



## Transport Study of Some Transition Metal Cations Through a Bulk Liquid Membrane Using Dicyclohexyl-18-crown-6 as Carrier

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Selectivity and transport efficiency for competitive transport of some transition metal cations containing Zn(II), Cd(II), Cr(III), Co(II), Ag(I), Pb(II) and Cu(II) across different bulk liquid membranes have been determined by dicyclohexyl-18-crown-6 as carrier. The membrane solvents include: dichloromethane, chloroform, 1,2-dichloroethane, nitrobenzene and chloroform-dichloromethane, chloroform-dichloroethane binary mixed solvents. The effect of some surfactants on transport performance of the metal cations through bulk liquid membranes were also studied. The results show that the selectivity and the efficiency of transport for these heavy metal cations change with the nature of the organic solvents which were used as liquid membranes in these experiments. A good transport efficiency and high selectivity was observed for Pb(II) cation under the experimental conditions.

**Key Words:** Liquid membrane transport, Transition metal cations, Dicyclohexyl-18-crown-6, Dichloromethane, 1,2-Dichloroethane.

### INTRODUCTION

Heavy metal contamination exists in the vast majority of industrial effluents and waste waters, such as mining effluents, etching or pickling baths, dilute leaching solutions generated during hydrometallurgy, electroplating rinse liquors, etc. As heavy metals are not biodegradable they tend to accumulate in living organisms causing various diseases and disorders. So extraction techniques for the removal and recovery of heavy metals are of increasing interest with a variety of processes being studied. Compared with solvent extraction, liquid membranes have shown great potential since they combine the process of extraction and stripping in a single unit operation<sup>1,2</sup>.

Liquid membranes have been successfully used to treat aqueous streams contaminated with heavy metal ions like copper, zinc, cadmium, nickel, mercury and lead<sup>3-9</sup>.

Bulk liquid membrane (BLM) is one of the simplest designs for performing liquid membrane processes, which has been studied for pre-concentration and separation of different metal ions with a variety of liquid membrane phase<sup>10-15</sup>.

Ion extraction by liquid membranes is normally facilitated by carrier mediated transport, in which an ion exchange reagent is incorporated into the membrane phase in order to carry the diffusing species from source phase across the membrane to the receiving phase. To utilize a system in separation chemistry, a receptor should have high extraction efficiency as well as high decomplexation efficiency towards a specific

metal ion. A receptor is of no use if it binds very strongly to the metal ion and it do not undergo decomplexation by any methods. This would waste the receptor after a single use and it would be difficult to recycle the receptor. Hence, the decomplexation of metals is one of the important factors in the solvent extraction. The present investigation is an investigation on the effect of the nature of the cation and crown ether and also the solvent properties on the liquid membrane transport and we report the results of liquid membrane transport of some of the transition metal cations using dicyclohexyl-18-crown-6 as an ion carrier in various liquid membranes.

### EXPERIMENTAL

Dicyclohexyl-18-Crown-6 (Merck) (Fig. 1), cobalt(II) nitrate (BDH), chromium(III) nitrate (BDH), copper(II) nitrate (BDH), zinc(II) nitrate (Merck), silver(I) nitrate (Merck) and cadmium(II) nitrate (Riedel), lead(II) nitrate (BDH), sodium acetate, sodium hydroxide, palmitic acid (all from Riedel) and stearic acid (BDH) and oleic acid (Merck) were used without further purification. The solvents chloroform (BDH), dichloromethane, 1,2-dichloroethane and nitrobenzene all from Merck with the highest purity were used as liquid membranes. Acetic acid (Merck), formic acid (Merck) and nitric acid (BDH) were used as received. All aqueous solutions were prepared using double distilled deionized water.

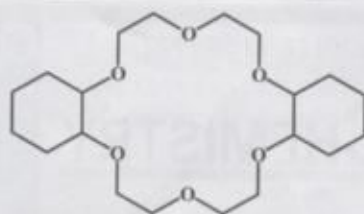


Fig. 1. Structure of dicyclohexyl-18-crown-6

**Procedure:** The transport experiments employed standardized concentric cells in which the aqueous source phase (10 cm<sup>3</sup>) and receiving phase (30 cm<sup>3</sup>) separated by an organic phase (50 cm<sup>3</sup>). Bulk membrane transport studies were carried out by stirring the organic phase of bulk liquid membrane cell at a constant slow speed (at 250 rpm) so that the interfaces between the organic membrane and two aqueous phases remained flat and well defined. The aqueous source phase consisted of a buffer solution at pH 4.9 ± 0.1 (6.95 cm<sup>3</sup> of 2 mol dm<sup>-3</sup> sodium acetate solution and 3.05 cm<sup>3</sup> of 2 mol dm<sup>-3</sup> acetic acid made up to 100 cm<sup>3</sup>)<sup>16</sup> containing an equimolar mixture of the metal ions, each at the concentration of 1.0 × 10<sup>-2</sup> mol dm<sup>-3</sup>. The organic phase contained the macrocycle at 10<sup>-3</sup> mol dm<sup>-3</sup>. The receiving phase consisted of a buffer solution at pH 3.0 ± 0.1 (56.6 cm<sup>3</sup> of 1 mol dm<sup>-3</sup> formic acid and 10 cm<sup>3</sup> of 1 mol dm<sup>-3</sup> sodium hydroxide) made<sup>16</sup> up to 100 cm<sup>3</sup>. All transport runs were terminated after 24 h and atomic absorption spectroscopy was used to determine the amount of each metal ion transported over this period. Both source and receiving phases were analyzed (using a Shimadzu spectra AA-670 spectrometer) after each transport run. This also enabled the amount of the metal cations remaining in the membrane phase to be calculated.

## RESULTS AND DISCUSSION

In order to test the selectivity of the system under investigation, the transport of the seven metal cations, were carried out by placing equimolar amounts of the cations in the source phase. The results for competitive transport of the cations from

a source phase at pH 4.9 through four liquid membranes: chloroform, dichloromethane (DCM), 1,2-dichloroethane (DCE) and nitrobenzene (NB) containing dicyclohexyl-18-crown-6 (DCH18C6) into acidic receiving phase (pH 3) are listed in Table-1. The results obtained in the case of chloroform:1,2-dichloroethane and chloroform-dichloro-methane binary mixtures containing dicyclohexyl 18-crown-6 as membrane phase are listed in Tables 2 and 3, respectively. A pH gradient was utilized to facilitate the transport of the metal cations across the membrane by counter transport of protons. The results in presence of surfactants in organic phase are listed in Table-4. The graphical results for competitive transport of cations across organic liquid membranes are shown in Figs. 2 and 3. The change in transport rate of Pb<sup>2+</sup> cation *versus* the mole fraction of 1,2-dichloroethane in chloroform:1,2-dichloroethane and dichloromethane in chloroform-dichloromethane binary solvent are shown in Figs. 4 and 5.

It is well known that the liquid membrane technique contains two processes in a single stage: extraction of an ion from the aqueous donor solution to the organic phase containing the carrier molecules (membrane) and reextraction of this ion from the membrane to the aqueous acceptor phase. The overall transport process consists of a mixture diffusion steps and complexation/decomplexation reactions at two independent and possible different interfaces.

### Effect of carrier structure on the cation transport:

Carrier mediated transport through liquid membrane is well known as one of the most powerful tools for concentration, separation and recovery. In the carrier-mediated transport, the carrier plays the role of a phase transfer catalyst. It forms a lipophilic extractable complex diffusing in the membrane and then, releases the analyte into the receiving phase<sup>17</sup>.

There has been considerable interest in the use of proton ionizable carriers for the transport of metal cations between two aqueous solutions through an organic membrane<sup>18</sup>. These pH-regulated ligands described until now present the fact that transfer of the counter-anion from the aqueous phase into the organic medium is avoided. Macrocyclic compounds such as

TABLE-I  
DATA FOR COMPETITIVE TRANSPORT OF SEVEN METAL CATIONS ACROSS BULK CHLOROFORM, DICHLOROMETHANE, 1,2-DICHLOROETHANE AND NITROBENZENE MEMBRANES BY DICYCLOHEXYL-18-CROWN-6 AT 25 °C

Membrane	Co(II)	Cr(III)	Cu(II)	Zn(II)	Ag(I)	Cd(II)	Pb(II)
Chloroform							
%(Transport receiving) <sup>a</sup>	—	—	—	—	2.05	—	33.68
%(Membrane) <sup>b</sup>	6.33	20.01	15.58	3.21	13.99	—	65.92
J (mol per 24 h) <sup>c</sup>	—	—	—	—	0.25	—	4.20
Dichloromethane							
%(Transport receiving) <sup>a</sup>	—	—	—	—	15.51	—	28.28
%(Membrane) <sup>b</sup>	10.33	21.23	23.91	24.85	46.39	—	70.86
J (mol per 24 h) <sup>c</sup>	—	—	—	—	1.94	—	3.53
1,2-Dichloroethane							
%(Transport receiving) <sup>a</sup>	—	—	—	—	—	—	21.50
%(Membrane) <sup>b</sup>	10.68	20.82	18.69	41.22	43.87	4.65	40.07
J (mol per 24 h) <sup>c</sup>	—	—	—	—	—	—	2.69
Nitrobenzene							
%(Transport receiving) <sup>a</sup>	—	—	—	—	2.82	—	22.86
%(Membrane) <sup>b</sup>	16.12	26.52	25.30	58.64	21.77	—	50.26
J (mol per 24 h) <sup>c</sup>	—	—	—	—	0.35	—	2.86

<sup>a</sup>Per cent of total metal cations in the receiving phase after 24 h; <sup>b</sup>Per cent of total metal cations in the membrane phase after 24 h;

<sup>c</sup>All J values are × 10<sup>5</sup>

TABLE-2  
DATA FOR COMPETITIVE TRANSPORT OF SEVEN METAL CATIONS ACROSS BULK CHLOROFORM-  
1,2-DICHLOROETHANE BINARY MIXED SOLVENT MEMBRANES BY DICYCLOHEXYL-18-CROWN-6 AT 25 °C

Membrane	Co(II)	Cr(III)	Cu(II)	Zn(II)	Ag(I)	Cd(II)	Pb(II)
Pure CHCl <sub>3</sub>							
%(Transport receiving) <sup>a</sup>	—	—	—	—	2.05	—	33.68
%(Membrane) <sup>b</sup>	6.33	20.01	15.58	3.21	13.99	—	65.92
J (mol per 24 h) <sup>c</sup>	—	—	—	—	0.25	—	4.20
75 % CHCl <sub>3</sub> -25 % 1,2-DCE							
%(Transport receiving) <sup>a</sup>	—	—	—	—	0.38	—	29.61
%(Membrane) <sup>b</sup>	30.40	20.52	24.76	38.89	72.70	28.46	70.01
J (mol per 24 h) <sup>c</sup>	—	—	—	—	0.05	—	3.70
50 % CHCl <sub>3</sub> -50 % 1,2-DCE							
%(Transport receiving) <sup>a</sup>	—	—	—	—	0.80	—	25.04
%(Membrane) <sup>b</sup>	29.62	19.71	25.86	74.31	73.8	24.17	72.07
J (mol per 24 h) <sup>c</sup>	—	—	—	—	0.10	—	3.13
25 % CHCl <sub>3</sub> -75 % 1,2-DCE							
%(Transport receiving) <sup>a</sup>	—	—	—	—	2.16	—	28.41
%(Membrane) <sup>b</sup>	17.02	7.14	16.12	44.17	75.08	14.62	70.39
J (mol per 24 h) <sup>c</sup>	—	—	—	—	0.27	—	3.55
Pure 1,2-DCE							
%(Transport receiving) <sup>a</sup>	—	—	—	—	—	—	21.50
%(Membrane) <sup>b</sup>	10.68	20.82	18.69	41.22	43.87	4.65	40.07
J (mol per 24 h) <sup>c</sup>	—	—	—	—	—	—	2.69

<sup>a</sup>Per cent of total metal cations in the receiving phase after 24 h; <sup>b</sup>Per cent of total metal cations in the membrane phase after 24 h;

<sup>c</sup>All J values are  $\times 10^6$

TABLE-3  
DATA FOR COMPETITIVE TRANSPORT OF SEVEN METAL CATIONS ACROSS BULK DICHLOROMETHANE-  
1,2-DICHLOROETHANE BINARY MIXED SOLVENT MEMBRANES BY DICYCLOHEXYL-18-CROWN-6 AT 25 °C

Membrane	Co(II)	Cr(III)	Cu(II)	Zn(II)	Ag(I)	Cd(II)	Pb(II)
Pure CHCl <sub>3</sub>							
%(Transport receiving) <sup>a</sup>	—	—	—	—	2.05	—	33.68
%(Membrane) <sup>b</sup>	6.33	20.01	15.58	3.21	13.99	—	65.92
J (mol per 24 h) <sup>c</sup>	—	—	—	—	0.25	—	4.20
75 % CHCl <sub>3</sub> -25 % DCM							
%(Transport receiving) <sup>a</sup>	—	0.94	—	—	0.53	—	27.05
%(Membrane) <sup>b</sup>	26.22	2.58	16.45	62.80	69.45	21.48	72.26
J (mol per 24 h) <sup>c</sup>	—	0.12	—	—	0.07	—	0.69
50 % CHCl <sub>3</sub> -50 % DCM							
%(Transport receiving) <sup>a</sup>	—	—	—	—	1.71	—	0.51
%(Membrane) <sup>b</sup>	29.61	19.71	26.72	59.60	73.87	13.42	99.49
J (mol per 24 h) <sup>c</sup>	—	—	—	—	0.21	—	0.06
25 % CHCl <sub>3</sub> -75 % DCM							
%(Transport receiving) <sup>a</sup>	—	—	—	—	2.40	—	0.51
%(Membrane) <sup>b</sup>	29.61	4.52	21.58	59.60	73.18	18.12	99.49
J (mol per 24 h) <sup>c</sup>	—	—	—	—	0.30	—	0.06
Pure DCM							
%(Transport receiving) <sup>a</sup>	—	—	—	—	15.51	—	28.28
%(Membrane) <sup>b</sup>	10.33	21.23	23.91	24.85	46.39	—	70.86
J (mol per 24 h) <sup>c</sup>	—	—	—	—	1.94	—	3.53

<sup>a</sup>Per cent of total metal cations in the receiving phase after 24 h; <sup>b</sup>Per cent of total metal cations in the membrane phase after 24 h;

<sup>c</sup>All J values are  $\times 10^6$

crown ethers, azacrowns, cryptands and calixarenes have been well known for selective recognition of specific metal ions or molecules<sup>19-24</sup>.

In the present study, we used dicyclohexyl-18-crown-6 as a carrier and the binding ability of this ligand for some transition metal cations was examined by competitive bulk liquid membrane transport experiments. The results show that dicyclohexyl-18-crown-6 preferentially transport the Pb<sup>2+</sup> cation. As expected, the nature of the macrocyclic crown ether used as carrier in the organic membrane phase was found to have a pronounced effect on the permeability of the membrane

system designed for Pb<sup>2+</sup> transport. It seems that according to the hard and soft acid-base concept, the Pb<sup>2+</sup> cation can form a more stable complex with oxygen donor atoms of dicyclohexyl-18-crown-6 as a hard base. Moreover, it bears a high charge density which results in a strong interaction with this macrocyclic ligand.

**Effect of solvent upon fluxes and selectivity in bulk liquid membrane transport:** However, the degree of selectivity in transport, as in solvent extraction, is found to be strongly influenced by the identity of organic solvent, the thickness of the unstirred boundary layers in the membrane (*i.e.*, the diffusion

TABLE-4  
EFFECT OF SURFACTANTS ON LEAD CATION TRANSPORT  
ACROSS ORGANIC SOLVENT SYSTEM AS BULK MEMBRANE  
WITH DICYCLOHEXYL-18-CROWN-6 AS IONOPHORE AT 25 °C

Membrane	Fatty acid	Per cent (receiving) <sup>a</sup>	Per cent (membrane) <sup>b</sup>	J (mol/24 h) <sup>c</sup>
DCM	—	28.28	70.86	3.53
	Stearic acid	29.21	70.38	3.65
	Palmitic acid	32.22	62.83	4.03
	Oleic acid	19.34	51.33	2.42
CHCl <sub>3</sub>	—	33.68	65.92	4.20
	Stearic acid	27.62	71.55	3.45
	Palmitic acid	31.21	67.16	3.90
	Oleic acid	31.68	67.76	3.96
NB	—	22.86	50.26	2.86
	Stearic acid	0.53	96.31	0.07
	Palmitic acid	25.15	71.24	3.14
	Oleic acid	20.85	48.55	2.61
DCE	—	21.50	40.07	2.69
	Stearic acid	13.48	10.16	1.68
	Palmitic acid	23.80	46.09	2.97
	Oleic acid	26.22	71.58	3.28

<sup>a</sup>Per cent of total metal cations in the receiving phase after 24 h;

<sup>b</sup>Per cent of total metal cations in the membrane phase after 24 h;

<sup>c</sup>All J values are  $\times 10^{-6}$

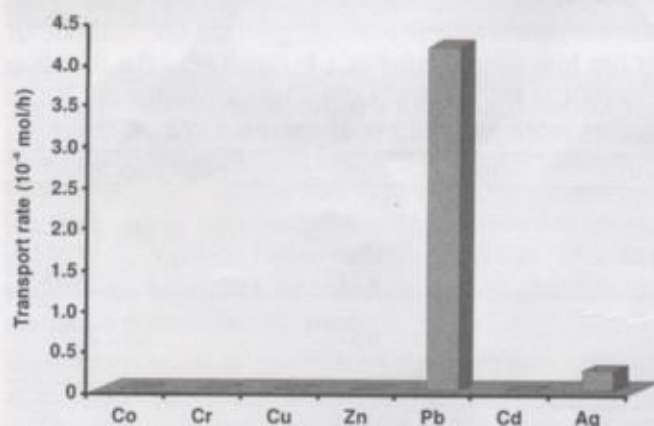


Fig. 2. Results of metal ion transport (water/chloroform/water) studies for dicyclohexyl-18-crown-6. Source phase: pH 4.9 (CH<sub>3</sub>COOH/CH<sub>3</sub>COONa) containing metal ions at 0.01 M (10 mL). Membrane phase: contains ligand (0.001 M) (50 mL). Receiving phase: pH 3.0 (HCOOH/HCOONa), (30 mL) stirred for 24 h

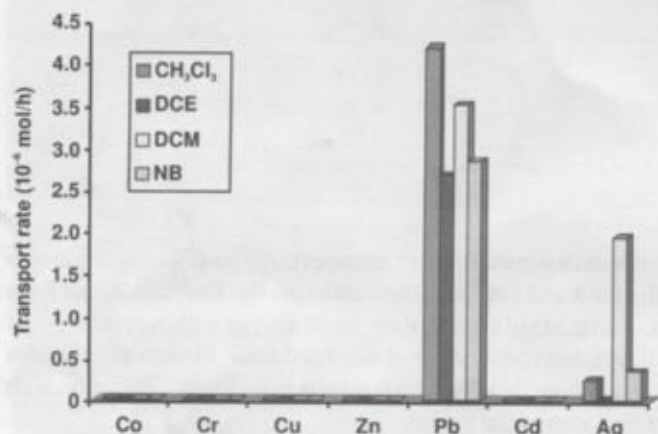


Fig. 3. Comparison of the results of metal ion transport (water/organic solvent/water) studies for dicyclohexyl-18-crown-6. Source phase: pH 4.9 (CH<sub>3</sub>COOH/CH<sub>3</sub>COONa) containing metal ions at 0.01 M (10 mL). Membrane phase: contains ligand (0.001 M) (50 mL). Receiving phase: pH 3.0 (HCOOH/HCOONa), (30 mL) stirred for 24 h

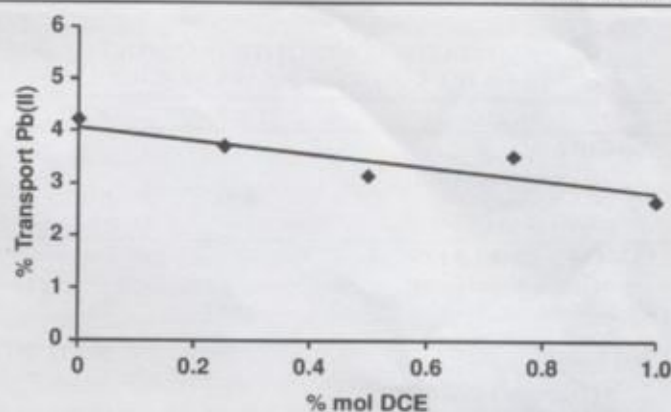


Fig. 4. Changes of lead(II) ion transport with the composition of the CHCl<sub>3</sub>-1,2-DCE binary systems. Source phase: pH 4.9 (CH<sub>3</sub>COOH/CH<sub>3</sub>COONa) containing metal ions at 0.01 M (10 mL). Membrane phase: contains dicyclohexyl 18-crown-6 (0.001M) (50 mL) in CHCl<sub>3</sub>-1,2-DCE binary solvents. Receiving phase: pH 3.0 (HCOOH/HCOONa), (30 mL) stirred for 24 h

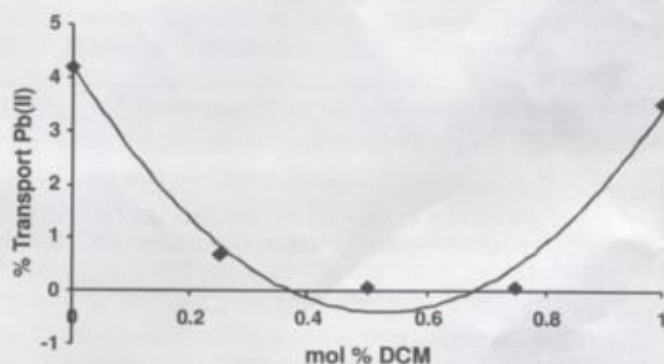


Fig. 5. Changes of lead(II) ion transport with the composition of the CHCl<sub>3</sub>-DCM binary systems. Source phase: pH 4.9 (CH<sub>3</sub>COOH/CH<sub>3</sub>COONa) containing metal ions at 0.01 M (10 mL). Membrane phase: contains dicyclohexyl-18-crown-6 (0.001 M) (50 mL) in 1,2-CHCl<sub>3</sub>-DCM binary solvents. Receiving phase: pH 3.0 (HCOOH/HCOONa), (30 mL) stirred for 24 h

path length), the diffusion coefficients of all mobile species in these boundary layers and the log K value for formation of the cation-ligand complex in the organic phase. Additional parameters are the partition coefficients of the macrocycle and the cation-ligand complex. The thickness of the boundary layers is a function of the rate at which the solvent is stirred, as well as, its viscosity. The composition of the boundary layers is a function of the mutual water-solvent solubilities<sup>25,26</sup>.

The rationalization of solvent effects often requires organic solvents. To take into account more than one property of the solvent, a multiparameter approach of the general form:

$$A = a + b.B + c.C + d.D \quad (1)$$

is applied, where A is the solvent-dependent property, such as the fluxes or selectivity ratios and B, C, D, ... represent the independent solvent parameters with a, b, c, d, ... as regression coefficients. Such equation can be utilized only if data are available for a large number of solvents and its success requires the application of proper statistical methods<sup>27</sup>.

The performance of various solvents including: chloroform, dichloromethane, 1,2-dichloroethane and nitrobenzene, were examined in this investigation. As is evident in Table-1, the rate of transport of Pb<sup>2+</sup> cation through different organic solvents was found to be in order: chloroform > dichloromethane

> nitrobenzene > 1,2-dichloroethane. The partitioning of ions into the organic phase should also be affected by the dielectric constant of the solvent<sup>25,27</sup>.

The dielectric constants of these solvents decrease in order: 1,2-dichloroethane > nitrobenzene > dichloromethane > chloroform. Since chloroform has the lowest dielectric constant, therefore, the partitioning of ions into it is easier than the other organic solvents. Also this results in stabilization of the ion pair in the organic phase causing to increase the rate of transport of  $Pb^{2+}$  through chloroform as liquid membrane. Since chloroform also has higher donor and acceptor numbers than DCE, DCM, NB, it solvates the  $Pb^{2+}$  cation and DCH18C6 stronger than the other organic solvents. This character may be lead to that this solvent can easily release metal cation to receiving phase. However other parameters can also be effective in chloroform performance in transport efficiency of  $Pb^{2+}$  cation.

**Effect of binary mixed solvent membrane on transport efficiency of the metal cations:** The results of transport from a source phase containing seven metal cations through chloroform:1,2-dichloroethane and chloroform-dichloromethane binary mixtures containing the macrocyclic ligand are listed in Tables 2 and 3, respectively. As is evident in Tables 2, the transport of  $Pb^{2+}$  cation increases as the concentration of chloroform is increased in binary chloroform:1,2-dichloroethane solvents. This result may be due to the lower dielectric constant of chloroform and, therefore formation of stable ion pairs and higher partitioning coefficient of  $Pb^{2+}$  into chloroform which leads to increase the transport of  $Pb^{2+}$  through liquid membrane with increasing the concentration of chloroform in this binary solution.

The change of transport percent of  $Pb^{2+}$  versus the composition of chloroform-1,2-dichloroethane and chloroform-dichloromethane binary solvents are shown in Figs. 4 and 5, respectively. As is obvious from Fig. 4, a linear relationship is observed for variation of transport per cent of this cation with the composition of binary chloroform-1,2-dichloroethane mixed solvents which indicates that there is no interaction between chloroform and 1,2-dichloroethane molecules in this solvent system. But in the case of chloroform-dichloromethane binary solution, a non-monotonic behaviour is observed between the transport rate of  $Pb^{2+}$  with the composition of this binary solutions. This behaviour may be due to strong solvent-solvent interactions between  $CHCl_3$  and DCM molecules which lead to change some of the physicochemical properties of this mixed binary solvent.

**Effect of fatty acids on transport efficiency of the metal cations:** It is expected that addition of a lipophilic counter ion such as a fatty acid to the organic membrane phase, can impart a greater degree of lipophilicity to the charged species and also removes the need for a simple counter ion to cross the source phase/membrane interface on metal cation uptake<sup>29</sup>.

In this study, we investigated the influence of three fatty acids (stearic acid, palmitic acid, oleic acid) as surfactant (0.004 M) in the membrane phase on  $Pb^{2+}$  cation transport process. The results are given in Table-4. As is obvious from these data, in a few cases, the presence of fatty acids leads to increase the transport rate. In different solvents may be due to

different fatty acid, crown ether and solvent structures and their stereo orientation, different solvation and interactions occur between them and this phenomenon lead to easier cation transport across bulk liquid membrane and therefore increasing transport rate. But in most cases the cooperative behaviour in the presence of these long-chain hydrocarboxylic acids is decreased. This may be due to formation of hydrogen bonds between the donor atoms of the macrocyclic ligand and acidic proton of carboxylic acids in membrane phase which results in formation a weaker complex between  $Pb^{2+}$  and crown ether in the presence of these fatty acids, or it may be due to formation of micelles of fatty acids in the membrane phase which trap the crown-cation complex<sup>30</sup>.

## Conclusion

The liquid membrane transport experiments of cobalt(II), chromium(III), copper(II), zinc(II), cadmium(II), silver(I) and lead(II) metal cations using dicyclohexyl-18-crown-6 as an ion carrier in various organic membranes showed that it is possible to separate and purify the  $Pb^{2+}$  cation by this macrocyclic ligand and the rate of this cation transport is strongly influenced by the nature and composition of the membrane solvents. The transport rate of the  $Pb^{2+}$  cation is influenced by the composition of the mixed solvents and chloroform-dichloromethane binary solution, a non-linear behaviour was observed between the transport per cent and the composition of this membrane system. The results obtained for competitive liquid membrane transport of the studied metal cations, show that in most cases, the transport rate of  $Pb^{2+}$  cation through organic membrane phases decreases in the presence of stearic acid, palmitic acid, oleic acid as surfactant in different organic phases.

The simplicity, low cost, high efficiency and selectivity for  $Pb^{2+}$  transport obtained by the liquid membrane systems, demonstrate its potential applicability to selective removal, concentration or purification of lead(II) cation from other interfering cations, present in solution.

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