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Solvent Extraction Process for the Recovery Cobalt and Nickel from Low-Grade Laterite using Batch Recycle System

Proses Extrasi Cair-cair Recoveri Kobalt dan Nikel dari Bijih Laterit Kadar Rendah menggunakan Sistem Batch Recycle

Sudibyo[1,*], Agus Junaedi[1], Muhammad Amin[1], Slamet Sumardi[1], Fajar Nurjaman[1], Bramantyo Bayu Aji[1], Yayat Iman Supriyatna[1], dan Lilis Hermida[2]
[1]Research Unit for Mineral Technology, Indonesian Institute of Sciences, Lampung, Indonesia
[2]Department of Chemical Engineering, Lampung University, Lampung, Indonesia
[*]*E-mail*: sudibyo@lipi.go.id

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Abstrak

Laterite kadar rendah yang memiliki kandungan nikel kurang dari 0.5% sangat sulit untuk diolah menggunakan proses pirometalurgi. Salah satu cara untuk mengatasi masalah ini adalah proses ekstraksi cair-cair. Pada penelitian ini, proses ekstraksi cair-cair menggunakan metode batch recycle telah berhasil memisahkan nikel dan cobalt dari laterite kadar rendah. Asam sulfat digunakan untuk melarutkan laterite pada tekanan atmosfir. Sedangkan cyanex dalam toluen digunakan sebagai pelarut organik. Desain eksperimen Taguchi telah dipilih untuk menganalisis dan mengoptimasi proses extraksi cair-cair menggunakan metode batch recycle. Hasil analisis Taguchi menunjukkan bahwa kondisi optimum adalah pada pH 7, lama operasi 3 jam, laju operasi 0,9 liter/menit dan perbandingan volume organik/aqueous 0,75.

Kata kunci: *Ekstraksi cair-cair, Batch recycle, Laterite, Ekstraksi kobalt-nikel, Aplikasi taguchi*

Keywords:
Solvent extraction
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Taguchi applications.

Abstract

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Low-grade laterite which contains nickel lower than 0.5% is difficult to be processed using pyrometallurgy. One method which is able to solve this problem is solvent extraction. In this study, a solvent extraction process using batch recycle methods has been successfully applied to separate nickel and cobalt from low-grade laterite. Sulphuric acid was used to leach laterite at atmospheric pressure. Meanwhile, cyanex in toluene was used as an organic solvent. Taguchi experimental design has been used to analyze and optimize the solvent extraction process using batch recycle methods. Taguchi analysis results show that the optimum conditions are at 7 of pH, 3 hours of time operation, 0.9 liter/minute of flow rate and 0.75 of organic/aqueous solutions ratio.

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INTRODUCTION

Indonesia is rich in laterite nickel ores. Indonesia has many low-grade laterite resources which have around 0.5% nickel and 0.04% cobalt. However, this laterite is difficult to be processed using pyrometallurgy methods (Astuti et al. 2016). One of the methods that can be applied to produce both nickel and cobalt is hydrometallurgy method. The solvent extraction method is hydrometallurgy method which is able to solve this problem.

In this study, Cyanex 272 in toluene was used as an organic solvent. Cyanex 272 has good performance to separate cobalt from nickel solution in solvent extraction process (Iliev et al. 2012; Kursunoglu, Ichlas, and Kaya 2017; Guimarães, Da Silva, and Mansur 2014; Park and Mohapatra 2006)4,4- trimethylpentyl. Cobalt ion will transfer to the organic solution. Meanwhile, nickel ions will stay in aqueous solution. This extraction process is environmentally friendly because the organic solvent can be recycled (Iliev et al. 2012). In addition, solvent extraction produces pure material. Therefore, solvent extraction has a bright prospect to solve the problem.

To increase a performance of solvent extraction process, a batch recycle methods was chosen to treat a low-grade laterite. This method uses the flow rate of the aqueous solution from the top of extraction column as a stirrer which hit the organic solution. The aqueous solution is recycled continuously at the certain duration of time to ensure the extraction process runs perfectly. Although the batch recycles method is a new method in the extraction process,it has been successfully used in another process such as in electrocoagulation process (Sudibyo, Hermida, and Suwardi 2017). This process is also able to process a large volume of aqueous solution in a single extraction column.

Taguchi experimental design was applied to select the optimum operating conditions and also to analyze the effect of all parameters towards extraction process (Zainal, Shukor, and Razak 2015); (Rosa et al. 2009). Taguchi method was able to reduce the time and cost because it just needs a few numbers of experiments which is less than another designed experiment. The purpose of this research is to get a higher value of the percentage of the increase of nickel concentration in aqueous solution (% Ni) by varying and optimizing the parameters such as an operating duration, flow rate, ratio of organic/aqueous, and pH.

METHODOLOGY

Materials used in this research were laterite ores South East Sulawesi Island (Integra Mining Co. Ltd), sulfuric acid (H_2SO_4) from Merck, Cyanex 272 ($C_{16}H_{35}O_2P$) from Sigma Aldrich, Toluene (C_6H_5-CH_3) High Gloss, water. X-Ray Fluorescence (XRF, Panalytical Epsilon 3 XLE from Netherland), Atomic Absorption Spectroscopy (AAS, Hitachi ZA-3000 form Japan), X-Ray Diffraction (XRD, Pan Alytical X'pert PRO MRD l from Netherland), and pH indicator (Merck) were used to analyze ores and product. Sulfuric acid was used to leach the laterite, then the pregnant leaching solution was extracted using cyanex 272 (10%) which dissolved into toluene. The experiments were conducted using Taguchi as listed in Table 1.

The extraction process was operated using the batch recycle methods. The aqueous solution was circulated into extraction column with the desired flow rate. The organic solution flowed into the extraction column from the top of the extraction column. Hence, the organic solvent layer was at the upper aqueous layer, meanwhile, the aqueous flow was contacted the organic solution layer from the top of extraction column as shown in Fig. 1. This batch recycle methods was operated at the certain duration time as shown in Table 2. At the end of batch recycle process, the organic solutions were separated using the three valves at the side of extraction column.

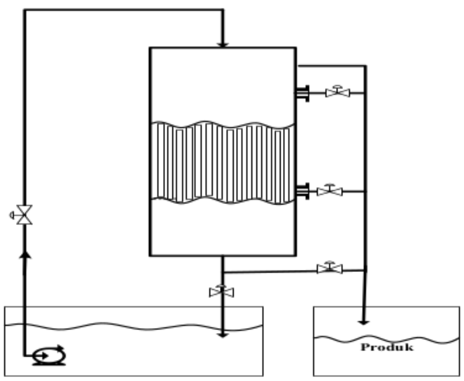

Figure 1. Batch recycling extraction scheme

The result of this process was the percentage of increase nickel concentration on the aqueous solution since cobalt was moved to organic solvents. The percentage of increase in nickel concentration was calculated using the following equation:

$$\%Ni = (W_1 - W)/W \times 100\% \qquad (1)$$

%Ni is the percentage of increase nickel concentration on the aqueous solution(%), W is initial nickel concentration in aqueous solution before the extraction. W_1 is nickel concentration in aqueous solution after the extraction. The results of the experiment were analyzed using a signal-to-noise (S/N) ratio of "larger the better" which using equation as follow:

$$S/N = -10*\log(\Sigma(1/Y2)/n) \qquad (2)$$

where Y = responses for the given factor level combination and n = number of responses in the factor level combination (Rosa et al. 2009).

Tabel 1. The composition of laterite feedstock

Component	Concentration (%)
Fe	10.97
LE	78.25
Si	5.427
K	1.529
Cl	1.419
Ca	1.776
Al	0.578
Ni	0.514
Cr	0.323
Mn	0.177
Co	0.04
S	0.022
Sb	0.022
Sn	0.016
Zn	0.0087
Cd	0.015

.

Tabel 2. Experiment design and experiment results

	Design of Experiment				Result
Run	Time (hour)	Flow Rate (liter/ minute)	Organic Volume/ Aqueous volume	pH	Increasing Ni (Ni%)
1	1	0.3	0.25	4.5	114.53
2	1	0.6	0.5	5.5	6.70
3	1	0.9	0.75	6.5	230.15
4	2	0.3	0.5	6.5	188.47
5	2	0.6	0.75	4.5	194.31
6	2	0.9	0.25	5.5	53.92
7	3	0.3	0.75	5.5	51.97
8	3	0.6	0.25	6.5	309.99
9	3	0.9	0.5	4.5	193.73

RESULT AND DISCUSSION

XRF and XRD Analysis for Nickel Ore

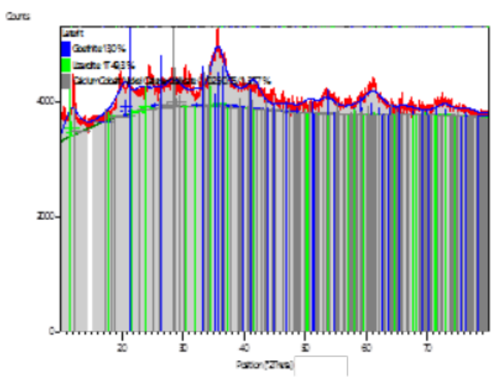

Figure 2. XRD analysis for Nickel Ore

Nickel laterite was analyzed using XRF and XRD. The result of XRF was listed in Table 1. The table shows that the laterite contains 0,514% of nickel, 0,04% of cobalt dan 1,776% of calcium. The nickel content found in this ore is classified as low grade, hence it called as limonite ore. This ore able to process using hydrometallurgy method. Figure 2 shows the XRD analysis of limonite ore. This ore contains gheotite, lizardite, and catena disilicate.

The Effect of Operation Duration to The Increase of Nickel Percentage in Aqueous Solution (% Ni)

Operation duration was varied from 1 to 3 hours as shown in Figure 2. The figure shows that the optimum condition is at 3-hour operation. The result shows that the increase of operation duration will increase %Ni because it will improve contact between aqueous and organic solution (Ndlovu and Mahlangu 2008)Ni, Mg and Ca. The pH(50. It will increase the movement of the cobalt and others metals from aqueous solution to the organic solution. Meanwhile, the Ni ions still stay in aqueous solution. As a result, the nickel concentration in aqueous solution increased.

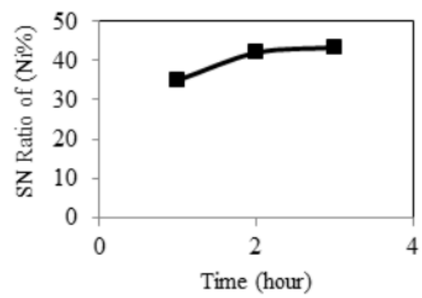

Figure 3. The effect of the duration of operation towards the SN ratio of the increase of nickel concentration in aqueous solution (% Ni)

The Effect of Flowrate to the Increase of Nickel Concentration in Aqueous Solution (% Ni)

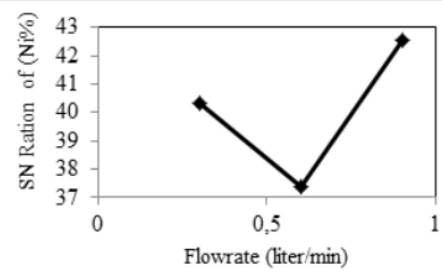

Figure 4. The effect of flow rate towards the SN ratio of the increase of nickel concentration in aqueous solution (% Ni)

The flow rate was varied from 0.3 to 0.9 liter/minute as shown in Figure 3. The figure shows that the optimum flow rate is 0.9 liter/minutes. The flow rate of aqueous from the top of extraction column in batch recycle process has a function as a stirrer to mix the organic and aqueous solution. The increase of flow rate will improve the increase of nickel in aqueous solution as shown in Figure 3. The higher of flowrate will rise the contact between the organic and aqueous solution in an extraction column that causes the mass transport of cobalt and other metal from aqueous to organic. Cyanex in the organic solvent was able to attract cobalt and other metal, meanwhile, ion Nickel, and iron were rejected to move inside organic solvent (Iliev et al. 2012; Kursunoglu, Ichlas, and Kaya 2017; Guimarães, Da Silva, and Mansur 2014; Park and Mohapatra 2006)4,4- trimethylpentyl. Hence, the nickel concentration was increased in aqueous solution. Figure 3 show that S/N ratio of 0.6 liter/minute is lower than 0.3 liter/minute since at the flowrate of 0.6 lter/minute cause any impurities not attract into organic solvent hence %Ni in aqueous solvents was decreased.

The Effects of Ratio Organic/Aqueous on The Increase of Nickel Percentage (%Ni) on The Aqueous Solution

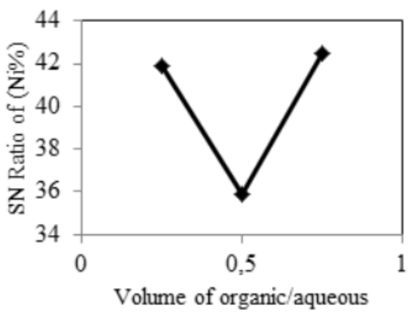

Figure 5. The effect of the organic/aqueous volume towards the SN ratio of increasing percentage of nickel

In order to learn the effect of the organic/aqueous ratio towards SN ratio of the increase of nickel percentage (%Ni), the organic/aqueous ratio was varied from 0.25 to 0.75 as shown in Figure 4. From the figure, it is found that the optimum organic/aqueous ratio is 0.75. The result shows that the increase of organic/aqueous ratio will cause the nickel increasing (%) in aqueous solution. The increase of organic solution volume will increase the cyanex amount in the organic solvent that creates the increase of mass transport of cobalt from aqueous to organic solution (Iliev et al. 2012). Finally, nickel amount in aqueous solution will increase since many metal ions have transferred to the organic solvent. Figure 4 also show that the S/N ratio of 0.5 is lower than 0.25 since at organic/aqueous ratio of 0.5 will decrease the ablity of organic solvent to attract any impurities hence %Ni in aqueous solvents was decreased.

The Effect of pH on the Increase of Nickel Percentage (%Ni) on the Aqueous Solution

pH of aqueous solvents was varied from 4.5 to 6.5 as shown in Figure 5. The figure shows that the optimum condition is at 6.5 of pH. Other researchers also found that Cobalt extraction will increase as the increase of pH when pH varied between 5 to 6 (Preez and Preston 2004). The cobalt and other metal will move from aqueous to organic solvents. Meanwhile, nickel ion is still in the aqueous solution. Hence, %Ni in an aqueous increase at 6.5 pH.

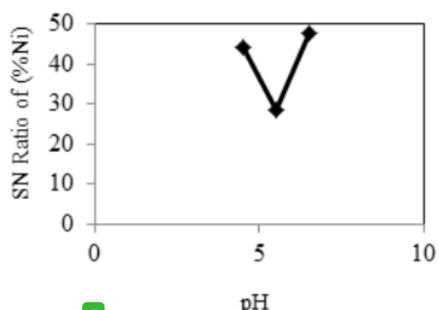

Figure 6. The effect of the pH towards the SN ratio of increasing percentage of nickel

The results of this study were analyzed using one of Taguchi's method of SN (Signal to Noise) ratio of larger better are listed in Table 3. The table also shows the value of delta for each parameter used in this research. The highest value of delta indicates that the parameter has the highest effect towards the increasing of nickel percentage. From the table, it is found that the first, second, third and fourth ranks are pH, time duration, organic/aqueous volume and flow rate, respectively.

Table 3. Response tabel signal to noise ratios larger is better

Level	Time	Flowrate	Org./aqu.	pH
1	34.98	40.333	41.8801	44.23
2	41.97	37.373	35.9234	28.49
3	43.30	42.54	42.4416	47.52
Delta	8.32	5.1671	6.5182	19.03
Rank	2	4	3	1

CONCLUSION

Solvent extraction to separate cobalt from nickel solution has been conducted using batch recycle methods. Optimum condition of this process has been carried out using Taguchi methods. The Taguchi analysis methods show that the optimum conditions are at 7 of pH, 3 hours of time operation, 0.9 liters/minute of flow rate, and 0.75 of organic/aqueous ratio. The Taguchi analysis also shows the first, second, third, and fourth ranks of a parameter which affect the increasing nickel percentage are pH, time duration, organic/aqueous volume and flow rate, respectively.

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