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Application of Taguchi Optimization for Nickel Electrowinning Using Batch Recycle Methods

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Abstract. Low-grade laterite which has nickel content 0.5% has been processed using solvent extraction and followed by electrowinning process. Cyanex which diluted in toluene was used as an organic solvent to separated cobalt from nickel solvent. In this work, nickel solvent which frees from cobalt was processed using electrowinning. Electrowinning was conducted using batch recycle methods to ensure the electrowinning performed well. Meanwhile, cylindrical cathode electrode was used to increase the performance of electrowinning, easy to operate and easy to harvest. The parameters which have a significant effect in batch recycle methods such as flow rate, voltage and process duration. Taguchi analysis showed the optimum condition and the parameter which has the highest effect towards the mass of nickel electrodeposits.

INTRODUCTION

Low-grade laterite which contains nickel less than 0.5% is very difficult to process using pyrometallurgy. Solvent extraction has become the methods chosen for the separation of cobalt and nickel from low-grade laterite [1–5]. At the end of the solvent extraction process, the nickel should separate from acid solution. One of the methods which able to take a nickel from solution is electrowinning. This method is more effective than other methods take positive electroactive ions such nickel ions [6–8]. In this work, electrowinning was conducted using batch recycle methods, hence able to treat the solvent optimally. The cylinder electrode type as a cathode was chosen in batch recycle electrowinning process because low operational cost, friendly for the environment, easy to install, design is compatible, it can be operated in large-scale process, acid electrolyte can be recycle, it can produce high purity of metal and it can be operated in low concentration of metal solution [6].

Cyanex in toluene was chosen in the solvent extraction process prior to the electrowinning. Cyanex good performance to separate cobalt from nickel solution [1–5]. Hence, higher purity of nickel able to recover using electrowinning. The batch recycle method was used in the nickel electrowinning to increase the nickel recovery. In this batch recycle methods, the electrolyte enters from the bottom of electrowinning cell using a pump, then the effluent of the electrolyte at top of electrowinning cell flowed back to electrolyte tank as shown in Fig. 1. This batch recycle method was conducted for certain duration of time to ensure the electrowinning was performed well.

In this research, parameters which affected the nickel electrodeposits amount at cathode were studied and optimized using Taguchi method. The parameters which studied in this research such as the flow rate, the process duration, and the voltage. Taguchi is one of the designs of an experiment which use few numbers of experiments to analyze the effect of each parameter towards the performance of the process and also to optimize the parameters simultaneously [9,10].

EXPERIMENTAL PROCEDURE

In this research, laterite from south-east Sulawesi (from Integra Mining Co. Ltd) was sieved using a sieve tray (mesh 150). Leaching process was conducted using vat leaching (atmospheric pressure) to leach 700 gram of laterite in 7 liters 1 M H₂SO₄ (from Sigma Aldrich Co. ltd.). Leaching process was ended until cobalt present in leaching solvent. Then the leaching solvent was filtered using filter press. This leaching solvent was extracted using Cyanex 272 (10% v/v) in toluene (70%) and the comparison of the organic/aqueous volume was 3/4. Aqueous solvent of extraction process was processed using electrowinning. In this research, electrowinning process was operated using batch recycle methods, meanwhile, the cathode was aluminum cylindrical electrode as shown in Fig. 1. The direct current (DC) power supply was used in this study. In this method, the electrolyte was pumped from the bottom electrowinning reactor and the outlet electrolyte was recycled from the top of electrowinning reactor. In this work, the voltage from DC power supply was monitored using a potentiometer. The parameters were optimized using Taguchi methods such as flow rate, operation duration, and voltage as shown in Table 2.

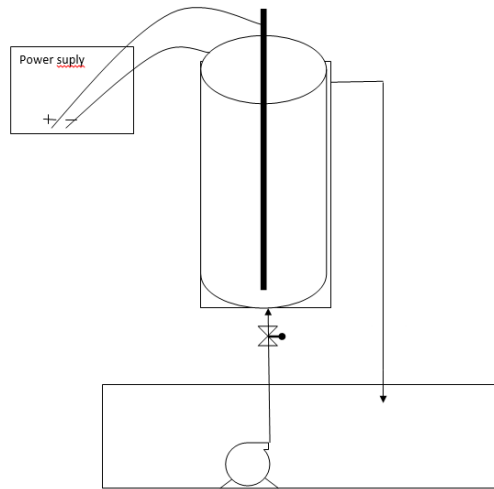


FIGURE 1. Batch recycling electrowinning scheme

TABLE 1. Design of experiment using Taguchi method

Run	Flowrate (Litre/minute)	Process Duration (Hour)	Voltage (V)
1	0,5	2	2
2	1	3	2
3	1,5	4	2
4	1	2	3
5	1,5	3	3
6	0,5	4	3
7	1,5	2	4
8	0,5	3	4
9	1	4	4

TABLE 2. The result of an experiment using Taguchi method

Run	Electrodeposit weight (gr)	Ni composition (%)	Ni (mg)
1	8	2.16	172.8
2	0.5	0.8	4
3	2	2.02	40.4
4	1	1.52	15.2
5	17.41	2.95	513.6
6	2	2.02	40.4
7	1	7.15	71.5
8	4	4.69	187.6
9	15	14.59	2188.5

RESULTS AND DISCUSSION

Analysis of Experimental Design

An orthogonal array was used to discover the optimal condition and to choose the parameters that have the biggest effect on the nickel electrodeposit. 9 runs were conducted in order to optimize the parameters used as shown in Table 1. The results of electrowinning experiments were electrodeposited in the cathode electrode. The total mass of electrodeposits was calculated from a weight of cathode after electrowinning minus the weight before electrowinning. Meanwhile, Nickel concentration was calculated using XRF. Hence, total nickel mass on electrodeposits can be determined as listed in Table 2.

The experiment results were analyzed using the signal/noise (S/N) ratio of larger the better which calculated using following equation:

$$S/N = -10 \cdot \log(\Sigma(1/Y^2)/n) \quad (1)$$

where Y = responses for the given factor level combination and n = number of responses in the factor level combination [9]. The value of S/N ratios were listed in Table 3. A delta value was the highest S/N ratio value minus the lowest S/N ratio value in the same parameter. The highest value of delta indicate the most significant parameters towards the mass of nickel electrodeposits. The table shows that the most significant parameter is the flow rate followed by the process duration and the last is the voltage.

TABLE 3. Data Response Tabel Signal to Noise Ratios Larger is better

Level	flowrate	Process Duration	Voltage
1	29.6399	35.158	40.7811
2	36.659	37.2394	34.1603
3	49.7846	43.6861	41.1421
Delta	20.1447	8.5281	6.9817
Rank	1	2	3

The Effect of Flow Rate towards the Mass of Nickel Electrodeposits (% Ni)

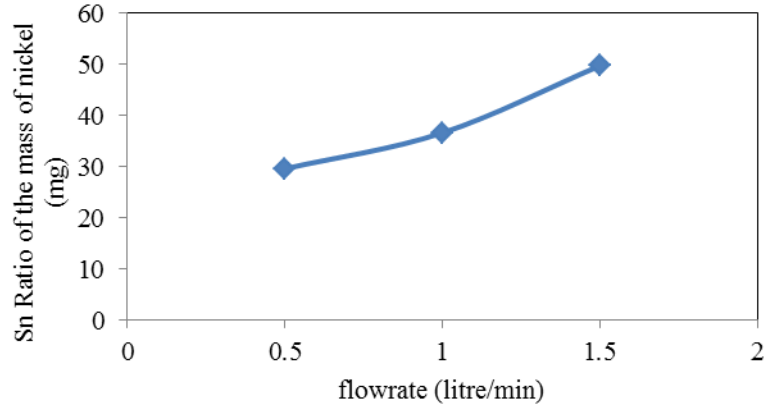


FIGURE 2. The effect of the flow rate towards the SN ratio of the mass of nickel electrodeposits (% Ni)

Electrowinning using cylindrical electrode able to process a large volume of electrolyte since electrolyte was pumped into the electrochemical reactor from electrolyte vessel. The electrolyte inlet was from the bottom of the electrochemical reactor. In this work, the volume of electrolyte is 2 liter, meanwhile, the electrochemical capacity was 1 liter as shown in Fig. 1. In order to know the effect of electrolyte flow rate towards the mass of nickel electrodeposits, the electrolyte flow rate was varied from 0.5 to 1.5 liter as shown in Fig. 2. The Figure shows that the increase in flow rate will increase the mass of nickel electrodeposits. In the batch recycle methods, increasing of flowrate will increase the volume of electrolyte which through the electrochemical reactor, hence it will increase the contact of the electroactive ions inside the electrolyte with the electrically charged electrode [11]. The positive electroactive ions as like nickel will be deposited into the negative electrode (cathode) [7–9]. This cathode was the cylindrical electrode which the electrolyte has flowed between the cathode and anode. The S/N ratio analysis was determined that the optimum flow rate was at 1.5 liters/minute as shown in Fig. 2.

The Effect of Operation Duration towards the Mass of Nickel Electrodeposits (% Ni)

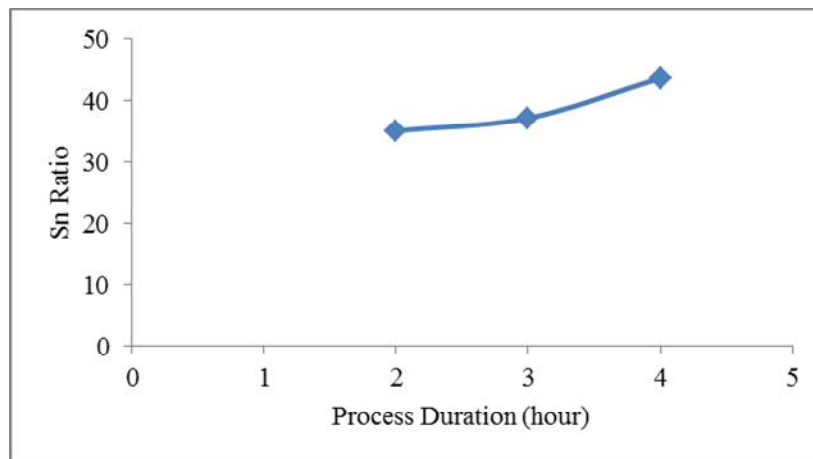


FIGURE 3. The effect of the process duration towards the SN ratio of the mass of nickel electrodeposits (% Ni)

In order to study the effect of process duration towards the mass of nickel electrodeposits (% Ni), the process duration was varied from 2 until 4 hours as shown in Fig. 3. The figure shows that the increase of process duration will enhance the mass of nickel electrodeposits. The increasing of process duration will increase the volume of electrolyte which flows in the electrochemical reactor. This will increase the contact between the electroactive ions inside the electrolyte with the electrode⁷. Hence, the number of nickel ions electrodeposits at the cathode increased. The Taguchi analysis result confirms that the optimum process duration is at 4 hours as shown in Fig. 3.

The Effect of Voltage towards the Mass of Nickel Electrodeposits (% Ni)

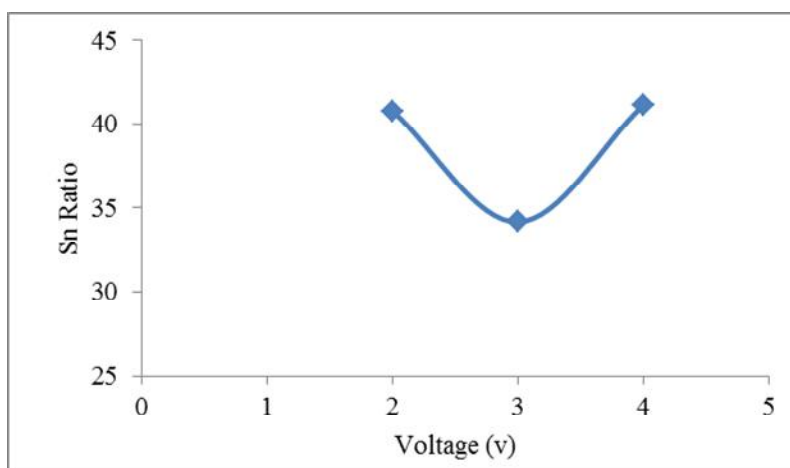


FIGURE 4. The effect of the voltage towards the SN ratio of the mass of nickel electrodeposits (% Ni)

In order to identify the effect of voltage towards the mass of nickel electrodeposits (% Ni), the voltage was varied from 2 to 4 volt as shown in Fig. 4. The figure shows that the increase in voltage will increase the mass of nickel electrodeposits (% Ni). The increasing of voltage will increase the mass transport of ions from anode to cathode. Hence, it will increase the electroactive ions in the electrolyte which be deposited at the cathode [7]. However, the mass of nickel electrodeposited at 3 V was decreased from 2 V. Since at that voltage, the hydrogen evolution reactions were dominant, hence it will disturb the deposition process of electroactive ions at the cathode [8,9]. The Fig. 4 also shows that the optimum voltage is at 4 V.

CONCLUSION

Nickel electrowinning from low-grade laterite has been conducted using the cylindrical cathode electrode which operated using batch recycle methods. Optimum condition of this process has been determined using Taguchi methods. The Taguchi analysis methods show that the optimum conditions are at 1.5 liters/minute of flow rate, 4 hours of process duration and 4 voltage. The Taguchi analysis also shows the first, second, third and fourth ranks of a parameter which affects the mass of nickel electrodeposits are flowrate, process duration, and voltage, respectively.

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