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Separation of Cadmium and Zinc by Supported Liquid Membrane Using TOPS-99 as Mobile Carrier

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ABSTRACT

The separation of cadmium and zinc from a dilute aqueous sulfate media using supported liquid membrane (SLM) technique has been studied. The microporous polypropylene film Celgard-2400 was used as the solid support for the liquid membrane and TOPS-99 was used as the mobile carrier. The effect of different parameters such as flow rate, pH of feed solution, extractant concentration in membrane phase, acid concentration in strip solution, and Cd(II) and Zn(II) concentration in feed solution on metal ion flux was studied. It was observed that a flow rate of 100 mL/min was sufficient to minimize the resistance due to an aqueous boundary layer at the feed solution membrane interface. The zinc flux increased with an increase of pH from 1.5 to 3.5 and then decreased with a further increase of pH. The cadmium permeation

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started at a higher pH, i.e., 3.0 and increased with increase of pH up to 3.5 followed by a plateau. Also, with an increase of TOPS-99 concentration in the membrane phase up to 200 mol/m³, J_{Zn} increased and then decreased. The separation factors for cadmium and zinc were calculated at different experimental conditions and are reported.

Key Words: Liquid membrane; Cadmium; Zinc; TOPS-99; Mobile carrier; Permeation.

INTRODUCTION

Several metal, such as As, Cd, Cu, Cr, Hg, Pb, Se, and Zn, are generated as industrial effluents that are hazardous to health and the environment. Out of these, cadmium is considered to be one of the most toxic elements. Cadmium is disposed off by different industries in the form of CdO, Cd(OH)₂, CdCO₃, CdCl₂, Cd(NO₃)₂ · 4H₂O, and CdSO₄. Cadmium poisoning occurs through inhalation and ingestion, leading to damage of the pulmonary and the renal systems. In comparison to cadmium, zinc has very little adverse effect on the environment, but, due to its thermodynamic properties, it has an affinity for cadmium. Many industrial products, namely batteries, electroplating, stabilizers, pigments, pesticides, fertilizers, cement, etc., contain both cadmium and zinc in their effluents, and these need to be removed before disposal.

By using precipitation methods, cadmium and zinc can be removed as carbonates and as hydroxides^[1] from most effluent streams. But such precipitation processes have inherent problems of solid–liquid separation, high cost, and slow kinetics. Solvent extraction and ion exchange processes also can be used for the removal of these heavy metals. However, due to low levels of these metals, these techniques may not be cost effective. As an alternative, a supported liquid membrane (SLM) technique could have several advantages. They are operational simplicity, low solvent inventory, low energy consumption, etc. The SLM technique is used for extraction/separation of different metal ions such as Cu, Zn,^[2–4] Cr,^[5–7] Al,^[8] Ce,^[9] Au,^[10] Pt,^[11] and Fe(III).^[12]

Several extractants, such as Cyanex 923, Aliquat 336, D2EHPA (di-2ethyl-hexyl phosphoric acid), LIX84, etc., are used for the extraction of cadmium and zinc.^[13–16] The systems, such as Zn–Cd–Co–Ni,^[17] Zn–Cd,^[18] and Zn–Cd–Hg,^[19,20] also have been investigated for separation of cadmium and zinc by using D2EHPA, carboxylic acid, and caprylic acid as extractants.

Although a good deal of work has been reported for the liquid–liquid extraction of cadmium and zinc, few studies concerning the separation of cadmium and zinc by using SLM are reported. In these studies, extractants,

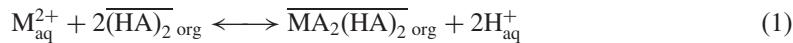
such as trilaurylamine–kerosene,^[21] Cyanex 923,^[22] tricaprilamine,^[23] etc., are used. The effect of paraffin and a surfactant on coupled transport of Cd(II) through bulk liquid membrane was studied by He and Ma.^[24] D2EHPA is a widely used metal extractant for separation of different metal ions, but the work on separation of cadmium and zinc by using D2EHPA as a mobile carrier is scanty.

The present work examines the possibility of using a SLM technique to separate cadmium and zinc from dilute solutions by using TOPS-99 as an extractant. (TOPS-99 is D2EHPA produced by Heavy Water Plant, Talcher, Orissa, India.) The equilibrium studies were carried out to find out the extraction mechanism of Cd(II) and Zn(II) with TOPS-99. In SLM studies, the effect of various process variables, such as flow rate, pH of feed solution, extractant concentration in membrane phase, acid concentration in strip solution, and Cd(II) and Zn(II) concentration in feed solution on the metal ion flux, was studied.

THEORY

The complex formation of a divalent metal ion with TOPS-99 at the feed side–membrane interface involves monomerization of (TOPS-99)₂, distribution of monomer between aqueous interface and organic medium, and acid dissociation of monomer at the interface.^[25]

The mechanism by which a divalent metal ion is extracted from an aqueous phase by using TOPS-99 can be written as:



where the subscript aq is the aqueous phase and subscript org is the organic phase.

The equilibrium constant, K , of the reaction can be written as:

$$K = \frac{\overline{[MA_2(HA)_2]}_{org} [H^+]_{aq}^2}{[M^{2+}]_{aq} \overline{[(HA)_2]}_{org}^2} \quad (2)$$

or

$$K = \frac{D[H^+]_{aq}^2}{\overline{[(HA)_2]}_{org}^2} \quad (3)$$

where distribution ratio $D = \overline{[MA_2(HA)_2]}_{org} / [M^{2+}]_{aq}$.

$$\log D = \log K + 2 \text{pH} + 2 \log \overline{[(HA)_2]}_{org} \quad (4)$$

Analyzing the experimental value of the distribution ratio (D) as a function of equilibrium pH and extractant concentration at constant value of other parameters allows estimation of the number of extractant molecules associated with the extractant metal complex.

The relationship that correlates the membrane flux, J ; concentration of the feed solution $[M]$; volume of the feed solution, V ; and the membrane area, A is

$$J = \left(\frac{V}{A}\right) \frac{d[M]}{dt} \quad (5)$$

The permeability coefficient of the membrane, P , is defined as:

$$P = \frac{J}{[M]} \quad (6)$$

Two or more metal ion species present in the feed solution can be separated when their permeability coefficient values are different. The separation factor, α , is defined by the following equation:

$$\alpha = \frac{P_1}{P_2} = \frac{(J_{M_1}/[M_1]_f)}{(J_{M_2}/[M_2]_f)} \quad (7)$$

where J_{M_1} and J_{M_2} are the fluxes for the major and the minor transporting components, respectively, and $[M_1]_f$ and $[M_2]_f$ are their feed concentrations.

EXPERIMENTAL

Chemicals/Reagents

All chemicals used in this study, namely $3\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$, ZnSO_4 , H_2SO_4 , NaOH , CH_3COONa , CH_3COOH , etc., were of analytical reagent grade. Stock solutions of CdSO_4 and ZnSO_4 (5 kg/m^3) were prepared and standardized against an ethylene diamine tetra-acetate (EDTA) solution. The working solutions were prepared by dilution.

The commercial extractant TOPS-99 was used as received, without any further purification. Distilled kerosene (bp $190\text{--}210^\circ\text{C}$) was used as the diluent. Tri-*n*-butyl phosphate 5 vol% was used as a modifier.

Membrane

Celgard 2400, a microporous polypropylene film, was used as the solid support for the liquid membrane. Hoechst Inc., Charlotte, USA, supplied this film. As per the specification provided by the supplier, the

membrane has a porosity of 41%, thickness of 25.4 μm , and pore dimensions ($W \times L$) = 0.04 \times 0.12 μm .

Methods

Liquid Membrane Experiments

A plate- and a frame-type of cell with an effective membrane area (geometrical membrane area \times porosity) of 0.0217 m^2 was used for the liquid membrane experiment. The length, width, and depth of the channels of both the feed and the strip solutions were 13, 7, and 0.1 cm, respectively.^[26] The volumes of the strip solution and the feed solution used were 200 mL each. A buffer solution of $\text{CH}_3\text{COOH}-\text{CH}_3\text{COONa}$ was used to maintain the required pH of the feed solution in the range of 3.42–5.89 and $\text{HCl}-\text{CH}_3\text{COONa}$ was used to maintain the pH in the range of 2.0–3.09. Both the solutions were circulated through the module with the help of a peristaltic pump (Watson Marlow 501 S). The feed solution was kept under agitation by using a mechanical stirrer. Equal volumes of samples (1 mL) were withdrawn from both the solutions at the desired time interval. The samples were analyzed for Zn(II) and Cd(II) concentration by using an atomic absorption spectrophotometer (Perkin Elmer, model 372).

Equilibrium Study

For equilibrium studies, 10 mL of the aqueous solution containing 0.89 mol/m³ of cadmium and 1.53 mol/m³ of zinc was equilibrated with an equal volume of TOPS-99 in a separating funnel for 10 min. After complete phase disengagement, the aqueous phase was separated and was analyzed for Cd(II) and Zn(II) concentrations. The concentrations of Cd(II) and Zn(II) in the organic phase were calculated from the difference between the metal ion concentrations in the aqueous phase before and after extraction.

RESULTS AND DISCUSSION

Equilibrium Study

Effect of Equilibrium pH

The extractions of cadmium and zinc from sulfate media containing 0.89 mol/m³ Cd(II) and 1.53 mol/m³ Zn(II) were studied by using TOPS-99

(5 mol/m³) within the equilibrium pH range 1.0–5.45 (Fig. 1). The percentage extraction of cadmium was very low, within a pH range of 1.0–3.0, but it increased from 7.02 to 87.35 with an increase of equilibrium pH from 3.0 to 4.5. The percentage extraction of zinc increased from 8 to 99.365 with an increase of equilibrium pH from 1.0 to 3.95 and then decreased to 79.09 with a further increase of pH to 5.6. Figure 2 shows the plots of $\log D$ vs. equilibrium pH. The plots are linear, with slopes of 1.8 and 1.7 for zinc and cadmium, respectively, indicating the exchange of 2 mol of H⁺ with 1 mol of the extracted metal species for both zinc and cadmium, which supports the reaction mechanism shown in Eq. (4). On comparing the extraction data for Zn and Cd when using D2EHPA, it is observed that the extraction takes place at a higher pH with SLM, compared with normal solvent extraction in a shake flask.^[17]

Effect of Extractant Concentration

The effect of TOPS-99 concentration on the extraction of cadmium (0.89 mol/m³) and zinc (1.53 mol/m³) was studied in the range 1.0–30 mol/m³.

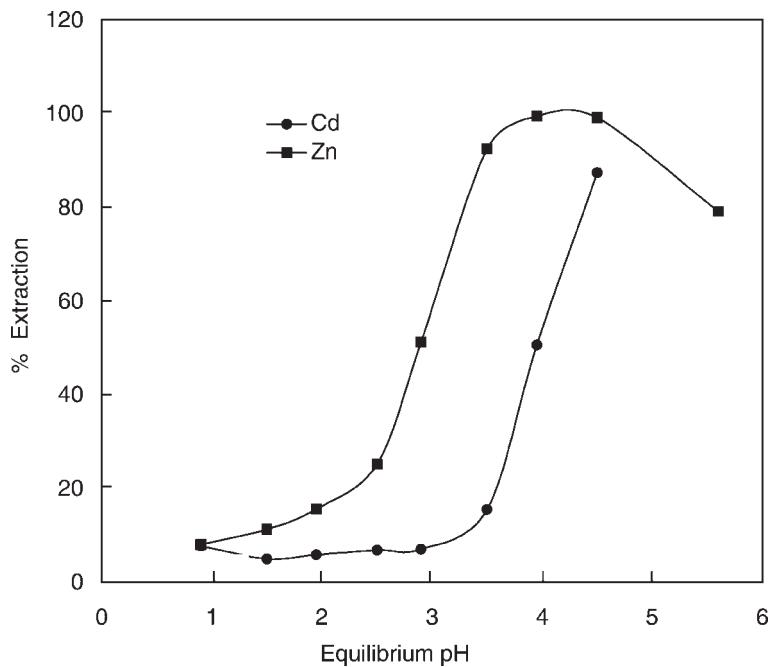


Figure 1. Effect of equilibrium pH on extraction of Cd and Zn.

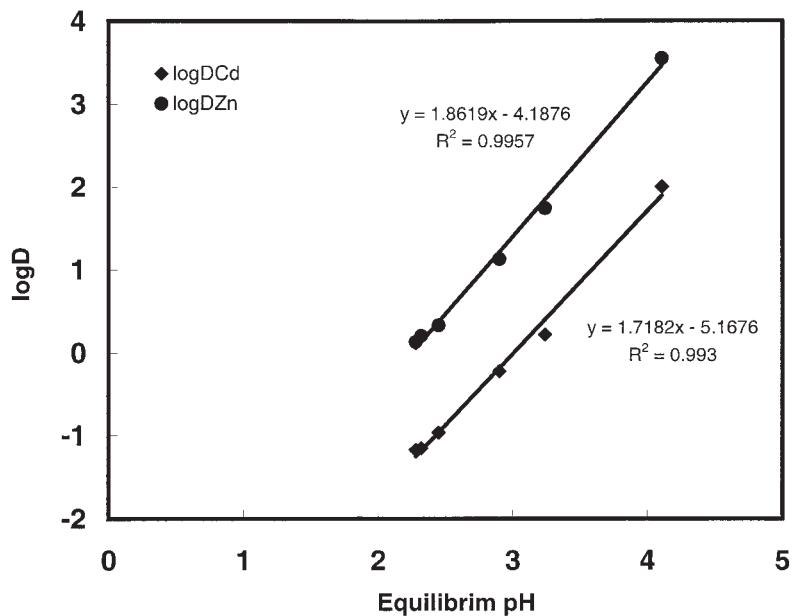


Figure 2. Plot of $\log D$ vs. equilibrium pH.

It was observed that the percentage extraction of Zn(II) increased with an increase of extractant concentration (Fig. 3). The pH of aqueous phase was kept constant at 2.9, so that cadmium permeation was minimum. The plot of $\log D$ vs. $\log[\text{HA}]$ was a straight line with a slope of 1.89 (Fig. 4). This indicates that 2 mol of extractant are associated with the extracted metal species of zinc. Similar results also were obtained for cadmium.^[27]

Transport of Metal Ions by SLM

It was observed that the concentrations of zinc and cadmium decreased in the feed compartment linearly with time for 1 hr and increased with time in the strip compartment at the same rate as it decreased in the feed solution. The fluxes of the metal ions (J_{Zn} and J_{Cd}) were calculated from the slopes of the initial straight lines of the $(V/A) d [\text{Cd(II)}] / dt$ plots (figures are not shown).

Some preliminary experiments were carried out with different flow rates for feed and strip solutions, and it was observed that a flow rate of 100 mL/min.

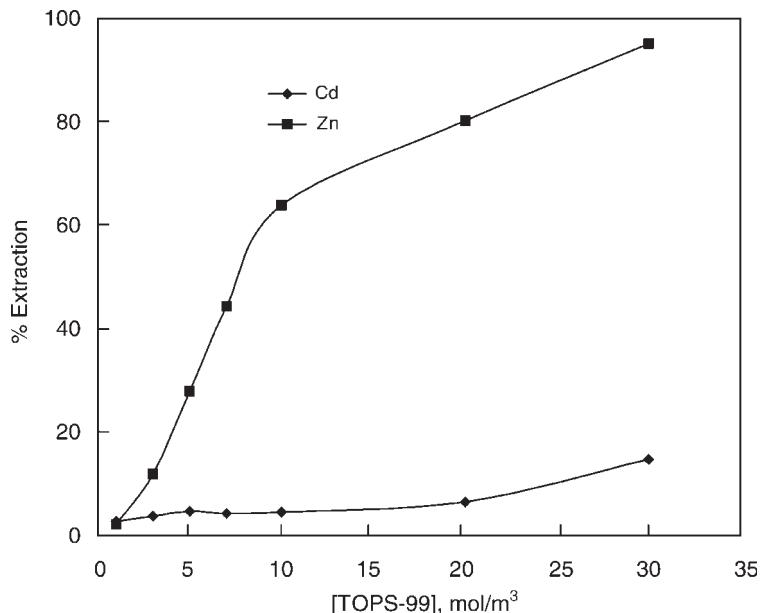


Figure 3. Effect of extractant concentration on percentage extraction of Zn and Cd.

was sufficient to reduce the resistance due to the aqueous boundary layer. So all other experiments were carried out at this flow rate.

Determination of Diffusivity of Zn-TOPS-99 and Cd-TOPS-99 Complex

For determination of diffusivity of Zn-TOPS-99 and Cd-TOPS-99 complex, the membrane was impregnated with kerosene. The Zn-TOPS-99 and Cd-TOPS-99 complexes, and the kerosene were taken as the feed and strip solutions, respectively. Samples were collected from the strip side at the desired time interval and were analyzed for concentration of zinc and cadmium after stripping with acid. The amounts of complexes transferred were plotted against time t (Fig. 5). From the time lag of the straight lines, diffusion coefficients of the Cd-TOPS-99 and Zn-TOPS-99 complexes were determined to be 3.58×10^{-12} and 4.3×10^{-12} m/sec, respectively.^[28]

Effect of pH

The effect of pH on extraction and separation of Cd(II) and Zn(II) was studied in the pH range of 1.5–5.0. The concentrations of Cd(II) and Zn(II)

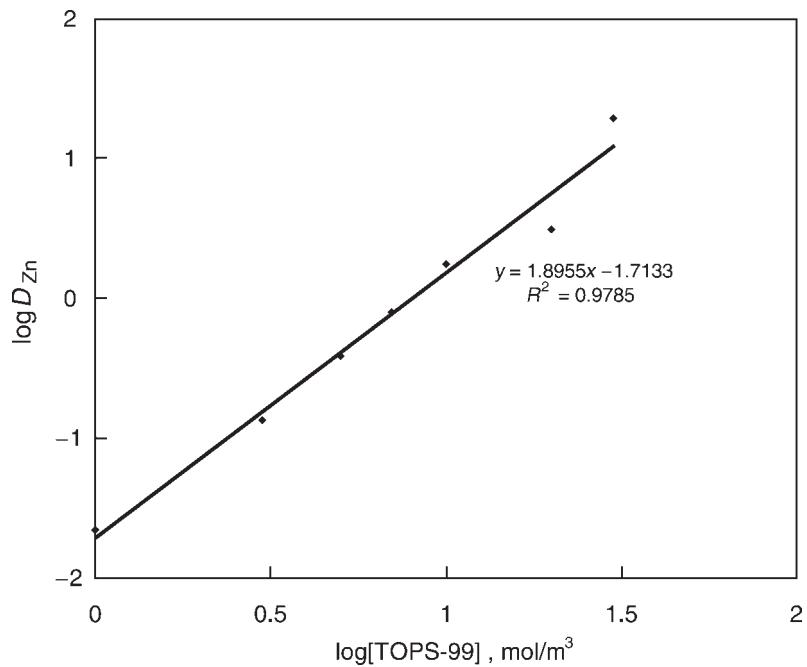


Figure 4. Plot of $\log D$ vs. $\log[\text{TOPS-99}]$.

in the feed solution were kept constant at 0.89 and $1.53 \text{ mol}/\text{m}^3$. J_{Zn} and J_{Cd} were plotted against the feed solution pH in Fig. 6, which showed that zinc flux increased with an increase of pH from 1.5 to 3.5. After pH 3.5, zinc flux values decreased. The cadmium permeation started at a higher pH, i.e., 3.0 and increased with an increase of pH up to 3.5 followed by a plateau. It was observed that the permeation behavior of zinc and cadmium for binary solution in the feed solution were the same as it is with a single metal ion (either Zn or Cd).^[26,27] The separations factors were calculated by using Eq. (7) and are given in Table 1. At pH 2.25, maximum separation factor was obtained, since the copermeation of Cd with Zn was less at this pH. At pH above 2.25, cadmium copermeates along with zinc and, therefore, the separation factor decreased sharply from 23.243 to 1.08.

Effect of the Extractant Concentration

The TOPS-99 concentration in the membrane phase varied from 25 to $300 \text{ mol}/\text{m}^3$. The Cd(II) and Zn(II) concentrations in the feed solution were kept constant at 0.89 and $1.53 \text{ mol}/\text{m}^3$, respectively. The pH of the feed solution and the other parameters also were kept constant. Figure 7 shows

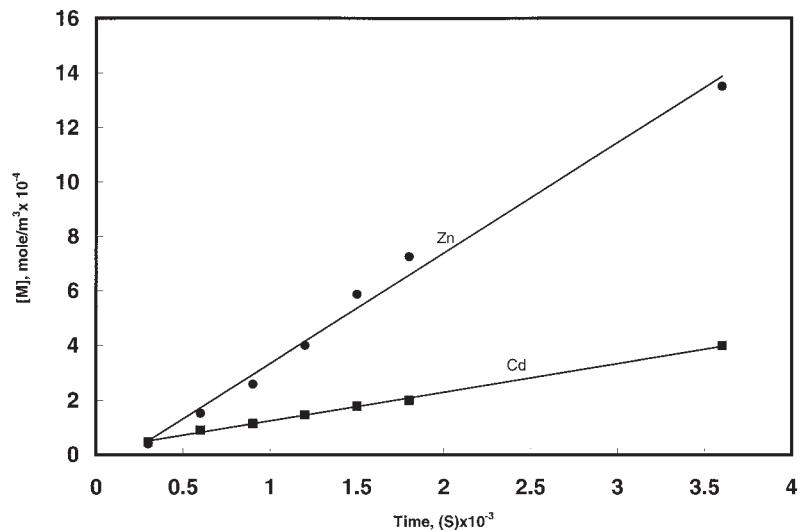


Figure 5. Time-dependent concentration of Cd-TOPS-99 and Zn-TOPS-99 complex in strip side.

the relationship of $J_{\text{Cd(II)}}$ and $J_{\text{Zn(II)}}$ with concentration of TOPS-99. Zinc flux increased with an increase of TOPS-99 concentration up to 200 mol/m^3 ($[\text{HAs}]_2 = 100 \text{ mol/m}^3$) and with further increase of extractant concentration, the flux decreased. As per Eq. (4), with an increase of TOPS-99 concentration, the formation of Zn(II)-TOPS-99 complex increased at the feed side-membrane interface, and, since at a lower extractant concentration the same interface was not saturated by the extractant, the flux increased with an increase of the extractant concentration. With a further increase of TOPS-99 concentration, viscosity of the membrane phase increased, which decreased the metal ion flux. The pH of the feed solution was maintained at 2.25, so the copermeation of cadmium along with zinc was minimum. The separation factors were calculated at different TOPS-99 concentrations and are given in Table 2. It was observed that the separation factor increased from 11.67 to 38.80 with an increase of TOPS-99 concentration from 10 to 200 mol/m^3 and then decreased to 29.053.

Effect of Zinc and Cadmium Concentrations in the Feed Solution

The effect of zinc concentration on the separation factor of cadmium and zinc was studied in the range of 0.765 – 7.65 mol/m^3 . The concentration of

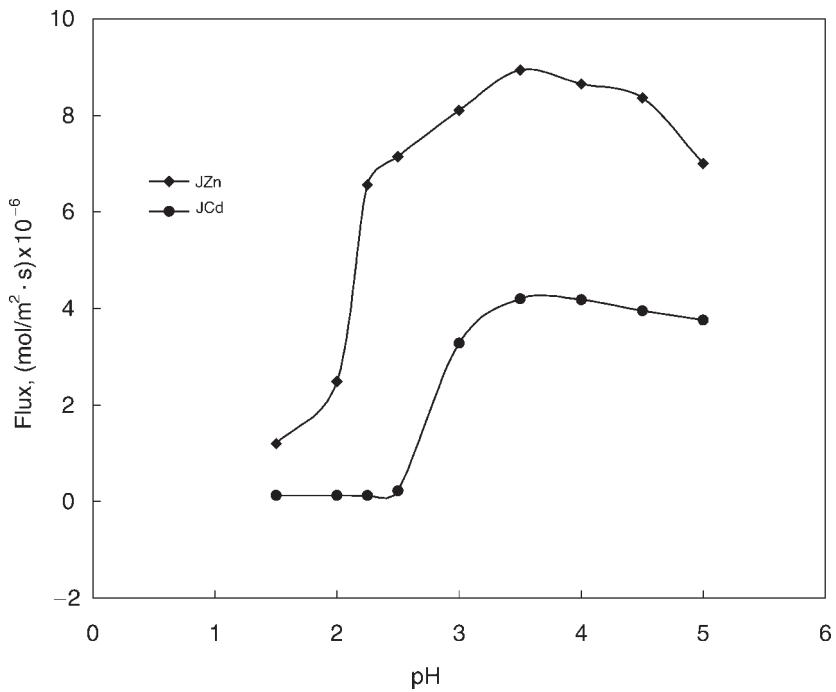


Figure 6. Effect of pH on metal ion flux.

Table 1. Separation factor of cadmium and zinc at different pH values.

pH	J_{Zn} (mol/m ² sec) $\times 10^{-6}$	J_{Cd} (mol/m ² sec) $\times 10^{-6}$	P_{Zn} (m/sec) $\times 10^{-6}$	P_{Cd} (m/sec) $\times 10^{-6}$	$\alpha = P_{Zn}/P_{Cd}$
1.5	1.2	0.125	0.784	0.1406	5.576
2.0	2.438	0.125	1.593	0.1406	11.33
2.25	5.0	0.125	3.268	0.1406	23.243
2.5	7.14	0.22	4.666	0.2475	18.8525
3.0	7.784	3.2	5.0875	3.599	1.4135
3.5	8.941	4.2	5.844	4.725	1.236
4.0	7.090	4.18	4.6339	4.70	0.98
4.5	8.364	3.95	5.4667	4.443	1.23
5.0	7.0	3.76	4.575	4.229	1.08

Note: Experimental condition: $[Zn]_{\text{feed}} = 1.53 \text{ mol/m}^3$; $[Cd]_{\text{feed}} = 0.889 \text{ mol/m}^3$; $[\text{TOPS-99}]_{\text{membrane}} = 100 \text{ mol/m}^3$; $[\text{H}_2\text{SO}_4]_{\text{strip}} = 900 \text{ mol/m}^3$.

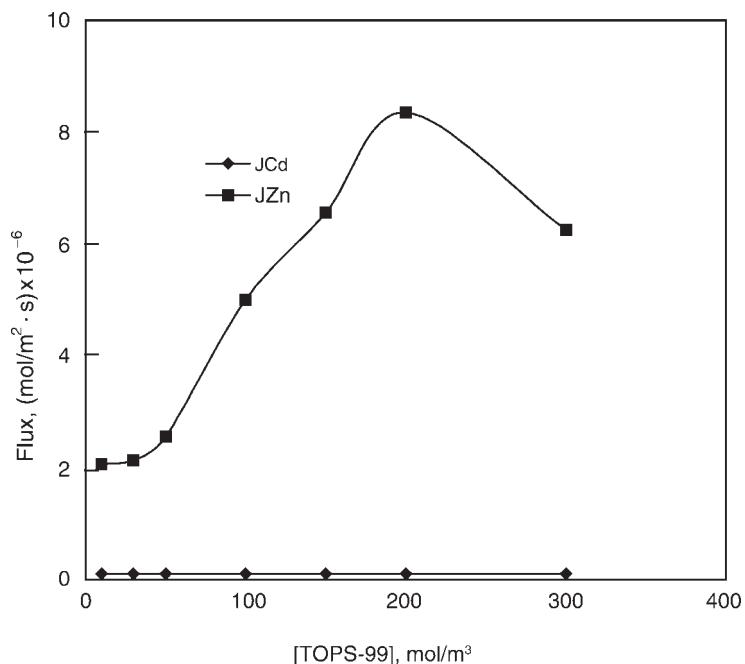


Figure 7. Effect of [TOPS-99] on metal ion flux.

cadmium in the feed solution was kept constant at $0.89 \text{ mol}/\text{m}^3$. The TOPS-99 concentration in the membrane phase and pH of the solution also were kept constant at $200 \text{ mol}/\text{m}^3$ and at 2.25, respectively. Within this concentration range of zinc in the feed solution, the availability of zinc ion at the feed side–membrane interface increased with an increase of zinc concentration. So the interfacial chemical reaction became faster [Eq. (1)], which led to an increase of J_{Zn} from 3.667×10^{-6} to $41.739 \times 10^{-6} \text{ mol}/\text{m}^2 \text{ sec}$ (Fig. 8). But the cadmium flux remained constant at $1.25 \times 10^{-7} \text{ mol}/\text{m}^2 \text{ sec}$, because the pH of the feed solution was maintained at 2.25 and, at that pH, cadmium extraction was minimum. The separation factors obtained at different concentrations of zinc are given in Table 3. The highest separation factor (53.854) was obtained with $0.765 \text{ mol}/\text{m}^3$ of zinc and $0.89 \text{ mol}/\text{m}^3$ of cadmium in the feed solution. Cadmium concentration in feed solution was varied from 0.44 to $4.4 \text{ mol}/\text{m}^3$, keeping the zinc concentration constant at $1.53 \text{ mol}/\text{m}^3$ in the feed solution. It was observed that with the increase of cadmium concentration in the feed solution, there was not much change in the zinc and the cadmium fluxes. The separation factors for cadmium and zinc at different concentrations of cadmium are given in Table 3.

Table 2. Separation factor of cadmium and zinc at different TOPS-99 concentrations.

[TOPS-99] (mol/m ³)	J_{Zn} (mol/m ² sec) $\times 10^{-6}$	J_{Cd} (mol/m ² sec) $\times 10^{-7}$	P_{Zn} (m/sec) $\times 10^{-6}$	P_{Cd} (m/sec) $\times 10^{-6}$	$\alpha = P_{Zn}/P_{Cd}$
10	2.060	1.025	1.346	0.1155	11.67
30	2.129	1.025	1.391	0.1155	12.0433
50	2.55	1.025	1.667	0.1155	14.458
100	5.00	1.25	3.268	0.1406	23.244
150	6.56	1.25	4.288	0.1406	30.498
200	8.348	1.25	5.456	0.1406	38.80
300	6.25	1.25	4.0849	0.1406	29.0533

Note: Experimental condition: pH = 2.25; [Zn]_{feed} = 1.53 mol/m³; [Cd]_{feed} = 8.89 mol/m³; [H₂SO₄]_{strip} = 900 mol/m³.

Effect of Acid Concentration in the Strip Solution

The stripping reaction at the membrane–strip solution side plays a vital role in the transfer of metal ion from feed side to strip side. So the stripping kinetics were studied with different concentrations of sulfuric acid (180, 540, 900, 1260, and 1800 mol/m³). All other parameters were kept constant.

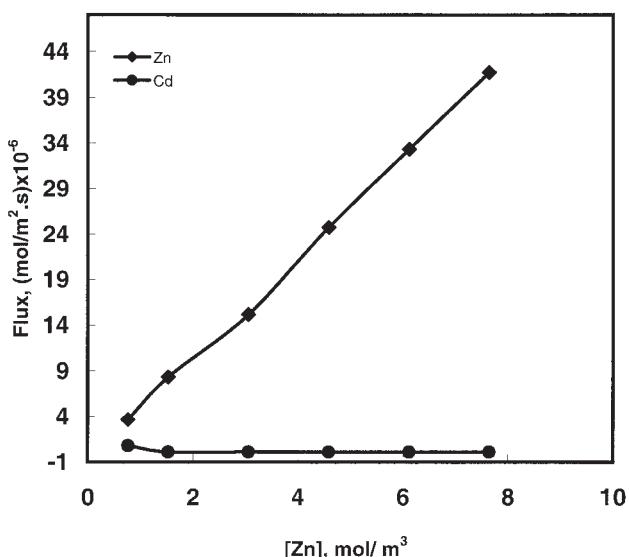
**Figure 8.** Effect of zinc ion concentration on metal ion flux.

Table 3. Separation factor of cadmium and zinc at different concentrations of zinc and cadmium in feed solution.

Sl. no	[Zn] (mol/m ³)	[Cd] (mol/m ³)	J_{Zn} (mol/m ² sec) $\times 10^{-6}$	J_{Cd} #(mol/ m ² sec) $\times 10^{-6}$	P_{Zn} (m/sec) $\times 10^{-6}$	P_{Cd} (m/sec) $\times 10^{-6}$	α
1	0.765	0.889	3.667	0.08	4.793	0.089	53.854
2	1.53	0.889	9.0	1.25	5.883	1.406	4.1842
3	3.058	0.889	15.172	1.25	4.9614	1.406	3.5388
4	4.588	0.889	24.762	1.25	5.397	1.406	3.8385
5	6.117	0.889	33.334	1.25	5.449	1.406	3.8755
6	7.65	0.889	41.739	1.25	5.456	1.406	3.8805
7	1.53	0.445	9.230	0.4	6.033	0.898	6.712
8	1.53	0.889	9.00	0.125	5.882	0.140	41.835
9	1.53	1.779	9.636	1.058	6.298	0.595	10.582
10	1.53	2.668	7.68	1.043	5.019	0.390	12.841
11	1.53	3.558	8.00	2.702	5.228	0.759	6.885
12	1.53	4.448	9.25	3.764	6.045	0.846	7.143

Note: Experimental condition: pH = 2.25; [TOPS-99]_{membrane} = 200 mol/m³; [H₂SO₄]_{strip} = 1260 mol/m³.

Figure 9 shows the plot of J_{Cd} and J_{Zn} against H_2SO_4 . It indicates that J_{Zn} increased from 7.08×10^{-6} to 9.0×10^{-6} mol/m² sec with an increase of acid concentration from 180 to 1260 mol/m³. A further increase of acid concentration to 1800 mol/m³ has no significant effect on metal ion flux.

CONCLUSIONS

Separation of zinc and cadmium was carried out by a liquid membrane technique by using TOPS-99 as mobile carrier. Also, equilibrium studies with a shake flask were carried out. The following conclusions were drawn from the above studies.

- The slope analysis studies of $\log D$ vs. equilibrium pH and $\log D$ vs. $\log [\text{TOPS-99}]$ indicate that 2 mol of the hydrogen ion and extractants were involved with 1 mol of the extracted metal species of both zinc and cadmium.
- The zinc flux increased with an increase of pH from 1.5 to 3.5, and cadmium flux increased from 3.0 to 3.5. The extraction of cadmium below pH 3.0 was minimum.

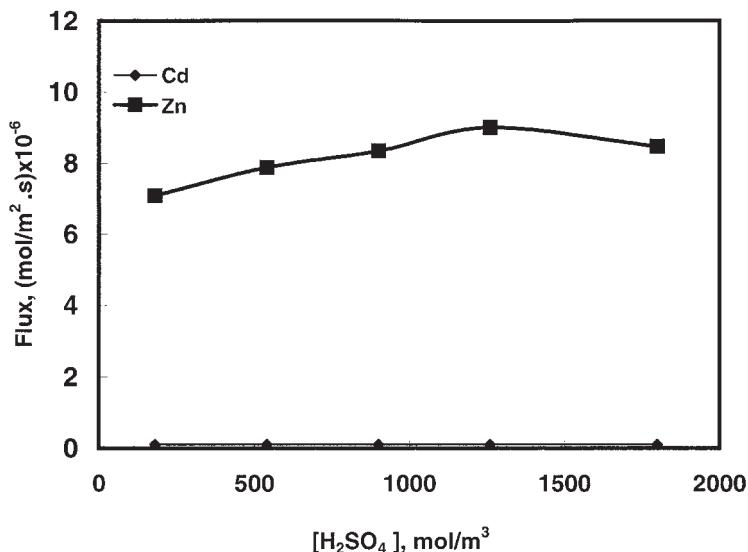


Figure 9. Effect of H_2SO_4 in strip solution on metal ion flux.

- c. Also, J_{Zn} increased with an increase of the extractant concentration up to 200 mol/m^3 and then decreased.
- d. It was observed that the maximum separation factor of 53.854 was obtained with 0.765 and 0.89 mol/m^3 of zinc and cadmium in the feed solution.
- e. The best separation of cadmium and zinc is obtained at pH 2.25, 200 mol/m^3 of TOPS-99 and 1260 mol/m^3 of H_2SO_4 in the strip solution.

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