Separation of Hg(II) from Its Aqueous Solution Using Bulk Liquid Membrane

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The focus of this paper is the separation of Hg(II) ions in anionic form from its aqueous solution through bulk liquid membrane (BLM). The efficiency of transport and extraction of Hg (II) through BLM was probed under various experimental criteria such as choice of solvent, operating conditions, and configuration of the system that would render optimal functioning of the BLM. The solvent−carrier combination, dichloroethane−trioctylamine (TOA), shows promising result in the extraction of Hg(II). Investigation was carried out at various process conditions, such as different combinations of pH, initial feed and strip phase concentrations and carrier concentration, and their effects on the equilibrium distribution as well as on the transport of Hg(II) were studied. It was observed that mercury transport phenomenon highly dependent on feed phase pH. Increase of carrier concentration and strip phase concentration increases the transport of mercury up to a certain limit. Initial feed concentration has no significant effect on the extraction of mercury up to 1 mg L^{-1}.

1. Introduction

Mercury and its compounds have always been a great concern for human health, marine organisms, and elements of biogeochemical cycle. Though the most common source of mercury in water is natural erosion of soil and ore deposits, runoff from factories and refineries can leak mercury into surface water sources too.[1] Short-term exposure to mercury in water can result in kidney damage, whereas longtime exposure can lead to impairments in neurological functioning. Oil and natural gas deposits in the oil fields are found to be contaminated with mercury.[2] Other sources of mercury are wastewater from coal-fired power plants, industrial boilers, plants producing gold, nonferrous metal and cement, typical smelters, municipal and hazardous wastes, crematoria, sewage sludge incinerator, caustic soda plant, and mercury production plant for batteries.[3] The environmental protection act[4] limits the concentration of mercury in water to 10 ppb. Hence, the separation of mercury from water and wastewater is of great importance in regard to pollution abatement and recovery. Conventional separation methods, which include ion exchange,[5] carbon adsorption,[6] precipitation,[7] etc., described the procedures for removal of mercury but they do not present the techniques of recovery. However, recovery of mercury is very important, as it would enable the scope of its reuse and that may provide economic and environmental benefits in terms of reduced disposal costs and raw material requirements. Liquid-membrane-based separation of mercury from its aqueous solutions is an attractive option for the simultaneous extraction and recovery of mercury. A liquid membrane in general is a liquid barrier separating two aqueous phases, viz. feed phase and strip phase, of components with different composition. The components can move through this liquid membrane from one phase to the other by means of diffusional process because of their own concentration gradient.[8]

A few researchers have already employed liquid membrane for separation of mercury. Shamsipur et al.[9] investigated the transport of Hg^{2+} ions through a chloroform bulk liquid membrane (BLM) containing tetrathia-12-crown-4 (TT12C4) as an ion carrier. Mercury ion is transported across the liquid membrane in the presence of ethylenediaminetetraacetic acid (EDTA) as a suitable stripping agent in the receiving phase,

the amount of mercury transported across the liquid membrane after 2 h is 99.0 ± 1.0%. Weiss et al.[10] reported the separation of mercury from wastewater by means of the emulsion liquid membrane (ELM) process employing dibutylbenzoylthiourea as carrier and Rofetan OM as surfactant. In the majority of cases of their study, more than 95% of the mercury in the external phase was transferred across the liquid membrane within three minutes. Warshawsky et al.[11] studied the separation of mercury through a liquid membrane containing bis-di (2-ethylhexyloxy) thiophosphoryl disulfide (BTPD) as carrier. Mercury transfer was found to be accelerated in presence of thiourea or thiosulfate in the receiver stripping solutions. Safavi et al.[12] concluded that methyl red can be used as carrier for the uphill transport of Hg(II) through the BLM. By using iodide as a metal acceptor and in the presence of sulfuric acid in receiving phase, the amount of mercury transport was about 90% after 180 min. Ersoz[13] achieved significant separation by developing calixarene derivative carriers for selective extraction and transport of mercury from aqueous phase. The extraction efficiency of mercury ions was found to be higher than 76%.

From the review of the literature, it is observed that the separation of Hg (II) achieved with various combination of solvent-carrier is about 90−95%. However, in all the above-mentioned cases, mercury is transported in cationic form. Li et al.[14] studied separation of Hg(II) in anionic form ($HgCl_4^{2-}$) through an emulsion liquid membrane (ELM) using TOA as carrier and toluene as the membrane phase and achieved 99% extraction in 10 min. The literature shows that less work has been done on the separation of Hg(II) in anionic form, although it has shown promising enough results to warrant further investigation in this direction to find a better membrane−carrier combination and to better understand the mechanism of such transport.

In the present work, an attempt has been made to examine the efficiency of a low viscous BLM for the extraction and recovery of Hg (II) ions as $HgCl_4^{2-}$ from its aqueous solution. A two-phase equilibrium study is carried out initially to choose a suitable membrane phase and to study the effects of parameters such as pH and carrier concentration on the equilibrium distribution of Hg(II) in the feed phase and membrane phase. On the basis of the outcomes of equilibrium study, an appropriate optimum operating condition is found out. Further experimentation is then conducted on the three-phase BLM and the

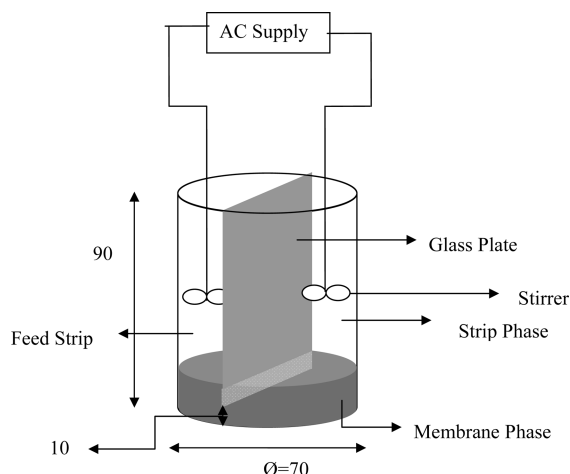* Corresponding author. Tel: + 91-361-2582252. Fax: +91-361-2582291. E-Mail: aloke@iitg.ernet.in.

Figure 1. Experimental setup for bulk liquid membrane.[8]

separation performance is assessed against various parameters such as carrier concentration, feed concentration, and strip phase concentration.

2. Theoretical Backgrounds

In the present work, an alkaline extractant, Tri-*n*-octylamine (TOA) is found to be the most promising carrier agent (discussed later in section 4.1.1) and is employed to transport Hg(II) ions through the organic phase of the BLM system. TOA reacts with the Hg(II) only when it is in anionic complex form. In this mechanism, the transport of mercury and the H^+ ions occur in the same direction by virtue of the concentration gradient of the ions across the aqueous−organic−aqueous phases. This transport mechanism is called coupled cotransport. The mechanism of cotransport mode is discussed elsewhere in the literature.[8] The reaction mechanism for the separation of mercury is as follows.
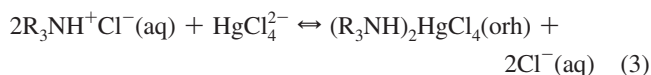
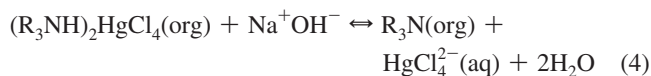Complexation of Hg (II) by chloride ions in the feed solution

$$Hg^{+2} + 4Cl^- \leftrightarrow HgCl_4^{-2} \tag{1}$$

Reaction of TOA (denoted as R_3N) at the feed/membrane interface with HCl in the feed solution

$$R_3N(org) + H^+Cl^-(aq) \leftrightarrow R_3NHCl(org) \tag{2}$$

In the feed, $HgCl_4^{2-}$ exchange with Cl^- of $R_3NH^+Cl^-$ in the membrane phase

$$2R_3NH^+Cl^-(aq) + HgCl_4^{2-} \leftrightarrow (R_3NH)_2HgCl_4(orh) + 2Cl^-(aq) \tag{3}$$

The stripping reaction

$$(R_3NH)_2HgCl_4(org) + Na^+OH^- \leftrightarrow R_3N(org) + HgCl_4^{2-}(aq) + 2H_2O \tag{4}$$

3. Experimental Apparatus and Method

3.1. Apparatus for Experimental Studies. A bulk type liquid membrane cell shown in Figure 1[8] was used in this study. All experiments were carried out at ambient temperature (303 K). The BLM experimental setup comprises of a glass beaker of dimension 70 mm ID and 90 mm height, two stirrers of impeller length 12 mm, and two regulated motors (sewing motor, model YM-50). A thin glass plate of thickness 2 mm is placed

inside the cell beaker with to create two leak proof compartments. A clearance of 10 mm is kept at the bottom of beaker while fixing the glass plate. The heavier phase, i.e., organic phase, was placed first at the bottom of the beaker in such a way that the organic liquid fills up the bottom clearance completely and the glass plate gets immersed slightly in the organic liquid. The aqueous phases (source and receiving phases) were then placed carefully in the compartments in such a way that the aqueous solutions remain isolated from each other and float above the organic phase and they do not get mixed accidentally. A test was conducted using CCl_4 as solvent to ensure no leakage from one compartment to the other. Details can be seen elsewhere.[8] The clearance at the bottom of the cell enables the transport of solute from feed phase to strip phase through membrane phase. Both the aqueous solutions were stirred continuously by motor driven stirrers and their speeds were regulated by voltage controllers to prevent the undesired mixing. Care was also taken to prevent disturbance of membrane phase due to uncontrolled stirrer speed in the aqueous phases, which might form emulsions between the organic−aqueous phases.

3.2. Analytical Instruments. The determination of Hg(II) concentration in solution was done by cold vapor atomic absorption spectrometry (Varian, model AA 240 FS). The pH measurements were carried out with a CP 901 digital pH meter.

3.3. Chemicals and Reagents. All reagents used in this experiment were of GR grade. Aqueous solutions were prepared by using Milli-Q deionized water (Millipore, USA). Mercury chloride was obtained from Merck (India), trioctylphosphine oxide (TOPO) from Alfa Aesar (Lancaster), 2-ethyl hexanol from Loba Chemical (India), and trioctylamine (TOA) was procured from Merck (Germany). All other chemicals such as 1,2-dichloroethane, carbon tetrachloride, hydrochloric acid, toluene, *n*-heptane, cyclohexane, hexane, sodium hydroxide, and sodium bicarbonate were obtained from Merck (India).

The stock solution of mercury was prepared by dissolving 1.35 mg of mercury chloride in 1000 mL of Milli-Q deionized water. The feed phases of desired concentration were prepared from the stock solution by diluting with water. The stripping phase was prepared by dissolving the required amount of sodium hydroxide (or sodium bicarbonate) in 100 mL of Milli-Q deionized water. By dissolving the appropriate amount of TOA in different pure solvents, the organic phase is developed for equilibrium studies and for three-phase studies.

3.4. Two-Phase Equilibrium Distribution. Two-phase equilibrium distribution studies provide knowledge of best performing conditions. Ten ml stock solution of mercury chloride was added to equal volume of organic phase. The mixture was then continuously agitated at constant speed (100 rpm) for about 6 h on an orbital shaker. Hg(II) concentration in aqueous phase was then determined by cold vapor atomic absorption spectrometry at 253.7 nm. Assuming no loss of mercury in the process, the Hg(II) concentration in membrane phase is then determined by calculating the balance amount of Hg(II) from initial condition. Distribution coefficient (i.e., separation factor) of Hg (II) (*m*) was then calculated via method described in ref 8. The experiments were repeated with different solvents such as *n*-heptane, hexane, toluene, dichloroethane, and cyclohexane. Organic solvent with best performance was identified by observing the distribution coefficient and the same was picked out for advanced studies to learn the effects of carrier concentration and pH on the equilibrium Hg(II) distribution.

3.5. Three-Phase Experiment. The three-phase experiments were carried out in the BLM setup described in section 3.1.

Table 1. Effects of Various Solvents and Carriers on the Equilibrium Distribution Coefficient of Mercury

| solvent	distribution coefficient (m)			
	no carrier	TOA	TOPO	2-ethylhexanol
hexane	5.6095	119.625	29.156	0.87925
dichloroethane	2.9443	26.9710	0.9693	9.0520
cyclohexane	1.0423	6.0181	5.2057	2.6142
toulene	0.4745	4.6268	0.6273	1.2209
n-heptane	0.8792	2.1028	1.3450	3.0631

Area of membrane-aqueous phase interface on either compartment of the BLM setup was about 1820 mm². Equal volume of liquid (50 mL) have been taken in all three phases of BLM viz feed, receiving, and organic phases. Uninterrupted stirring was provided to maintain uniform bulk concentration in the aqueous phases. The pH was constantly monitored and adjusted by adding hydrochloric acid as and when required. For further analysis, 1 mL of each aqueous phase was collected at every 20 min.

4. Results and Discussion

4.1. Two-Phase Equilibrium Study. A suitable liquid membrane of high separation factor was identified by performing the experiments as per procedures reported in the section 3.4, through two phase equilibrium studies. With the selected solvent, effects of various process parameters such as carrier concentration and pH on the equilibrium distribution of Hg(II) have also been examined.

4.1.1. Choice of Solvent−Carrier Combination. In liquid membrane separation processes, the selection of solvent (i.e., membrane phase) has always been a major area to be looked into. The ideal solvent must possess certain properties such as immiscibility with the aqueous phase, low viscosity, low volatility, and high distribution coefficient.[15,16] Various solvents, viz. n-heptane, hexane, toluene, cyclohexane, and dichloroethane, were employed and tested for the extraction of Hg(II). Extraction of metal ions by liquid membrane is enhanced by carrier mediated transport. Therefore, careful integration of a suitable carrier into the liquid membrane is also very important to enhance the effective solubility of the metal species in the membrane phase that sharply increases the metal flux. As reported in the literature with TOA as carrier the separation of mercury achieved was 99% when it is in anionic form. Therefore, in this study, besides TOA, two other carriers used in the transport of anions i.e., trioctylphosphine oxide (TOPO) and 2-ethylhexanol, were employed to study their efficiency in the transport of mercury. The results are reported in Table 1 in terms of distribution coefficients.

The distribution coefficients (m) of Hg(II) in these solvents in absence of any carrier are also reported in table in order to compare the performance of carrier agent. It is observed that transport of mercury is not significant when n-heptane and toulene were used as membrane. Significant separation is observed with hexane, followed by dichloroethane and cyclohexane. Because vapor pressures of hexane and cyclohexane at 25 °C are 201 and 130 mbar respectively, they prove to be more volatile than the dichloroethane having vapor pressure 105 mbar at 25 °C. Therefore, hexane and cyclohexane have not been considered, and dichloroethane has been selected as the suitable solvent for the subsequent studies. It is observed that TOA as carrier shows promising result in the extraction of Hg(II) from aqueous solutions when compared to other carriers used in this study (i.e., TOPO and 2-ethylhexanol). Hence, dichloroethane−TOA duo has been selected as the solvent−carrier combination in the BLM for further experiments.
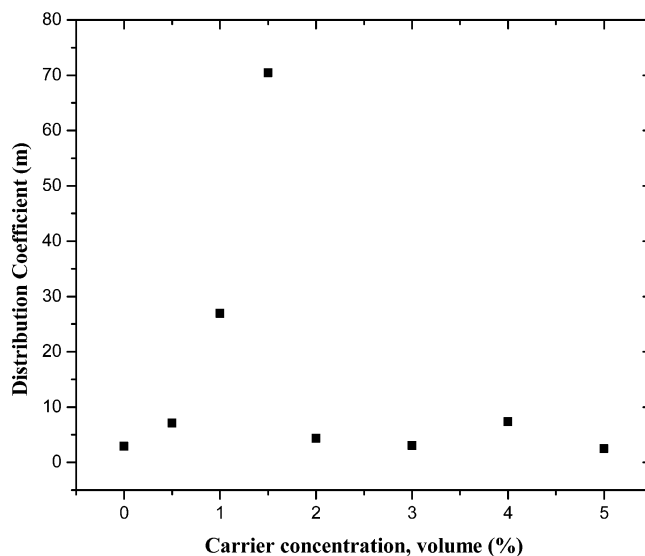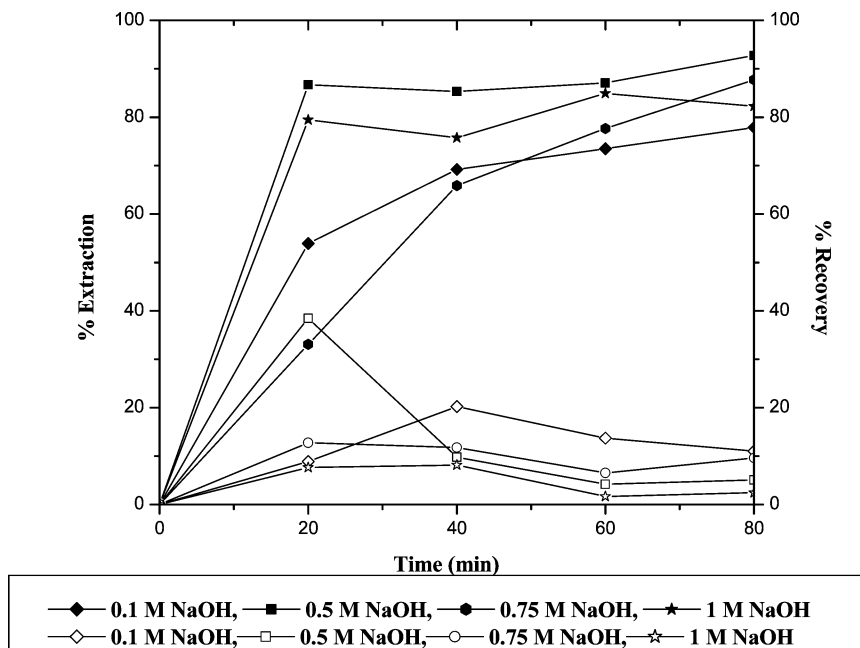

Figure 2. Effect of carrier concentration on the equilibrium distribution of mercury, $V_F = V_O = 10$ mL, time = 6 h, agitation speed = 100 rpm, temperature = 303 K.
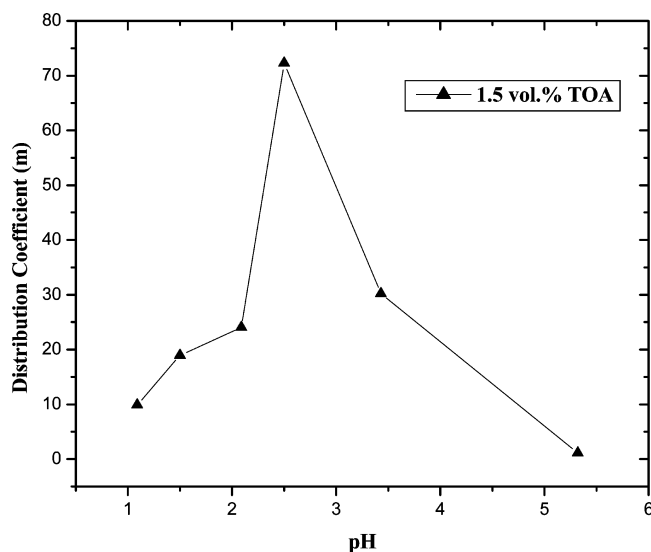
Figure 3. Effect of pH on the equilibrum distribution on Hg(II) $V_F = V_O = 10$ mL, carrier (TOA) concentration = 1.5 vol %, time = 6 h, speed = 100 rpm, temperature = 303 K.

4.1.2. Effect of Carrier Concentration. The effect of concentration of TOA (mobile carrier) in the organic phase on the equilibrium distribution of Hg (II) was studied in the range of 0−5 vol %. The results are reported in Figure 2. It is found that the optimum carrier concentration attains at 1.5 vol % for the present case. It is observed that when the carrier is imparted to the membrane phase, the formation Hg (II)−amine complex (reaction 3) in the membrane phase takes place, which thereby increases the mass transfer through the interface of feed-membrane and eventually yields higher distribution coefficient.

The optimality in the distribution coefficient at 1.5 vol % carrier concentration indicates the saturation capacity of the membrane phase for the complex. It is also observed that the saturation capacity of the complex in the membrane phase reduces due to the presence of excess amount of carrier. This downfall of membrane phase capacity for the complex is reflected from the lower distribution coefficient obtained at higher carrier concentration. However, the optimum carrier concentration should also depend on the ratio of membrane-

Figure 4. Effect of strip phase concentration on extraction and recovery of Hg(II), $C_{Hg} = 500\ \mu g\ L^{-1}$, $V_F = V_O = V_S = 50$ mL, carrier (TOA) concentration = 1.5 vol %, pH = 2.5, time = 80 min, speed = 400 rpm, temperature = 303 K (filled symbols represent extraction and empty symbols represent recovery).

phase volume to feed-phase volume and the initial feed-phase concentration.[8]

4.1.3. Effect of pH. To understand the effect of pH of the feed phase on separation, we performed two-phase equilibrium experiments with pH variation in the range of 1 to 6 in the presence of carrier, TOA. The experimental results are depicted in Figure 3. It is observed that at pH 2.5, the distribution coefficient of Hg(II) is the maximum. The reaction mechanism (section 2) indicates that acidic condition is needed for the protonation of TOA as it binds with Hg(II) in anionic form only, which in turn justifies the maximum transport of Hg(II). At much lower pH, an increase in proton concentration in the feed solution forms H_2HgCl_4, which does not dissociate enough to form adequate $HgCl_4^{-2}$ that forms the complex with the cation (R_3NH^+).[17,18] Hence, extraction is not effective at very low pH. Again, at much higher pH, protonation of TOA to form R_3NH^+ that makes the complex with $HgCl_4^{-2}$ becomes inadequate. Therefore, pH 2.5 is selected for the subsequent studies. Similar observation has been reported in our recent work on separation of mercury using supported liquid membrane.[18]

4.2. Three-Phase Studies. The BLM setup discussed in section 3.1 was used to study the influence of various process variables, such as feed- and strip-phase concentrations and carrier concentration on the transport of Hg (II). The effects of process variables on the transport of Hg (II) were estimated in terms of % extraction and % recovery, calculated as follows

$$\% \text{ extraction} = \frac{C_0 - C_t}{C_0} \times 100 \qquad (5)$$

$$\% \text{ recovery} = \frac{C_{st}}{C_0} \times 100 \qquad (6)$$

Where C_0 is the concentrations of Hg(II) in feed at time $t = 0$ and C_t is the concentration of mercury at time t. C_{st} is the concentration of Hg (II) in strip phase at a particular time.

4.2.1. Effect of Strip Phase Concentration. The effect of NaOH concentration on the transport of Hg(II) was studied in

the range of 0.1−1 M. The results of both extraction and recovery are shown in Figure 4. The NaOH behaves as a strip-extractant in the stripping phase. From Figure 4, it is observed that the percentage extraction after 80 min lies between 78 and 94% for variation of NaOH concentration between 0.1 and 1 M. Percentages extraction after 80 min for 0.5−1 M NaOH are almost the same, around 90%. According to the Le Chatelier principle, an increase in hydroxyl ions results in faster decomposition of mercury complex and removal of protons from amine molecule, which results in higher recovery. The percentage recovery is found to increase for some time and then starts decreasing. The apparent downfall of recovery at higher concentration (Figure 4) is due to precipitation of mercury as HgO in the strip phase.[14,18] However, both percentage and extraction and recovery were found to be more for 0.5 M NaOH strip solution.

4.2.2. Effect of Carrier Concentration. The actual transport phenomena in BLMs are quite complex and are strongly influenced by the both carrier concentration and physiochemical properties of carrier in the membrane phase. The percentage extraction of mercury ions increased with increase in TOA concentration from 1 to 1.5% (v/v), as shown in Figure 5. However, when the TOA concentration exceeded 1.5% (v/v), both % extraction and % recovery decreased (Figure 5). This phenomenon occurred because of the enhanced viscosity of membrane phase with increase in TOA concentration. Higher viscosity retards the diffusion speed of the complex and affects the mass transfer process through the membrane phase.[8,19] Hence, similar to the two phase study, 1.5% (v/v) TOA has been found to be the optimum carrier concentration for the three-phase study, too.

4.2.3. Effect of Initial Feed Concentration. The relation between the feed phase concentration and the percentage of extraction and recovery of mercury is shown in Figure 6. The effect of initial feed concentration was examined over concentration range of 200 ppb to 4 ppm. From the figure it is seen that with increase in feed concentration, the initial rate of transport of Hg(II) increases. But the feed concentration above
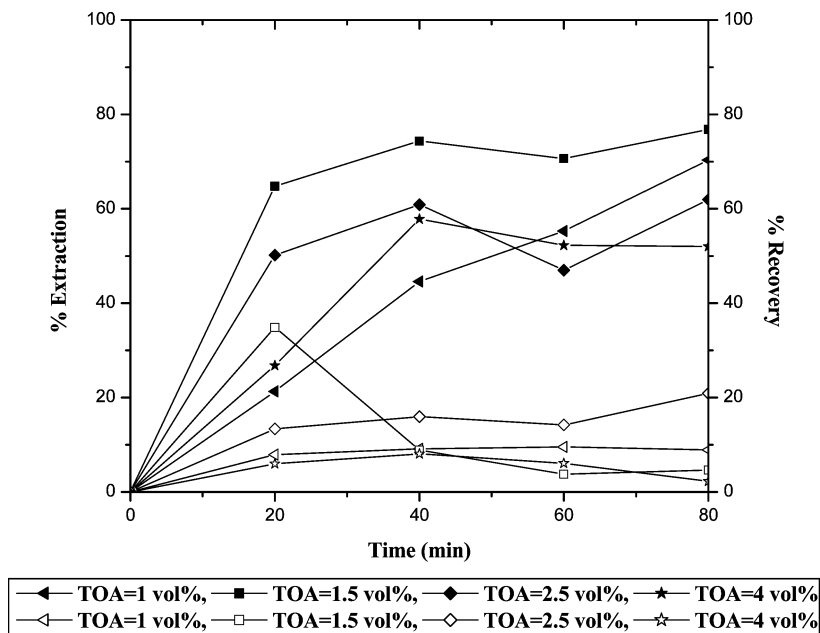
Figure 5. Effect of carrier concentration on extraction and recovery of Hg(II), $C_{Hg} = 500~\mu g~L^{-1}$, $V_F = V_O = V_S = 50$ mL, strip concentration = 0.5 M, pH = 2.5, time = 80 min, speed = 400 rpm, temperature = 303 K (filled symbols represent extraction and empty symbols represent recovery).
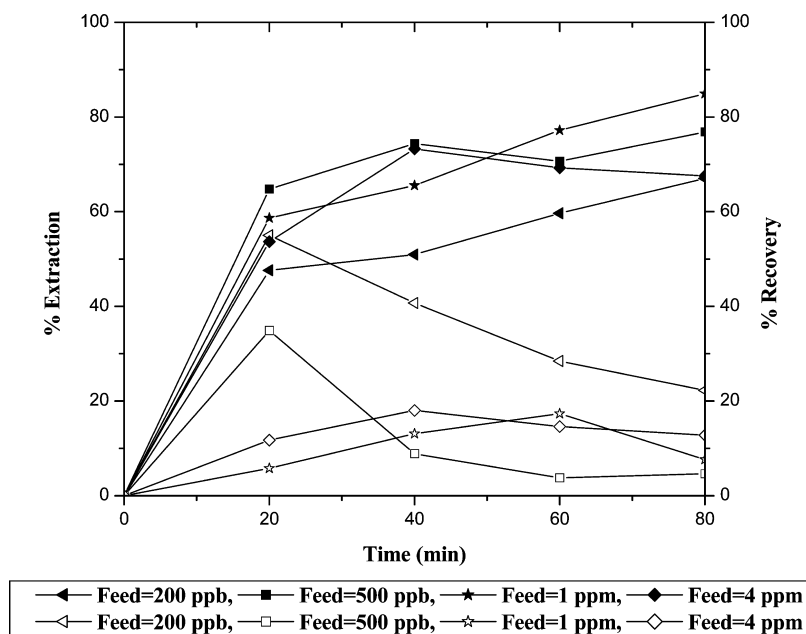
Figure 6. Effect of initial feed concentration on extraction and recovery of Hg (II), $V_F = V_O = V_S = 50$ mL, strip concentration = 0.5 M, carrier (TOA) concentration = 1.5 vol %, pH = 2.5, time = 80 min, speed =400 rpm, temperature = 303 K (filled symbols represent extraction and empty symbols represent recovery).

1 ppm shows a declining trend, which may be attributed to the fact that at higher concentration, complex of Hg(II) ions get accumulated at feed−membrane interface and consequently leads to a longer diffusion path of complex and/or lowers the complexation rate. Observing the percentage of recovery behavior (Figure 6), it can be concluded that lower concentration of Hg(II) in feed leads to a higher recovery. But as the concentration of Hg(II) in feed increases, the recovery decreases because of the slower diffusion of complex from feed to strip as the path becomes longer. However, on adequate supply of Hg (II) at the strip-side, precipitation reaction is enhanced. This can be explained by the combined effect of diffusion through the membrane phase and the precipitation of mercury to HgO at the strip side. At lower initial feed concentration, the diffusion

rate of the complex through the membrane is higher and at the same time provides inadequate Hg(II) ion to facilitate the precipitation reaction.

5. Conclusion

Suitable solvent−carrier combination is selected for the extraction and recovery of Hg(II) from contaminated water. Optimum conditions for the cotransport of mercury ion through a BLM are found out. The three-phase transport results are suitably explained with the help of the reaction and transport mechanism. The solvent−carrier combination, hexane−TOA, has the highest distribution coefficient for Hg(II). Because of highly volatile nature of hexane, dichloroethane−TOA has been

chosen as the best solvent-carrier combination for the transport of Hg(II). Feed-phase pH affects the mercury extraction process significantly. Precipitation of Hg(II) as HgO in alkaline solution decreases the transport efficiency of mercury at higher strip phase concentration. The present system is found to be independent of initial mercury concentration up to 1 mg L^{-1}. The optimized conditions for transport of mercury through dichloroethane-TOA BLM is found as pH of 2.5 in feed phase and carrier concentration of 1.5 vol % in liquid membrane, and 0.5 M NaOH in the stripping solution. At optimum conditions, extraction of mercury achieved is about 94% and that of recovery is about 38%.

Nomenclature

A	membrane area, cm^2
V_F	volume of feed phase, mL
V_S	volume of strip phase, mL
V_O	volume of organic phase, mL
C_{st}	concentration of Hg (II) in strip phase at particular time, mg L^{-1}
C_{Hg}	concentration of mercury, mg L^{-1}
C_0	concentration of Hg(II) in feed phase at time $t = 0$, mg L^{-1}
C_t	concentration of Hg(II) in feed phase at time $t = 0$, mg L^{-1}

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