

Transport of Pb(II) through bulk liquid membrane containing tri-n-butyl phosphate as a carrier

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Abstract:-

Lead contamination of industrial effluents and wastewaters is a significant environmental problem. This study was carried out to remove and recover lead from aqueous solution using bulk liquid membrane. The effects of several parameters including carrier concentration (2.5, 5, 7.5, 10 and 12.5%), pH of the feed phase (2, 3, 4, 5 and 6.5), pH of the strip phase (2, 3, 4, 5, 6.5, 8, 9, 10, 11 and 12), lead concentration (25, 50, 100, 200, 300, 400 and 500 ppm), volume ratio (S:F) of (1:1), (1:2) and (2:1) and EDTA concentration (0.001, 0.01 and 0.1M) on the performance of the bulk liquid membrane were studied to find out optimum working conditions. The highest efficiency of extraction (87%) and stripping (92%) were achieved for 100 ppm initial concentration of Pb²⁺, 12.5% (v/v) carrier concentration, pH 5 of the feed phase, pH 2 of the strip phase, 0.1 M of EDTA and volume ratio (S:F) (1:2). The kinetics of Pb²⁺ transport through a bulk liquid membrane was investigated. This study proved that bulk liquid membrane is effective technique to remove lead from aqueous solutions.

Keywords:- Bulk liquid membrane, Carrier / Complexing agent, Tri-n-butyl phosphate, Stripping agent

1. Introduction

The discharge of organic and metal pollutants into the environment is a serious problem facing numerous industries. Because heavy metals are biodegradable not in natural conditions, they tend to accumulate in living organisms causing various diseases and disorders [11]. Therefore their concentrations must be reduced to acceptable levels before discharging them into environment. A variety of industries are responsible for the release of heavy metals into the environment through their wastewaters, among them mining, hydrometallurgy, pulp

and paper, petrochemical, refinery, fertilizer and electroplating, etc. [6]. methods Several have been described for the removal of heavy metals from wastewater, including precipitation chemical [14]. adsorption [10, 13], biosorption [2], ion exchange [12], complexation pressure-driven membrane [17], processes [4], and electrochemical processes [5, 8]. These techniques have their own inherent limitations such as less efficiency, sensitive operating conditions, production of secondary sludge, high capital and operating costs, and further the disposal is a costly affair [1].



Liquid membrane (LM) techniques are recently developed and have attracted the world as these yield active and selective transport of inorganic ionic and organic species in pure form. A liquid membrane, in non-porous. general, is a homogeneous, hydrophobic, thin film, typically of organic liquid held between two aqueous phases of different compositions. Due to the concentration gradient, the solute is transferred through the LM from one other. phase the Liquid to membranes are usually of three i.e.. supported liquid types, liquid membrane (SLM). bulk (BLM) and emulsion membrane liquid membrane (ELM).

Candela et al., [3] studied the use of bis-(2-ethyl hexyl) phosphoric acid (D2EHPA) as carrier in kerosene solvent in both BLM and SLM studies of uranium and it was seen that maximum extraction values obtained were above 95%, and recovery yields were in the range 57-79% for the BLM and 90% for the SLM.

The selective transport of Cu^{2+} ion was obtained from a mixture containing cations like Cd^{2+} , Pb^{2+} , Zn^{2+} , Ni^{2+} , and Co^{2+} using 5-nitro-8quinolinol as carrier. Nezhadali et al., [16] showed that under optimum circumstances, the amount of Cu^{2+} transport was 33.05% after 24 hr. The appositeness of the method was observed for the separation of copper(II) ions from real river samples and the transport efficiency was found to vary between 29% and 32% after 24 hr.

The objectives of the present work were studying the extraction and stripping behavior of lead on the transport process through the liquid membrane using TBP, studying the effect of various parameters like carrier concentration, pH of the feed phases, strip lead ions and concentration, stripping agent concentration and feed to strip volume ratio, and Applying the kinetic model for the transport of metal ions through liquid membrane by using the mechanisms of two consecutive irreversible first order reactions.

2. Material and Methods

2.1. Chemicals

Lead acetate $Pb(C_2H_3O_2)_2.3H_2O$ of purity 99.5%, obtained from (BDH Chemicals Ltd company, England) was used as a source of Pb²⁺ ions in solution, Tri-n-butyl aqueous the phosphate $C_{12}H_{27}O_4P$ of purity 98.5%. Commercially available kerosene was used without further purification.

2.2. Analysis

Atomic Absorption Spectrometer was used to measure concentration of soluble lead (GBC, Germany).

2.3. Bulk liquid membrane setup

The experimental setup of bulk liquid membrane used in this work is shown in (**Fig.** 1). It consists of a rectangular glass vessel (80 mm height \times 180 mm length \times 60 mm width) divided into two compartments by a 60-mm-height



glass plate of thickness 2 mm. One compartment was filled with an aqueous feed (F) phase and another one with an aqueous strip (S) phase. These aqueous phases are layered with a membrane (M) phase above them. The glass plate was placed at specific positions so that it gives S:F ratios of 1:1, 1:2, and 2:1.



Fig. 1 Schematic diagram of bulk liquid membrane.

2.4. Sample analysis

At regular intervals of 1, 2, 3, 4, and 5 hours, samples (each of volume 2 mL) from the feed and stripping phases were taken by pipetting out the aqueous phases, and after appropriate dilution the samples of both phases were analyzed for Pb²⁺ concentrations by a flame atomic absorption spectrophotometer.

The percentage of extraction (%E) of lead can be calculated according to equation (1):

% E =
$$\frac{(C_{F_0}) - (C_F)}{(C_{F_0})} \times 100\%$$
 (1)
Where:

 C_{F_0} : initial heavy metals

concentrations in the feed phase (ppm).

 C_F : heavy metals concentration at any time in feed phase (ppm).

The percentage of stripping (%S) of lead was given by equation (2):

$$\% S = \frac{(C_S)}{(C_{F_0}) - (C_F)} \times 100\%$$
(2)
Where:

 C_S : is the heavy metals concentration at any time in strip phase (ppm).

2.6. Kinetic procedure

The kinetic behavior for the variation of Pb²⁺ concentrations with time were directly determined on both feed and strip phases using AAS at regular time intervals of 1 hour for a total of 5 hours. The corresponding change of heavy metals concentrations ion in the membrane phase was determined from the material balance between dimensionless the phases. The reduced concentrations in the feed (C_F) and strip (C_S) phases were used for experimental purposes [7]:

 $R_{F} = \frac{C_{F}}{C_{F_{0}}}, R_{S} = \frac{C_{S}}{C_{F_{0}}}, R_{M} = \frac{C_{M}}{C_{F_{0}}}$ (3) Where:

R_s, R_M R_F, : represent the dimensionless reduced concentrations of the feed, strip and membrane phases, respectively, C_{Fo} : initial heavy metal concentration in the feed phase (ppm), C_F , C_S , C_M : represent the heavy metal concentrations (ppm) at any time in feed, strip and membrane phases, respectively.

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The initial concentration of heavy metals in both the membrane and strip phases was considered zero. The material balance with respect to the dimensionless reduced concentrations can be expressed as [7]:

 $R_F + R_S + R_M = 1$ (4) From this expression, the kinetic behavior of the consecutive irreversible first order reactions can be described as follows [7]:

$$C_F \xrightarrow{k_1} C_M \xrightarrow{k_2} C_S$$
 (5)

Where k_1 and k_2 are the apparent first-order rate constants of metal extraction and stripping processes, respectively. The kinetic scheme, Eq. (3) for consecutive irreversible reactions can be described by the following rate equations [7]:

$$\frac{dR_F}{dt} = -k_1 R_f \equiv J_F$$
(6)
$$\frac{dR_M}{dt} = k_1 R_F - k_2 R_M \equiv J_M$$
(7)
$$\frac{dR_S}{dt} = k_2 R_M \equiv J_S$$
(8)

Where: J_F , J_M and J_S are the instantaneous solute fluxes in the feed, strip and membrane phases, respectively.

When $k_1 \neq k_2$, and integrating the above differential equations gives [18]: **P** = $\exp(|k|t)$ (0)

that the time dependence of R_F is

decreasing monoexponential and the time dependence of both R_M and R_s biexponentail. The actual numerical analysis was carried out by non-linear curve fitting.

The maximum value of R_m (when $dR_M/dt = 0$) and the corresponding t_{max} can be written as follows [18]:

$$R_{M}^{\max} = \left(\frac{k_{1}}{k_{2}}\right)^{\frac{k_{2}}{k_{2}-k_{1}}}$$
(12)
$$t_{max} = \frac{\ln \frac{k_{2}}{k_{1}}}{(k_{2}-k_{1})}$$
(13)

Combination Eqs. (12) and (13) the following relationship can be obtained:

$$k_2 = \frac{\ln\left(\frac{1}{R_{\rm M}^{\rm max}}\right)}{t_{\rm max}} \tag{14}$$

Numerical analysis of experimental results by non-linear curve fitting permits the rate constants of the kinetic process to be determined. The values of k_1 were directly obtained by iteration from Eq. (9). This value was introduced as constant value in Eqs. (10) and (11). Two values of the rate constant k_2 were obtained, one derived from Eq. (10), k_{2m} , and the other derived from Eq. (11), k_{2s} . Good agreement between k_{2m} and k_{2s} constants was observed in all experimental conditions.

First-order time differentiation of Eqs. (7-9) lead to the final form of the flux equation [9]:

$$\frac{dR_{F}}{dt} = -k_{1}exp(-k_{1}t)$$
(15)

$$\frac{dR_{M}}{dt} = \frac{k_{1}}{k_{2}-k_{1}}[k_{2}exp(-k_{2}t) - k_{1}exp(-k_{1}t)]$$
(16)



 $\frac{dR_{S}}{dt} = \frac{k_{1}k_{2}}{k_{2}-k_{1}} [exp(-k_{1}t) - exp(-k_{2}t)]$(17) By substituting the expression of t_{max} given for Eq. (9) in Eqs. (15-17), max fluxes can be obtained [9]: $\left(\frac{dR_{F}}{dt}\right)\Big|_{max} = -k_{1}\left(\frac{k_{1}}{k_{2}}\right)^{-k_{1}/(k_{1}-k_{2})}$(18) $\left(\frac{dR_{S}}{dt}\right)\Big|_{max} = k_{2}\left(\frac{k_{1}}{k_{2}}\right)^{-k_{2}/(k_{1}-k_{2})} \equiv J_{S}^{max}$(19) $\left(\frac{dR_{M}}{dt}\right)\Big|_{max} = 0$(20) $-\left(\frac{dR_{F}}{dt}\right)\Big|_{max} = \left(\frac{dR_{S}}{dt}\right)\Big|_{max}$(21) It may be noted that at t = t_{max}, the system is in a steady state because

system is in a steady state because the concentration of metal ion in the membrane did not change with time Eq. (20). As a result, the exit and entrance fluxes are equal with opposite signs Eq. (21).

3. Results and discussion

3.1. Effect of carrier concentration In this study the influence of TBP concentration in the organic phase on the transport efficiency of lead were studied. The experiments were carried out using different Concentrations of TBP as the carrier in organic membrane phase within the range of (2.5, 5, 7.5, 10 and 12.5%) (v/v) in kerosene at ambient temperature and mixing speed at 200 rpm. The experimental results presented in Fig. 2 and Fig. 3. These figures shows that the carrier concentration in the membrane phase plays an important role on the metal ions removal.

has been observed that the It extraction and stripping efficiency of lead were increased from 13.32% to 30.297% and from 54.68 to 74% respectively when the carrier concentration is increased from 2.5% to 12.5% (v/v). It means that the increase in carrier concentration in the liquid membrane will increase the transport efficiency, thus the feed-membrane interface heightens formation of the metal-carrier complex, the same behavior was noticed by Fozia et al., [7]. Hence concentration of 12.5% (v/v) was selected as optimum value for further experiments.









Fig.3 Carrier concentration dependence of R_F, R_S and R_M for the transport Pb²⁺.

3.2. Effect of the feed phase pH One of the most important parameters that is expected to affect the transport efficiency from the feed phase to strip phase is the pH of the aqueous feed solution. This is because the first step in the transport of metal ion is the formation of metal-carrier complex at the feedmembrane interface. The pH of the aqueous feed phase was varied in the range of (2, 3, 4, 5 and 6.5) using 1M of HCL. The results are plotted in Fig.4 and Fig.5. From these figures it can be seen that lead had maximum extraction efficiency which is about 47% at pH 5 and the stripping efficiency reach 69%.

When the pH of the aqueous feed phase is decreased, i.e. high acidic there is a competition region, between the metal ions and the protons of the carrier and so the of metal transport amount is decreased. At pH 5 of the feed phase, the complexation of the metal ions with the carrier is the dominant process and the amount of metal transport is increased. At pH value up to 5 the extraction efficiency was reduced. The corresponding kinetic parameters for the transports of Pb²⁺ ions are presented in Table.1. As a results, the pH 5 was chosen as the optimum value for further experiments.





Fig.4 pH of the feed phase dependence of R_F, R_S and R_M for the transport of Pb²⁺.



Fig.5 Time dependence of R_F , R_S and R_M for the transport of Pb^{2+} at pH=5.

	phase pli .									
pН	k_1 (hr ⁻¹)	$k_2(hr^{-1})$	R _M ^{max}	t _{max} (hr)	J _F ^{max}	J _S ^{max}				
					(hr ⁻¹)	(hr ⁻¹)				
2	0.060665	0.3049	0.133231	6.6109	-0.04062	0.04062				
2	0.070124	0.2200	0.140040	5 7097	0.04749	0.04749				
3	0.072154	0.3390	0.140049	5.7987	-0.04/48	0.04748				
4	0.107718	0.4851	0.144515	3.9875	-0.0701	0.07010				
5	0.12735	0.527	0.153687	3.5537	-0.08099	0.08099				
6.5	0.099099	0.4443	0.144988	4.3464	-0.06442	0.06442				
From this Table it can be observed				k_2 and flux increased from (0.06066-						
that with increasing pH of feed				0.1273), (0.3049-0.0.527) and						
solut	ion from (2-5), the value	es of k_1 .	$(0.04062-0.0809 \text{ hr}^{-1})$, respectively						

Table.1 The kinetic parameters for the transports of Pb²⁺ at different feed phase pH^a.



Also the maximum time required to reach saturation decreased from (6.610-3.55 hr). Further increase in pH i.e. above 5 lead to decrease k_1 , k_2 , flux and increase the time required to reach saturation (t_{max}).

3.3. Effect of pH of the strip phase

The strength of the strip phase pH for the transport of metal ions could significant effect have on the efficiency and the transport selectivity of lead to transport. The pH of strip phase was varied in the range of (2, 3, 4, 5, 6.5, 8, 9, 10, 11 and 12). The experimental results presented in Fig. (6) shows that lead maximum extraction has and stripping efficiency which was about

77% and 95% respectively, at pH 2. This may be due to the increase of hydrogen ion concentration (H^+) responsible in carrying the metalcarrier complex from the organic membrane phase to the strip phase enhance which the transport efficiency. The transport efficiency decreases considerably when the pH of the strip phase is decreased to neutral condition. The corresponding kinetic parameters for the transports of Pb²⁺ ions are presented in Table (2). According to the obtained results, the optimum pH of the strip phase was found to be 2. This pH value for further was used experiments.



Fig.6	pH of	the strip	phase	dependence	of R _F ,	R _S and	R _M for	the trai	nsport o	of Pb ²⁺ .
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pН	k_1 (hr ⁻¹)	$k_2(hr^{-1})$	R_M^{max}	t _{max} (hr)	J _F max	J _S ^{max}
					(hr ⁻¹)	(hr ⁻¹)
2	0.292704	1.9803	0.106093	1.132879	-0.2101	0.2101
3	0.210456	1.2753	0.115586	1.691946	-0.14741	0.14741
4	0.188147	1.1853	0.112163	1.845781	-0.13295	0.13295
5	0.177599	0.894	0.133076	2.255968	-0.11897	0.11897
6.5	0.12735	0.527	0.153687	3.553767	-0.08099	0.08099

Tuble a the mileter purameters for the transports of the attained one ship phase pri	Table. 2 The	kinetic parameter	s for the transpo	orts of Pb ²⁺ at o	different strip	phase pH ^a
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8	0.070992	0.368	0.130181	5.540296	-0.04791	0.04791
9	0.095891	0.4784	0.133969	4.201819	-0.06409	0.06409
10	0.112336	0.5055	0.144598	3.825511	-0.07309	0.07309
11	0.193249	1.222	0.111838	1.792724	-0.13667	0.13667
12	0.1983	1.243	0.112601	1.756966	-0.13996	0.13996

3.4. Effect of initial concentration of Pb^{2+}

The results obtained are presented in Fig.7 clearly indicates that with increasing the initial concentration of Pb^{2+} in the feed phase from 50 to 500 ppm, the transport of metal ions decreased. This decrease may be due to saturation of the membrane at high metal concentration, similar behavior was observed by Muthuraman al., [15]. The et maximum transport rate occurred at

50 ppm in which the extraction efficiency reach 80% and at 100 ppm reach 77%. At initial concentration of 25 ppm the decreased. transport rate The stripping rate was high at initial concentration of 100 ppm which was about 95% and start to decreases with the increase in metal ions concentration. Furthermore. an initial concentration of 100 ppm in the aqueous feed phase was chosen further experiments. for



Fig.7 Extraction and stripping efficiency versus Heavy metals concentrations of Pb²⁺.

3.5. Effect of stripping agent concentration

One of the important parameters that can affect the transport rate is the nature of the stripping reagent. This is because the stripping will also decide the overall transport rate. The complexation of metal ions by the carrier at the first interface is through proton exchange mechanisms and thus the selection of the stripping agent in the stripping phase is suitable for the stripping process. The stripping agent used to



enhance the stripping process was EDTA, and the effect of its concentration on the removal efficiency was studied in the range of (0.001, 0.01 and 0.1 M).

The influence of EDTA concentration in the stripping phase on the transport rate of Pb^{2+} ion are shown in **Fig.** (8) and **Fig.** (9). There was marginal increase in stripping and extraction rate occurs at EDTA concentration of 0.001 M compared with the case of no EDTA. The transport of Pb^{2+} ions also increased

with increasing the concentration of EDTA to 0.1 M, in which the stripping and extraction efficiency were 97% and 79%, respectively. This slight increase was also related to the formation of complex between lead and EDTA at membrane-strip which interface. enhance the Pb^{2+} . transport rate of The corresponding kinetic parameters for the transports of Pb^{2+} ions are presented in Table (3). As a results, the concentration of 0.1 M of EDTA was chosen for further experiments.



Fig.8 Time dependence of R_F, R_M and R_S for the transport of Pb²⁺ at 0.1M of EDTA.



Fig.9 EDTA concentrations dependence of R_F, R_S and R_M for the transport of Pb²⁺.



concenti ations.								
EDTA	k ₁ (hr ⁻¹)	$k_2(hr^{-1})$	R _M ^{max}	t _{max} (hr)	J_F^{max} (hr ⁻¹)	J _S ^{max}		
Conc.						(hr ⁻¹)		
(M)								
0	0.292704	1.9803	0.106093	1.132879	-0.2101	0.2101		
0.001	0.296341	2.066	0.103619	1.097307	-0.21408	0.21408		
0.01	0.308627	2.24	0.100376	1.026263	-0.22484	0.22484		
0.1	0.312396	3.2268	0.075376	0.801184	-0.24322	0.24322		

Table.3 The kinetic parameters for the transports of Pb2+ at different EDTAconcentrations.

3.5. Effect of strip to feed volume ratio

The experimental results were presented in Fig.10 and Fig. 11 show that the transport rate increases when S:F ratio changed from 1:1 to 1:2. This increase may be due to the increase in the interfacial area available at the feed-membrane interface for the transport of metal ion through liquid membrane, which became 72 cm^2 for S:F of 1:2 compared with 54 cm² for S:F of 1:1. The extraction and stripping efficiency were 87% and 91%, respectively at (S:F) 1:2 compared

with 79% and 97% at (S:F) 1:1, and when the volume ratio (S:F) was changed from 1:1 to 2:1 the transport rate decreases considerably, in which extraction and stripping efficiency were 66% and 94%, respectively at (S:F) 2:1 compared with 87% and 91% at (S:F) 1:2. This decrease was because of the small available interfacial area at the feedmembrane interface which was 36 cm^2 compared with 72 cm^2 for S:F ratio (1:2).

According to the obtained results, the volume ratio (S:F) of (1:2) was selected as the optimum ratio.



Fig. 10 Time dependence of R_F, R_M and R_S for the transport of Pb²⁺ at S:F 1:2.





Fig. 11 Volume ratio dependence of R_F, R_S and R_M for the transport of Pb²⁺.

4. Conclusions

- 1. The obtained experimental results from the present research indicated that the liquid membrane technique is effective for removal of lead ions contaminated in aqueous solution.
- 2. The kinetic model was applied and the two consecutive irreversible first-order apparent rate constants k_1 , k_2 and R_M^{max} , t_{max} and flux value J_F^{max} , J_S^{max} had been determined.
- 3. It was found that the presence of carrier in the membrane phase is very important for the metal ion to transport from the feed phase to the strip phase, because it forms a complex with the metal ion at the feedmembrane interface and carry the metal to the strip phase. The optimum carrier

concentration (v/v) was 12.5%.

- 4. A pH gradient between the feed and the strip phases is the driving force for the transport of metal ions across the membrane phase. The experimental results indicated highest transport that the occured when the pH of the feed and strip phases were 5 adjusted to and 2. respectively.
- 5. Increased heavy metal concentration decreased the transport of metal ions across the membrane phase, this decrease may be due to the saturation of the membrane at high metal concentration. As well as at very low concentrations the transport rate was also decreased, which may be due to the lower concentration driving force for the transport of metal ion



through bulk liquid membrane.

- 6. The extraction and stripping efficiency reached 79% and 97% when adding 0.1 M of EDTA to the strip phase as stripping agent. The maximum flux value at the strip phase J_S^{max} was 0.24322 hr⁻¹.
- 7. Strip to feed volume ratio (S:F) of (1:2) was found to be the optimum value for lead ions to transport from the feed phase to the strip phase.

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أنتقال الرصاص خلال الاغشية المائية الكتلية باستخدام tri-n-butyl phosphate كناقل

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الخلاصة