



Facilitated Liquid-Liquid Extraction and Stripping of Nickel and Cadmium from Aqueous Solutions by Ionic Liquid

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ABSTRACT

Application of an ionic liquid (4-Methyl-2-pentanone) carrier on liquid-liquid extraction of nickel and cadmium from aqueous solutions is reported. 4-Methyl-2-pentanone was found to dissolve in octanol (as diluent) and was capable of extracting both nickel and cadmium ions, however in terms of extraction efficiency, 4-Methyl-2-pentanone has higher capability for nickel extraction in compare to cadmium. The effects of feed phase pH, carrier concentration, mixing time, stripping phase pH and inert salt concentration on extraction of nickel and cadmium were analyzed using factorial design for screening experiment and face centered composite design of experiments for optimization. The results of liquid-liquid extraction process led to removal of 91% of nickel ions and 32% of cadmium from mixed Ni-Cd aqueous feed phase with carrier concentration of 250 mM, feed phase pH of 4, mixing time of 5 minutes and 1.5 M of sodium sulphate as inert salt. In stripping phase, both nickel and cadmium were highly stripped while stripping phase pH was set at alkaline range of 10-11. The effect of feed phase pH on distribution ratio (Feed/Org) for both metals was investigated and the results revealed that one nickel ion is extracted into the organic phase by an average 3.2 of 4-Methyl-2-pentanone molecules to form nickel-4-Methyl-2-pentanone complex, while for cadmium extraction, the carrier molecules involved in metal-carrier complexation is equal to 2.

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INTRODUCTION

Water pollution is one of the most serious and important issues in water quality. Industrial wastewater treatment has been studied for many years and by many researchers [1-10]. Due to high consumption rate in modern life and the growing demand on the environment natural sources, heavy metals are not only refined from mineral sources but also can be recovered and pre-concentrated from industrial wastewaters as secondary sources. The recovery of heavy metals from waste solutions or pre-concentration techniques at low levels has a prominent value in economy and environment. It is important to introduce and substitute some conventional heavy metals removal methods with new techniques and approaches with higher efficiency and at the same time more environmental friendly aspects. For the recovery or refining of metal ions,

green solvent extraction is a suitable method with advantages such as high selectivity, sludge-free, precious metal recovery potential and low energy consumption. Industrial waste waters are considered as a potential source of heavy metal pollution in the environment which may accumulate the toxicity level and cause serious environmental problems [11]. There are various techniques and methods in order to remove heavy metals from industrial wastewater such as chemical precipitation, ion exchange, adsorption separation, liquid membranes, solvent extraction, etc. [12-16]

Amongst these techniques and methods, solvent extraction is a promising method in wastewater treatment. The term solvent extraction refers to the distribution of a solute between two immiscible liquids in contact with each other. For this process, International Union of Pure and Applied Chemistry (IUPAC) recommended the term liquid-liquid extraction (LLE) [17]. Solvent extraction is the basic step for

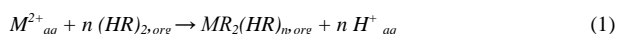
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liquid membrane (LM) process in which two aqueous phases named as feed and stripping phases are separated by an immiscible organic phase. LM has advantages such as low operation costs, high efficiency and potential usage of environmental friendly materials as diluent or carrier. Based on the mechanism of solute transport from the feed phase through the liquid membrane into the stripping phase, LM technique can be divided into four major types:

- Simple transport: in this case the solute solubility is the main reason for solute to move through the organic phase [18, 19].
- Facilitated transport in which a carrier mediated organic phase accelerates the solute diffusion through the liquid membrane [20, 21].
- Coupled counter transport (co-transport) is a specific type of *facilitated transport* in which feed and stripping phases acidity play the major role in solute-carrier complexation, decomplexation and couple transport of protons [22, 23].
- Active transport which is based on oxidation-reduction and biochemical conversions in feed-membrane and membrane-strip phase interfaces [24, 25]. The most common and conventional solute transport mechanism in LM is the facilitated type and many researchers have investigated the effect of various carriers, diluents, solutes and LM modules to find the best and most effective solvent extraction condition. Recently, application of ionic liquids as carrier in facilitated removal and recovery of heavy metals has attracted attention of many researchers. Ionic liquids (IL) have shown specific and individual physical and chemical characteristics which have made them interesting and useful materials for investigation and research. For instance high thermal stability, negligible volatility, thermal stability and non-flammability of IL have convinced the researchers to accept ionic liquids as a new green solvent, but the main advantage of these media over organic solvents which makes them a very suitable and attractive material in solvent extraction studies is their near-zero vapor pressure which increases the liquid membrane organic phase stability drastically [26-31].

Metal extraction process in a facilitated liquid-liquid extraction system follows the basic rule of metal-carrier complexation at feed-organic interface and the formed complex decomplexation at the organic-stripping phase interface. The main complexation reaction between metal ion and carrier is



where M^{2+} represents metal ion, HR represents carrier in organic phase, and n is the number of carrier molecules (either dimeric or monomeric) engaged in the reaction [32].

In this work, nickel and cadmium extraction and stripping from aqueous solution were studied using 4-Methyl-2-pentanone as carrier and 1-octanol as diluent in the organic phase. 4-Methyl-2-pentanone ($C_6H_{12}O$), abbreviated as 4M2P, with high stability against acidic and basic aqueous solutions and very low solubility in water (1.91 g/100 mL) is considered a suitable green carrier (extractant) for metals extraction [33]. 1-octanol is immiscible with water and 4M2P is soluble in it which makes 1-octanol an appropriate diluent for the current study.

MATERIALS AND METHODS

Materials

4-Methyl-2-pentanone (99%, Alfa Alesar) and 1-octanol (Sigma Adrich) were used as carrier and diluent, respectively. Cadmium sulfate ($3CdSO_4 \cdot 6H_2O$, System), nickel sulfate ($NiSO_4 \cdot 6H_2O$, Merck), hydrochloric acid (HCl) (98% purity) and sodium hydroxide (NaOH) (99% purity, Merck) were used as received. Na_2SO_4 (Analyticals, $\geq 99\%$ purity) was used as inert salt to minimize aqueous-organic phase solubility. Addition of an excess inert salt to the aqueous solutions provides a 'salting-out' effect which decreases the solubility of nonelectrolytes such as organic compounds and, thereby, improves the phase separation in aqueous-organic systems [34]. Sodium acetate ($C_2H_3NaO_2$, R&M) and acetic acid (CH_3COOH , System) were used in the feed phase buffer solution preparation to prevent changes in the desired pH. An orbital shaker (WiseShake, SHO-20) was used to shake the aqueous and organic phase mixture while a pH meter (HACH) was used for pH measurement of aqueous phase before and after extraction. The concentrations of Ni and Cd in the aqueous phase before and after extraction and after stripping were measured with a flame atomic absorption spectrophotometer (FAAS) (Perkin Elmer AANALYST 100) at a wavelength of 232 nm.

Extraction and stripping by LLE

In LLE experiments the aqueous feed phase was prepared by dissolving specific amount of $NiSO_4$ and $CdSO_4$ in distilled water. The organic phase was prepared by dissolving desired amount of 4M2P in octanol. Several factors (mixing time, carrier concentration, feed phase pH, stripping phase pH) were examined to determine the highest nickel and cadmium ion extraction and stripping. For extraction, initially a volume (10-50 mL) of the prepared organic phase containing octanol loaded with 4M2P was mixed with the prepared aqueous phase at a specific organic to aqueous phase ratio in a conical flask. The flasks were shaken by an orbital shaker. Samples were taken every 3

minutes from the aqueous phase to determine the minimum mixing time that the system required to reach equilibrium. The aqueous phase pH was measured before and after each run with a pH meter. After determination of the equilibrium time, the mixture was transferred to a separating funnel and allowed to settle for 10 minutes. The organic phase was then separated for stripping experiments. In the stripping process, aqueous solutions with different pH were prepared and mixed with the loaded organic phase with highest nickel and cadmium concentrations from the extraction process. Samples were taken by syringe from 2 cm below the surface of the aqueous phase for metal concentration measurement using atomic absorption spectrophotometer (AAS).

The removal efficiency (E%) of metal ion was calculated according to:

$$E\% = ([M^{2+}]_{org, eq} / [M^{2+}]_{aq, ini}) \times 100 \quad (2)$$

where $[M^{2+}]$ represents the concentration of metal ion and the subscripts *org*, *eq*, *aq* and *ini* denote organic, equilibrium, aqueous and initial states, respectively.

The ratio of metal concentration in the organic phase to that in the aqueous phase attained at equilibrium, known as distribution ratio (*D*), is expressed as

$$D = [M^{2+}]_{org, eq} / [M^{2+}]_{aq, eq} = ([M^{2+}]_{aq, ini} - [M^{2+}]_{aq, eq}) / [M^{2+}]_{aq, eq} \quad (3)$$

Table 1 gives the range of each factor in the stage of preliminary studies and the results were taken for the optimization process using RSM.

TABLE 1. Selected factors used in LLE and the related ranges.

Factor	Range
Feed phase pH	1-6
Mixing time	5-20 minutes
[4M2P]	0.05-0.2 M
[Na ₂ SO ₄]	0.15-0.3 M
[NaOH]	0.5-2 M

Design of experiments

Factorial design is a statistical method to find the relationship between studied parameters (factors), each at two levels (low and high) or more. Prior to optimization, preliminary studies and screening experiment provide necessary information needed for optimization process. This information in fact limits the range of studied factors and leads the design of experiments towards the best operational condition performance [35].

Response surface methodology (RSM) is a combination of mathematical and statistical methods which is considered to be a useful tool for modelling and analysis of data [36]. In RSM a response "y" (such as removal efficiency) is considered to be influenced by different independent variables " x_i " (such as pH, carrier

concentration, etc.). The purpose of using RSM is to find the best levels of each independent variable and interaction between variables that optimize the response [37].

The response is a function of the levels of independent variables:

$$y = f(x_1, x_2, \dots, x_k) \quad (4)$$

Experimental data was analyzed to fit the following second-order model:

$$y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i < j} \beta_{ij} x_i x_j \quad (5)$$

where β_0 , β_i , β_{ii} and β_{ij} are regression coefficients, and x_i and x_j are the coded variables.

In the present study, RSM was used to determine the optimum and experimental design matrix specified according to the face centered composite design (FCCD) method. FCCD method includes axial points placed on the face centers of a cube and each factor has three levels [38]. FCCD allows the desired response to be predicted by a second order equation which gives estimation of the main, quadratic and interactive effects of the factors on the factors under investigation [39].

The significance of each test and regression model has been analyzed using analysis of the variation (ANOVA). The regression model is said to be statistically significant when the P-value is lower than 0.05, and that of the lack-of-fit is higher than the significance level employed in the analysis [35].

RESULT AND DISCUSSION

Screening experiment

2⁴ factorial design of experiments for LLE including: Feed phase pH, mixing time, [4M2P], [Na₂SO₄], coded as *A_E*, *B_E*, *C_E* and *D_E*, respectively, were carried out in order to screen the best studied range for nickel and cadmium ions extraction. The subscript *E* represents the term extraction. It was found that for both nickel and cadmium extraction, feed phase pH and carrier concentration were significant, along with the interaction between feed phase pH and carrier concentration. Although inert salt concentration (Na₂SO₄) had no significant effect on the metal extraction, it should be mentioned that for aqueous electrolytes, the activity coefficients vary with the ionic strength of the solutions and this ionic strength can be kept constant by addition of an inert salt [20, 40].

Figure 1 shows the normal plot of the factors in their related ranges. It indicates that amongst the four studied factors of LLE, inert salt concentration ([Na₂SO₄]) and mixing time had no significant effect on nickel and cadmium extraction.

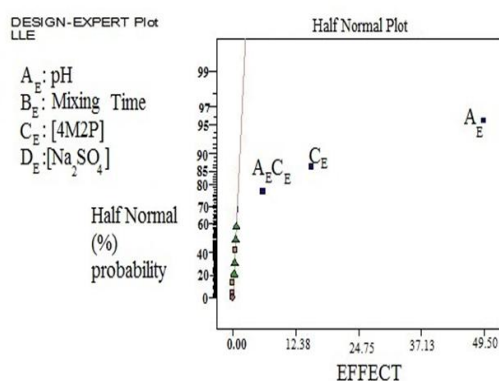


Figure 1. Normal plot of the design for significant factors

Hence, mixing time and inert salt concentration were kept fixed at their minimum level (5 minutes and 150 mM, respectively) and variables pH and carrier concentration were chosen for optimization process experiment.

Optimization of Cadmium extraction and stripping

The results of ANOVA for the optimized quadratic model of cadmium extraction and stripping are presented in Tables 2 and 3; which summarize the analysis of each response and show the significant model terms.

Table 2 shows the ANOVA for cadmium extraction as the response of quadratic model, including feed phase pH (A_E), [4M2P] (C_E) and interaction between pH and carrier concentration (A_EC_E). Linear effect of feed phase pH and carrier concentration and quadratic effect of carrier concentration with p-values of less than 0.05 have significant effect on cadmium extraction while quadratic effect of feed phase pH had insignificant effect on the response. R^2 value of 0.973 indicates that 97.3% of the response variation (cadmium extraction) can be explained by the combination of settings for the selected factors.

Based on the results of ANOVA, the optimum regression equation for cadmium extraction by LLE is

$$Cd\ extraction(\%) = +19.07 - 5.83A_E + 1.67C_E + 0.26A_E^2 + 2.76C_E^2 - 3.00A_EC_E \quad (6)$$

Factors with positive coefficient at high levels and those with negative coefficient at low levels maximize cadmium extraction. Figure 2 shows the effect of feed phase pH and carrier concentration on cadmium extraction. For cadmium stripping, based on the preliminary results, stripping phase pH (A_S) and mixing time (B_S) were chosen as the significant factors affecting the cadmium stripping process.

Table 3 shows the ANOVA of cadmium stripping optimization process. It is obvious that both linear and quadratic effects of selected factors (stripping phase pH

and mixing time) have significant effect on cadmium stripping process.

TABLE 2. ANOVA for selected factorial model analysis of variance for optimization of cadmium extraction

Source	Sum of squares	DF	Mean square	F-value	P-value
Model	283.39	5	56.68	50.61	< 0.0001
A_E	204.17	1	204.17	182.31	< 0.0001
C_E	16.67	1	16.67	14.88	0.0068
A_E^2	0.18	1	0.18	0.16	0.6968
C_E^2	21.02	1	21.02	18.77	0.0034
A_EC_E	36.00	1	36.00	32.15	0.0008
Lack of fit	1.84	3	0.61	0.41	0.7559
Residual	7.84	7	1.12		
Pure error	6.00	4	1.50		
Total	291.23	12			
R-Squared	0.9731				

High R^2 value indicates that the model is well fitted for the design and 99.84% of the response (cadmium stripping) can be explained by the combination of stripping phase pH and mixing time.

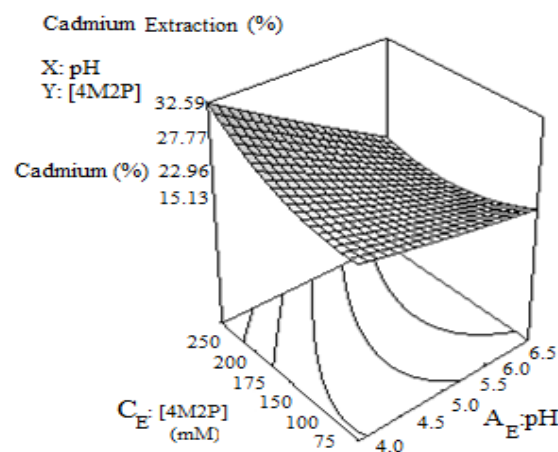


Figure 2. Three-dimensional response surface for cadmium extraction as a function of pH and [4M2P]

Figure 3 shows a three dimensional schematic of cadmium stripping as a response of aqueous solution pH and mixing time.

TABLE 3. ANOVA for optimization of selected factorial model for cadmium stripping

Source	Sum of squares	DF	Mean square	F-value	P-value
Model	7224.78	5	1444.96	877.35	<0.0001
AS	3266.67	1	3266.67	1983.45	<0.0001
BS	130.67	1	130.67	79.34	<0.0001
A2S	3070.90	1	3070.90	1864.58	<0.0001
B2S	15.19	1	15.19	9.22	0.0189
ASBS	25.00	1	25.00	15.81	0.0059
Residual	11.53	7	1.65		
Total	7236.31	12			
R-Squared	0.9984				

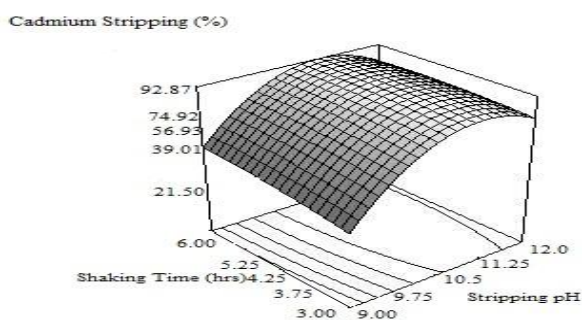


Figure 3. Three-dimensional response surface for cadmium stripping from the organic phase into the stripping phase

The optimum regression equation for cadmium stripping by LLE is:

$$Cd \text{ stripping } (\%) = +87.24 + 23.33A_5 + 4.67B_5 - 33.34A_5^2 - 2.34B_5^2 - 2.50A_5B_5 \quad (7)$$

Optimization of Nickel extraction and stripping

Nickel extraction and stripping processes were optimized based on preliminary and screening results. For extraction, it was found that linear effect of feed phase pH (A'_E), linear effect of the carrier concentration (C'_E) and quadratic effect of carrier concentration (C'^2_E) have significant effect on nickel extraction, while unlike the cadmium extraction, the interaction between pH and carrier concentration had no significant effect on nickel extraction. Table 4 presents the results of ANOVA for nickel extraction optimization.

The optimum regression equation for nickel extraction by LLE is:

$$Ni \text{ extraction } (\%) = +63.48 - 20.17A'_E + 2.17C'_E - 0.19A'^2_E + 4.81C'^2_E - 1.00A'_E C'_E \quad (8)$$

Figure 4 shows how nickel extraction efficiency varied as a function of pH and carrier concentration. It shows that 91.44% of nickel extraction was achieved when aqueous feed phase was set 4.0 with the carrier concentration of 250 mM.

Extraction optimization process of nickel was followed by stripping optimization process. In this process, stripping phase pH (A'_S) and mixing time (B'_S), both in linear and quadratic form, showed significant effect on nickel ion stripping from the organic phase into the aqueous stripping phase (Table 5).

TABLE 4. ANOVA for selected factorial model analysis of variance for optimization of nickel extraction

Source	Sum of squares	DF	Mean square	F-value	P-value
Model	2544.96	5	508.99	221.10	< 0.0001
A'E	2440.17	1	2440.17	1059.96	< 0.0001
C'E	28.17	1	28.17	12.24	0.0100
A'2E	0.099	1	0.099	0.043	0.8414
C'2E	63.91	1	63.91	27.76	0.0012
A'E C'E	4.00	1	4.00	1.74	0.2289
Lack of fit	10.11	3	3.37	2.25	0.2250
Residual	16.11	7	2.30		
Pure error	6.00	4	1.50		
Total	2561.08	12			
R-Squared	0.9937				

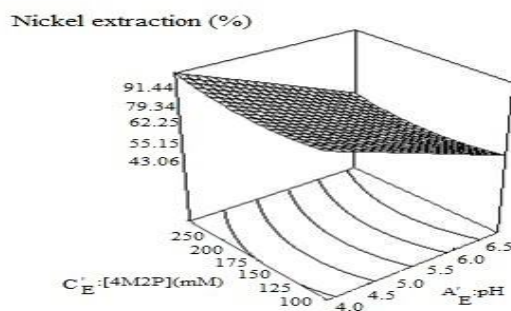


Figure 4. Response surface for nickel extraction as a function of aqueous feed phase pH and the carrier concentration

TABLE 5. ANOVA for optimization of response surface quadratic model for nickel ions stripping

Source	Sum of squares	DF	Mean square	F-value	P-value
Model	1351.27	5	270.25	427.49	< 0.0001
A'S	560.67	1	560.67	886.87	< 0.0001
B'S	32.67	1	32.67	52.67	< 0.0002
A'2S	704.00	1	704.00	1113.61	< 0.0001
B'2S	11.43	1	11.43	18.08	0.0038
A'SB'S	1.00	1	1.00	1.58	0.2488
Residual	4.43	7	0.63		
Total	1355.59	12			
R-Squared	0.9967				

Figure 5 shows the three dimensional response surface for nickel stripping. It demonstrates that with pH value of 10.5 in the stripping phase, after 7.5 hours, almost 93% of the extracted nickel ions were stripped from the organic phase into the aqueous stripping phase.

The optimum regression equation for nickel stripping is

$$Ni \text{ stripping } (\%) = +87.28 - 9.6A'_S + 2.33B'_S - 5.97A'^2_S + 2.03B'^2_S + 0.50A'_S B'_S \quad (9)$$

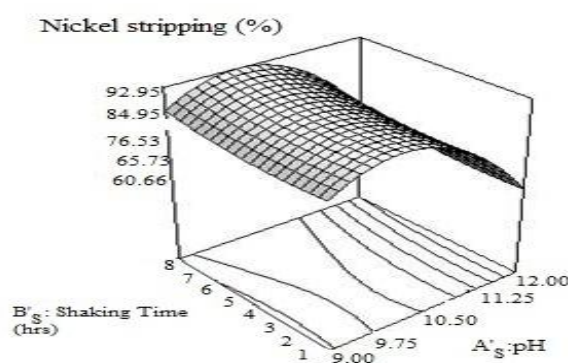


Figure 5. Response surface for nickel stripping as a function of stripping phase pH and mixing time.

Effect of pH on distribution ratio

After optimum condition determination, the effect of feed phase pH on distribution ratio was plotted in terms of log D versus pH. Figure 6 demonstrates the log D

versus pH for cadmium and nickel extraction under optimum condition.

The slope value of 2.07 suggests that two molecules of 4M2P is able to extract one molecule of cadmium during extraction process, while for nickel extraction, the obtained slope value of 1.5 suggests that the average ratio $[Ni^{2+}]/[4M2P]$ in the metal complex is 2:3.

The observed difference between log D versus pH plot for nickel and cadmium confirms the obtained results in the extraction optimization phase, in which the effect of pH and carrier concentration as an interaction variable for cadmium extraction showed itself as a significant parameter while for nickel extraction the interaction between pH and carrier concentration was insignificant. The obtained results will be useful for further studies on the application of 4M2P on nickel extraction using different types of liquid membrane such as emulsion liquid membrane, bulk liquid membrane or supported liquid membrane.

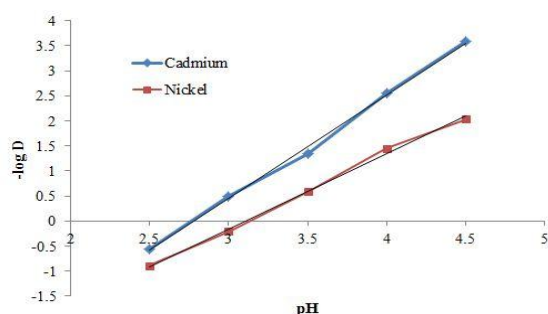


Figure 6. Effect of pH on distribution ratio of cadmium and nickel extraction; [4M2P]:250 mM; initial metal concentration: 100 mg/L

CONCLUSION

Liquid-liquid extraction and stripping of nickel and cadmium ions from aqueous solutions using 4-Methyl-2-pentanone (4M2P) as carrier and octanol as diluent were investigated, performed and optimized. For cadmium extraction linear and quadratic effects of pH and carrier concentration along with interaction between pH and carrier concentration had significant effect on metal extraction. The results of extraction optimization revealed that 4M2P can be considered an effective carrier for nickel extraction but it might not be the best extractant for cadmium extraction. The optimum condition for cadmium extraction process led to 32.59% of cadmium removal with pH value of 4 and [4M2P] of 250 mM while the highest nickel extraction achieved at this optimum condition was 91.44% with the same conditions of aqueous feed phase pH and carrier concentration. For nickel extraction, linear effect of feed phase pH, linear effect of the carrier concentration and quadratic effect of carrier concentration have significant effect on extraction. For stripping process, both nickel and cadmium showed high stripping capacity in case of

alkaline pH of the stripping phase, with 93% of cadmium stripping at pH value of 11 and 91% of nickel stripping at pH value of 10. The results of distribution ration against aqueous feed phase acidity (log D versus pH) for both metals extraction revealed that two molecules of 4M2P was able to extract one molecule of cadmium during extraction process, while for nickel extraction, each nickel molecule was extracted by 3/2 of 4M2P molecules.

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Persian Abstract

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چکیده

در این گزارش، استفاده از یک مایع یونی (۴-متیل-۲-پنتانن) به عنوان حامل در استخراج مایع-مایع نیکل و کادمیوم از محلول های آبی گزارش شده است. ۴-متیل-۲-پنتانن قادر به حل شدن در اکتانل (به عنوان رقیق کننده) و همچنین قادر به استخراج یون های نیکل و کادمیوم است. با این حال از نظر میزان قابلیت استخراج، ۴-متیل-۲-پنتانن دارای توانایی بیشتری برای استخراج نیکل در مقایسه با کادمیوم میباشد. اثر pH در فاز ورودی (دهنده)، غلظت حامل، مدت زمان تکانش، pH فاز خروجی (گیرنده) و غلظت نمک خنثی به عنوان عوامل تاثیر گذر در فرایند استخراج با کمک طرح آزمایشات عاملی مورد بررسی و بهینه سازی قرار گرفت. نتایج فرآیند استخراج مایع-مایع منجر به حذف ۹۱٪ از یون های نیکل و ۳۲٪ از کادمیوم از مخلوط نیکل-کادمیم فاز آبی دهنده با غلظت حامل ۲۵۰ میلی مولار، pH ۴ در فاز دهنده، زمان تکانش ۵ دقیقه و ۱/۵ مولار سولفات سدیم به عنوان نمک خنثی شد. در مرحله جداسازی، هم نیکل و هم کادمیم با سطحی بالا قابل به جدا شدن از فاز آبی بودند در حالی که pH فاز خروجی در محدوده قلبایی ۱۱-۱۰ تعیین شده است. اثر pH فاز "دهنده" بر ضریب توزیع برای هر دو فلز مورد بررسی قرار گرفت و نتایج نشان داد که یک یون نیکل توسط به طور میانگین ۳/۲ مولکول های ۴-متیل-۲-پنتانن استخراج می شود تا مخلوط نیکل-۴-متیل-۲-پنتانن را تشکیل دهد، در حالی که این رقم برای استخراج کادمیوم، مولکول های حامل درگیر در کمپلکس های حامل فلز برابر با ۲ است.