

UDC 669.2

DOI: 10.15587/1729-4061.2020.193885

REVERSE LEACHING OF MAGNESIUM FROM FERRONICKEL SLAG USING ALKALI SOLVENT NaOH

A. B. Prasetyo

Master of Engineering, Doctorate Candidate*

E-mail: chencen_abp@yahoo.com

D. Rahadian

Bachelor of Engineering**

W. Mayangsari

Master Candidate*

E. Febriana

Master of Science***

S. Permana

Doctor of Engineering*

A. Maksum

Doctor of Engineering

Department of Mechanical Engineering

Politeknik Negeri Jakarta

Depok 16424, Indonesia

O. Soesaptri

Master of Engineering**

F. Firdiyono

Doctor of Engineering, Professor***

J. W. Soedarsono

Doctor of Engineering, Professor*

*Department of Metallurgy and Material Engineering

Universitas Indonesia

Kampus Baru UI Depok, Jawa Barat, Indonesia, 16424

**Department of Metallurgy Engineering

Universitas Sultan Ageng Tirtayasa

Cilegon Banten, Indonesia

***Research Center of Metallurgy and Material

Indonesia Institute of Sciences

Building 470, Kawasan Puspiptek Serpong, Tangerang

Selatan 15314, Indonesia

Проведено дослідження з вилучення магнію з феронікелевого шлаку, обробленого зворотним вилуговуванням розчинами гідроксиду натрію (NaOH). Феронікелевий шлак в основному складається з силікату магнію і силікату заліза. Першим етапом дослідження була підготовка феронікелевого шлаку до подрібнення за допомогою кульового млина до розміру –200 меш. По-друге, прожарювання феронікелевих шлаків для видалення кристалічної води і збільшення пористості для полегшення процесу вилуговування. Наступним кроком було зворотне вилуговування з використанням гідроксиду натрію (NaOH) для розчинення кремнезему. При розчиненні кремнезему очікувалося збільшення вмісту в залишку таких елементів, як магній і залізо. Змінними в даному дослідженні з вилуговування феронікелевого шлаку були час вилуговування, концентрація розчинника і температура вилуговування. Зворотне вилуговування феронікелевого шлаку проводили зі зміною часу від 15 до 240 хвилин, температурою 30 °C, 70 °C і 100 °C, концентраціями NaOH 9 M, 10 M і 11 M. Для вивчення вихідних характеристик феронікелевого шлаку і результатів процесу вилуговування використовували рентгеноструктурний аналіз, рентгенофлуоресцентний аналіз і мас-спектрометрію з індуктивно-зв'язаною плазмою. Результати визначення характеристик зразків феронікелевого шлаку методом рентгеноструктурного аналізу показують, що в складі домінуючих з'єднань присутні форстерит (Mg_2SiO_4), енстатит ($MgSiO_3$) і фаяліт (Fe_2SiO_4). Крім того, результати також підтверджуються рентгенофлуоресцентним аналізом і растровою електронною мікроскопією. Кількісний РФА аналіз показує, що феронікелевий шлак містить 45,69 % SiO_2 , 29,32 % MgO і 16,5 % Fe_2O_3 . Результати растрової електронної мікроскопії показують, що Mg, Si, Fe і O зв'язуються разом, що вказує на присутність силікату магнію і силікату заліза. Найбільший відсоток вилучення магнію становить 73,10 % в умовах експериментальної температури 100 °C протягом 240 хвилин, концентрації розчинника 10 M і швидкості перемішування 300 об/хв. Збільшення відсотка вилучення магнію обумовлено розчиненням кремнезему в процесі вилуговування. Розчинення кремнезему підтверджується наявністю гідроксиду магнію і гідроксиду заліза (II) в залишку, що показано рентгеноструктурним аналізом. Це призвело до значного збільшення вмісту MgO в залишку до 42,8 %, як показав рентгенофлуоресцентний аналіз. Крім того, растрова електронна мікроскопія показує, що Mg і O зв'язуються разом, що вказує на присутність MgO . Також можна визначити, що MgO є домінуючим

Ключові слова: феронікель, шлак, форстерит, магній, кремнезем, вилуговування, зворотне вилуговування, гідроксид натрію, фільтрат, залишок, % вилучення

Received date 03.01.2020

Accepted date 17.02.2020

Published date 29.02.2020

Copyright © 2020, A. B. Prasetyo, D. Rahadian, W. Mayangsari,

E. Febriana, S. Permana, A. Maksum, O. Soesaptri, F. Firdiyono, J. W. Soedarsono

This is an open access article under the CC BY license (<http://creativecommons.org/licenses/by/4.0>)

1. Introduction

Slag utilization is one of the ways that can be taken to extract valuable elements from secondary sources other than primary mineral sources. Some previous studies utilized secondary sources from byproducts of slags in the

metallurgical process [1–3]. Some secondary sources that can be used from the byproduct of slags in the metallurgical process are tin slags, steel slags, aluminum slags, and ferronickel slags. The ferronickel slag is a byproduct of the smelting of nickel ores categorized as hazardous and poisonous to the environment [4]. The current use of ferronickel slag is only

as construction material and most of its remaining slags are only piled around its environment [5–9].

The ferronickel slag still contains some valuable minerals such as silica, magnesium, nickel, iron, cobalt and some rare-earth elements [4, 10–14]. The major element content in the ferronickel slag is Si about 30 %, others are 20 % of Mg, 12 % of Fe, 1–2 % of Al, and other elements [15]. The condition where the content of magnesium is high, it is possible to extract its element from the ferronickel slags so that it will increase the value of the ferronickel slag waste. Magnesium can be used in several applications, for example, as aluminum alloys, foundries, steel desulfurization, and other applications in the chemical industry [16, 17]. The aluminum industry that uses magnesium as an alloy material aims to increase strength, ductility and corrosion resistance.

The explanation above is some of the utilization of processed ferronickel slag waste so that it gets a more economic value. From the available literature, it is explained that some of the metal content can still be taken for use. Especially Si and Mg which is a content that has high levels. So that the best technique is needed to separate the precious metals contained in the ferronickel slag waste.

2. Literature review and problem statement

Research that has been done to process ferronickel slag waste has been carried out by several researchers, both with pyrometallurgical, hydrometallurgical processes and a mixture of both pyro-hydrometallurgy. Several studies with pyrometallurgical processes are by roasting or reducing ferronickel slag with a mixture of additives NaOH [13], Na₂CO₃ [18] and Na₂SO₄ [12]. The roasting process is carried out by adding an additive of Na₂CO₃ for the decomposition of forsterite and enstatite into magnesium oxide and sodium silicate so that it will facilitate the process of separating precious metals, especially magnesium [4]. The hydrometallurgical process using the nickel slag leaching method was carried out at a leaching temperature of 140–200 °C with sulfuric acid as a leaching agent. Co and Ni metal extraction results obtained more than 97 % and Cu extraction of more than 95 % [19]. The pyro-hydrometallurgical process with the alkali roasting method is followed by leaching using water. The optimum percent extraction yield of 92.33 % under conditions of a roasting temperature of 600 °C for one hour, followed by washing water under conditions of research temperature at 50 °C for one hour at a ratio of 10 ml/g of liquid-solid [5].

Theoretically, the ferronickel extraction can be done by dissolving the silica with an acid or base solution to separate it with magnesium. Dissolving the silica using NaOH solution was carried out to separate silica from vanadium steel slag using the leaching process concentrated in the polytetrafluoroethylene (PTFE) autoclave at a temperature of 140–240 °C. Most of the silica can be dissolved effectively and just left 4.28 % low silica content in the residue [20]. Dissolving silica using NaOH solution at a lower temperature, 25–110 °C, can decrease energy consumption, however, it resulted in lower dissolved silica at about 31.48 % for 4 hours leaching process [21]. Increasing dissolution temperature can increase the solubility of silica from raw materials, thus it affects silica recovery. Moreover, the dissolution of ferronickel slag with acid was also carried out

by the hydro-pyrometallurgy process. The leaching process of ferronickel slag using the hydrochloride acid solution followed by the calcination process was carried out [22, 23]. The concentration of the solvent, leaching temperature and time as well as calcination condition affect the percentage of MgO resulted. Utilization of HCl in the leaching process at 30 °C for 30 minutes followed by the precipitation process using NH₄OH and calcination process at 600 °C for 30 minutes, resulted in 95 %MgO [23]. Increasing leaching temperature and time up to 80 °C and 24 hours resulted in the high content of Mg in the filtrate [22]. Furthermore, sulfuric acid [24, 25] and nitric acid were also used for the magnesium leaching process [24]. Even though dissolving ferronickel slag using the acid solution can dissolve magnesium directly, but an irregular tridimensional framework of silica gel would form in the filtrate and caused a dramatically longer filtration process to separate filtrate and residue from the leaching process previously [26]. Recently, the recovery of magnesium from ferronickel slag has been carried out by vacuum reduction followed by condensation at the temperature of 1573 K and 10 Pa for 3 hours. Silica and CaO were added as a reducing agent. Gaseous magnesium resulted from the process since it is the lightest metal present in the raw materials. 97.74 % Mg resulted from the condensation of the gaseous metal [27]. However, this process needs high energy consumption since the condition process is performed at high temperatures and vacuum. Moreover, additional equipment such as condenser is needed to convert gaseous magnesium resulted.

Silica separation is essential for higher results of magnesium recovery since it has the highest content in the ferronickel slag. Dissolving silica using NaOH solution is likely effective for those purposes since longer filtration processes can be avoided. Moreover, extraction in the autoclave needs higher energy than atmospheric leaching even though it allows it to obtain a higher recovery. However, lower energy for the optimum condition in the overall processes of magnesium recovery needs to discover to efficiency. In this study, determining optimum atmospheric leaching is a critical issue to be solved previously to obtain a high recovery of silica that is beneficial to a higher result of magnesium recovery subsequently.

3. The aim and objectives of the study

The aim of this study is to upgrade the value of ferronickel slag as a result of a byproduct of nickel ore processing, especially for magnesium element. To achieve this aim, the reverse leaching processes are carried out by varying the leaching temperature and time as well as the concentration of the NaOH solution used. It is expected to determine the optimum process condition of reverse leaching processes, then the highest magnesium recovery is accomplished.

To achieve that goal, the following was applied:

- characterization of ferronickel slag using XRF, XRD and SEM mapping;
- reverse leaching using NaOH to find the optimum leaching conditions for variations in the concentration of NaOH 9 M, 10 M and 11 M, temperature leaching 30, 70 and 100 °C and leaching time 15–240 minutes;
- characterization of residual results of reverse leaching using XRF, XRD and SEM mapping.

4. Materials and method for investigation of reverse leaching magnesium from ferronickel slag

4. 1. Materials and apparatus used in the experiment

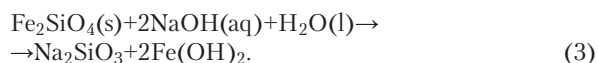
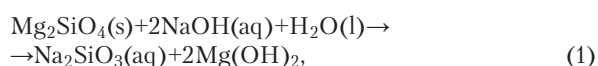
The sample used in this research is the ferronickel smelting slag from Morowali, Central Sulawesi, Indonesia. The ferronickel slag was crushed to obtain a grain size fraction of -200 mesh. The compound composition of the slag sample was analyzed using X-ray fluorescence (XRF) from the Bruker S2 Puma brand, while X-ray diffraction (XRD) analysis from Shimadzu MAXima XRD-7000 brand with Cu $K\alpha$ radiation ($\lambda=1.54$) was used to determine the composition in it. The SEM (Scanning Electron Microscope) mapping test from the JEOL Jsm 6390A brand was conducted to determine the mineralogical structure and the distribution of elements in the particles of the ferronickel slag sample.

4. 2. Procedure for conducting the experiments

The initial procedure of the study is the preparation of samples using a ball mill to get a grain size of -200 mesh by a grinding process. Secondly, the calcination process was carried out at a temperature of 800 °C for 60 minutes using a carbolite type of muffle furnace. The temperature and calcination time conditions are based on previous studies conducted from TG/DTA analysis towards ferronickel slag [4]. This calcination process is carried out to remove crystal water and to increase the porosity of the slag so it will facilitate the leaching process.

The next procedure is the process of reverse leaching using NaOH solutions. The reverse leaching of ferronickel slag was carried out with a time variation of 15 to 240 minutes, the temperature of 30 °C, 70 °C, and 100 °C and NaOH concentrations are 9 M, 10 M, and 11 M. The reverse leaching aims to dissolve the impurity which is silica so that it is separated from the valuable metal settle. The silica dissolved in the NaOH leaching agent reduces silica content in the residue so that it will increase the magnesium content. According to previous research, the reason for a leaching process using strong base solutions is to reduce the deposition of the metal elements that must be carried out if it is using acid solvents because valuable metals are found in the filtrate [21]. Moreover, the use of base solvents can reduce the damage of the leaching tank caused by acid solvents due to corrosion.

The leaching process reactions that might occur are as follows:



Reaction 1 shows the formation of sodium silicate solution (Na_2SiO_3) in the filtrate in the leaching process with NaOH while the solid magnesium hydroxide ($\text{Mg}(\text{OH})_2$) separated on the residue subsequently [28].

The next procedure is dissolving the residue from the leaching results with aqua regia and diluting it. Then, the results will be analyzed using ICP OES from Agilent Technologies 700 brand to determine the magnesium content.

5. Experiment result of the initial characterization of ferronickel slag, reverse leaching magnesium from ferronickel slag and characterization of residual results of reverse leaching

Sub-sections of the results of the study are as follows:

- 1) characterization of ferronickel slag with XRF, XRD, and SEM;
- 2) reverse Leaching uses NaOH with variations in leaching time, leaching temperature and NaOH concentration;
- 3) characterization of residue results from reverse leaching with NaOH.

5. 1. Initial characterization of the ferronickel slag samples

The initial characterization of ferronickel slag was carried out using the XRF analysis. XRF analysis was performed to determine the compounds contained in the ferronickel slag that is shown in Table 1. XRD analysis was performed to determine the composition of dominant compounds in the ferronickel slag. XRD analysis is shown in Fig. 1. SEM mapping analysis is used to determine the distribution of elements in the ferronickel slag, shown in Fig. 2.

Table 1

Data Results of XRF Analysis on the Ferronickel Slag

Compound	Content (%)
Na ₂ O	0.294
MgO	29.324
Al ₂ O ₃	4.811
SiO ₂	45.69
P ₂ O ₅	0.055
SO ₃	0.092
CaO	0.934
TiO ₂	0.121
Cr ₂ O ₃	1.403
MnO	0.575
Fe ₂ O ₃	16.503
NiO	0.121
ZnO	0.035
V ₂ O ₅	0.010

Table 1 shows the results of the XRF analysis of the initial ferronickel slag with compounds including SiO₂, MgO, Fe₂O₃, Al₂O₃, Cr₂O₃ and the remaining compounds with small levels. Fig. 1 shows the graphs of the peaks XRD analysis results of ferronickel slag dominated by magnesium silicate and iron silicate compounds, namely forsterite (Mg_2SiO_4), enstatite (MgSiO_3), and fayalite (Fe_2SiO_4).

Fig. 2 shows the results of SEM mapping analysis of ferronickel slag. The distribution of the elements contained in the ferronickel slag, especially the distribution of Mg, Si, Fe, Al, and O can be seen.

The distribution of Mg, Si, and O is very strongly marked by a bright red color. From Fig. 2, *a-d*, it can be concluded that Mg, Si, and O are in the same area, showing that these elements form a bond as magnesium silicate. This is in accordance with the result of the XRD analysis in Fig. 1 that the main phase of the ferronickel slag is magnesium silicate.

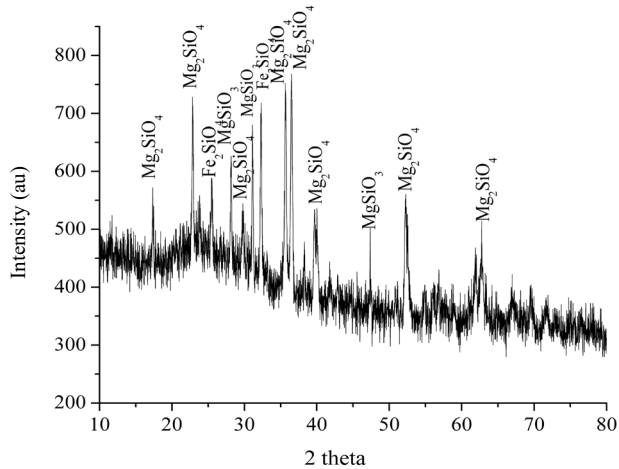


Fig. 1. Results of XRD characterization on ferronickel slag

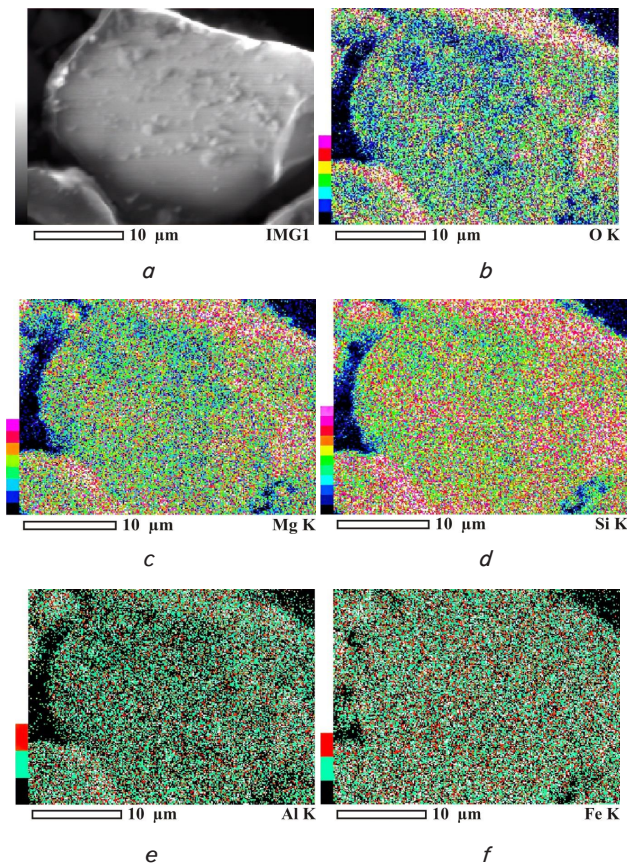


Fig. 2. SEM mapping towards the distribution of elements in the ferronickel slag: *a* – ferronickel slag; *b* – O distributions; *c* – Mg distributions; *d* – Si distributions; *e* – Al distributions; *f* – Fe distributions

5. 2. Effect of leaching time on magnesium extraction percentage

The leaching calcination result of ferronickel slag was carried out with a time variation of 15 to 240 minutes. The leaching process used variable temperature of 30 °C, 70 °C, and 100 °C. The ferronickel slag leaching process was carried out by the agitation leaching method using a hotplate with a magnetic stirrer, stirred at 300 rpm and consisted of 5 % solid. By doing a long time variation in the ferronickel slag leaching process, it is expected to affect the value produced

in magnesium extraction percentage. The NaOH concentrations are 9 M, 10 M, and 11 M.

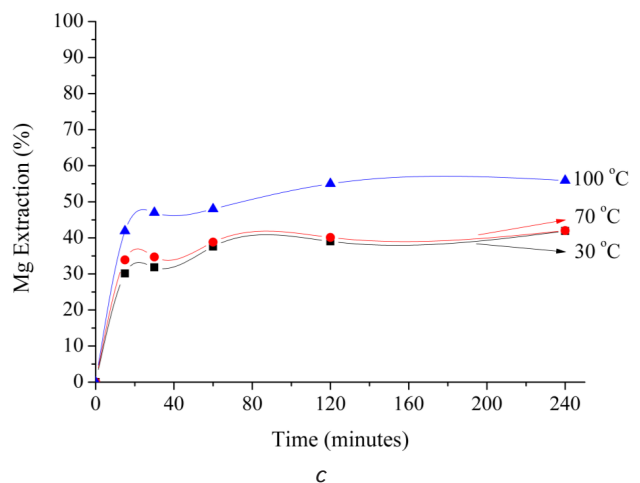
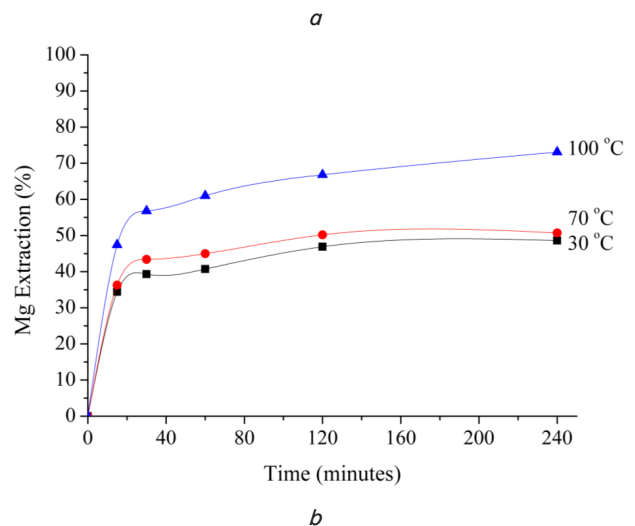
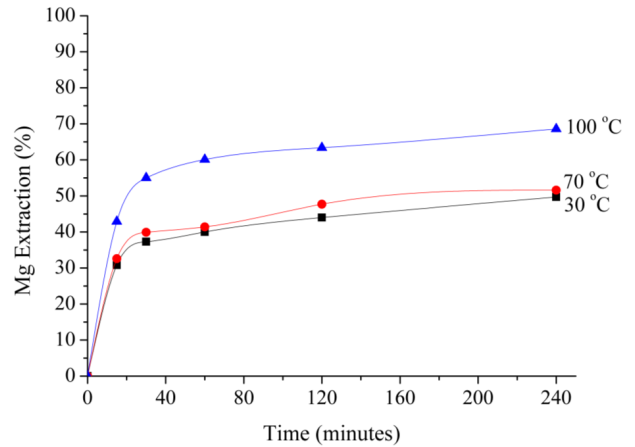


Fig. 3. Graph of the effect of leaching time on magnesium extraction percentage: *a* – 9 M, *b* – 10 M, *c* – 11 M

Fig. 3, *a-c* shows the effect of leaching time on the percentage of magnesium extraction from ferronickel slag. Fig. 3, *a-c* shows the percent extraction of magnesium obtained with 9 M, 10 M and 11 M of NaOH solution. The results show that the concentration of NaOH 9, 10 and 11 M at a leaching time of 240 minutes and the temperature of

100 °C increase the percent extraction of magnesium significantly.

5.3. Effect of leaching temperature on magnesium extraction percentage

The ferronickel slag leaching temperature variety ranges from 30 °C, 70 °C, and 100 °C. Leaching was performed using a fixed variable leaching time of 240 minutes, stirring speed of 300 rpm and percent solid 5 %. Fig. 4 shows a graph of the effect of temperature on the percent of magnesium extraction found in ferronickel slag with a concentration variable of NaOH solution and a fixed variable of leaching time for 240 minutes.

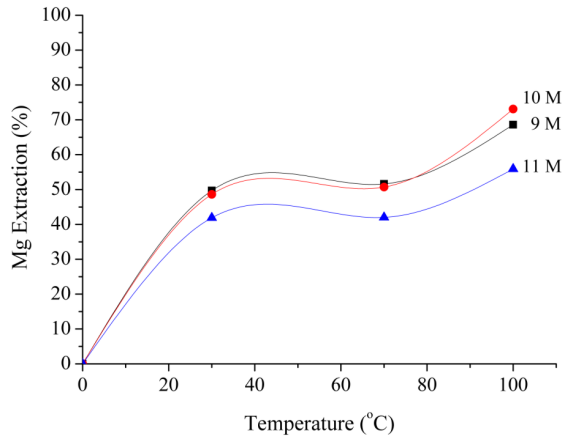


Fig. 4. Graph of the effect of leaching temperature on magnesium extraction percentage

The best results obtained by magnesium extraction occurred at a temperature of 100 °C at a concentration of 10 M.

5.4. Effect of solvent concentration on magnesium extraction percentage

The solvent concentration in the ferronickel slag leaching process varies from 9 M, 10 M, and 11 M at 100 °C of a leaching temperature with 300 rpm of stirring speed and percent solid 5 %. It can be seen in Fig. 5 that the highest magnesium extraction value is when 10 M NaOH concentration is used as a solvent in the leaching process.

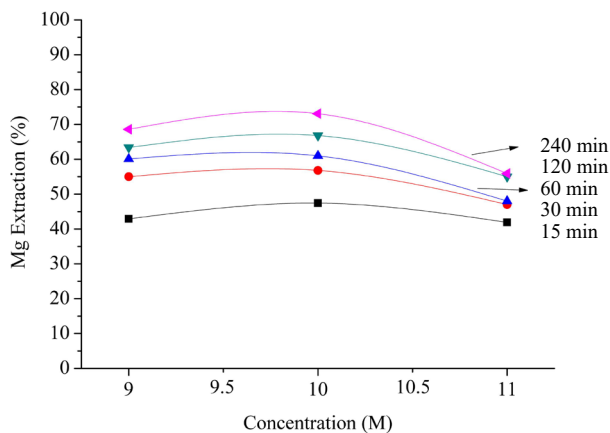


Fig. 5. Graph of the solvent concentration effect on magnesium extraction percentage

Fig. 5 shows the effect of different concentrations of NaOH solution on the percent extraction of magnesium with

a variable leaching time of 15–240 minutes and a temperature of 100 °C.

5.5. Characterization of ferronickel slag residues after leaching process

After identifying the compounds and compositions contained in the residue, the best results of the leaching process occurred under research conditions of 10 M solvent concentration at a temperature of 100 °C and for 240 minutes with 300 rpm of stirring speed and percent solid 5 %. The silica that associates with magnesium is still detected even though its intensity is less than before the dissolving.

Table 2

XRF analysis results towards residue samples after the leaching process

Compound	Content (%)
Na ₂ O	3.467
MgO	42.886
Al ₂ O ₃	7.78
SiO ₂	32.19
SO ₃	0.096
CaO	0.946
TiO ₂	0.143
Cr ₂ O ₃	1.307
MnO	0.695
Fe ₂ O ₃	10.295
NiO	0.051
ZnO	0.018
V ₂ O ₅	0.034

Table 2 shows the results of the XRF analysis of the residues from the leaching process with the MgO as greater the compound formed. Fig. 6 shows the results of XRD characterization of the residual leaching results from the ferronickel slag which produces compounds such as magnesium hydroxide and iron hydroxide.

Fig. 6. XRD characterization results of ferronickel slag after

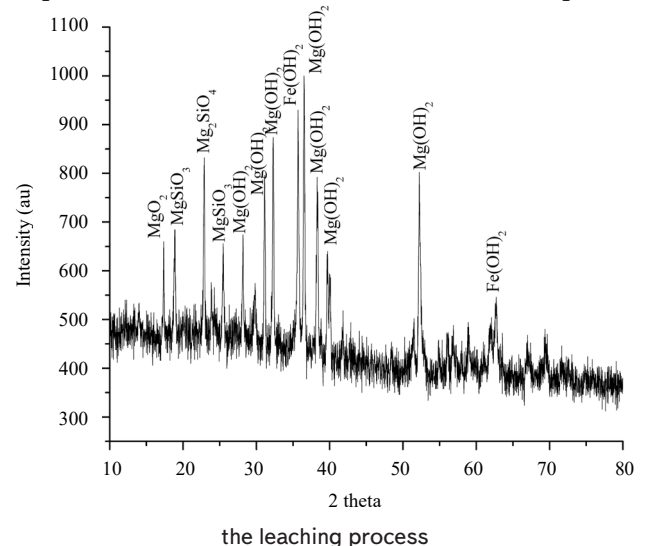


Fig. 7 shows the results of SEM mapping analysis of the distribution of elements in the residue from the reverse

leaching process. The distribution of Mg and O dominates in spots marked in bright red.

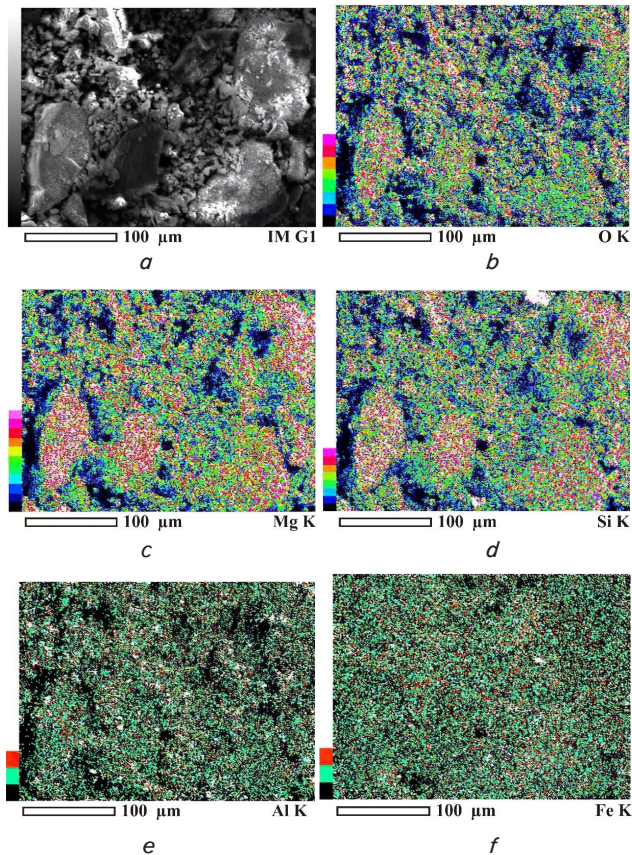


Fig. 7. SEM mapping towards the distribution of elements in the ferronickel slag after leaching:
a – residue; *b* – O distributions; *c* – Mg distributions;
d – Si distributions; *e* – Al distributions;
f – Fe distributions

6. Discussion of the results of reverse leaching magnesium from ferronickel slag

Characterizations of raw ferronickel slag, effect of leaching time and temperature as well as a concentration of NaOH solution are discussed here. Moreover, the residue which was resulted from the leaching process is also characterized to determine the effect of a series processing carried out.

Characterizations of ferronickel slag were performed by three kinds of analysis consisting of XRF, XRD and SEM mapping that have conformance with each other. Table 1, it is known that the dominant contained compound in ferronickel slag is silica (SiO_2) 45.69 %. Other elements contained in ferronickel slag are magnesium, iron, and aluminum with the content of 29.32 %, 16.5 %, and 4.8 % respectively. These XRF analysis results are consistent with the previous study that carried out the analysis even though it has a different use of sources in the analyzed samples [21]. The compounds identified in the ferronickel slags are associated with different mineral phases that were characterized using X-Ray diffraction instruments shown in Fig. 1. The composition of silica is SiO_2 forsterite (Mg_2SiO_4), enstatite (MgSiO_3) and fayalite (Fe_2SiO_4) which are dominant. This can be seen in the graphs of Fig. 1 that the three

strongest diffraction peaks are forsterite (Mg_2SiO_4), fayalite (Fe_2SiO_4) and enstatite (MgSiO_3) compounds. This XRD result is appropriate with previous researchers who have characterized the ferronickel slag [15, 18, 22]. Moreover, morphological structure and mapping from SEM-EDS analysis, Fig. 2, have also a similar tendency that show these phases from XRD analysis. Fig. 2, *a* shows that the ferronickel slag is dense and glossy. Based on the mapping analysis, the elements are distributed throughout the area. However, Fig. 2, *a-c* show O, Mg and Si mapping elements are distributed on the edge area. It can be interpreted as Mg_2SiO_4 and MgSiO_3 phases. Meanwhile, Fig. 2, *d, e* shows Al and Fe distribution throughout the area and can be interpreted associating in the Al_2O_3 and Fe_2SiO_4 phases.

In Fig. 3, *a-c* it appears that the longer the leaching process time, the higher the percent extraction produced. It can be seen in Fig. 3, *a*, on the addition of 9 M concentration of sodium hydroxide, the highest percent extraction was obtained at 240 minutes leaching process time with 100 °C of temperature condition and produced 68.62 % percent extraction, while the lowest percent extraction was obtained at 15 minutes leaching process time with 30 °C of temperature condition and produced 30.77 % percent extraction. In Fig. 3, *b*, on the addition of 10 M concentration of sodium hydroxide, the highest percent extraction was obtained at 240 minutes leaching process time with 100 °C of temperature condition and produced 73.10 % percent extraction, while the lowest percent extraction was obtained at 15 minutes leaching process time with 30 °C of temperature condition and produced 34.42 % of magnesium extraction percentage. In Fig. 3, *c*, on the addition of 11 M concentration of sodium hydroxide, the highest percent extraction was obtained at 240 minutes leaching process time with 100 °C of temperature condition and produced 55.92 % percent extraction, while the lowest percent extraction was obtained at 15 minutes leaching process time with 30 °C of temperature condition and produced 30.13 % of magnesium extraction percentage. This is appropriate with the previous research which states that the longer the leaching time, the more silica dissolves in sodium hydroxide as there is more opportunity to contact reactions between the sodium hydroxide samples. Silica that is dissolved in the solutions increases and causes an increment of magnesium content in the residue from the leaching results [21].

It can be seen in Fig. 4 that the magnesium extraction percentage increases along with the rise of the leaching temperature. In heterogeneous reactions, the increment of temperature is one of the parameters that greatly affect the solubility rate in the leaching process with reactions controlled by chemical reactions [29]. Fig. 4 shows that the higher the leaching temperature, the higher the magnesium extraction percentage produced. On the addition of 9 M NaOH concentration, it can be seen that the highest percent extraction was obtained at 100 °C of temperature condition and produced 68.62 % percent extraction, while the lowest percent extraction was obtained at 30 °C of temperature condition and produced 49.65 % magnesium extraction percentage. Then, on the addition of 10 M NaOH concentration, it is shown that the highest percent extraction was obtained at 100 °C of temperature condition and produced 73.10 % percent extraction, while the lowest percent extraction was obtained at 30 °C of temperature condition and produced 48.62 % magnesium extraction

percentage. On the addition of 11 M NaOH concentration, it is shown that the highest percent extraction was obtained at 100 °C of temperature condition and produced 55.92 % percent extraction, while the lowest percent extraction was obtained at 30 °C of temperature condition and produced 41.86 % magnesium extraction percentage. The increment of magnesium extraction percentage in the reverse leaching process is due to the rise of silica solubility in the NaOH solution as the process temperature increases. This is appropriate with the theory that the solubility rate is directly proportional to the temperature [29]. These results are consistent with the studies that have extracted silica from olivine with a basic solution, that the higher the temperature of the silica extraction results, the higher the magnesium content increase in residues [28].

Fig. 5 shows that the percent extraction of magnesium increased from 9 M concentration of sodium hydroxide to 10 M concentration of sodium hydroxide and then decreased at 11 M concentration of sodium hydroxide. A similar trend occurred in previous studies which stated that the dissolution of silicon from the ferronickel slag increased significantly with increasing NaOH concentration until it got a certain concentration, then decreased significantly as well [11, 21]. They conclude that the higher the concentration used in the solutions, the aluminosilicate precipitate formation will occur which can inhibit the silica dissolution at its higher NaOH concentrations. Aluminosilicate does not dissolve in the leaching agent but it settles in the residue with magnesium and iron so that it will cause the decrease of magnesium contents [11, 21].

Characterizations to the residue resulted from the leaching process were performed by XRF, XRD and SEM-EDS analysis. It can be compared with the characterizations of raw ferronickel slag. Fig. 6 is the XRD analysis results showing that magnesium hydroxide and iron(II) hydroxide are formed as a result of reverse leaching on the ferronickel slag. This is appropriate to the reaction (1)–(3), in which the magnesium hydroxide and iron hydroxide are formed. It also indicated that the sodium silicate solution is formed in the ferronickel slag reverse leaching process using NaOH and be separated into the filtrate. The silica is not dominant because a lot of silica has been dissolved during the leaching process with NaOH solutions. It can be proved by XRF analysis, which can be seen in Table 2. The XRF result shows the magnesium content in the residue that resulted from the leaching process on ferronickel slag increased significantly to 42.8 % and silica content decreased to 32 %. This is consistent with XRD analysis, which shows the dominant peak of magnesium hydroxide $\text{Mg}(\text{OH})_2$. However, silica still exists in the form of Mg_2SiO_4 . Moreover, the existence of $\text{Mg}(\text{OH})_2$ and Mg_2SiO_4 can also be proved by SEM and mapping analysis that is shown in Fig. 7, *a–f*. Fig. 7, *a* shows the morphology of leaching residue that is more porous in some parts than the raw ferronickel, even though dense morphology still exists. It indicated that the dissolution of silica does not occur completely. Fig. 7, *b–d* indicated the presence of $\text{Mg}(\text{OH})_2$ and Mg_2SiO_4 in both forms, porous and dense. The dense one indicates silica that does not dissolve in the NaOH solution through the leaching process. Fig. 7, *e, f* shows the aluminum and iron that are still distributed entirely in the area. It indicated that both of the elements are just slightly affected by the leaching process using NaOH solu-

tion. However, the mapping of the aluminum shows the accumulation in some areas that might be growth when the calcination process is carried out. Based on the characterizations of leaching residue, it can be discovered that high magnesium content and low silica content resulted from calcination followed by the leaching process using NaOH solution.

Based on the results of this study, extraction of magnesium from ferronickel slag by calcination followed by the reversed leaching process using NaOH solution can produce percent extraction of magnesium up to 73.10 %. However, the calcination process was performed at high temperature, which needs high energy consumption. The issue of energy is attractive to be solved. The lower the energy consumption, the more profitable it will be.

Process development can be carried out to increase the percent extraction of magnesium. The presence of Mg_2SiO_4 and MgSiO_3 shows that silica does not dissolve completely in the leaching process. Therefore, the optimum process to dissolve the whole silica from ferronickel needs to determine previously to obtain higher percent extraction of Mg, however it is still challenging to solve. Moreover, water glass results from the leaching process. It provides the additional advantage of the process since another beneficial product, precipitated silica, can be resulted. However, the precipitation process by acid and filtration processes must be added subsequently.

7. Conclusions

1. The result of XRD shows ferronickel slag is composed of forsterite (Mg_2SiO_4), enstatite (MgSiO_3) and fayalite (Fe_2SiO_4) structures. The XRF analysis showed that most of the ferronickel slag contains 45.69 % of SiO_2 , 29.32 % of MgO and 16.5 % of Fe_2O_3 , and any other oxides leftover. The SEM mapping analysis shows the distribution of elements of Mg, Si, and O is very dominant.

2. The results showed that the highest percentage of magnesium extraction was 73.10 % under experimental temperature conditions of 100 °C, 10 M of solvent concentration for 240 minutes with 300 rpm of stirring speed and percent solid 5 %.

3. The XRD analysis results of the residue show that magnesium hydroxide and iron(II) hydroxide are formed as a result of reverse leaching on the ferronickel slag. The XRF result shows the magnesium content contained in the residue during the leaching process on ferronickel slag increased significantly to 42.8 % and silica content decreased to 32 %. The results of the SEM mapping analysis showed that Mg and Si bonds were not dominant because most of the silica dissolved as filtrate.

Acknowledgments

The author would like to thank the Ministry of Research and Higher Education as a provider of scholarships and financial support through the Doctoral Dissertation Research Grant with contract number 234/PKS/R/UI/2019. In addition, this research was also supported by the Research Center for Metallurgy and Materials, Indonesian Institute of Sciences with its laboratory facilities.

References

1. Permana, S., Vincia, S. F., Amilia, A., Maksum, A., Widana, K. S., Soedarsono, J. W. (2018). Enrichment on Bangka tin slag's tantalum and niobium oxide contents through non-fluoride process. *Eastern-European Journal of Enterprise Technologies*, 5 (12 (95)), 56–64. doi: <https://doi.org/10.15587/1729-4061.2018.129914>
2. Piatak, N. M. (2018). Environmental Characteristics and Utilization Potential of Metallurgical Slag. *Environmental Geochemistry: Site Characterization, Data Analysis and Case Histories*, 487–519. doi: <https://doi.org/10.1016/b978-0-444-63763-5.00020-3>
3. Ma, N., Houser, J. B. (2014). Recycling of steelmaking slag fines by weak magnetic separation coupled with selective particle size screening. *Journal of Cleaner Production*, 82, 221–231. doi: <https://doi.org/10.1016/j.jclepro.2014.06.092>
4. Prasetyo, A. B., Maksum, A., Soedarsono, J. W., Firdiyono, F. (2019). Thermal characteristics of ferronickel slag on roasting process with addition of sodium carbonate (Na₂CO₃). *IOP Conference Series: Materials Science and Engineering*, 541, 012037. doi: <https://doi.org/10.1088/1757-899x/541/1/012037>
5. Gu, F., Zhang, Y., Peng, Z., Su, Z., Tang, H., Tian, W. et. al. (2019). Selective recovery of chromium from ferronickel slag via alkaline roasting followed by water leaching. *Journal of Hazardous Materials*, 374, 83–91. doi: <https://doi.org/10.1016/j.jhazmat.2019.04.002>
6. Huang, Y., Wang, Q., Shi, M. (2017). Characteristics and reactivity of ferronickel slag powder. *Construction and Building Materials*, 156, 773–789. doi: <https://doi.org/10.1016/j.conbuildmat.2017.09.038>
7. Saha, A. K., Sarker, P. K. (2017). Sustainable use of ferronickel slag fine aggregate and fly ash in structural concrete: Mechanical properties and leaching study. *Journal of Cleaner Production*, 162, 438–448. doi: <https://doi.org/10.1016/j.jclepro.2017.06.035>
8. Choi, Y. C., Choi, S. (2015). Alkali–silica reactivity of cementitious materials using ferro-nickel slag fine aggregates produced in different cooling conditions. *Construction and Building Materials*, 99, 279–287. doi: <https://doi.org/10.1016/j.conbuildmat.2015.09.039>
9. Gu, F., Peng, Z., Zhang, Y., Tang, H., Ye, L., Tian, W. et. al. (2018). Facile Route for Preparing Refractory Materials from Ferronickel Slag with Addition of Magnesia. *ACS Sustainable Chemistry & Engineering*, 6 (4), 4880–4889. doi: <https://doi.org/10.1021/acssuschemeng.7b04336>
10. Tangahu, B. V., Warmadewanthi, I., Saptarini, D., Pudjiastuti, L., Tardan, M. A. M., Luqman, A. (2015). Ferronickel Slag Performance from Reclamation Area in Pomalaa, Southeast Sulawesi, Indonesia. *Advances in Chemical Engineering and Science*, 05 (03), 408–412. doi: <https://doi.org/10.4236/aces.2015.53041>
11. Maragkos, I., Giannopoulou, I. P., Panias, D. (2009). Synthesis of ferronickel slag-based geopolymers. *Minerals Engineering*, 22 (2), 196–203. doi: <https://doi.org/10.1016/j.mineng.2008.07.003>
12. Patrick, J., Prasetyo, A. B., Munir, B., Maksum, A., Soedarsono, J. W. (2018). The effect of addition of sodium sulphate (Na₂SO₄) to nickel slag pyrometallurgical process with temperature and additives ratio as variables. *E3S Web of Conferences*, 67, 03053. doi: <https://doi.org/10.1051/e3sconf/20186703053>
13. Pangaribuan, R. H., Patrick, J., Prasetyo, A. B., Maksum, A., Munir, B., Soedarsono, J. W. (2018). The effect of NaOH (sodium hydroxide) to slag nickel pyrometallurgy in different temperature and additive ratio. *E3S Web of Conferences*, 67, 03052. doi: <https://doi.org/10.1051/e3sconf/20186703052>
14. Perederiy, I. (2011). Dissolution of Valuable Metals from Nickel Smelter Slags by Means of High Pressure Oxidative Acid Leaching. University of Toronto, 122.
15. Demotica, J. S., Jr., R. F. A., Malaluan, R. M., Demayo, C. G. (2012). Characterization and Leaching Assessment of Ferronickel Slag from a Smelting Plant in Iligan City, Philippines. *International Journal of Environmental Science and Development*, 3 (5), 470–474. doi: <https://doi.org/10.7763/ijesd.2012.v3.269>
16. Survey, G. (2016). Mineral commodity summaries 2016. 2016: Government Printing Office.
17. Royani, A., Sulistiyono, E., Prasetyo, A. B., Subagja, R. (2018). Extraction of magnesium from calcined dolomite ore using hydrochloric acid leaching. doi: <https://doi.org/10.1063/1.5038299>
18. Setyabrata, A. C., Maksum, A., Prasetyo, A. B., Priyono, B., Wahyuadi Soedarsono, J. (2019). Effect of Sodium Carbonate on the Reduction Process of Nickel Slag from Sulawesi. *IOP Conference Series: Materials Science and Engineering*, 553, 012028. doi: <https://doi.org/10.1088/1757-899x/553/1/012028>
19. Huang, F., Liao, Y., Zhou, J., Wang, Y., Li, H. (2015). Selective recovery of valuable metals from nickel converter slag at elevated temperature with sulfuric acid solution. *Separation and Purification Technology*, 156, 572–581. doi: <https://doi.org/10.1016/j.seppur.2015.10.051>

20. Xiao, Q., Chen, Y., Gao, Y., Xu, H., Zhang, Y. (2010). Leaching of silica from vanadium-bearing steel slag in sodium hydroxide solution. *Hydrometallurgy*, 104 (2), 216–221. doi: <https://doi.org/10.1016/j.hydromet.2010.06.007>
21. Mufakhir, F. R., Mubarak, M. Z., Ichlas, Z. T. (2018). Leaching of silicon from ferronickel (FeNi) smelting slag with sodium hydroxide solution at atmospheric pressure. *IOP Conference Series: Materials Science and Engineering*, 285, 012003. doi: <https://doi.org/10.1088/1757-899x/285/1/012003>
22. Mubarak, M. Z., Yudiarto, A. (2017). Synthesis of Magnesium Oxide from Ferronickel Smelting Slag Through Hydrochloric Acid Leaching-Precipitation and Calcination. *Energy Technology* 2017, 247–258. doi: https://doi.org/10.1007/978-3-319-52192-3_24
23. Chu, Y.-S., Lim, Y.-R., Park, H.-B., Song, H., Lee, J.-K., Lee, S.-H. (2010). Extraction of Mg ion and Fabrication of Mg Compound from Ferro-Nickel Slag. *Journal of the Korean Ceramic Society*, 47 (6), 613–617. doi: <https://doi.org/10.4191/kcers.2010.47.6.613>
24. Son, H.-J., Lee, W.-K. (2015). Leaching Characteristics of Magnesium from Mine Residues by H₂SO₄ and HNO₃. *Proceedings of the World Congress on New Technologies*.
25. Song, H.-Y., Seo, J.-B., Kang, S.-K., Kim, I.-D., Choi, B.-W., Oh, K.-J. (2014). CO₂Fixation by Magnesium Hydroxide from Ferro-Nickel Slag. *Clean Technology*, 20 (1), 42–50. doi: <https://doi.org/10.7464/ksct.2014.20.1.042>
26. Stopic, S., Dertmann, C., Koiwa, I., Kremer, D., Wotruba, H., Etzold, S. et. al. (2019). Synthesis of Nanosilica via Olivine Mineral Carbonation under High Pressure in an Autoclave. *Metals*, 9 (6), 708. doi: <https://doi.org/10.3390/met9060708>
27. Zhang, X., Gu, F., Peng, Z., Wang, L., Tang, H., Rao, M. et. al. (2019). Recovering Magnesium from Ferronickel Slag by Vacuum Reduction: Thermodynamic Analysis and Experimental Verification. *ACS Omega*, 4 (14), 16062–16067. doi: <https://doi.org/10.1021/acsomega.9b02262>
28. Raza, N., Raza, W., Madeddu, S., Agbe, H., Kumar, R. V., Kim, K.-H. (2018). Synthesis and characterization of amorphous precipitated silica from alkaline dissolution of olivine. *RSC Advances*, 8 (57), 32651–32658. doi: <https://doi.org/10.1039/c8ra06257a>
29. Habashi, F. (1997). *Handbook of extractive metallurgy*. Wiley-Vch.