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THE EFFECT OF ADDITIVES AND REDUCTORS IN SELECTIVE REDUCTION PROCESS OF LATERITE NICKEL ORE

F. Nurjaman[1], A. Sa'adah[2], A. Shofi[1], W. Apriyana[3] and B. Suharno[2]

[1] Research Unit for Mineral Technology - LIPI
Jl. Ir. Sutami, Km 15 Tanjung Bintang, Serdang, Lampung 35361
[2] Departement of Metallurgical and Materials Engineering, University of Indonesia
Kampus Baru UI, Depok 16424
[3]Research Unit for Natural Technology - LIPI
Daerah Istimewa Yogyakarta 55861
E-mail: fajar.nurjaman@lipi.go.id

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ABSTRACT

THE EFFECT OF ADDITIVES AND REDUCTORS IN SELECTIVE REDUCTION PROCESS OF LATERITE NICKEL ORE. Selective reduction of laterite nickel ore followed by magnetic separation was carried out to produce ferronickel products. The effect of adding additives and reductor types in the selective reduction process was studied in this study. Reductors used were anthracite coal and palm shell charcoal with variations of 5 to 15% by weight, while the additive used was sodium sulfate (Na_2SO_4). The reduction process was carried out at temperatures of 950 ºC, 1050 ºC and 1150 ºC for 60 minutes. The addition of 10% sodium sulfate additives by weight in the reduction process of laterite nickel ore produced higher concentrations of nickel as 6.09%, compared to no additives, i.e. 2.45%. The addition of reductors in the selective reduction process of laterite nickel ore shows that the higher the amount of reductors causes a decrease in the concentrate level of nickel. Furhtermore, the type of reductors used shows that the concentrate from the reduction result using anthracite coal reductor produces higher level of nickel and lower level of iron compared to the use of palm shell charcoal reductor.

Keywords: Laterite nickel ore, Selective reduction, Ferronickel, Sodium sulfate, Anthracite coal, Palm shell charcoal

ABSTRAK

PENGARUH ADITIF DAN REDUKTOR PADA PROSES REDUKSI SELEKTIF BIJIH NIKEL LATERIT. Reduksi selektif bijih nikel laterit diikuti dengan pemisahan magnetik dilakukan untuk menghasilkan produk ferronikel. Pengaruh penambahan aditif dan jenis reduktor dalam proses reduksi selektif telah dipelajari dalam penelitian ini. Reduktor yang digunakan, yaitu batubara antrasit dan arang cangkang sawit dengan variasi 5 hingga 15% berat, sedangkan aditif yang digunakan adalah sodium sulfat (Na_2SO_4). Proses reduksi dilakukan pada temperatur 950 ºC, 1050 ºC dan 1150 ºC selama 60 menit. Penambahan aditif sodium sulfat sebanyak 10% berat dalam proses reduksi bijih nikel laterit menghasilkan kadar nikel dalam konsentrat yang lebih tinggi yaitu 6,09%, jika dibandingkan tanpa aditif, yaitu 2,45%. Pada penambahan reduktor dalam proses reduksi selektif bijih nikel laterit, menunjukkan bahwa semakin tinggi jumlah reduktor menyebabkan penurunan kadar nikel dalam konsentrat. Disamping itu dari jenis reduktor yang digunakan menunjukkan bahwa konsentrat hasil reduksi menggunakan reduktor batubara antrasit menghasilkan kadar nikel yang lebih tinggi dan besi yang lebih rendah jika dibandingkan dengan penggunaan reduktor arang cangkang sawit.

Kata kunci: Bijih nikel laterit, Reduksi selektif, Ferronikel, Sodium sulfat, Batubara antrasit, Arang cangkang sawit

INTRODUCTION

Nearly 81%, nickel is used as one of the alloying elements in the manufacture of stainless steel and alloy steel [1]. In Indonesia, a large scale of nickel laterite deposits is found in Sulawesi islands. [2]. Laterite ores account for 72 % of global nickel sources, but only 42 % are used for primary nickel metal production. In contrast, sulfide ore accounts for 58 % of nickel production [3]. This is because the technological challenge in processing laterite nickel ore is more difficult compared to nickel sulfide ore [1]. To meet the high demand for nickel in the future, laterite nickel can be used as an alternative raw material, due to the depletion of nickel sulfide ore reserves [4].

One method for processing laterite nickel ore into ferronickel is a reduction process at a temperature of 1000-1200 ºC, followed by a magnetic separation method, otherwise known as the selective reduction method [5]. Reduction of laterite nickel ore is carried out using carbon-based reductors, which will produce CO gas which will reduce metal oxide compounds, as shown in equation (1).

$$C + CO_2 \rightarrow 2CO \quad \text{...........} \quad (1)$$

$$2(Fe,Ni)O.OH \rightarrow (Fe,Ni)_2O_3 + H_2O \quad \text{..............} \quad (2)$$

$$6(Fe,Ni)O.OH \rightarrow 2(Fe,Ni)_3O_4 + 3H_2O + 0{,}5O_2 \quad \text{......} \quad (3)$$

$$NiFe_2O_4 + CO \rightarrow Ni + Fe_2O_3 + CO_2 \quad \text{...........} \quad (4)$$

$$3Fe_2O_3 + CO \rightarrow 2Fe_3O_4 + CO_2 \quad \text{...........} \quad (5)$$

$$Fe_3O_4 + CO \rightarrow 3FeO + CO_2 \quad \text{...........} \quad (6)$$

$$FeO + CO \rightarrow Fe + CO_2 \quad \text{...........} \quad (7)$$

Nickel in the limonite ore is substituted in the goethite crystal structure which at 300 °C goethite dehydroxylation occurs by reaction in equation (2) [6]. After dehydroxylation, nickel is formed in the hematite spinel structure. Nickel will join into the magnetite spinel phase under the condition of reduction through the reaction in equation (3) which is then reduced, through the reaction in equation (4) to be Ni metal and iron hematite compound [7]. The process of reducing iron oxide to iron metal is shown in the reaction in equation (5-7).

Addition of additives in the selective reduction process is carried out to increase the content and yield of Ni in nickel ore concentrates. Additives containing sulphur are generally used to increase the levels and yield of nickel in concentrates, including elemental sulphur [6,8], sodium sulfate [9-11], calcium sulfate [12,13], and pyrite [14]. Sodium sulfate provides a more effective reduction result compared to other additives. Jiang et al. [15] conducted a selective reduction process of laterite nickel ore as 1.49% Ni by adding 10% Na_2SO_4 additive and 2% reductor of bituminous coal at a temperature of 1200 °C for 50 minutes, and obtained ferronickel concentration of 9.87% Ni and nickel yield as 90.9%. Li et al. [16] also conducted a selective reduction process of 1.9% Ni laterite nickel by adding an additive of 20% Na_2SO_4 and lignite coal reductor at a temperature of 800-1200 ºC for 60 minutes, and obtained optimum ferronickel concentrate with a content of 9.48% Ni and a nickel yield of 83.02% at a reduction temperature of 1100 ºC.

The use of reductors in the reduction selective process of laterite nickel ore aims to reduce the metal oxide compounds found in laterite nickel ore. Table 1 shows several types of reductors and additives used in the reduction process of laterite nickel ore. Anthracite coal has a higher caloric value than bituminous and lignite, so its use in the industrial world as fuel is quite extensive that the selling value of low-sulphur anthracite coal is much higher than both of them. A problem arises in anthracite coal with high sulphur content, where its use as fuel in electric power plants can trigger corrosion of boiler pipe metals, so the use of this type of coal is very little.

In this study, 10% sodium sulfate additives were used to reduce nickel ore (1.4 Ni) with high Fe and Si contents (50.5 Fe and 16.5 Si, respectively). The effect of using sodium sulfate additives on the reduction process nickel ore was studied in this research. Anthracite coal with high sulphur content was used as a reductor in this study. In addition, as shown in Table 1, the use of biomass as material reductor is also not widely used. Therefore, this research also used palm shell charcoal reducing agent (with 0% sulphur content) in the reduction process of nickel ore. The effect of the sulphur content in reductors was studied in this research.

Table 1. Selective reduction of Laterite Nickel Ore as Additive and reduction

Laterite Nickel Ore Type	Ore composition (%)				Reductor	Aditive	Literature
	Ni	Fe	MgO	SiO_2			
Limonite	1.33	56	0.83	2.98	Bituminus	Sulfur and pirit	Elliot *et al.* 2017 [4]
Limonite	1.11	48.68	3.04	14.84	Bituminus	$CaSO_4$	Mayangsari dkk 2016 [17]
Saprolite	1.49	34.69	12.28	20.05	Bituminus	Na_2SO_4	Jiang *et al.* 2013 [15]
Saprolite	1.91	22.1	13.4	26.49	Lignit	Na_2SO_4	Li *et al.* 2012 [16]
Limonite	0.97	40.90	4.65	12.55	Bituminous,	$CaSO_4$	Zhu et al., 2012 [12]
Saprolite	1.42	23.16	17.65	27.74	graphite		
Limonite	1.8	16.54	6.81	20.97	LPG	-	Itao and Anacleto, 2011 [18]
Saprolite	1.38	25.3	14.99	19.07	H_2:70%, N_2:30%	Na_2SO_4	Lu et al. 2013 [19]

EXPERIMEN METHOD

Materials

Laterite nickel ore in this study is derived from Southeast Sulawesi, Indonesia. The chemical composition of laterite ore is based on an analysis using XRF as shown in Table 2. Anthracite coal and palm shell charcoal are used as reductors. The proximate analysis of reductors is shown in Table 3. Sodium sulphate was used as an additive in the selective reduction process of laterite nickel ore in this study.

Table 2. Chemical composition of Nickel ore

Element	Ni	Fe	Si	Mg	Al	Ca	Cr	Mn	Co
% Weight	1.4	50.5	16.5	1.81	4.86	0.177	2.68	0.847	0.0662

Table 3. Proximate analysis redactor (% weight)

Reductor	Moisture	Component (%)	Ash content	Fixed carbon
		Volatile content		
Antrasit Coal	3.14	18.25	18.25	60.35
Palm Shell Charcoal	0.43	22.57	21	77

Procedure

Nickel ore and reductors are crushed to a size of less than 149 µm (-100 mesh). As much as 100 grams of laterite nickel ore, 5% of reductors weight and 10% of sodium sulphate weight are mixed and made into pellets 10-15 mm in diameter. The pellets were dried in an oven at a temperature of 120 °C for 4 hours to remove the water content, then put into graphite crucible for further reduction process using muffle furnace at temperatures of 950 ºC, 1050 ºC and 1150 °C for 60 minutes. After the reduction process, the pellets were removed from the muffle furnace and cooled quickly using water.

The magnetite separation process was carried out at the next stage to separate the concentrate and tailings. Concentrates that are rich in iron and nickel alloy metals are magnetic while tailings containing impurities (non metallic oxides) are non magnetic. The reduced nickel ore was crushed to a size of 74 µm before the magnetic separation process is carried out in a wet manner using magnets of 500 Gauss.

Phase transformations or changes in compounds in laterite nickel ore during the reduction process were analyzed using XRD. XRF analysis was conducted to determine the levels of nickel and iron in ferronickel concentrates, while SEM-EDS was used for ferronickel particle morphology. The recovery of nickel and iron is calculated based on equations (8) and (9).

$$Ni\ Acquisition = \frac{\%Ni\ concentrate}{\%Ni\ Laterite\ Nickel\ Ore} \quad .... \quad (8)$$

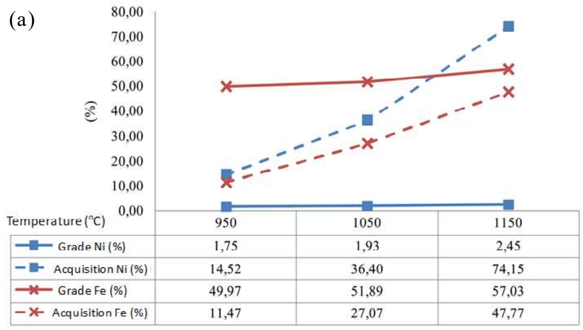
$$Ni\ Acquisition = \frac{\%Fe\ concentrate}{\%Fe\ Laterite\ Nickel\ Ore} \quad .... \quad (9)$$

RESULTS AND DISCUSSION

The Effect of Additives Addition

Figure 1 shows the levels and yields of Ni and Fe from the concentrations of magnetic separation results with the addition of 10% Na_2SO_4 with and without additives at a reduction temperature of 950 ºC, 1050 ºC and 1150 °C for 60 minutes with anthracite coal reductor. The level and yield of Ni and Fe in the concentrate without additive (Figure 1(a)) increase along with the increasing reduction temperature from 950 ºC to 1150 °C. This is also indicated by the results of XRD analysis (Figure 2), where the intensity of FeNi peak increases along with the increasing reduction temperature. This is also indicated by the decreasing intensity of FeO, which shows that the rate of reduction of iron oxide compounds into Fe metal increases with the increasing of reduction temperature. Furthermore, Fe metal will associate with Ni metal to form ferronickel (FeNi). However, the optimum levels of Ni and Fe are only 2.45% and 57.03%, respectively, which are obtained at a reduction temperature of 1150 °C.

(a)



(b)

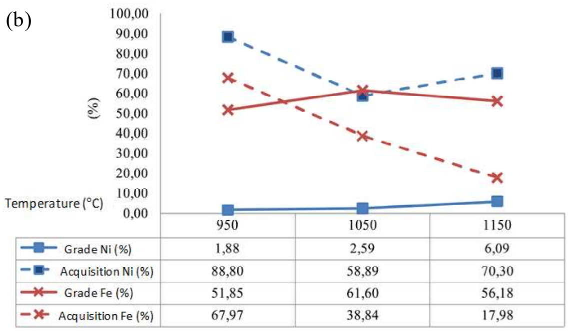


Figure 1. The effect of additive addition on the levels and recovery of Ni and Fe in concentrates at temperatures of 950 to 1150 °C for 60 minutes: (a). without Na_2SO_4, (b). with Na_2SO_4C

With the presence of sulphur-based additives, troilite (FeS) compounds are formed [10], as shown by the results of XRD analysis (Figure 3), where FeS compounds are present at reduction temperatures of 950 °C, 1050 ºC and 1150 ºC. the reduced Na_2SO_4 produces Na_2S. Then Na_2S reacts with FeO to form FeS with a reaction in equation (6-7).

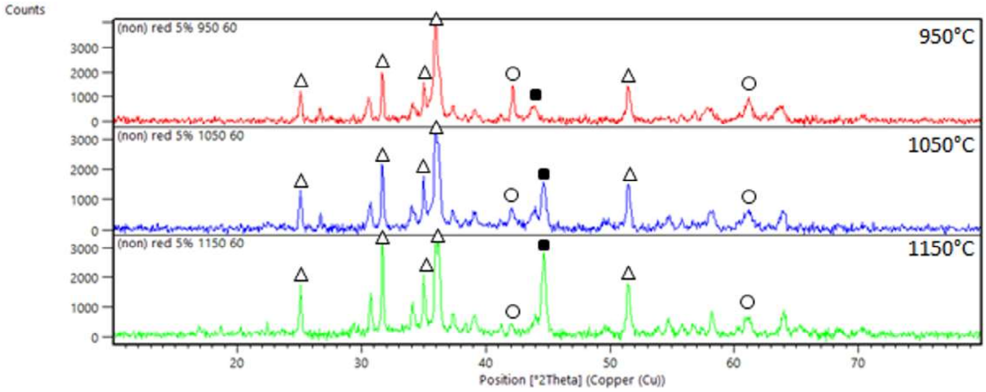


Figure 2. The results of XRD analysis for the reduction of nickel lat-erite ore without additives for 60 minutes with the addition of 5% anthracite coal reductor (Δ = $(Fe,Mg)_2SiO_4$, ■ = FeNi, ○ = FeO).

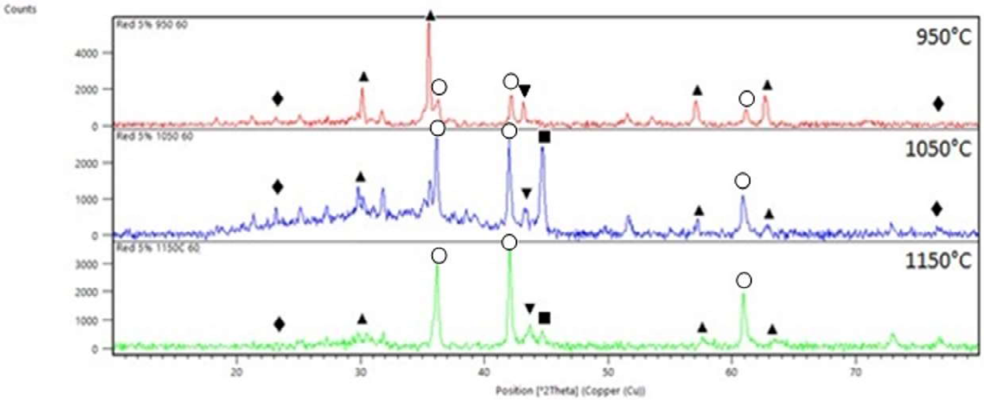


Figure 3. The results of XRD analysis for the reduction of nickel laterite ore with 10% Na_2SO_4 additive for 60 minutes with the addition of 5% anthracite coal reductor (▼ = FeS, ▲ = Fe_2SiO_4, ■ = FeNi, ○ = FeO, ¨= SiO_2).

$$\tfrac{1}{4} Na_2SO_4 + CO \rightarrow \tfrac{1}{4} Na_2S + CO_2 \quad \text{.............} \quad (6)$$

$$Na_2S + FeO + 2SiO_2 \rightarrow Na_2Si_2O_5 + FeS \quad \text{............} \quad (7)$$

When laterite ore was reduced at temperatures of 950 ºC, 1150 °C and 1150 °C for 60 minutes with the addition of 10% Na_2SO_4 (Figure 2b), Ni and Fe increased respectively from 1.4% and 50.85% (in nickel ore) to 6.09% and 56.18% (in concentrate). From these results, it can be seen that the ore which is reduced by the addition of additives produces concentrates with higher nickel and lower iron compared to without additives.

The presence of non magnetic FeS phase can reduce the yield of Fe in concentrates, so that the Ni content will increase. In addition, with the higher reduction temperature, FeS compounds can also inhibit the reduction process of wustite (FeO) compounds into Fe metal. This is indicated by the increasing intensity of FeO compounds, as shown in Figure 3, along with the increase in reduction temperature. FeO is paramagnetic, so it will be grouped into tailings with FeS after the magnetic separation process is carried out. The low peak of FeNi in Figure 3 shows that FeNi concentrates are rich in nickel content (with less iron content).

From the results of microstructure analysis, as shown in Figures 4 and 5, bright grains show ferronickel formed from the reduction of nickel ore, while dark grains are impurities with the dominant elements Si and O (quartz/SiO_2). From the figures, changes in grain distribution and size formed when the ore is reduced with and without additives at temperatures of 950 ºC to 1150 °C are visible. The grain size of ferronickel in both types of concentrates increases with increasing

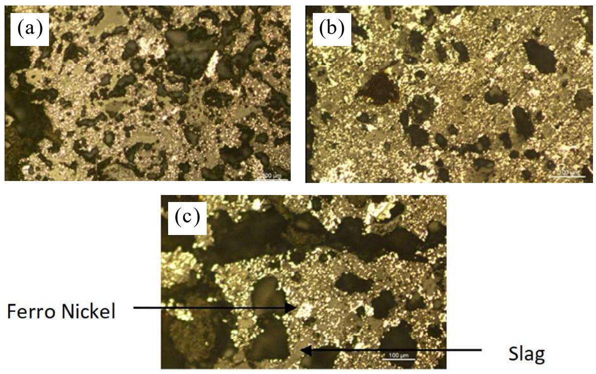


Figure 4. Pellet microstructure which was reduced with and without Na_2SO_4 at different temperatures for 60 minutes: (a). 950 °C, (b). 1050 °C, and (c). 1150 °C.

temperature, but the ferronickel content in the concentrate with the addition of 10% sodium sulfate (Figure 5) has a larger size than the concentrate without additive (Figure 4). This is due to the low eutectic temperature of the FeFeS phase formed by sodium sulphate additive, which is 985 °C, so that the liquid FeS phase is formed at a reduction temperature of 1050 ºC. In the liquid phase, the metal transfer movement becomes easier so that the ferronickel undergoes agglomeration. The diffusion rate of metal elements will be higher as the temperature increases, so the higher reduction temperature can accelerate the growth of ferro nickel grain so that the grain size of ferronickel becomes larger [10].

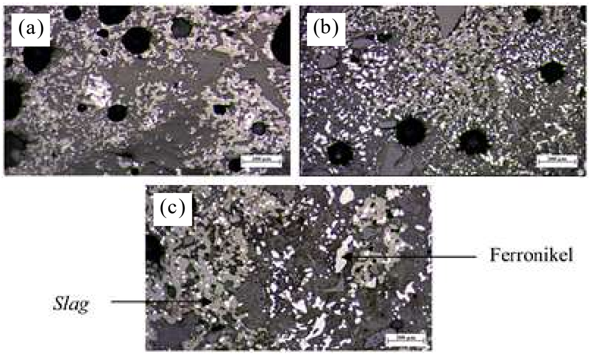
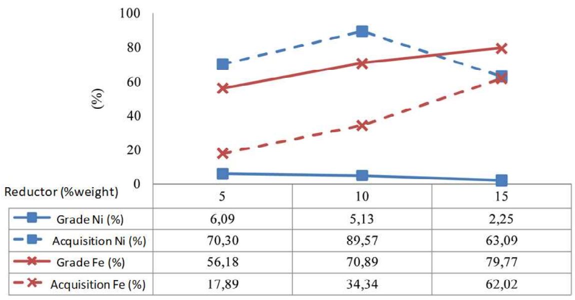


Figure 5. Pellet Microstructure which wash reduced by Na_2SO_4 with different temperature for 60 minutes: (a). 950 ºC, (b). 1050 ºC, dan (c). 1150 ºC.

The Effect of Amount and Types of Reductors

The greater the amount of reductors used, the higher the concentration produced [20]. Figure 6 shows the level and yield of Ni and Fe concentrates from the



Reductor (%weight)	5	10	15
Grade Ni (%)	6,09	5,13	2,25
Acquisition Ni (%)	70,30	89,57	63,09
Grade Fe (%)	56,18	70,89	79,77
Acquisition Fe (%)	17,89	34,34	62,02

Figure 6. The effect of the number of reductors on concentrates with additives of 10% Na_2SO_4 at a temperature of 1150 °C and for 60 minutes against: (a). Ni and Fe levels and (b). Ni and Fe recovery.

reduction of laterite nickel ore using anthracite coal reductors. Based on the graph, the iron content increases along with the increase in the amount of reductors, from 56.18% to 79.77%. Likewise on the yield of iron metal. The more reductors are added, the CO gas produced during the reduction process will also increase and cause the reduction process of iron oxide into metal become more optimal. However, this phenomenon is not desirable in a selective reduction process, where the expected conditions are high levels and yields of nickel by obtaining low iron levels and yields on the concentrate. From Figure 6, nickel content tends to decrease from 6.1% in the addition of 5% reductor to 2.25% in the addition of 15% reductor. The same thing happened to the yield of nickel metal, although it had increased at the addition of 10% reductor. The higher the addition of reductor, the lower the level and yield of nickel [12]. The increase in the level of reductor to reach the optimal amount of carbon can contribute to the reduction of iron and nickel so that the metal yield increases, but excessive carbon can resist the increase in nickel levels

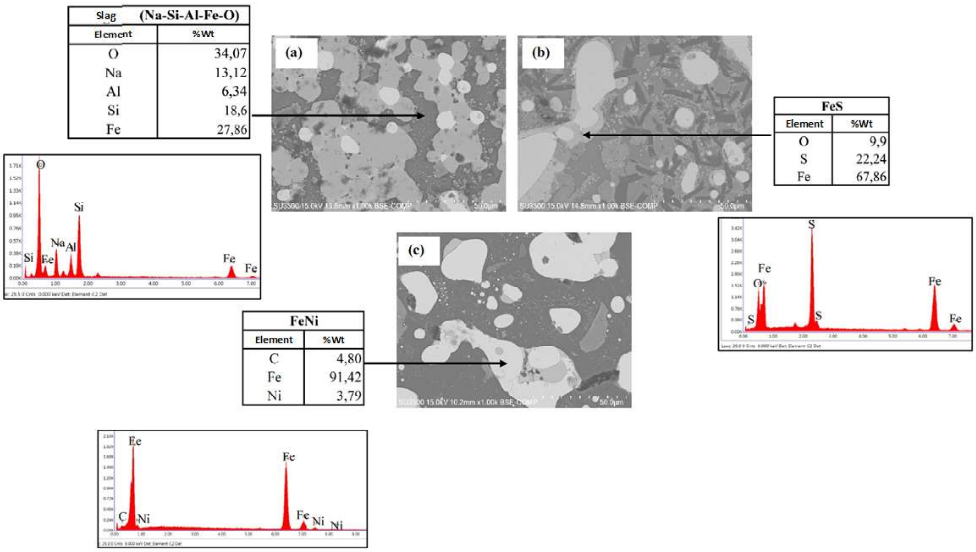


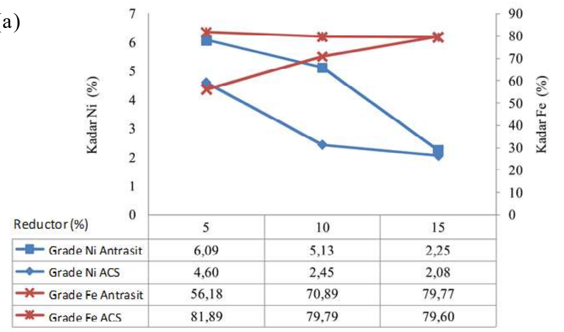
Figure 7. Microstructure (SEM-EDAX photo magnification of 500 BSE) of pellets which has been reduced at a temperature of 1150 °C for 60 minutes with 10% Na_2SO_4 with anthracite coal reductors: (a). 5, (b) 10, and (c). 15% by weight.

and also lead to decreased nickel yield [21]. Nickel levels decrease due to the formation and increase in the amount of iron [22]. The addition of carbon (derived from reducing reductors) which is greater than that needed for the purpose of obtaining high nickel yields can actually reduce nickel levels due to an increase in reduction of iron oxide to metallic iron [3].

From the results of microstructure analysis (Figure 7), bright grains are from metal (ferronickel) particles formed, while dark grains are identified as impurities. From Figure 7, it appears that there is a change in the grain size formed due to the reduction process of laterite nickel ore with the addition of anthracite coal reductor as 5-15%. In addition to the amount of reductor 5% by weight, the ferronickel grain is not too much. The more additions to the number of reductor, the more ferronickel grains get larger and bigger because the more iron oxide is reduced to iron metal and into ferronickel, which is not desirable.

Figure 8 shows the effect of using the types of palm shell charcoal and antithetite coal reductors on the levels and yields of iron and nickel at various reduction temperatures. From the experimental results it appears that, the higher the reduction temperature, the higher the level of nickel in the concentrate. The highest nickel level in both concentrates was obtained at 1150 ºC reduction temperature. The ore reduced by anthracite coal has a higher nickel level, which is 6.1% Ni, compared to palm shell charcoal, which is 4.6% Ni. For iron level,

(a)



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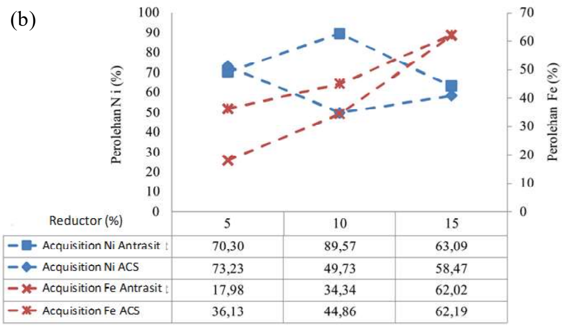

Figure 8. (a). The effect of the types of reductors on the levels and recovery of Ni and Fe in concentrates of 10% Na_2SO_4 at temperatures of 950 °C, 1050 °C and 1150 °C for 60 minutes with reductors: (a). Anthracite, (b). Palm shell charcoal

the nickel ore reduced by anthracite produces lower levels of iron, which is 61.6% compared to palm shell charcoal, which is 81.89%.

The same thing happened to the yields of nickel and iron. At a temperature of 1150 °C, the yield of nickel from both types of concentrate is not much different. As for iron yield, concentrates with anthracite coal reductor produce lower iron yield compared to concentrates with palm shell charcoal reductor.

From the results of these experiments, it was found that laterite nickel ore which was reduced by anthracite coal produced better concentrations, which concentrates with high nickel content and low iron content were obtained. This is due to the higher sulphur content found in anthracite coal reductor compared to palm shell charcoal, as shown in Table 4.

Table 4. The analysis results of anthracite coal and palm shell char-coal

Reductor	Component	
	Sulphur (%)	Calory (Cal/g)
Anthracite Coal	3.14	18.25
Palm Shell Charcoal	0.43	22.57

The presence of sulphur, both from additives and reductors will increase the rate of kinetic reduction and reduce iron metallization and facilitate the formation of FeS which can increase nickel selectivity so that nickel yield increases [12]. Therefore, the more sulphur content, the more FeS is formed and FeS can increase the nickel level in concentrate by suppressing the formation of iron into metal which causes the iron to enter the tailings so that the iron yield in concentrate becomes low [16].

CONCLUSION

Addition of Na_2SO_4 additive to the selective reduction of low level laterite nickel ore with a content of 1.4% Ni can increase nickel content in concentration up to 6.09% Ni at a reduction temperature of 1150 ºC for 60 minutes. In addition, the presence of Na_2SO_4 contributes to an increase in the particle size of ferronickel, where the size becomes larger and thus has a positive impact on the magnetic separation process which results in increased nickel metal concentrations. While the addition of reductors results in decreased levels of nickel in concentrates due to the optimal reduction of iron oxide to metal. The highest nickel level is obtained at the amount of 5% reductor. Ore, which is reduced by anthracite coal reductor, produces higher concentrations of nickel level and lower iron compared to ore reduced by palm shell charcoal. This is due to higher sulphur content in anthracite coal. Sulphur can react with iron and form FeS which can reduce iron metallization to increase the nickel level in concentrate.

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