



## Recovery of nickel ions by supported liquid membrane (SLM) using D2EHPA as carrier

Kemla Othmen<sup>a</sup>, Sana Ncib<sup>a</sup>, Afef Barhoumi<sup>a</sup>, Lassaad Dammak<sup>b</sup>, Wided Bouguerra<sup>a,c,\*</sup>

<sup>a</sup>U.R Matériaux, Environnement et Energie (ME2) (UR 14 ES26), Département de Chimie, Faculté des Sciences de Gafsa, Campus Universitaire Sidi Ahmed Zarroug – 2112 Gafsa, Tunisia, email: bg\_Wided@yahoo.fr

<sup>b</sup>Institut de Chimie et des Matériaux Paris-Est (ICMPE), UMR 7182 CNRS - Université Paris-Est Créteil, 2 Rue Henri Dunant, 94320 Thiais, France

<sup>c</sup>Faculté des Sciences de Gafsa, Université de Gafsa, Gafsa, Tunisia

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### ABSTRACT

This research is dealing with the liquid–liquid extraction and the facilitated transport through a supported liquid membrane (SLM) system of nickel ions. Di(2-ethylhexyl) phosphoric acid (D2EHPA) was used as a carrier dissolved in chloroform. The effect of critical parameters such as the amount of D2EHPA (in membrane), the initial concentration of nickel as well as feed phase pH which affect the transport of nickel(II) through the SLM system, were investigated. Experimental results revealed that Ni(II) flux across the membrane tends to increase with the concentration of these metal ions. The optimum conditions for Ni(II) transport are: feed phase pH of 5, stripping phase of 0, 5 M HNO<sub>3</sub>, and 30% D2EHPA (v/v). At optimal conditions, the transport of Ni(II) was achieved with an efficiency of about 60% within 24 h and 100% within 3 h for initial nickel concentration of 2.5 and 0.25 mM, respectively. A concluding aging test was carried out to check the stability of the membrane and the evolution of the percentage of nickel transported in the function of time.

*Keywords:* Nickel; Extraction; Supported liquid membrane; D2EHPA; Transport

### 1. Introduction

Nickel(II) which is considered as a toxic heavy metal can affect the bodyweight of organs and inhibits plant growth [1,2]. Nickel is also harmful to human health; it causes cancer, heart problems, and digestive disorders [3–5]. Otherwise, nickel has many successful applications in different fields such as metallurgy, batteries, pigment, and electroplating [6,7]. Various methods have been developed for the removal of nickel(II) such as chemical precipitation [8], solvent extraction [9], ionic exchange [10], and adsorption [11].

These techniques are today reconsidered in view of their possible substitution by processes based on different types of membranes such as; the emulsion liquid membrane (ELM), the bulk liquid membrane (BLM), and the supported liquid membrane (SLM) [12–14].

An SLM is expected to be one of the most efficient membranes for separation processes [15]. In fact, it combines the process of extraction and stripping in a single operation [16]. Furthermore, it consists of bringing into contact two aqueous solutions (source and receiving phases) separated by an organic membrane solution. These two phases are agitated separately to ensure the removal of the metal from the solution [17–19].

SLM systems present several advantages mainly: high permeability, short experiment time, low consumption of organic solvents for extraction, and energy savings [20,21].

This paper is focused on the recovery of nickel with two extraction processes: the liquid–liquid extraction (LLE) and the SLM using di-(2-ethylhexyl) phosphoric acid (D2EHPA) as a carrier. Several parameters were investigated in order to determine the optimal condition of selective extraction

\* Corresponding author.

of Ni(II) such as; the effect of pH source phase, (D2EHPA) concentration, the concentration of the receiving phase, and nickel concentration in the source solution. Once optimal parameters were determined, the membrane stability was studied.

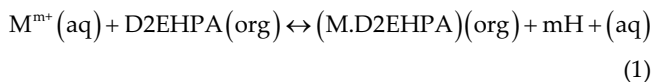
## 2. Experimental

### 2.1. Reagents

The carrier for the liquid membrane was di-(2-ethyl-hexyl) phosphoric acid (D2EHPA) which is an organophosphorus extractant (Fig. 1). The D2EHPA used in this study is a product purchased from Sigma Aldrich (USA) with purity greater than 97%. Its molar mass is equal to 322.43 g mol<sup>-1</sup>.

It has a boiling point of 155°C to 1 atm. In polar solvents, D2EHPA is in the form of a monomer, whereas in apolar solvents it is found as a dimer form according to the following structure [22].

The reaction of D2EHPA with metal ions can be described by the following equation:



The tested diluents are chloroform (CHCl<sub>3</sub>), hexane, dichloromethane, toluene, and butanol, respectively, purchased from Merck Prolabo (France), Scharlau (Spain), Carlo Erba (France), and VWR Prolabo (France). Other inorganic chemical products: HNO<sub>3</sub>, NaOH from analytical grade agents were used.

The support used for the liquid membranes is Celgard 2500 type. It is a flat sheet, polymeric, hydrophobic, and microporous polypropylene [23]. Its physical characteristics are collected in Table 1.

### 2.2. Experimental procedure

#### 2.2.1. LLE experiments

To effect separation, 2 mL of an aqueous solution containing 2.5 mM of nickel(II) adjusted at pH = 5 with a few drops of HNO<sub>3</sub> as well as 1 mL of D2EHPA and 1 mL of solvent organic, were mixed in clogged glass tubes. The mixture was then agitated at 30 rpm for 24 h. After that, the solution was centrifuged and the two phases were well separated. The nickel concentration was determined via atomic absorption spectrometry with flame.

The percentage of extraction (%E) described through Eq. (2) was calculated using the concentration of nickel ions before and after extraction:

$$\%E = \frac{[\text{Ni}^{2+}]_{\text{aq},i} - [\text{Ni}^{2+}]_{\text{aq}}}{[\text{Ni}^{2+}]_{\text{aq},i}} \quad (2)$$

The recovery of the extracted nickel in the organic phase was executed using 2 mL of an HNO<sub>3</sub> aqueous solution (0.5 M). Then, the percentage has been calculated from the concentration of recuperated and initial nickel ([Ni<sup>2+</sup>]<sub>aq,r</sub>) in the aqueous phase ([Ni<sup>2+</sup>]<sub>aq,i</sub>). It can be described by Eq. (3):

$$\%R = \frac{[\text{Ni}^{2+}]_{\text{aq},r}}{[\text{Ni}^{2+}]_{\text{aq},i}} \times 100 \quad (3)$$

The distribution coefficient of the extracted metal ion is given by the equation below:

$$D = \frac{\sum C_{\text{Ni}^{2+}}(\text{org})}{\sum C_{\text{Ni}^{2+}}(\text{aq})} \quad (4)$$

where  $\sum C_{\text{Ni}^{2+}}(\text{org})$  and  $\sum C_{\text{Ni}^{2+}}(\text{aq})$  correspond, respectively, to concentrations of the nickel ions in the organic and aqueous phases.

#### 2.2.2. Transport membrane experiments

The experiments were carried out in a two-compartment Teflon transport cell. The SLM was prepared by impregnating during 24 h in a D2EHPA – chloroform mixture. The obtained SLM was placed in a circular window (diameter of 3 cm) between two-compartments with a maximum capacity of 100 mL [25,26]. Both aqueous feed and stripping solutions were magnetically stirred at room temperature (25°C). The active surface area of the membrane was 7.10 cm<sup>2</sup> (Fig. 2). Samples were collected, from feed and receiving solutions, at regular time intervals during the 24 h of the experiment. A Jeulin pH meter (pH 211 J-SA) was used to measure the pH of the aqueous phases. An atomic absorption spectrophotometer (Analytic Jenna model NoVAA400) (AASF) was used to determine the nickel concentration.

### 2.3. Analytical method

The nickel concentration was determined by atomic absorption spectroscopy with the flame (AASF) method. In the context of this study, it was the most feasible, selective, and could be adequately adapted to a higher concentration of nickel, with detection limits consistent with the objectives of the analysis of natural water [27]. For this reason, nickel concentrations were measured using an Analytic Jenna model Novaa 400 apparatus (Germany).

Table 1  
Physical characteristics of polymeric support [24]

Characteristics	Values
Thickness, μm	25
Porosity, %	55
Pore size, μm	0.064
Puncture strength, g	>335
TD tensile strength, kgf cm <sup>-2</sup>	135

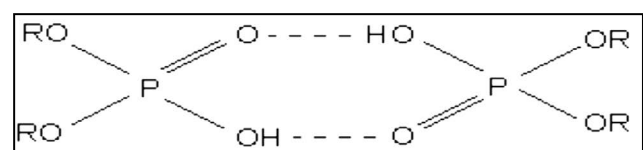


Fig. 1. Dimer structure of D2EHPA.

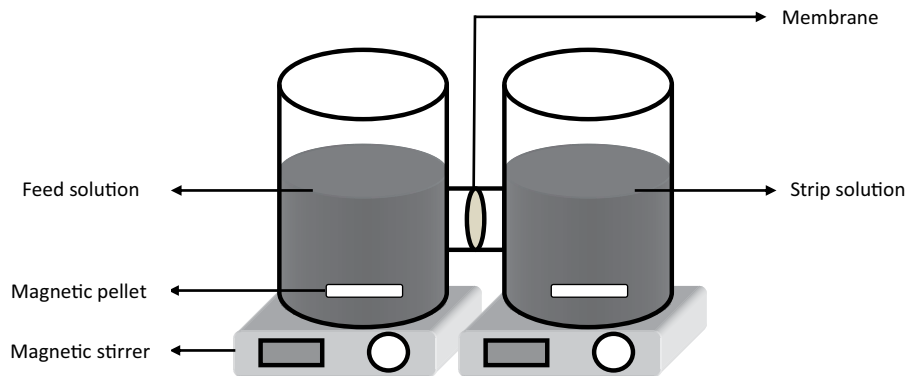


Fig. 2. Simultaneous extraction and stripping cell.

From a stock solution of nickel concentration equal to  $1,000 \text{ mg L}^{-1}$  and the standard solutions are prepared: 0, 3, 5, 7, and  $10 \text{ mg L}^{-1}$ . These solutions are preserved in plastic bottles. Then, the absorbance of these standards was measured at the wavelength  $\lambda_{\text{max}} = 232 \text{ nm}$ . The value of the correlation coefficient for the calibration curve was found higher than 0.99.

### 3. Results and discussion

#### 3.1. Liquid–liquid extraction

##### 3.1.1. Nature effect of the solvent on nickel extraction

In order to determine the optimal solvent for Ni(II) extraction by D2EHPA, various LLE tests were achieved using five organic solvents (hexane, dichloromethane, butan-2-ol, toluene, and chloroform). Recovery of nickel from the organic phase was studied using a nitric acid solution ( $0.01 \text{ mM}$ ). Extraction (%*E*) and reextraction (%*R*) efficiencies and the distribution coefficients *D* expressed as  $\log D$ , are summarized in Table 2.

As summarized in Table 2, the results have revealed that the extraction yield does not exceed 70% for tested solvents. The best extraction yield was obtained using a mixture of D2EHPA and chloroform phase. However, butan-2-ol and dichloromethane have the lowest extraction yields ( $E < 20\%$ ). Didi et al. [28] found similar results for the LLE of amino acids. Also, Azzoug et al. [29] showed that the chloroform is a better solvent for the LLE of lead(II) and cadmium(II). Table 2 depicted that the aqueous solution of  $\text{HNO}_3$  causes nickel stripping for all tested solvents except butan-2-ol. Similar results were reported by Buch [30]. By comparing the distribution coefficients of nickel, it is clear that the D2EHPA-chloroform mixture has the most important distribution coefficient  $D = 1.32$  ( $\log D = 0.28$ ).

##### 3.1.2. Kinetics of extraction

The extraction kinetics of nickel(II) was studied using D2EHPA as an extractant for various solvents. According to the experimental protocol previously described, the mixture of the two aqueous and organic phases was stirred for 24 h. The variation of the percentage of extraction as a function of time is given in Fig. 3.

Table 2

Extraction and recovery yield and the distribution coefficients expressed as  $\log D$  of nickel using a mixture of D2EHPA and various tested organic solvent

Solvent tested	% <i>E</i>	$\log D$	% <i>R</i>
Hexane	62.15	0.21	53.50
Dichloromethane	18.88	−0.63	26.25
Chloroform	65.60	0.28	45.30
Toluene	31.40	−34.00	1.78
2-butanol	8.12	−1.09	0.00

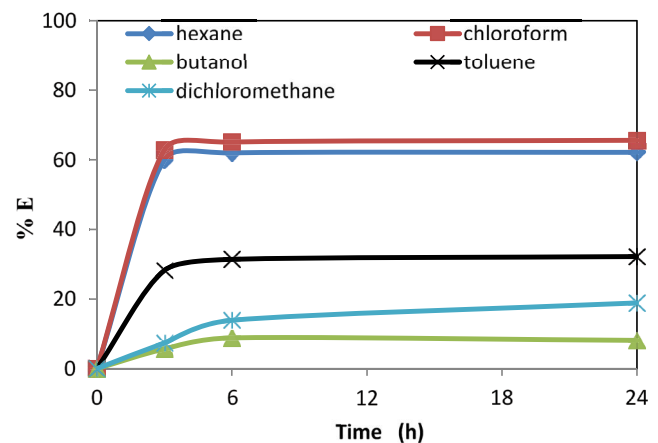


Fig. 3. Variation of the extraction yield as a function of time (0–24 h). Aqueous solution: 2 mL of nickel ( $2.5 \text{ mM}$ ) at  $\text{pH} = 5$ , organic phase: a mixture of 1 mL of D2EHPA and 1 mL of solvent.

Based on this Fig. 3, it can be noticed that the percentage of extraction shows a clear increase during the first 3 h. A contact time between the two phases greater than 3 h results in a slight improvement of the extraction efficiency. Beyond 6 h, the percentage of extraction was almost constant for all the solvents studied. In addition, it can be observed that the optimal extraction of nickel (over 60%) was obtained when chloroform was used as an organic solvent.

According to the nature of the diluents, the efficiency of nickel extraction follows the following order:

Chloroform > hexane > toluene > dichloromethane > butan-2-ol (5)

From these results, it can be concluded that chloroform offers the best efficiency when it was used as an organic solvent for the extraction of Ni(II).

The solvent in the SLM system plays a key role in the recovery of Ni(II) ions. High polarity, low viscosity, high surface tension, low volatility, and low water solubility are essential specifications for solvent in SLM system [18]. For this reason and regarding the obtained results, chloroform was chosen to study the transport of nickel through the SLM.

### 3.1.3. Influence of aqueous phase pH

The effect of pH of the aqueous phase on the extraction of Ni(II) using a mixture of D2EHPA-chloroform (v/v) was studied for a range of 4–6. The results are shown in Table 3.

According to these results, it can be noticed that the increase of pH of the aqueous phase from 4 to 5 results in an increase of extraction efficiency from 15% to over 65%. For pH equal to 6, we can observe a fall of the percentage of extraction down to 2%. This could be explained by the precipitation of nickel hydroxide  $\text{Ni}(\text{OH})_2$ . Tayeb et al. [25] and Vasilyev et al. [31] observed similar behavior for the LLE of Cr(III) and Ni(II), respectively.

## 3.2. Facilitated transport of nickel using D2EHPA carrier through the SLM system

### 3.2.1. Effect of source phase pH

In order to assess the influence of source phase pH on the transport of nickel through the SLM system, the separation of  $\text{Ni}^{2+}$  was studied in the pH range of 3–6 using D2EHPA as a carrier. Fig. 4 shows the pH effect on the percentage of nickel extraction. It can be perceived that the amount of  $\text{Ni}^{2+}$  transported increases with increasing pH up to 5. From this result, it is clear that the increase of hydrogen ions in the feed solution increases the complex formation at reaction equilibrium, which enhanced the transfer of nickel ions.

Beyond  $\text{pH} = 5$ , the amount of nickel transported decreases. This phenomenon can be explained by the

precipitation of Ni(II) ions in the form of hydroxide for a higher pH value ( $\text{pH} = 6$ ) as shown in Fig. 5 realized through MEDUSA software. The source pH was maintained at  $\text{pH} = 5$  as optimal value. Similar results were observed for the transport of nickel by Cyanex [32] and the transport of copper by D2EHPA [33].

### 3.2.2. Effect of D2EHPA concentration in the membrane phase

The effect of D2EHPA concentration in the membrane phase on the separation of nickel was studied in the range of 20%–50% (v/v). The results in Fig. 6 showed that the extraction efficiency increased when the concentration of D2EHPA in the membrane increases from 20% to 30% (v/v). The ion permeability increased with the concentrations of D2EHPA [34]. For 30% (v/v) of concentration, the extraction percentage reaches a maximum value of 58%.

Beyond 30% (v/v), the percentage of nickel transported after 24 h decreases despite the increase in the

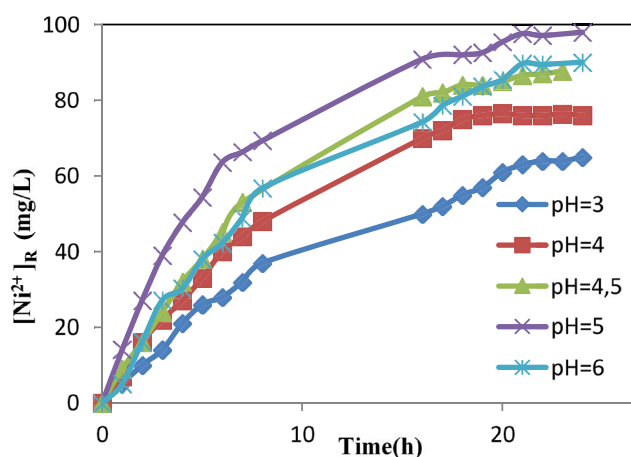


Fig. 4. Concentration of nickel transported as a function of time for different pH values. Phase source: aqueous solution of nickel (2.5 mM) with different pH values, receiving phase:  $\text{HNO}_3$  (0.01 M), and membrane phase: D2EHPA (25%) (v/v) in chloroform.

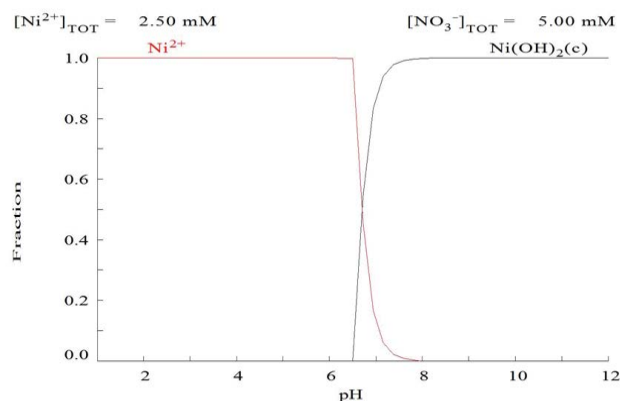


Fig. 5. Speciation diagram of nickel species in nitrate media.

Table 3  
Effect of aqueous phase pH on the extraction of nickel.

pH	%E		
	t = 3 h	t = 6 h	t = 24 h
4	15.03	15.03	15.03
5	62.80	65.10	65.60
6	0.00	0.00	2.09

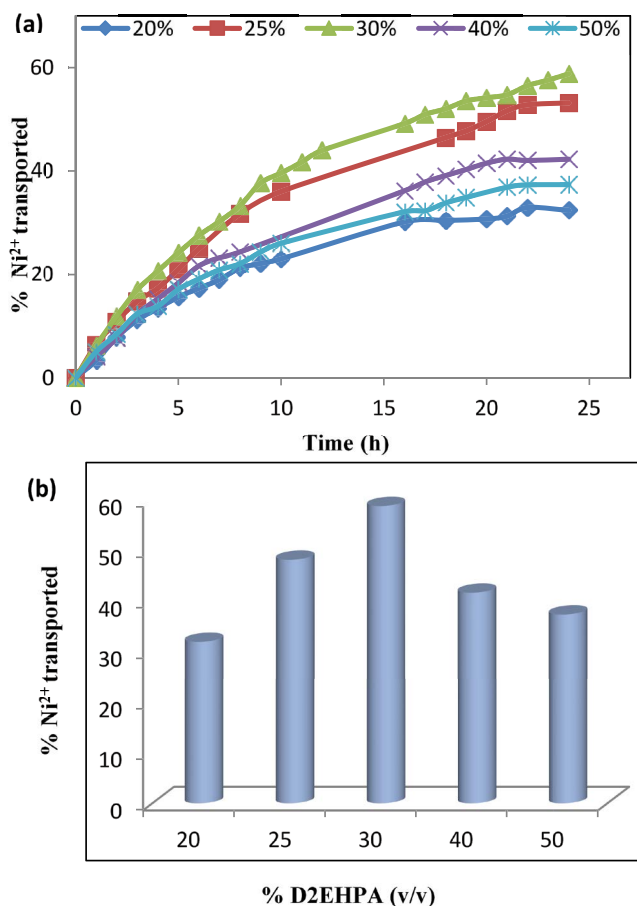


Fig. 6. Variation of an extraction efficiency of nickel as a function of (a) time and (b) D2EHPA concentration. Source phase: aqueous nickel solution (2.5 mM) at pH = 5, receiving phase: HNO<sub>3</sub> (0.01 M), and membrane phase: D2EHPA 25% (v/v) in chloroform.

concentration of D2EHPA. These results can be explained by two phenomena. When the concentration of D2EHPA increases from 20% to 30% (v/v), the concentration of the Ni-D2EHPA complex increases and then facilitates the transport of nickel.

The decrease of the amount extracted from nickel concentration can be explained by the increase of the viscosity of the organic solvent, which increases the resistance of the membrane and decreases the migration speed in the membrane.

Dingsheng et al. [35] have shown that the transport flux of Zn(II) through an SLM increases with increasing concentration of D2EHPA until an optimal value and beyond this value, the percentage of extraction decreases. Thus, 30% (v/v) of D2EHPA will be chosen as the optimal concentration and then used in our later tests.

### 3.2.3. Effect of initial nickel concentration in the source phase

The effects of nickel concentration on the behavior of the SLM stability and removal of nickel were investigated in the range of 0.25, 1.5, 2.5, 5, and 25 mM. The pH

of these solutions has been adjusted to 5. The receiving phase is HNO<sub>3</sub> solution (0.5 M). These different results are exhibited in Fig. 7.

It can be observed from these curves that the percentage of extraction decreased successively with the increase of the initial nickel concentration. For a 0.25 mM concentration, Ni(II) was totally extracted after only 3 h. On the other hand, for 25 mM concentration, there is difficulty in the transport of Ni(II) ions from the source phase to the receiving phase. This is probably due to the membrane saturation phenomenon. Similar behavior was noticed in later work on the recovery of Cu(II) and Tb(III) using D2EHPA carrier through the SLM system [36,37].

### 3.2.4. Effect of the receiving phase composition

The stripping reaction at the membrane receiving solution plays a vital role in the transfer of metal ions from source to receiving phase. We studied the effect of HNO<sub>3</sub> concentration on Ni(II) transport. Fig. 8 illustrates the variation of the Ni(II) concentration transported in the receiving phase as a function of time for different concentrations of HNO<sub>3</sub> (0.01, 0.05, 0.1, and 0.5 M).

This result is expected because the increase of H<sup>+</sup> concentration increases the speed of decomplexation at the membrane strip interface. This reduces the extraction time from more than 24 to only 12 h for a concentration of 0.5 M of HNO<sub>3</sub>. The concentration of nitric acid in the receiving phase needed was 0.5 M for effective transport of nickel(II). The obtained result is in accordance with those obtained in other previous studies [36–39]

### 3.2.5. Stability of SLM

In order to study the stability of the SLM and the long-term nickel transport efficiency, we have examined the functioning of the membrane in a continuous way

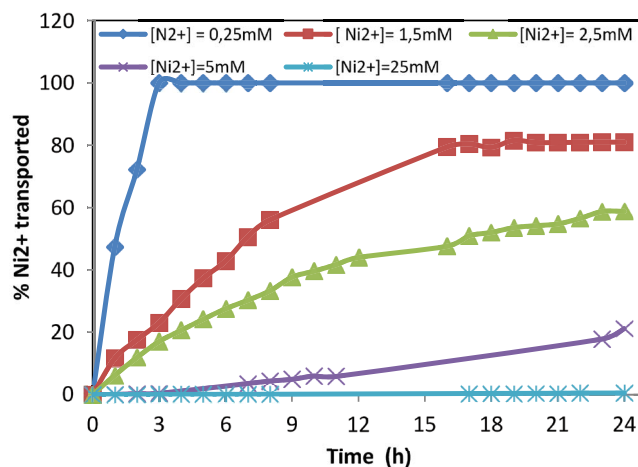


Fig. 7. Variation of the percentage of nickel transported as a function of time. Source phase: aqueous solution of nickel at different concentrations (pH = 5), receiving phase: HNO<sub>3</sub> (0.01 M), and membrane phase: D2EHPA (30%) (v/v) dissolved in chloroform.

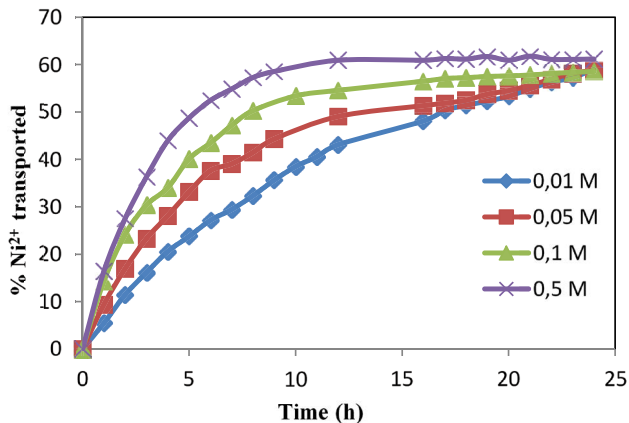


Fig. 7. Variation of the percentage of nickel transported as a function of time. Source phase: aqueous solution of nickel ( $2.5 \times 10^{-3}$  M) at pH = 5, receiving phase: aqueous solution of HNO<sub>3</sub>, membrane phase: D2EHPA 30% (v/v) dissolved in chloroform.

without re-impregnating it. At the same time, all the other experimental conditions are kept at their optimal levels. The percentage of nickel extraction was measured every 24 h for 5 d. After this period, the depleted source phase and the enriched receiving phase were replaced by fresh solutions. The obtained results are summarized in Fig. 8.

Fig. 8 shows a decrease in the percentage of extraction during the first 24 h from 68% to 52%. This decrease would result in a flux or permeability decrease of nickel and it is an indication for membrane instability. The reduced percentage of transport of the Ni(II) extracted may be due to the loss of the liquid membrane phase (carrier and/or solvent) out of the pores of the support [40]. Van de Voorde et al. [41] and Kemperman et al. [42] have corroborated this hypothesis and similar behavior was observed for the recovery of nickel ions by SLM using different types of extractants [41]. Moreover, the MLS

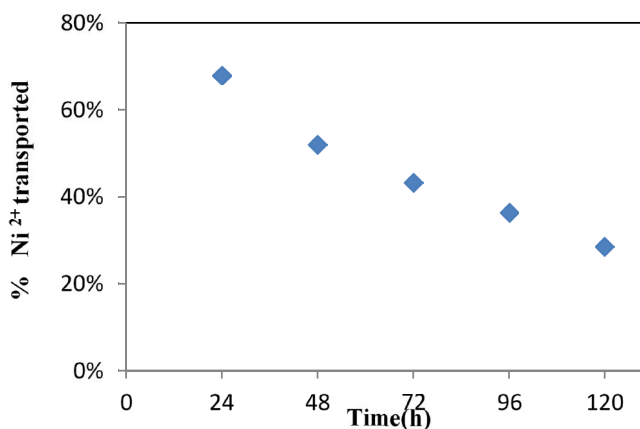


Fig. 8. Stability of the SLM system: percentage of nickel transported as a function of time. Source phase: aqueous solution of nickel ( $2.510^{-3}$  M) for pH = 5, receiving phase: HNO<sub>3</sub> (0, 5 M), and organic phase: D2EHPA 30% (v/v) dissolved in chloroform.

system using D2EHPA as a carrier shows a significant decrease of copper ions flux during the first cycles [43].

SLM systems usually consist of an organic solvent, immobilized in the pores of a hydrophobic microporous membrane. The reason for an SLM to become unstable is generally loss of liquid phase (carrier and/or solvent) out of the pores of the support [42]. Fig. 9 shows SEM images of Celgard 2500 membrane, impregnated Celgard 2500 membrane before and after nickel transport.

Fig. 9a confirms the microporous structure of the Celgard 2500 membrane [44]. Impregnation of the membrane by the liquid organic phase (solvent+ D2EHPA) resulted in a total coating of the membrane surface and a large amount of the organic phase was observed on the surface of the Celgard 2500 membrane (Fig. 9b). Therefore, the carrier seems to be to a certain extent uniformly distributed. As shown in Fig. 9c, after nickel transport by the SLM system, the carrier excess was shriveled and we noticed that the surface of the membrane contains several microparticles. Consequently, the decline of transport ratio could be attributed to the gradual loss of the membrane's liquid phase in the aqueous phase at the interface of the membrane on both sides by emulsion formation [41] and blockage of the membrane pores by precipitation of the carrier [40] and/or nickel hydroxide. Nevertheless, the latter assumption needs to be investigated.

#### 4. Conclusion

SLM process using D2EHPA to extract and separate nickel from acidic solutions was investigated. Preliminary studies of the LLE were carried out in order to determine the most efficient organic solvent for nickel extraction by the SLM system. Afterward, the effect of several parameters such as source solution pH, D2EHPA concentration, initial nickel concentration, and receiving phase concentration on nickel transport was investigated experimentally. From this study, the following conclusions can be drawn:

- The best nickel extraction yield is obtained using chloroform as an organic solvent. The D2EHPA-chloroform mixture has the most important distribution coefficient.
- Increasing pH up to 5 resulted in an enhancement of nickel extraction by SLM. Above or under this optimal pH value, the transport rate decreased.
- The optimal extraction of nickel(II) was accomplished using 30% D2EHPA (v/v) in chloroform as membrane phase.
- The use of a significant initial amount of Ni(II) causes an appearance of the phenomenon of membrane saturation. The Ni<sup>2+</sup> ions remain accumulated in the matrix of the membrane.
- At the optimum conditions, Ni(II) was totally extracted within 3 h for an initial nickel concentration of 0.25 mM.
- Finally, it can be observed that nickel recovery using SLM decreases with time during the first continuous cycle (24 h) of operation.

Despite the lack of stability of the SLM, this system provides a high-efficiency separation with a small amount



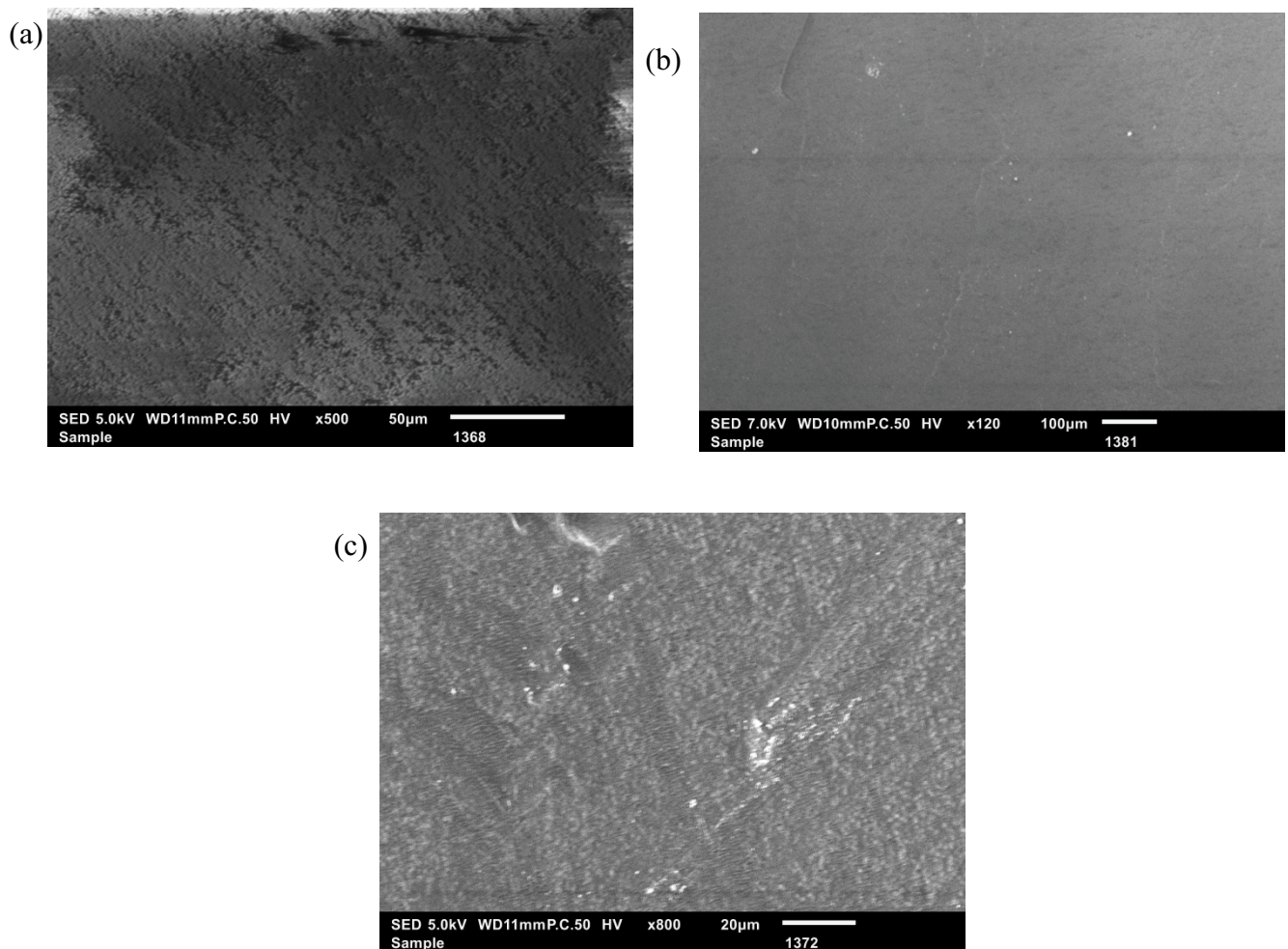


Fig. 9. SEM images of (a) Celgard 2500 membrane, (b) impregnated Celgard 2500 membrane, and (c) impregnated Celgard 2500 membrane after nickel transport.

of liquid membrane. SLM system would afford a suitable alternative to the existing conventional separation processes.

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