Optimizing Emulsion Liquid Membrane Process for Extraction of Nickel from Wastewater Using Taguchi Method

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***Abstract*--The main objectives of this research were focused on extracting nickel ions from waste water using emulsion liquid membrane as well as determining the optimal conditions for the extraction process. Taguchi experimental design method was applied to determine the optimum extraction conditions. The controllable factors of the emulsion liquid membrane process were carrier; surfactant; and internal phase concentration, treating ratio, stirring time, and feed phase acidity were optimized. The contribution of each controllable factor was also explored. The results indicated the greatest effect of the carrier concentration in comparison to other parameters. The five other parameters slightly affected the extraction percentage of nickel. The optimum conditions for the extraction was found to be carrier concentration (M) of 0.25, surfactant concentration (v %) of 10, internal phase concentration (M) of 0.1, external / emulsion ratio (v/v) of 5, stirring time (min.) of 1, and feed phase pH of 0.5**

***Key words*: nickel, emulsion liquid membrane, TOPO, Taguchi.**

I. INTRODUCTION

The growth in the use of portable equipment such as cellular phones, notebook computers, video cameras, etc. had greatly stimulated the production and consumption of portable batteries. The generation of metal-containing hazardous waste from spent batteries was an environmental concern, and their recycling would reduce the amount of waste sent to landfill and improve the utilization of the world's metal resources [1].

So extraction techniques for the removal and recovery of heavy metals were of increasing interest. According tothe World Health Organization, the most toxic metals were aluminum, chromium, iron, cobalt, nickel, copper, zinc, cadmium, mercury, and lead. A limitation in traditional solvent extraction was that a large inventory of solvent was required, especially when processing dilute solutions. Another limitation in traditional solvent extraction was also high capital cost. The use of liquid membranes containing specific metal ion carriers offered an alternative method to the solvent extraction processes for selective separation and concentration of the metal ions from aqueous dilute solutions [2].From different types of liquid membranes available in literature, Emulsion Liquid Membrane (ELM) had the advantage of having higher surface areas, fast extraction/stripping process and high efficiency. ELM had been fast extraction/stripping process and high efficiency.

ELM had been recognized as an effective method for ion separation and concentration, when the component to be extracted was present in very low concentration. Choosing a suitable extractant or carrier, between other operational conditions was one of the key factors that control the ion extraction efficiency [3].Some literatures reported more about the nickel separationfrom Co-Ni using TOPO and TOA as a carrier [4, 5], Ni-MH batteries [6-9], and nickel/cadmium batteries [10].

Recently, great attention was observed for applying Taguchi method as an experimental design method [11-14]. Applying Taguchi method for systematic design of experiments led to determination of the optimum quantity of design parameters and produces stability and reproducibility rarely found in most statistical methods [15].The aim of this research was devoted to apply theTaguchi design method for the first time on the extraction of nickel from aqueous sulfate solutions by emulsion liquid membrane.

II. METHODOLOGY

Taguchi experimental design method allowed several effects of factorsto be simultaneously determined effectively. Taguchi's design of experiments was used to get informationsuch as main effects and interaction effects of design parametersfrom minimum number of experiments [16–19].Keeping the experimental cost at a minimum level [20], reducingthe required time for experimental investigation and studying the influenceof individual factors to determine which had the most effective,in comparisonwith traditional optimization techniques [18,19]. The term “signal to noise” representedthe relation of desirable values to undesirable values for the outputcharacterization. In other words, the S/N ratio was used to verify thedeviation of the characteristics from the desired values [21]. The S/Nratios were calculated from the following Equation:

$$S/N = -10\log\left(\frac{1/Y_1^2}{n} + \frac{1/Y_2^2}{n} + \ldots\ldots\ldots + \frac{1/Y_i^2}{n}\right) \quad (1)$$

Wherey_i stands for the result of each experiment and n is thenumber of experiments.

For design of experiments with six factors (carrier concentration, surfactant concentration, internal phase concentration, feed/emulsion volume ratio, extraction time, and feed phase acidity) and five levels for each one, standard L25 orthogonal array was used. The rows of the matrix represent experimental runs which were carriedout in a random sequence. The factors and their levels wereassigned in Table 1. Table 2 showed the standard L25 orthogonal array.Factors A, B, C, D, E, and F are arranged in column 1, 2, 3, 4, 5, and 6 respectively.For analysis of the results and optimization of condition to arrangethe control factors, Minitab 13 software was used.

Table I. Levels of Different Variables for Extraction of Cadmium

Independent variable	Levels				
	1	2	3	4	5
Carrier concentration, A (M)	0.05	0.10	0.15	0.20	0.25
Surfactant concentration, B (v %)	3	5	7	8	10
Internal phase concentration, C (M)	0.10	0.30	0.50	0.70	1.00
External/emulsion ratio, D (v/v)	1	5	10	15	20
Stirring time, E (min)	1	5	10	20	30
Feed phase acidity, F	0.5	1	2	3.5	5

Table II. Experimental Design Orthogonal Array

Run	Input parameters												Observed values	
	A TOPO, M		B Span80, v%		C Int. phase, M		D Ext/ Emulsion Ratio		E Stirring time, min.		F Ext. pH		E%	S/N ratio
	Coded	Uuencoded	Coded	Uuencoded	Coded	Uuencoded	Coded	Uuencoded	Coded	Uuencoded	Coded	Uuencoded		
1	1	0.05	1	3	1	0.10	1	1	1	1	1	0.5	68.5719	36.7229
2	1	0.05	2	5	2	0.30	2	5	2	5	2	1	63.3097	36.0294
3	1	0.05	3	7	3	0.50	3	10	3	10	3	2	60.0000	35.5630
4	1	0.05	4	8	4	0.70	4	15	4	20	4	3.5	60.0000	35.5630
5	1	0.05	5	10	5	1.00	5	20	5	30	5	5	62.2705	35.8856
6	2	0.10	1	3	2	0.30	3	10	4	20	5	5	65.2706	36.2944
7	2	0.10	2	5	3	0.50	4	15	5	30	1	0.5	74.0190	37.3869
8	2	0.10	3	7	4	0.70	5	20	1	1	2	1	72.0000	37.1466
9	2	0.10	4	8	5	1.00	1	1	2	5	3	2	74.0000	37.3846
10	2	0.10	5	10	1	0.10	2	5	3	10	4	3.5	75.3990	37.5473
11	3	0.15	1	3	3	0.50	5	20	2	5	4	3.5	77.2000	37.7523
12	3	0.15	2	5	4	0.70	1	1	3	10	5	5	87.0000	38.7904
13	3	0.15	3	7	5	1.00	2	5	4	20	1	0.5	93.3037	39.3980
14	3	0.15	4	8	1	0.10	3	10	5	30	2	1	93.0397	39.3734
15	3	0.15	5	10	2	0.30	4	15	1	1	3	2	93.5000	39.4162
16	4	0.20	1	3	4	0.70	2	5	5	30	3	2	98.7000	39.8863
17	4	0.20	2	5	5	1.00	3	10	1	1	4	3.5	94.5038	39.5090
18	4	0.20	3	7	1	0.10	4	15	2	5	5	5	95.9078	39.6371
19	4	0.20	4	8	2	0.30	5	20	3	10	1	0.5	99.5000	39.9565
20	4	0.20	5	10	3	0.50	1	1	4	20	2	1	95.8000	39.6273
21	5	0.25	1	3	5	1.00	4	15	3	10	2	1	98.1591	39.8386
22	5	0.25	2	5	1	0.10	5	20	4	20	3	2	97.2119	39.7544
23	5	0.25	3	7	2	0.30	1	1	5	30	4	3.5	98.7000	39.8863
24	5	0.25	4	8	3	0.50	2	5	1	1	5	5	98.2599	39.8475
25	5	0.25	5	10	4	0.70	3	10	2	5	1	0.5	97.7487	39.8022

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III. EXPERIMENTAL

3.1 Materials

Tri octyl phosphine oxide (TOPO), Sigma-Aldrich Chemicals Co., was used as a carrier. Carrier with certain concentration ranged from 0.05 to 0.25 M dissolved in an organic solvent (Kerosene). The nonionic surfactant (sorbitanmonooleate) which is commercially known as Span 80 and a product of Sigma-Aldrich Chemicals Co. is used. Nickel(II) nitrate hexahydratewith a formula $Ni(NO_3)_2.6H_2O$, is used to prepare a stock solution of nickel. Aqueous Sulfuric acid solutions were used as an internal phase.

3.2 Extraction Experiments

Stable emulsion was prepared by emulsifying aqueous solution of sulfuric acid (internal phase) of different concentrations (Table 1) with an organic phase (membrane phase) containing carrier, surfactant and kerosene, using an ultraturax T25 homogenizer at 7500 rpm for 10 min. The emulsion was then immediately dispersed into a glass beaker (7.0 cm diameter) containing aqueous feed phase of nickel ions with a magnetic stirrer for different time intervals. After stirring, phase separated in a cylindrical separating funnels and samples of about 10mL were withdrawn and filtered through a filter paper to separate the remaining emulsion droplets and the aqueous feed phase, and then analyzed. The concentrations of nickel ions in the external phase were measured at a characteristic wavelength (231.604 nm) using an ICP (Perkin Elmer, Optima 7000 DV). A Horiba pH meter (model 713) with a combined glass electrode was used for pH measurements. The batch ELM experiments were conducted at room temperature of 25 °C. Extraction % is calculated according to eq. 2:

$$E\% = \frac{C_e^o - C_e^t}{C_e^o} \times 100 \qquad (2)$$

where C_e^o and C_e^t were the initial Ni ions concentration of the external phase and at time t respectively. Emulsion liquid membrane steps were shown in Fig.1.

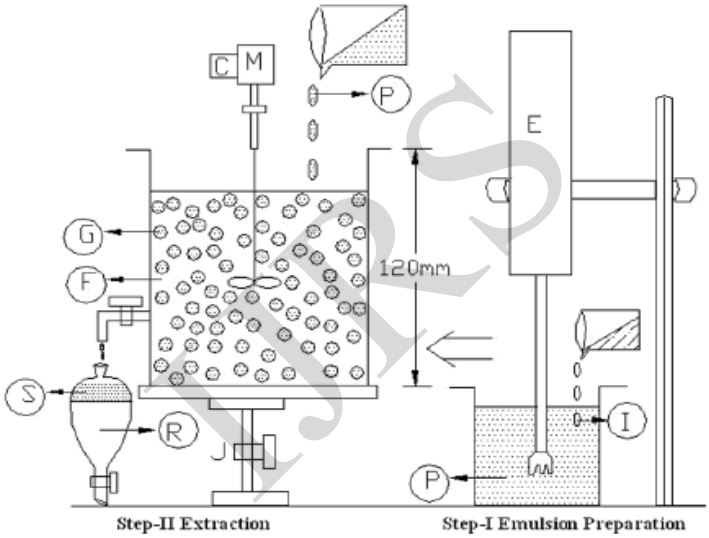


Fig. 1: Experimental steps of emulsion liquid membrane: I: internal phase, E: homogenizer, P: emulsion phase, M: stirrer, G: emulsion globules, F: feed phase, S: loaded emulsion, R: raffinate aqueous phase

IV. RESULTS AND DISCUSSION

4.1 Effect of Emulsion Liquid Membrane Parameters on Nickel Extraction

4.1.1 Effect of TOPO Concentration

Carrier concentration (TOPO) played an important role in the extraction process. Increasing TOPO concentration in the organic phase can increasefree sites for Ni ions complexation, which increasedthe extraction efficiency. It was found that the maximum extraction wasobtained at 0.25M concentration. Fig. 2clearly showed that temperatureat level 5 is suitable for Ni extraction.

4.2.1 Effect of Span80 Concentration on Ni Extraction

The effect of Span 80 on the extraction efficiency of Ni using ELM had been investigated. According to Fig. 4, the highest extraction efficiency and stable emulsion was obtained for 5.0% (v/v) SPAN80. At low concentration range, the stability of ELM was so low. Further increase of surfactant concentration resulted in an increaseof the emulsion viscosity that hinder the transfer of Ni ions to the internal phase through the emulsion which can cause lower extraction efficiency.Investigation of proper surfactant concentration gives us remarkable information about the behavior of emulsion stability. This phenomenon was explained by Valenzula et al. [22] and they showed that

excessive surfactant concentration in ELM tended to increase the resistance at the interface and this could be attributed to the increase in viscosity of ELMs. As shown in Fig.4, maximum extraction wasobserved when Span 80 concentration was at the level 3 (5v%).

4.3.1 Effect of Internal Phase Concentration

Figure 4 illustrated the effect of internal phase concentrations towards the performance of Ni ion extraction. The results showed that 85% of Ni ions were extracted. This could be explained by the fact that the increase in internal phase concentration would increase the concentration of hydrogen ion in internal phase, hence enhancing the ionic strength of internal phase. This condition hadled to the occurrence of osmotic pressure gradient between both the phases, which allowed the transportation of more water molecules from the feed phase into internal phase. Another observation also reported by Goyal et al. [23] who indicated that the higher concentration of stripping agent will lead to the reaction with surfactant and reduces the number of surfactant in the membrane which causes emulsion breaking.

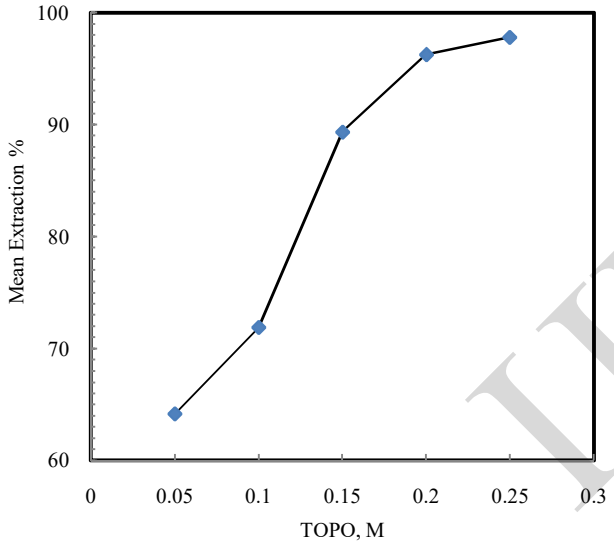


Fig 2: Effect of TOPO concentration on Ni extraction based on the Mean

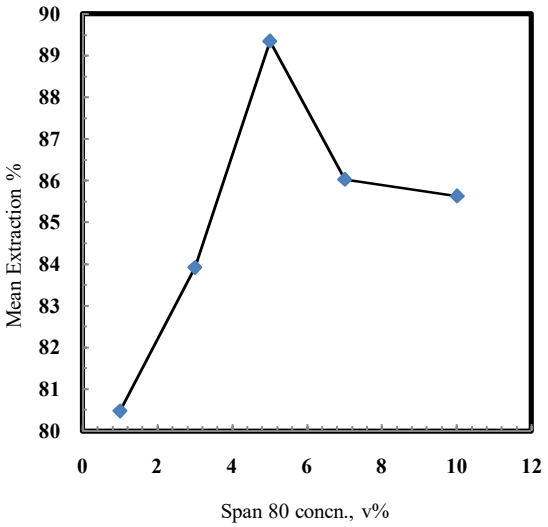


Fig. 3: Effect of Span 80 concentration on Ni extraction based on the Mean

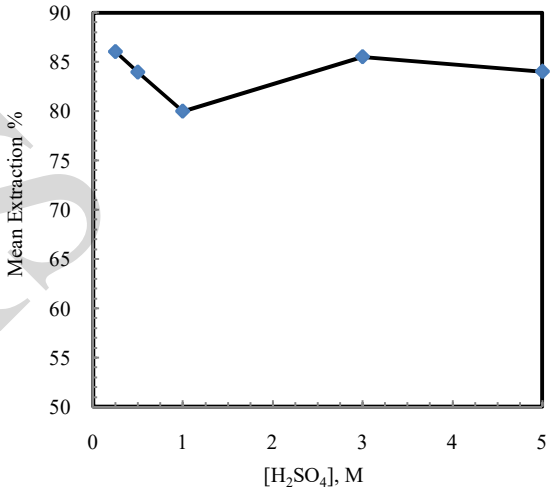


Fig. 4: Effect of internal phase concentration on Ni extraction based on the Mean

4.4.1 Effect of Feed to Emulsion Ratio

The feed to emulsion ratio was varied by changing the amount of feed phase and keeping the volume of the emulsion constant. The ratio controlled the interfacial mass transfer across emulsion liquid membrane. The result depicted in Fig. 5 observed that about 85% of Ni ion was extracted at a low treat ratio of 5.This was due to the emulsion phase did not disperse very welland the contact area between both phases significantly decreased. A further increase in the ratio up to 10 had slightly increased the extraction of Ni ions. Thisindicated that at this ratio, ELM system had achieved the effectivenessof the stability. In addition, increasing treat ratio also increased theemulsion phase hold up which simultaneously increased in theextraction capacity [4]. Higher ratios provided higher spacefor the emulsion dispersion, hence increasing the

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contact area between the two phases [5, 24, and 25]. Further increase in the ratio to 30,the emulsion got breakage. This condition might lead to theincrement of the surface area for mass transfer owing to theformation of a high number of emulsion globules. However, there was a continuous movement of water molecules into the internal phasewith the help of hydration surfactant which led to the emulsionbreakage.

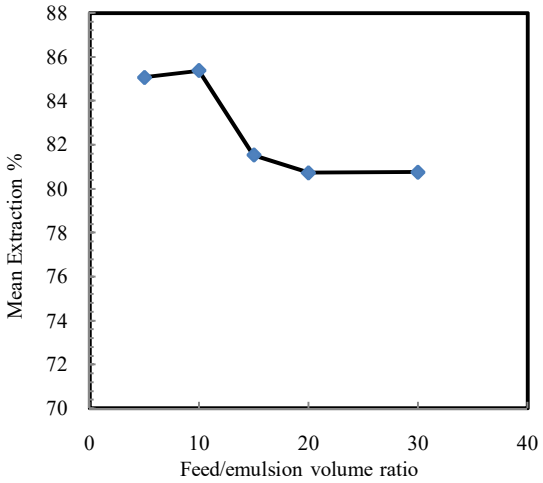


Fig. 5: Effect of Feed/emulsion volume ratio on Ni extraction based on the Mean

4.5.1 Effect of Stirring Time

The effect of stirring time on Ni ion extraction was depicted in Fig. 6. Theresult demonstrated that 84% of Ni ion was extracted after 10 mins.Emulsion breakage was observed at the first 5 min. This indicatedthat at the short contact time, the hydration of surfactant significantlyincreased the co-transport of water molecules into the internalphase, hence leading to the emulsion breakage. As the extractiontime increased up to 10 min, emulsion swelling slightly increaseddue to the surfactant which carried water molecules continuouslyinto internal phase. With further increasing the extraction time upto 30 min, emulsion swelling slightly increased.

4.6.1 The Effect of Feed Solution ph

Figure 7 showed the effect pH of feed phase towards the extraction of Ni ions. As can be observed, 86% of Ni ion was extracted and the extraction efficiency decreased as pH of the feed phase increased. Acidic medium provided the stronger complexation between the solute and the carrier hence increasing the extraction efficiency. On the other hand, extraction of Cr(VI) species were not possible at moderately acidic pH, because the chromium species were anionic only at low acidic pH. It was proved with many scientific studies [26–28]. Finally, maximum extraction percentage was obtained at pH 0.50. Thus, the acidic medium, pH 2 was used in the feed phase in order to enhance the extraction efficiency.

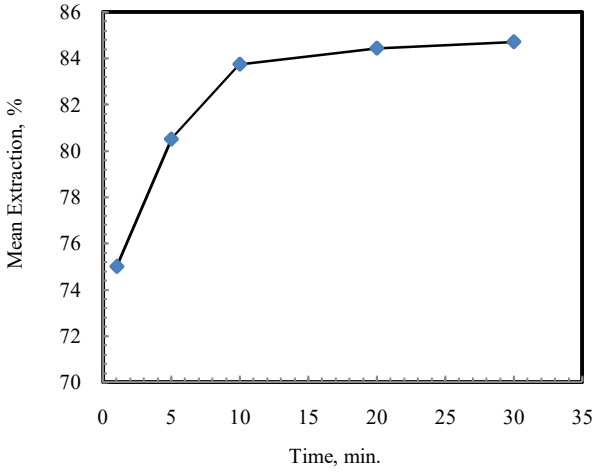


Fig. 6: Effect of stirring time on Ni extraction based on the Mean

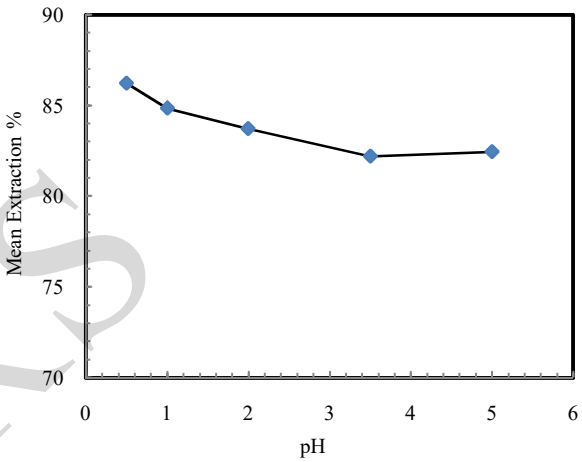


Fig. 7: Effect of feed phase acidity on Ni extraction based on the Mean

4.2 Analysis of Experimental Results Based on the Taguchi Methodology

Figure 8 showed the main effects of ELM parameters on the mean values of extraction % of nickel. Based on the experimental results showed in Table 2, an analysis of variance (ANOVA) was performed in order to estimate the predictive accuracy of the model. Also, by using Eq. 2, the S/N ratios for the five levels of each control factor were computed and tabulated in Table 3 to determine the relative significances of the different parameters. From the analysis of the E% data (see Table 3) and Fig. 8, it was observed that carrier concentration (TOPO) (91% contribution) played significant role in determining the E%. Furthermore, the feed/emulsion phase ratio (2.282% contribution) and $[H_2SO_4]$int (2.175% contribution) were less-significant parameters. Further, from Table 3 it can be seen that the optimal extraction performance for the ELM (maximum signal to noise ratios value) was obtained at TOPO concentration 0.25 M (Level 5), Span 80 concentration 10 v% (Level 5), internal sulfuric acid concentration 0.1 M

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(Level 1), feed/emulsion phase ratio 5 (v/v), stirring time 1 min. (level 1), and feed phase acidity (pH) 0.5 (Level 1).

According to Fig. 2, the increase of the TOPO concentration led to sharply increase in the E%. We noticed from Fig. 4 that, internal phase concentration led, in general, to a decrease in the E%, reaching to the lower value then E% started to increase with a further increase in each of the above mentioned ELM parameters. This is partly due to the swelling and breaking of emulsion.

Table III. Analysis of Ni Extraction Results

Factor	Signal-to-noise (S/N) ratio by factor level					Rank	DF	Seq SS	Adj MS	Contribution %
	1	2	3	4	5					
TOPO conc., M	35.95	37.15	38.95	39.72	39.83*	1	4	57.2775	14.3194	90.96
Span 80, v%	38.10	38.29	38.33	38.43	38.46*	6	4	0.8619	0.2155	1.367
$[H_2SO_4]_{int.}$	38.61*	38.32	38.04	38.24	38.40	3	4	1.3694	0.3424	2.175
Feed/emulsion ratio	38.48	38.54*	38.11	38.37	38.10	4	4	1.4371	0.3593	2.282
Stirring time, min.	38.53*	38.12	38.34	38.13	38.48	5	4	1.0156	0.2539	1.163
Feed phase pH	38.65*	38.40	38.40	38.05	38.09	2	4	1.0105	0.2526	1.604
Total							24	62.9719		100

Overall mean= 36.186 * Optimum level Contribution %=[Seq SS/Seq SS(Total)]x100

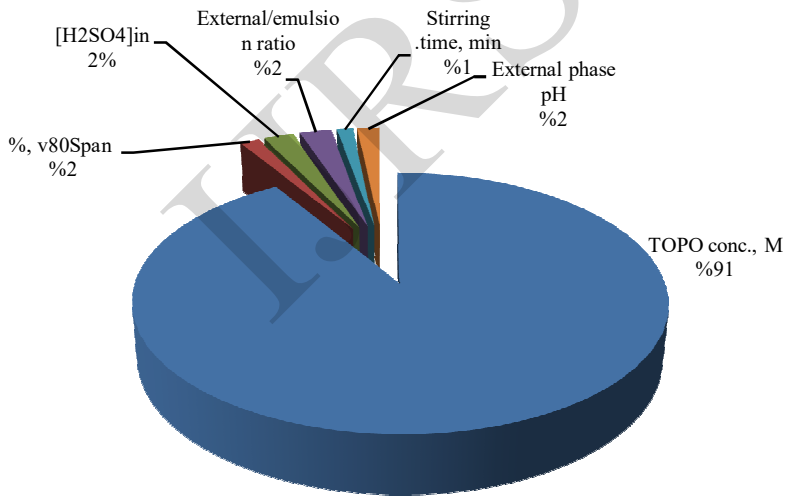


Fig. 8: The contribution pie chart of extraction of nickel

V. CONCLUSIONS

The results of this work clearly showed that ELM provided the possibilityof extracting nickel ions from wastewater. This method is simple, selective, andsensitive which can be a good alternative for previously appliedmethods like liquid-liquid extraction. ApplyingTaguchi method for optimization of Ni extraction by ELM showed that carrier concentration (90.96%)has the highest contribution on process performance and feed/emulsion ratio (2.282%), internal phase concentration (2.175%) are the next effective parameters, respectively.

According tothe analysis of results, a TOPO concentration of 0.25M, Span80 concentrationof 10v%, an internal phase concentration 0.5M, feed/emulsion phase ratio 5/1, stirring time of 1 min and a feed pH 0.5 are the optimum extraction conditions.

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