

Ferronickel slag is a by-product of the nickel smelting process. Recycling of ferronickel slag is required since it contains valuable elements besides its potency to pollute the environment. In order to take advantage of the valuable materials and reducing the potential hazard, beneficiation of ferronickel slag is essential. Alkali fusion of ferronickel slag using Na_2CO_3 in the roasting process was carried out. This study aims to determine the decomposition of the mixture of ferronickel slag- Na_2CO_3 in the roasting process. Roasting temperature and time were 800–1,000 °C and 60–240 minutes, respectively. Characterizations of the ferronickel slag were conducted by XRF, ICP-OES, XRD and SEM-EDS. Meanwhile, roasted products were characterized using ICP-OES, XRD and SEM-EDS. Characterization of the ferronickel slag indicates that Mg and Si are the main elements followed by Fe, Al and Cr. Moreover, olivine is detected as the main phase. The roasting process caused percent weight loss of the roasted products, which indicates decomposition occurred and affected the elements content, phases and morphology. The roasting process at about 900 °C for 60 minutes is a preferable decomposition base on the process conditions applied and the change of elements content. Aluminum (Al) and chromium (Cr) content in the roasted products upgraded significantly compared to iron (Fe) and magnesium (Mg) content. Olivine phase transforms to some phases, which were bounded with the sodium compound such as $\text{Na}_2\text{MgSiO}_4$, Na_4SiO_4 and Na_2CrO_4 . The rough layer is observed on the surface of the roasted product as a result of the decomposition process. It indicates that liquid-solid mass transfer is initiated from the surface

Keywords: ferronickel, slag, alkali fusion, roasting, thermal, decomposition, Na_2CO_3 , olivine, aluminum, chromium, iron, magnesium

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DECOMPOSITION OF FERRONICKEL SLAG THROUGH ALKALI FUSION IN THE ROASTING PROCESS

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1. Introduction

East of Indonesia is an area that contains an abundant resource of lateritic nickel. Moreover, some producing plants have been built there processing lateritic ore to produce ferronickel or nickel mate, as well as its by-product, ferronickel slag.

Ferronickel slag is the by-product resulted from the lateritic nickel ore smelting process in the blast furnace or electric furnace. Generally, it contains SiO_2 , MgO and FeO as the main compounds [1–3], which are fused in the olivine phase [4], as well as small amounts of Al [3, 5–7] and Cr [4, 8] that are fused in the spinel phase.

Currently, ferronickel slag is used for reclamation [9–11]. However, it has been discovered that heavy metal was detected in the soil, which used ferronickel slag for reclamation [11]. This means that the potential for environmental contamination is detected [4, 8, 12]. On the other hand, ferronickel slag contains valuable materials, which can be extracted even though it is still challenging. Moreover, the data of Indonesia nickel production from USGS [13–16] 2015–2017 increase from 130,000 up to 345,000 metric ton. Since the production of 1 ton ferronickel results in 8 ton ferronickel slag [17], slag generated and area needed to accommodate the waste can be calculated. Therefore, beneficiation of ferronickel slag is necessary, in order to recover valuable materials and to protect the environment from the hazard materials accumulation.

However, ferronickel slag has a high melting point, about 1,450–1,650 °C [1, 7], which is influenced by its properties such as Si, Mg and Fe [1]. It causes ferronickel slag needs high energy to treat. Therefore, decomposition by materials addition is expected to decompose ferronickel slag bonding that can decrease the process temperature and reduce the energy used subsequently. Moreover, decomposition changes the phases contained and simplifies the extraction process afterward.

The previous study [5] discussed the thermal decomposition of the mixture of ferronickel slag- Na_2CO_3 at various temperatures. However, roasting time, which is an essential variable as well, has not been investigated in thermal decomposition. In order to overcome that gap, roasting processes at various temperatures and time were conducted. This is related to the mass transfer in the reactions occurred that influence decomposition, including weight loss, elements content, phases and morphology. Moreover, the proposed reaction mechanism is discussed based on the thermodynamic analysis and phases formed from the XRD analysis. Therefore, studies that are devoted possess scientific relevance.

2. Literature review and problem statement

In order to beneficiate ferronickel slag, some studies have been conducted to decompose ferronickel slag in the form that is easy to separate.

The reduction process using coal as a reducing agent against ferronickel slag with the addition of Na_2SO_4 resulted in phase transformation, Fe liberated from silicates and reacted with Na_2SO_4 to form FeS [18]. Meanwhile, FeO resulted from the reduction of ferronickel slag with NaOH addition [19]. Both previous studies show that reduction using sodium compound as an additive selective to the decomposition of Fe element. Decomposition of Mg or Si elements to the easily separated form is likely more beneficial since Mg and Si content dominates ferronickel slag. Sodium magnesium silicate formation could increase difficulties in the separation of Mg and Si elements.

Thermodynamic analysis of the sintering process of ferronickel slag in the presence of magnesia at 1,200–1,500 °C was carried out. Forsterite and spinel (Mg, Fe-spinel, Mg, Al-spinel, and Mg, Cr-spinel) formed from the sintering process, which can be used as refractory materials. Both of them have a high melting point that rectifies the refractoriness of refractory materials [20]. This process used high temperature for sintering, it needs much energy. Forsterite also resulted from the roasting process of the ferronickel

slag- Na_2CO_3 mixture at 1,000 °C [5]. Na_2CO_3 addition suppresses the temperature of forsterite formation due to its melting point, thus it needs less energy. However, the transformation of element content and reaction mechanism were not discussed in this study.

Some phases that were bonded to sodium compound were observed in the roasted products. These are disodium magnesium silicate [4, 7] and sodium chromate [4], which were observed from the roasting process that used sodium compound such as Na_2O_2 [4] and NaOH [7] as an additive in the roasting process at about 500–600 °C for 1 hour. Moreover, selective extraction of elemental chromium from ferronickel slag via microwave roasting with sodium peroxide addition followed by water leaching yielded 94.21 % soluble chromium, while only 0.06 % by weight of chromium remained in the leach residue [21]. Sodium chromate formation is beneficial since it can be separated by water leaching and prevent environmental pollution. However, low recovery might be obtained since Cr content in the raw ferronickel slag is low. The removal of magnesium from ferronickel slag by vacuum reduction using CaO to remove SiO_2 in the slag was carried out. The result obtained was 97.74 % of magnesium in the experimental conditions at 1300 °C for 3 hours and 10 Pa with the addition of 30 % CaO [22]. This process needs much energy for vacuum, high pressure and high-temperature conditions.

3. The aim and objectives of the study

This study aims to determine the thermal decomposition of ferronickel slag with Na_2CO_3 addition in the roasting process. Furthermore, the proposed reaction mechanism is determined based on the thermodynamic analysis and phase transformation.

The objectives are achieved by the following methods:

- characterization of ferronickel slag using XRF analysis;
- roasting of the mixture of ferronickel slag- Na_2CO_3 followed by calculating the weight loss percentage;
- characterization of ferronickel slag and roasted product using ICP-OES to determine the effect of the roasting process on the element content;
- characterization of ferronickel slag and roasted product using XRD analysis to determine the phase transformation;
- characterization of ferronickel slag and roasted product using SEM-EDS to determine the morphology transformation.

4. Materials and method for investigation of thermal decomposition of ferronickel slag with Na_2CO_3 addition in the roasting process

4.1. Materials

Ferronickel slag from Morowali, Central Sulawesi, Indonesia is used for this study. Analytical chemical grade of Na_2CO_3 from Merck is used for the additive that was mixed with ferronickel slag prior to the roasting process.

4.2. Methodology

Ferronickel slag was crushed and milled to reduce the size to 149 μm . It was then mixed with Na_2CO_3 in a 1:1 ratio by physical blending. The mixture was weighted

then placed into a graphite crucible for roasting, which was performed in the CWF 1300 carbolite muffle furnace. Heating was started subsequently when the samples were in the furnace. Roasting processes were carried out at a specific temperature (800, 900 and 1,000 °C) and restrained for a specific time (60–240 minutes). The furnace was switched off after process conditions were obtained and cooling processes were performed with the samples in the furnace until reaching ambient temperature. Weighting of the roasted product was carried out to calculate the weight loss percentage caused by the roasting process. Characterizations were then performed with the roasted products.

4. 3. Characterization

Chemical composition of ferronickel slag in the form of its oxide was characterized by XRF from Bruker S2 Puma, shown in Table 1. Few amounts of raw ferronickel slag and roasted products were dissolved in the strong acid (the mixture of nitric acid pure analytic and hydrochloric acid pure analytic with a 1:3 ratio) and diluted using distilled water. That method is supported for Al, Cr, Fe and Mg analysis, therefore, in this study observations are limited for these contents. The diluted solutions were then characterized using ICP-OES 725 from Agilent to determine the upgrading of the elements content. XRD analysis was carried out at a 2θ angle, using Cu anode and wavelength of 1.541487Å. XRD was performed for the raw ferronickel slag and roasted product to determine phase transformation. The morphological transformation and semi-quantitative elemental content of the raw ferronickel slag and roasted product were analyzed using SEM-EDS JOEL Jsm 6390A.

5. Experimental results of decomposition of ferronickel slag through alkali fusion in the roasting process

Roasting processes of ferronickel slag with Na₂CO₃ addition were conducted at 800, 900 and 1,000 °C for 60–240 minutes. Characterization of raw materials and roasted products was carried out to determine the effect of roasting processes on the decomposition occurred. Analysis of the raw ferronickel slag, weight loss percentage, upgrading of elements content, phase and microstructure transformation was carried out.

5. 1. Characterization of ferronickel slag

XRF analysis is the initial characterization of the ferronickel slag that was carried out to determine the chemical compositions, shown in Table 1. It is critical to perform since it defines the appropriate process.

It can be seen from Table 1 that Mg and Si oxides dominate the ferronickel slag followed by the oxides of Fe, Al and Cr. Meanwhile, the content of other oxides is less than 1 % wt.

Table 1

Chemical compositions of ferronickel slag

Component	Content (% wt)	Component	Content (% wt)
Cl	0.0600	Fe ₂ O ₃	10.78
MgO	41.04	NiO	0.0535
Al ₂ O ₃	4.04	ZnO	0.0255
SiO ₂	40.74	Cr ₂ O ₃	1.47
P ₂ O ₅	0.2470	Ta ₂ O ₅	0.0030
SO ₃	0.0120	WO ₃	0.0105
CaO	0.7050	CuO	0.0045
TiO ₂	0.0975	Sc ₂ O ₃	0.0020
LOI	0.7095	-	-

5. 2. Weight loss percentage due to the roasting process

Weight loss occurs due to decomposition in the roasting process of the mixture of ferronickel slag-Na₂CO₃. It can be seen in Fig. 1 that the percent weight loss of roasted products increases as a function of temperature and time.

The highest weight loss percentage, about 25.30 %, results from the roasting process at 900 °C for 240 minutes. Increasing roasting temperature from 800 °C to 900 °C for the roasting time of 60–240 minutes shows a considerable increase in weight loss percentage. However, when the roasting temperature rises from 900 °C to 1,000 °C, a different tendency of weight loss occurs along with escalating roasting time, about 14–18 %. It tends to be stable for escalating roasting temperature from 900 °C to 1,000 °C with the roasting time of 60–120 minutes. On the other hand, increasing roasting time further tends to cause weight loss decrease. Moreover, it is shown that the weight loss percentage of roasted products from the roasting process at 1,000 °C is not significant as a function of time.

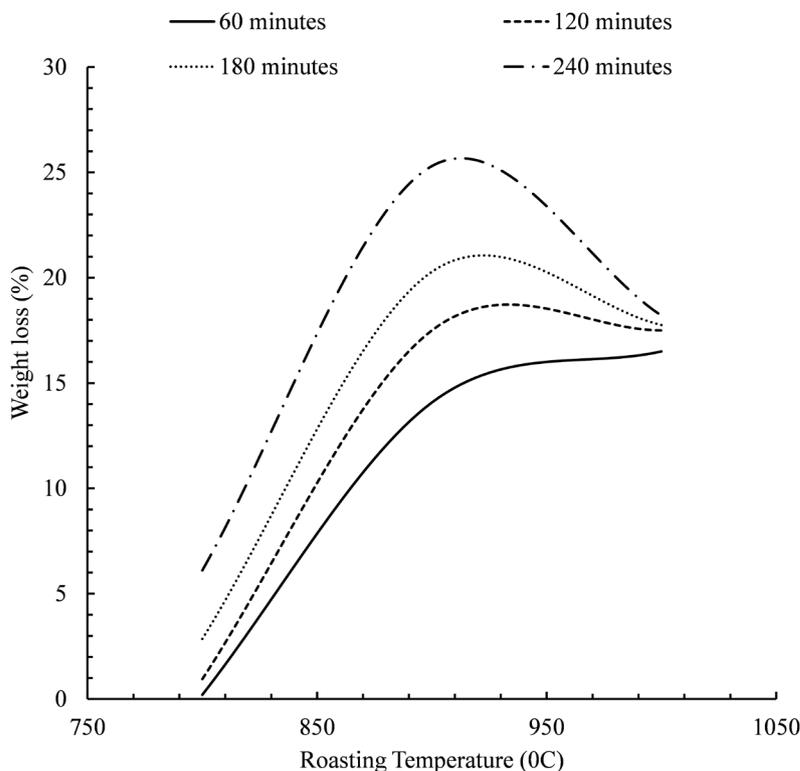


Fig. 1. Weight loss percentage due to roasting process

5.3. Effect of roasting process on the elements content of ferronickel slag and roasted product

In order to determine the effect of roasting temperature and time on the elements content of the mixture of ferronickel slag-Na₂CO₃, the ferronickel slag and roasted product were analyzed using ICP-OES. Table 2 presents the elements content in the ferronickel slag especially for Al, Cr, Fe and Mg content.

Table 2

Characterization of Al, Cr, Fe and Mg content in the ferronickel slag using ICP-OES

Element	Al	Cr	Fe	Mg
Raw ferronickel slag (% wt)	1.74	0.90	6.46	13.04

Fig. 2 shows the Al, Cr, Fe and Mg content of roasted products from the roasting process at 800–1,000 °C for 60–240 minutes. Some data show that increasing elements content is in line with increasing roasting temperature and time. However, roasting processes at 800–1,000 °C for 60 and 120 minutes have a distinct tendency, the highest elements content is generated at a roasting temperature of 900 °C. Moreover, roasting at 1,000 °C shows a good in line tendency of the elements content by increasing the roasting time.

Al contents of roasted products from roasting at 800–900 °C for 60 minutes are slightly higher than in the other roasted products with longer roasting time, as shown in Fig. 2, *a*. The highest Al content of the roasted product, 2.18 %, results from roasting at 1,000 °C for 240 minutes. The highest content of Cr, 1.42 %, is generated from roasting at 900 °C for 240 minutes, Fig. 2, *b*. However, decreasing roasting time to 60 minutes produces just a slightly lower content of Cr, 1.21 %. Moreover, the Fe and Mg contents, Fig. 2, *c, d*, have a similar tendency along with the escalation of roasting temperature and time. The higher contents of Fe and Mg result from roasting at 900 °C for 60 minutes, 1.63 % and 1.99 %, respectively. Increasing roasting temperature and time further would decrease the Fe and Mg content.

5.4. Phase transformation of ferronickel slag and roasted product

Magnesium iron silicate, which is fused in the olivine phase, is observed in the XRD analysis of the ferronickel slag, Fig. 3. Phase transformation occurred when the roasting process was performed. XRD analysis of the roasted product from roasting at 900 °C for 60 minutes, Fig. 3, is chosen to illustrate the phase transformation.

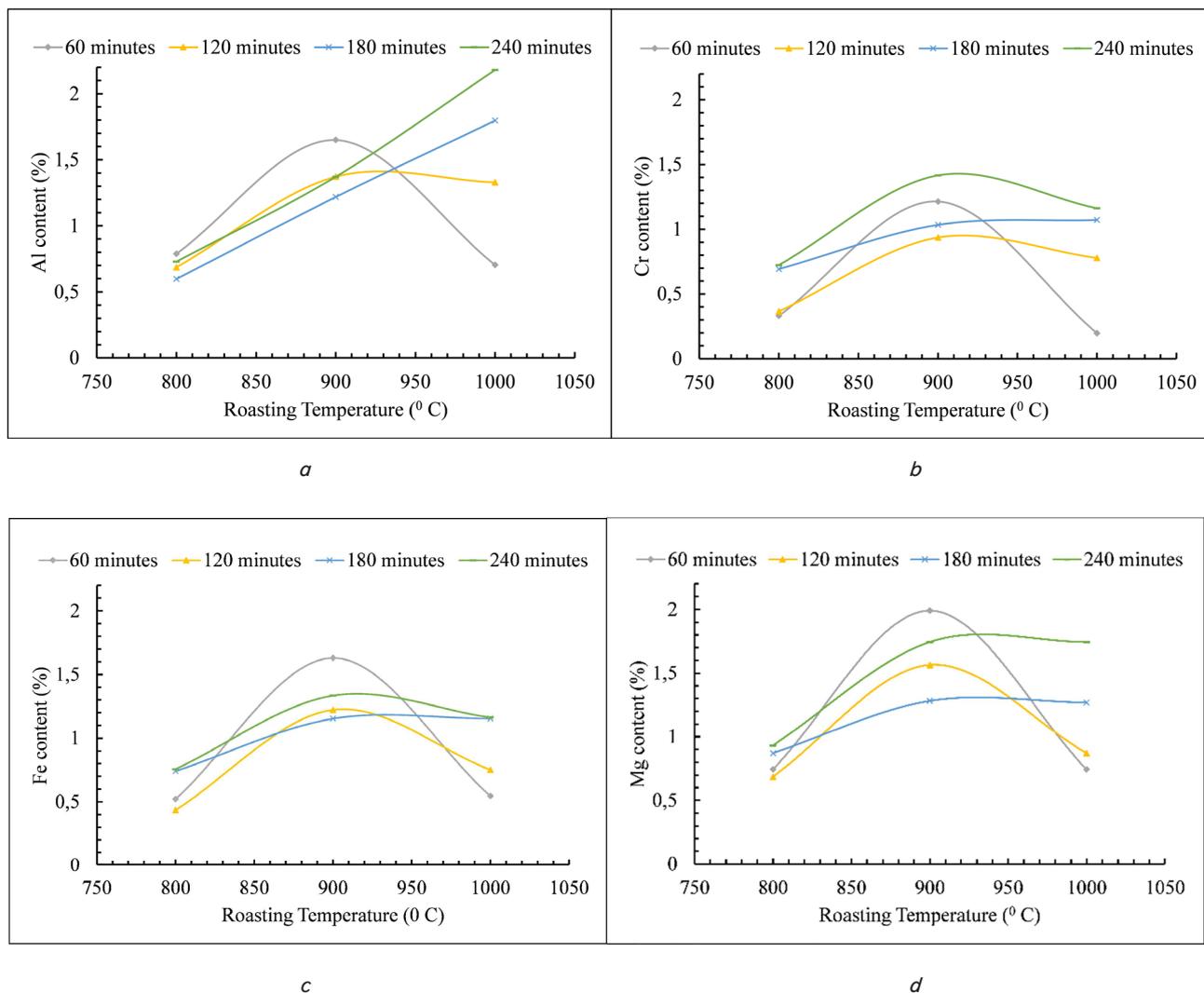


Fig. 2. Element content of the roasted product: *a* – Al content; *b* – Cr content; *c* – Fe content; *d* – Mg content

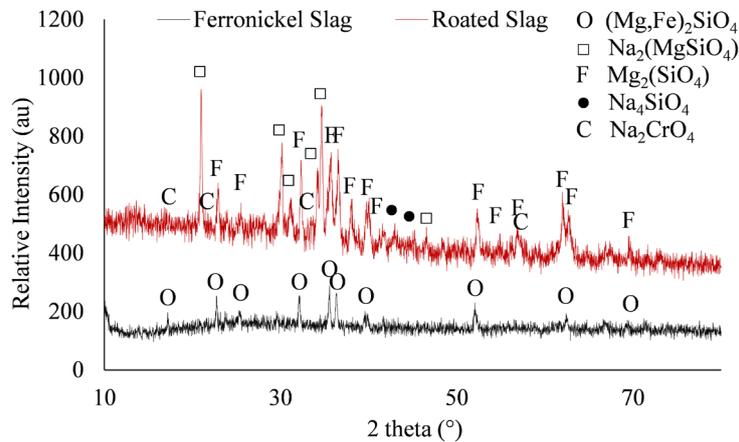
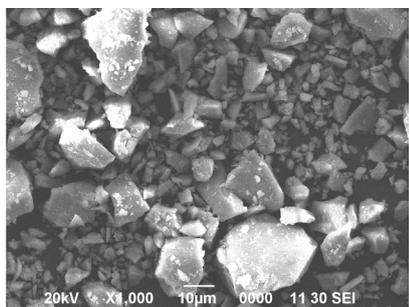


Fig. 3. XRD analysis of ferronickel slag and roasted product from roasting process at 900 °C for 60 minutes

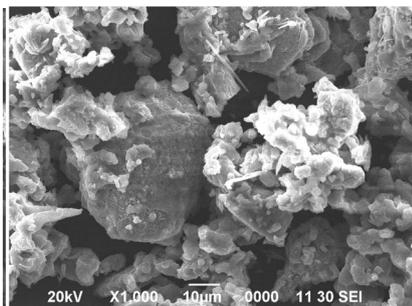
Mg₂SiO₄, Na₂MgSiO₄, Na₄SiO₄, Fe₂O₃ and Na₂CrO₄ are observed as phases formed in the roasted product. Mg₂SiO₄ and Na₂MgSiO₄ are dominated with high intensity. The presence of Na₂MgSiO₄ is in good agreement with previous studies [4, 7], as well as for Na₂CrO₄ [4].

5. 5. Morphology transformation of ferronickel slag and roasted product

SEM-EDS was conducted with the raw ferronickel slag, Fig. 4, a, 5, a and roasted product of the mixture of ferronickel slag-Na₂CO₃ from roasting at 900 °C for 60 minutes, Fig. 4, b, 5, b. The morphology transformation between ferronickel slag and its roasted product can be seen clearly.

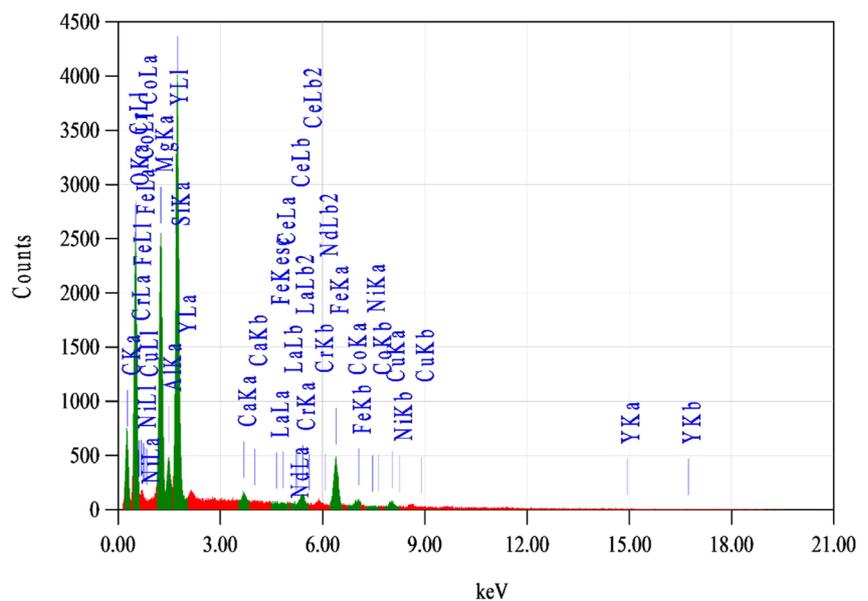


a

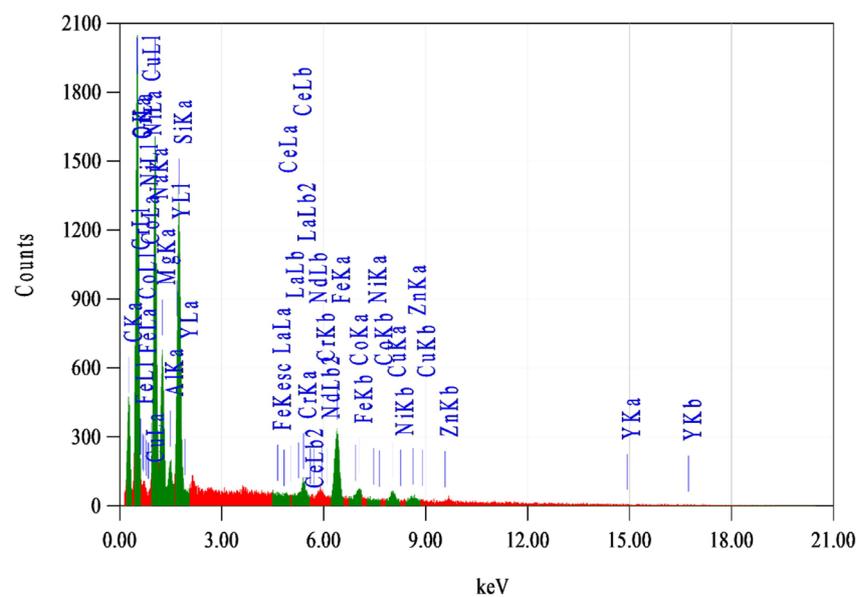


b

Fig. 4. SEM: a – ferronickel slag; b – roasted product of roasting process at 900 °C for 60 minutes



a



b

Fig. 5. EDS: a – ferronickel slag; b – roasted product of roasting process at 900 °C for 60 minutes

The dense and sharp morphology with various particle sizes is observed in the raw ferronickel slag. Meanwhile, there is a rough layer growth on the surface of the roasted product. It indicates that decomposition occurred. In addition, the intensity of the roasted elements such as Al, Fe, Mg and Si significantly declines compared to the raw ferronickel slag due to Na_2CO_3 addition. Meanwhile, the intensity of Cr increases slightly. This corresponds to ICP-OES and XRD analysis where Cr content is increased and Na_2CrO_4 is observed.

6. Discussion of the results of thermal decomposition of ferronickel slag with Na_2CO_3 addition in the roasting process

Alkali fusion of ferronickel slag using Na_2CO_3 addition in the roasting processes was conducted at 800, 900 and 1,000 °C for 60–240 minutes. The results of the analysis of the raw ferronickel slag, weight loss percentage, upgrading of elements content, phase and microstructure transformation are discussed here.

Characterization of the ferronickel slag using XRF indicates that the main components are Mg, Si, Fe, Al and Cr. It is in good agreement with the EDS results. Moreover, the XRD analysis shows olivine ((Mg, Fe) $_2\text{SiO}_4$) as the main phase. The characterizations of raw ferronickel slag correspond to the previous studies [5, 23, 24].

Roasting processes at 800–1,000 °C for 60–240 minutes cause weight loss due to decomposition. Mostly, the highest weight loss percentage is obtained from the roasting process at 900 °C for various roasting times, as shown in Fig. 1. It might be related with % TG-DTA analysis of the mixture of ferronickel slag- Na_2CO_3 performed previously [5], which resulted in a dramatic increase of weight loss percentage and endothermic reaction at 845–860 °C with a total mass loss of 49.30 %. Meanwhile, the weight loss percentage of roasted products from roasting at 1,000 °C was almost similar and endothermic reaction was not observed [5]. However, the highest weight loss resulted in this study is 25.30 %. It might be caused by the fact that the elements change, Fig. 2, is not large enough generally compared to the raw ferronickel slag.

In addition, the melting point of Na_2CO_3 is at 851 °C, thus, the liquid phase accelerates the mass transfer of the mixture and pushed the reaction further. Therefore, the change of elements content is observed distinctively in the roasted products resulted from roasting at 900 °C, as shown in Fig. 2. However, as the roasting temperature and time increase, CO_2 generated also escalates. It might cause reductive conditions with the presence of O_2 and react to form a new substance decreasing the weight loss percentage. It might cause a relatively stable Cr, Fe and Mg content of roasted products from roasting at 1,000 °C or lower Cr, Fe and Mg content of roasted products from roasting at 900 °C. Since Al reacts easily with O_2 , Al content has different trends compared to the Cr, Fe and Mg content. It increases constantly with increasing roasting temperature and longer roasting time.

In the air atmospheric condition, sintering of the olivine in the presence of O_2 is as follows [4]:



Based on the reaction (1) and the main components contained in the ferronickel slag, the reactions proposed that

might occur between ferronickel slag and Na_2CO_3 in the roasting process are as follows:

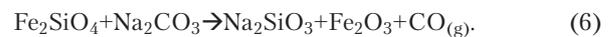
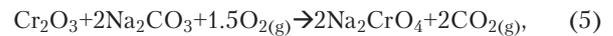
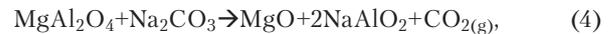


Fig. 6 shows the graph of Gibbs free energy of reaction (2)–(6), which indicates the spontaneity of these reactions.

Based on the Gibbs free energy for reactions (2)–(6), the reactions of Cr and Al are much more spontaneous compared to Fe and Mg, which have a negative value of Gibbs free energy at a temperature of about 700 °C and 900 °C, respectively. It might cause much better upgrading of the elements content in the roasted products for Al and Cr than Fe and Mg compared to its initial elements content in the raw ferronickel slag.

However, Fig. 3 shows the formation of $\text{Na}_2\text{MgSiO}_4$ and Na_4SiO_4 instead of Na_2SiO_3 and MgO. This may be due to the presence of $\text{O}_{2(\text{g})}$ in the roasting process that leads to Na_4SiO_4 formation as reaction (7) [25]. Meanwhile, the formation of $\text{Na}_2\text{MgSiO}_4$ might occur as reaction (8) and (9) [26]. This means that reactions (2) and (4) that produce MgO occur as a transition product to form $\text{Na}_2\text{MgSiO}_4$.



Moreover, the presence of Na_2CrO_4 in the XRD results supports the ICP-OES results that show the upgrading of Cr content in the roasted product compared to its initial content in the raw ferronickel slag.

Fig. 4, 5 show the sharp transition of ferronickel slag and roasted product. Roasting is carried out at a higher temperature than Na_2CO_3 melting temperature, thus, it forced mass transfer better since the reaction occurred at solid-liquid phases and pushed decomposition, as well as the reaction occurred, forming white and glossy aggregate.

Alkali fusion of ferronickel slag using Na_2CO_3 in the roasting process confirmed the occurrence of decomposition of the ferronickel slag at a lower temperature than the melting point of the ferronickel slag. This can be proved by the weight loss, the change of elemental content, phase and morphology transformation, as well as proposed reaction mechanism. Previous studies investigated the recovery of Al [7], Cr [4] and Mg separately [23, 27]. In this study, the changes of elements content for Al, Cr, Fe and Mg are determined simultaneously. It is more attractive when the change of silicon content is also investigated since silicon is the main component of ferronickel slag besides magnesium. However, it needs an additional procedure to prepare the sample for ICP-OES analysis since silicon is not dissolved in the acid solution.

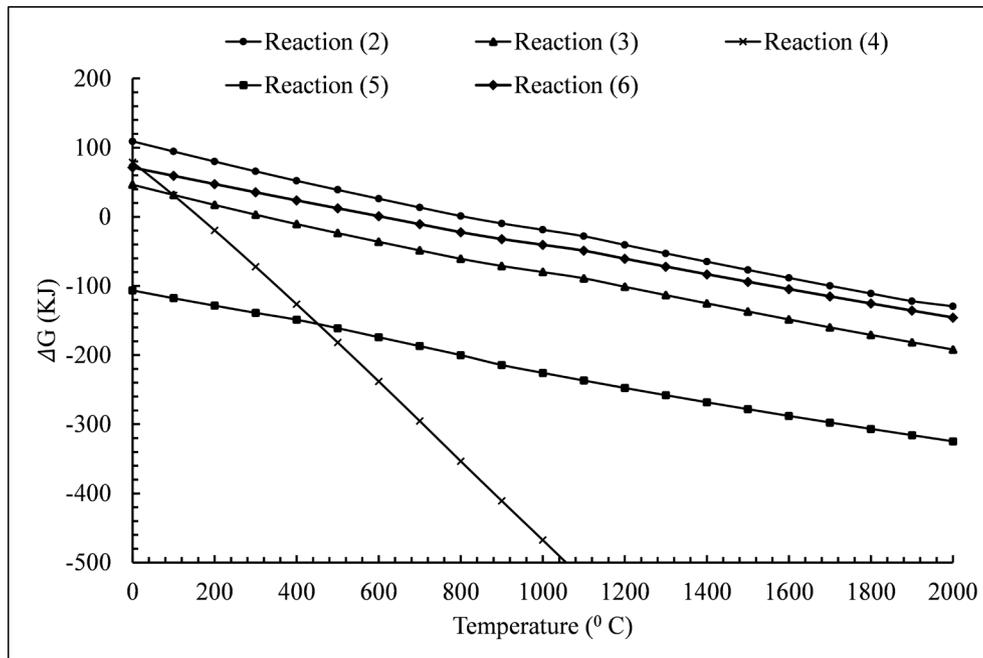


Fig. 6. Gibbs free energy of reactions (2)–(6)

Moreover, variation of Na_2CO_3 addition is needed to determine the optimum Na_2CO_3 addition in the alkali fusion of the roasting process since sodium silicate and Na_2CrO_4 formation that is shown in the XRD analysis is still low and $\text{Na}_2\text{MgSiO}_4$ and Mg_2SiO_4 tend to form. This would cause difficult separation of Mg and Si elements. The development process to decompose $\text{Na}_2\text{MgSiO}_4$ and Mg_2SiO_4 needs to be carried out to obtain the optimum magnesium and silicon separation in the further process.

The formation of Na_2CrO_4 and Na_4SiO_4 in the roasted product indicates that Cr and Si can be separated firstly by dissolving the roasted product in water since both of them are water-soluble. Meanwhile, Al, Fe and Mg remain as residue.

7. Conclusions

1. Characterization of the ferronickel slag using XRF indicates that the main components are Mg and Si, followed by Fe, Al and Cr.

2. Weight loss, elements content, phase and microstructure transformation occurred due to the roasting process. Roasting at 900°C for 60 minutes brings out the most effective results to the weight loss percentage and elements content of selective metal compared to the other roasting conditions since escalating temperature and time generate just a bit alteration.

3. The upgrading of the elements content in the roasted products for Al and Cr is more significant than for Fe and Mg. The upgrading of Al and Cr content in the roasted product is up to 1.25 and 1.35 times, respectively, compared to the raw ferronickel slag.

4. The XRD analysis shows olivine ($\text{Mg, Fe}_2\text{SiO}_4$) as the main phase of the ferronickel slag. It transforms to Mg_2SiO_4 , $\text{Na}_2\text{MgSiO}_4$, Na_4SiO_4 , Fe_2O_3 and Na_2CrO_4 in the roasting process at 900°C for 60 minutes.

5. SEM analysis of ferronickel slag illustrates the morphology transformation of the ferronickel slag and the roasted product indicates the decomposition process occurred due to liquid-solid mass transfer on the surface of the mixture.

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