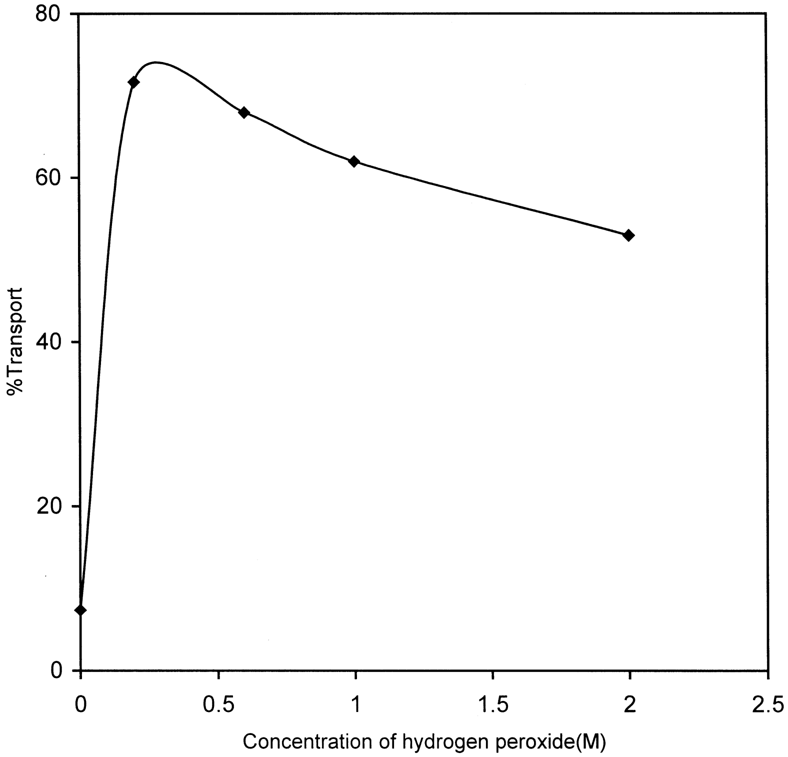

FIG. 3 Effect of H_2O_2 concentration in the receiving phase on molybdenum ion transported into the RP. Conditions: Source phase, 5 mL of 10^{-4} mol/L Mo(IV) and 6 mol/L HNO_3; membrane phase, 15 mL of 0.05 mol/L of D2EHPA in CCl_4; receiving phase, 10 mL of 0.5 M HNO_3 and varying concentration of H_2O_2; time of transport, 2 hours.

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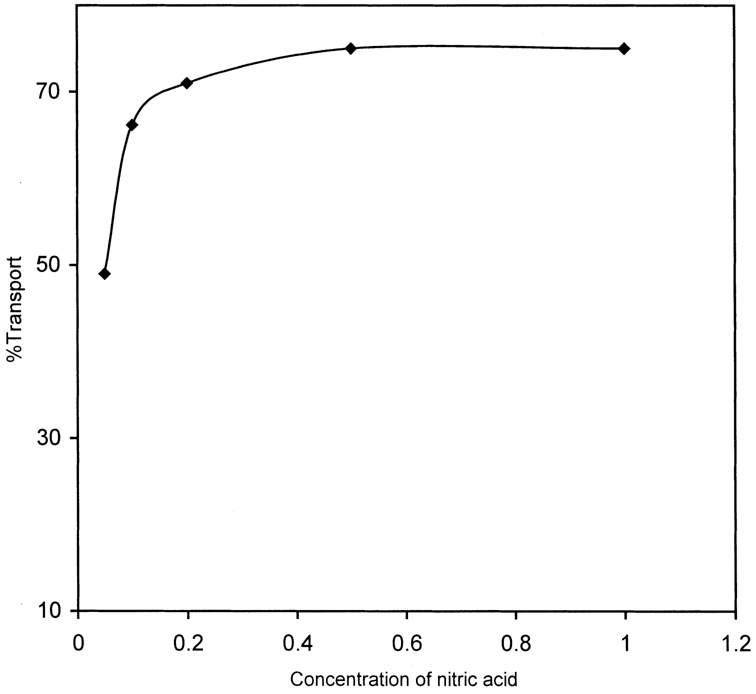


FIG. 4 Effect of HNO_3 concentration in the receiving phase on molybdenum ion transport into the RP. Conditions: Source phase, 5 mL of 10^{-4} mol/L Mo(IV) and 6 mol/L HNO_3; membrane phase, 15 mL of 0.05 mol/L of D2EHPA in CCl_4; receiving phase, 10 mL of 0.2 M H_2O_2 and varying concentration of HNO_3; time of transport, 2 hours.

HNO_3 concentrations above 0.5 M, thus further transport studies of molybdenum were carried out with 0.5 M HNO_3. It is obvious from the proposed mechanism that an increasing concentration of HNO_3 in the RP is expected to facilitate proton transfer from H_2O_2 to R^- to form neutral RH for the completion of the transport cycle.

Figure 5 shows the time dependence of molybdenum transport through the liquid membrane under the optimal experimental conditions. It is seen that complete molybdenum transport occurs after 4 hours. The reproducibility of molybdenum transport was investigated, and the percent of metal ion transport after 4 hours from three replicate measurements was found to be 99.5 ± 0.7%. The transport efficiency of 99.5% obtained here is equivalent to a flux of $J = 3.2 \times 10^{-8}$ mol/m²/s.

For investigation of the selectivity of the membrane system for the transport of molybdenum, an aliquot of a solution containing molybdenum and the competing ion to be tested was taken and HNO_3 was added so that its concentration was 6 M in a total volume of 5 mL. The concentration of molybdenum and the competing ion were determined in the RP after 6 hours (Table 1). As seen among the different ions tried, Ti and W were transported 1.6 and 12.2%, respectively, and the transport of all the other ions was less than 1%. This is most probably due to similarities of the properties of Mo and W in many re-

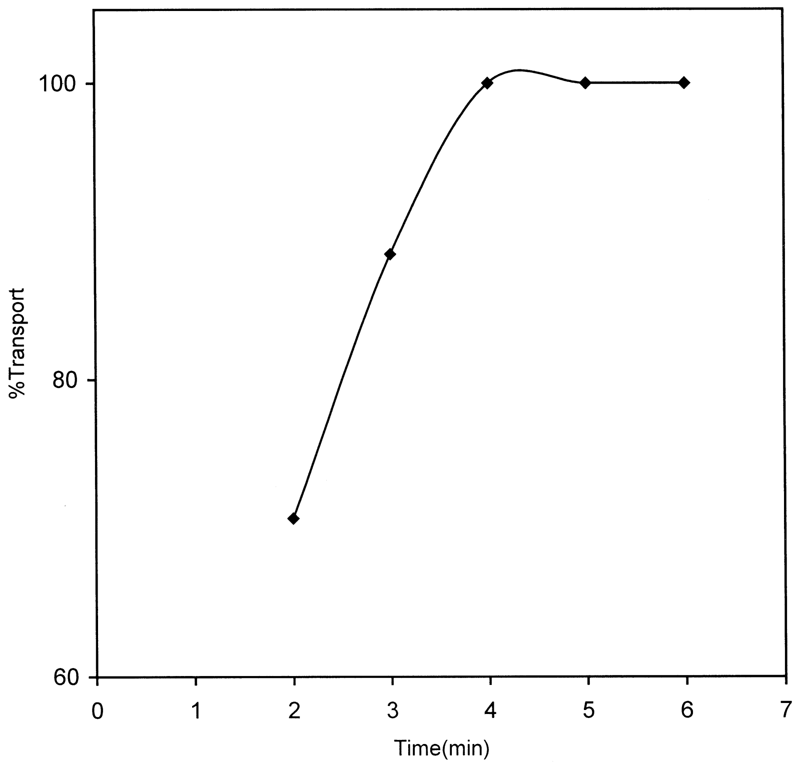


FIG. 5 Time dependence of molybdenum ion transport into the RP. Conditions: Source phase, 5 mL of 10^{-4} mol/L Mo(IV) and 6 mol/L HNO_3; membrane phase, 15 mL of 0.05 mol/L of D2EHPA in CCl_4; receiving phase, 10 mL of 0.2 M H_2O_2 and 0.5 M of HNO_3.

TABLE 1
Amount of Ion Transported from Mixtures[a] of Molybdenum and a Competing Ion through a Membrane

Ion	Percentage transport into receiving phase	Ion	Percentage transport into receiving phase
Ag^{+}[b]	0.4	K^{+}	0.2
Al^{3+}	0.3	Li^{+}	0.2
B	0.6	Mg^{2+}	0.2
Ba^{2+}	<0.1	Mg^{2+}	0.2
Bi^{3+}	0.1	Na^{+}	0.4
Ca^{2+}	0.5	Ni^{2+}	0.2
Cd^{2+}	<0.1	Pb^{2+}	0.1
Co^{3+}	0.2	Sr^{2+}	0.0
Cr^{3+}	0.2	Te(IV)	0.0
Cu^{2+}	0.3	Ti^{3+}	1.5
Fe^{3+}	0.1	W(VI)	12.1
Ga^{3+}	0.3	Zn^{2+}	0.4
In^{3+}	0.1	U(VI)	0.1

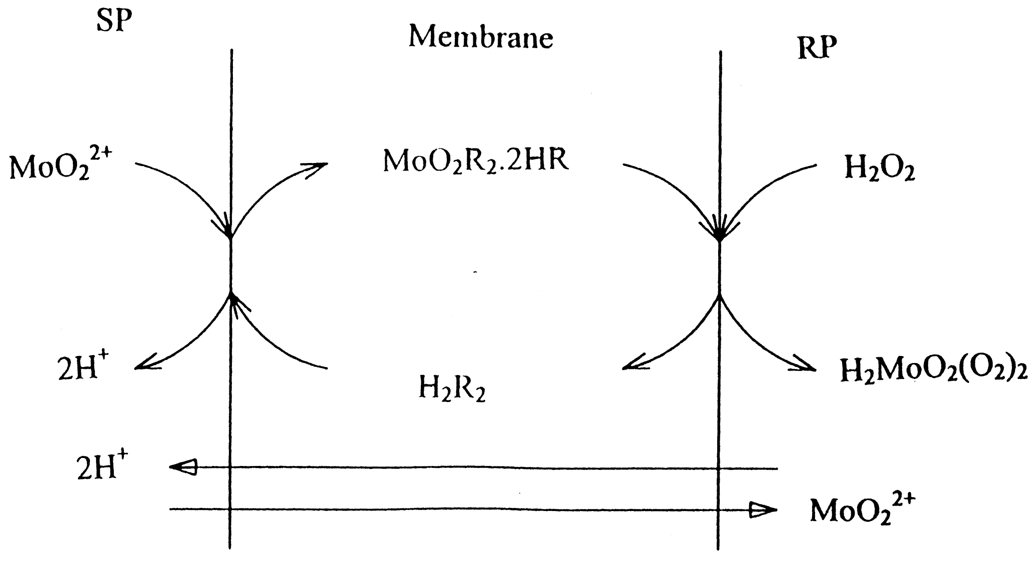
[a] Conditions: Source phase, 5 mL of 10^{-4} mol/L Mo(IV), 4×10^{-2} mol/L foreign ion, and 6 mol/L HNO_3; membrane phase, 15 mL of 0.05 mol/L of D2EHPA in CCl_4; receiving phase, 10 mL of 0.2 M H_2O_2 in 0.5 M HNO_3; time of transport, 6 hours.

[b] Molybdenum ion was quantitatively transported through a membrane in all mixtures.

spects (43). However, the interfering effect of these ions was successfully eliminated using 0.5 M citric acid as a masking agent in the RP, as reported elsewhere (44). To investigate the applicability of the proposed method for the separation of molybdenum from complex matrices, the method was applied to the separation of molybdenum from a solution containing 27 ions at 400 ppm concentrations (data not shown). The results showed that quantitative separation of molybdenum from competing ions will occur during the time period less than 5 hours.

Based on the results obtained in this study and those reported in the literature by different researchers (5, 10, 11, 42) the mechanism summarized in Scheme 1 is proposed for the molybdenum transport through the liquid membrane used. The molybdenum ion transport can be explained as follows:

1. At the source phase–membrane interface, the $MoO_2R_2{\cdot}2HR$ complex is formed, and this complex distributes preferentially into the organic membrane.
2. The complex thus formed diffuses across the membrane.
3. At the membrane–receiving phase interface, H_2O_2, with its high affinity for MoO_2^{2+} ion, completes the transport by removing MoO_2^{2+} from its complex by forming a very stable complex as $H_2MoO_2(O_2)_2$ (43).
4. The free carrier absorbs H^+ to form a neutral carrier and diffuses back across the membrane to the source phase–membrane interface, where the cycle starts again.



SCHEME 1

CONCLUSION

Separation of molybdenum by using a bulk liquid membrane was examined with D2EHPA as the carrier. The maximum transport of molybdenum from the source phase (SP) to the receiving phase (RP) was done in the presence of nitric acid (6 M) in the SP and D2EHPA (0.02 M) in the liquid membrane. Various material such as SCN^-, $P_2O_7^{3-}$, PO_4^{3-}, and H_2O_2, were used as stripping agents; the best result was observed in the presence of H_2O_2 (0.2 M) in HNO_3 (0.5 M). 99.5% of the molybdenum was transported from the SP to the RP in optimized conditions after 4 hours. The method give accomplished the separation of molybdenum from excess uranium and other interfere elements.

The excellent efficiency and high degree of selectivity for molybdenum transport demonstrated by the liquid membrane system studied reveals its potential for application to the selective removal and purification of molybdenum ion from mixtures.

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