

MAGNESIUM EXTRACTION OF FERRONICKEL SLAG PROCESSED BY ALKALI FUSION AND HYDROCHLORIC ACID LEACHING

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Abstract

A research using ferronickel slag, the by-product of ferronickel production, as raw material for magnesium extraction was carried out. It is essential to upgrade the value of ferronickel slag instead of used directly for reclamation materials. Moreover, the accumulation due to increasing ferronickel demand as well as the environmental contamination due to various potentially toxic elements contained in the ferronickel slag could be prevented. The general objective of this study is to utilize the ferronickel slag for magnesium materials. The specific objective is to determine the optimum conditions of magnesium extraction in the process of alkali fusion followed by hydrochloric acid leaching. A novel method for magnesium extraction from ferronickel slag was carried out through reducing silica content followed by acid leaching method. Alkali fusion of the mixture of ferronickel slag and Na_2CO_3 at 1000 °C for 60 minutes, followed by water leaching at 100 °C for 60 minutes with solid to liquid percentage of 20 % was carried out to separate the silica. The leaching residue resulted from water leaching was then leached using hydrochloric acid solution to extract magnesium. The leaching temperature and time as well as the hydrochloric acid concentration were varied in the acid leaching process. Alkali fusion process proved the sodium silicate can be generated and that it can be separated in the water leaching to the leached solution. Meanwhile, the leaching residue was leached using hydrochloric acid to extract the magnesium. The highest magnesium extraction percentage is 82.67% that resulted from an optimum acid leaching condition with temperature of 80 °C for 30 minutes using 2M HCl solution. Based on the kinetics study, the activation energy (E_a) for the leaching reaction of magnesium at atmospheric pressure between 32 °C to 80 °C is 9.44 kJ/mol and affected by diffusion and chemical reactions.

Keywords: Ferronickel; Slag; Alkali-fusion; Leaching; Magnesium; Silica; Extraction

1. Introduction

The production of ferronickel from nickel laterite ore processing produces a by-product called ferronickel slag. It is used directly as a reclamation material for mining access since it increases soil bearing capacity [1, 2], the mixture for concrete industry [3], cement industry [4, 5], construction industry [6], geopolymer industry [7], and fertilizer industry [8]. On the other hand, there are many valuable materials contained in the ferronickel slag that can be extracted and have higher economic value. However, the studies about extraction of valuable materials from ferronickel slag have not been carried out considerably. Therefore, developing an economic technology process that can recycle ferronickel slag to

be valuable material needs to be performed, and thus the accumulation of ferronickel slag as an effect of increasing ferronickel production can be resolved. Moreover, potentially toxic elements contained in the ferronickel slag such as Ni, Cr, Co [9], Cd, Cu, and Zn [10] are not released into the environment due to long accumulation period.

Magnesium and silica are the major elements contained in the ferronickel slag [11]. Hence, by extracting those elements, magnesium and silica products (leached solution or oxide) can be obtained and valuable elements that have small content such as REE (Rare Earth Elements) can be extracted subsequently. Therefore, the enlargement of utilization of ferronickel slag can be carried out.

Prior research studies to increase the value of the

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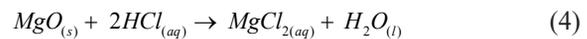
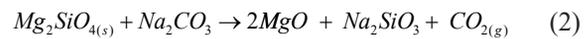
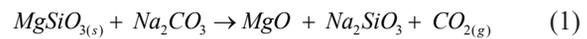


ferronickel slag were carried out using pyrometallurgy process [12-14] hydrometallurgy process [15, 16] as well as combination of both processes [17-19]. Generally, flotation process is a commercially pre-treatment that is carried out prior to pyrometallurgy or hydrometallurgy process to separate or upgrade the specific element. However, it is usually selected for low grade raw sulfide minerals [20-22]. Meanwhile, ferronickel slag is a by-product of smelting process that has complex oxide phases and high content of some elements such as magnesium and silica. Therefore, thermal decomposition is more beneficial than flotation to use in the pre-treatment of ferronickel slag processing. Thermal characterization of ferronickel slag with Na_2CO_3 addition confirmed the formation of sodium silicate at minimal temperature of 900 °C [12]. Sodium silicate formation was also obtained from the reduction of ferronickel slag using NaOH [13] and Na_2SO_4 [14] as additives. However, the utilization of NaOH to form sodium silicate needs high quantity [18, 23] and leads to high base condition which can affect further treatments. Hydrometallurgy process by leaching the ferronickel slag using NaOH solution can produce silicon leached solution. However, leaching the ferronickel slag directly without thermal and decomposition processes could result in low extraction percentage of silicon, about 30 % [15]. On the other hand, the leaching process of ferronickel slag using acid solution resulted in leached solution that contained high Co, Ni, Cu [16], and Mg [24] as well. Further processes such as precipitation and calcination to the magnesium leached solution resulted in magnesium oxide product [17, 25]. However, without reducing silica content prior to acid leaching, it produces a gel leached solution that is difficult to separate.

In this study, magnesium extraction was carried out since magnesium and its derivatives have various application in the industry such as for alloys, liquid iron desulfurization, insulation materials, fertilizers, refractory materials, constructions as well as for medical purposes (drugs and bone implants) [26, 27]. The general objective of this study is to utilize the ferronickel slag, for magnesium materials. The specific objective is to determine the optimum conditions of magnesium extraction in the process of alkali fusion followed by hydrochloric acid leaching.

Due to reaching the objectives of this study, a novel method of magnesium extraction from ferronickel slag was carried out in two stages which consisted of reducing silica content by decomposition through alkali fusion with Na_2CO_3 addition followed by water leaching and upgrading of magnesium content by acid leaching using hydrochloric acid solution. Therefore, silica and magnesium leached solution were produced from this method.

The proposed reaction that occurred in the alkali fusion process of ferronickel slag with the addition of Na_2CO_3 is shown by reactions (1) – (3). Meanwhile reaction (4) is proposed reaction that occurred in the magnesium leaching using hydrochloric acid. Utilization of hydrochloric acid leads to MgCl_2 formation where Cl ion can be eliminated from Mg easily by heat treatment.



2. Materials and Methods

The basic material used was the slag from a by-product of the ferronickel processing in Indonesia. Ferronickel slag was crushed by using a crusher and a disk mill to obtain a 200 mesh of particle size. The initial process was alkali fusion of 200 mesh of ferronickel slag with the addition of sodium carbonate (Na_2CO_3). It was intended to increase the slag porosity and transform silica-bonded phases in the easy separated phase, sodium silicate (Na_2SiO_3). The operation condition of alkali fusion process was based on the previous study [12]. It was carried out in the muffle furnace carbolite at a temperature of 1000 °C for 60 minutes with ratio 1:1 of ferronickel slag and Na_2CO_3 . Water leaching performed to the product resulted from alkali fusion process at temperature of 100 °C for 60 minutes, solid to liquid ratio 20 %, and stirring speed 400 rpm. It was aimed at eliminating sodium silicate that separated in the leached solution. Meanwhile, the leaching residue was leached using hydrochloric acid solution to extract magnesium.

The leaching process was undertaken by varying the leaching temperature and time as well as leaching agent concentration. Those three variables generally have an important role in the upgrading of magnesium contents in the acid leaching process [17]. Leaching process was carried out in the three-neck flask with solid to liquid ratio 1:10 and stirring speed 300 rpm. Leaching temperature and time were varied at ambient temperature up to 80 °C and for 15 – 240 minutes, respectively. Hydrochloric acid concentration was varied from 2 – 8 M. Filtration was then performed to separate the leached solution and leaching residue.

XRF (X-Ray Fluorescence) analysis using XRF Bruker-S2 Puma was performed to the ferronickel slag and the product of alkali fusion process to determine its composition. The phase transformation of ferronickel slag, the product of alkali fusion process, and leaching residue resulting from water leaching process were analysed using XRD (X-Ray



Diffraction) Panalytical Epsilon 1. Moreover, the morphology and the mapping were characterized using SEM (Scanning Electron Microscope)-mapping analysis JEOL Jsm 6390A. Mg content in the leached solutions from acid leaching process as an effect of leaching temperature, leaching time, and acid concentration were determined using ICP-OES Agilent 725 series. In addition, the kinetic study of magnesium extraction in the acid leaching process was investigated.

3. Results and Discussions

3.1. Thermodynamic Studies on Alkali Process

Alkali fusion process of the mixture of ferronickel slag and Na_2CO_3 in the present study was carried out at a temperature of 1000 °C for 60 minutes. The determination of temperature was engaged with ΔG calculation using HSC chemistry 6 software to find out the temperature of the spontaneous reaction start to occur. Due to Mg_2SiO_4 being the dominant phase in the ferronickel slag, it was used as a phase representative that reacted with Na_2CO_3 in the alkali fusion process. The results of the ΔG calculation using HSC chemistry 6 software can be seen in Table 1.

Table 1. Thermodynamic properties of reaction between Mg_2SiO_4 and Na_2CO_3

$\text{Mg}_2\text{SiO}_4 + \text{Na}_2\text{CO}_3 = \text{Na}_2\text{SiO}_3 + 2\text{MgO} + \text{CO}_2(\text{g})$					
T, C	ΔH , kcal	ΔS , cal/K	ΔG , kcal	K	Log (K)
0	35.7716	35.35706	26.11381	1.27E-21	-20.8956
100	35.63969	34.95267	22.59711	5.81E-14	-13.236
200	35.38021	34.34528	19.12974	1.46E-09	-8.83683
300	34.87582	33.3874	15.73984	9.95E-07	-6.00231
400	34.01745	32.01569	12.46609	8.96E-05	-4.04767
500	33.17956	30.83234	9.341534	0.002286	-2.64083
600	32.88732	30.47921	6.274399	0.026877	-1.57061
700	32.39594	29.94843	3.251625	0.186076	-0.73031
800	31.69006	29.25973	0.289975	0.872853	-0.05906
900	23.77103	22.20636	-2.28036	2.659811	0.424851
1000	23.03717	21.60568	-4.47011	5.853362	0.767405

Based on the ΔG calculation using HSC chemistry 6 software (see Table 1), the reaction between Mg_2SiO_4 and Na_2CO_3 can occur starting at a temperature of 900 °C. Since the ΔG has a negative value, that indicates that a spontaneous reaction occurred. Therefore, alkali fusion process at a temperature of 1000 °C was in good agreement with the thermodynamic study. The use of temperature at

1000 °C for 60 minutes was appropriate according to previous studies that recommended the alkali fusion of ferronickel slag with Na_2CO_3 at a temperature above 900 °C [12].

3.2. XRF Analysis

The identification of chemical compositions and contents in the ferronickel slags was analyzed by XRF (X-Ray Fluorescence) with the Bruker-S2 Puma brand. The chemical composition of ferronickel slag, a product of alkali fusion and the residue from water leaching process can be seen in Table 2.

As can be seen from Table 2, the dominant element found in ferronickel slag is silica dioxide (SiO_2) with the content of 45.69%. Other elements contained in the ferronickel slag are magnesium oxide (MgO), iron (III) oxide (Fe_2O_3), and nickel (II) oxide (NiO) with the content of 29.332%, 16.503%, and 0.121%, respectively. The results of elemental contents in the ferronickel slag were not much different from some works from the literature that show an analysis using XRF on ferronickel slag samples [12, 15, 17]. The XRF results after an alkali fusion show that the contents of SiO_2 , MgO, Fe_2O_3 , and NiO decreased due to the addition of sodium carbonate as an additive. Meanwhile, decreasing SiO_2 content in the leaching residue of water leaching process indicated that water leaching process was able to dissolve decomposed SiO_2 .

Table 2. The results of XRF analysis of ferronickel slag

Compound	Content (%)		
	Ferronickel Slag	Result of alkali fusion	The leaching residue of water leaching process
SiO_2	45.7	37.6	30.23
MgO	29.3	23.3	28.29
Fe_2O_3	16.5	13.8	16.40
Al_2O_3	4.81	4.22	4.54
Cr_2O_3	1.40	1.01	0.94
CaO	0.93	0.82	1.03
MnO	0.58	0.47	1.04
Na_2O	0.29	18.3	17.14
TiO_2	0.12	0.12	0.14
NiO	0.12	0.11	0.12
SO_3	0.092	0.051	-
P_2O_5	0.055	0.001	-
V_2O_5	0.039	0.02	0.022
ZnO	0.035	0.035	0.051



ferronickel SEM results. This is also reinforced by the XRD results towards the ferronickel slag fusion process which adds the sodium carbonate additive substance shown in Figure 1 (b). In Figure 1 (b) sodium silicate is formed and the detection of SiO_2 and MgO as a single-compound.

Figure 2 (c) shows the SEM mapping of the residual leach with water. The distribution of silica decreased, shown in a less bright red color, while magnesium and oxygen had an increase in the distribution of reddening colors in one spot area. This result indicates the decreasing content of silica in the

residue of water leaching is in good agreement with the XRF results of the residue of water leaching in Table 2.

3.5. Effect of Leaching Temperature toward Magnesium Extraction Percentage

The leaching process was done by varying the leaching temperatures at room temperature (32°C), 60°C , and 80°C with 300 rpm of stirring speed and a solid/liquid ratio 1:10. The leached solution was analyzed using ICP (Inductively Coupled Plasma)-

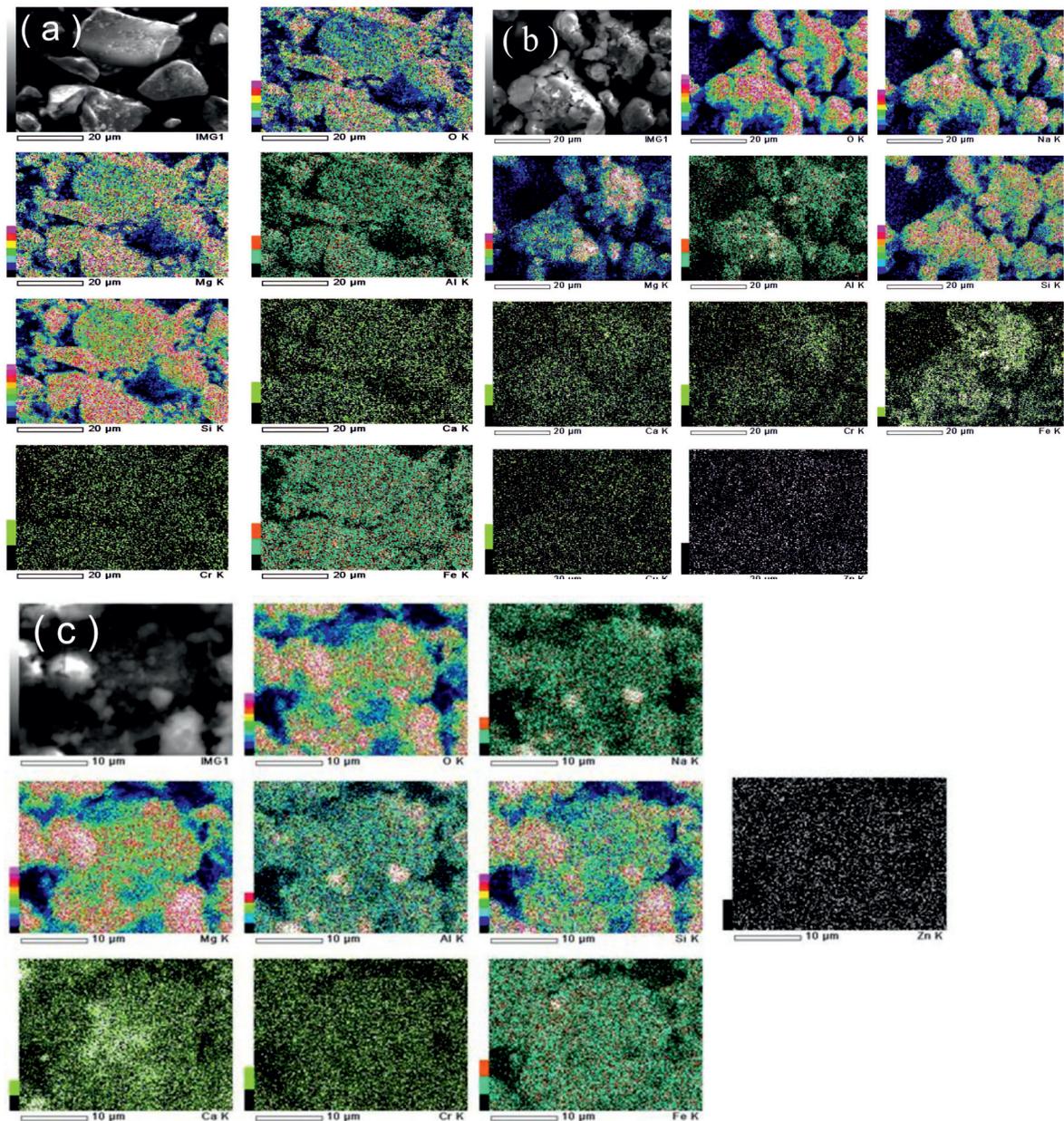


Figure 2. SEM Analysis results towards ferronickel slag samples (a), ferronickel slag alkali fusion results (b) and residue of water leaching process (c)

OES, an Agilent Technologies 5000 brand to determine the soluble magnesium contents.

As shown in Figure 3, the percentage of magnesium extraction increases along with the rise of leaching temperatures. The leaching process with a temperature of 80°C produced the leached solution with the highest magnesium extraction percentage, 82.67%, at thirtieth minute. It is in accordance with the previous research, which explained that the leaching temperature for the leaching of magnesium silicate should be done at 50-105°C [24], since low temperature of the leaching process becomes less effective due to silica gel formation that can reduce magnesium extraction. Moreover, the statement is in accordance with the present research that shows the highest magnesium extraction percentage in the leaching process at a room temperature (< 50 °C) for 240 minutes was only 54.12%. In addition, the research conducted by A. Royani in extracting magnesium from dolomite shows the similar trend, the higher the temperature is, the higher magnesium extraction percentage is obtained. Still, in some ways temperatures above 75°C are considered giving less significant impact [26]. However, the economic factor should be perceived, as the addition of temperature is less economical since it substantially increases the energy and the loss of acid used [26].

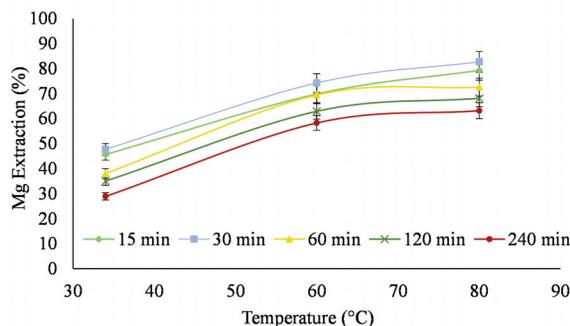


Figure 3. Graphic of temperature effect towards magnesium extraction percentage at various time

3.6. Effect of Solution Concentration towards Magnesium Extraction Percentage

The leaching processes with the variations of hydrochloric acid concentration were carried out at 80°C for 15 – 240 minutes using 2M, 4M, 6M, and 8M of hydrochloric acid. Figure 4 shows that the highest value of magnesium extraction percentage was at 2M of hydrochloric acid concentration, 82.67%, for 30 minutes. Increasing hydrochloric acid concentration to 4M, 6M and 8M, led to decreasing the magnesium extraction percentage to 72.48%, 69.36%, and 62.89% respectively. The higher acid concentration in the leaching process, the more elements would dissolve: it corresponds with the

research by Raschman et al. that the magnesium extraction percentage in the leached solution decreases with the increasing HCl concentration [28].

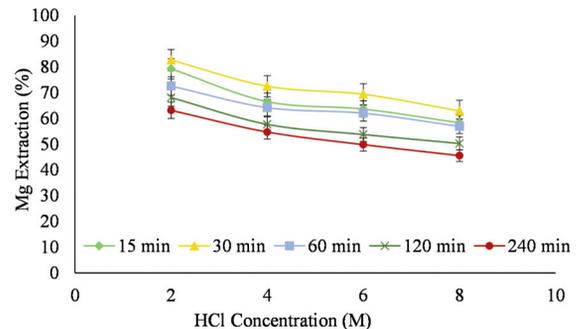


Figure 4. Graphic of concentration-effect towards magnesium extraction percentage at various time

3.7. Effect of Leaching Time towards Magnesium Extraction Percentage

Figure 5 shows that the highest magnesium extraction percentage, 82.67%, was reached at leaching temperature and time of 80 °C and for 30 minutes using 2 M of hydrochloric acid solution. The magnesium extraction percentage tends to decrease for further leaching time, > 30 minutes, at all temperature variations. Extending the times into the process could cause dilution of other components. It is consistent with the research of Ozdemir et al. That shows an increasing magnesium recovery in the leaching process by increasing leaching time to 20 minutes, then the magnesium recovery in the leaching process decreases for further leaching time at all variations of HCl concentration [29].

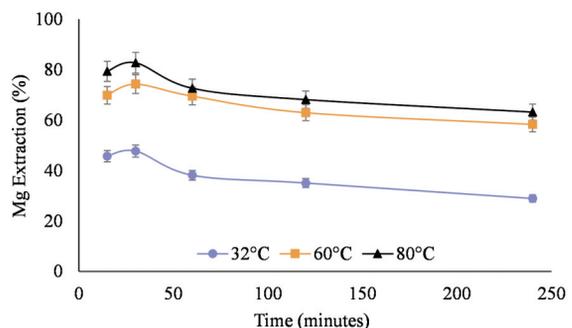


Figure 5. Graphic of leaching time effect towards magnesium extraction percentage at various temperature

3.8. XRF analysis of residue from acid leaching

Chemical composition of the residue of HCl leaching using a concentration of 2M and a leaching temperature of 80 °C was carried out using XRF analysis. It can be seen in the Table 3 that the

remaining contents of magnesium (Mg), iron (Fe), aluminum (Al), chrome (Cr) and nickel (Ni) are 0.42%, 1.49%, 0, 56%, 1.53% and 0.03%, respectively. It shows that the majority of magnesium can be extracted in the dissolved solution and have leftover silica in the residue.

Table 3. The Result of the residue of acid leaching process using XRF analysis

Compound	Residue of acid leaching process
SiO ₂	92.46
MgO	0.42
Fe ₂ O ₃	1.49
Al ₂ O ₃	0.56
Cr ₂ O ₃	1.53
NiO	0.03
MnO	0.09
Na ₂ O	0.10
TiO ₂	0.16
SO ₃	0.01
V ₂ O ₅	0.02

3.9. Kinetics Studies of Leaching

Kinetics Leaching can be learned using the kinetics Shrinking-core approach model [30-33] In this kinetics model, the speed of the reaction, whether it is controlled by chemical reactions on the surface or by diffusion through the liquid boundary layer, is determined quantitatively based on the suitability between the elements fraction data that reacts at each time using the following equations (6) and (7) [30-33]:

$$Kc = 1 - (1 - \alpha)^{(1/3)} = k_c \tag{6}$$

$$Kd = 1 - \frac{2}{3}\alpha - (1 - \alpha)^{2/3} = k_d t \tag{7}$$

α is the fraction of the reacting elements, t is the leaching time equation (6) assumes that the leaching speed is controlled by chemical reactions occur on the surface of mineral particles with the reaction-rate constant k_c , while equation (7) assumes that the leaching rate reaction control is the diffusion on the surface of mineral particles with the reaction-rate constant k_d . Furthermore, the activation energy is determined based on the Arrhenius equation:

$$\ln k = \ln A - \frac{Ea}{R} \left(\frac{1}{T} \right) \tag{8}$$

Where, A = pre-exponential factor, R = universal

gas constant (8.314 J/(mol K)), T = absolute temperature (K) and Ea = activation energy (kJ mol) [34, 35].

The data plot between α and t using equations (6) and (7) for the leaching reaction of Mg element from nickel slags with 2M of HCl solution concentration can be seen in Figure 6 (a) and (b).

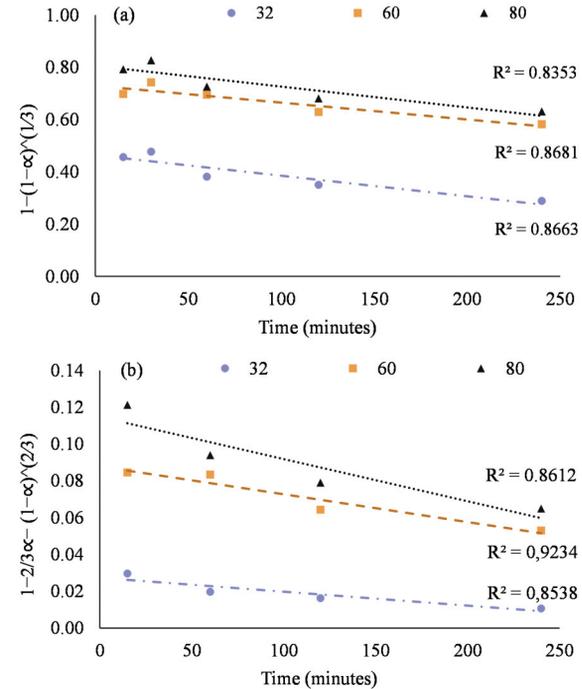


Figure 6. The a plot towards time on the leaching process (a) chemical reactions (b) diffusion on the surface of mineral particles

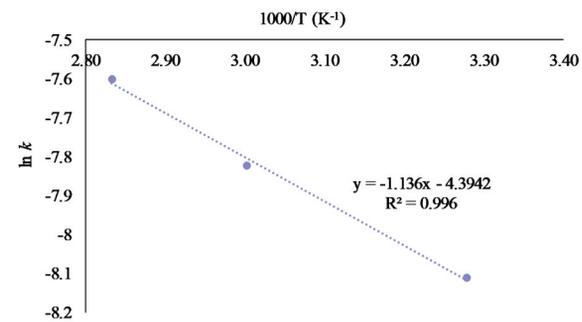


Figure 7. Arrhenius plot on Mg dissolution

The optimum linear regression is obtained in the data plot using equation (6) as shown in Figure 6 (a). The Arrhenius plot according to the equation (8) is based on the rate constants determined from the slope of the linear regression in Figure 6 (a). From the straight-line slope in Figure 7, the activation energy (Ea) can be determined for the leaching reaction of Mg element at atmospheric pressure between 32°C to



80°C is 9.44 kJ/mol. Based on the extraction percentage data in Figure 3 and the kinetics data in Figure 6, the reaction rates increase significantly at temperatures of 32°C to 60°C, so the average extraction percentage increases by $\pm 30\%$ for each reaction time. Subsequent temperature increases up to 80°C, only give an extraction percentage of $\pm 6\%$ on average. Furthermore, the concentration of hydrochloric acid solution also did not have a significant effect on the Mg extraction percentage as shown in Figure 4. This condition indicates that the Mg leaching process from nickel slag is affected by diffusion and chemical reactions.

4. Conclusion

Magnesium extraction of ferronickel slag processed by alkali fusion and hydrochloric acid leaching was carried out. It was proved that ferronickel slag can be used as magnesium materials in two-steps, reducing silica content and upgrading magnesium content. Sodium silicate was confirmed to be formed due to alkali fusion of ferronickel slag with sodium carbonate addition at temperature of 1000 °C for 60 minutes. It was then separated by water leaching. XRD analysis of the leaching residue resulted from water leaching process showed the disappearance of sodium silicate phase that beneficiate magnesium extraction in the acid leaching process. An optimum condition of magnesium extraction in the hydrochloric acid leaching was reached at a temperature of 80 °C for 30 minutes using 2M of hydrochloric acid solution with stirring speed 300 rpm and solid to liquid ratio of 1:10. The reaction was affected by diffusion and chemical reactions, with the activation energy (E_a) of 9.44 kJ/mol. The highest magnesium extraction obtained was 82.67 %.

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IZDVAJANJE MAGNEZIJUMA IZ ŠLJAKE FERONIKLA ALKALNIM TOPLJENJEM I LUŽENJEM HLOROVODONIČNOM KISELINOM

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Apstrakt

Izvršeno je istraživanje u kojem je kao sirovina za ekstrakciju magnezijuma korišćena šljaka feronikla, nusprodukt proizvodnje feronikla. Od izuzetne je važnosti da se vrednost šljake feronikla poboljša umesto da se direktno koristi kao materijal za meliorizaciju. Štaviše, moguće je sprečiti akumulaciju nastalu zbog sve veće potražnje za feronikom, kao i zagađenje okoline usled različitih potencijalno toksičnih elemenata koji se nalaze u šljaci feronikla. Opšti cilj ovog istraživanja je da se šljaka feronikla upotrebi za materijale magnezijuma. Poseban cilj je da se odrede optimalni uslovi ekstrakcije magnezijuma u procesu alkalnog topljenja posle kojeg sledi luženje hlorovodoničnom kiselinom. Urađen je novi metod ekstrakcije magnezijuma iz šljake feronikla smanjenjem sadržaja silicijum oksida posle čega je usledio metod luženja kiselinom. Da bi se odvojio silicijum oksid, posle alkalnog topljenja mešavine šljake feronikla i Na_2CO_3 pri temperaturi od 1000 °C u trajanju od 60 minuta usledilo je luženje vodom pri temperaturi od 100 °C u trajanju od 60 minuta gde je procenat čvrstog prema tečnom bio 20%. Ostatak luženja koji je dobijen luženjem vodom je nakon toga lužen rastvorom hlorovodonične kiseline da bi se izdvojio magnezijum. U procesu luženja kiselinom menjani su kako temperatura i vreme luženja, tako i koncentracija hlorovodonične kiseline. Proces alkalnog topljenja dokazao je da se može generisati natrijum silikat i da se može odvojiti tokom luženja vodom do rastvora za luženje. U međuvremenu, ostatak luženja je lužen uz pomoć hlorovodonične kiseline da bi se izdvojio magnezijum. Najveći procenat ekstrakcije magnezijuma je 82.67% i rezultat je optimalnih uslova luženja kiselinom pri temperaturi od 80 °C i u trajanju od 30 minuta, korišćenjem 2M HCl rastvora. Na osnovu kinetičkih ispitivanja, aktivaciona energija (E_a) za reakciju luženja magnezijuma pri atmosferskom pritisku od 32 °C do 80 °C je 9.44 kJ/mol i na nju utiču difuzija i hemijske reakcije.

Ključne reči: Feronikl; Šljaka; Alkalna topljenje; Luženje; Magnezijum; Silicijum oksid; Ekstrakcija

