

## Metallic ions extraction and transport in supported liquid membrane using organo-phosphoric compounds as mobile carriers

S. Azzoug<sup>a</sup>, O. Arous<sup>a,b,\*</sup>, H. Kerdjoudj<sup>a</sup>

<sup>a</sup> Laboratory of Hydrometallurgy and Inorganic Molecular Chemistry, U.S.T.H.B.-Faculty of Chemistry, Bab Ezzouar, Algeria

<sup>b</sup> Center of Research in Physical and Chemical Analysis, CRAPC, PO Box 248, Algiers 16004, Algeria

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### ABSTRACT

The extraction and transport of cadmium (II) and lead (II) in supported liquid membrane (SLM) using tributylphosphate (TBP), tris-ethylhexyl phosphate (TEHP), trioctylphosphine oxide (TOPO) and di-2-ethylhexyl phosphoric acid (D<sub>2</sub>EHPA) as mobile carriers was studied. The liquid–liquid extraction tests showed a maximum distribution coefficient when the carrier concentration was greater than 10<sup>-3</sup> M. In transport experiments the transmembrane flux increased with increasing carrier concentration reaching a limiting value ( $\sim 5 \times 10^{-10}$  mol cm<sup>-2</sup> s<sup>-1</sup>) at greater than 10<sup>-3</sup> M concentration. An impregnation time of 1 h of the support in the organic phase is necessary for giving a good efficiency to the supported liquid membrane (SLM). The other fundamental parameters optimum (carrier concentration, metal concentration and support characteristics) were determined. The permeability was significantly higher using Celgard 2400 support with respect to Celgard 5550, 3401, 2500 and 2402. A comparative study of the transport across such a membrane containing TBP, TEHP, TOPO or D<sub>2</sub>EHPA has shown that the cadmium (II) and lead (II) transport efficiency was decreased using a mixture of D<sub>2</sub>EHPA with other carriers.

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### Introduction

Industry generates large amounts of wastes which can harm nature and human health. Metal ions such as cadmium and lead are some of the harmful species found in these wastes [1]. Selective removal of metal ions from industrial and waste solutions is frequently required in hydrometallurgical processing. In this case, the concentration of metal ionic species is above 10<sup>-4</sup> M. In order to avoid the uncontrolled waste of this type of pollutants, it is necessary to develop techniques that allow their recovery and reutilization of treated wastewater for irrigation.

In the liquid–liquid extraction technology, the extractant plays a characteristic role for the separation of metal ions. A suitable choice of the extractant decides the success of the extraction process. Several extractants were developed and their properties of extraction were evaluated for various metal ions [2]. Acid organo-phosphorus compounds are excellent extractants. They are particularly used for the purification of the liquid effluents likely to contain heavy metals [3]. Numerous extractants, such as tributylphosphate [4], trioctylphosphine oxide [5], triphenyl-phosphine oxide [6], have

been used for the liquid–liquid extraction of Zn(II). Di(2-ethylhexyl)phosphoric acid is available commercially. It has been widely used in the extraction of a range of metals including nickel, cobalt, copper, chromium and bismuth [7].

In general, membrane techniques of separation, purification and recovery using Donnan dialysis [8,9], liquid membrane [10], electrodialysis [11–15] and electro-deionisation [16] have been used as “cleaner” processes. It is possible to remove some species present in the feed or improve their permeation through the membrane after modification of the ion exchange membrane properties [17,18]. In all the membrane separation process, membrane used is basically a thin film and porous in nature, which acts as a semi-permeable barrier for allowing certain component to transport and others to reject. Depending on the feed and the process the product may be permeated or may be rejected by the membrane.

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 [19,20]. Hence, more efficient and cost-effective removal and recovery methods are sought after to overcome these difficulties. Of all these techniques, liquid membrane (LM) has been given considerable attention by the researchers for removal and recovery of heavy metals from aqueous solutions. However, LM techniques have not been adopted for large-scale industrial processes [21–26], principally

\* Corresponding author at: Laboratory of Hydrometallurgy and Inorganic Molecular Chemistry, U.S.T.H.B.-Faculty of Chemistry, Bab Ezzouar, Algeria.

Tel.: +213 21 24 79 52; fax: +213 21 24 73 11.

E-mail address: [omararous@yahoo.fr](mailto:omararous@yahoo.fr) (O. Arous).

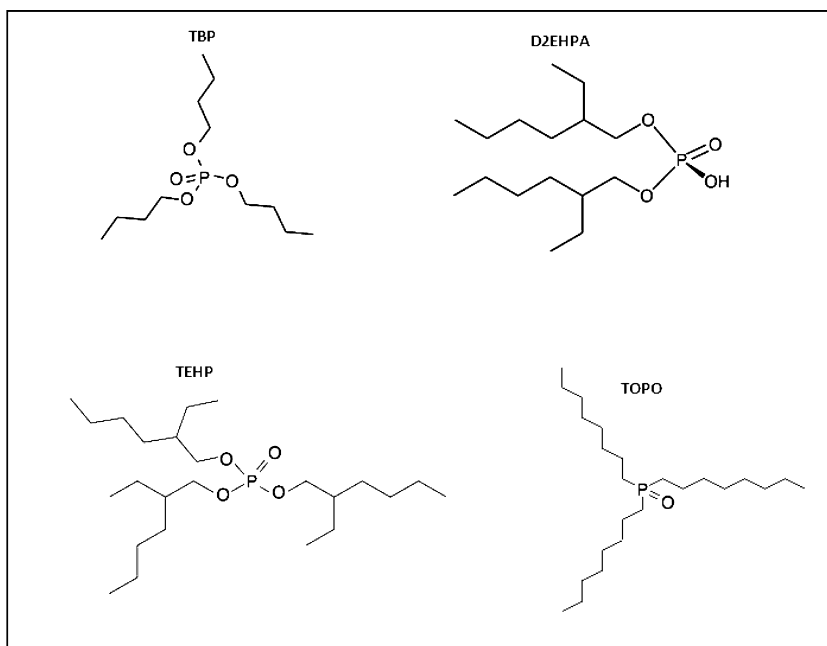


Fig. 1. Organo-phosphorous carriers' structures.

due to the lack of longtime stability, difficulty in operation of the emulsification and de-emulsification.

Liquid membrane processes have become an attractive alternative to conventional solvent extraction for selective separation and concentration of compounds such as metals and acids from dilute aqueous solution because they combine in a single stage, an extraction and a stripping operation. Development of supported liquid membrane (SLM) is now of significant importance in separation, purification, or analytical applications in areas such as biomedical technology or water treatment [27–34]. SLM can carry out extraction and stripping processes simultaneously, and they show advantages of non-equilibrium mass transfer and up-hill effect, which means that solute, can be moved from low-to-high concentration solution [35–39].

The main use of organo-phosphorus compound is in solvent extraction of metals. The high lipophilicity and high polarity are properties key to this application. Their high polarity, which results from the dipolar phosphorus–oxygen bond, allows this compound to bind to metal ions. The octyl groups confer solubility in low polarity solvents such as kerosene. The major uses of organo-phosphorus compound in industry are as a component of aircraft hydraulic fluid and as a solvent for rare earth extraction and purification.

In the present work, we compared the behavior of a di(2-ethylhexyl)phosphoric acid (D<sub>2</sub>EHPA), a trioctylphosphine oxide (TOPO), a tributylphosphate (TBP) and a tris-ethylhexyl phosphate (TEHP), toward liquid–liquid extraction and facilitated transport of lead and cadmium through a supported liquid membrane.

## Experimental

Analytical-grade inorganic chemicals: lead nitrates and cadmium nitrate (Pb(NO<sub>3</sub>)<sub>2</sub>, Cd(NO<sub>3</sub>)<sub>2</sub>) were obtained from CARLO ERBA.

Organic reagents: the ion carriers (2-ethylhexyl) phosphoric acid (D<sub>2</sub>EHPA), tributylphosphate (TBP) and trioctylphosphine oxide (TOPO) were obtained from Aldrich. Tris-ethylhexyl phosphate (TEHP) was purchased from Merck. The chemical structures of ligands are presented in Fig. 1, and Table 1 regroups formula, molecular weight and water solubility of used organic carriers.

Double distilled water was used as the stripping solution. The aqueous solutions are prepared by dissolving different reagents in bi-distilled water (~2 MΩ cm). Bi-distilled water is prepared by double distillation of water. It was the de facto standard for highly purified laboratory water for biochemistry and, by the method of trace analysis until combination methods of purification became widespread. The water does not contain greater amounts of impurities, apart from certain gases, such as carbon dioxide from the air which can slightly affect its pH.

Analysis by atomic absorption spectroscopy (AAS) requires the use of high purity reagents and solvents to ensure the accuracy and precision of measurements. Water is used to prepare blanks, standards, and in the sample preparation procedure, hence it should be free of the elements under investigation, as well as any elements and compounds whose presence could interfere with the analysis.

Equilibrium investigations were carried out by liquid–liquid extractions: equal volumes of the aqueous phases (10 mL), containing the investigated metal, and of the organic phases containing organo-phosphorus carriers in chloroform at different concentrations were contacted for 1 h at a stirring speed of 2400 rpm using a mechanical stirrer (Heidolph TOP-MIX 94323). Then the two phases were completely separated by centrifugation. All the experiments were performed at 25 °C.

The experimental studies on lead and cadmium extraction were carried out using organic feed concentrations of 10<sup>−5</sup>–10<sup>−1</sup> M,

Table 1  
Carriers' characteristics.

Carrier	TOPO	TBP	TEHP	D <sub>2</sub> EHPA
Formula	C <sub>24</sub> H <sub>51</sub> OP	C <sub>12</sub> H <sub>27</sub> O <sub>4</sub> P	C <sub>24</sub> H <sub>51</sub> O <sub>4</sub> P	(C <sub>8</sub> H <sub>17</sub> O) <sub>2</sub> P(O)OH
Molecular weight (g mol <sup>−1</sup> )	386.63	266.31	434.63	322.43
Solubility in water (g L <sup>−1</sup> )	Immiscible	0.4	≤0.1	≤0.01

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