

Removal of Lead (II) from Battery Industry Wastewater by HFSLM

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Abstract—This work presents the extraction of lead (II) from wastewater of battery manufacturing (trace concentration) using hollow fiber supported liquid membrane (HFSLM). There are polypropylene as supporter, D2EHPA as carrier, kerosene as solvent and HNO₃ as stripping agent. The experiments were performed as various conditions such as organic phase concentration, stripping phase concentration, feed pH, and volumetric flow rate of feed and stripping solutions to obtain the best condition and to give the maximum efficiency. The extraction and stripping percentage of lead (II) from aqueous feed and stripping phase are 99.40% and 97.15%, respectively. The values of extraction equilibrium constant (K_{ex}), distribution ratio (D), permeability coefficients (P) were determined. The concentration of lead (II) is measured by Atomic Absorption Spectroscopy (AAS).

Index Terms—Hollow fiber, liquid membrane, lead extraction, battery manufacturing wastewater.

I. INTRODUCTION

Nowadays Lead contamination in an environment is a very important problem of worldwide concerning due to its highly toxic and nonbiodegradable in nature [1]. There are many ways that lead is released into aquatic system such as natural phenomenon, urban, agricultural and industrial activity. The industrial activity is the major source of lead contamination in water. There are various industries those are pertaining lead such as pulp and paper, petrochemicals, refineries, printing, pigments, photographic materials and explosive manufacturing, ceramic, glass, oil, metal, phosphate fertilizer, electronic, wood production, combustion of fossil fuel, forest fires, mining activities, sewage wastewater, automotive, coating, painting, storage batteries, aeronautical, alloy and steel industries [2]-[5]. When lead is released into the environment, it will be accumulated in food chain and exist in the nature. Lead can cause the severe health hazard even in the trace concentration. For instance, lead is extremely toxic and can damage to the nervous system, kidney, organ, and reproductive system [6], [7]. Moreover it is toxic to human via interaction with the sulfhydryl group of proteins, resulting

in disruption of the metabolism and biological activities of many proteins and it also impairs hemoglobin synthesis [8]. The national and international health authorities such as World Health Organization (WHO) try to control the level of lead emission from any sources are in the allowable concentrations. The latest information of the maximum contamination of lead level in drinking water from the United State Environmental Protection Agency (EPA) is 0.015 mg/l [9]. While the highest levels that the Pollution Control Department (PCD) of Thailand allows in drinking and ground water qualities are 0.05 mg/l and 0.2 mg/l for industrial effluent [10].

This paper interested in wastewater from battery manufacturing due to the most of lead has been consumed in this field. Lead is raw material which presents the specified characteristics such as resistant to corrosion, conductivity and the special reversible reaction between lead oxide and sulfuric acid. The growth of lead-acid battery is increase with the number automotive vehicles. A recent survey of three storage battery manufactures showed that the pH of wastewater at the source ranged 1.6 to 2.9, while the concentration of lead was dissolved in the ranged 5-15 mg/L [11]. There are many methods have been proposed for removal lead ions from wastewater, including chemical precipitation [12], [13], electrochemical reduction [14], ion exchange [15], [16], activated carbon [17], adsorption [18], [19], and biosorption [20]. The appropriate treatment method depends on effluent characteristics such as lead concentration, pH, temperature, flow volume, biological oxygen demand, and the economics involved and the social factor like standard set by government agencies [21]. But all of these methods can be only used for metal extraction when the concentration is less than 100 mg/L [22]. Recently, many attentions have been attracted to liquid membrane techniques due to the specific characteristics, liquid membrane can carry out simultaneous extraction and stripping processes in the same stage, and it has benefits of nonequilibrium mass transfer and up-hill effect, where solute can move from low to high concentration solutions [23].

Liquid membrane is a liquid barrier separating two aqueous phase of different composition that consist of feed phase and strip phase. The feed phase and strip phase can move through the liquid membrane from one phase to another phase due to the driving force between the two opposite side of the aqueous. The liquid membrane can be divided into two types, i.e. a supported liquid membrane (SLM) and a non-supported liquid membrane (non-SLM). For non-supported liquid membranes consist of bulk liquid membrane (BLM) and emulsion liquid membrane. For supported liquid membranes consist of an organic extractant solution that is held in polymeric microspore by capillary force [24], [25]. There are

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many types of supported liquid membranes such as flat-plate type (FLM), spiral-type, and hollow fiber supported liquid membranes (HFSLM). In recent years, the extraction of metal ions with a trace concentration has been concentrated on liquid membrane (LM) techniques. Especially, the HFSLM is the excellent techniques with a several advantages over traditional separation techniques such as low energy requirements, low operating costs, easy scale up and operation, high selectivity, single step operation rather than three steps as in solvent extraction [26], nondispersive contacting of the phases, large area to volume ratio for mass transfer, smaller production of secondary wastes, easy regeneration of the organic media [27]. But, there have been a few reports about lead extraction by using hollow fiber supported liquid membrane. Zeng, C. et al. [28] studied the determination of trace lead from hollow fiber supported liquid membrane by portable tungsten coil electrothermal atomic. Bautista-Flores, A. et al, [29] studied the hollow fiber supported liquid membrane sampling and speciation of lead (II) from aqueous.

All of the above reports show the great potential of the HFSLM. Hence, this work has selected this separation technique to study the removal of lead (II) from wastewater of battery manufacturing and propose it to be the alternative to conventional process. The HFSLM system consists of polypropylene as supporter, D2EHPA as carrier, kerosene as solvent and HNO_3 as stripping agent. The influence of organic phase concentration, stripping phase concentration, feed pH, and volumetric flow rate of feed and stripping solutions were investigated whereas the recycling mode was employed to obtain the optimum condition for lead (II) extraction. The values of extraction equilibrium constant (K_{ex}), distribution ratio (D), permeability coefficients (P) were determined.

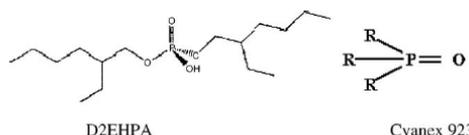


Fig. 1. Chemical structure of di(2-ethylhexyl) phosphonic acid (D2EHPA) and Cyanex 923 [30]

II. EXPERIMENTAL PROCEDURE

A. Reagents

The extractant di(2-ethylhexyl) phosphonic acid, commercial name: D2EHPA and Cyanex[®] 923 were obtained from Merck. The structures of carriers were shown in Fig. 1. They were used as the fixed carrier without further purification. The aqueous solution was provided from Thai Petroleum and Trading Co., Ltd. (Automotive Battery Manufacturing Plant). All other chemicals such as hydrochloric acid, sulfuric acid, nitric acid and kerosene were obtained from SR-LAB (Thailand). The feed phase was not diluted but only adjusted pH by sulfuric acid. The receiving or stripping phase was prepared from the stock solution by dilution with distilled water to the desired concentration. The organic phase is used as a carrier and dissolved in kerosene.

B. Characteristic of HFSLM

The hollow fiber module (Liqui-Cel extra-flow module)

was manufactured by Polypore (USA). This module uses Celgard microporous polyethylene fibers that are woven into fabric and wrapped around a central tube feeder that resembles a shell and tube configuration, with inlet/outlet ports for the shell and tube sides. The membrane is made from polypropylene, which is a hydrophobic material. The woven fabric provides more uniform fiber spacing, which in turn leads to a higher mass transfer coefficient than those obtained with individual fibers [32]. The properties of a hollow fiber module are listed in Table I.

TABLE I: THE PROPERTIES OF THE HOLLOW FIBER MODULE

Property	Description
Material	Polypropylene
Inside diameter of hollow fiber (cm)	24.0×10^{-3}
Outside diameter of hollow fiber (cm)	30.0×10^{-3}
Effective length of hollow fiber (cm)	1.4×10^4
Number of fiber	10,000
Pore size (μm)	0.03
Porosity (%)	25
Tortuosity	2.6
Area per unit volume (cm^2/cm^3)	29.3
Module diameter (cm)	6.3
Module length (cm)	20.3

C. HFSLM preparation and methods

The single module operation is shown in Fig. 2. At first, the carrier was diluted in kerosene and then it was impregnated into hollow fiber module by circulation into shell and tube for 40 min. After that the feed solution was fed into the tube side whereas the stripping solution was fed into shell side but it was in counter current direction. The lead ions in the sulphate solution were moved across the liquid membrane to stripping phase and were accumulated in the stripping phase. The concentrations of lead ions in sample from the feed and the stripping solutions were analyzed by AAS to determine the percentage of extraction and stripping.

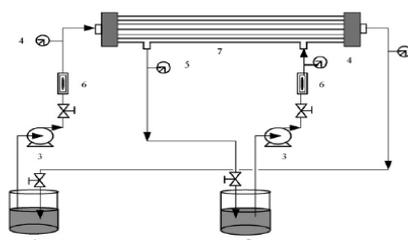


Fig. 2. Schematic counter-current flow diagram for a single-module operation in the HFSLM: (1) feed reservoir; (2) stripping reservoir; (3) gear pumps; (4) inlet pressure gauges; (5) outlet pressure gauges; (6) flow meter; (7) hollow fiber module

III. THEORETICAL BACKGROUND

A. Principle and Theory of the HFSLM System

The HFSLM system consists of 3 phases those are feed phase, organic membrane phase and stripping phase. The aqueous feed solution was lead (II) ions (Pb^{2+}) and HNO_3 was used as stripping phase. The aqueous feed phase and stripping phase were in contact with the organic membrane phase. The extractant (bis(2-ethylhexyl) phosphoric acid, D2EHPA in kerosene) is filled in the membrane pores. The aqueous feed flows inside the tube and stripping solution flows in the shell

of module. The flow between tube and shell sides is in counter current direction. The transport mechanism of lead (II) ion is so called *couple facilitated counter-transport*, as shown in Fig. 3. It involves various equilibrium reactions which are described as follows:

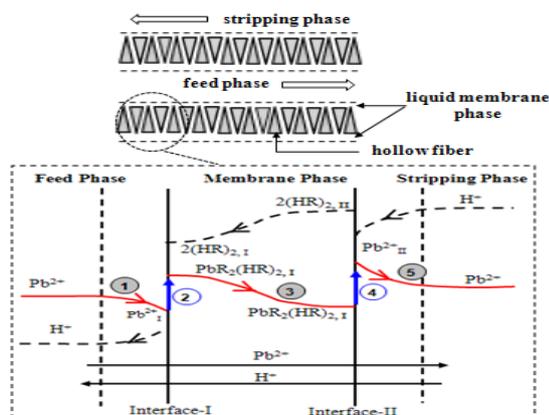
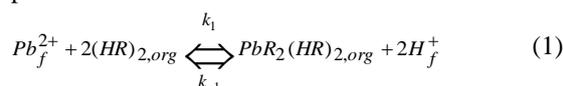


Fig. 3. Transport scheme of extraction and stripping in a liquid membrane process using D2EHPA as a carrier

(i) Pb^{2+} diffuses from the feed phase to the feed-membrane phase interface.

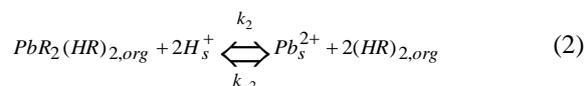
(ii) On the feed-membrane phase interface, the extraction reaction of Pb^{2+} with carrier D2EHPA in kerosene can be expressed as:



where f and org are the feed and organic phase, respectively; $(HR)_2$ indicates that the D2EHPA in kerosene; k_1 and k_{-1} are forward and backward reaction rate constant at the feed-membrane phase interface.

(iii) The ^{metal} complex ($PbR_2(HR)_2$) diffuses through the membrane phase from interface-I to interface-II by the concentration gradient of metal complex.

(iv) The metal complex reacts with the hydronium ions at the membrane-stripping phase interface. Pb^{2+} are stripped by stripping agent. The stripping reaction on the stripping side of the HFSLM is written as follows:



where s represents the stripping phase; k_2 and k_{-2} are forward and backward reaction rate constant at the membrane - stripping phase interface.

(v) Carrier D2EHPA returns from interface-II to interface-I.

B. The Percentages of Extraction and Stripping Calculation

$$\% \text{Extraction} = \frac{C_{f,in} - C_{f,out}}{C_{f,in}} \times 100 \quad (3)$$

$$\% \text{Stripping} = \frac{C_{s,out}}{C_{f,in}} \times 100 \quad (4)$$

where $C_{f,in}$ and $C_{f,out}$ are the inlet and outlet concentration of metal ion in the feed solution; $C_{s,out}$ is the outlet concentration of metal ion in the stripping solution.

C. Extraction Equilibrium Constant and Distribution Ratio

The extraction equilibrium constant (K_{ex}) of lead ion that was extracted by D2EHPA in (1), we can determine by using (6),

$$K_{ex} = \frac{[PbR_2(HR)_2][H^+]^2}{[Pb^{2+}][(HR)_2]^2} \quad (6)$$

The distribution ratio (D) of lead was given by

$$D = \frac{[PbR_2(HR)_2]}{[Pb^{2+}]} \quad (7)$$

According to the distribution ratio in (7), can be expressed as a function of the extraction equilibrium constant as follows:

$$D = \frac{K_{ex}[(HR)_2]^2}{[H^+]^2} \quad (8)$$

D. Permeability Coefficient

The permeability coefficient (P), fast interfacial reactions, and the distribution of lead between the feed and membrane phase were calculated to be higher than those between the membrane and stripping phase. The equation for determining the permeability coefficient was expressed by Denesi [32]:

$$-V_f \cdot \ln\left(\frac{C_f}{C_{f,0}}\right) = AP \frac{\beta}{\beta + 1} t \quad (9)$$

$$\beta = \frac{Q_f}{PL\epsilon\pi N r_i} \quad (10)$$

where P is the permeability coefficient (cm/s); V_f is the volume of the feed reservoir (cm³); $C_{f,0}$ is the initial lead concentration (mol/L); C_f is the lead concentration at any time (mol/L); A is the effective area of the hollow fiber module (cm²); t is time (min); Q_f is the volumetric flow rate (cm³/s); L is the length of hollow fiber (cm); ϵ is the porosity of hollow fiber; N is the number of hollow fibers in the module; and r_i is the internal radius of the hollow fiber (cm).

IV. RESULTS AND DISCUSSION

A. The Influence of PH in Feed Solution

The pH of feed phase is an important factor for the separation of lead ions due to metal can precipitate at an approximate pH value 7 [33]. Therefore, the pH value in feed solution was studied in the range 1-5. As shown in Fig. 4, pH of feed solution has an effect on the extraction of lead (II) and at pH 3 the maximum extraction yield is obtained. This maybe because at high concentration of H_2SO_4 which was used for adjust pH compete with D2EHPA to form complex [34].

B. The Influence of Carrier Concentration in the Membrane Phase

The concentration of carrier in the membrane phase has a significant influence on the extraction of lead ions through the HFSLM. The experiments were performed for the difference carriers in the concentration range 0.003-0.12 mol/L. Those

carriers are D2EHPA and Cyanex[®] 923. Both extractants are alkylphosphoric acid which mostly be used in acidic extractant group [35], [36]. It is indicated that at studied condition D2EHPA can extract lead (II) much higher than Cyanex[®] 923. From Fig. 5, the result show that extraction increase with increasing concentration D2EHPA of 0.003 to 0.12 mol/L, follow the reaction between metal and the organic extractant which can be described by (1), [37]. And the percentage of lead (II) extraction is slightly constant at above 0.12 mol/L of D2EHPA. Therefore, 0.12 mol/L of D2EHPA is the optimum concentration to extract lead (II) from wastewater.

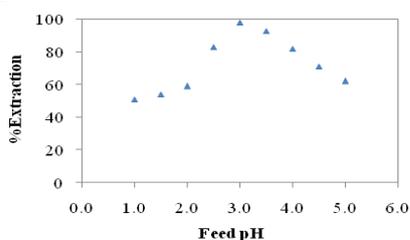


Fig. 4. The influence of feed pH on the extraction of lead ions

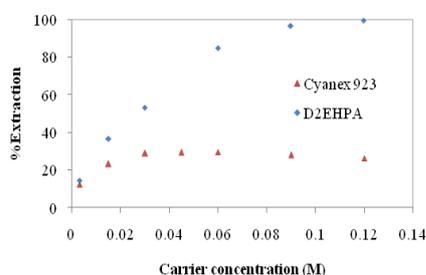


Fig. 5. The relation of carrier concentration and percentage of lead ions extraction

C. The Influence of the Types of Stripping Agents

The stripping agent in the stripping phase plays an important role in transport of metal ions from the organic membrane phase to the stripping phase. So the influence of different of stripping agent in the stripping phase on the transport of lead (II) was studied. The initial concentration of lead (II) in the feed phase, carrier concentration, pH value and volumetric flow rate of feed and stripping solution were adjusted to 7.5 mg/L, 0.12 mol/L, 3.0, and 100 mL/min, respectively. The experiments were performed under the different types of stripping agents that consist of nitric acid (HNO₃), sulfuric acid (H₂SO₄), and hydrochloric acid (HCl) in the concentration range 0.1-2.0 mol/L. The percentage of stripping by various concentrations of stripping agents was illustrated in Fig. 6.

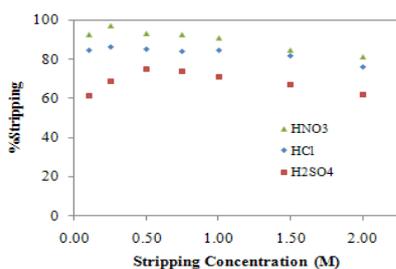


Fig. 6. Percentage of lead (II) stripping against concentration of stripping agents

The experimental results indicated that the stripping efficiency of lead decreased in the following order: HNO₃ > H₂SO₄ > HCl. Nitric acid provided the highest stripping efficiency and it obtained the maximum yield at the concentration 0.25 mol/L. So nitric acid can be used as the optimum stripping agent in these experiments.

D. The Influence of the Volumetric Flow Rate of Feed And Stripping Solution

Fig. 7 illustrated the percentages of extraction and stripping as a function of volumetric flow rate. The feed solution flows in the tube and the stripping solution flows in the shell as the counter current direction. The volumetric flow rates were studied at 100, 200, 300, and 400 mL/min under the condition of D2EHPA 0.12 mol/L, 0.25 mol/L HNO₃ as a stripping solution, and operation time 90 min.

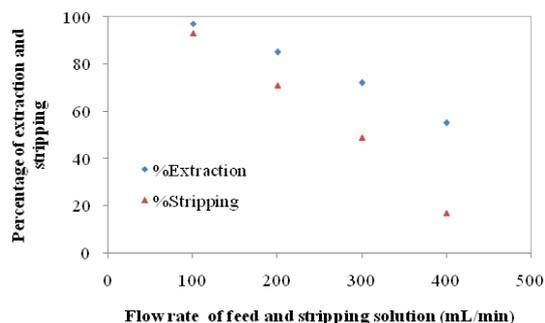


Fig. 7. The influence of various volumetric flow rates on the extraction and recovery of lead ions

The results indicated that the volumetric flow rate of feed and stripping solution at 100 mL/min obtained the highest percentage of extraction and stripping of lead ions about 99.40% and 97.15%, respectively. The percentage of extraction and stripping decreased with the increasing of the volumetric flow rate. The volumetric flow rate of feed solution plays an important role on the percentage of extraction and stripping of lead (II). Due to the low volumetric flow rate, the resident time of the relevant molecule in the reaction is higher than high volumetric flow rate. The high volumetric flow rate may remove the carrier from the support of liquid membrane and deteriorate the membrane system that can be seen from poor liquid membrane stability and lower percentages of extraction [37], [38].

E. The Extraction Equilibrium Constant and Distribution Coefficients

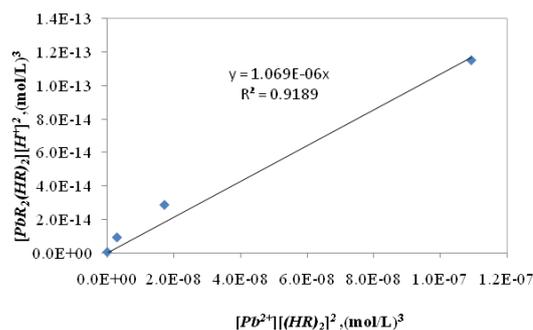


Fig. 8. The Equilibrium constant: plot of $[Pb^{2+}][HR_2]_2$ versus $[PbR_2(HR)_2][H^+]^2$

The extraction equilibrium constant of lead (K_{ex}) was evaluated from (6), by plotting $[PbR_2(HR)_2][H^+]^2$ against $[Pb^{2+}][HR]_2^2$ at the equilibrium condition and It should obtain straight line with the intercept at zero. The value of K_{ex} would be obtained from the slope of the graph that was shown in the Fig. 8. The value of K_{ex} was 1.069×10^{-6} . The distribution coefficients (D) of lead extraction from HFSLM system were presented in the Table 2. All of the distribution coefficients were calculated by obvious K_{ex} and (8). It was observed that the distribution coefficients increased with the concentration of D2EHPA.

TABLE II: THE DISTRIBUTION RATIO (D) OF LEAD (II) AT THE CONCENTRATION RANGE OF D2EHPA 0.003-0.12 MOL/L

D	D2EHPA (mol/L)					
	0.003	0.015	0.030	0.060	0.090	0.120
	0.086	0.338	0.639	1.016	1.755	2.952

F. The Permeability Coefficients

The permeability coefficients of lead as function of D2EHPA concentration were studied in the range 0.003-0.12 mol/L. We used (9), to evaluate the permeability coefficients of lead by plotting $-V_f \ln(C_f/C_{f,0})$ as a function of time (t) as shown in Fig. 9.

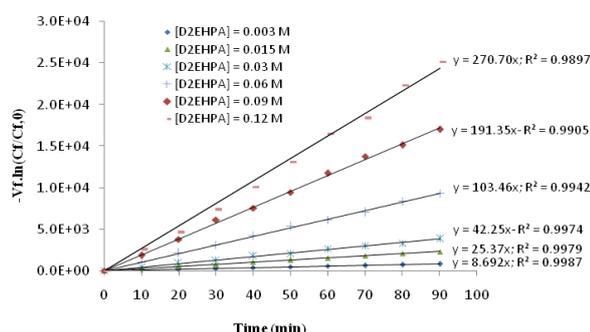


Fig. 9. Plot of $-V_f \ln(C_f/C_{f,0})$ of lead (II) in feed solution against the time at the difference concentration of D2EHPA

TABLE III: THE PERMEABILITY COEFFICIENTS (P) AT THE CONCENTRATION RANGE OF D2EHPA 0.003-0.12 MOL/L

P (*10 ⁴ cm/s)	D2EHPA (mol/L)					
	0.003	0.015	0.030	0.060	0.090	0.120
	0.105	0.310	0.525	1.375	2.824	4.435

It should obtain straight line with the intercept at zero and slope was $AP(\beta/\beta+1)$. The permeability coefficients can be obtained from (10), and all of values were presented in Table 3. The results showed that the permeability coefficients increase with the carrier concentration that related with obvious report from Pancharoen et al. [38].

V. CONCLUSION

This work focused on the study of the removal of lead (II) from wastewater of battery manufacturing by using the hollow

fiber supported liquid membrane. The influences of various parameters on the transport of lead (II) were studied to obtain the optimum conditions. The highest percentage of extraction and stripping of lead (II) at the optimum condition are 99.40% and 97.15%, respectively. It was achieved in 90 minutes at the concentration 0.12 mol/L D2EHPA, stripping concentration of 0.25 mol/L HNO₃. The volumetric flow rate of feed and stripping were maintained at 100 mL/min and keeping feed:strip ratio 1:1 ($V_f = 5000 \text{ cm}^3$ and $V_s = 5000 \text{ cm}^3$). In these experiments, feed and stripping solutions were continuously replaced with renewing solution in order to achieve high concentration of lead (II) in stripping phase. The results of this work can be concluded that hollow fiber supported liquid membrane process is a valuable alternative to conventional process for lead (II) stripping from wastewater of battery manufacturing.

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