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Separation Study of Molybdenum Through an Emulsion Liquid Membrane Using Tris (2-Ethylhexyl) Phosphate as Mobile Carrier

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ABSTRACT

A study of the separation of Mo(VI) ions by transport through a tris (2-ethylhexyl) phosphate (TEHP)—sorbitan monooleate (Span-80)—toluene liquid membrane was performed with varying concentrations of HCl, TEHP, Span-80, and NaOH containing Na_2CO_3 in the feed, membrane, and strip solutions, respectively. Maximum transport was observed with 3.5 to 5.5 mol dm^{-3} HCl, 50 to 70% (v/v) TEHP in toluene, 7 to 8% (v/v) Span-80, 0.005 to 0.015 mol dm^{-3} NaOH, and 1.4 to 2.0 mol dm^{-3} Na_2CO_3. Mo(VI) was completely separated from binary mixtures containing Fe(II), Co(II), Ni(II), Mn(II), Cr(VI), Al(III), W(VI), Ti(IV),

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V(V), Sn(IV), U(VI), and Th(IV) ions with this system. Preconcentration of Mo(VI) from dilute aqueous solution is presented and transport mechanism is discussed. The method was employed for the separation and recovery of Mo(VI) from solutions of samples such as spent catalyst, low-grade molybdenite concentrate, and ferrous scrap.

Key Words: Emulsion liquid membrane; Molybdenum (VI); Tris (2-ethylhexyl) phosphate.

INTRODUCTION

Emulsion liquid membrane (ELM) is a versatile technique for the selective separation and preconcentration of metal ions,[1–4] due to its advantage of high efficiency and low expense. Separation of Mo(VI) with ELM using 2-ethylhexylphosphonic acid mono-2-ethylhexyl ester (PC-88A),[5] trioctyl amine (TOA)[6] and aliquat-336[7] as a mobile carriers are reported. Tris (2-ethylhexyl) phosphate (TEHP) has been studied as an extractant for the liquid–liquid extraction of some metal ions.[8–13] However, it has not been employed so far as a mobile carrier in ELM.

In this research, ELM with TEHP as a mobile carrier in toluene was studied for the separation of Mo(VI). Various parameters influencing the transport of Mo(VI) across the membrane were optimized to preconcentrate and separate it from binary mixtures containing various associated metal ions. The transport mechanism is discussed and applications of this method for the recovery of molybdenum from solutions of spent catalyst, low-grade molybdenite concentrate, and ferrous scrap were studied.

EXPERIMENTAL

Instrumentation

A motor driven emulsifier (range 0 to 6000 rpm), magnetic stirrer (range 0 to 600 rpm), and a spectronic 20-D spectrophotometer (Milton Roy & Co.) were used in this work.

Chemicals and Reagents

A standard solution (1 mg cm^{-3}) of molybdenum was prepared by dissolving 0.460 g of ammonium molybdate in 250 cm^3 of distilled water.

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This solution was standardized titrimetrically[14] and further diluted with distilled water as required for working solutions.

TEHP (Aldrich, B.P. 215°C) dissolved in toluene (AR grade) and sorbitan monooleate (Span-80) (S.D. Fine Chemical Ltd., India) were used. All other chemicals used were of AR grade.

Procedure

Preparation of ELM

Emulsion was prepared by drop-wise addition of strip phase (1.8 mol dm^{-3} aqueous solution of Na_2CO_3 containing 0.01 mol dm^{-3} NaOH) to a stirred solution of organic phase containing 60% TEHP in toluene and 7.5% surfactant (Span-80) at an O/A phase ratio of 1:2 for 20 minutes at 2000 rpm stirring speed on a motor driven emulsifier to get stable milky white emulsion.

Transport of Metal Ions

In a 150-cm^3 beaker containing 75-cm^3 of feed solution containing 5 to 300 μg of Mo(VI) ion, 15 cm^3 of ELM prepared as above was added and the contents were stirred at 200 rpm on a magnetic stirrer for a given transfer time; emulsion was separated from the aqueous phase in a separating funnel and finally, it was broken down by treating with 0.5 cm^3 of n-butanol at room temperature ($28 \pm 2°\mathrm{C}$). The Mo(VI) content in the strip phase was analyzed spectrophotometrically.[14] The Mo(VI) content remaining in the feed solution was calculated by mass balance. The percentage transport (%T) was then calculated using the following formula.

$$\%\mathrm{T} = \frac{\text{Conc. of Mo(VI) transported in the strip phase}}{\text{Initial conc. of Mo(VI) present in the feed phase}} \times 100$$

Determination of Molybdenum

To an aliquot of Mo(VI) solution neutralized previously by HCl, 2 cm^3 of concentrated HCl, 1 cm^3 of 10% ferrous ammonium sulphate, 3 cm^3 of 10% stannous chloride, and 3 cm^3 of 10% ammonium thiocyanate solutions were added in a 125-cm^3 separating funnel and equilibrated with 10 cm^3 of isoamyl alcohol and the absorbance was read at 465 nm against the reagent blank.[14]

RESULTS AND DISCUSSION

Influence of Hydrochloric Acid Concentration in Feed

The relationship between the concentration of hydrochloric acid in the feed solution and transport of molybdenum into strip phase was studied. An acid concentration of 3.5 to 5.5 mol dm^{-3} was found to be the best for the transport of molybdenum through ELM. Mo(VI) forms various species depending on the conditions of the aqueous solutions. In neutral or alkaline aqueous medium, the molybdate anion $[MoO_4\ (H_2O)_2]^{2-}$ is the only Mo(VI) species. In the acid solution, its protonation takes place stepwise and $[MoO_2\ (H_2O)_2]^{2+}$ is formed in strongly acidic medium as the final mononuclear product.[15] The transport process of MoO_2^{2+} in presence of HCl from feed to the internal strip phase containing Na_2CO_3 and NaOH solution through an ELM containing TEHP in toluene and Span−80, may be illustrated by the following equations:

$$[MoO_2(H_2O)_2]^{2+} + 2Cl^- \rightleftarrows MoO_2Cl_2(H_2O)_2 \quad \text{(In feed phase)} \tag{1}$$

$$MoO_2Cl_2(H_2O)_2 + 2TEHP \rightleftarrows MoO_2Cl_2 \cdot (TEHP)_2 \quad \text{(In membrane phase)} \tag{2}$$

$$MoO_2Cl_2 + 2OH^- \rightleftarrows MoO_4^{2-} + 2H^+ + 2Cl^- \quad \text{(In strip phase)} \tag{3}$$

The expected mechanism of Mo(VI) transport in the present case may be illustrated as shown in Figure 1. This is a facilitated, coupled cotransfer mechanism. MoO_2Cl_2 and H^+ ions from the feed phase were cotransported to the strip phase, as evidenced by decrease of H^+, Cl^-, and Mo(VI) ions in the feed phase and their increase in strip phase. The transport of H^+ ions from feed to the strip phase is due to the difference in concentration gradients, which provide energy to MoO_2Cl_2 to drive it to the surface of the membrane where it reacts with TEHP to form $MoO_2Cl_2(TEHP)_2$ complex, which diffuses inside the membrane. When the concentration of HCl in the feed was 3.5 to 5.5 mol dm^{-3}, the transport of Mo(VI) was found to be 99.8%. However, it was less when the concentration of HCl was below 3.5 mol dm^{-3} and above 5.5 mol dm^{-3}. These results may be explained on the basis of the H^+ ions transporting from feed to the strip. Below 3.5 mol dm^{-3} HCl, since the H^+ ions transporting were less, the amount of MoO_2Cl_2 contacting the surface of the membrane was also less. Thus,

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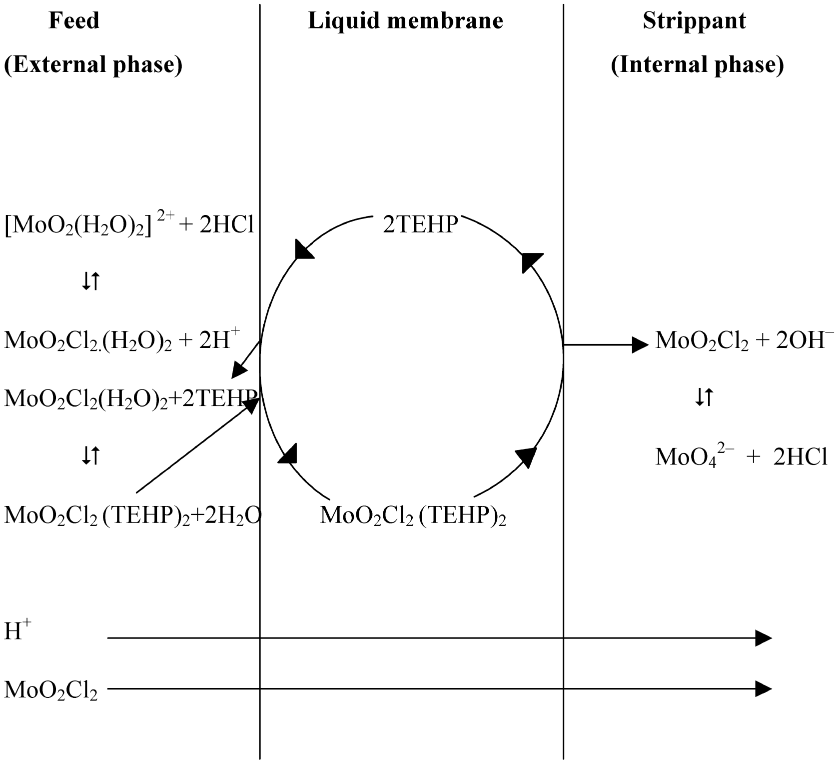

Figure 1. Transport mechanism of Mo(VI) through the ELM process. Concentration of Mo(VI) = 10 μg; concentration of HCl = 4.5 mol dm^{-3}. Concentration of strip phase = 0.01 mol dm^{-3} NaOH in 1.8 mol dm^{-3} aq. Na_2CO_3. Concentration of TEHP in toluene = 60 % (v/v); conc. of Span-80 = 7.5 % (v/v). Stirring time = 25 min; stirring rate = 200 rpm.

formation of $MoO_2Cl_2(TEHP)_2$ and its diffusion inside the membrane and, hence, the percentage transport will decrease. Above 5.5 mol dm^{-3} HCl concentration, H^+ ions transporting from feed to strip will be more. This will neutralize a greater amount of NaOH in the strip, reducing the concentration of NaOH to strip MoO_2Cl_2. Thus, the percentage transport of Mo(VI) will decrease.

The pH of the strip phase was found to decrease from 11.9 to 10.0, 9.5, and 9.1 when the HCl concentration in the feed phase was below 3.5 mol dm^{-3}, between 3.5 to 5.5 mol dm^{-3}, and above 5.5 mol dm^{-3}, respectively. These results were in accordance with the proposed mechanism just discussed.

Influence of Sodium Hydroxide and Sodium Carbonate in the Strip Phase

Sodium hydroxide in the strip (internal) phase functioned as a back-extraction solution. ELM with NaOH solution was unstable. However, emulsion was stable with Na_2CO_3 solution but the transport of Mo(VI) was not maximum with Na_2CO_3 alone. Therefore, a mixture of NaOH and Na_2CO_3 aqueous solution was used as the strip phase because it gave stable emulsion and clear and fast transport of Mo(VI). When the composition of emulsion was fixed, variation of NaOH from 0.005 to 0.015 mol dm^{-3} at the fixed concentration of 1.8 mol dm^{-3} Na_2CO_3 and variation of Na_2CO_3 from 1.4 to 2.0 mol dm^{-3} at the fixed concentration of 0.01 mol dm^{-3} NaOH in the internal phase, gave maximum transport of 99.8%.

Influence of Concentration of Span-80

Both the stability of the emulsion and the viscosity of the liquid membrane were altered by changing the proportion of surfactant in toluene. When the concentration of Span-80 was less than 6% (v/v), the ELM was found to leak with transfer time during stirring (Table 1). A concentration of 7 to 8% (v/v) Span-80 in the membrane resulted in maximum transport and stability. Therefore, 7.5% (v/v) Span-80 was used in the preparation of the ELM. Above 8% (v/v) Span-80, percentage transport of Mo(VI) decreased. This may be due to the increased viscosity of the membrane opposing the flow of solute inside the membrane.

Influence of Concentration of TEHP

The influence of concentration of TEHP (mobile carrier) in toluene on the transport efficiency of molybdenum was studied. The percentage transport of molybdenum increased with an increasing TEHP concentration

Table 1. Effect of concentration of surfactant.

Concentration of Span-80 (% v/v)	5	6	7	8	10
Percentage transport	94.0	99.3	99.8	99.8	92.0

Conditions:
Concentration of Mo(VI) = 10 µg; concentration of HCl = 4.5 mol dm^{-3}.
Concentration of strip phase = 0.01 mol dm^{-3} NaOH in 1.8 mol dm^{-3} aq. Na_2CO_3.
Concentration of TEHP in toluene = 60% (v/v); concentration of Span-80 = 7.5% (v/v).
Stirring time = 25 min; stirring rate = 200 rpm.

in toluene. Maximum transport occurred at 50 to 70% (v/v) of TEHP and additional TEHP had no significant effect on the transport efficiency. Subsequent transport studies of molybdenum were carried out with 60% (v/v) TEHP in toluene. It was observed that the amount of $[MoO_2Cl_2 (TEHP)_2]$ in the liquid membrane increased by increasing the concentration of TEHP in the membrane until a concentration of 50% (v/v) TEHP was reached. However, addition of an excess amount of the carrier, up to 70% (v/v), did not change the percentage transport of molybdenum. This may probably be due to the fixed stoichiometry of resultant 2:1 TEHP:Mo(VI) complex $[MoO_2Cl_2 (TEHP)_2]$ as well as the more or less constant viscosity of the membrane phase at this concentration range.

Influence of Time of Stirring

Time dependence of molybdenum transport through the membrane under the optimum experimental conditions was studied. It was observed that complete transport of molybdenum occurred after 25 minutes. The reproducibility of transport was investigated. The percentage of Mo(VI) ion transport after 25 minutes from triplicate measurement was found to be $99.8 \pm 0.5\%$.

When the concentration of NaOH and Na_2CO_3 were 0.01 mol dm^{-3} and 1.8 mol dm^{-3} respectively, in the strip phase, by changing the transfer time from 5 to 30 minutes, the natural logarithm of the ratio of initial concentration $[C_0]$ to given time concentration $[C_t]$ of molybdenum in the feed phase was measured (Table 2). For the kinetic curve, a plot of ln C_0/C_t against time t, a good straight line with $\ln C_0/C_t = 0.0023t + 0.0173$, $R^2 = 0.9946$ was obtained (Figure 2). This showed that transport of MoO_2^{2+} is a first-order reaction with a transfer rate constant of $0.00233 sec^{-1}$. This further showed that the transfer rate of MoO_2^{2+} was controlled by MoO_2Cl_2. 2TEHP concentration on the feed side interface. Its kinetic equation is illustrated by following:

$$-d[MoO_2^{2+}] = k[MoO_2Cl_2(TEHP)_2]dt$$

Influence of Stirring Rate on Transport

The stirring rate (speed of agitation) of the feed phase affects the performance of ELM process to a great extent. The effect of the stirring rate was studied in the range of 100 to 400 rpm. It was observed that with an increase in stirring rate from 100 rpm to 200 rpm, the rate of transport increased. This was due to an increase in volumetric mass-transfer coefficient in aqueous feed phase between the emulsion globules and aqueous

Table 2. Effect of time on extraction of Mo(VI).

Contact time (min)	Mo(VI) in external (feed) phase (μg)	Mo(VI) in internal (strip) phase (μg)	Percentage transport of Mo(VI)	ln $^*[C_o/C_t]$
0.0	10.0	—	—	0
5.0	5.13	4.87	48.76	0.676
10.0	2.20	7.80	77.99	1.514
15.0	1.25	8.75	87.50	2.080
20.0	0.60	9.40	94.00	2.813
25.0	—	9.98	99.80	—
30.0	—	9.98	99.80	—

Conditions:
Concentration of Mo(VI) = 10 μg; concentration of HCl = 4.5 mol dm^{-3}.
Concentration of strip phase = 0.01 mol dm^{-3} NaOH in 1.8 mol dm^{-3} aq. Na_2CO_3.
Concentration of TEHP in toluene = 60% (v/v); concentration of Span-80 = 7.5% (v/v).
Stirring time = 25 min; stirring rate = 200 rpm.
$^*[C_o]$: Initial concentration of Mo(VI) in the feed phase at zero time. $[C_t]$: concentration of Mo(VI) in the feed phase at time t.

feed solution. An increase in the stirring rate from 200 to 300 rpm did not change the percentage transport of molybdenum. However, above 300 rpm, percentage transport decreased, which was contrary to the general expectation. It was attributed to hydrodynamic instability of emulsion at high speed and increased stirring rate might have caused shear on emulsion globules. The resulting shear might be causing the breakage of emulsion. Therefore, a stirring rate of 200 rpm was used in all the experiments.

Separation of Molybdenum from Binary Mixtures

For investigation of the selectivity of the membrane system for the transport of molybdenum, an aliquot of solution containing 10 μg of Mo(VI) and competing ion to be tested with concentration, as mentioned in Table 3 was taken and 10 mol dm^{-3} HCl was added so that its concentration was 3.5 to 5.5 mol dm^{-3} in a total volume of 75 cm^3 of feed phase. The concentration of Mo(VI) in the strip phase and the concentration of the competing ion in the feed phase were determined after 25 minutes spectrophotometrically using the respective chomogenic reagents as mentioned in Table 3. From the results, the amount of competing ion cotransported along with Mo(VI) was calculated. As is seen, among the different ions tried, W(VI) was cotransported 35% and the cotransports of other ions

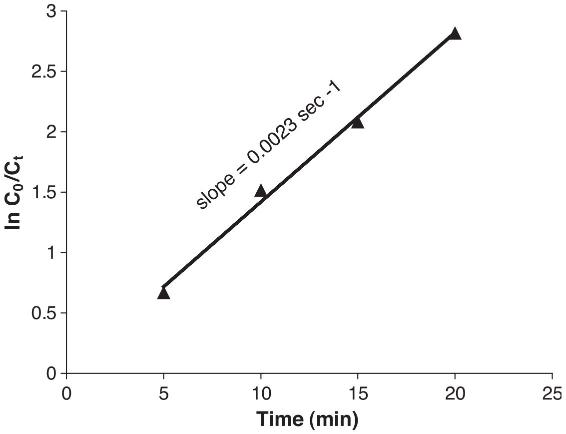

Figure 2. Kinetic curve of transport of Mo(VI) through the ELM process. Concentration of Mo(VI) = 10 μg; conc. of HCl = 4.5 mol dm^{-3}. Concentration of strip phase = 0.01 mol dm^{-3} NaOH in 1.8 mol dm^{-3} aq. Na_2CO_3. Concentration of TEHP in toluene = 60 % (v/v); conc. of Span-80 = 7.5 % (v/v). Stirring time = 25 min; stirring rate = 200 rpm.

were negligible. However, the interference effect of W(VI) was successfully eliminated using 1% potassium oxalate as a masking agent in the feed phase. These results indicate that the separation of Mo(VI) by the proposed procedure is selective.

Preconcentration of Mo(VI) from Dilute Solutions

Transport studies of 10 μg of Mo(VI) from aqueous feed solutions were carried out using the proposed procedure by increasing the volume of feed solution from 50 cm^3 to 200 cm^3. The observed results indicate that up to 150 cm^3 of feed phase percentage transport of Mo(VI) was 99.8%. Thus, 10 μg of Mo(VI) from 150 cm^3 of feed phase can be preconcentrated in 10 cm^3 of strip phase, the preconcentration factor being 15.

APPLICATIONS

Separation of Mo(VI) from Spent Catalyst

The catalyst WN 289, 0.1 g, was dissolved in aqua regia by boiling. The solution was evaporated to a paste and the residue was dissolved and diluted

Table 3. Effect of foreign ions on transport of Mo(VI).

		Molybdenum			Other metal ion		
Sr. no.	Composition binary mixtures (μg)	Found (μg) (in strip phase)	Recovery % (in strip phase)	% RSD (n = 3)	Recovery % (in feed phase)	% RSD (n = 3)	Chromogenic reagents (nm) for added metal ions
1	Mo(VI) (10) Mn(II) (100)	9.99	99.90	0.96	100.00	0.72	Formaldoxime (445)
2	Mo(VI) (10) Ni(II) (100)	9.94	99.40	1.43	100.00	0.84	DMG (445)
3	Mo(VI) (10) Cr(VI) (100)	10.0	100.00	0.63	99.80	1.08	DPC (540)
4	Mo(VI) (10) Fe(II) (100)	9.95	99.53	1.35	99.40	0.96	Thiocyanate (480)
5	Mo(VI) (10) Co(II) (100)	9.98	99.80	1.04	99.70	1.10	Nitroso R salt (415)
6	Mo(VI) (10) Sn(II) (100)	9.92	99.20	0.45	100.00	1.35	Pyrocatechol violet (552)

7	Mo(VI) (10) W(VI) (100)	8.50	85.00	0.84	64.76	0.63	Thiocyanate (400)
8	Mo(VI) (10) W(VI) (100)	9.98*	99.80*	1.18*	99.80	0.16	Thiocyanate (400)
9	Mo(VI) (10) V(V) (100)	10.00	100.00	1.71	99.80	0.20	H_2O_2 (450)
10	Mo(VI) (10) Al(III) (100)	10.00	100.00	1.10	100.00	0.96	Eriochrome cyanin R (535)
11	Mo(VI) (10) Ti(IV) (100)	10.00	100.00	1.10	100.00	0.84	H_2O_2 (410)
12	Mo(VI) (10) Th(IV) (100)	9.99	99.90	0.84	99.80	0.20	Arsenozo III (665)
13	Mo(VI) (10) U(I) (100)	9.98	99.80	0.96	99.40	0.16	PAR (530)

*In the presence of 1% potassium oxalate. RSD = relative standard deviation; n = number of analysis; DMG = dimethyl glyoxime; and DPC = diphenyl carbazide.

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to 100 cm^3 with 2 mol dm^{-3} of HCl. The molybdenum content of this solution was determined spectrophotometrically by the thiocyanate method.[14]

A 1-cm^3 portion of this solution was then taken in the feed phase and 10 mol dm^{-3} HCl was added so that its concentration was 4.5 mol dm^{-3} with respect to HCl in a total volume of 75 cm^3 of the feed phase. Agitation of this phase with 15 cm^3 of emulsion phase at 200 rpm for 25 minutes, as per the proposed procedure, was carried out. The amount of Mo(VI) transported in the aqueous solution of 1.8 mol dm^{-3} Na_2CO_3 containing 0.01 mol dm^{-3} NaOH as internal phase was determined spectrophotometrically.[14] The mean amount of Mo(VI) recovered was found to be 99.4%, with percentage RSD as 0.2% for n = 3 (Table 4).

Table 4. Separation of Mo(VI) from spent catalyst, ferrous scrap, and molybdenite concentrate.

		Molybdenum			
Sample	Composition	Expected (mg/100 mg)	Found* (mg/100 mg)	% Recovery	% RSD (n = 3)
Catalyst WN 289	Mo, 33 mg/g W, 10 mg/g Al, 235 mg/g Ni, 14 mg/g	3.30	3.28	99.40	0.20
Ferrous scrap	Fe, 64.4%; Cr, 18.2%; Ni, 12.7%; Mo, 2.7%; Mn, 1.1%; Cu, 0.1%; Co, 0.2%; P, 0.007%; C, 0.03%; S, 0.003%; Ti, trace	2.70	2.68	99.30	0.16
Molybdenite concentrate	Mo, 31.25%; Ni, 0.17%; Re, 0.014%; Cu, 0.35%; Fe, 1.62%; S, 23.25%	31.25	31.19	99.80	0.54

RSD = relative standard deviation; n = number of analysis.
*Average of triplicate analysis.

Separation of Mo(VI) from Ferrous Scrap

100 mgs of finely powdered ferrous scrap was treated with 25 cm^3 of conc. HCl in a 250 cm^3 round bottom flask fitted with a chlorine gas delivery tube and heated for about an hour on a boiling water bath. Chlorine gas was passed continuously during heating. The clear solution was then diluted to 50 cm^3 with distilled water. 0.5 cm^3 of this solution was used for the separation of Mo(VI) by the proposed procedure described as above. The mean recovery of Mo(VI) was found to be 99.30% with% R.S.D. as 0.16% for $n = 3$ (Table 4).

Separation of Mo(VI) from Low-Grade Molybdenite Concentrates

One hundred mg of the molybdenite concentrate sample was mixed with 100 mg of sodium carbonate in a graphite crucible and heated to 650 to 700°C for about an hour. The mass was then brought into solution by extraction with hot water. The solution was diluted to 250 cm^3 with water. Ten cm^3 of this solution was then diluted again to 50 cm^3 with water and 1 cm^3 of the diluted solution was used for the separation of Mo(VI) by the proposed procedure as above. The mean amount of recovery of Mo(VI) was found to be 99.8% with percentage RSD as 0.54% for $n = 3$ (see Table 4).

CONCLUSION

The study of the transport of Mo(VI) through a TEHP–Span-80–toluene ELM revealed that the optimum conditions for the transport of Mo(VI) should be 3.5 to 5.5 mol dm^{-3} HCl in the feed solution, 50 to 70% TEHP in toluene, and 7 to 8% (v/v) Span-80 in the liquid membrane, and 0.005 to 0.015 mol dm^{-3} NaOH and 1.4 to 2.0 mol dm^{-3} Na_2CO_3 in the stripping solution. The stirring rate and contact time should be 200 rpm and 25 minutes, respectively. It was also concluded that Mo(VI) could be separated selectively from its binary mixtures containing Fe(II), Co(II), Ni(II), Mn(II), Cr(VI), Al(III), W(VI), Ti(IV), Sn(IV), V(V), U(VI) and Th(IV). The method can be employed for the preconcentration of Mo(VI). Less than 10 μg of Mo(VI) from 150 cm^3 of diluted solution can be preconcentrated to 10 cm^3 by this method, the preconcentration factor being 15. The method can be applied for the recovery of Mo(VI) from solutions of spent catalyst, ferrous scrap, and low-grade molybdenite concentrate.

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