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IDENTIFYING THE EFFECT OF VARYING ACID CONCENTRATION AND SOLID/LIQUID RATIO IN THE LEACHING EXTRACTION OF MAGNESIUM FROM FERRONICKEL SLAG

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Indonesia is the country with the largest nickel reserves and production levels in the world. Each ton of nickel production can produce eight tons of by-products in the form of ferronickel slag, which continues to increase due to the minimal recycling process of these by-products. This study aimed to determine the impact of changes in acid concentration and solid/liquid ratio on the leaching extraction of magnesium from ferronickel slag and characterize the transformation of ferronickel slag at each stage of the process. The research was conducted using the alkali fusion method and continued with water leaching and acid leaching using Hydrochloric Acid (HCl) as the solvent. The first step in the investigation was milling to get a powder with the particle size ≤127 μm. The sample powder was subsequently mixed with the Na_2CO_3 additive in a 50:50 (w/w) ratio and roasted for 60 minutes at 1000 °C. The water leaching procedure was then conducted for 60 minutes at 100 °C, a 1:10 (w/v) ratio, and a stirring speed of 400 rpm. The filtrate and residue were then separated using a filtration process. An acid leaching utilizing HCl with concentration variations of 0.5, 1, and 1.5 M, and solid/liquid ratio (s/l) variations of 1:20, 1:30, and 1:40 (w/v) was performed on the residue. The results of acid leaching were then filtrated again. The filtrate was then characterized by ICP-OES testing. Based on the analysis results, it can be stated that the percentage of magnesium extraction increases as the solid/liquid ratio (s/l) increases but decreases with the increase in HCl concentration. The optimum percentage of magnesium extraction is 64.12 %, which was achieved with the leaching conditions of a solid/liquid ratio (s/l) of 1:40 (w/v) and a HCl concentration of 0.5 M

Keywords: ferronickel slag, recycling, alkali fusion, roasting, water leaching, acid leaching, extraction, magnesium, acid concentration, solid/liquid ratio

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1. Introduction

Indonesia is one of the countries that has bright prospects in the future with regard to its nickel reserves. Based on data from the Ministry of Energy and Mineral Resources in 2021, Indonesia was declared as the country with the largest nickel reserves in the world, with a total wealth of 72 tons out of a total of 139,419,000 tons of world nickel reserves or has a coverage of 52 % of the world's nickel reserves. According to data in the United States Geological Survey report (USGS) report, the level of nickel production in the world in 2022 reached 3.30 million metric tons. This amount increased by 20.88 % compared to 2021 with a production of 2.73 metric tons. Based on the report, Indonesia is listed as the country with the largest nickel production in the world in 2022 with total production reaching 1.60 million metric tons or can be said to cover 48.48 % of the total nickel production in the world [1].

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There are two types of nickel ore, namely sulfide nickel ore and laterite nickel ore with a percentage content of 30 % and 70 % of the total amount of nickel reserves in the world [2]. The by-product of the ferronickel production process from laterite nickel ore involving smelting through pyrometallurgical methods is called ferronickel slag [3]. Based on the data, every ton of nickel production produces eight tons of by-products in the form of ferronickel slag, which continues to increase due to the minimal recycling process of these by-products [4]. Ferronickel slag can be a serious problem for the environment today and for the future, so it needs to be handled. Several countries, including Indonesia, use the Basel Convention's Classification of Slag reference, which states ferronickel slag as a hazardous waste, so that very careful handling of ferronickel slag is needed [5].

Ferronickel slag contains many valuable materials that can be extracted and have a higher economic value [6]. The main chemical composition of ferronickel slag is 52.10 % SiO_2; 27.06 % MgO; and 11.26 % FeO and the minor composition consists of 6.49 % Al_2O_3; 1.22 % Cr_2O_3; 0.79 % CaO; 0.092 % NiO; 0.01 % CoO, and 0.003 % Sc_2O_3 [5]. Each of these elements can be separated and utilized in different industrial purposes, such silica for silica precipitate [4], iron for magnetite particles [7, 8], and so forth. Furthermore, magnesium is the second most dominant element in ferronickel slag. The magnesium content in this slag is higher than that of dolomite ($CaCO_3.MgCO_3$) and other natural minerals [9]. In accordance with Decree No 296.K/MB.01/MEM.B/2023 of the Ministry of Energy and Mineral Resources, magnesium is classified as a critical mineral. This classification is reserved for minerals that are crucial for the national economy and national defense and have limited reserves that have not yet been replaced. Magnesium extracted from ferronickel slag can be used as a material for making refractories [10], magnesium metal and ferroalloy [5], magnesium for CO_2 removal [11, 12], or magnesium chemical [13, 14].

The extraction method of valuable elements from ferronickel slag has not provided optimal results, mainly due to the difficulty in breaking down the silicate compounds from high-temperature melting. Therefore, research was conducted on the extraction of magnesium from ferronickel slag, focusing on separating magnesium from silicate compounds. To achieve this goal, a study was conducted on a combination of pyro-hydrometallurgy methods with alkali fusion followed by atmospheric leaching using very low acid concentrations. Ferronickel slag powder is first mixed with a certain amount of sodium carbonate (Na_2CO_3) to bind silica through the alkali fusion process, thus obtaining sodium silicate, which can be easily separated through the water leaching process [6]. This process produces residues with higher magnesium content. Thus, research on using magnesium from ferronickel slag will be very beneficial and increase the added value of waste materials from the metal processing that have not been utilized so far.

2. Literature review and problem statement

Numerous investigations have been carried out to recover important components that are still present in ferronickel slag using pyrometallurgy, hydrometallurgy, or a combination of the two processes. Previous research using the pyrometallurgical route had been conducted on the reduction of ferronickel slag at a temperature range of 800–1000 °C with the addition of sodium hydroxide (NaOH), which initiated the formation of sodium magnesium silicate (Na_2MgSiO_4), magnesium oxide (MgO), and wustite (FeO). These phases are not formed in the process without the addition of NaOH additives in which the magnesium element is still bound as a silicate compound in the forsterite ($FeMgSiO_4$) and olivine ($NiMgSiO_4$) phase structures [15]. Similar work was carried out using sodium sulfate (Na_2SO_4). Adding additives and raising the reduction temperature can lower the SiO_2 content and release the iron element, which binds to sulfur to form troilite (FeS) [16]. However, magnesium components cannot be directly separated from ferronickel slag using this pyrometallurgical processing approach. Even when some of the impurities have changed phase, further treatment is still necessary.

Hydrometallurgical method of dissolving silica from ferronickel slag directly using NaOH solution in the atmospheric leaching process for 4 hours was only able to dissolve 31.48 % [17]. Generally, increasing the temperature of the leaching process is known to increase the solubility of silica [18]. The process of separating silica using NaOH solvent with high pressure and temperature in a polytetrafluoroethylene (PTFE) autoclave reactor from vanadium steel slag can separate almost 95 % of the silica contained in it so that residue with a higher metal content is obtained [19]. However, since the silica in ferronickel slag is coupled as a complex silicate compound rather than existing as a stand-alone compound, this process will be very challenging to employ to extract metal elements from this material. Meanwhile, the leaching process which directly uses acid, either hydrochloric acid [13, 20] or sulfuric acid [9, 21] is susceptible to the formation of gel which will inhibit the separation process during the extraction process.

Silica dissolution from ferronickel slag can also be carried out by employing the pyro-hydrometallurgical approach with the alkali fusion followed by leaching process using sodium hydroxide as a solvent after an alkali fusion process at a temperature of 1000 °C with the addition of sodium carbonate (Na_2CO_3) [4]. This technique reveals that a significant amount of silica remains in the residue, which is identified as forsterite (Mg_2SiO_4), which is insoluble in NaOH. The addition of a significant amount of sodium from sodium carbonate and sodium hydroxide is likely to hinder the silica dissolution reaction, thereby reducing the magnesium concentration in the residue. Another investigation has been carried out to extract magnesium using the pyro-hydrometallurgical processing method by adding sodium carbonate and roasted the mixture for 60 minutes at 1000 °C then leached it with water followed by acid leaching using HCl solution. After leaching with a 2 M HCl solution for 30 minutes at 80 °C, up to 82.67 % of magnesium can be separated [6]. Indeed, there is a trend for magnesium extraction to decrease when the acid concentration employed for leaching increases from 2 M to 8 M. The higher the acid concentration, the more elements are dissolved, but the presence of silica in the aqueous solution environment tends to form a gel that will hinder the diffusion of the solvent to the surface of the ferronickel slag particles.

The fundamental issue, as evidenced by the numerous investigations, is how to liberate silica from the silicate structure in the form of enstatite ($MgSiO_3$) and forsterite (Mg_2SiO_4), the two primary phases that comprise ferronickel slag.

All this allows to assert that it is expedient to conduct a study on determining how the solid/liquid ratio and acid concentration affected the reaction, as well as how the phases involved in each reaction stage changed. This method, in addition to reducing operational costs, also has the potential to increase magnesium extraction by preventing the formation of gel that obstructs the reaction between the solvent and ferronickel slag.

3. The aim and objectives of the study

The aim of the study is to determine of possibility utilizing ferronickel slag to extract magnesium in the form of a concentrated solution. In this instance, some advances in identifying the most appropriate variation in acid concentrations including the solid/liquid ratio during the leaching extraction provides scientific evidence. This will allow the transformation of ferronickel slag characteristic and to benefit industrial practicability. To achieve this aim, the following objectives are accomplished:

– to determine characterization of ferronickel slag using XRD and XRF;

– to carry out alkali fusion using Na_2CO_3 with heating at a temperature of 1000 °C for 60 minutes to bind silica in ferronickel slag in the form of sodium silicate (Na_2SiO_3);

– to carry out water leaching to dissolve the sodium silicate from the alkali fusion followed by filtration to separate the residue;

– to carry out acid leaching to dissolve magnesium using HCl solution with concentrations of 0.5, 1, and 1.5 M, and solid/liquid ratios of 1:10; 1:20; and 1:40 at a temperature of 80 °C for 30 minutes;

– to determine impurities in acid leaching filtrate.

4. Materials and methods

4. 1. Object and hypothesis of the study

The object of this study is ferronickel slag, a by-product of ferronickel smelting at high temperatures (>1500 °C). The research hypothesis assumes that the alkali fusion process can bind silica in sodium silicate (Na_2SiO_3), which is soluble in water, producing residue with a greater metal concentration. Furthermore, acid leaching at low concentrations can improve the efficiency of metal extraction because it avoids the formation of gels that obstruct the reaction.

4. 2. Materials and apparatus used in the experiment

The sample used in this study was ferronickel slag from ANTAM Indonesia. Ferronickel slag, which was initially in the form of grains with sizes varying from fine to gravel with a diameter of about 1.5 cm, was ground with a disk mill to obtain fine particles measuring ≤127 μm. Analytical grades of Na_2CO_3 and 32 % Hydrochloric Acid (HCl) were purchased from Merck and were used as received without further purification. Laboratory reagent-grade distilled water produced from a water distillation machine unit was used throughout the experimental process.

4. 3. Experimental procedure

The experimental procedure involved pyrometallurgy with alkali fusion, followed by hydrometallurgy with leaching utilizing water and acid solution. Fig. 1 shows the research flow chart.

At the initial research stage, based on the Fig. 1, the powder sample was mixed with sodium carbonate (Na_2CO_3) additive with a proportion of 50:50 (w/w). Following the mixing step, the mixture was placed on a ceramic cup, placed in a furnace, and then roasted at 1000 °C to form sodium silicate products (Na_2SiO_3) by an alkali fusion phenomenon. The sodium silicate (Na_2SiO_3) compound was water-soluble and was diluted by water leaching. Then filtration was done using the vacuum filtration method to remove the silica content and get the residue with a high content of metal elements. The water leaching process was carried out at a temperature of 100 °C with a solid/liquid ratio (s/l) of 1:10 for 2 hours and a stirring speed of 400 rpm. The solid residue was then washed using aqua dest to maximize the dissolution of sodium (Na) and silica (Si) elements in attention to optimize the percentage of magnesium recovery in the HCl leaching process for the next stage. Acid leaching was carried out using HCl solution at a temperature of 80 °C for 30 minutes. The HCl solution concentration was kept as low as possible, ranging from 0.5 to 1 M, and the solid/liquid ratio (s/l) was set at 1:10, 1:20, and 1:40 to prevent the formation of a gel that would stop the solvent from reacting with ferronickel slag particles. It is envisaged that this will allow for the best possible proportion of magnesium extraction.

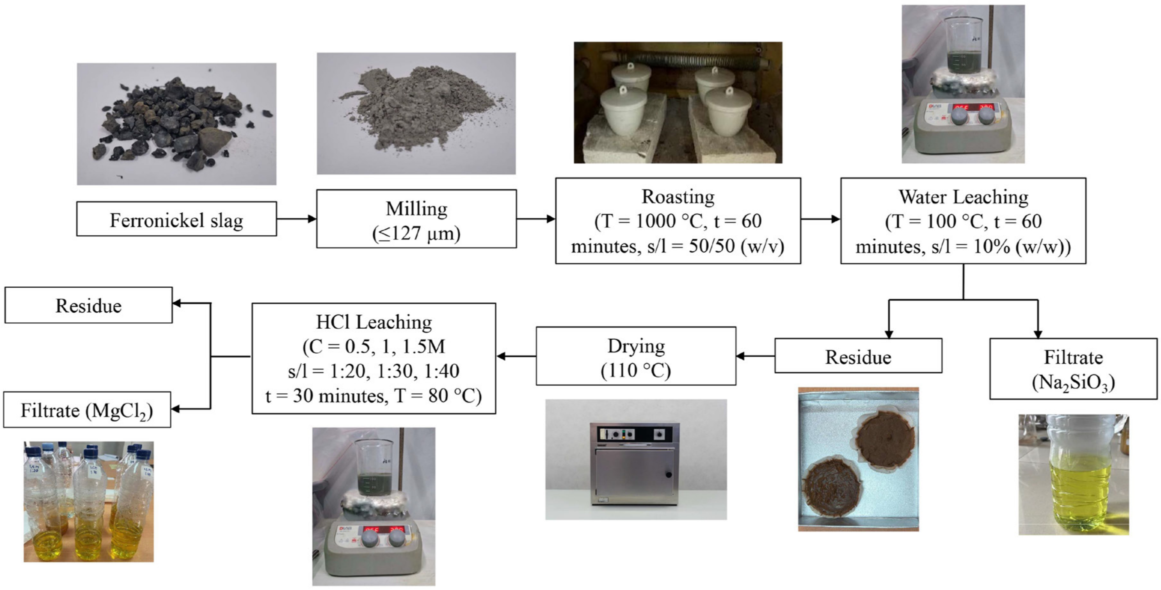

Fig. 1. Research flow chart

4. 4. Characterization

The transformation in chemical composition and phase structure of initial ferronickel slag, roasted sample after alkali fusion, and water leaching residue were analyzed using X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD), respectively. XRF analysis was performed using XRF Panalytical Epsilon 3 xle. A hydraulic press in the aluminum cup was used to compress many powder samples into pellets, which were subsequently put straight into the spectrometer. XRD analysis was carried out by using XRD Panalytical X'pert3 Powder at a 2Θ from 10 to 90° using Cu anode and wavelength of 1.54 Å. The leaching filtrate was analyzed by Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP – OES) Agilent 725 series to determine the content of dissolved elements (wt. %) by comparing with diluted standard solution.

5. Experiment results of the leaching extraction of magnesium from ferronickel slag

5. 1. Characterization of ferronickel slag

X-ray diffraction (XRD) characterization was carried out to determine the compounds contained in the sample, while X-ray fluorescence (XRF) analysis was performed to get chemical composition of the materials. Fig. 2 shows the XRD graph analysis for initial ferronickel slag, and Table 1 shows the results of the chemical composition analysis of ferronickel slag using XRF.

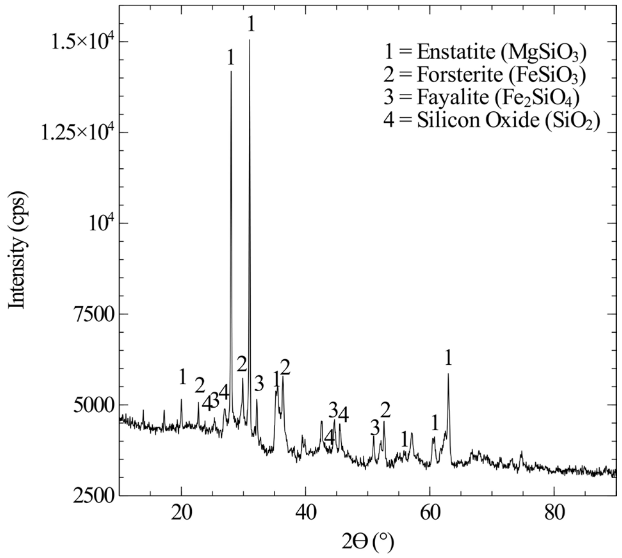

Fig. 2. X-ray diffraction analysis of initial ferronickel slag

From Fig. 2 it is possible to see that the peaks on the XRD graph indicate the presence of enstatite ($MgSiO_3$), forsterite ($MgSiO_4$), fayalite (Fe_2SiO_4), and silicon dioxide (SiO_2) phases.

The data obtained through XRF characterization as is possible to see in Table 1 are in the form of oxide compounds of metals along with the percentage of its content. The components that predominate in ferronickel slag are magnesium (MgO) at 11.35 %, iron (Fe_2O_3) at 30.64 %, and silica (SiO_2) at 44.44 %.

Table 1

Chemical composition of initial ferronickel slag

Compound	Content (%)
SiO_2	44.44
MgO	30.64
Fe_2O_3	11.35
Al_2O_3	2.67
CaO	2.47
Cr_2O_3	2.42
MnO	0.80
P_2O_5	0.55
NiO	0.23
SO_3	0.22
TiO_2	0.18
Co_3O_4	0.13

5. 2. Alkali fusion of ferronickel slag with Na_2CO_3 addition

After the ferronickel slag sample was mixed with Na_2CO_3 and roasted into alkali fusion process. This process aims to produce sodium silicate (Na_2SiO_3) following this reaction [6]:

$$MgSiO_3(s)+Na_2CO_3 \rightarrow MgO+Na_2SiO_3+CO_2(g), \quad (1)$$

$$Mg_2SiO_4(s)+Na_2CO_3 \rightarrow 2MgO+Na_2SiO_3+CO_2(g). \quad (2)$$

This product of alkali fusion is soluble in water so that metal elements will remain in the water-insoluble residue. Under specific conditions, a transition product known as sodium magnesium silicate (Na_2MgSiO_4) may also occur in this alkali fusion process. This Na_2MgSiO_4 formation reaction occurs according to the following reaction [21]:

$$MgO+SiO_2+Na_2CO_3 \rightarrow Na_2MgSiO_4+CO_2(g), \quad (3)$$

$$MgO+Na_2O+SiO_2 \rightarrow Na_2MgSiO_4. \quad (4)$$

Other reactions that may occur during the alkali fusion at the temperature of 1000 °C are as follows:

$$Na_2CO_3 \rightarrow Na_2O+CO_2, \quad (5)$$

$$Fe_2SiO_4+Na_2CO_3 \rightarrow Na_2SiO_3+Fe_2O_3+CO, \quad (6)$$

$$MgSiO_3 \rightarrow MgO+SiO_2. \quad (7)$$

Then XRD and XRF characterization were carried out on the results of the roasting stage. Fig. 3 shows the XRD characterization results of the roasted sample from the mixture of ferronickel slag and Na_2CO_3. The results of chemical composition analysis can be seen in Table 2.

Along with the fayalite, enstatite, hematite, and silicon dioxide phases seen in the initial ferronickel slag sample, Fig. 3 also showed the existence of sodium silicate (Na_2SiO_3) and sodium magnesium silicate (Na_2MgSiO_4) phases.

The data in Table 2 show that 40 wt. % of the chemical composition of the roasted sample after alkali fusion was dominated by iron oxide (Fe_2O_3). Compared to the initial ferronickel slag, the Fe_2O_3 content increased, and the SiO_2 content decreased.

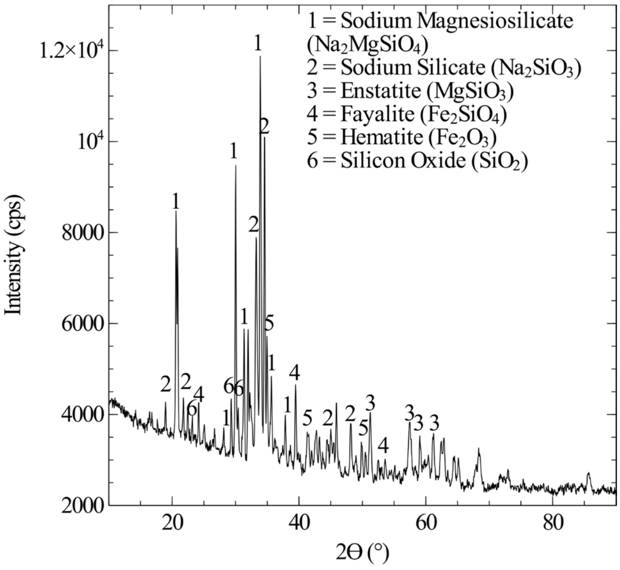

Fig. 3. X-ray diffraction analysis of roasted product from alkali fusion

Table 2

Chemical composition of the roasted sample after alkali fusion

Compound	Content (%)
SiO_2	29.07
MgO	9.01
Fe_2O_3	40.03
Al_2O_3	1.32
CaO	2.69
Cr_2O_3	2.82
MnO	1.03
P_2O_5	0.74
NiO	0.45
SO_3	0.29
TiO_2	0.21
Co_3O_4	0.19

5. 3. Water leaching of roasted ferronickel slag

Ferronickel slag is leached using water after undergoing the alkali fusion roasting stage. This process aims to dissolve the formed sodium silicate so that the silica component separates from the ferronickel slag, leaving a residue with a higher metal content. The residue of the water leaching process is first dried through the drying process in an oven with a temperature of 110 °C for 2 hours. Subsequently, the drying results were again crushed using a pestle and mortar to achieve a higher surface area of fine powder. The dried residue from the water-leaching filtering process was then subjected to XRD and XRF characterization before proceeding to the next stage. Fig. 4 shows the XRD characterization results of the water leaching residue and the chemical compositions in Table 3. These characterizations were carried out to determine changes in ferronickel slag's structural phases and chemical composition until it turns into residue.

Fig. 4 shows that the water-leaching residue sample still contained sodium silicate (Na_2SiO_3) and sodium magnesium silicate (Na_2MgSiO_4) phases.

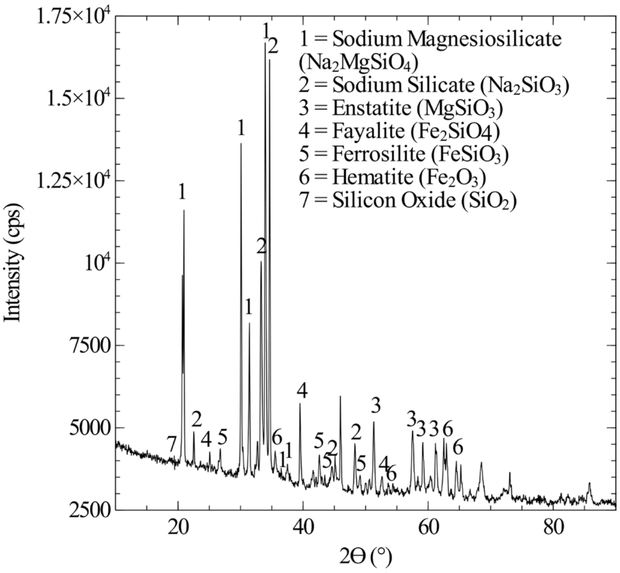

Fig. 4. X-ray diffraction analysis of water-leaching residue

Table 3

Chemical composition of water leaching residue

Compound	Content (%)
SiO_2	28.80
MgO	12.98
Fe_2O_3	46.27
Al_2O_3	2.08
CaO	3.29
Cr_2O_3	1.42
MnO	1.28
P_2O_5	0.53
NiO	0.62
SO_3	0.02
TiO_2	0.19
Co_3O_4	0.15

Based on Table 3, the percentage of SiO_2 content has decreased compared to the previous roasting product.

5. 4. Effect of HCl concentration and solid/liquid ratio (s/l) on magnesium content

Characterization of Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) on the filtrate of acid leaching results aims to determine the magnesium content that dissolves in HCl in the form of magnesium chloride ($MgCl_2$) so that the effect of concentration and solid/liquid ratio (s/l) can be known to the percentage of magnesium content obtained. ICP-OES characterization results are obtained with units of ppm (part per million).

The ICP-OES test data will be processed to determine the extraction yield value of magnesium from each variable variation using formula of:

$$\text{Extraction}(\%) = \\ = \frac{\text{Initial flask volume} \times \text{ICP result} \times \text{Dilution factor}}{\text{Weight of solid sampel}} \times 100\,\%.$$

The data processing results using those formulas will display the metal extraction percentage in graphical form based on concentration and solid/liquid ratio (s/l) variations.

Residue samples from water leaching that have been dried and prepared will be continued in the leaching process using acid solutions with concentrations of 0.5, 1, and 1.5 M and solid/liquid ratios (s/l) of 1:10, 1:20, and 1:40 at a temperature of 80 °C for 30 minutes. Test results that have been processed to determine the amount of extraction obtained are displayed in the form of graphical images in Fig. 5, 6.

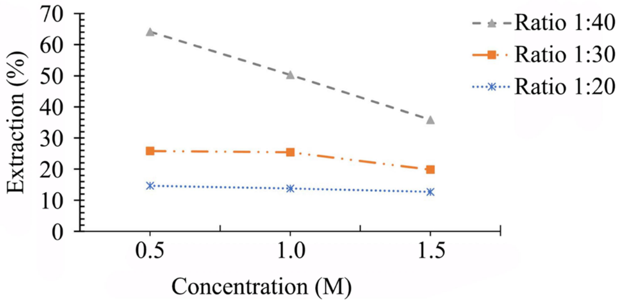

Fig. 5. Effect of HCl concentration variation on magnesium content

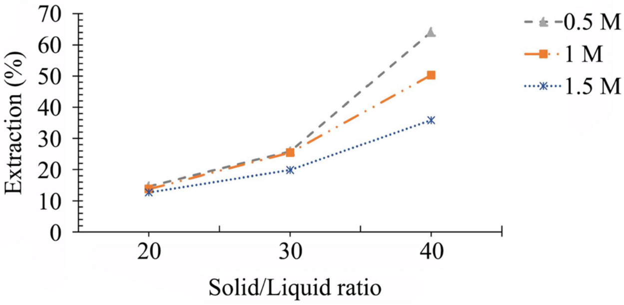

Fig. 6. Effect of solid-to-liquid ratio (s/l) on magnesium content

Fig. 5 shows the magnesium extraction due to the influence of variation in acid concentration, which consists of 0.5 M, 1 M, and 1.5 M, and Fig. 6 shows the effect of solid/liquid ratio (1:10, 1:20, and 1:40 (w/v)) on the magnesium extraction results from the acid leaching process.

5. 5. Impurities in acid leaching filtrate

Table 4 shows the result of the analysis of impurities in the filtrate of acid leaching.

Table 4

Elemental content of Na&Si in HCl leaching filtrate

No	Na (ppm)	Si (ppm)
1	61.73	–
2	90.10	13.90
3	58.23	42.19
4	13.52	–
5	91.47	52.42
6	66.93	43.48
7	117.00	78.81
8	99.60	50.72

The presence of sodium (Na) and silicon (Si) elements remain detected from the results of ICP-OES analysis in the acid leaching filtrate. The presence of these two elements can be a prime factor that influences the percentage of magnesium extraction in the form of $MgCl_2$.

6. Discussion of the effect of varying acid concentration and solid/liquid ratio for extracting magnesium from ferronickel slag

Based on the experimental results, this discussion can give details about how ferronickel slag is initially described and how its phase and chemical makeup change after being roasted in the alkali fusion and water-leaching processes. It can also talk about the condition of the application result, where the effect of acid concentration and the solid/liquid ratio in the acid leaching process is studied. This potential effect will influence the amount of magnesium extracted from the processed ferronickel slag.

Characterization of ferronickel slag based on the analysis results using XRD according to Fig. 2, ferronickel slag is composed of forsterite (Mg_2SiO_4), enstatite ($MgSiO_3$), fayalite (Fe_2SiO_4), and quartz (SiO_2) compounds. These four compounds are the compounds that dominate the composition of the ferronickel slag. In addition, it can also be seen that most of the graph's peaks result from silicate oxides that bind to magnesium and iron elements in the form of magnesium and iron elements in the form of forsterite, enstatite, and fayalite. The three compounds above dominate the chemical composition of ferronickel slag [22]. Broadly speaking, the chemical composition of this ferronickel slag is composed of silicate compounds, which will be broken down to obtain the desired valuable metals through the next process.

Based on the XRF results of the initial ferronickel slag contained in Table 1, there are three elements that dominate with the largest levels in ferronickel slag, namely silica (SiO_2) at 44.44 %, iron (Fe_2O_3) of 30.64 %, and magnesium (MgO) of 11.35 %. These results are in accordance with previous research where the three elements with the highest levels in ferronickel slag are the same elements as these XRF results [17].

After the ferronickel slag sample was mixed with Na_2CO_3 and roasted to the alkali fusion process, XRD and XRF characterization were carried out on the results of the roasted sample. The results of the XRF characterization of the roasting product can be seen in Table 2. XRF characterization was conducted to determine changes in the content of the chemical composition of ferronickel slag samples after mixing with Na_2CO_3 additives and roasting. XRD characterization of the roasted ferronickel slag in Fig. 3, *b* shows the compounds that change due to high-temperature decomposition and the addition of Na_2CO_3 (alkali fusion) to ferronickel slag. Based on that figure, one of the results of the roasting process is Na_2SiO_3, which is one of the parameters of the success in creating alkali fusion phenomenon to be able to bind silica (Si) and dissolve it together with sodium (Na) derived from additives, in the leaching process using distilled water as shown in reaction (1) and (2).

Both reactions cause a decrease in the intensity of the magnesium silicate compounds due to the tendency of sodium (Na) to bind to silica (Si). The success of the alkali fusion process is a decrease in the intensity of the magnesium silicate bond and the formation of sodium silicate compounds (Na_2SiO_3). The decrease in magnesium content is caused by decomposition due to high temperature during roasting and the addition of Na_2CO_3 additives. In addition, there are also results from the roasting process that occupy a high peak position in the figure above, namely sodium magnesiosilicate (Na_2MgSiO_4). This is in accordance with previous research, which states that the product transition compound from roasting a mixture of ferronickel slag with Na_2CO_3 consists of Na_2MgSiO_4, Mg_2SiO_4, and Fe_2O_3, where Na_2MgSiO_4 becomes the dominant compound that holds high intensity in roasting products [21].

Based on reactions (3) and (4), it can be said that the product in the form of MgO is the transition product to form Na_2MgSiO_4, which occurs due to the distribution of magnesium, which decreases or thins out at 1000 °C and has a weaker bond with Si and O. This makes the decomposition that occurs to the Mg_2SiO_4 to MgO at 800 °C. In addition, at temperature ±851 °C, there is also a decomposition of Na_2CO_3 into Na_2O and CO_2 [22] as in reaction (5).

This is related to the formation of more CO_2 as the temperature rises, which causes a reductive reaction. Due to the decomposition as the temperature increases, the distribution of Na, Si, and O elements tend to be in the same place along with Mg, Si, and O, which experience a decrease [22]. This is what makes the products of reactions (1) and (2), namely MgO, re-experience a reaction with Na, Si, and O, as shown in reaction (4), thus creating the Na_2MgSiO_4 product, which is the dominant compound in the roasting product.

There is an increase in iron (Fe) in the roasted product, which is the result of the reaction of Fe_2SiO_4 (fayalite) with Na_2CO_3 additive as the reaction (6) [21].

This reaction changes iron from a silicate compound to an oxide compound, which increases the iron content detected by XRF. Through the reactions above, the formation of Na_2SiO_3 can become more significant as the temperature increases. The formation of Na_2SiO_3 is significant because the roasting process binds Si and Na from the additive before weakening the silicate bonds of the valuable element to undergo extraction further. The more silica bonded, the higher silica content will be dissolved in the subsequent water-leaching process, which can increase the effectiveness of HCl leaching results.

In addition, the decrease in magnesium content after roasting is due to the decomposition reaction that occurs at high temperatures along with the addition of Na_2CO_3 additives, of magnesium transition from Mg_2SiO_4 (forsterite) to Na_2MgSiO_4 (sodium magnesiosilicate) according to reaction (3) and reaction (4) above. In addition, the $MgSiO_3$ (enstatite) compound, which is one of the dominating silicate compounds in ferronickel slag, tends to undergo a decomposition reaction as the roasting temperature increases according to reaction (7) [6].

The product, MgO, decomposes and tends to react with Na_2CO_3 to form sodium magnesiosilicate (Na_2MgSiO_4), decreasing the magnesium levels detected in oxide form.

Upon the roasting completion stage, the water-leaching process is carried out on the sample. The residue from the water-leaching filtering process was then subjected to XRD and XRF characterization before proceeding to the next stage. Fig. 4 shows the XRD characterization results of the residue of the water-leaching process. Based on the graph above, there is a decrease experienced by the Na_2MgSiO_4 and Na_2SiO_3 compounds in which its related to previous research where after water leaching, no Na_2SiO_3 and Na_2MgSiO_4 are found in the XRD graph, to show that the compound is water soluble.

In this study, there is still a certain amount of Na_2SiO_3 on the XRD graph, where the compound must be further minimized through washing with the decantation method, namely by soaking the residual solid in a distilled water solution with a large intensity to maximize the dissolution of Na and Si compound bonds such as Na_2SiO_3 [6].

XRF analysis show the residue of water leaching, and the result is also provided in Table 3. Based on the chemical analysis, there is an increase in the level of Fe_2O_3 due to the reaction between Fe_2SiO_4 and Na_2CO_3 additives (reaction 6), which makes an increase in Fe_2O_3 levels through the products resulting from the reaction. In addition, the decrease in SiO_2 levels is due to the tendency for SiO_2 elements to react with Na_2CO_3 additives and produce water soluble sodium silicate (Na_2SiO_3). The decrease in silica content through the XRF data above indicates the success of the alkali fusion phenomenon, which results in an increase in Na content in ferronickel slag. The decrease in silica (Si) levels can increase the effectiveness of the acid leaching process using HCl, because the less silica content, the smaller the potential for gel formation due to the reaction between silica and acid solution, so that the separation process between filtrate and residue can be more straightforward [6]. The decrease in silica content can also increase the percentage of recovery of valuable elements including magnesium (Mg) contained in ferronickel slag in the leaching process with HCl because the valuable elements the majority of which previously existed in the form of silicates [22], can experience chain breaks with the element silica (Si) and tend to form oxide compounds. This can make the reaction between valuable elements such as magnesium (Mg) can experience contact and a more effective reaction with HCl in the leaching process. This is in accordance with research conducted previously stated that the decrease of SiO_2 content in the residue obtained after the water leaching process indicates that the water leaching process has the ability to dissolve SiO_2 which has been decomposed [6]. In addition, the decrease in the level of SiO_2 is also in accordance with the previous XRD graph in Fig. 3 which shows the presence of SiO_2 that is binds to other elements also experienced a decrease in peak, which means a decrease in intensity in the sample.

In the leaching process using a solution with a s/l ratio of 1:20 with a variation of concentrations of 0.5 M; 1 M; and 1.5 M, the extraction percentage was 14.66 %; 13.78 %; and 12.69 %, respectively. Then in the leaching process using solution with a s/l ratio of 1:30 with a concentration variation of 0.5 M; 1 M; and 1.5 M, the extraction percentage were 25.83 %; 25.44 %; and 19.85 %. And for leaching using a solution with a s/l ratio of 1:40 with a concentration variation of 0.5 M; 1 M; and 1.5 M, the extraction percentage were 64.12 %; 50.31 %; and 35.85 %. These findings demonstrate that when the concentration of the HCl solution used in the leaching process rises, the amount of magnesium (Mg) extracted will decrease. This outcome is consistent with other studies, which found that when the concentration of HCl is low, the rate at which magnesium is displaced into the solution increases, and vice-versa. [23]. A lower HCl concentration will accelerate the rate at which magnesium reacts with the solution, promoting a higher amount of $MgCl_2$ formation products in the filtrate. The reduction in HCl concentration decreases the amount of magnesium (Mg) recovery as it may make it less likely for other components or contaminants to dissolve in the filtrate.

In addition, based on the ICP-OES characterization data in Table 4 on the filtrate of the acid leaching results with HCl, the presence of sodium (Na) and silica (Si) elements is still detected where these two elements can inhibit the percentage of magnesium extraction in the form of $MgCl_2$ with maximum levels due to the reactivity of sodium (Na^+) to the chloride (Cl^-) from HCl, which makes the $MgCl_2$ formation reaction not maximized because Cl^- will tend to react with Na^+ to form NaCl rather than react with Mg to form the product $MgCl_2$. The presence of the element silica (Si) can also inhibit the percentage of obtaining magnesium in the form of $MgCl_2$ with maximum levels due to the result of the reaction between silica and aqueous solution in acid leaching will produce a product in the form of a gel, which will complicate the process of separating magnesium from ferronickel slag. In addition, the formation of the gel will increase the viscosity of the solution which will inhibit particle movement, reaction and mass transfer for the particles, reactions, and mass transfer for the formation of $MgCl_2$ so that the percentage of magnesium extraction cannot be obtained maximally. To be able to minimize the presence of Na and Si elements to a minimum in the residue of the water leaching process before acid leaching is carried out, it is necessary to wash properly. The washing method that is considered effective is decantation method, which is a method carried out by soaking the residue in the form of solids in a container using warm distilled water with a large enough amount with the aim of maximizing dissolution evenly.

Leaching conditions with an HCl concentration of 0.5 M were not able to provide maximum results with an extraction percentage of 64.12 %, however, this condition minimized the formation of gel that would inhibit the process and thus made handling easier during the process. In addition, the solid/liquid ratio appears to play a major role in the extraction process where the more liquid phases involved can increase the extraction percentage. However, the involvement of the quantity of reacting materials will affect the capacity of the equipment needed.

7. Conclusions

1. Based on the results of XRD and XRF characterization, it can be concluded that the compound SiO_2 is the compound with the largest composition in ferronickel slag.
2. The roasting process at 1000 °C creates an effective alkali fusion phenomenon, where more Na_2SiO_3 is formed from the reaction between silicate compounds and Na_2CO_3 additives.
3. The water leaching is effective in reducing the amount of sodium silicate (Na_2SiO_3) through the process of alkali fusion despite the XRD results showing traces of them, including magnesium silicate (Na_2MgSiO_4).
4. The smaller ratio of solid to liquid (s/l) used, the greater percentage of magnesium extraction obtained. S/l ratio of 1:40 give the optimum result on magnesium extraction.
5. Based on the elemental composition test, there is evidence that the acid-leaching impurities exhibit a small portion of sodium (Na) and silicon (Si) in each sample, indicating the success of magnesium extraction. The decreased amount of Si content is directly proportional to the percentage of magnesium recovery.

Conflict of interest

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research, and its results presented in this paper.

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Data availability

Data cannot be made available for reasons disclosed in the data availability statement.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the current work.

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