Extraction of Heavy Metal Using Span 80 as Surfactant Process Optimization

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Abstract—The technique of emulsion ionic liquid membrane (EILM) was used as chemical process for Pb(II) recovery, with nitrate ions as counter-ions, optimizing one experimental parameter at a time. This process was carried out using a quaternary amine extractant (Aliquat 336) and a dispersive non-ionic surfactant (Span 80), commonly employed for emulsion formation and stabilizing water-in-oil (W/O) emulsions due to its low HLB value of approximately 4.3.

The most influential experimental factors such as extractant concentration, surfactant concentration, solution pH, and the nature and concentration of both internal and external phases were systematically studied. The results showed that Pb(II) ions were completely extracted (100%) by Aliquat336, in aqueous solution of the nitric acid, from a feed phase of lead(II) nitrate of 100 ppm at pH equal to 6.3, in presence of 12% w/w Aliquat336 and 0.2% w/w of Span 80 under 50 min of stirring at 250 rpm. The lead (II) separation experiments were conducted under the optimal conditions for lead (II) recovery. A comparative table shows the results and differences between several techniques.

Keywords— **Emulsion liquid, membrane, Span 80, Aliquat 336, lead**

I-Introduction

In environmental toxicology, exposure to heavy metals is a major problem due to their toxicity to ecosystems. This exposure can be natural in origin, but it is mainly linked to human activities such as industry, agriculture and fossil fuel combustion. Heavy metals can accumulate in the body and cause adverse effects on human health, as they can also be easily absorbed by marine animals and enter the human food chain directly, posing a high risk to consumers.

For this reason, rigorous control and monitoring are necessary. [1, 2]

The complexity and difficulty associated with effluents containing heavy metals have prompted researchers to explore numerous possible solutions.

Faced with these risks, it is essential to recover and eliminate these pollutants from various industrial effluents in order to meet increasingly stringent environmental quality standards and enable the recycling and recovery of heavy metal resources. [2]

Currently, in the field of hydrometallurgy, the extraction and preconcentration of metals is a challenge and a major economic issue, and heavy metals such as lead pose a serious threat and are a major concern for flora and fauna.[2]

Among these heavy metals, lead stands out as being non-biodegradable and highly toxic. Its presence in the body causes carcinogenic effects, high blood pressure, kidney disease, teratogenic effects, fertility problems and pathologies affecting the physical and mental integrity of human beings. [1, 2]

Traditional methods for extracting metal ions from environmental matrices include physical processes such as adsorption, filtration, and flotation; chemical processes such as coagulation-flocculation, oxidation, reduction, electrolysis, and photochemistry; and biological processes such as aerobic and anaerobic degradation.

Despite their widespread use, these methods have disadvantages such as high operating costs, low efficiency at low metal concentrations, the need for slower processing times and the use of large quantities of chemicals.

The extraction techniques based on the liquid membranes can be an alternative solution to overcome these constraints (Hou et al., 2015; Lu and Dreisinger, 2014). The technique of emulsion liquid membrane (EILM)was invented by Li in 1968. This technique has drawn a great attention by many scientific and industrial researchers for the hydrometallurgical processes of heavy metals recovery. EILM has presented a high efficiency, compared to conventional techniques, in the recovery of metal ions and hydrocarbons from wastewater (Chakrabarty et al.,2010; Kitagawa et al., 1977). EILM process consists in a three-phase format (W/O/W). A continuous aqueous phase (W) enters in contact with a hydrophobic organic liquid; called, a membrane phase (O) containing a strip agent solution (W), dispersed in the form of fine droplets to create the emulsion globule which offers an important mass transfer due to the high interfacial area (Gameiro et al., 2007).[3-4]. An adequate surfactant should be added to the organic liquid to ensure the formation and stability of emulsion, and enhance the selectivity and permeability of the membrane (Frankenfeld and Li, 1987).

The concentration of metal ion at membrane/aqueous interface must be maintained at zero to keep a continuous driving force of permeation (Park and Chung, 2003). The transport of metal ion is governed by the kinetic rather than the conditions of equilibrium as in solvent extraction processes (Frankenfeld and Li, 1987; Pellegrino and Noble, 1990).

In recent years, ionic liquids (ILs), often referred to as “green solvents,” have emerged as an alternative to traditional hydrometallurgical methods for metal ion separation, increasingly replacing many organic solvents that are costly, toxic, or harmful to the environment.

This is attributed to their characteristics such as zero volatility, excellent miscibility and solubility with both organic and inorganic compounds, and low toxicity. They remain in a liquid state over a broad temperature range. (Abbott and McKenzie, 2006; Coll et al., 2012;Tian, 2012).

Aliquat 336 is a commercially available ionic liquid, also known as Starks's catalyst. It is a quaternary ammonium salt, insoluble in water, and is used both as a phase-transfer catalyst and as a carrier for metal ions. [article mimi]

The experiments of extraction of lead (II)) were realized by Aliquat336 and Span 80 using the technique of emulsion ionic liquid membrane (EILM). The optimization method of one experimental parameter at the time was adopted for determination of the best conditions of lead (II) recovery, from its nitrate aqueous medium.

II-experimental

1- Reagents

Aliquat 336 is a quaternary ammonium salt, insoluble in water, resulting from the methylation of tri-octyl/decylamine, was used without further purification.

The non-ionic surfactants used is: Sorbitan monooleate (Span 80) with the following properties: the critical micellar concentration is equal to 0.01–0.1 g/L (20–25◦C), the HLB balance is of 4.3. These products were purchased from Sigma–Aldrich (Steinheim, Germany).

Heptane was pur-chased from Merck (Darmstadt, Germany). Sulfuric acid was provided from Prolabo (VWR International France). The stock of lead nitrate was supplied by Fluka (Germany).

2- Apparatus

The experiments of metal ion recovery were conducted by a mechanical stirring during the equilibrium time of the extraction reaction using a platform agitator; type A Haier (Beijing,China). The study of the pH effect on extraction and pre-concentration of metal ions, was carried out with a pH-meter; type Consort C831 (Turnhout, Belgium). An analytical balance; type Kern ABS (Balingen, Germany), was used in the weighing operation. The experiments of the emulsion formation were carried out by using a homogenizer; type Vortex (Germany)at 2500 rpm. An Ultra-8TL centrifuge model (LW Scientific, Lawrenceville, USA) was used for the phase separation. UV-Visible Spectrophotometer: Hach Lange DR 5000 model, used to measure the absorbance of the PAR-Pb(II) colour complex.

3- Preparation of emulsion

The membrane used for metal extraction consists of an extractant, Aliquat 336, dissolved in a volume of heptane solution, with the surfactant Span 80, An organic solution of 7.5 mL was prepared. The purification solution used is 0.5M hydrochloric acid (HCl), which is added drop by drop to the membrane phase. The emulsion is formed using a Vortex mixer, maintaining a rotation speed of 1800 revolutions per minute (rpm) for a period of 20 minutes.

4- Batch ELM experiment

The emulsion phase (W/O emulsion) was added to a volume of62.5 mL of feed solution of metal ions then, obtained solution is mixed during a time of extraction. This block of emulsion liquid membrane was used in the study of the permeation of the metal ion.

5- Analytical response

The study of recovery process of metal ion was evaluated by the yield of extraction (Eq. (1))

$$\text{Yield of Extraction, Y(\%)} = \frac{C_{0-}\ C}{C_0} \text{ x } 100 \qquad (1)$$

III. Result and discussion

1- Influence of surfactant concentration

Surfactants play a crucial role in the development and extraction process of MILE. The choice of surfactant is an important parameter that depends on its HLB (Hydrophilic Lipophilic Balance) value.

In a MILE system, the addition of a surfactant as an emulsifier in the liquid membrane phase affects not only the stability of the membrane, but also the swelling of the emulsion and the transport rate of metals. [5]

In our experiment, lead(II) ions were extracted with 0.1% m/m Aliquat 336 while varying the concentrations of the surfactant (Span 80) from 0.2 to 14% m/m.

The figure shows the influence of the concentration of the Span 80 surfactant in the membrane phase on the extraction yield of lead(II).

The results show that extraction efficiency reaches a maximum value of 70.68% at a concentration of 0.2%. Above this concentration, there is a clear decrease in extraction efficiency until a point of equilibrium is reached. This is due to the increase in membrane viscosity and resistance to mass transfer caused by the interfacial film of the surfactant. [6]

These results are consistent with previous studies. [6,7]

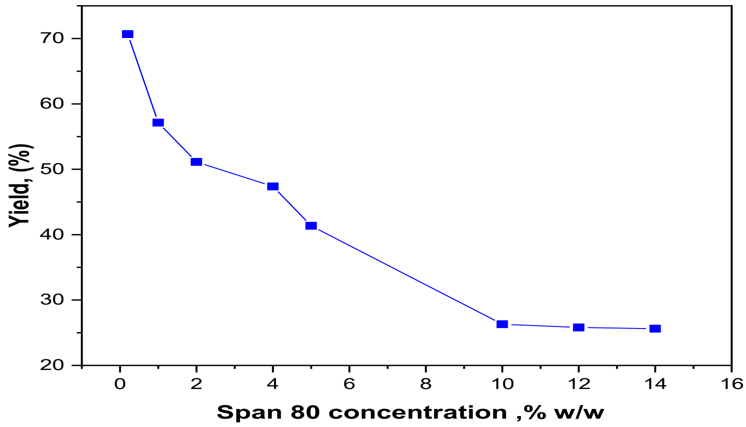

Fig. 1 Influence of surfactant concentration on the Pb(II)recovery. [Aliquat336] = 0.1% w/w; [Pb(II)] = 200 ppm; pHi = 6.3; stirring speed = 180 rpm, time = 30 min.

2- Influence of carrier concentration

In extraction using the liquid emulsion membrane (EILM) technique, the choice of carrier or extractant plays a vital role, facilitating the transport of metal species across the membrane through the formation of stable and reversible complexes. It must be soluble in the organic membrane phase and insoluble in the aqueous phases. This encourages the transfer of species from the external feed phase to the internal purification phase.

In our work, lead (II) ions are extracted with 0.2% m/m Span 80 while varying the concentration of the extractant (Aliquat 336) in the range of 0.5 to 16% m/m.

The results are illustrated in figure 2.

Figure 2 shows that the extraction yield increases with the increase in the amount of extractant (Aliquat 336) in the membrane phase. A maximum yield of 98.24% is obtained at a concentration of 12% m/m; above this value, the extraction yields remain constant.

This behaviour can be explained by the fact that increasing the concentration of Aliquat 336 initially promotes the formation of a greater number of extractant-metal complexes, thereby improving extraction efficiency.

In fact, there will be more extractant sites available: a higher concentration of Aliquat 336 in the organic phase allows more complexes to form with the target ion (Pb^{2+}), thereby increasing the amount extracted. [8]

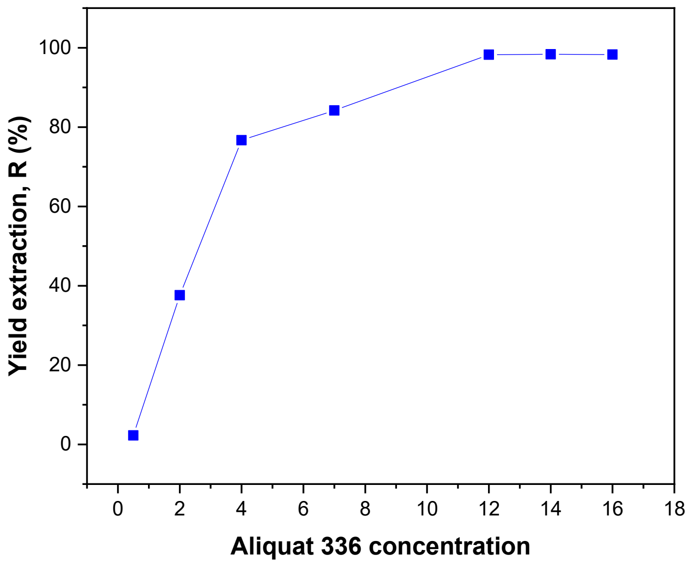

Fig. 2 Influence of carrier concentration on the Pb(II)recovery. [Pb(II)]=200 ppm; pHi = 6.3; stirring speed = 180 rpm, time = 30 min; [Span80]= 0.2%w/w.

However, beyond a certain concentration, the organic phase becomes saturated and the yield stabilises. This stabilisation is linked to the saturation of the active extraction sites within the membrane; more specifically, the interface can no longer accommodate any more extracting molecules, thus limiting any further improvement.
Furthermore, above a certain threshold, all available metal ions have already formed complexes. The system then reaches a plateau (stabilisation), where further increases in the extractant no longer improve the yield, as the limiting factor becomes the availability of the metal ion or the capacity for release into the internal phase. [9]
These studies are consistent with our results. [10,11]

3- Effect of the nature of the internal phase

In the emulsified liquid membrane extraction process, the extraction and purification stages occur simultaneously. It is therefore essential to study the influence of the nature of the purification phase in which the metal ions will be trapped, as numerous treatment solutions can be implemented in EILM systems. The choice of purification solution is crucial to ensuring the effectiveness of the EILM system. [12]
In this study, we worked with five different solutions: three acids and two bases are used in the extraction of metal ions. These solutions are: sulphuric acid, nitric acid, hydrochloric acid, sodium hydroxide and ammonium hydroxide.
The results are illustrated in figure 3.

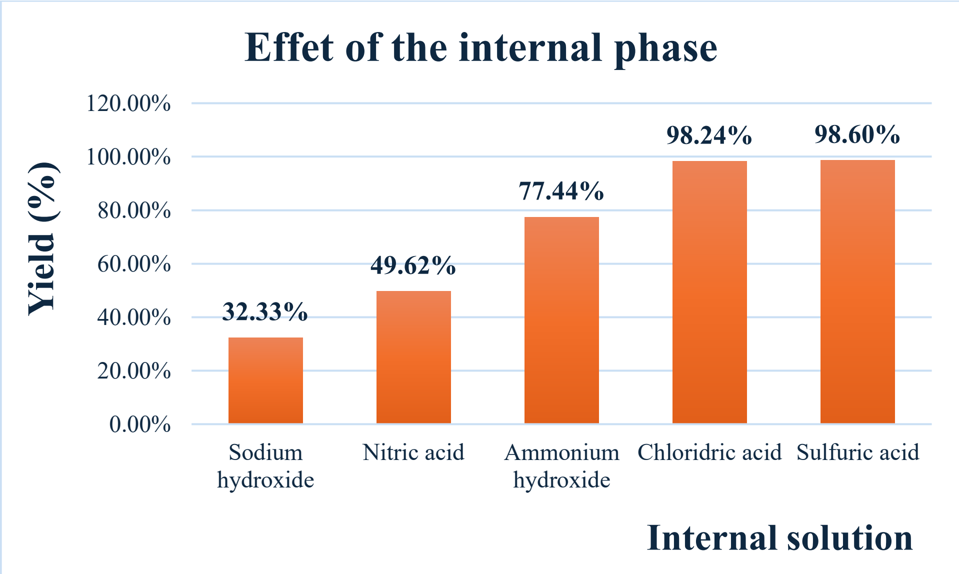

Fig. 3 Influence of nature of internal phase on the Pb(II)recovery. [Pb(II)]=200 ppm; pHi = 6.3; stirring speed = 180 rpm, time = 30 min; [Span80]= 0.2%w/w; [Aliquat336] =12%.

Analysis of the results shows that using sulphuric acid as the purification phase achieves an extraction yield of 98.6%. This performance can be explained by the high acidity of H_2SO_4, which promotes rapid and efficient deprotonation at the membrane/internal phase interface, thereby improving the breakdown of the metal-extractant complex and facilitating the transfer of Pb^{2+} ions.

Indeed, its high protonating power allows it to release more H^+ ions compared to other acids of equivalent concentration, which promotes the decomplexation of the metal (Pb^{2+}–Aliquat 336) and its effective release into the internal phase. [13]

The significant decrease in extraction yield observed with nitric acid, sodium hydroxide and ammonium hydroxide can be attributed to similar phenomena related to the disruption of emulsion stability. Hydroxides ($NaOH$; NH_4OH) often cause the metal to precipitate in the form of hydroxide ($Pb(OH)_2$), which blocks transfer and impairs yield. In fact, despite its acidic nature, nitric acid does not provide a sufficiently high proton gradient to ensure effective breakdown of the metal-extractant complex, which slows down the desorption of pb^{2+} ions. [13, 14]

4- *Effect of the concentration of the purification phase*

In an emulsified liquid membrane (EILM) system, the metal ion extraction process relies on two key interfaces: the interface between the aqueous feed phase and the membrane, where extraction occurs, and the interface between the membrane and the purification phase, where metal release and extractant regeneration take place.

The membrane's ability to extract metal is limited by the concentration of the internal phase. The influence of sulphuric acid concentration on Pb(II) extraction is shown in the following figure.

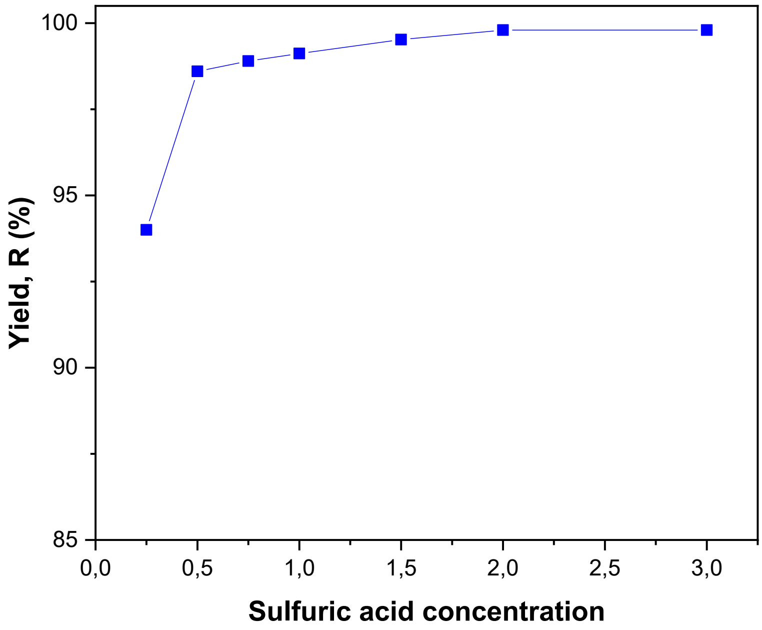

Fig. 4 Influence of concentration of purification phase on the Pb(II)recovery. [Pb(II)]=200 ppm; pHi = 6.3; stirring speed = 180 rpm, time = 30 min; [Span80]= 0.2%w/w; [Aliquat336] =12%.

According to the figure, the extraction yield of lead by the emulsified liquid membrane increases gradually with the increase in sulphuric acid concentration in the internal phase, reaching 99.8% at 2M and then stabilising.
This improvement is explained by the increase in the proton (H^+) gradient between the external phase and the internal phase, which facilitates the decomplexation of lead from the complex formed with the extractant and accelerates its transfer to the internal phase. In addition, more efficient metal release: a higher concentration of H_2SO_4 increases the availability of H^+ ions, which promotes the decomplexation of the metal-Aliquat 336 complex (by ion exchange) and thus the transfer of the metal to the internal phase. [15]
Recent studies have revealed that increasing the acid concentration not only promotes the release of the metal from the complex, but also the stability of the emulsion up to a certain critical threshold, thereby optimising extraction yield. Thus, at high acidity, once released, the metal remains stable in acid solution (Pb^{2+}), preventing any return to the membrane.[16]

5- Effect of the nature of the solvent

The study began by evaluating the influence of the diluent on the extraction of Pb(II) by three solvents with the following characteristics: Dichloromethane (polarity: p = 3.1), point d'ébullition = 40 °C, viscosité : η(cP) = 0.44 et densité (g/cm3) : ρ = 1.327) ; l'hexane (polarity : p = 0.1), boiling point = 69 °C, viscosity : η(cP) = 0.32 and density (g/cm^3) : ρ = 0.6548 , for heptane (polarity : p = 0.1), boiling point = 98 °C, viscosity : η(cP) = 0.386 et density (g/cm^3) : ρ = 0.684).

The figure 5 below shows the evolution of the extraction yield of Pb(II) as a function of the nature of the solvent.

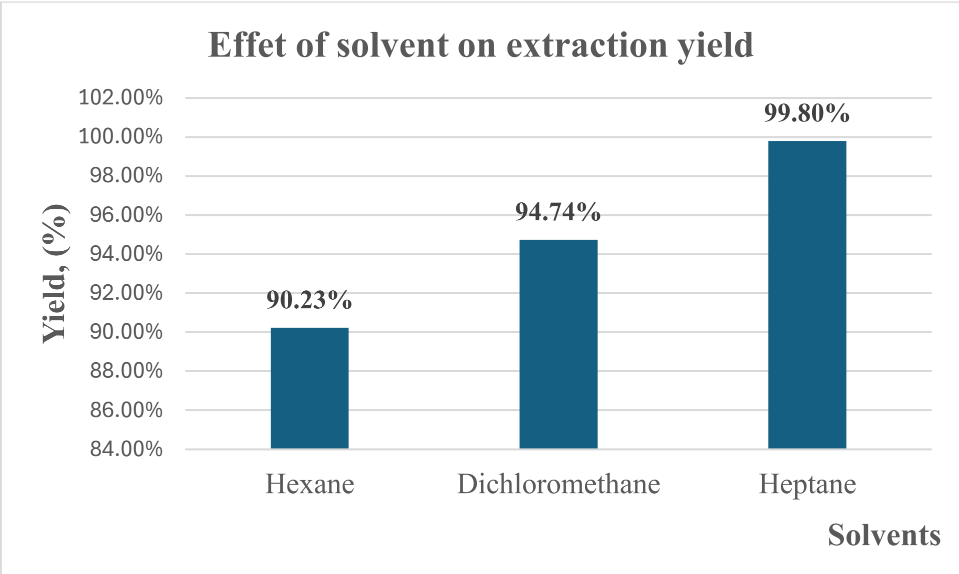

Fig. 5 Influence of solvent on the Pb(II)recovery. [Pb(II)]=200 ppm; pHi = 6.3; stirring speed = 180 rpm, time = 30 min; [Span80]= 0.2%w/w; [Aliquat336] =12%.

The evaluation of the effect of the nature of the solvent on the extraction of Pb^{2+} showed that the best extraction yield was obtained using heptane (99.8%). This result can be explained by its advantageous physicochemical properties, in particular its low volatility. Heptane evaporates less quickly than hexane or dichloromethane, which makes the membrane more stable throughout the extraction process, especially in open systems, and offers good compatibility with Aliquat 336: it allows for better solubility and dispersibility of the extractant (Aliquat 336), promoting the formation of stable complexes with the metal ion. Dichloromethane (94.74%) and hexane (90.23%) showed slightly lower efficacy. Dichloromethane, although polar with the ability to solubilise metal complexes, tends to destabilise the emulsion due to its high volatility. Hexane, on the other hand, is very low in polarity, more volatile and more toxic, limiting the solubility of the extracted complex and thus reducing mass transfer.

Heptane stands out as the most effective solvent, as it offers a good compromise between emulsion stability and pb(II) transport efficiency. [17]

6- Influence of initial pH of feed phase

pH is a critical parameter in the transport of metal ions through the liquid emulsion membrane (EILM). Its role is evident in the nature of the metal species present in aqueous solution and in the behaviour of the extractant's functional group. [18]

The study of the influence of the initial pH of the metal solution on EILM extraction is carried out for the metal ion pb(II).

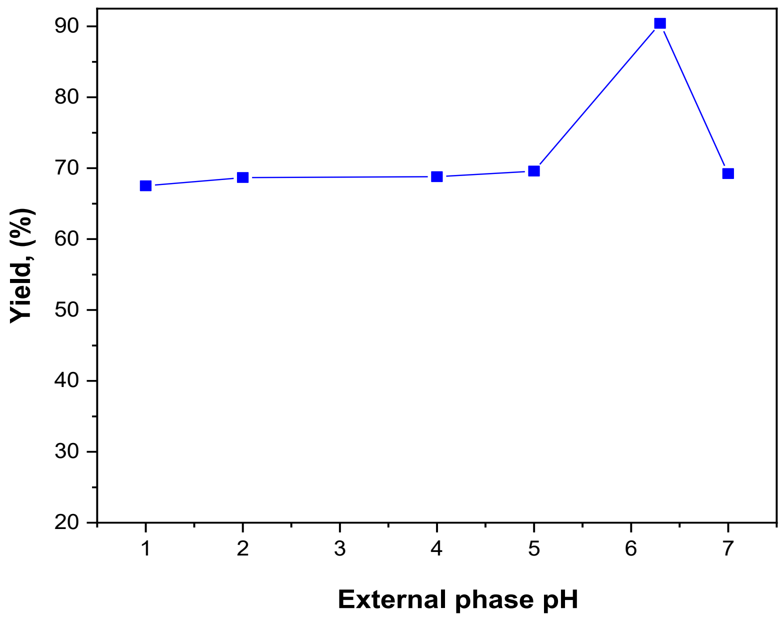

Fig. 6 Influence of initial pH on the Pb(II)recovery. [Pb(II)]=200 ppm; stirring speed = 180 rpm, time = 30 min; [Span80]= 0.2%w/w; [Aliquat336] =12%.

The extraction of Pb(II) ions increases with the elevation of the initial pH of the feed phase, rising from 1 to 6.3 (figure 6). The maximum extraction yield Y = 99.42% is achieved at a pH of 6.3. This can be explained by:

At low pH (acidic): lead ions (Pb^{2+}) are well dissolved, but the medium is too rich in H^+, which competes with lead for ion exchange with the extractant (Aliquat 336), reducing extraction efficiency.

At moderately acidic pH (optimal): there is less competition from H^+, and the lead remains in the form of free Pb^{2+}. This is the optimal extraction zone, as the extractant interacts effectively with Pb^{2+}.

At high pH (basic): lead begins to precipitate as $Pb(OH)_2$, which reduces its availability in ionic form for extraction, thus decreasing the yield. [19]

7- *Effect of stirring speed and time*

The stirring speed plays a crucial role in the formation of emulsion droplets during the preparation of the emulsified phase. It also has an impact on membrane stability and the transfer rate of metal ions in the emulsified liquid membrane technique.

The effect of this parameter was studied for a range of stirring speeds between 0 and 250 rpm. The results obtained are shown in figure 7.

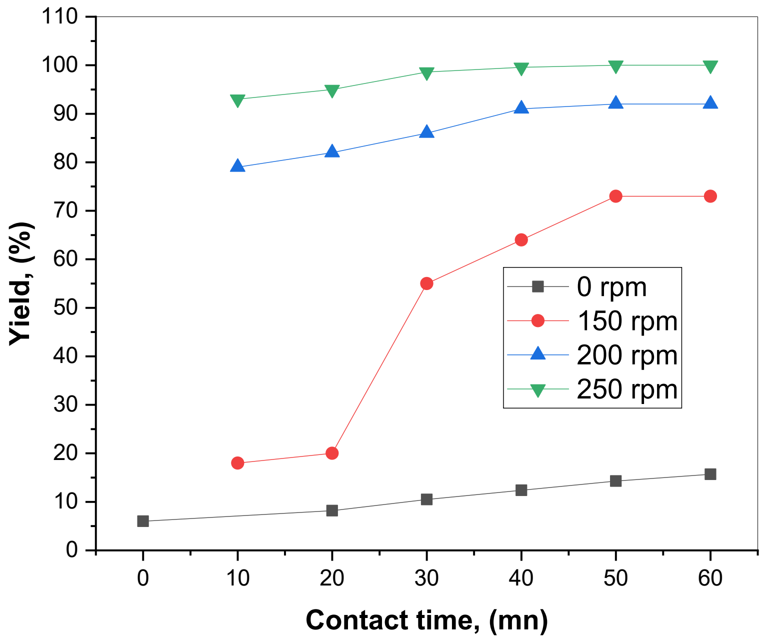

Fig. 7 Effect of time and stirring speed on the extraction of Pb(II). [Pb(II)]=200 ppm; pHi = 6.3; [Span80]= 0.2%w/w; [Aliquat336] =12%.

According to the figure, it can be seen that the lead extraction yield increases with increasing stirring time and speed until it reaches a state of equilibrium with a maximum yield (100%) at 250 rpm and a contact time of 50 minutes.

This could be attributed to the increase in shear rate, promoting the formation of smaller emulsion globules, which leads to an increase in the interfacial area between the external aqueous phase and the globules, thus promoting greater mass transfer. [20]

This can be explained by the fact that agitation promotes renewal of the interface between phases, reducing the diffusion boundary layer and accelerating the transfer of the Pb-extractant complex across the membrane.

Furthermore, moderate to rapid agitation promotes a finer and more stable emulsion, increasing the interfacial exchange surface available for extraction. [21]

However, it is important to note that excessive agitation could theoretically lead to droplet coalescence and emulsion breakdown beyond a certain threshold, but in this specific case, stability appears to be maintained up to 250 rpm. [22]

8- Effect of the initial concentration of the feed phase

The variation in solute concentration affects the driving force of transport as well as the mass transfer rate through the emulsified liquid membrane. [23]

The influence of the initial concentration of the feed phase (external phase) on Pb(II) extraction is shown in figure 8.

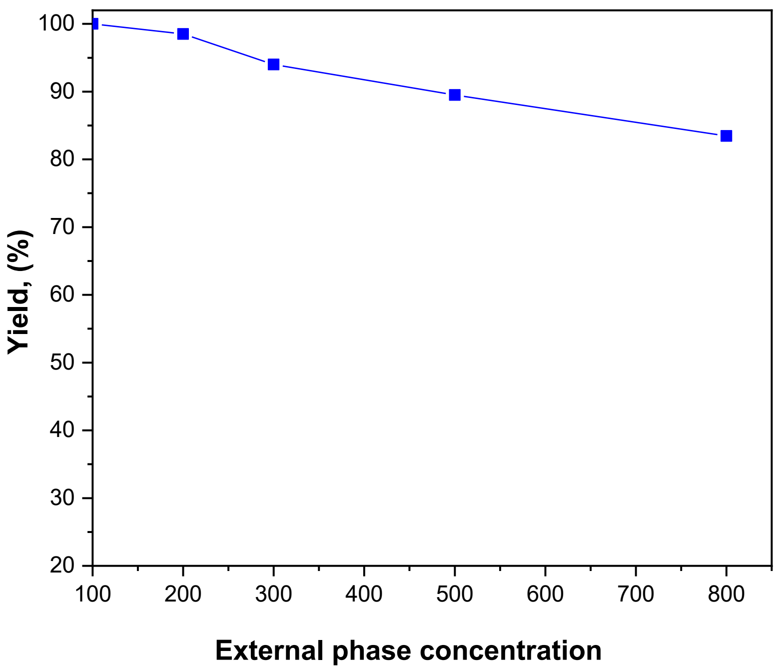

Fig. 8 Effect of intial concentration of the feed phase on the extraction of Pb(II). pHi = 6.3; [Span80]= 0.2%w/w; [Aliquat336] =12%.

The figure shows that the extraction yield of Pb is maximum (100%) at a concentration of 100 ppm and gradually decreases as the concentration of the feed phase increases.

When the feed phase is concentrated further, extraction yields decrease. This is attributed to an increase in osmotic pressure. This pressure difference between the internal and external phases causes the emulsion droplets to swell, destabilising the emulsified liquid membrane and causing a decrease in mass transfer.[24]

IV. Comparative study on lead extraction

A comparative study for lead extraction is given in table below:

TABLE 1

Comparative study for Pb (II) extraction

Reference	Metal	Surfactant	Extractant carrier	Internal phase	Diluent	Extraction Yield
[25]	Cr(II), Mn(II),Co(II),Pb(II),Ni(II), Cu(II)	Span 80	8-Quinolinol	HCl	Toluene	90
[26]	lead	Span 80	D2EHPA	-	Kerozene	99.50
[27]	Lead and cadmium	ECA 5025	D2EHPA	HCl, H_2SO_4	Tetradecane	-
[3]	Lead	TritonX-100 and Tween-20	Aliquat 336	HNO_3	heptane	82.16
[28]	lead	Span 80	D2EHPA	H_2SO_4	Kerozene	99
Present work	lead	Span 80	Aliquat 336	H_2SO_4	heptane	100

V. Conclusion

The experimental study of recovery process of Pb(II), from nitrate aqueous solution, by the ionic liquid carrier (Aliquat336) using emulsion liquid membrane technique, showed that the lead ions were extracted at 100% in ionic liquid membrane

This was obtained by optimizing one parameter at the time. The optimal experimental conditions were: [Aliquat336] = 12% w/w; [Span 80] = 0.2% w/w; extraction time: 50 min; stirring speed of extraction: 250 rpm; [Pb(II)] = 100 ppm and pH of feed phase: 6.3.

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