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Study of Co(II) Transfer through a Ceramic Supported Liquid Membrane Impregnated with Di-(2-ethylhexyl)phosphoric Acid (D_2HEPA)

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THE permeation of cobalt (II) transport through a ceramic disc supported liquid membrane impregnated in D_2HEPA as a carrier was studied at 25^0C. The effects of Co (II) ions, HNO_3 in feed phase and D_2HEPA. The transport of Co(II) was studied as a function of different parameters like: effect of divalent ions, HNO_3 concentration, effect of D_2HEPA and effect of permeation rate, stirring speed of the feed (300-5000 min^{-1}) and receiving (300-1200 min^{-1}) phases, EDTA as stripping solution (0.1-2) mol/L in ceramic membrane concentrations have been studied. Co(II) ions concentration increase in the feed due to an increase in flux from $(15.8\text{-}21.2)\times10^{-9}$ in the Co(II) ions, concentration range $(7.98–26.45)\times10^{-4}$ mol/L for cobalt at 2mol/L HNO_3 in the feed phase . The optimum conditions for Co (II) ion transport are 2mol/L HNO_3 in feed phase and 2mol/L D_2HEPA in disc ceramic membrane. It shows that cobalt ions in flux via the membrane due to increase with these different ions concentration increases. Applying the studied conditions to cobalt in waste solutions indicated more than 98.7% removal .

Keywords: Ceramic disc, Supported membrane , EDTA, D_2HEPA, Permeation.

Introduction

Wastewaters from metal industries contain various toxic heavy metals. These heavy metals present a high health risk when they enter the human food chain[1,2]. Cobalt is a very toxic element affecting the environment. Cobalt is present in the wastewater of nuclear power plants and many other industries such as mining, metallurgical, electroplating, paints, pigments and electronic industries. High levels of cobalt may affect several health problems[3-10].

Whereas membrane technologies are one of the most important topics in today´s research and practical use, liquid membrane processes have been proposed as clean ones owing to their characteristics of high specificity, low energy utilisation, the extraction and stripping steps are combined in a single operation etc.; thus, the use of liquid membranes has gained a general interest in the treatment of effluents were solute concentrations are low and large volume of solutions must be processed, and if possible, without generating any secondary waste[11-18]. Recommended in the report which applied to different equations of permeation behavior since before and after treating membrane, calculations of β(separation factor) Co(II)/Pb(II), porosity calculation in an aqua ceramic membrane system and the Δp (difference pressure) in an aqua membrane drop for these experiments was in the range of 10–354 kPa found the concentration of [D2HEPA] is stable and the layer not affected temperature, so that and It has also been used as a protective layer for ceramic disc and it can be used in ceramic painting. In previous works, the extraction of cobalt (II) from aqueous solutions using different mixtures of acidic reagents were

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studied [19]. The performance of these extractants mixtures on cobalt extraction, *i.e.* allowing the metal extraction at more acidic values than each separate reagent, had led to implement one of these extraction systems in a ceramic supported liquid membrane design in order to investigate the cobalt transport using the mixture of different extractants such as D_2HEPA and DP-8R extraction reagents in Exxsol D100 as mobile carrier phase.

Ceramic Membrane Materials supported disc was used different composition and impregnated in extractant di-(2-ethylhexyl) phosphoric acid (D_2EHPA soluble in 15% kerosene in used in this work.

For this extraction system, the extraction reaction of cobalt or lead ions can be given by Eq. (1):

$$Co(II)_{aq} + (2+n)NO_3^- + nH_{aq}^+ + \overline{nR} \Leftrightarrow [(LR)_n CoNO_3^{2-})]_{2n} \ldots . 1$$

Ceramic disc supported liquid membrane used in separation processes in two metals. Figure.1. The system consists of two aqueous phases and an organic phase[20-24], which contains [D_2HEPA] as a carrier dissolved in 15% kerosene as diluent. The ceramic disc renders for both a supported membrane for the organic and bulkhead from ceramic between the two aqueous phases. Theoretically the following different steps depends on the permeability of two metals from feed bulk solution into ceramic pores of cobalt andlead ions with D_2HEPA or permeation of the complex in the organic membrane phase.To the other side of the ceramic disc membrane and another suggested the dissociation of two ions after the advent in contact with EDTA stripping phase. Equation (1); show the react between the metal ion and the carrier of membrane interface is predominated by the equilibrium, the extraction distribution coefficient D and equilibrium constant k_{ex} are given by

$$D = \frac{\overline{[(RH)_{3n-1}.MR_n]}}{[M]^{+2}} \ldots\ldots 2$$

Ddistribution coefficient for different metal ions in the organic and aqueous phases, and K_{ext}

$$K_{ext} = \frac{D}{[H^+][M]^n[R^-]^{3n-1}} \ldots\ldots 3$$

Where $[H^+]$, $[M]^n$ and $[R]^{3n-1}$ are activities of Co and Pb ions species.

Experimental

Di-(2-ethylhexil) phosphoric acid (D_2EHPA) was purchased from Merck (*German*) and was used as received. HNO_3 (0.5M) was used as kerosene from Misr Petroleum Co. (Egypt Oil&Gas) a diluent for the organic phase. The Co $(NO_3)_2$ was used in this study EDTA as stripping agents was supplied by analytical grade. Stock solutions were prepared by dissolving the required amount of cations in HNO_3 (0.5-3) mol/L. Another chemicals used in the work were of analytical reagent grade. The ά-Al_2O_3 with SiO_2 and TiO_2 was obtained as nature, sources of feldspar, Aswan clay and Abu-Eldarag clay to fabricated disc ceramic supported liquid membrane previously manufactured [19] with a pore size of 0.1µm, thickness of 100µm and a porosity of 80%); surface area 6.25 cm^2, was also employed in the study and was obtained from after polishing in one side. The concentration of two metals solutions was determined using Atomic absorption/Emission Spectrophotometer/210/VGP, Buck Scientific, USA, was used for determination of cobalt and lead ion concentration and determination of particle size by Quanta chrome instruments model Nova 1000e .An apparatus used for membrane extraction.

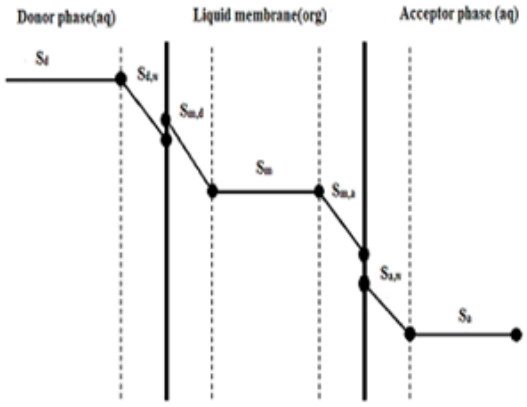

Fig. 1. Schematic diagram in supported liquid membrane.

Ceramic disc membrane supported preparation

The Ceramic disc membrane was prepared from nature clays containing SiO_2 and ὰ-Al_2O_3 (high quality) impregnated the 100-µm membrane support disc with D2EHPA for 8 h before use. The supported CDM was then taken out from the SLM and the excess liquid attached to the surface of the membrane by polishing process in one side Ra < 0.1 µm. The CDM prepared was fixed into the SLM cell in Figure (2). Thickness 100µm, pore diameter 0.1µm, surface area 6.25 cm^2 and porosity 80%.Circular pieces in pertraction apparatus of ceramic microporous as nature materials [20-22].

Figure 2 S. Scheme for pertraction apparatus. (1) Double shield glass outer vessel; (2) Teflon cross-stirring blade; (3) supported membrane (cellulose nitrate); (4) feed compartment; (5) strip compartment. The SLM cell consisted of two square deal of a cylindrical chamber (65 mm internal diameter, 75 mm length) separated from the CD supported membrane. A magnetic stirrer agitated the HNO_3 (0.5-3M) aqueous solution in each chamber. The effective volume of each chamber was 100 ml. With the central part in contact with the aqueous solution having a 40 mm diameter. Transport of Co(II) ion experiments were carried out in a pertraction CDs membrane supported cell in which the membrane disc was clamped tightly[22].

Determination of permeation coefficient

The flux ($J_{mCo\ (II)\ \&Pb\ (II)}$) was obtained using equation [22]

$$J_m = -\frac{VdC_{Co,Pb}}{Adt} \ldots .4$$

Where V is the volume of the feed solution, A is the membrane area in contact with the aqueous solution, C is the concentration of Cobalt and Lead ions and t is the neglected time. The permeability coefficient (P) was then calculated using equation[20]:

$$\ln {C_t}/{C_0} = -\frac{AP_t}{V} \ldots .5$$

Where C_t and C_0 are concentration of Cobalt and Lead ions in the acidic medium at a given time and t_0, respectively, A is the effective ceramic membrane area and V is the volume of the feed phase and the t = 0.

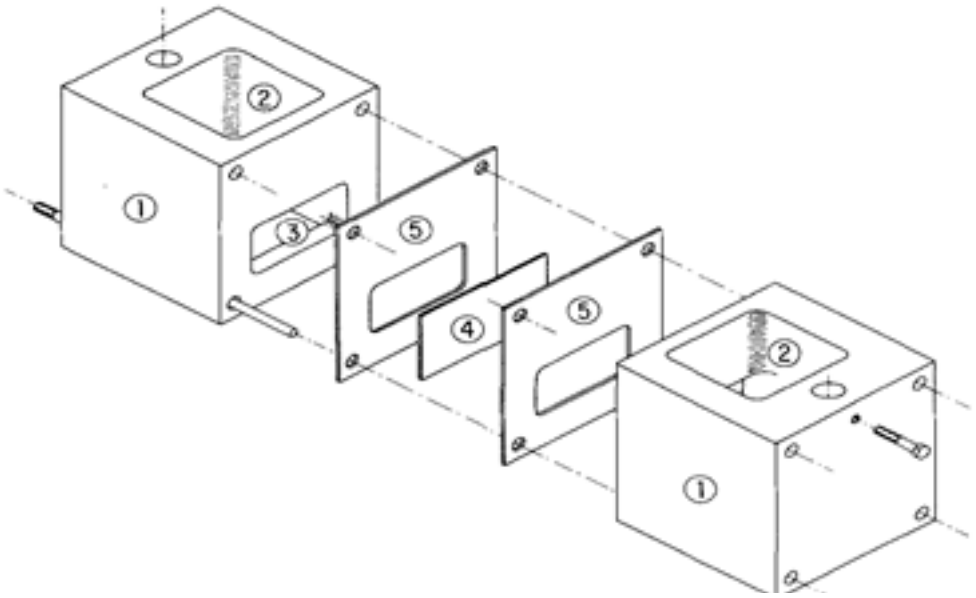

Fig. 2. Scheme for pertraction apparatus. (1) Double shield glass outer vessel; (2) Teflon cross-stirring blade; (3) supported membrane (cellulose nitrate); (4) feed compartment; (5) strip compartment[22].

Results and Discussion

A study of diffusion experiments was perfected to investigate the effect of Co(II) ion concentration, carrier concentration, acid concentration and stripping solution (EDTA) on metals transport.

Effect of divalent ion concentration

Figures 3-6 show the plot of cobalt (II) ions concentration vs time. It is clear that the transports of metal ions are affected by the Co ions concentration existent in the feed phase of the CDs membrane. The stripping solution used is (0.1-2) mol/L EDTA. As the concentration of cobalt ions of feed solution are increased and the extraction of metal ions. It can also be seen that Co (II) ions are transported via the CDs membrane, although the concentration of cobalt ions and lead ions in the feed phase are less than that in the stripping phase.

Figures 5 and 6 show the effect of feed concentration solution of the transport of cobalt (II) ions through the membrane using 2M EDTA as a stripping agent. It is obvious in Fig. 7 that there is a slight increase in flux with an increase in cobalt concentration and maximum flux is found when 20.54×10^{-4}mol/L for cobalt ion concentration is used. According reaction (6) and Eq. (7). The amounts of stripping (EDTA) concentration lightly influences the flux. About 2 M EDTA concentrations are adequate to strip cobalt ion.

$$Co(II)_{aq} + (2+n)NO_3^- + nH_{aq}^+ + \overline{nR} \Leftrightarrow [(LR)_n CoNO_3^{2-})]_{2n} \ldots . -6$$

$$J_m = \frac{D_E}{\iota C_1} = \frac{D_E \lambda C_1^0}{\iota} \ldots \ldots 7$$

Effect of HNO_3 concentration

Figures 7 and 8. The explicates the effect of HNO_3 concentration of metal ions in the feed solution on the transport of cobalt ions through the existing of CDs membrane. It is clear that the flux of metal ions increases up to a 2 mol/L HNO_3 concentration and then decreases. The concentration, extent studied for HNO_3 is from 0.1 to 2.0 mol/L. According to equations Eq. (8), the cobalt ions obtain to convert into $[CoNO_{3+1}]_{1-}$ in the presence of 2mol/L HNO_3. So the cobalt or lead anions associates with D_2HEPA ions in the presence of protons to form a $[(D_2HEPA)_2O]_nCoNO_{3+n}$ type complex, which permeate towards the other side of the CDs membrane, resulting in Co(II) transport.

$$R + nH^+ \Leftrightarrow \overline{[RH_n]^{n+}} \ldots . 8$$

$$\log J_m = \log A^- + n\log[\overline{R}] + n\log[H^+] + [(3-n)\log NO^-] + \log C_t^0 \ldots . 9$$

An increase in HNO_3 concentration as the concentration of the cobalt(II) ions in carrier complex at the feed –ceramic disc membrane interface increases, according to the equilibrium relationship seen the reaction in equation. 9.

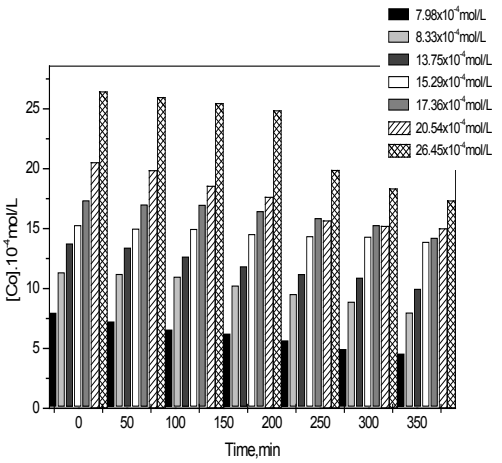

Fig. 3. [Co] decrease in feed phase against time,min at different C0 with 2 mol/L EDTA as stripping phase.[EDTA] 0.1-2 mol/L, [HNO3] 0.5-3 mol/L ,[D2HEPA] 1.5 mol/L, [Co] (7.98-26.45). 10-4 mol/L at pH 6.03±0.03.

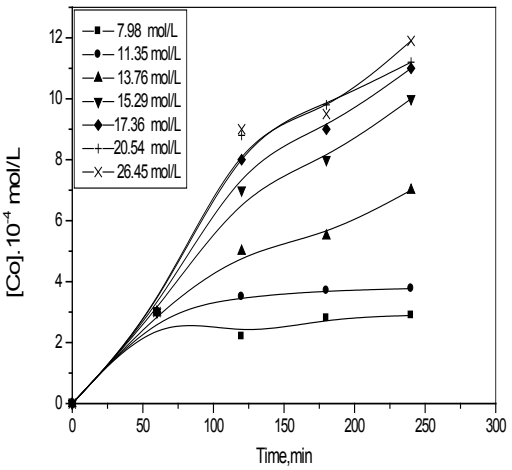

Fig. 4. [Co] increase in EDTA strip against Time, min at different C0 of Co(II) in feed solution, [EDTA] 2 mol/L as stripping solution , [HNO_3] 0.5-3 mol/L,[Co] (7.98-26.45).10-4 mol/L at pH 6.03±0.03.

From the Figure.9. Showed that the before treated membrane flux increased by[HNO_3] increase from 1.0 to 1.5 mol/L after 1.5 mol/L concentration of [HNO_3] before treated of membrane decreased. The second curve showed that after treating membrane increased in flux from 1.5-2mol/L [HNO_3] and the permeation behavior science before and after treating membrane in the Table (1). The separation factors were calculated inequation (10) and are given in Table(1) when solution containing Pb(II) with Co ((II) .

$$\beta = \frac{P_1}{P_2} = \frac{J_{mb}/[M_{Co,Pb}]_{feed}}{J_{mA}/[M_{Co,Pb}]_{feed}} \ldots\ldots \mathbf{10}$$

Where J_{mb},J_{mA} flux before and after treating

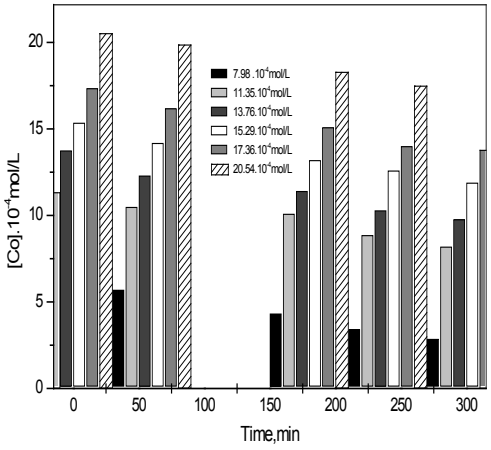

Fig. 5. [Co] decrease in feed phase against time,min at different C0 in feed phase of solution against 2 mol/L EDTA as stripping phase. [EDTA] 2 mol/L ,[HNO_3] 0.5-3 mol/L , [D_2HEPA] 2.5 mol/L, [Co] (7.98-20.54).10^{-4} mol/L,at pH 6.03±0.03.

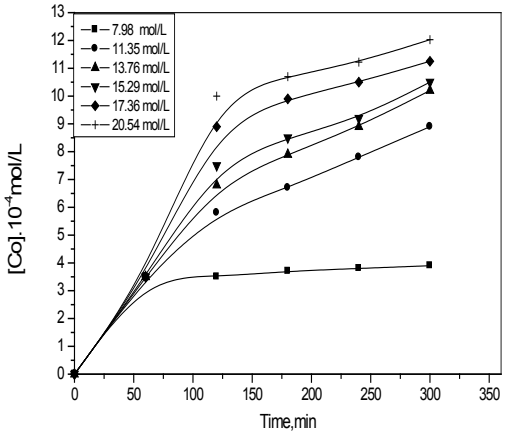

Fig.6. [Co]strip increase against Time,min at Cc feed , 2 mol/L [EDTA]stripping phase. [HNO_3] 2 mol/L,[D_2HEPA] 2mol/L, [Co] (7.98-20.54).10^{-4} mol/L at pH 6.03±0.03.

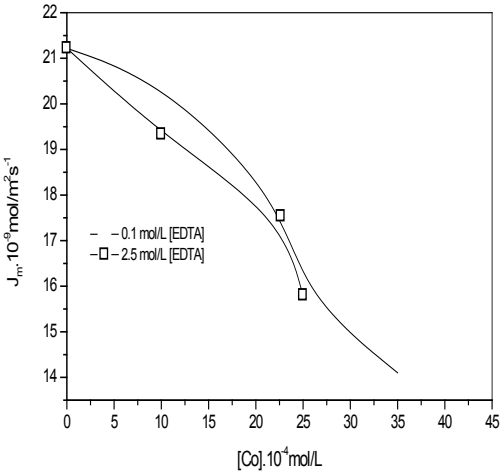

Fig.7. Effect of [Co]$_{permeation\ rate.}$[HNO_3] =2 mol/L, [D_2HEPA] =1.5 mol/L,[EDTA]0.1-2.5mol/L at pH 6.03±0.03.

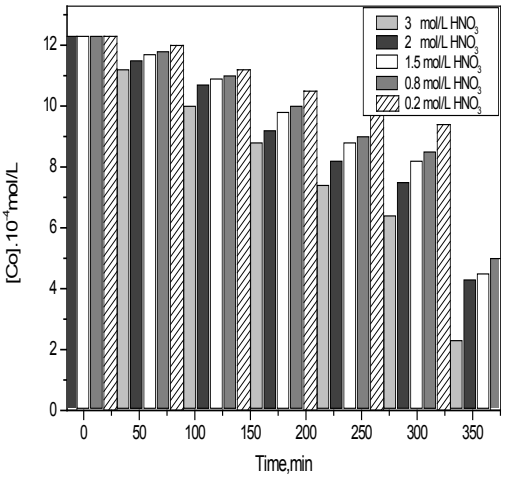

Fig. 8. Effect of [Co]$_{feed}$ when decrease against time, min at different [HNO_3]$_{feed}$ 0.2-3 mol/L, [D_2HEPA] 1.5 mol/L,[EDTA]=2 mol/L.,[Co] 12.33x10^{-4} mol/L, pH 6.03±0.03

membrane, respectively, show the maximum separation factor at [HNO_3] higher separation factor in 2 mol/L and less a separation factor at 0.19 mol/L.

Effect of permeation Rate

The determination of the permeation rate is known to be due to the carrier concentration reaches at saturation at the feed membrane interface. According to Eq. (9), if ${}^{f}C_0$ and [D_2HEPA] are preserved constant, i.e. at the same divalent ions like cobalt and lead ion concentration in feed solution and at the stable concentration of [D_2HEPA] in the CDs liquid membrane, a plot of log J_m versus log[H^+] can be obtain base a straight line and the value of '*n*' from its slope. It can be shown that up to about 2 mol/L HNO_3, it is a straight line (Fig. 10). Having a slope of 0.66. The fact that *n* is found to be 0.66 suggests the extraction mechanism which is based on the complexation of cobalt ions with a [H^+]^1 proton. To study the transport of H^+ ions with time, pH of feed phase was determined at regular duration's time. It can be shown that pH of feed phase increases with time, due to the transport of [H]$^+$ from the feed phase (Fig. 11). The effect of D_2HEPA concentration on the extraction of cobalt (7.98-20.54).10^{-4} mol/L, and lead (5.66-12.68).10^{-4} mol/L was studied in the range 1.0–4mol/L. It was shown that the percentage extraction of Co (II) increased with an increase of D_2HEPA concentration. The D_2HEPA concentration in the membrane phase varied from1 to 4 mol/L. The Co (II) and Pb (II) concentrations in the feed solution were kept constant at C_0 mol/L for the two metals. Fig.12. Show the extraction of Co (II) 98.7% ,while Pb (II) 66.4% respectively. Table 2. It was shown that the separation factor increased from 2.27 to 16.39 with an increase of [D_2HEPA] concentration from 0.25 to 1.5 mol/L and then decreased from 1.39 to 1.34 of [D_2HEPA] 2-3mol/L.

TABLE.1. Separation factor of $J_{mbefore\ treated\ membrane}$ and $J_{mAfter\ treated\ membrane}$ for Co and Pb at different [HNO_3].

[HNO_3]	J_{Co}mol/m^2.sec	J_{Pb}mol/m^2.sec	P_{Co}m/sec.10^{-4}	P_{Pb}m/sec.10^{-4}	$\beta_{Co/Pb}$
0.25	1.596	0.33	0.528	1.19	0.277
0.5	2.87	0.25	0.708	1.26	0.198
1	2.752	0.36	0.684	1.06	0.34
2	3.058	0.37	0.57	0.7	0.53

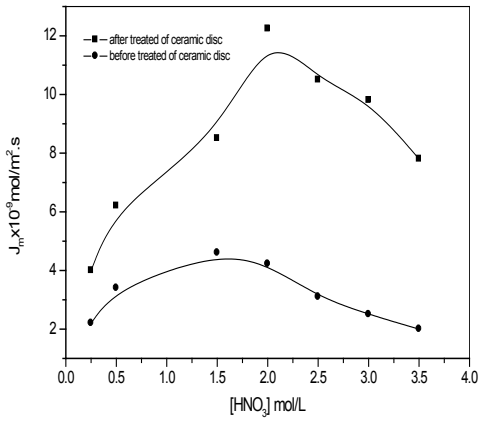

Fig.9. Effect of [HNO_3] permeation rate .[HNO_3] = 2 mol/L, [D_2HEPA] 1.5 mol/L, [EDTA] 2 mol/L.

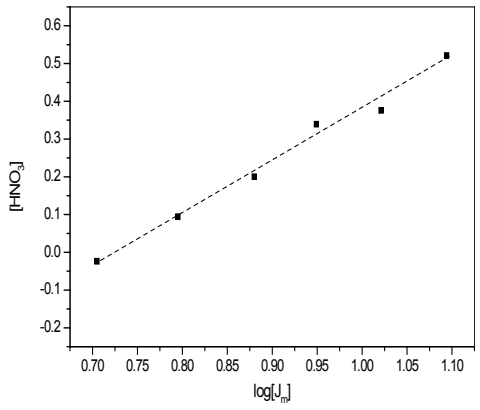

Fig. 10. plot log Jm vs.log [HNO_3] , slope= 0.7.

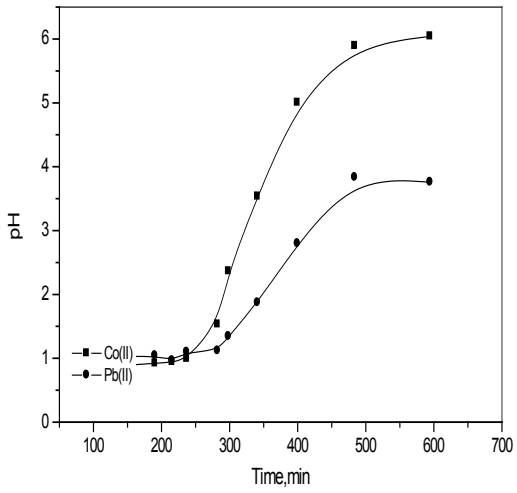

Fig. 11. Effect of pH of feed phase vs time, min. [HNO_3] 3 mol/L,[D_2HEPA]2mol/L,[EDTA]=2mol/L, [Co]12.33x10^{-4} at pH 6.03±0.03,[Pb]6.33x10^{-4} at pH 3.5±0.03 respectively.

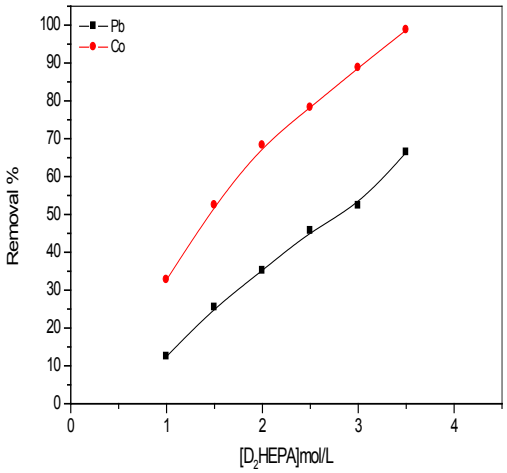

Fig. 12. Effect of D_2HEPA concentration on percentage extraction of Co 98.7 % and Pb 66.4%.

TABLE.2. Separation factor for cobalt and lead ions at different [D_2HEPA].

[D_2HEPA]	J_{Co}mol/m^2.sec	J_{Pb}mol/m^2.sec	P_{Co}m/sec.10^{-4}	P_{Pb}m/sec.10^{-4}	$\beta_{Co/Pb}$
0.25	4x10^{-9}	2.9x10^{-9}	0.5	0.22	2.27
0.5	6x10^{-9}	3.1x10^{-9}	0.68	0.277	2.15
1	8.2x10^{-9}	3.4x10^{-9}	0.83	0.269	3.08
1.5	9.8x10^{-9}	3.6x10^{-9}	1.05	0.062	16.93
2	10.2x10^{-9}	3.8x10^{-9}	1.07	0.769	1.39
2.5	11.2x10^{-9}	3.9x10^{-9}	1.007	0.83	1.21
3	12x10^{-9}	4x10^{-9}	1.12	0.832	1.34

Conclusions

Transport of cobalt and lead divalent ions was carried out by a ceramic disc supported liquid membrane technique by using nature of clays impregnated in D_2HEPA/ kerosene 15% as carrier. Study of various parameters Explained in this work. Studies the slope analysis studies of log $J_m$vs. [HNO_3] indicate that 0.7 or approach ˷1 mol/L of the hydronium ion in nitrate media and extractants were contributed to extracted metal ion species of both cobalt and lead. The cobalt and lead ions flux increased with an increase of [HNO_3] 0.25-3 mol/L, flux increased from 12.21x10^{-9} mol/m^2.s for [Co] and 4.3x10^{-9} mol/m^2.s for [Pb], the effect of pH of [$Co_{extraction}$ and [$Pb]_{extraction}$ 6.03-3.5±0.03 respectively. $J_{Co,Pb}$ increased with the increase of the [D_2HEPA] as carrier concentration at 2 mol/L and then decreased. It was seen that the maximum separation factor of J_m(membrane treated before and after) for [$Co]_{feed}$ and [Pb_{feed} at different [HNO_3] increased at 2 mol/L and less than o.5 mol/L, for study separation factor for [$Co]_{feed}$ with [$Pb]_{feed}$ at [$D_2HEPA]_{extractant}$ at 1.5 mol/Where separation factor for divalent metal ions in feed phase increased from [7.98-17.39] x10^{-4} mol/L for [Co] and [5.66-11.23] x10^{-4}mol/L for [Pb] ions respectively. And the best separation of cobalt and lead ions in wastewater or (feed solution) is obtained pH 3.5-6.03±0.03,1.5 mol/L of [D_2HEPA] and 2mol/L[$EDTA]_{strip}$ in aqueous solution. The divalent ions are transported via [D_2HEPA]/kerosene ceramic supported membrane and hydronium ion will be decreased from feed solution.. Nearly all cobalt and lead

ions have been found to be removed when ceramic supported membrane system is used for removal of this ion from waste solutions into feed. Ceramic membrane is on the promising technology in removing heavy metals from wastewater such as cobalt ions by 98.7% and lead ions by 66.4% respectively .

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