

# OPTIMIZATION OF NICKEL EXTRACTION FROM LATERITIC ORE IN HYDROCHLORIC ACID SOLUTION WITH HYDROGEN PEROXIDE BY TAGUCHI METHOD

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**ABSTRACT:** Taguchi optimization method was used to determine optimum conditions for the extraction of nickel from lateritic ore in hydrochloric acid solution with hydrogen peroxide. Leaching time, stirring speed, temperature, hydrochloric acid concentration and hydrogen peroxide concentration were chosen as parameters. The optimum conditions for dissolution were found as leaching time of 240 min, a temperature of 70°C, hydrochloric acid concentration of 3 M, hydrogen peroxide concentration of 0.1 M and without stirring. The experimental results under optimum leaching conditions, showed that the extraction of nickel from lateritic ore was 90.66%. Analysis of variance (ANOVA) was applied to experimental results. Percentage contributions of each factor for the extraction of nickel were determined.

Key Words: Leaching, Lateritic ore, Nickel; Hydrochloric acid, Hydrogen peroxide; Taguchi method

# Nikelin Lateritik Cevherden Hidrojen Peroksitli Hidroklorik Asit Çözeltisinde Çözündürülmesinin Taguchi Yöntemiyle Optimizasyonu

ÖZ: Nikelin lateritik cevherden hidrojen peroksitli hidroklorik asit çözeltisinde çözündürülmesinin optimum koşullarını belirlemek için Taguchi optimizasyon yöntemi kullanılmıştır. Deney parametreleri olarak, süre, karıştırma hızı, sıcaklık, hidroklorik asit derişimi ve hidrojen peroksit derişimi seçilmiştir. Çözündürme için optimum koşullar, karıştırma yapılmadan, 240 dakika liç süresi, 70 °C sıcaklık, 3 M hidroklorik asit derişimi ve 0.1 M hidrojen peroksit derişimi olarak bulunmuştur. Optimum koşullarda yapılan deney sonuçları, nikelin lateritik cevherden çözündürülmesinin %90.66 olduğunu göstermiştir. Deney sonuçlarına varyans analizi (ANOVA) uygulanmış ve her faktörün nikel çözündürülmesine olan katkısı belirlenmiştir.

Anahtar Kelimeler: Liç, Lateritik cevher, Nikel; Hidroklorik asit; Hidrojen peroksit; Taguchi metodu

## INTRODUCTION

Nickel is a widely used metal due to its chemical and physical properties, such as corrosion resistance, alloying, electrical, thermal and catalyst properties. The main use area of nickel is the production of stainless steel and alloys, other uses are electroplating and chemistry. Laterites are oxide ores widely distributed in the equatorial regions. Lateritic nickel deposits occur during laterization, namely, a weathering process of ultramafic rocks containing minerals such as olivine, pyroxene and amphibole (Golightly, 1981). Ni-laterites can be classified in three main groups as oxide, hydrous silicate and clay silicate deposits (Golightly, 1981; Gleeson, et al., 2003; Brand et al., 1998; Sagapoa et al., 2011;

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Georgiou and Papangelakis, 1998). Nickel laterites constitute an important part of world reserves of nickel. About 70% of the world's nickel reserves occur from laterite deposits, the rest of sulphide deposits (Elias, 2002; Landers et al., 2009; Soler et al., 2008). However, about 40% of the world's nickel production is provided from lateritic ores (Gleeson, et al., 2003; Elias, 2002; Brand et al., 1998; Dalvi et al., 2004). Nickel production from lateritic ores contains pyrometallurgical and hydrometallurgical processes (Deepatana et al., 2006). Generally, pyrometallurgical processes (ferronickel and matte smelting) involve drying, calcining/reduction and electric furnace smelting. However, hydrometallurgical processes can be applied as the Caron process, HPAL (High pressure acid leaching), AL (Atmospheric leaching) and acid heap leaching (Dalvi et al., 2004). In the literature, there are several studies related to lateritic nickel leaching in different solutions using high pressure or atmospheric pressure (agitation or heap leaching). The solutions used in these studies can be given as sulphuric acid (Georgiou and Papangelakis, 1998; Agacayak and Zedef, 2012; Luo et al., 2010; Stopic et al., 2002; Ayanda et al., 2011; Rubisov et al., 2000; Agatzini-Leonardou and Zafiratos, 2004; McDonald and Whittington, 2008a; Thubakgale et al., 2013; Landers et al., 2009; Mohammadreza et al., 2014), hydrochloric acid (Ayanda et al., 2011; Park and Nam, 2008; Agacayak et. al., 2011; Olanipekun, 2000; Wang et al. 2012; Guo et al., 2015; McDonald and Whittington, 2008b; Li et al., 2012), nitric acid (Agacayak and Zedef, 2013; Ayanda et al., 2011; Ma et al., 2013), ammonia (Zhai et al., 2010; Chen et al., 2010; Zuniga et al., 2010), citric acid, oxalic acid and acetic acid (Sahu et al., 2011; Behera et al., 2010; Sukla and Panchanadikar, 1993, Kursunoglu and Kaya, 2015). The main aim in leaching studies is to provide metal extraction with high recovery. Therefore, optimization of leaching parameters is very important. Taguchi method is widely used in optimization studies, both leaching studies and other scientific fields (Bese et al., 2010; Abali et al., 2006; Demir and Donmez, 2008; Babaei-Dehkordi et al., 2013; Asl et al., 2015; Dogan and Yartasi, 2014; Copur, 2002; Abali et al., 2011; Moghaddam et al., 2006; Ata et al., 2001; Safarzadeh et al., 2008; Ilyas et al., 2010; Zolfaghari et al., 2011). One of the advantages of Taguchi method is to keep costs to a minimum level compared to conventional experimental design methods and closer performance to the desired level. Another advantage is to adapt optimum conditions obtained from laboratory studies to real production conditions (Taguchi, 1987; Demir and Donmez, 2008; Donmez et al., 1998; Ata et al., 2001; Roy, 1995; Safarzadeh et al., 2008).

The dissolution of lateritic nickel ore in hydrochloric acid solution with hydrogen peroxide was investigated. As a result of the literature review, no study was found on the optimization of the dissolution of lateritic nickel ore for this medium. Hence, it was aimed to determine optimum leaching condition of lateritic nickel ore by Taguchi method.

#### MATERIAL AND METHOD

Lateritic nickel ore sample was taken from the Gördes (Manisa) region of Turkey. The sample used was a typical limonitic laterite ore with high iron content. According to the mineralogic analysis, the sample contains goethite, hematite and quartz. The XRD pattern of the sample was given in Figure 1.

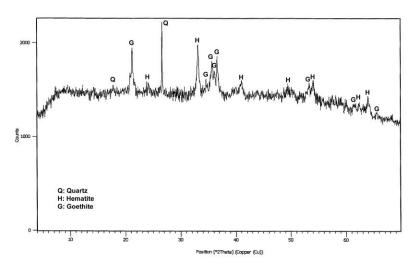


Figure 1. XRD pattern of the sample

First, the sample was ground to  $-212~\mu m$  in order to use in the experiments. Then, the sample was wet sieved to obtain different particle size fractions as -212+150, -150+106, -106+75 and  $-75+53~\mu m$ . Nickel and iron contents were determined with Atomic Absorption Spectrophotometer (AAS). Nickel and iron contents of each size fraction were given in Table 1.

Table 1. Nickel and it of contents of each size if action								
Particle size, µm	Ni content, %	Fe content, %						
-212+150	1.05	36.01						
-150+106	1.02	37.03						
-106+75	1.00	36.40						
-75+53	1.01	38.25						

**Table 1.** Nickel and iron contents of each size fraction

Leaching experiments were carried out in a 1 L glass vessel placed in a thermostatically-controlled water bath. For the stirring process, Heidolph brand RZR 2021 model mechanical stirrer equipped with a Teflon-covered propeller was used. For each experiment, 5 g lateritic nickel ore sample with a fraction size of 75x53  $\mu$ m was used in 500 mL HCl+H<sub>2</sub>O<sub>2</sub> solution. Taguchi method was used in the design of leaching experiments. Experimental parameters were applied as stirring speed (0–600 rpm), temperature (40–70 °C), HCl concentration (0.5–3.0 M), H<sub>2</sub>O<sub>2</sub> concentration (0.1–1 M) and leaching time (30–240 min). At the end of leaching time, 5 mL solution was taken and diluted with distilled water to 100 mL in a volumetric flask. The amount of Ni in the leaching solution was determined using a GBC mark SensAA model flame atomic absorption spectrometer (AAS).

#### RESULT AND DISCUSSION

# Taguchi Method and Optimization Studies

For optimizing a process by using Taguchi method, the design of an experiment involves the following stages: selection of independent process parameters; determination of the number of levels for the process parameters; selection of the suitable orthogonal array and assignment of the process parameters; performing the experiments proper to the orthogonal array; calculation of the performance characteristics; analyzing the data using the performance characteristics; determination of the optimum levels of the process parameters; performing the confirmation experiment using the optimum process parameters (Bese *et al.*, 2010; Phadke, 1989; Abali *et al.*, 2006). The change in the quality characteristics in reply to a factor presented in the experimental design is the signal of the requested effect. The noise is

the effect of external factors that were not designed in experiments on the outcome. The signal to noise ratio (S/N) shows the sensibility of the quality characteristic to noise factors (Roy, 1995). Usually, three different equations of S/N ratio were used depending on performance characteristics. These are, lower is better, nominal is best and higher is better (Roy, 1995; Atil and Unver, 2000; Zolfaghari *et al.*, 2011). The aim of this study was to provide maximum metal extraction, "higher is better" which given in Equation (1) was used,

$$S/N = -10\log_{10}\left[\frac{1}{n}\sum_{EoM_i}(\frac{1}{EoM_i})^2\right]$$
(1)

where n is the number of repetitions of the experiments and  $EoM_i$  (the extraction of metal) is the result of the experiment. Experimental factors and their levels were given in Table 2. The orthogonal array (OA) experimental design plan (L<sub>16</sub>) was given in (Table 3).  $EoM_1$  and  $EoM_2$  show the extraction of metal for the first and second leaching test, respectively. As seen from Table 3, the maximum S/N value which obtained from test 10 was represented in bold. To investigate the optimum conditions, the analysis of the means (ANOM) statistical method was used. For that reason, the mean of the S/N ratios calculated and the mean of the S/N ratios of factor I in level i, is given by Equation (2):

Table 2. Experimental factors and their levels

Factor	Description	Level 1	Level 2	Level 3	Level 4
A	Leaching time (min)	30	60	120	240
В	Stirring speed (rpm)	0	200	400	600
C	Temperature (°C)	40	50	60	70
D	Acid concentration (M)	0.5	1	2	3
E	H <sub>2</sub> O <sub>2</sub> concentration (M)	0.1	0.3	0.5	1

Table 3. Experimental results and L<sub>16</sub> (4<sup>5</sup>) plan table

	Factor				E	EoM (%)		
Test	A	В	C	D	E	EoM <sub>1</sub>	EoM <sub>2</sub>	
Test 1	30	0	40	0.5	0.1	24.23	24.80	27.79
Test 2	30	200	50	1	0.3	25.67	26.42	28.31
Test 3	30	400	60	2	0.5	30.93	30.95	29.81
Test 4	30	600	70	3	1	57.04	62.37	35.49
Test 5	60	0	50	2	1	27.96	31.09	29.37
Test 6	60	200	40	3	0.5	26.94	31.21	29.20
Test 7	60	400	70	0.5	0.3	29.23	31.15	29.58
Test 8	60	600	60	1	0.1	30.40	30.93	29.73
Test 9	120	0	60	3	0.3	49.92	51.74	34.12
Test 10	120	200	70	2	0.1	60.97	62.41	35.80
Test 11	120	400	40	1	1	28.58	28.56	29.12
Test 12	120	600	50	0.5	0.5	29.33	29.05	29.30
Test 13	240	0	70	1	0.5	48.38	48.50	33.70
Test 14	240	200	60	0.5	1	33.75	33.91	30.59
Test 15	240	400	50	3	0.1	56.44	57.04	35.08
Test 16	240	600	40	2	0.3	32.75	32.45	30.26

For example,  $(M)_{Factor=I}^{Level=i}$ , the mean of the S/N ratio of factor I in level i, is given by Equation (2):

$$(\mathsf{M})_{\mathsf{Factor}}^{\mathsf{Level}} = \frac{1}{n_{li}} \sum_{j=1}^{n_{li}} \left[ \left( \frac{S}{N} \right)_{\mathsf{Factor}}^{\mathsf{Level}} = i \right]_{j}$$
 (2)

In Equation (2),  $n_{Ii}$  shows the number of views of factor I in the level i.  $\frac{\binom{S}{N}^{\text{Level}=i}}{\text{Factor}=I}$  is the S/N ratio of factor I in level i, and its sequence of view in Table 3 is the jth. Subsequently, the values of the S/N ratio were transferred into Equation (2) and the mean of the S/N ratios of a certain factor in the ith level,  $\binom{(M)^{\text{Level}=i}}{\text{Factor}=I}$ , was obtained (Table 4). As seen from Table 4, the bold values show the maximum of the mean of the S/N ratios. Thus, it shows the optimum conditions for Ni extraction from lateritic ore.

**Table 4.** S/N ratio response table for Ni extraction

	[(S/N	) Level Factor	$(M)_{Factor}^{Level}$		
Factor/Level					
	<i>j</i> =1	j=2	j=3	j=4	
A/1	27.79	28.31	29.81	35.49	30.35
A/2	29.37	29.20	29.58	29.73	29.47
A/3	34.12	35.80	29.12	29.30	32.09
A/4	33.70	30.59	35.08	30.26	32.41
B/1	27.79	29.37	34.12	33.70	31.24
B/2	28.31	29.20	35.80	30.59	30.98
B/3	29.81	29.58	29.12	35.08	30.90
B/4	35.49	29.73	29.30	30.26	31.20
C/1	27.79	29.20	29.12	30.26	29.09
C/2	28.31	29.37	29.30	35.08	30.52
C/3	29.81	29.73	34.12	30.59	31.06
C/4	35.49	29.58	35.80	33.70	33.65
D/1	27.79	29.58	29.30	30.59	29.32
D/2	28.31	29.73	29.12	33.70	30.22
D/3	29.81	29.37	35.80	30.26	31.31
D/4	35.49	29.20	34.12	35.08	33.47
E/1	27.79	29.73	35.80	35.08	32.10
E/2	28.31	29.58	34.12	30.26	30.57
E/3	29.81	29.20	29.30	33.70	30.50
E/4	35.49	29.37	29.12	30.59	31.14

Also, analysis of variance (ANOVA) statistical method was applied to experimental results to investigate the effect of factors on Ni extraction. The percentage contribution of each factor, QF, was given by Equation (3).

$$\rho_{\rm F} = \frac{SS_{\rm F} - (DOF_{\rm F}V_{\rm Er})}{SS_{\rm T}} \times 100 \tag{3}$$

In Equation (3),  $DOF_F$  is produced by subtracting one from the number of levels. The total sum of squares, the factorial sum of squares and the variance of error are symbolized as  $SS_T$ ,  $SS_F$  and  $V_{Er}$ , respectively.

$$SS_{T} = \sum_{j=1}^{m} \left( \sum_{i=1}^{n} EoM_{i}^{2} \right)_{j} - mn(\overline{EoM}_{T})^{2}$$
(4)

In Equation (4), m and n represents the number of experiments and the number of repetitions, respectively.  $\overline{\text{EoM}}_T$  represents the average of total EoM.

$$SS_{F} = \frac{mn}{L} \sum_{k=1}^{L} \left( \overline{\text{EoM}}_{k}^{F} - \overline{\text{EoM}}_{T} \right)^{2}$$
(5)

In Equation (5),  $\overline{\text{EoM}}_{k}^{F}$  is the average value of the experimental results of a certain factor in the kth level. The variance of error,  $V_{\text{Er}}$ , is given by Equation (6):

$$V_{\rm Er} = \frac{SS_{\rm T} - \sum_{F=A}^{E} SS_{\rm F}}{m(n-1)} \tag{6}$$

## **Optimization Conditions**

For optimization of nickel extraction from lateritic ore, 16 tests were performed according to conditions given in Table 2. By taking into account the number of experimental repetitions and results of metal extraction (EoM), the S/N ratios of each test condition were determined (Table 3). In Table 3, the maximum S/N value was represented in bold. Subsequently, the S/N ratio values were transferred into Equation (2) and the mean of the S/N ratios were given in Table 4. (M)<sup>Level</sup> refers to result of the effect of each level of each factor and the maximum values of these show the optimum conditions for nickel extraction. As a result of this, optimum conditions were given in Table 5. As seen from Table 5, an experiment was carried out to compare the results of test 10 and obtained optimum conditions. Consequently, it was determined that the value of the S/N ratio of optimum conditions was greater than the value of the S/N ratio of test 10. Additionally, the average of EoM rises from 61.69% to 90.66%. Each level of each factor and corresponding the mean of the S/N ratio values which obtained from Table 4 were plotted and shown in Figure 2–6.

**Table 5.** The optimum condition for Ni extraction

	Α	В	C	D	E	$EoM_1$	$EoM_2$	S/N
Test 10	120	200	70	2	0.1	60.97	62.41	35.80
Optimization condition	240	0	70	3	0.1	89.60	91.71	39.15

**Table 6.** The average of the experimental results of a certain factor in the kth level and the average of total EoM (EoM<sub>T</sub>) for Ni extraction

	total Low (Lown) for the extraction									
Level	$\overline{\operatorname{EoM}}_K^A$	$\overline{\mathbf{EoM}}_K^{\mathrm{B}}$	$\overline{\mathbf{EoM}}_K^{\mathbf{c}}$	$\overline{\mathbf{EoM}}_K^{\mathrm{D}}$	$\overline{\mathbf{EoM}}_K^{\mathrm{E}}$	$\overline{\text{EoM}}_{\text{T}}$				
Level 1	35.30	38.33	28.69	29.43	43.40	37.66				
Level 2	29.86	37.66	35.38	33.43	34.92					
Level 3	42.57	36.61	36.57	38.69	34.41					
Level 4	42.90	38.04	50.00	49.09	37.91					

### Leaching time

Figure 2 shows the effect of leaching time on the S/N ratio for extraction of nickel from lateritic ore in hydrochloric acid solution with hydrogen peroxide. As seen from Figure 2, it was determined that the optimum leaching time was 240 min. It may be concluded that an increase in time of the presence of ore particles in solution causes to increase the extraction of nickel. Thus, the amount of metal ions dissolved from particle that passes into solution increases with time.

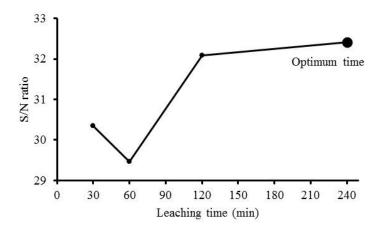


Figure 2. Effect of leaching time on the S/N ratio for extraction of nickel from lateritic ore

## Stirring speed

In Figure 3, effect of stirring speed on the S/N ratio for extraction of nickel from lateritic ore in hydrochloric acid solution with hydrogen peroxide was shown. As seen from Figure 3, it was determined that no significant effect of stirring speed on the extraction of nickel. Thus, the optimum condition was provided by no stirring. Although a kinetic investigation was not performed in this study, it may be considered the negligible effect of stirring speed on extraction of nickel from lateritic ore could stem from dissolution mechanism. Georgiou and Papangelakis (1998) expressed that stirring speed had a negligible effect on the rate of nickel dissolution from limonitic laterite.

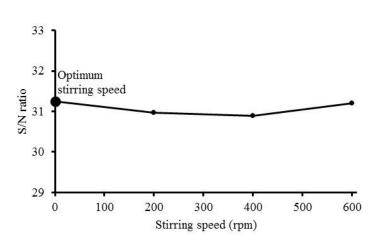


Figure 3. Effect of stirring speed on the S/N ratio for extraction of nickel from lateritic ore

# **Temperature**

The plot of the effect of temperature on the S/N ratio for the extraction of nickel from lateritic ore in hydrochloric acid solution with hydrogen peroxide was shown in Figure 4. As seen from Figure 4, it was determined that the optimum leaching temperature was 70°C. The extraction of nickel increased with an increase in temperature of solution. As known, temperature increases reaction rate and equilibrium constant. However, rapid decomposition of hydrogen peroxide occurs at higher temperatures (Antonijevic *et al.*, 1997; Mahajan *et al.*, 2007).

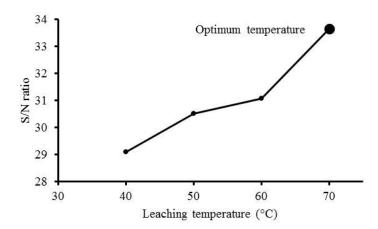


Figure 4. Effect of temperature on the S/N ratio for extraction of nickel from lateritic ore

#### Acid concentration

Figure 5 shows the effect of acid concentration on the S/N ratio for extraction of nickel from lateritic ore in hydrochloric acid solution with hydrogen peroxide. As seen from Figure 5, it was determined that the optimum hydrochloric acid concentration was 3 M. The results presented in Figure 5 show that, S/N ratio values increase as acid concentration increases. Aydogan (2006) and Antonijevic *et al.* (1997) expressed that hydrogen peroxide shows oxidative action in acidic solutions.

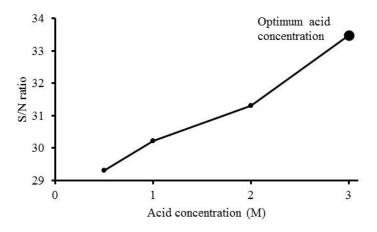


Figure 5. Effect of acid concentration on the S/N ratio for extraction of nickel from lateritic ore

#### H<sub>2</sub>O<sub>2</sub> concentration

The plot of the effect of  $H_2O_2$  concentration on the S/N ratio for extraction of nickel from lateritic ore in hydrochloric acid solution with hydrogen peroxide was shown in Figure 6. As seen from Figure 6, it was determined that the optimum  $H_2O_2$  concentration was 0.1 M. The results presented in Figure 6 show that, S/N ratio values decreases as  $H_2O_2$  concentration increases.

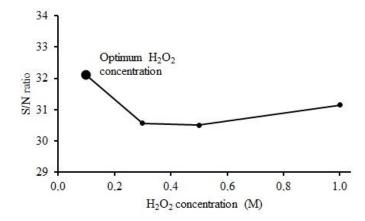


Figure 6. Effect of H<sub>2</sub>O<sub>2</sub> concentration on the S/N ratio for extraction of nickel from lateritic ore

## Percentage of Contribution

In order to determine the percentage of the contribution of each factor, the average values of the experimental results of a certain factor in the kth level ( $\overline{\text{EoM}}_K^F$ ) were obtained using EoMi values in Table 3 and given in Table 6. After this, the factorial sum of squares,  $SS_F$ , for each factor was calculated. The total sum of squares,  $SS_T$ , was determined. After this process, using  $SS_F$  and  $SS_T$  values,  $V_{Er}$  was obtained. Consequently, the percentage contribution of each factor,  $\rho_F$ , was determined. All of the obtained values were given in Table 7. As seen from Table 7, the importance order can be seen as; temperature, acid concentration, leaching time,  $H_2O_2$  concentration and stirring speed.

Factor	$DOF_{\rm F}$	$SS_{\mathrm{F}}$	ρε(%)	SS <sub>T</sub>	$V_{ m Er}$
Leaching time (min)	3	943.80	18.693	5048.95	1.96
Stirring speed (rpm)	3	13.57	0.269		
Temperature (°C)	3	1912.98	37.889		
Acid concentration (M)	3	1738.65	34.436		
H <sub>2</sub> O <sub>2</sub> concentration (M)	3	408.64	8.094		
Error		31.30	0.620		

**Table 7.** Percentage contribution of each factor

#### **CONCLUSION**

The optimum conditions of extraction of nickel from lateritic ore in hydrochloric acid solution with hydrogen peroxide were determined by Taguchi method. The effects of some parameters (leaching time, stirring speed, temperature, hydrochloric acid concentration and hydrogen peroxide concentration) on extraction of nickel were investigated. The optimum conditions determined by Taguchi method were; leaching time of 240 min, a temperature of 70 °C, hydrochloric acid concentration of 3 M, hydrogen peroxide concentration of 0.1 M and without stirring. As a result of experimental results, it was demonstrated that, extraction of nickel was obtained with a recovery of 90.66% in optimum conditions.

In addition, analysis of variance (ANOVA) was applied to experimental results. Percentage contributions of each factor for extraction of nickel were: temperature (37.889%), acid concentration (34.436%), leaching time (18.693%),  $H_2O_2$  concentration (8.094%) and stirring speed (0.269%). In point of these results, the extraction of nickel is most dependent by temperature.

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