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Selective reduction for nickel laterite using sodium sulfate and palm kernel shell charcoal followed by magnetic separation

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Abstract The selective reduction process of laterite nickel ore using sodium sulfate (Na_2SO_4) and palm shell charcoal has been studied to enhance the separation of nickel from impurities at high temperatures**.** This study aims to optimize the reduction process of laterite nickel by utilizing sodium sulfate (10% by weight) as fluxing agent and palm shell charcoal (5% by weight) as an environmentally friendly carbon source to achieve efficient reduction. The study used variables such as particle size fraction (-60# (mesh) +80# (mesh), -80 (mesh) #+100# (mesh), and -100# (mesh)). Each particle size fraction was then formed into pellets and reduced at temperature of 950 °C, 1050 °C, and 1150 °C for 60 minutes by using a muffle furnace. Magnetic separation was subsequently performed using varying magnetic field strengths of 500 G, 1000 G, and 1500 G. the results yielded ferronickel concentrate (magnetic) and tailing (non-magnetic). The optimal result with the highest nickel concentrate (5,332%) was obtained under high temperature condition (1150 °C), particle size fraction of -80# (mesh) +100# (mesh), and a magnetic field strength of 1000 G.

1. Introduction

Nickel is the fifth most abundant element on Earth with high concentrations in the Earth's core and lowest concentrations in the Earth's crust. Naturally occurring nickel is generally found in the form of oxides (laterite), sulfides, or silica. Nickel ore is mined in approximately 25 countries across all continents, and smelting or refining is carried out in approximately 26 countries as of 2024 [1]. Primary nickel is produced and utilized in various forms, such as ferronickel, nickel oxide, nickel pig iron (NPI), nickel sulfate, other chemicals, and pure nickel metal in various degrees of purity. Furthermore, nickel is easily recycled in many of its applications, and large quantities of secondary nickel or scrap used to supplement the supply of newly mined nickel ore [2].

Production of nickel oxide (laterite) is lower when compared to nickel sulfide production. This is caused by the processing of nickel oxide (laterite) ore which is more difficult than nickel sulfide [3]. The difficulty of processing nickel oxide can be seen from the high cost of processing nickel oxide compared to nickel sulfide [4].

The high costs required to process nickel oxide (laterite) are due to the fact that laterite nickel ore has a more complex mineral and chemical structure than nickel sulfide, so more stages are required in processing laterite nickel ore [5]. Laterite nickel ore is generally processed using pyrometallurgy [6]. However, this process requires very high energy consumption because it involves melting at temperatures between 1500 ºC and 1600 ºC [7].

Due to the high energy consumption of nickel laterite processing using pyrometallurgy, a selective reduction method has been developed. This method involves reducing the laterite nickel ore at a specific temperature, followed by magnetic separation, and then pyrometallurgy. This reduction process is carried out at low temperatures, ranging from 1100 ºC to 1200 ºC, with the addition of a number of additives to the laterite nickel ore [8].

The reduction reaction that occurs during the selective reduction process can be seen in the equation below [9].

$$2Fe_3O_4 + 2Co(g) = 6FeO + 2CO_2(g) \quad (1)$$
$$2/5Fe_3O_4 + S_2(g) = 6/5FeS + 4/5SO_2(g) \quad (2)$$
$$4/3FeO + S_2(g) = 4/3FeS + 2/3SO_2(g) \quad (3)$$
$$2FeO + 2CO(g) = 2Fe + 2CO_2(g) \quad (4)$$
$$2NiO + 2CO(g) = 2Ni + 2CO_2(g) \quad (5)$$

The use of selective reduction methods in the processing of laterite nickel ore is still widely developed, by reducing laterite nickel ore by adding a number of reducing agents and additives to limit the

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reduction of iron oxide to iron metal, thus obtaining a ferronickel concentrate that is richer in nickel. Previous research conducted by Cao (2010); Li (2021); and Zhao (2012) used coal as a reducing agent with additives Sodium Carbonate, Sodium Sulfate, and Calcium Sulfate. At Hafid (2022), similar research has been done with adding NaCl as additive with exactly same methode, instrument, and variabels.

The addition of sulfate additives increases the grain size of ferronickel, thereby increasing the degree of liberation of ferronickel from impurities [10]. The use of sodium sulfate gives better results compared to sodium carbonate and chloride [11]. Sodium sulfate reduces Fe metallization by sulfidation reaction by forming FeS compound [12].

Indonesia, as one of the largest palm oil producer, produces palm kernel shell charcoal, a byproduct of its palm oil processing industry, which was used as a reducing agent in this study [13]. Palm kernel shell charcoal was used as a reducing agent because it is relatively more economical than coal, yet still has a high fixed carbon content which is quite large, namely 77% [3].

Research on selective reduction using palm shell charcoal as a reducing agent has been conducted previously, however the emphasis in this research is on the influence of the grain size of laterite nickel ore, reduction temperature, and the strength of the magnetic field as well as the use of additives in the form of sodium sulfate which are predicted to have an effect on the selective reduction process of laterite nickel from Southeast Sulawesi, which not many studies have discussed previously [9].

2. Method

2.1 Material

The raw material used in the research was limonite laterite nickel ore from Southeast Sulawesi. The chemical composition of the research raw material (as-received) with a content of 50.5% Fe and 1.4% Ni was determined by XRF, while palm kernel shell charcoal is used as a reducing agent. Sodium sulfate (Na) is used as an additive (Na_2SO_4) pro-analysis (p.a). The use of sodium sulfate containing sulfur compounds aims to increase the content and recovery of nickel in the final separation results [14]. Previous research done by Fajar et al [11, 14] showed that using palm kernell shell charcoal as reducing agent and sodium sulfate (Na_2SO_4) as an additive could increase the nickel grade up to 6%, while this study focuses on the effects of grain size, reduction temperature, and magnetic field strength.

2.2 Reduction Process

The reaction equation for selective nickel reduction using sodium sulfate additive is shown in equation (6–10) [12].

$$Na_2SO_4 + 4CO(g) = Na_2S + 4CO_2(g) \quad (6)$$

$$Na_2S + FeO + SiO_2(g) = Na_2SiO_3 + FeS(g) \quad (7)$$

$$NaS + NiO + SiO_2(g) = Na_2SiO_3 + NiS(g) \quad (8)$$

$$NiS + FeO + CO(g) = FeS + Ni + CO_2(g) \quad (9)$$

$$Na_2SO_4 + SiO_2 + CO(g) = Na_2SiO_3 + SO_2 + CO_2(g) \quad (10)$$

The sample repair process begins by grinding the laterite nickel ore with 1,2% nickel grade to a size of (60+80#, -80+100#, and -100#). Palm kernel shell charcoal is used as a reducing agent and Na_2SO_4 as an additive, ground to a size of -100#. The three ingredients are then mixed and stirred until evenly distributed and agglomerated into a powder form pellet with 10 – 15mm diameters. The pellet then reduced by using a muffle furnace at temperatures of 950 °C, 1050 °C, and 1150 °C for 60 minutes [7].

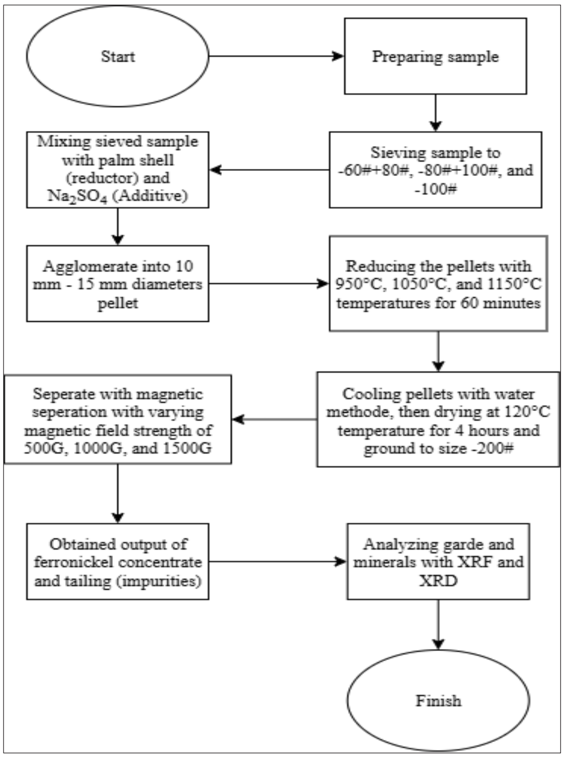

Fig. 1 Flowchart of the research

After being reduced, pellet then cooled using the water method (water quench), then continued with drying at a temperature of 120 °C for 4 hours and ground to a size of -200#. Next, separation is carried out using the method magnetic separation in a wet state with varying magnetic field strengths of 500 G, 1000 G, and 1500 G, until obtained output in the form of ferronickel concentrate which is magnetic and tailing (impurities) that are non-magnetic. Nickel grade determined by XRF and nickel recovery was calculated based on the ratio of nickel content in the magnetic concentrate to the total nickel content in the feed material, considering both magnetic and nonmagnetic fractions.

To determine the effect caused by grain size, the selective reduction process was carried out at a temperature of 1050 °C with the addition of additives in the form of Na_2SO_4 as much as 10% by weight and palm shell charcoal as much as 5%. Reduction time is a fixed variable, while the variable that changes is the grain size and magnetic field strength.

To determine the effect caused by reduction temperatures of 950 °C, 1050 °C, and 1150 °C on the selective reduction process, an experiment was conducted using laterite nickel ore as-received plus Na_2SO_4 as much as 10% of the weight and a reducing agent in the form of palm shell charcoal as much as 5% of the weight. The operating variable of the magnetic field strength used is 1000 G.

To determine the effect of magnetic field strength, the selective reduction process was carried out at a temperature of 1050 °C with the addition of additives in the form of Na_2SO_4 as much as 10% by weight and palm shell charcoal as much as 5%. Reduction time is a fixed variable, while the variable that changes is the grain size and magnetic field strength.

2.3 Testing

Testing is carried out to determine the levels of elements contained in raw materials, concentrates, and tailing. Based on research results using an XRF Microstructural analysis was performed using an XRD tester to determine the phase composition and morphological structure of the resulting ferronickel. The elemental content of the research raw materials can be seen in Table 1 (by XRF) and Fig. 2 (by XRD) below.

Table 1. The results of the analysis of raw materials using XRF (as-received)

Element	% Weight
Ni	1,4
Fe	50,5
Si	16,5
Mg	1,81
Al	4,86
Ca	0,177
Cr	2,68

The mineral content of the research raw materials can be seen in Fig. 2 below.

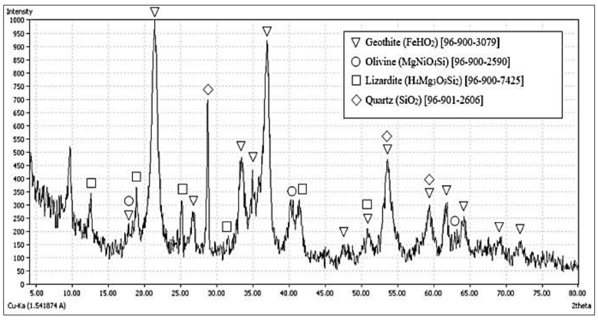

Fig. 2 XRD analysis results for raw material using XRD (as-received)

The XRD results (Fig. 2) on the laterite nickel ore sample showed that this ore is limonite nickel ore, which is dominated by geothite, olivine, lizardite, and quartz compounds. The presence of these compounds is evident from the peaks that appear on the graph. So, it can be concluded that the sample can be include in the limonite nickel ore category.

2.4 Characterization

Characterization was carried out at the beginning of the research for ore samples and after the reduction and magnetic separation processes. Characterization is carried out to determine the characteristics of the compounds and chemical elements contained, as well as the surface morphology of the reduced samples. Samples were analyzed using several analytical tools, such as X-Ray Diffraction (XRD) X'Pert Highscore type from PAN, X-Ray Fluoroscence (XRF) Portable, optical microscope, and Scanning Electro Microscopy-Energy Dispersive Spectroscopy (SEMEDS).

3. Results and Discussion

3.1 Effect of Grain Size

Fig. 3 shows that the -100# grain size fraction produces a higher content than the -60+80# and 80#+100# grain size fractions of each grain size fraction, the grain size -80#+100# produces a lower Ni content than other grain size fractions. The highest nickel content is obtained in the grain size fraction 100# with a magnetic field strength of 1000 G used at 4.463%.

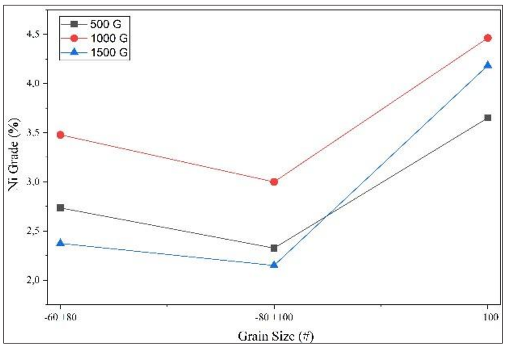

Fig. 3 Graph of the effect of grain size fraction on nickel grade

The nickel recovery results shown in Fig. 4 indicate that the highest recovery (93.93%) was obtained for the −80+100 mesh size fraction at a magnetic field strength of 1000 G, although the nickel grade was relatively low (3%). An inverse relationship between nickel grade and recovery was observed, where higher recovery corresponded to lower nickel grade. In the -100# size fraction, it can be seen that the magnetic strength of 1000 G produces the greatest recovery with 84,77% and the content is 4,46%, so the -100# size fraction is considered optimal in this reduction process.

SEM observations (Fig. 6) reveal that ferronickel particles formed in the −100 mesh size fraction are larger and exhibit a more granular morphology compared to those in coarser size fractions. So, ferronickel is easily liberated by magnetic separation.

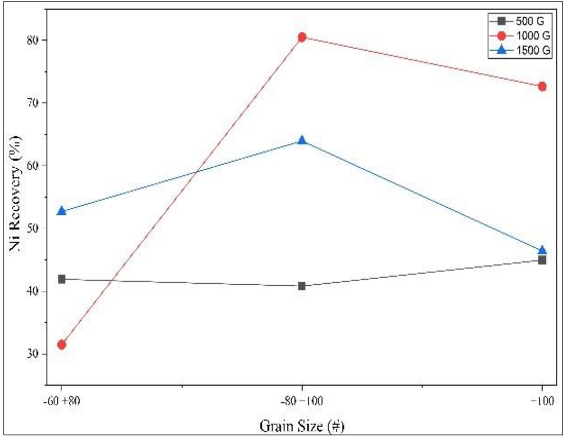

Fig. 4 Graph of the effect of grain size fraction on nickel recovery

Based on the XRD results (Fig. 5), magnetite was identified as the dominant phase in the −80+100 mesh size fraction, indicating that iron oxide reduction was less effective compared to other size fractions. Also, based on the SEM analysis results (Fig. 6), it can be seen that ferronickel particles observed at 500x magnifiction appear to be smaller in size compared to other size fractions. The position of ferronickel in the -80#+100# fraction is still very close to other compounds. It can be concluded that the finer the grain size, the higher the Ni content obtained. This occurs because the finer the grain size, the better the degree of liberation of nickel. The better the degree of liberation obtained, the better the nickel will be separated from the impurity minerals.

In previous research [15], the optimum nickel grade was obtained at -60#+80# grain size fraction with 1000 G magnetic field strength at 2,912% and 77,22% recovery rate.

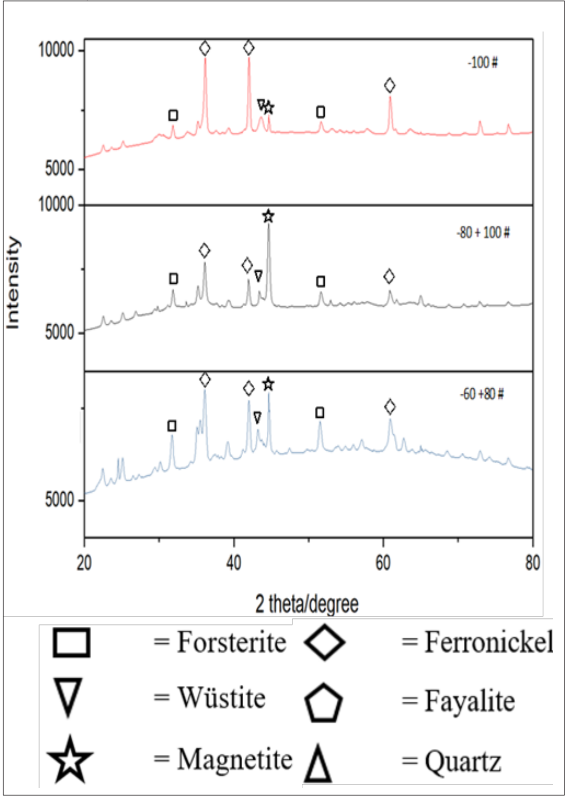

Fig. 5 XRD analysis results for grain size fraction variations

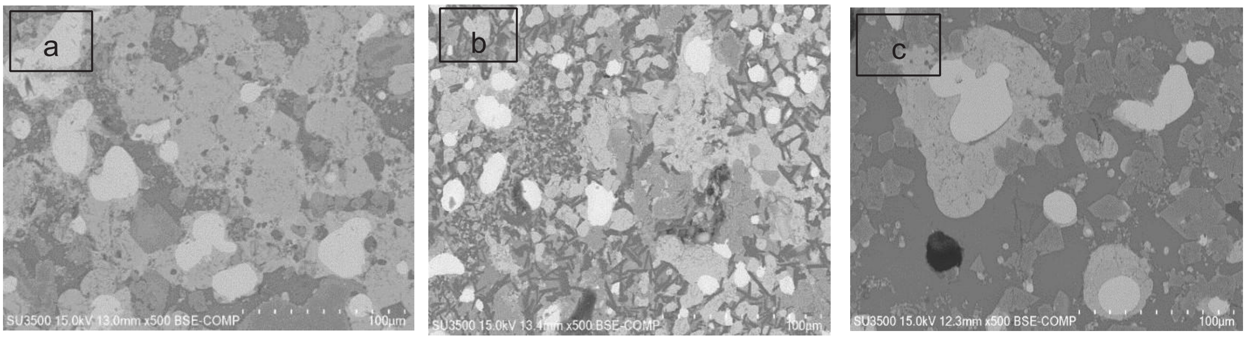

Fig. 6 SEM-EDS analysis results for grain size fraction variations (a) -60#+80#, (b) -80#+100#, and (c) -100# with 500x magnification

3.2 Effect of Reduction Temperature

Fig. 7 shows that the higher the reduction temperature, the lower the Ni content obtained. However, this decreasing trend is reversed for the 80#+100# grain size fractions. In these grain size fractions, the higher the reduction temperature, the higher the nickel content obtained, even increasing quite sharply. The highest percentage of nickel content was obtained from the combination of the

100# grain size fraction with a reduction temperature of 950 °C.

It can be seen in Fig. 8, when the reduction temperature is increased from 950 °C to 1050 °C, there is an increase in the percentage recovery for grain size fractions -80#+100# and -100#.

However, when the temperature was increased to 1150 °C, there was a significant decrease of the recovery percentage. The highest nickel recovery content was obtained at a temperature of 1050 °C with a grain size fraction of -80#+100#.

Fig. 10 shows the microscopic structure of the reduced nickel ore, showing that the ferronickel particle size increases with increasing reduction temperature. This is most likely due to the increasing volume of the liquid phase, resulting in mass transfer that increases the grain growth rate. The results of the XRD analysis presented in Fig. 8.

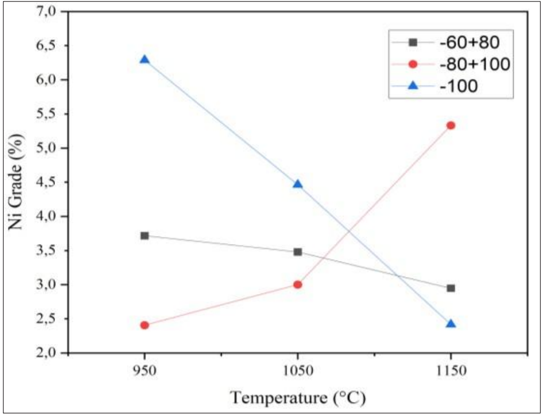

Fig. 7 Graph of the effect of temperature reduction on nickel grade

Based on the results of optical microscope analysis (Fig. 10), the largest grain size was obtained at a reduction temperature of 1150 °C, and there was also magnetite (Fig. 9), which of course reduces the formation of ferronickel so it is possible that due to incomplete reduction of iron oxide the nickel content is reduced.

Based on Fig. 9, magnetite is seen at reduction temperatures of 1050 °C and 1150 °C. The presence of magnetite indicates a reduction in ferronickel formation, which allows for incomplete reduction of iron oxide so that the nickel content is reduced. At a temperature of 950 °C, magnetite has not been found, but when the reduction temperature is increased to 1050 °C, magnetite is seen and when the temperature is increased again to 1150 °C, there is a decrease in the amount of magnetite. The optimal results obtained at 1050 °C, with 4,46% nickel grade and 84,77% recovery in -100# size fraction.

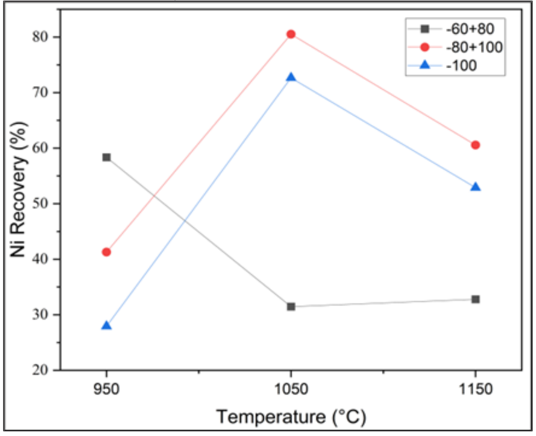

Fig. 8 Graph of the effect of reducing temperature on nickel recovery

In previous research [15], the optimum nickel grade was obtained at 1150 °C with -60#+80# grain size fraction at 2,912% nickel grade and 77,22% recovery rate.

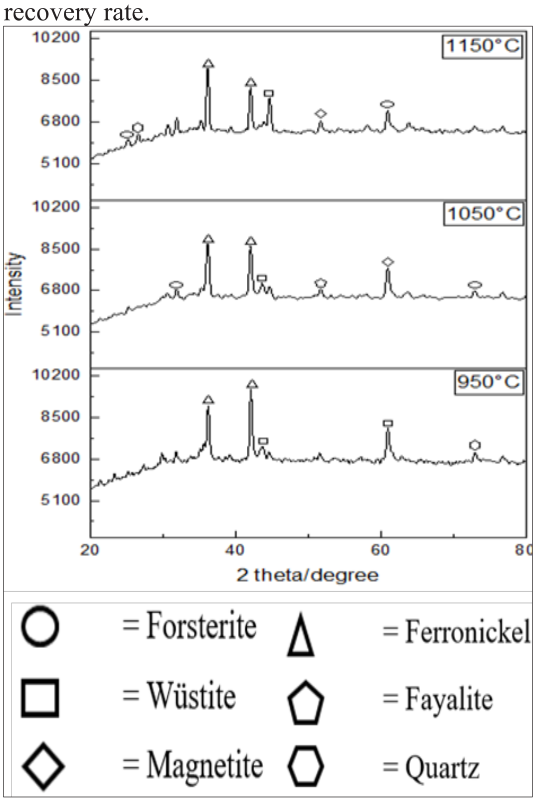

Fig. 9 XRD analysis results for reduction temperatures variations

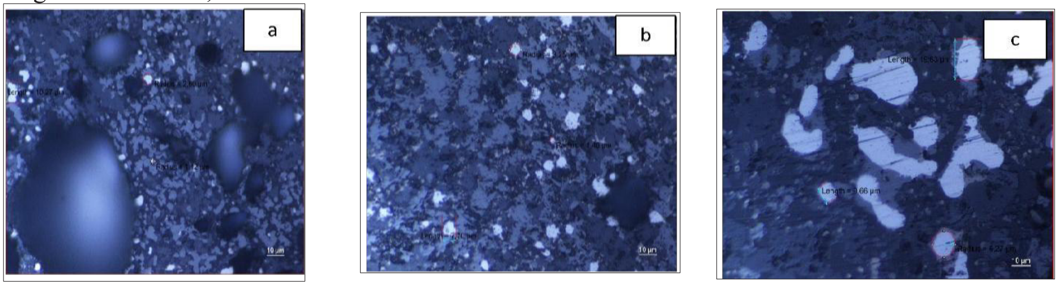

Fig. 10 Optical microscope results for reduction temperature variations (a) 950 °C, (b) 1050 °C, (c) 1150 °C

3.3 Effect of Magnetic Field Strength

Based on Fig. 11 below, it can be seen that there is an increasing trend of nickel content in each nickel ore grain size fraction when the magnetic field strength is increased from 500 G to 1000 G.

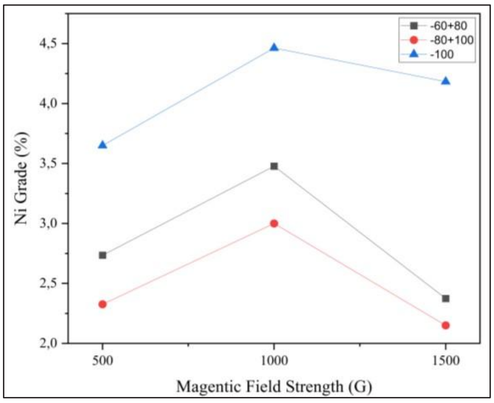

Fig. 11 Graph of the effect of magnetic field strength on nickel grade

However, when the magnetic field strength is increased from 1000 G to 1500 G, there is a decrease in Ni content from each nickel ore grain size fraction. At higher magnetic field strength (1500 G), excessive attraction of Fe-rich magnetic phases may lead to entrainment of nickel-bearing particles that are not fully liberated, resulting in a decrease in nickel grade despite stronger magnetic separation. It can be seen that the optimal magnetic field strength used is produced at a strength of 1000 G. While the lowest results are obtained at a magnetic field strength of 500 G.

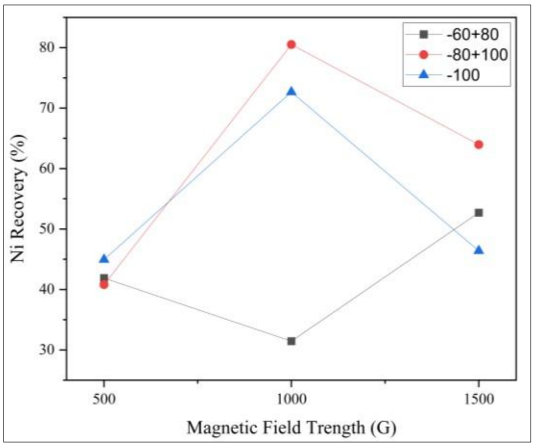

Fig. 12 Graph of the effect of magnetic field strength on nickel recovery

In Fig. 12 it can be seen nickel recovery showed an increasing trend when the magnetic field strength was increased from 500 G to 1000 G, and decreased when the magnetic field strength was increased to 1500 G. The highest nickel recovery was obtained in the grain size fraction -80#+100# with a magnetic field strength of 1000 G. The optimal nickel grade, accompanied by relatively high recovery, was achieved at a magnetic field strength of 1000 G. It can be concluded that the higher nickel grade obtained, the lower nickel recovery will be produced.

Previous research [15], showed that the optimum nickel grade was obtained at 1000 G magnetic field strength with -60#+80# grain size fraction at 1150 °C reduction temperature by using sodium chloride (NaCl) as an additive.

4. Conclusion

The research was conducted using a selective reduction process of laterite nickel ore added with Na_2SO_4 as an additive and palm kernel shell charcoal as a reducing agent. The use of palm kernel shell charcoal to replace coal as a reducing agent in the selective reduction process provides quite good results and it also environment friendly. Increasing the temperature can increase the nickel content, but after reaching the optimal point, if the reduction temperature is increased again, the results obtained will decrease compared to when the optimal point is reached. The nickel content tends to decrease as the reduction temperature is further increased. A fine particle size produces a higher nickel content compared to a larger particle size. The fine particle size causes a good degree of liberation, so that nickel is easily separated from impurities. Therefore, the magnetic separation process becomes more efficient. In addition, the magnetic field strength also affects the selective reduction process of laterite nickel ore. Nickel grade increased with increasing magnetic field strength up to an optimal value (1000 G), beyond which excessive magnetic force led to a decrease in nickel grade. Adding sodium sulfate (Na_2SO_4) as an additive may increase the particle size of the ferronickel, where has positive effect on magnetic separation process which results in increased nickel metal concentration. This phenomenon occurs because sulfur reacts with iron to form FeS, which suppresses iron metallization and consequently increases the nickel concentration in the ferronickel phase. However, there is a possibility that what is obtained is not ferronickel but nickel in another form (caused by incomplete reduction of iron oxide), depending on the results of the reduction process, which are influenced by grain size and reduction temperature.

A magnetic field strength of 1000 G is the most optimal in separating the paramagnetic phase (containing iron elements) so that it can produce a fairly high nickel content (4,463%) and recovery (84,77%) in the concentrate with a grain size of 100# and 1050 °C. Compared to previous research when using sodium chloride (NaCl) as an additive, using sodium sulfate (Na_2SO_4) is better. Sodium sulfate can generate higher nickel grade and recovery at lower reduction temperatures. Thus, it lowered the energy consumption.

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