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D2EHPA-Sulfuric Acid System for Simultaneous Extraction and Recovery of Nickel Ions via Supported Liquid Membrane Process

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A B S T R A C T

This research addresses the extraction and recovery of nickel ions from real electroplating wastewater using supported liquid membrane (SLM) process. The process involves three main phase system which are feed, organic and stripping phase. The feed phase containing the nickel electroplating wastewater whereas the organic phase containing the liquid membrane which was immobilized in the membrane support. The liquid membrane was prepared by dissolving certain concentration of D2EHPA in kerosene which acts as a carrier and diluent, respectively. Meanwhile, the membrane support employed was commercial polypropylene membrane with features of 100 μm thickness, 71.9% porosity and 0.10 μm effective pore size. On the other hand, the stripping phase consisting of sulfuric acid (H_2SO_4) solution which acted as a stripping agent. Parameters such as carrier and stripping agent concentration and feed phase flowrate were examined to obtain the best condition for the extraction and recovery efficiency of nickel. The results revealed that about 44 and 55% of nickel ions successfully extracted and recovered, respectively at the best conditions of 1.0 M of D2EHPA, 3.0 M of H_2SO_4 and 70 ml/min flowrate of feed phase.

1. INTRODUCTION

In the recent years, nickel compounds and their alloys have been used in various industrial processes such as production of stainless steel, nonferrous alloys, nickel-based superalloys, rechargeable and non-rechargeable batteries and electroplating. This is due to the good features of nickel which are high corrosion and temperature resistance especially in galvanizing and catalyzing processes [1]. Electroplating industries consumed numerous types of chemicals to electroplate parts with one or a combination of high number metallic coatings. Consequently, a huge amount of waste in the forms of wastewater, spent solvent, spent process solutions, and sludge have been generated [2]. The primary hazardous nature of electroplating wastewater is due to the presence of heavy metals which are being harmful to aquatic fauna, livestock and human beings. In addition, they possess high toxicity, non-biodegradability and high solubility in the aquatic environment which can be rapidly absorbed by living organisms. Previously, several methods on the removal and recovery of metal ions have been reported such as ion exchange resin [3], leaching [4], biological treatment [5], electrodeposition [6], nanofiltration [7], adsorption [8], solvent extraction [9] and membrane separation [10-12]. Among them, membrane separation was regarded as one of the most effective techniques. Liquid membrane technology have been widely used for the extraction of abundant metals and organics complexes from aqueous waste solution. This technology has several benefits includes simultaneous extraction and stripping process, less consumption of chemicals and energy saving with minimum supervision [13]. Out of three, supported liquid membrane is one of the main configuration in liquid membrane technology. Theoretically, this system

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composed of three main phase which are feed, membrane and stripping phase. Two different aqueous phase which are feed and stripping phase are in contact with the membrane phase. The membrane phase is practically insoluble, hence creating a barrier between both of feed and stripping phase. In supported liquid membrane, the feed and stripping phase are separated by polymeric support containing organic liquid membrane phase [14].

The primary importance components in liquid membrane system are carrier, stripping agent and diluent. In supported liquid membrane system, the membrane support is impregnated with carrier in diluent for the extraction process. The carrier aids the transportation of metal ions from the feed-membrane interface to the membrane-stripping interface by forming complexation with the targeted solute ion. The characteristic features of a good carrier are rapid kinetic formation and decomposition of complexes in the membrane interfaces, low solubility in the feed and the stripping phase, no side reaction, and acceptable price for industrial application [15]. In fact, every specific solute needs its own specific carrier. The extractions are classified as (i) extraction by compound formation, (ii) extraction by ion-pair formation, and (iii) extraction by solvation [16]. Commonly, the carriers used for the extraction by compound formation composed of chelating and acidic carrier whereas the basic carriers involve via extraction by ion pair formation. Meanwhile, the extraction by solvation employed with the solvating or neutral carriers. In addition, an anion is needed for the complexation with the metal cation from an aqueous waste solution. Thus, an acidic carrier are highly suitable for the separation of cations by replacing their protons for the metal ion through a cation exchange mechanism. D2EHPA is one of the acidic as well as the most versatile carriers for various metal cation extraction. For instance, Talebi et al. [17] claimed that D2EHPA has been the most favored carrier for nickel extraction due to its chemical stability, low aqueous solubility and high loading characteristics, which makes D2EHPA one of the most versatile extractants. This is in agreement with Zhang et al. [18] who reported that D2EHPA provided high acidity, hence the proton in P–O–H bond of D2EHPA is easily substituted by metal cation. On the other hand, metal ion extracted by acidic carrier usually stripped using high acidic solution to create chemical potential among both membrane and stripping phase [19].

The main objective of this research is to observe the performance of D2EHPA-sulfuric acid system for simultaneous extraction and recovery of nickel ions from electroless nickel plating wastewater via supported liquid membrane process. The fundamental parameters which influencing the extraction performance of nickel ion were investigated include effect of carrier and stripping agent concentration and feed phase flow rate.

2. THEORITICAL BACKGROUND

Fundamentally, Figure 1 illustrates the mass transfer of metal ion through SLM which involving several important steps which are [20-23];

❖ Diffusion of metal ion from the bulk of the feed phase, $C_{B,f}$ to the diffusion layer of feed phase, $C_{i,f}$

❖ Complexation reaction between the metal ion and the carrier at the feed-membrane interface, $\overline{C_{i,f}}$

❖ The metal-carrier complex diffuses through the membrane phase towards strip-membrane interface, $\overline{C_{i,s}}$

❖ Upon reaching at the strip-membrane interface, the strip reaction takes place, regenerating the carrier and liberating the metal ions into the stripping phase, $C_{i,s}$

❖ The stripped metal diffuses to the bulk of the stripping solution, $C_{B,s}$

❖ The regenerated carrier diffuses through the membrane to the feed/membrane where the transport process is repeated.

The mass transfer of the metal ion passing through the membrane is described considering the diffusional parameters. The interfacial flux due to the chemical reaction is neglected as the chemical reaction seems to take place at the aqueous feed/membrane and membrane/strip interface which occur instantaneously relative to the diffusion process [22-23]. Therefore, the metal transport is determined by the rate of diffusion of the metal containing species through the feed diffusion layer and the rate of diffusion of metal-carrier complex though the membrane. Under steady state conditions, the flux of metal ion through the aqueous feed film, J_{aq} and membrane flux of metal-carrier complexes, J_{org} may be derived by applying Fick's first diffusion law [21-23]:

$$J_{aq} = K_{aq}\ (C_{Bf} - C_{if}) \tag{1}$$

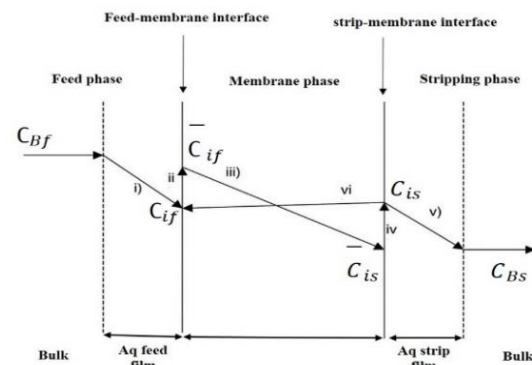$$J_{org} = K_{org}\ (C_{i,f} - C_{i,s}) \tag{2}$$

Figure 1. Concentration profile of metal ion   transportation in SLM [23]

Considering distribution coefficient of metal ion, K_d between liquid membrane and strip solution much lower than that between the feed solution and the membrane, Equation (2) can be rewritten as

$$J_{org} = K_{org}C_{i,f} \qquad (3)$$

Also the transport rate can be determined by nickel ion diffusion through feed film and diffusion of its complex through membrane neglecting the aqueous film resistance of strip side state $(J_{aq} = J_{org} = J)$, flux expression of nickel can be given as,

$$J = -\frac{V}{A}\left(\frac{dC_B}{dt}\right) = \frac{K_d C_{i,f}}{K_d K_{aq}^{-1} + K_{org}^{-1}} \qquad (4)$$

where V is volume of aqueous feed solution is, A is effective membrane area, K_{aq} and K_{org} denote mass transfer of metal ion in aqueous and organic phase, respectively. From Equation (4) permeability of metal ion, P can be obtained as,

$$P = \frac{K_d}{K_d K_{aq}^{-1} + K_{org}^{-1}} \qquad (5)$$

Rearranging Equation (5), next expression can be obtained,

$$\frac{1}{P} = K_{aq}^{-1} + \frac{K_{org}^{-1}}{K_d} \qquad (6)$$

By plotting $\frac{1}{P}$ versus $\frac{1}{K_d}$, the mass transfer coefficient value K_{aq} and K_{org} will be obtained. Diffusivity, D_{org} is related to the mass transfer coefficient by following relation,

$$K_{org} = \frac{D_{org}}{d_0 \tau} \qquad (7)$$

where d_0 and τ are thickness and turtoisity of the membrane. Alternatively, the permeability coefficient can also be obtained experimentally using the following equation:

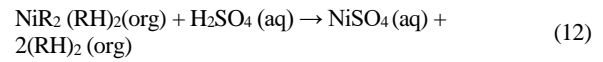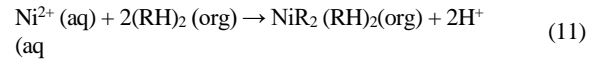$$In\frac{C_t}{C_i} = -\left(\frac{A}{V}\right)Pt \qquad (8)$$

Thus, the permeability coefficient (P) can be obtained from the slope of ln $([Ni]_t / [Ni]_o)$ as a function of time. The extraction and recovery percentage of nickel ions for a period of time were calculated by the following equation:

$$\text{Extraction, E (\%)} = \frac{C_i - C_f}{C_i} x\ 100\% \qquad (9)$$

where C_i represents initial concentration of nickel at feed phase; C_f is the final concentration of nickel at feed phase; C_s is the nickel concentration at the stripping phase; C_t is the nickel concentration at certain time; A represents effective area of membrane (cm^2); V is the volume of aqueous feed phase (cm^3), t is the time (min) and p is the permeability value (cms^{-1}).

Figure 2 demonstrates the transport mechanism of nickel ions extraction through D2EHPA-sulfuric acid system via supported liquid membrane process. There are three phase involve which are feed, membrane and stripping phase. D2EHPA in the membrane phase acts as a carrier which chemically reacts with the nickel ions at the feed-membrane interface, hence forming nickel-D2EHPA complexes as shown in Equation (11). Subsequently, these complexes permeate through the membrane phase to the membrane-stripping interface to be stripped out by sulfuric acid, thus producing nickel sulfate complexes in the stripping phase as exhibited in Equation (12). D2EHPA tends to act as a shuttle in a reversible reaction which diffusing back to the feed-membrane interface to react with another nickel ion in the feed phase .
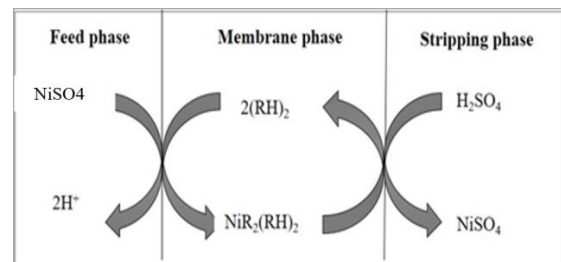At the feed-membrane interface,

$$\text{Ni}^{2+}\ (aq) + 2(RH)_2\ (org) \rightarrow NiR_2\ (RH)_2(org) + 2H^+ \ (aq \qquad (11)$$

$$NiR_2\ (RH)_2(org) + H_2SO_4\ (aq) \rightarrow NiSO_4\ (aq) + 2(RH)_2\ (org) \qquad (12)$$

3. METHODOLOGY

3. 1. Materials   Wastewater from the electroless nickel plating was obtained from Seagate, Senai, Johor. Di (2-ethylhexyl) phosphoric acid (D2EHPA) (95% purity), sulphuric acid (98% purity) and kerosene (78% purity) as a carrier, stripping agent and diluent, respectively were suplied by Merck (Darmstadt, Germany). Besides, the hydrophobic commercial polypropylene (PP) membrane which was purchased from Accurel Membrana was used as a membrane support with porosity of 71.9%, thickness of 100 μm and average pore size of 0.1 μm. All chemicals were directly used without further purification.

3. 2. Supported Liquid Membrane Process   The main components involved in supported liquid membrane rig set up include membrane cell, feed and strip vessels, double head peristaltic pump, tubing, and flow meter.

Figure 2. Supported liquid membrane mechanism for nickel extraction; $(RH)_2$ represents D2EHPA

During the process, polypropylene membrane was immobilized with the organic liquid membrane prepared by dissolving of D2EHPA in kerosene for 24 hours. The immobilized membrane was allowed to drain off for a few minutes to remove the excess amount of the organic solution from the membrane surface. Henceforth, the polypropylene membrane was put between two blocks membrane cell and clamped with the four stainless steel Teflon blocks. Meanwhile, the feed and strip vessels were filled with 300 mL of nickel wastewater and sulfuric acid solution, respectively. The membrane cell containing polypropylene membrane was set in between two aqueous phase system which were feed and stripping phase. Besides, the peristaltic pump pumped the feed and strip solution into the membrane phase with recycled operation where the flow rate of the solution was controlled by the flow meter. The samples from the feed and the stripping solutions were drawn after regular time intervals and analyzed by atomic absorption spectrophotometry.

4. RESULTS AND DISCUSSION

4. 1. Electroless Nickel Plating Wastewater Analysis
The chemical compositions of the electroless nickel plating analysed by Sulaiman and Othman. [9] was tabulated in Table 1. The main components involved in the plating bath solution were nickel sulfate, ammonium sulfate and sodium hypophosphate.

Sodium hypophosphate play a paramount role as a reducing agent during the operation. Meanwhile, nickel and hypophosphate ions were gradually decreased during the process, hence nickel sulphate and sodium hypophosphate were added up to reload back these ions. Consequently, the high concentration of several ions were identified such as sodium (Na), nickel (Ni), sulfate (SO_4), ammonium (NH_4) and phosphate (PO_4).

4. 2. Effect of D2EHPA Concentration
The effect of D2EHPA concentrations towards extraction performance of nickel is studied in the range of 0.5 to 2.0M as depicted in Figure 3(a). It is observed that the percentages of nickel extraction linearly increased from 33 to 49% upon increasing D2EHPA concentrations from 0.5 to 1.5M, respectively. However, beyond 1.5M the percentage of nickel extraction decreased to 22%. Meanwhile, the permeability values with respect to D2EHPA concentrations are tabulated in Table 2. As can be seen, the permeability values also increased with D2EHPA concentration from 8.7 x10^{-3} ms^{-1} to 14.1 x10^{-3} ms^{-1} and greatly decreased thereafter at 2.0M to 6.0 x10^{-3} ms^{-1}. The possible explanation is the low concentration of 0.5M provides inadequate D2EHPA molecules present in the membrane phase. At this condition, more nickel ions have been accumulated in the feed-membrane interface without being transported, hence causing less number of nickel-D2EHPA complexes permeating through the membrane. Otherwise the increment of D2EHPA concentration up to 1.5M seems to provide more available D2EHPA molecules for nickel ion complexation; thus,; encouraging higher permeation through the membrane phase. Surprisingly, the permeability value of 14.1 x10^{-3} ms^{-1} unchanged as enhancing D2EHPA concentration from 1.0 and 1.5M; which means there is no difference in the mass transfer of nickel permeation through the membrane phase. This can be attributed that at this condition, the high number of carrier molecules is unnecessary as they are transporting nickel ions in a reversible reaction through the membrane phase. It can be seen that 1.0M of D2EHPA is enough to permeate 400 ppm of nickel ions from the feed to the stripping phase. In contrast, further increase D2EHPA concentration up to 2.0M provide the inefficiency of nickel extraction owing to the viscosity effect. The membrane phase tend to be viscous with the existing free D2EHPA which resist the nickel ion transportation in the membrane phase. This is strongly supported by Rehman et al. [24] who indicated that the viscosity of the membrane phase increase with carrier concentration. Besides, Zidi et al. [25] also revealed that the diffusivity of complex species decreased with higher carrier concentration in the membrane phase.

Henceforth, Figure 3(b) exhibits the effect of D2EHPA concentrations towards recovery performance of nickel. Based on the result obtained, the recovery percentages of nickel ion notably increased from 43 to 56% with increasing of D2EHPA concentrations from 0.5 to 1.5M, respectively. Conversely, further increase of
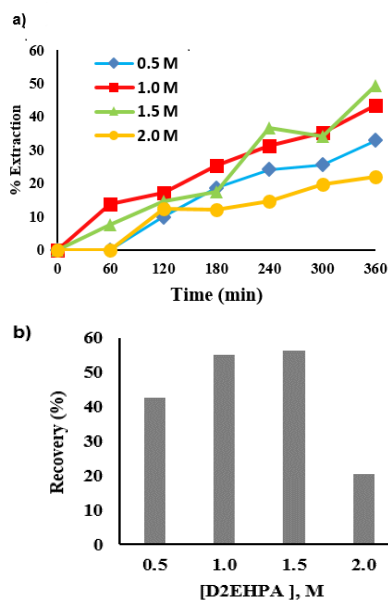
TABLE 1. Nickel electroplating waste analysis [9]

Cation	Concentration (ppm)
Na	34060
NH$_4$	17220
K	79
Ca	16
Mg	1
Ni	4156

Anion	Concentration (ppm)
F	10110
Cl	12.887
Br	204
NO$_3$	62
PO$_4$	56144
SO$_4$	30410

D2EHPA up to 2.0M, the recovery percentage decreased to 20%. In fact, the recovery performance shows the similar trend with the extraction part since the simultaneous extraction and stripping completely occurred during the SLM process. Fundamentally, the higher D2EHPA concentrations promote higher opportunity of nickel-D2EHPA complexes to be stripped by sulfuric acid at the membrane-stripping interface, hence leading to the higher efficiency of nickel recovery. This is due to the higher quantity of carrier molecules present in the membrane phase, hence forming more complexes with the nickel ions at the feed-membrane interface. On the contrary, the excess of D2EHPA concentrations only lead to the clogging of membrane phase, which inhibits the permeation of nickel-D2EHPA complexes to be stripped out by stripping agent. Thus, this condition lead to the inefficiency of recovery performance. Therefore, 1.0M D2EHPA which shows not much differences in terms of removal and recovery performance with 1.5M D2EHPA is enough to be used for the further investigation.

4. 3. Effect of H$_2$SO$_4$ Concentration Stripping agent play a significant role in SLM process to recover the nickel ion from the nickel-D2EHPA complexes at the membrane-stripping interface.

In this study, the effect of H$_2$SO$_4$ concentration towards nickel extraction is investigated by varying H$_2$SO$_4$ concentration from 1.0 to 3.5M as shown in Figure 4(a).

TABLE 2. Permeability coefficient value with respect of D2EHPA concentration

[D2EHPA], M	Permeability, 10^{-3} ms^{-1}
0.5	8.7
1.0	14.1
1.5	14.1
2.0	6.0

As can be observed, increasing H$_2$SO$_4$ concentration from 1.0 to 3.0M have led to the increasing of 37 to 44% nickel extraction, respectively. However, raising the H$_2$SO$_4$ concentration up to 3.5M have led to a slightly decline of 39%. Besides, Table 3 illustrates the permeation rate of nickel through the membrane phase which gradually increase from 12.2 x10^{-3} ms^{-1} to 14.1 x10^{-3} ms^{-1} with enhancing 1.0 to 3.0M H$_2$SO$_4$ concentration. This can be attributed to the higher acidic condition in the stripping phase tend to provide higher proton concentrations which act as a driving force to aid the transportation of nickel via counter transport mechanism. This condition leads to the high numbers of available nickel-D2EHPA complexes permeating through membrane and being stripped out to the stripping phase.

Nevertheless, the excess concentration of 3.5M H$_2$SO$_4$ has only caused the saturation of the stripping phase.
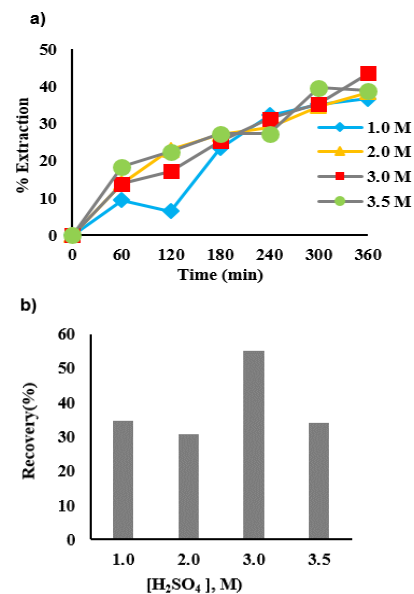

Figure 3. (a) Effect of D2EHPA concentration on the nickel extraction (Experimental condition: Feed phase=400ppm; H$_2$SO$_4$=3.0M, flow rate=70ml/min) and (b) Effect of D2EHPA concentration on the nickel recovery at 360 min (Experimental condition: Feed phase= 400 ppm, D2EHPA =1.0M; H$_2$SO$_4$=3.0M, flow rate=70ml/min

Figure 4. (a) Effect of H$_2$SO$_4$ concentration on the nickel extraction(Experimental condition: Feed phase=400ppm; D2EHPA=1.0M; flow rate=70ml/min) and (b) Effect of H$_2$SO$_4$ concentration on the nickel recovery at 360 min (Experimental condition: Feed phase= 400ppm, D2EHPA= 1.0M; flow rate=70ml/min)

TABLE 3. Permeability coefficient value with respect of H_2SO_4 concentration
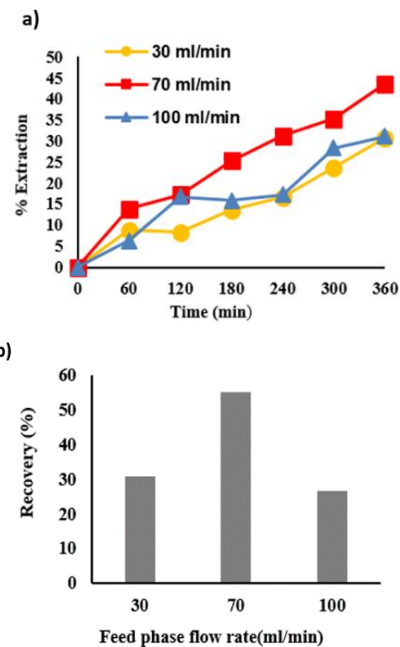
[H_2SO_4], M	Permeability, 10^{-3} ms^{-1}
1.0	12.2
2.0	13.1
3.0	14.1
3.5	13.1

Consequently, the high volume of nickel-D2EHPA complexes have accumulated in the membrane-stripping interface without being stripped. Thus, clogging the membrane phase as well as reducing the permeability value to 13.1 x10^{-3} ms^{-1}. On the other hand, Figure 4(b) exhibits the effect of stripping agent concentrations on the recovery performance of nickel. The result shows that the recovery percentage proportionally increased with the H_2SO_4 concentration from 35 to 55% at 1.0 to 3.0M, respectively. Further increase up to 3.5M, the recovery percentage seemed decreased to 34%. This can be explained by the fact that the low stripping concentration is inadequate for the stripping reaction otherwise an increment of the H_2SO_4 concentration up to certain limit retarded the stripping reaction due to the stripping reaction already reached the plateau. Hence 3.0M is enough for further experiment.

4. 4. Effect of Feed Phase Flow Rate   The effect of feed phase flow rate towards nickel extraction was examined in the ranges of 30 to 100 ml/min as depicted in Figure 5(a). Meanwhile the permeability values of different flow rate are tabulated in Table 4. It can be seen, the lowest flowrate of 30 ml/min provided the low permeability value of 7.9 x 10^{-3} ms^{-1}. Basically the complexation between nickel ions and D2EHPA in the membrane-feed interface occur very fast [22-23]. However, the low flow rate may retard the reaction, hence reducing the formation of the nickel-D2EHPA complexes permeating through membrane phase. Further increase the flow rate up to 70 mL/min, also increased the permeability value up to 14.1 x 10^{-3} ms^{-1}. At this stage, the higher flow rate on the feed side leads to a thinner aqueous boundary layer. Besides, the higher shear force and turbulence can reduce the mass transfer resistance; thus providing higher permeation rate of nickel-D2EHPA complexes. This is in agreement with Jagdale et al. [26] who revealed that the high flow rate is capable of diminishing the thickness of the boundary layer; hence facilitating the strontium ion transportation. Nevertheless, upon increasing flow rate up to 100 ml/min the extraction efficiency seemed slightly reduced to 31% as well as the permeability value which is 8.7 x 10^{-3}ms^{-1}. This can be attributed that too high flow rate extremely encouraged the quick accumulation of nickel-D2EHPA

complexes in the membrane phase, competing among each other to be stripped out into the stripping phase. This condition will resist the permeation of the transported nickel, hence affecting the extraction efficiency.

On the other hand, the recovery percentage also show similar trend with the extraction. As can be observed from Figure 5(b), the lowest flow rate of 30 ml/min provided the low efficiency of recovery and raising the flow rate up to 70 ml/min, the recovery performance increased to 55%. Thereafter beyond 70 ml/min, the recovery inefficiency is observed. Basically the reaction rate involved in the SLM process is non-equilibrium. The fast complexation occur among the nickel and D2EHPA at the feed-membrane interface is controlling rate as previously described in Section 2. Therefore 70 ml/min is the best flow rate which provide the highest simultaneous removal and recovery of nickel.

Figure 5. (a) Effect of feed phase flow rate on the nickel extraction(Experimental condition: Feedphase=400ppm; D2EHPA=1.0M; H_2SO_4=3.0M) and (b) Effect of feed phase flow rate on the nickel recovery at 360 min (Experimental condition: Feed phase= 400 ppm, D2EHPA =1.0M;H_2SO_4=3M

TABLE 4. Permeability coefficient value with respect of feed phase flow rate

Feed phase flow rate, ml/min	Permeability, 10^{-3} ms^{-1}
30	7.9
70	14.1
100	8.7

5. CONCLUSION

In conclusion, at the best conditions of 1.0 M of D2EHPA, 3.0 M of H_2SO_4 and 70 ml/min flowrate of feed phase, about 44 and 55% of nickel ions were successfully extracted and recovered, respectively. All parameters studied significantly affect the extraction and recovery performance of nickel ions from the electroplating wastewater. As for recommendations, the parameters such as types of stripping agent and carrier as well as flow rate of stripping phase should be studied to improve the performance of extraction and recovery of nickel.

6. ACKNOWLEDGEMENT

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D2EHPA-Sulfuric Acid System for Simultaneous Extraction and Recovery of Nickel Ions via Supported Liquid Membrane Process

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چکیده

این تحقیق به استخراج و بازیافت یون های نیکل از آبکاری حقیقی پساب با استفاده از فرایند غشاء مایع تقویت شده (SLM) می پردازد. این فرایند شامل سه مرحله اصلی سیستم است که مراحل خوراک، ارگانیک و خارج سازی می باشند. مرحله خوراک عبارتند از آبکاری نیکل پساب درحالیکه مرحله ارگانیک عبارتند از غشاء مایع که در غشاء کمکی تثبیت شد. غشاء مایع با حل کردن غلظت مشخصی از D2EHPA در کروسن آماده شد که به ترتیب به عنوان حامل و رقیق کننده عمل می کنند. در این میان، غشاء کمکی استفاده شده، غشاء پلی پروپیلن تجاری با مشخصات ضخامت ۱۰۰ μm، تخلخل ۷۱/۹٪ و اندازه منافذ مؤثر ۱۰/۰ μm بود. از طرف دیگر، مرحله خارج سازی شامل محلول اسید سولفوریک (H₂SO₄) بود که به عنوان ماده خارج کننده عمل کرد. شاخص هایی مانند غلظت ماده خارج کننده و حامل و شدت جریان مرحله خوراک، به منظور به دست آوردن بهترین شرایط استخراج و بازده بازیافت نیکل سنجیده شدند. نتایج نشان دادند که در بهترین شرایط ۱/۰ M از D2EHPA، ۳/۰ M از H₂SO₄ و شدت جریان ۷۰ ml/min در مرحله خوراک، به ترتیب حدود ٤٤ و ٥٥٪ از یون های نیکل با موفقیت استخراج و بازیافت شده اند.

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