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THE EFFECT OF ALKALI ROASTING OF FERRONICKEL SLAG PRIOR TO THE LEACHING AND PRECIPITATION PROCESS

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Abstrak

Terak feronikel dihasilkan sebagai produk samping dari produksi feronikel. Terak feronikel mempunyai potensi untuk dimanfaatkan sebagai bahan baku beberapa komponen berharga karena komposisinya melalui proses bertahap. Tujuan dari penelitian ini adalah untuk mengetahui pengaruh dari proses pemanggangan campuran terak feronikel dan Na₂CO₃ sebelum pelindian dengan air panas dan presipitasi untuk menghasilkan endapan silika. Proses pemanggangan terak feronikel dengan penambahan Na₂CO₃ telah dilakukan untuk pembentukan natrium silikat. Kemudian dilarutkan melalui proses pelindian menggunakan air panas 90 °C selama 120 menit. Endapan silika didapatkan dengan proses presipitasi sodium silikat terlarut diikuti dengan pemeraman selama tiga hari. Berdasarkan hasilnya, pemanggangan menyebabkan perubahan komposisi yang mempengaruhi persen pelindian dari RAF (*roasted of alkalinized ferronickel slag*). Natrium silikat dalam bentuk Na₄SiO₄ diketahui terlarut pada tahap pelindian dengan air panas. Pengendapan dan pemeraman larutan natrium silikat telah menghasilkan endapan silika dengan ukuran partikel lebih dari 100 μ m. Proses pemanggangan pada 1000 °C selama 240 menit menghasilkan perulehan silika tertinggi.

Kata Kunci: Feronikel, terak, pemanggangan, pelindian, pengendapan, silika

Abstract

As a by-product of ferronickel production, ferronickel slag was created. Because of its composition, it has the potential to be used as a raw material for some valuable elements through a series of processing. The purpose of this research is to determine the effect of roasting ferronickel slag and Na₂CO₃ prior to hot water leaching and precipitation to obtain silica precipitate. To produce sodium silicate, ferronickel slag was roasted with Na₂CO₃ addition. It was then dissolved by leaching for 120 minutes in hot water at around 90 °C. Silica precipitate is made by precipitating dissolved sodium silicate and aging it for three days. According to the findings, roasting causes a change in composition, which influences the leaching percentage and silica recovery. Size reduction of leaching residue from its RAF (roasted alkalinized ferronickel slag) demonstrated that reactions occurred from the surface to the core. When water is leached, sodium silicate in the form of Na₄SiO₄ is observed and dissolved. The precipitation and aging of sodium silicate solution produced silica precipitate with particle sizes greater than 100 µm. The highest silica recovery is obtained by roasting at 1000 °C for 240 minutes.

Keywords: Ferronickel, slag, roasting, leaching, precipitation, silica

1. INTRODUCTION

The smelting process of lateritic nickel ore produces ferronickel or nickel matte as the main product and ferronickel slag as a by-product. Ferronickel has been used mostly as material for stainless steel. Meanwhile, nickel matte is commonly used for producing pure nickel as well as nickel sulfate for cathode materials in battery which has been developed recently. However, mostly ferronickel slag is just used as reclamation materials [1] without further processing,

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© 2021 Metalurgi. This is an open access article under the CC BY-NC-SA license (<u>https://creativecommons.org/licenses/by-nc-sa/4.0/</u>) Metalurgi is Sinta 2 Journal (<u>https://sinta.ristekbrin.go.id/journals/detail?id=3708</u>) accredited by Ministry of Research & Technology, Republic Indonesia therefore it does not own the added value. Ferronickel slag contains various valuable elements such as Al, Cr, Fe, Mg, Si [2]-[4] as well as rare earth elements [5] that potentially recover. However, presently research for ferronickel slag mostly tends to make it as construction materials [6]–[10] due to its properties. The methodology to recover its valuable elements is still rare and challenging, therefore study about the recovery of valuable materials from ferronickel slag is essential to be carried out.

Fang et. al., [11] tried to recycle Si and Al from ferronickel slag with sodium hydroxide addition through alkali roasting and water leaching processes. The extraction percentage of Si increases as roasting temperature and time increase. About 80% Si can be recovered from the water leaching process of roasted mixture of ferronickel slag and sodium hydroxide at 550 °C. Gu et. al., [12] obtained 92.33% of chromium from the selective recovery of ferronickel slag with Na₂O₂ addition. The optimum result is obtained from roasting at 600 °C for one hour with the ratio of ferronickel slag and Na_2O_2 is 1:1, leaching temperature of 50 °C for one hour, and liquid/ solid ratio 10 ml/g. Prasetyo et. al., [5] carried out roasting processes to the mixture of ferronickel slag and Na₂CO₃. Sodium silicate is observed in the XRD (x-ray diffraction) analysis of the roasted products. Moreover, magnesium was also recovered by the calcination process followed by leaching using NaOH solution to dissolve silica and recover magnesium, in which 73.10% of magnesium can be obtained from this process [13].

In the previous study [14], alkali fusion of ferronickel slag using Na₂CO₃ through the roasting process was carried out. Weight loss percentage due to the roasting process, reaction mechanism proposed as well as quantitative analysis of Al, Fe, and Mg in the roasted ferronickel slag are discussed. In this study, hot water leaching of RAF and precipitation were carried out. Na₂CO₃ was added as an additive to lower the melting point of the mixture [5],[14] since the melting point of ferronickel slag is high, at about 1400 °C, depends on the compositions [2]. Moreover, it provides better separation of Ca to the leaching residue instead of to the leachate due to CaCO₃ formation [15]. Meanwhile, the sodium ion is expected to react with SiO₂ to form sodium silicate which is dissolved in the water when the leaching process is carried out, and then it can be separated and precipitated by acid to produce silica precipitate. This study aims to determine the effect of roasting processing of

ferronickel slag with Na₂CO₃ addition prior to hot water leaching and precipitation to obtain silica precipitate.

2. MATERIALS AND METHODS

Ferronickel slag used is from a smelting plant at Morowali, Centre of Sulawesi, Indonesia. Size reduction was conducted to the ferronickel slag to obtain 0.15 mm fine grain. It was then physically mixed with the analytical grade of sodium carbonate (Na₂CO₃) from Merck with a 50: 50 by weight. Roasting processes at specific temperatures and times were conducted to the weighted ferronickel slag and Na₂CO₃ mixture in the CWF 1300 muffle furnace to produce RAF. Roasting temperature and time used for this study are 800 - 1000 °C and for 60, 120, and 240 minutes. Roasted slag resulted from the roasting process at a temperature of 800, 900, and 1000 °C is then referred to as RAF 800, RAF 900, and RAF 1000 respectively.

Leaching processes using hot water accompanied by heating at about 90 °C and stirring, were carried out to 10 g of the RAF and 100 ml hot water for 120 minutes in the beaker glass which was heated in a hot plate. The process was then continued with filtration to separate leachate and leaching residue. The mass differences of RAF weight for leaching and residue leaching were used to calculate the leaching percentage. The leaching residues were dried in the oven at 110 °C for characterization. Meanwhile, silica precipitation using 20 ml of HCl was performed on the leachates. HCl has added drops-wisely together to obtain a homogeneous mixture, which was then aged for three days for precipitations subsequently. The precipitates formed were then filtered to separate it with the solution and dried in the oven at 110 °C for 6 hours. The precipitates resulted were then weighted to calculate the recovery and characterize.

To determine the effect of roasting processes before the leaching, SEM-EDS (scanning electron microscope-energy dispersive spectroscopy) analysis was performed on the raw ferronickel slag, RAF, residue leaching, and silica precipitate to determine the microstructure of ferronickel slag and its transformations due to roasting and leaching processes. Furthermore, XRD (x-ray diffraction) analysis was performed to determined phases transformations of roasted product and leaching residue. Figure 1 shows the process flow diagram of this research.

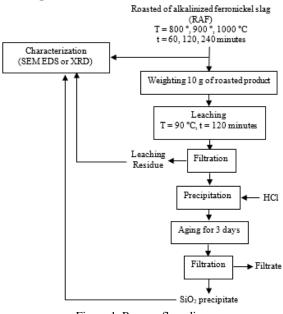


Figure 1. Process flow diagram

3. RESULTS AND DISCUSSIONS

A characterization of the raw ferronickel slag was carried out by SEM-EDS (scanning electron microscope-energy dispersive analysis spectroscopy) to determine the morphology and the compositions. Figure 2 shows the morphology of the ferronickel slag and its EDS analysis in the captured area summarized in Table 1. The morphology is rough and has various particle sizes, while the EDS shows that Si and O are the main components followed by Mg and Fe.

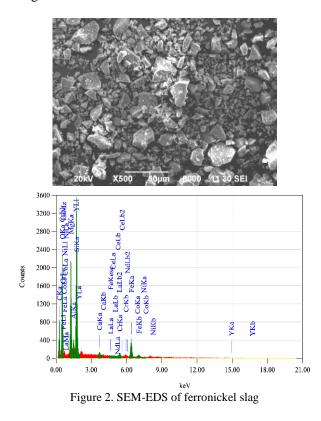


Table 1. Compositions of ferronickel slag

Element	Wt.%
0	45.96
Mg	14.59
Al	3.08
Si	20.84
Ca	1.60
Cr	1.60
Fe	11.84
Co	0.39
Ni	0.058
Cu	0.97

The leaching process was conducted at 90 °C for 120 minutes to the RAF which was previously roasted at various temperatures and times. The leaching percentage was then calculated to determine the ratio of dissolved materials to the initial mass of the roasted slag which is used in the leaching process. The effect of alkali roasting prior to the leaching process toward the leaching percentage, microstructure transformations, and its compositions as well as phase transformations is discussed here.

3.1 The Effect of Alkali Roasting Prior to Leaching Process toward Leaching Percentage

The leaching processes were carried out to the RAF 800, RAF 900, and RAF 1000 which were roasted previously for 60-240 minutes by using hot water. The leaching was performed at a temperature of 90 °C for 120 minutes with a solid/liquid ratio of 1 g/ 10 ml.

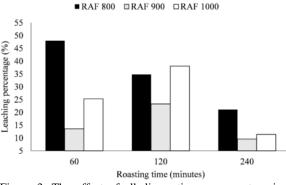


Figure 3. The effect of alkali roasting process at various temperature and time prior to leaching process toward leaching percentage

Figure 3 shows the effect of the alkali roasting process at various temperatures and times before the leaching process toward the leaching percentage. The higher leaching percentage means more RAF dissolved in the hot water during leaching. The trend of leaching percentage resulted from the leaching process of RAF 800 is different, the longer leaching time, the lower leaching percentage resulted. Meanwhile, RAF 900 and RAF 1000 have a similar tendency, showing the highest leaching percentage at 120 minutes.

The melting point of Na₂CO₃ is 851 °C, roasting at a temperature of 800 °C might be affecting the lower decomposition of the mixture of ferronickel slag and Na₂CO₃ in the roasting process due to solid-solid reactions. Moreover, most of Na₂CO₃ remains in the initial form, the previous study shows that the mapping of the roasted product of the mixture of ferronickel slag and Na₂CO₃ at a similar process condition demonstrating sodium distributes on the specific area [5]. The presence of Na_2CO_3 in the initial form contributes to the rise in the leaching percentage of the RAF due to the initial characteristic of Na₂CO₃ that easily dissolves in the water, it is not caused by a good decomposition that resulted from the desired compositions. Furthermore, as the contact of the mixtures was longer by increasing leaching time, the reaction might be better, resulting product that is more difficult to dissolved in the hot water such as sodium magnesium silicate as illustrates in Fig. 5. Therefore, the leaching percentage was higher in the first 60 minutes and constantly decrease as longer leaching time was performed.

When the roasting process was carried out at the temperature 900 and 1000 °C, higher than the melting point of Na_2CO_3 , the solid-liquid reactions might have occurred and better decomposition has resulted. Based on the previous study, the sharp peak of an endothermic reaction is observed on the TG-DTA analysis of ferronickel slag and Na_2CO_3 at 900 °C [5]. RAF 900 and RAF 1000 have a similar tendency, showing the highest leaching percentage at 120 minutes. RAF at 900 °C has a lower leaching percentage, about 15%, than the leaching percentage of RAF 1000. It is in good agreement with the previous study that the leaching percentage increases as the roasting temperature rises. Moreover, the leaching percentage decreased as roasting time escalates more than 3 hours [12].

3.2 The Effect of Alkali Roasting Prior to Leaching Process toward Microstructure Transformation and Composition

In order to determine the effect of alkali roasting prior to the leaching process toward microstructure transformation and its composition, SEM-EDS was performed to the RAF 800, RAF 900, RAF 1000 (resulted from the roasting process for 60 minutes) and its leaching residue. Figure 4 shows the SEM analysis of the roasted products (RAF 800, RAF 900, RAF 1000) and their leaching residue. The differences between the three are obvious. RAF 800, RAF 900, RAF 1000 are shown in Figs. 4(a), 4(c), 4(e) have the greater particle size, white and porous microstructure on the surface. Moreover, Figs. 4(b), 4(d), 4(f) show the microstructure of leaching residue. It can be seen that the particle size is reduced to the smaller size, the white and porous sharp is also reduced, the dark and compact form leftover, which indicated some elements or compounds dissolved into hot water even though just on the surfaces, therefore affect the microstructure itself. Figures 4(a) and 4(b) change slightly. Figures 4(c) and 4(d) show the greater particle size. It is caused by elements content escalation (Table 2) at these process conditions, reducing the solubility to the hot water [16]. On the other hand, Fig. 4(e) shows the greatest melting component than the others and the greatest dissolved components into the hot water. It can be shown by the greater differences in particle size in Fig. 4(f) compared to the others.

Elements	RAF 800	Leaching residue RAF 800	RAF 900	Leaching residue RAF 900	RAF 1000	Leaching residue RAF 1000
0	32.30	28.79	51.18	32.29	41.96	30.20
Na	8.08	7.87	21.92	11.75	20.04	16.04
Mg	18.41	18.36	9.04	13.38	7.71	13.61
Al	3.47	2.63	2.15	2.95	1.63	2.63
Si	21.00	15.11	13.00	15.31	12.37	19.34
Ca	0.87	1.27	-	-	-	-
Cr	1.20	1.95	2.12	3.91	1.67	1.19
Fe	13.19	18.69	13.78	16.43	10.77	12.21
Со	-	-	0.39	-	0.13	-
Ni	-	-	0.12	-	-	-
Cu	-	2.77	3.98	2.44	-	2.23
Zn	1.49	1.56	1.65	1.56	2.38	1.37
Mn	-	0.99	-	-	1.33	1.18

Table 2. Composition of RAF (roasted alkalinized ferronickel) and leaching residue

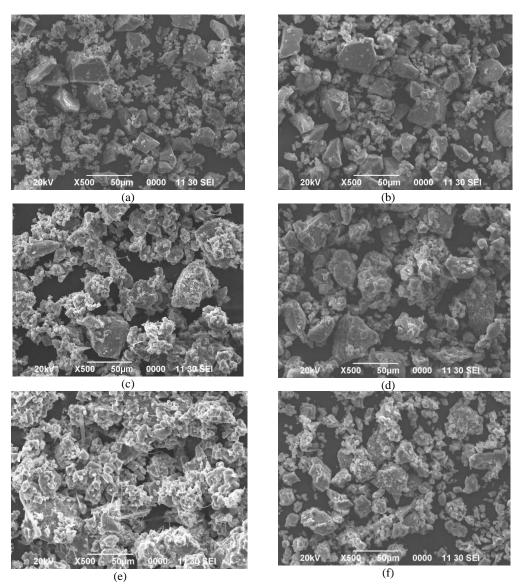


Figure 3. SEM analysis of (a) RAF 800 (b) leaching residue 800 (c) RAF 900 (d) leaching residue 900 (e) RAF 1000 (f) leaching residue 1000

As the EDS analysis of RAF 800, RAF 900, and RAF 1000 which were roasted for 60 minutes, and their leaching residue is shown in Table 2.

RAF 800 was slightly changed in the content compared of the element to the raw ferronickel slag. It might be caused by reactions that occurred to the spinel phases that bearing Al and Mg [14]. Moreover, Fe as Fe_2SiO_4 from olivine also reacted with Na_2CO_3 at about 610 °C [14]. Therefore, the content of Al, Mg, and Fe slightly increase in the RAF 800 compare with raw ferronickel slag.

The leaching process causes the content of O, Na, Al, and Si to slightly decrease. It shows that these elements dissolved in the hot water and increasing some elements content that remained in the leaching residue such as Ca, Cr, and Fe. Moreover, some elements such as Cu, Zn, and Mn can also be observed.

RAF 900 shows the greatest change of elements content. The previous study also shows the greatest changes in the content of the element Al, Fe, and Mg with similar process conditions [14]. Liquid-solid reactions occurred since the process was performed at a temperature higher than the melting temperature of Na₂CO₃. The higher change of O content shows that RAF 900 is covered by oxide. Moreover, Ni and Co just can be observed on the RAF 900. Increasing elements content of Fe, Co, Cu, Cr, and Ni are in line with increasing O content. It indicates that these elements are in the form of oxide. However, increasing elements content might be causing the lower leaching percentage since more components are not dissolved in hot water. It can be proved by increasing elements content of Al,

Cr, Fe, and Mg in the leaching residue compared to RAF 900 or raw ferronickel slag. Decreasing O, Na, and Si elements in the leaching residue indicated that these elements dissolved in the hot water thus some elements contained in the leaching residue increase.

The element's content of RAF 1000 has a similar trend with RAF 900. The element content of Al, Fe, Mg, and Si in the leaching residue was higher than its RAF. However, the content of Cr, Na, and O in the leaching residue was decreased. It indicates a part of the dissolution of these components in the leachate. Increasing roasting temperature rises reaction rat, the content of Na in the leaching residue of RAF 1000 shows the highest content than RAF 800 and RAF 900, it indicates that more ferronickel slag reacts with Na₂CO₃.

3.3 Phase Transformation of RAF and Leaching Residue

XRD (x-ray diffraction) analysis was used to determine the phase transformation of the roasted product and the leaching residue. It carried out to the roasted product that was roasted at 1000 °C (RAF 1000) for 60 minutes and its leaching residue.

Figure 5(a) shows the XRD analysis of RAF 1000 °C. It shows olivine that is an original phase of ferronickel slag [12],[14] reacts with Na₂CO₃ forming Na₂(MgSiO₄), Mg₂SiO₄, NaAlSiO₄, Na₄SiO₄, MgO, and Fe₃O₄. The proposed reactions are as follow [12], [14]:

$2[(Mg \cdot Fe)O \cdot SiO_2] + 3/2 O_2 \rightarrow Mg_2SiO_4 + SiO_2 + Fe_2O_3$	(1)
$Mg_2SiO_4 + Na_2CO_3 \rightarrow 2MgO + Na_2SiO_3 + CO_{2(g)}$	(2)
$Fe_2SiO_4 + Na_2CO_3 + \frac{1}{2}O_2 \rightarrow Na_4SiO_4 + Fe_2O_3 + CO_{2(g)}$	(3)
$Fe_2SiO_4 + Na_2CO_3 \rightarrow Na_2SiO_3 + Fe_2O_3 + CO_{(g)}$	(4)
$MgO + SiO_2 + Na_2CO_3 \rightarrow Na_2MgSiO_4$	(5)
$MgAl_2O_4 + Na_2CO_3 \rightarrow MgO + 2NaAlO_2 + CO_{2(g)}$	(6)
$NaAlO_2 + SiO_2 \rightarrow NaAlSiO_4$	(7)

Figure 5(b) shows the XRD analysis of the leaching residue from the leaching process at about 90 °C for 120 minutes of RAF 1000. It shows that Na₄SiO₄ was separated in the leachate since it is not presented on the XRD graph, Figure 5 (b), and leftover Na₂(MgSiO₄), Mg₂SiO₄, NaAlSiO₄, MgO and Fe₃O₄. The formation of Na₂(MgSiO₄) is similar to the reaction between ferronickel slag and Na₂O₂ [12]. The presence of Fe₃O₄ instead of Fe₂O₃ in the XRD graph might be caused a reduction of Fe₂O₃ by CO resulted from the reactions that occurred. Moreover, the presence of sodium aluminosilicate could prohibit the dissolution of silicon in the leaching process [17], resulted a lower leaching percentage of silicon.

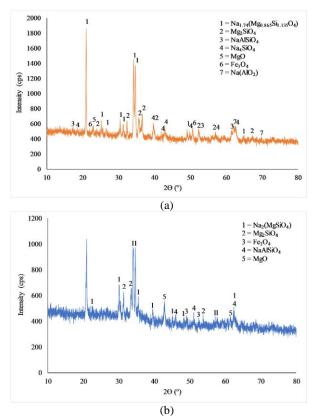


Figure 5. XRD analysis (a) roasted product at 1000 °C for 60 minutes and (b) the leaching residue

The semi-quantitative phases formed is tabulated in Table 3. The phases are dominated by $Na_2(MgSiO_4)$ and Mg_2SiO_4 . It shows that SiO_2 does not liberate overall from olivine. The reaction might occur just at the surface since Mg_2SiO_4 is still presented. Moreover, the microstructure transformation of the RAF and its leaching residue show the surface differences.

Table 3. Phase compositions of roasted product and leaching residue

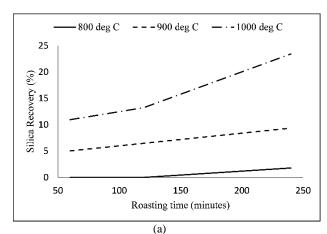
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Phases	Roasted product	Leaching residue
	(%)	(%)
Na ₂ MgSiO ₄	43.8	54.7
Mg ₂ SiO ₄	26.7	14.7
NaAlSiO ₄	12.1	9.9
Na ₄ SiO ₄	12.9	-
Fe ₃ O ₄	2.7	7.3
MgO	1.7	13.3

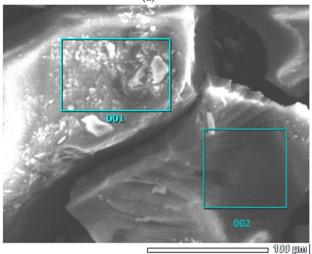
3.4 Precipitation Process of the Leachate

Precipitations were conducted to the leachate that produced from leaching processes of RAF 800, RAF 900, and RAF 1000. The reaction mechanism for leaching and precipitation are shown on reaction (8) - (10).

$SiO_{2(s)} + 2Na_2CO_{3(s)} \rightarrow Na_4SiO_{4(s)} + 2CO_{2(g)}$	(8)
$Na_4SiO_{4(s)} + H_2O_{(l)} \rightarrow Na_4SiO_{4(aq)}$	(9)
$Na_4SiO_{4(aq)} + 4HCl_{(aq)} \rightarrow 4NaCl_{(aq)} + SiO_{2(s)} + 2H_2O_{(1)}$	(10)

Figure 6(a) shows the precipitate recovered from a series of roasting, leaching, and precipitation processes. It shows that the higher the roasting temperature and time, the higher recovered. Leaching silica followed by precipitation processes of roasted alkalinized ferronickel slag (RAF) with roasting temperature and time of 1000 °C and 240 minutes can recover precipitate up to 23.43 %. The low recovery of silica indicates the optimization of a series of processes needs to be carried out further. Decomposition of silica from olivine bonding to be sodium silicate must be optimum, therefore a high leaching percentage especially for Si can be obtained for the precipitation process further.





(b)

Figure 6. (a) Recovery of the precipitate (b) SEM analysis of the precipitate

Characterization of precipitate recovered is shown in Fig. 6(b) and its compositions in Table 4. SEM-EDS analysis shows the dark and compact particle with a particle size is more than 100 μ m. The area 001 and 002 of EDS analysis show that the precipitate is silica. It shows that extraction of silica from ferronickel slag can be an option to provide added value for side products of nickel smelting.

	Table 4.	Compositions	of silica	precipitate
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Compositions	Area 001	Area 002
Si	39.83	46.38
0	60.17	53.62
Total	100	100

4. CONCLUSIONS

The roasting process affects the content of the element of RAF and subsequently affects the leaching percentage of RAF. Roasting at 800 °C causes solid-solid reactions that generate low decomposition. Roasting at 900 °C causes massively decompositions, generating the lowest leaching percentage due to increasing some undissolved elements. Increasing roasting temperature to 1000 °C gains a higher leaching percentage. The higher the roasting temperature, the deeper reactions occurred, resulting from the smaller particle size of leaching residue. The reaction starts from the surfaces to the core. Sodium silicate (Na₄SiO₄) is formed as an effect of the roasting process and separated in the leachate when leaching using hot water performed. However. the presence of Na₂MgSiO₄, Mg₂SiO₄, and NaAlSiO₄ prevent the further dissolution of silica in the leaching process and cause low leaching percentage that trigger low recovery of the silica precipitate. Alkali roasting prior to the leaching process affects the recovery of silica precipitate, the higher the roasting temperature and the longer the roasting time, the more silica precipitate recovered. The particle size of silica precipitate is more than 100 µm.

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