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Selective Reduction of High Alumina-Lateritic Nickel Ore (0.5 Ni-44Fe-16Al_2O_3)

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Abstract

In this present study, the effect of reductant dosage, temperature and holding time on selective reduction process of high alumina-lateritic nickel ore have been investigated clearly. The lateritic nickel ore was reduced with 5 until 15 wt. % anthracite and 10 wt. % sodium sulfate at reduction temperature of 950ºC, 1050ºC and 1150°C for 60, 90, and 120 minutes. Magnetic separation process was then conducted to separate the concentrate and tailing. The analysis of reduced nickel ore is performed by the Atomic Absorption Spectroscopy, X-Ray Diffraction, and Secondary Electron Microscopy. The optimal process resulted from the reduction of nickel ore with 10 wt. % anthracite at the temperature of 1050°C for 120 minutes which resulted in 0.84% nickel in concentrate. The troilite was not found in reduced ore. The iron grade increased along the increased of reduction temperature. The longer of holding time in selective reduction process increased the nickel grade but it decreased the iron grade.

Keywords: selective reduction, magnetic separation, nickel ore, sodium sulfate, anthracite

1. Introduction

Nickel is widely used in the form of pure metals or as ferronickel alloys with varying the iron content (Li et al., 2012). There are two types of nickel ore which are used as raw materials for making nickel metals, namely sulfidic and lateritic ore types. Today, nickel sulfide ore reserves containing high nickel content is continuing to decrease, therefore some industries turn their attention to laterite nickel ore to be used as raw materials (Subagja and Firdiyono, 2015). Indonesian is one of the countries which has a large number of deposits of laterite nickel ore. Indonesian laterite nickel ore is scattered in several regions such as South East Sulawesi, Halmahera, Maluku, and Papua. Indonesian is the fifth-largest nickel producer in the world which has 12% reserves of nickel laterite ore in the world.

It is difficult to improve the nickel grade in lateritic ore with physical beneficiation process. Hydrometallurgy is commonly used to generate pure nickel, while pyrometallurgy is used to produce ferronickel from lateritic nickel ore (Kyle, 2010). Chemical waste and high-temperature operation which resulted in high emission pollutant are the main problems on both mineral processing (Mudd 2009). The reduction process of nickel laterite to produce ferronickel has been developed by researchers (Bunjaku et al., 2011; Elliot et al., 2017). The used of additives such as sodium sulfate is added for lowering the reduction temperature process and suppressing the reduction of iron oxide into iron metals through the formation of troilite (Li et al., 2012). Thus the process is called a selective reduction process due to only limited iron oxide which reduced into iron, while most of the nickel oxide is reduced into nickel.

Silicon oxide, magnesium oxide and aluminum oxide are the most impurities in limonitic and saprolitic nickel ore. The selective reduction process by using sodium sulfate additives is very effective to process lateritic nickel ore either for limonite or saprolite with high in magnesium and silicon oxide as impurities. Jiang et al. (2013) has been used a 10% of sodium sulfate for reducing the limonitic nickel ore containing high silicon oxide and magnesium oxide which is 20.05 SiO_2-12.28 MgO at 1200 ℃ for 50 minutes which resulted ferronickel containing 9.87% Ni. Subagja et al. (2016) reducing the limonitic ore containing high silicon oxide which is 14.84 SiO_2 using 20% sodium sulfate at 1000 ℃ for 1 hour resulting ferronickel with 10.28% Ni.

However, there still less information about the effectiveness of sodium sulfate in selective reduction of low-grade lateritic nickel ore containing high aluminum oxide. The effect of sodium sulfate, temperatures and length of reduction time in the selective reduction of low-grade lateritic nickel ore containing high aluminum oxide were investigated clearly in this study.

2. Material and Methods

2.1. Materials

The low-grade lateritic nickel ore containing high aluminum oxide was from South East Sulawesi, Indonesia which owned by PT. Integra Mining Nusantara. The anthracite coal, which used as reductant, was obtained from Sawahlunto, Indonesia. The proximate analysis of the reductant is listed in Table 1. The sodium sulfate, which used as additive, was purchased from PT. Bratachem, Indonesia.

2.2. Ore Preparation

Figure 1 shows the schematic diagram of the experiment. The nickel ore and anthracite were crushed into a -100 mesh prior to mix homogenously together with the additives. The 5 wt. % until 15 wt. % of anthracite as reductant and 10 wt. % of sodium sulfate as additive was used in this experiment. The mixture was agglomerated into 10-15 mm in diameter of pellets. The pellets were drying at 100°C for 4 hours before continued to reduction process.

2.3. Reduction process

The reduction process of pellets was conducted in a muffle furnace at a temperature of 950ºC, 1050ºC and 1150°C for 60, 90, and 120 minutes. The reduced pellets were cooled rapidly in water and dried at 100°C for 4 hours. The X-Ray Diffraction (XRD) using *Pan Analytical X'Pert 3 Powder* was used to analyze the mineralogical phases of reduced pellets.

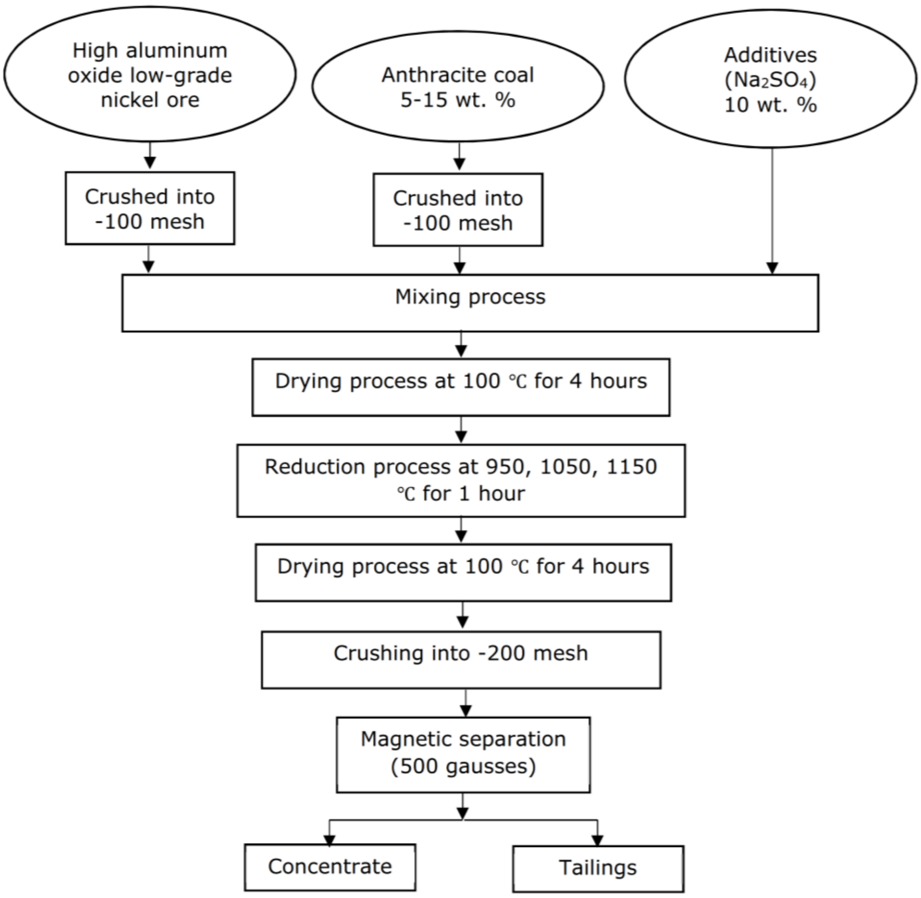

Figure 1. Schematic diagram of experiment

2.4. Magnetic Separation Process

The reduced pellets were ground into -200 mesh before the wet magnetic separation proces was performed and separated into the concentrate (magnetic) and tailing (non-magnetic). The magnetic field was 500 Gauss. The iron and nickel grade in concentrate was analyzed by Atomic Absorption Spectrophotometry (AAS) using *Shimadzu AA-7000*, while the morphological of ferronickel was observed using *JEOL*-Scanning Electron Microscopy (SEM).

3. Result and Discussion

3.1. Characteristic of low-grade lateritic nickel ore

The chemical composition of the nickel ore is listed in Table 2. The ore contains low-grade nickel and high aluminum oxide which is 0.45% and 16.87%, respectively. From XRD analysis, as shown in Figure 2, the nickel ore contains goethite which has aluminum content, hematite, and hercynite. The nickel content in nickel ore is present as the substitute of iron in goethite. The composition of each compound is listed in Table 3 as the Rietveld method which generated by using Highscore Plus software.

3.2. Effect of the amount of reductant on grade in ferronickel concentrate

Figure 3 presents as the result of various types and amount of sodium sulfate on iron-nickel grade in the concentrates resulting from the reduction process at 1050°C for 120 minutes followed by the magnetic separation process.

From Figure 3, it shows that the nickel content in concentrate is not increasing significantly as the increasing of reductant addition in reduction process of nickel ore. The highest nickel grade is found in 10 wt. % reductant which is 0.84%. According to (Foster et al., 2016), it was explained that the addition of more carbon or reductant to increase the yield of concentrate in reduction process of nickel ore would reduce the nickel grade in concentrate due to the increasing of iron oxide reduction to metallic iron. However, the addition of too small reductant would result in only a few of nickel oxide reduction into metallic nickel.

The XRD analysis, as shown in Figure 4, was performed to investigate the effect of reductant addititon to the compounds transformation which formed in reduced pellet. As shown in Figure 4, the XRD pattern of the reduced pellet at 1050°C consists of trinepheline ($NaAlSiO_4$), Hercynite (Al_2FeO_4), Tetrataenite (FeNi), Magnesioferrite (Fe_2MgO_4), Wustite (FeO) and Quartz Low (SiO_2). The FeNi peak at 10 wt.% of reductant is lower than 5 and 15 wt. %. Nevertheless, it contains higher nickel content in concentrate, which indicates that the FeNi in 10 wt. % of reductant has rich in nickel than iron content. At reductant 10 wt. % the wustite was formed due to the reduction process of hematite and magnetite in nickel ore. More addition of reductant resulted in more iron content in concentrate due to the more reduction of wustite into iron, thus it lowering the nickel grade in concentrate. As shown in Figure 4(c), the more iron content in concentrate, resulting in a higher peak of FeNi in XRD pattern.

Table 1. Proxymate analysis of anthracite coal

Element	Moisture	Volatile matter	Ash content	Fixed carbon
wt. %	3.14	18.25	18.25	60.35

Table 2. Chemical analysis of low-grade lateritic nickel ore

Elements	Fe	Ni	Al_2O_3	SiO_2	MgO
wt. %	34.96	0.45	16.87	9.77	0.28

Table 3. Rietveld analysis of lateritic nickel ore

Compounds	Ghoetite-Aluminan ($H_1Al_{0.17}Fe_{0.83}O_2$)	Hematite (Fe_2O_3)	Hercynite (Al_2FeO_4)
wt. %	34.96	0.45	16.87

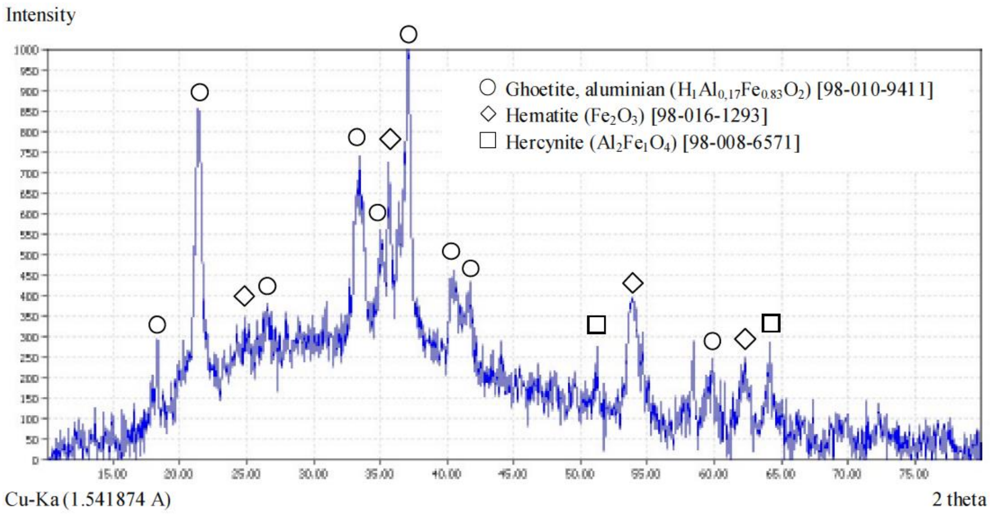

Figure 2. XRD analysis of low-grade lateritic nickel ore

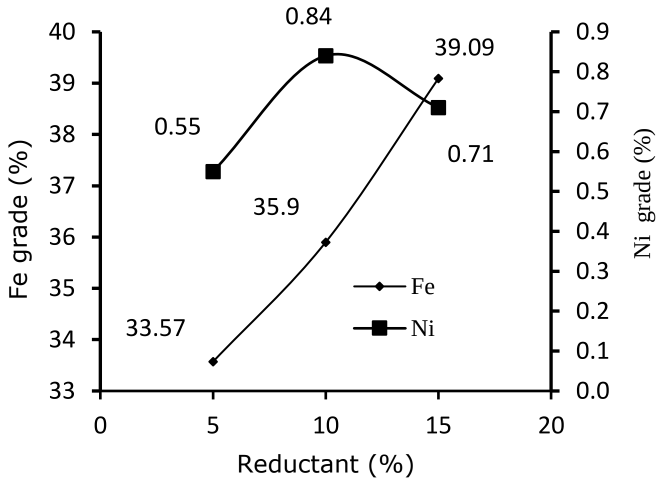

Figure 3. Effect of the number of reductant on iron and nickel grade with 10 wt. % additives of sodium sulfate at reduction temperature of 1050°C for 120 minutes

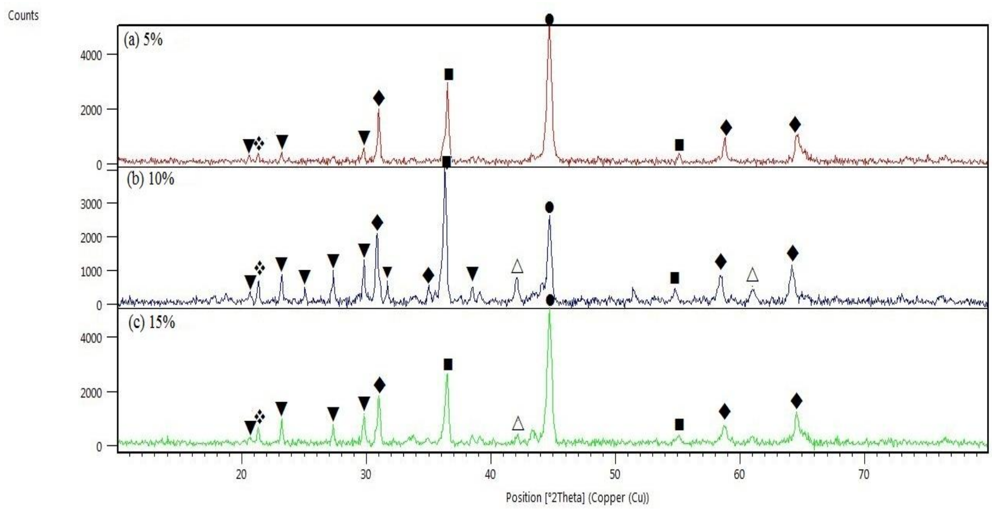

Figure 4. The XRD patterns of reduced pellets at 1050°C for 120 minutes with 10 wt.% additives of sodium sulfate with various number of reductant: (a) 5 wt.%., (b) 10 wt.%. and (c) 15 wt.%. (▼= $NaAlSiO_4$, ■=Al_2FeO_4, ●=FeNi, ◆= Fe_2MgO_4, △=FeO, ❖= SiO_2)

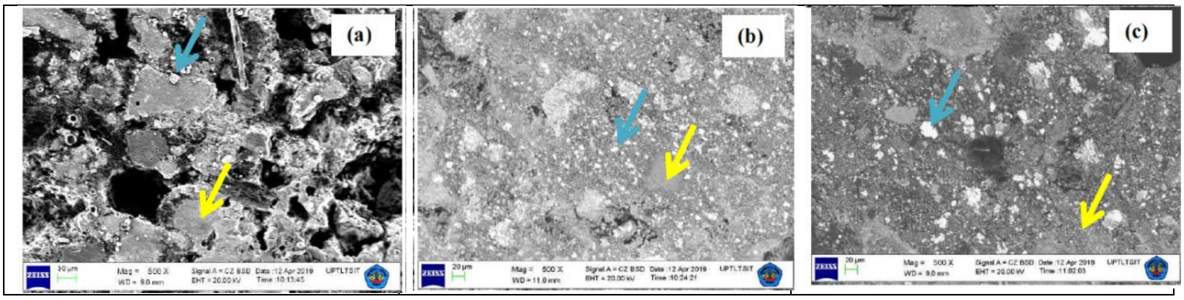

Figure 5. SEM analysis of reduced laterite pellets at 1050°C temperature for 120 minutes with 10 wt.% of additives Na_2SO_4 with variations in the number of reducing agents, that is (a) 5 wt.%, (b) 10 wt.% and (c)15 wt.%.

Figure 5 shows the microstructures of a sample with a temperature of 1050°C by using SEM in various reductant 5, 10 and 15 wt. % for 120 minutes. White area/blue arrow indicated as ferronickel, while the gray area/yellow arrow indicated as impurities. As shown in Figure 6, the ferronickel particle size looks getting larger as the increasing of reductant in nickel ore due to the increasing reduction rate of metal oxide which is agreed with the previous research (Li et al., 2012; Elliot et al., 2017).

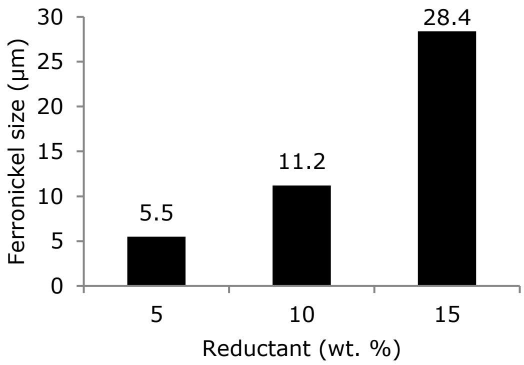

Figure 6. The effect of reductant amount in particle size of feronickel.

From the XRD pattern and SEM pictures, the wustite was not found. According to the previous result (Nurjaman et al., 2018), the wustite promotes the increasing of nickel content in concentrate due to the non-magnetic of wustite and also the growth of ferronickel particle due to the agglomeration particle in liquidus phase at low temperature (980ºC). The high aluminum oxide in low-grade nickel ore was tended to inhibit the wustite formation. From the XRD analysis, the iron more reacted with aluminum to form hernycite rather than with sulfur. Hercyinite also has a higher melting point than wustite, thus the agglomeration process of the ferronickel particle is not found in this selective reduction process.

3.2. Effect of the temperature on grade in ferronickel concentrate

Figure 7 presents the result of the reduction and magnetic separation process of low-grade lateritic pellets ore with the addition of 10 wt. % sodium sulfate additives. The reduction process of lateritic ore was conducted at 950°C to 1150°C for 60 minutes. It shows that the increase of nickel content in concentrate is not significant with the increase of reduction temperature. The highest nickel content was obtained at a reduction temperature of 1050°C which is 0.78%. The higher the reduction temperature, the higher of metal (iron) content in the concentrate, this is due to the increasing degree of metallization of metals (iron) so that more metal oxide is reduced into metal (Pan et al., 2013).

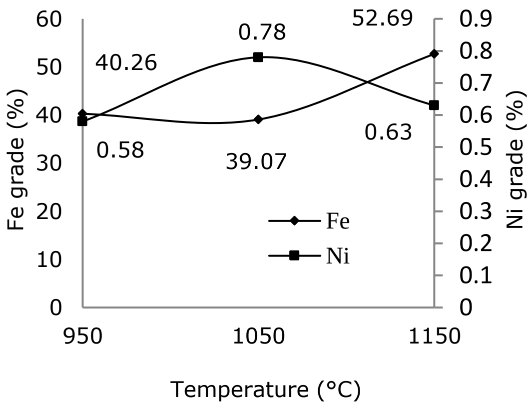

Figure 7. Effect of temperature on iron and nickel grade with 10 wt. % additives of Na_2SO_4 at reduction 60 minutes and reducing agents 10 wt. %

From XRD pattern in Figure 8, ferronickel is not found from the reduction of nickel ore at temperature 950°C. It also confirmed from the microstructure analysis in Figure 9. The iron and nickel still trapped in metallic and °Figure 8, where the iron is still trapped in hercynite and wustite. From Rietveld analysis

(Table 4), at higher temperature reduction, 1050°C and 1150°C, the ferronickel is formed and it is increased with the increase of reduction temperature, thus it increases the nickel grade in concentrate (Valix and Cheung, 2002).

From the microstructure (Figure 8), the ferronickel particle is growing larger by the increasing of reduction temperature. The diameter of the particle size is shown in Figure 10.

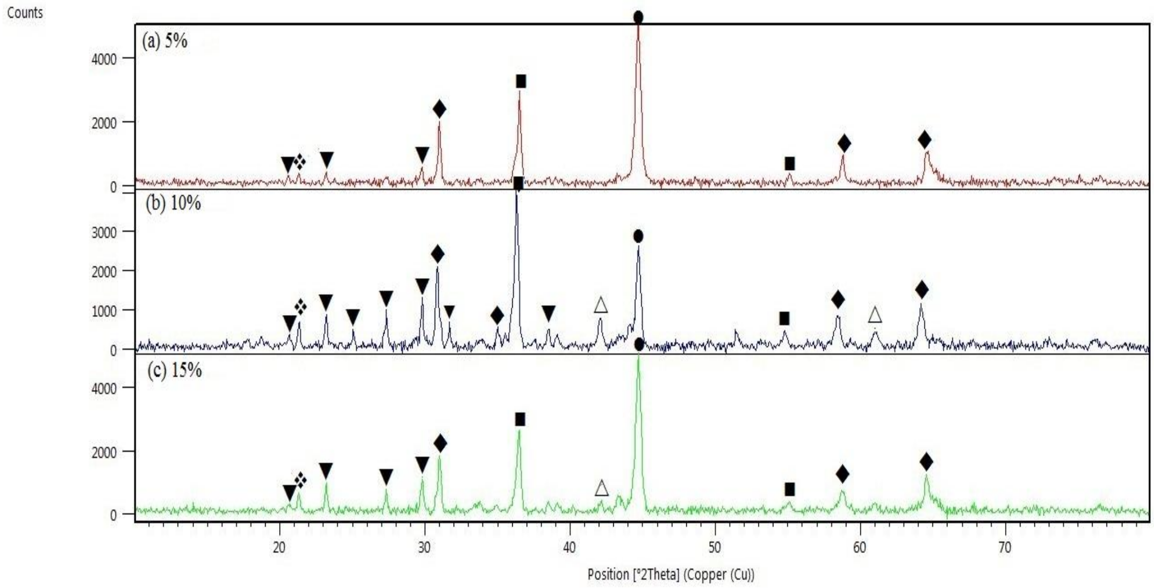

Figure 8. XRD pattern of reduced nickel ore with addition of 10 wt. % sodium sulfate additive and 10 wt. % reductant for 60 minute. (▼=$NaAlSiO_4$, ■=Al_2FeO_4, ●=FeNi, ◆= Fe_2MgO_4, △= FeO, ❖= SiO_2)

Table 4. R*ietveld analysis of* reduced nickel ore with various reductant temperature

Compounds	Reduction Temperature (°C)		
	950	1050	1150
$NaAlSiO_4$	30.3	37.9	40.3
Al_2FeO_4	9.8	10.3	8.4
FeNi	-	27.7	29.6
Fe_2MgO_4	46.9	19.8	19.3
FeO	12.9	3.8	1.7
SiO_2	-	0.4	0.6

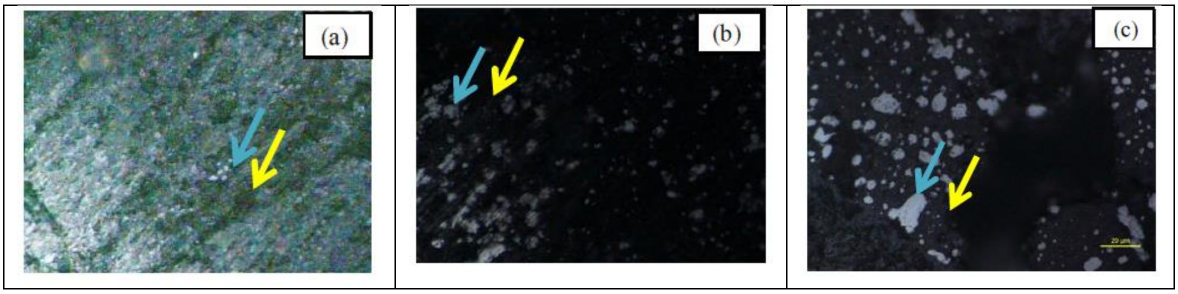

Figure 9. Microstructure of reduced nickel ore with 10 wt. % Na_2SO_4 additive and 10 wt. % reductant for 60 minute in various reduction temperatures: (a) 950°C, (b) 1050°C dan (c) 1150°C

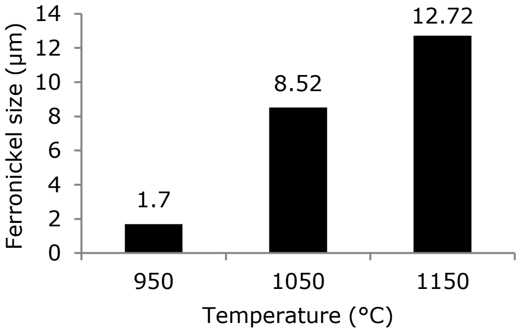

Figure 10. The effect of reduction temperature in particle size of feronickel

3.3. Effect of the holding time on grade in ferronickel concentrate

The results of the reduction and magnetic separation of reduced nickel ore at reduction time from 60 minutes to 120 minutes is shown in Figure 11. The reduction temperature was at 1050°C. From Figure 11 shows that the nickel grade increases with the increase of reduction time. The highest nickel grade is 0.84% which obtained from the reduction process for 120 minutes. Nevertheless, from the XRD pattern in Figure 12, it shows that the intensity of ferronickel at reduction time of 120 minutes is lower than others. It means that the intensity of ferronickel in the XRD pattern is determined by the metallic iron content in ferronickel, not the nickel content. Thus, the lower intensity of ferronickel in Figure 12 (c) contains more nickel content than iron content.

The longer of reduction time, the more reductant gas resulted, thus more metal oxide will be reduced into metallic phase (Jiang et al., 2013). However, with too long in reduction time, the reductant gas will run out and some of the metallic iron will re-oxidized into wustite (Foster et al., 2016). Therefore, it will result in a concentrate with higher nickel content. From Rietveld analysis, as shown in Table 5, it shows that the length of reduction time, the more hercynite formed. It means that some of the metallic iron was transformed into hercynite rather than wustite at the re-oxidation process due to the high aluminum oxide content in this low-grade nickel ore.

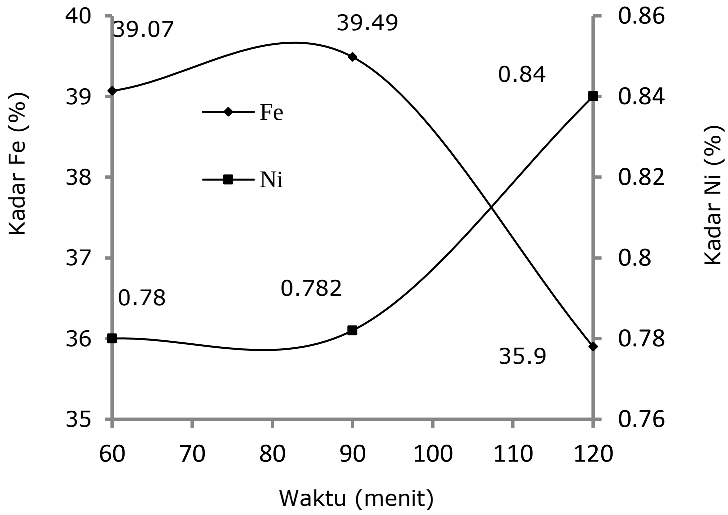

Figure 11. Effect of reduction time on selective reduction low-grade nickel ore with 10 wt. % additives of sodium sulfate and 10 wt. % of reductant at reduction temperature of 1050°C.

Table 5. Rietveld refinement of reduced nickel ore with the addition of 10 wt. % sodium sulfate and 10 wt. % of reductant at reduction temperature 1050◦C

Compounds (wt. %)	Reduction time (minutes)		
	60	90	120
$NaAlSiO_4$	36.9	26.7	30.9
Al_2FeO_4	10.3	12.2	28.9
FeNi	27.7	28.2	29.6
Fe_2MgO_4	19.8	18.8	2.7
FeO	4.8	13.5	7.2
SiO_2	0.4	0.5	0.6

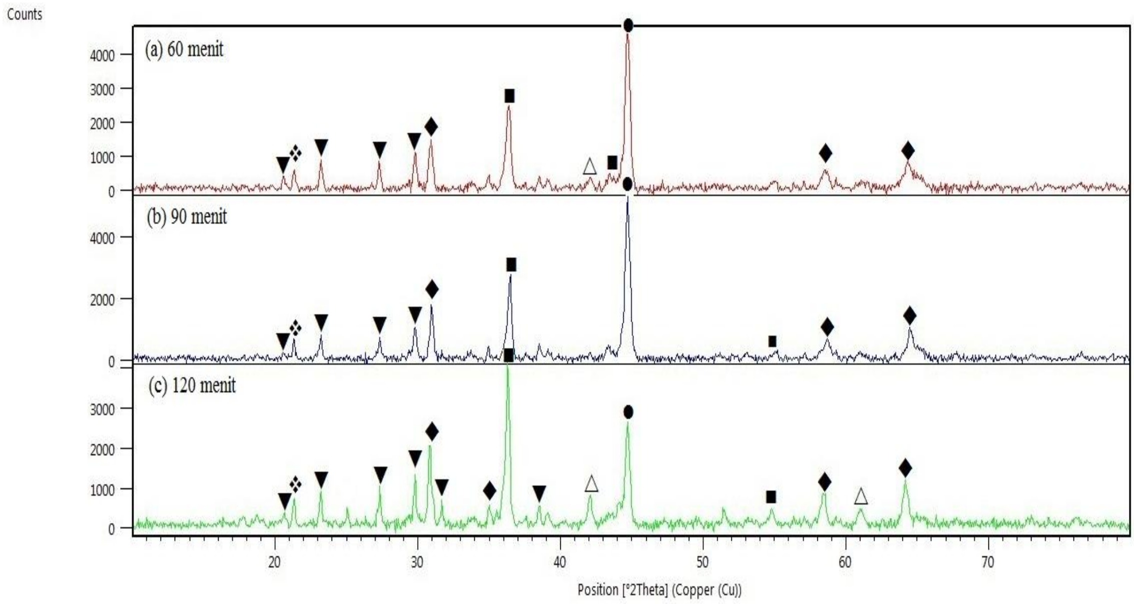

Figure 12. The XRD pattern of reduced nickel ore with the addition of 10 wt. % sodium sulfate and 10 wt. % of reductant at reduction temperature 1050°C for : (a) 60 menit, (b) 90 menit dan (c) 120 menit. (▼= $NaAlSiO_4$, ■=Al_2FeO_4, ●=FeNi, ◆= Fe_2MgO_4, △= FeO, ❖= SiO_2).

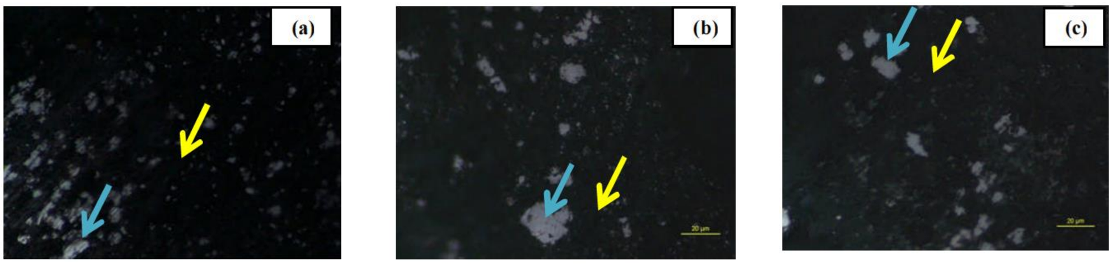

Figure 13. Microstructure analysis of reduced nickel ore with the addition of 10 wt. % sodium sulfate and 10 wt. % of reductant at reduction temperature 1050°C for: (a) 60 minutes, (b) 90 minutes dan (c) 120 minutes

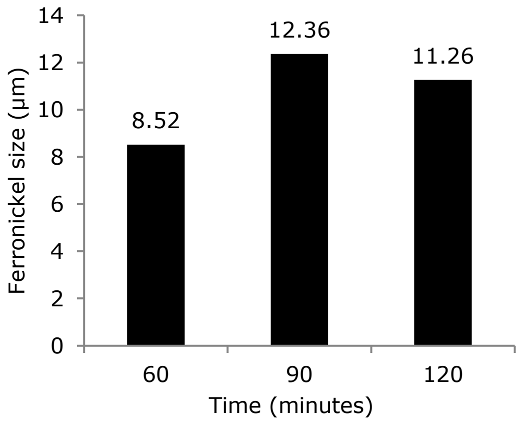

Figure 14. The effect of holding temperature in reduction process on the particle size of feronickel.

From Figure 13 and 14, it shows that the ferronickel particle getting larger when the reduction time increase from 60 minutes into 90 minutes. It looks the more reductant gas produced by the longer reduction time. However, the ferronickel particle is tended to decrease at 120 minutes of reduction time which means the reductant gas starts to run out.

4. Conclusion

The optimal process resulted from the reduction process of nickel ore with 10 wt.% reductant at the temperature of 1050°C for 120 minutes. The troilite was not found in reduced ore, thus it only obtained 0.84% nickel grade in concentrate. The addition of reductant increased the nickel grade at 10 wt.% dosage but decreased at more than 10 wt.% dosage. The iron grade increased along the increased of reduction temperature, while the optimum nickel grade found at 1050ºC. The longer of holding time in selective reduction process increased the nickel grade although it was contrary different from the iron grade.

Acknowledgments

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