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# EFFECT OF SULFUR IN THE REDUCTANTS ON SULFIDATION MECHANISM OF NICKEL LATERITE

## *PENGARUH SULFUR DALAM REDUKTAN TERHADAP MEKANISME SULFIDASI BIJIH NIKEL LATERIT*

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**ABSTRACT**

Processing nickel laterite conventionally, namely by pyrometallurgy method, requires high temperature and energy, results in a costive process. Due to its lower temperature reduction process, selective reduction with additives could be an alternative in nickel ore processing. Additives such as sulfur/sulfate have a critical role in promoting the low melting point phase. Sulfur is also found in coal. Therefore, it is important to investigate the effect of sulfur content in reductant on selective reduction of lateritic nickel ore. In this work, the effect of sulfur content (2.68% and 5% S) in anthracite coal as a reductant on selective reduction of limonitic ore was studied clearly. Nickel ore, reductant and sodium sulfate were mixed homogenously and pelletized up to 10-15 mm in diameter. Pellets were reduced using a muffle furnace at 950 to 1150°C for 60 min. Reduced pellets were crushed into -200 mesh before separating the ferronickel and its impurities using a wet magnetic separation process. The result showed that the anthracite coal with 5% S produced concentrate containing 3.56% Ni with 95,97% recovery, which is higher than 2.68% S. The sulfur content in reductant could replace the addition of sulfur/sulfate as the additives in the selective reduction of lateritic nickel ore.

Keywords: selective reduction, nickel laterite, reductant, sulfur, ferronickel.

***ABSTRAK***

*Pengolahan bijih nikel laterit secara konvensional (pirometalurgi) membutuhkan temperatur dan energi tinggi, sehingga proses akan menjadi mahal. Reduksi selektif dengan penambahan aditif dapat menjadi salah satu alternatif dalam pemrosesan bijih nikel laterit dikarenakan penggunaan temperatur proses yang lebih rendah. Aditif seperti sulfur/sulfat memiliki peran penting terhadap pembentukan senyawa logam dengan titik lebur rendah. Sulfur juga terkandung dalam batubara. Oleh karena itu, perlu dipelajari mengenai pengaruh sulfur dalam batubara terhadap proses reduksi selektif bijih nikel laterit. Dalam penelitian ini telah dipelajari mengenai pengaruh kandungan sulfur (2,68% dan 5% S) dalam batubara antrasit sebagai reduktan dalam proses reduksi selektif. Bijih nikel, reduktan dan natrium sulfat dicampur hingga homogen dan dibuat menjadi bentuk pellet berukuran diameter 10-15 mm. Pellet tersebut direduksi menggunakan* muffle furnace *pada temperatur 950 hingga 1150°C selama 60 menit. Pellet hasil reduksi tersebut digerus hingga berukuran -200 mesh sebelum dilakukan proses pemisahan ferronikel dari pengotor menggunakan proses separasi magnet basah. Hasil penelitian menunjukkan bahwa batubara antrasit dengan kandungan 5% S menghasilkan konsentrat mengandung 3,56% Ni dengan* recovery *95,97%, lebih tinggi dibandingkan dengan 2,68% S. Kandungan sulfur dalam reduktan dapat menggantikan penambahan aditif sulfur/sulfat pada proses reduksi selektif bijih nikel laterit.*

*Kata kunci: reduksi selektif, bijih nikel laterit, reduktan, sulfur, ferronikel.*

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## INTRODUCTION

In 2020, Indonesia was known as the largest nickel reserves in the world, accounting for 21 million metric tons (Zovko and Romic, 2011; Kuck, 2016). It is generally found in the form of nickel laterite. The nickel is one of the strategic metals. Mostly, it is used as ferronickel in stainless steel production (Hang, Xue and Wu, 2020). Nickel resources are found in sulfidic or lateritic ore. Many industries are preferred to process the sulfidic ore, which has higher nickel content, rather than the lateritic ore. However, depletion of sulfidic ore has promoted the use of lateritic ore (Dalvi, Bacon and Osborne, 2004). Its low nickel content and complex metallic oxide structure, associated with magnesium and silicate, have become a challenge in lateritic nickel ore processing.

Many industries have chosen pyrometallurgy for nickel ore processing due to its well-proven technology, high productivity, and non-dangerous chemical waste produced (Oxley and Barcza, 2013; Zhu *et al.*, 2016). Conventional pyrometallurgy in nickel ore processing to produce ferronickel using a blast furnace and rotary kiln electric furnace (RKEF) has been well known (Rao *et al.*, 2013). However, its high-temperature process, which means high energy needs, has been limited for high-grade nickel ore processing due to its economic consideration, especially for nickel laterite processing. Therefore, many researchers have developed a new method for low-temperature processes, such as the reduction process followed with magnetic separation process (Nurjaman *et al.*, 2018; Keskinkilic, 2019).

Thermodynamic reduction of nickel laterite is expressed in Reaction (1-7). The reductant is used to promote the reduction atmosphere to reduce the metal oxide into ferronickel. Carbonaceous material, such as coal, is commonly used as a reductant for the reduction process of nickel ore (Li *et al.*, 2013). Bouduard reaction, expressed in Reaction (1), will transform carbon into carbon monoxide (CO) as a reductant gas, then it will reduce the metallic oxide. From carbothermic reduction, illustrated in Figure 1, shows that the nickel oxide will be reduced into metallic nickel at 400°C, while the iron oxide at 648°C for $Fe_3O_4$ and 695°C for FeO. Therefore, the nickel oxide will be reduced earlier than the iron oxide (Rao *et al.*, 2013).

$$C + CO_{2(g)} \rightarrow 2CO_{(g)} \quad \text{.......} (1)$$

$$(Fe,Ni,Mg)_3Si_2O_5(OH)_4 \rightarrow$$
$$3/2(Fe,Ni,Mg)_2SiO_4 + ½SiO_2 + 2H_2O_{(g)} \quad \text{.......} (2)$$

$$(Fe,Ni)OOH \rightarrow (Fe,Ni)_2O_3 + H_2O_{(g)} \quad \text{.......} (3)$$

$$(Fe,Ni)_2O_3 + CO_{(g)} \rightarrow (Fe,Ni)_3O_4 + CO_{2(g)} \quad \text{..} (4)$$

$$(Fe,Ni)_3O_4 + CO(g) \rightarrow (Fe,Ni)O + CO_{2(g)} \quad \text{...} (5)$$

$$(Fe,Ni)O + CO_{(g)} \rightarrow (Fe,Ni) + CO_{2(g)} \quad \text{.......} (6)$$

$$Fe + Ni \rightarrow FeNi \quad \text{.......} (7)$$

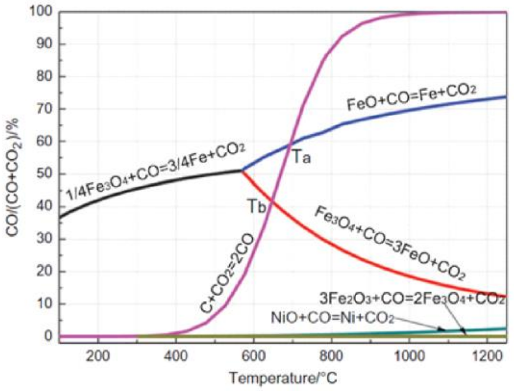

Figure 1. Gas equilibrium on carbothermic reduction for nickel and iron oxide (Rao *et al.*, 2013)

Ferronickel price depends on the nickel grade, while the iron content will pay for free. Therefore, it is very important to produce ferronickel with a high nickel grade by lowering the iron recovery or inhibiting iron's metallization. According to Rao *et al.* (2016), the initial temperature reduction of nickel is lower than that the iron. Thus, it is possible to control selectivity reduction between nickel and iron. Another method to suppress the iron metallization is by adding some additives in the reduction process of the nickel ore, such as carbonate, chloride, and sulfate (Dong *et al.*, 2018). However, according to the previous study, additive sodium sulfate has generated in the highest nickel grade due to the sulfidation mechanism, as expressed in Reaction (8-9) (Suharno *et al.*, 2021). The inhibition of iron metallization was due to the formation of iron sulfide or troilite (FeS), which has non-magnetic properties. Iron sulfide also has a low melting point temperature (988°C), which could promote the agglomeration particle of ferronickel (Li *et al.*, 2012). It is important for the next stage, i.e., the magnetic separation process, to separate ferronickel (magnetic) and impurities (non-magnetic). The large ferronickel size will enhance the liberation degree of ferronickel. Thus, it will result in high recovery of nickel.

$$Na_2SO_4 + 4CO \rightarrow Na_2S + 4CO_{2(g)} \ldots\ldots\ldots\ldots (8)$$
$$Na_2S + FeO + 2SiO_2 \rightarrow FeS + Na_2Si_2O_5 \ldots (9)$$

The use of sodium sulfate in the selective reduction process has also been reported by Jiang *et al.* (2013). Adding 10wt% of sodium sulfate and 2 wt% of bituminous coal has increased the grade of nickel ore from 1.49% to 9.87% with a recovery value of 90.90% after magnetic separation process. Therefore, sulfur/sulfate is very important in the selective reduction process. From the simulation thermodynamic of nickel laterite reduction process, Harjanto and Rhamdhani (2019) reported that sulfur in coal (as reductant) could promote the sulfidation mechanism in the selective reduction process. Nevertheless, there is still less information regarding the experiment to prove this study. Therefore, in this work, the effect of sulfur in anthracite coal on the selective reduction process of limonitic nickel ore has been investigated clearly.

## METHOD

### Materials

The studied limonitic nickel ore in this research was taken from Southeast Sulawesi, Indonesia. The main composition of the ore, characterized by the x-ray fluorescence (XRF), is listed in Table 1. The iron and nickel grades are 38.2% and 1.38%, respectively. An analysis of the mineral contained in nickel ore was carried out using the x-ray diffraction (XRD). The result is shown in Figure 2. It shows goethite (FeOOH), olivine ($MgFeSiO_4$), lizardite ($(Fe,Ni,Mg)_3Si_2O_5(OH)_4$), and quartz ($SiO_2$).

A two different anthracite coal was used as a reductant. Each coal was characterized by proximate and sulfur analysis, as listed in Table 2. Sodium sulfate was used in this research as an additive to promote the sulfidation mechanism in the selective reduction process, as expressed in Reaction (8-9).

Table 1. Chemical composition of limonitic nickel ore (wt%)

| Fe | Ni | $SiO_2$ | CaO | $Al_2O_3$ | MgO |
|---|---|---|---|---|---|
| 38.2 | 1.38 | 10.12 | 0.13 | 5.30 | 1.46 |

Table 2. Proximate and sulfur analysis of anthracite coal (wt%)

| Anthracite Type | Fixed Carbon | Volatile Matter | Moisture | Ash | S |
|---|---|---|---|---|---|
| A | 64.55 | 21.10 | 0.78 | 13.55 | 2.68 |
| B | 67.63 | 21.39 | 1.08 | 9.90 | 5 |

### Method

In this work, two kinds of anthracite coals, i.e., anthracite types A and B containing 2.68% and 5% of sulfur were used. Nickel ore, anthracite coal, and 10 wt% of sodium sulfate are crushed to less than 100 mesh. The anthracite coal was added in 0.0625; 0.125; and 0.25 of stoichiometry based calculation according to the metal oxide content in nickel ore. The materials were then homogeneously mixed before they pelletized into 10-15 mm of diameter. The reduction process of pellets was carried out using a muffle furnace at 950, 1050, and 1150°C for 60 min. The reduced pellets were then quenched rapidly in water to prevent the re-oxidation of the metallic phase. After that, the reduced pellets were crushed into -200 mesh before separating the ferronickel (as concentrate/magnetic) and impurities (as tailing/non-magnetic) using a wet magnetic separation process. Iron and nickel grade in concentrate was analyzed using XRF. They were also analyzed in tailing to obtain the recovery value using Equation 10.

$$\text{Recovery of Ni} = \frac{W_c \times Ni_c}{(W_c \times Ni_c) + (W_t \times Ni_t)} \ldots\ldots\ldots\ldots (10)$$

Where $W_c$ is the mass of concentrate, $Ni_c$ is nickel grade in concentrate, $W_t$ is the mass of tailing, $Ni_t$ is nickel grade in tailing. The same equation is also used to calculate the iron recovery. XRD and SEM-EDS analyses were performed to identify phase transformation and microstructure of reduced ore.

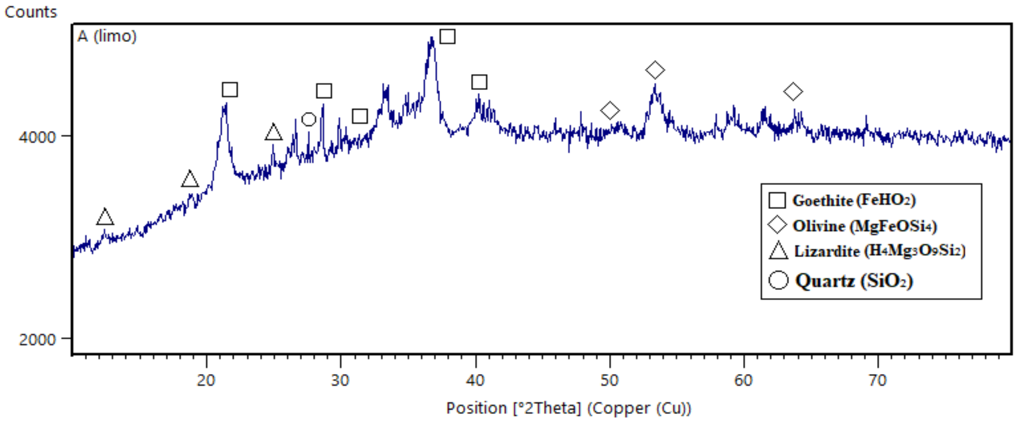

Figure 2. XRD pattern of limonitic laterite nickel ore

## RESULTS AND DISCUSSION

### Effect of Reduction Temperature

The effect of reduction temperature on the selective reduction of limonitic nickel ore was studied. Pellets with anthracite type B containing 5% S with 0.25 stoichiometry and 10 wt% of sodium sulfate were used. Figure 3 shows that the nickel grade and the recovery increase with the increase of temperature reduction. A similar trend is also found in the iron grades. However, the iron recovery slightly decreases with the increasing temperature to 1050°C. It increases after being reduced to 1150°C.

The reduction rate or metallization of metal oxide will increase with increasing the reduction temperature (Li *et al.*, 2012; Setiawan, Harjanto and Subagja, 2017). Thus, metal recovery will increase with the increase in temperature. However, the decrease of iron recovery at 1050°C is due to the sulfidation mechanism of iron sulfide or troilite (FeS) formation, as expressed in Reaction (9), which is occurred at 988°C (Elliott, Pickles and Peacey, 2017). Iron sulfide is a non-magnetic phase, and it will be collected in tailing. Therefore, it will lower the iron recovery. At 1150°C, the reduction mechanism is more powerful than that of the sulfidation mechanism (Febriana *et al.*, 2020). Thus, the iron recovery increases.

The XRD analysis is shown in Figure 4. As the reduction temperature rises, the intensity of magnesioferrite has decreased, indicating that the reduction rate increases with the increasing temperature. The magnesioferrite peak is no longer visible at 1150°C, which was transformed into wustite. Phases such as clinopyroxene and forsterite-ferroan are only observed at 950°C. The wustite phase was found in all temperatures. Wustite intensity is high at 950°C, but decreases at 1050°C, due to the formation of iron sulfide. An increase in wustite intensity can be observed at the increase in reduction temperature of 1050 to 1150°C. The ferronickel phase is observed at 1050 and 1150°C, but not at 950°C. The iron and nickel seem to be trapped in another phase at 950°C, such as nickel-bearing magnesioferrite and forsterite-ferroan. Therefore, the nickel grade and recovery are low at 950°C. Zhu *et al.* (2012) also found a nickel-bearing magnetite phase in the reducing nickel ore with the addition of 10 wt% sodium sulfate additive at 800°C. They also reported that the addition of sodium sulfate had weakened the reducing atmosphere due to the formation of iron sulfide (FeS) (Zhu *et al.*, 2012).

The microstructure analysis of reduced ore in various temperature reductions is presented in Figure 5. The ferronickel phase is shown with white grains, while the impurities are in a dark color. At 950°C (Figure 5a), the ferronickel grains are difficult to observe. When the reduction temperature has risen to 1050°C (Figure 5b), ferronickel grains began to grow. When the reduction temperature rose to 1150°C (Figure 5c), the small ferronickel particles were gradually agglomerated into larger grains. As the temperature increases, the average ferronickel diameter grows larger (Figure 6).

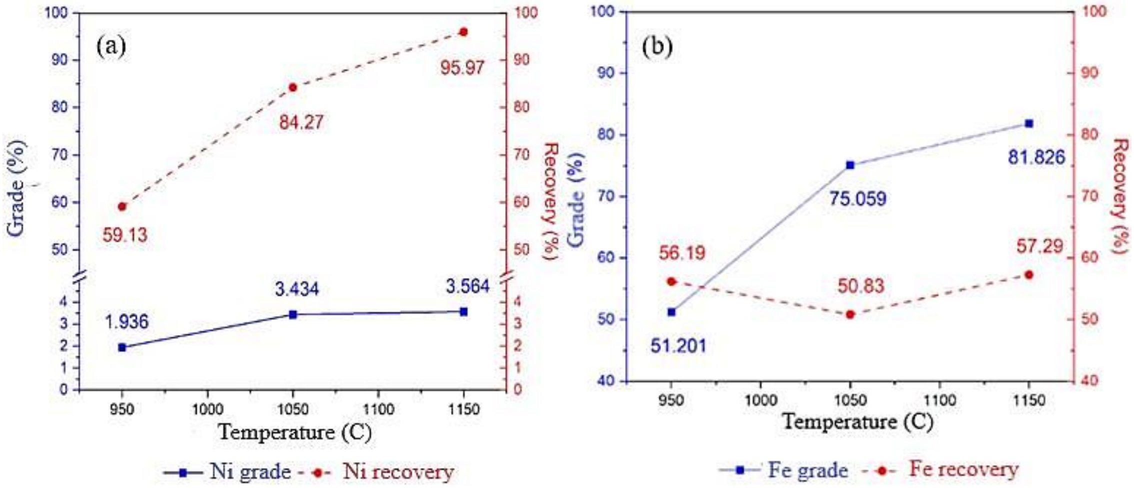

Figure 3. XRF analysis for grade and recovery of (a) nickel (b) iron at different temperatures on pellet type B with 0.25 stoichiometry

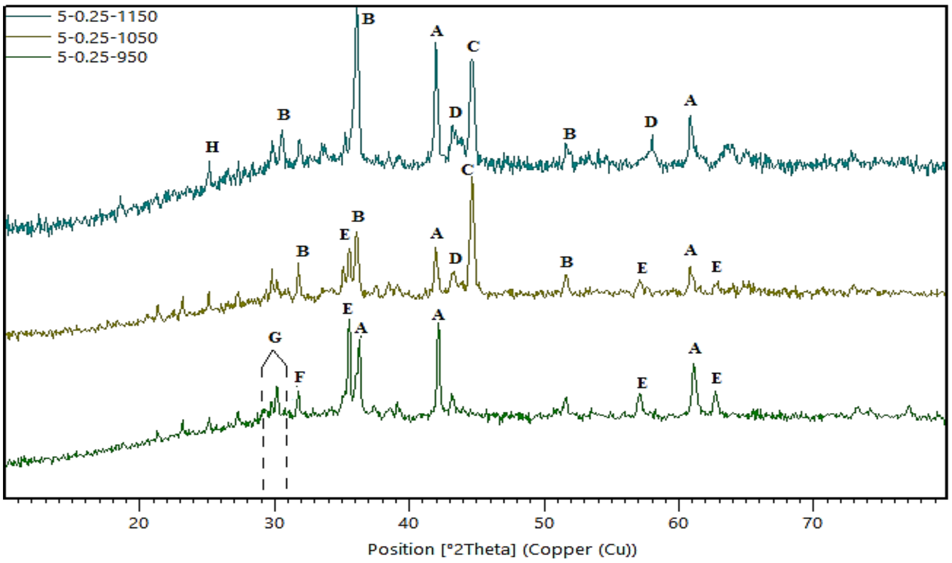

Figure 4. XRD pattern of the phases formed at different temperatures on pellet type B with 0.25 stoichiometry (A–Wustite (FeO); B–Olivine ($FeMgSiO_4$); C–Iron Nickel (FeNi); D–Iron Sulfide (FeS); E–Magnesioferrite (Ni bearing) ($(Mg,Ni)Fe_2O_4$); F–Forsterite-ferroan ($(Fe,Mg)SiO_4$); G–Clinopyroxene ($(Ca,Fe,Mg)Si_2O_6$); H–Quartz ($SiO_2$))

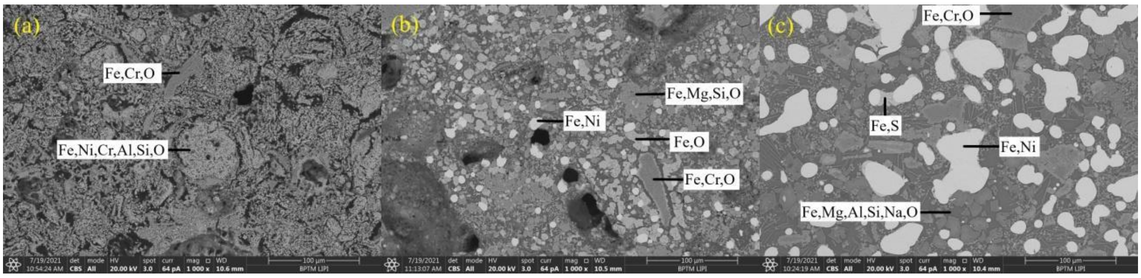

Figure 5. Microstructures of type-B reduced pellets with 0.25 stoichiometry at (a) 950, (b) 1050, and (c) 1150°C

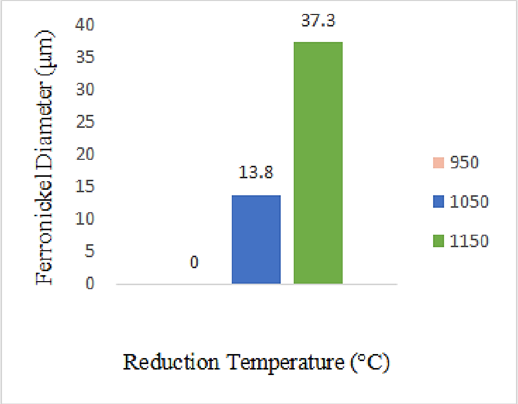

Figure 6. The average ferronickel size after reduction at various temperatures on pellet type B with 0.25 stoichiometry

## Effect of Reductant Amount

Effect of reductant amount based on stochiometric calculation, i.e., 0.0625, 0.125 and 0.25, has been investigated. The type-B reductant and 10 wt% of sodium sulfate was used in this selective reduction process at 1150°C for 60 min. Figure 7, it shows the nickel grade increase with the increase of reductant amount, while nickel recovery is slightly decreased and the iron recovery significantly decreases. It might be due to the promotion of the sulfidation process, whereas sulfur content increases with more coal addition. From the XRD analysis, as shown in Figure 8, troilite intensity increases with the increase in reductant addition. Ferronickel peaks decreases due to the low recovery of nickel, which indicates high nickel grade within the concentrate. The more iron sulfide formed, the more liquidus phase resulted, which could suppress the reducing atmosphere. It was indicated with additional wustite and forsterite-ferroan observed by the increasing of reductant addition.

From microstructure analysis in Figure 9, the reduced pellet with 0.25 stoichiometry of reductant addition has the largest ferronickel size. Nepheline ($Na_2Si_2O_5$), shows a black area in Figure 9(c), has also low melting point phase. It was formed due to the sulfidation mechanism as expressed in Reaction (8-9). The presence of the liquidus phase in the reduction process will promote the agglomeration particle of ferronickel. Figure 10 shows that the ferronickel size increase with the increase of reductant.

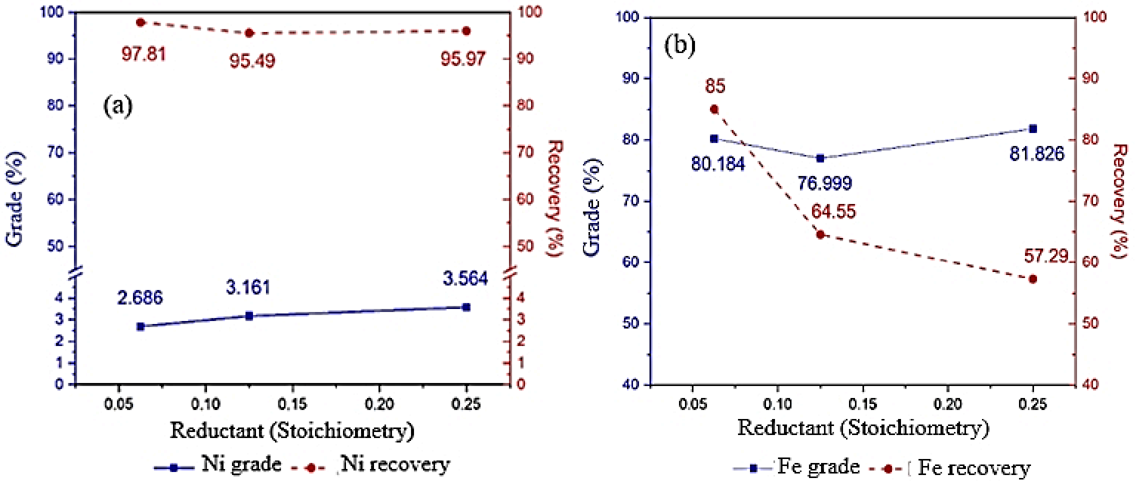

Figure 7. XRF analysis for grade and recovery of (a) nickel, (b) iron at various reductant amounts on pellet type B

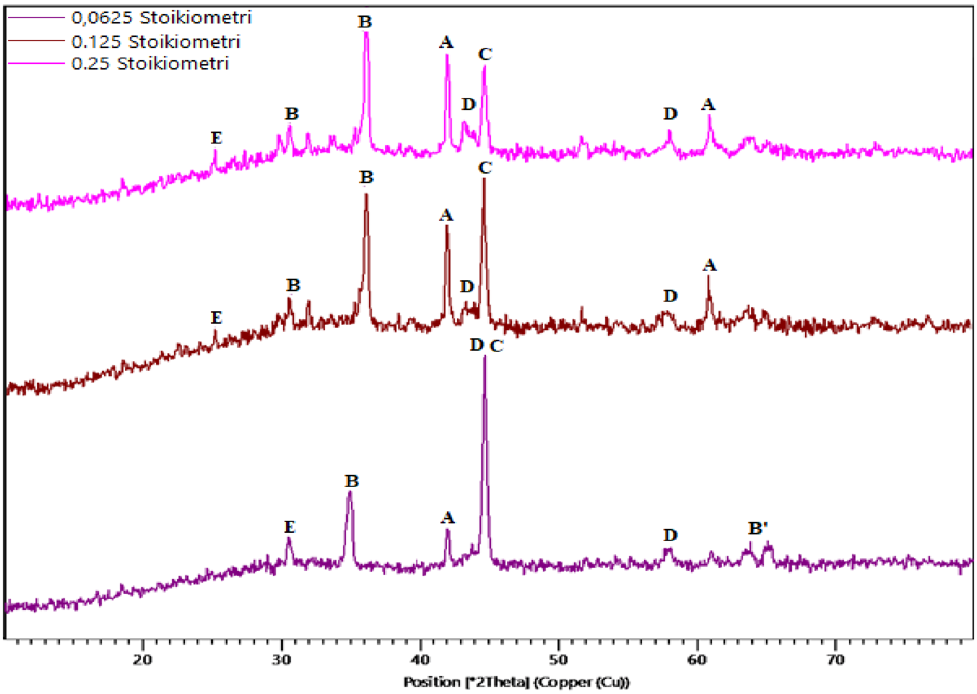

Figure 8. XRD pattern of the phases formed at different additions of reductant on pellet type B. (A–Wustite (FeO); B–Olivine ($FeMgSiO_4$); B'–Forsterite-ferroan ($(Fe,Mg)_2SiO_4$); C– Iron Nickel (FeNi); D–Iron Sulfide (FeS); E–Quartz ($SiO_2$))

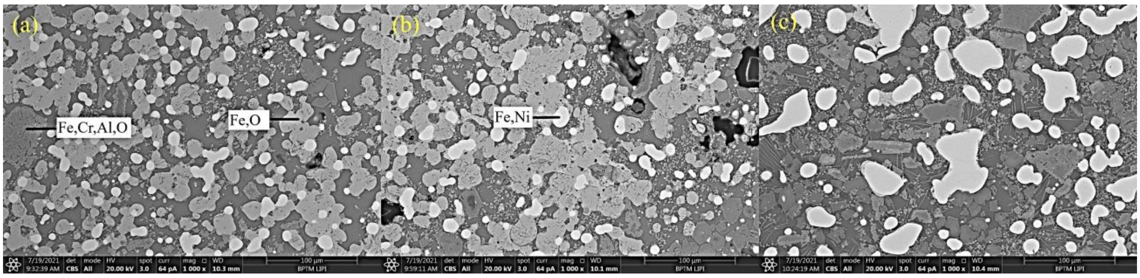

Figure 9. Microstructures of reduced pellets with various reductant addition of (a) 0.0625, (b) 0.125, and (c) 0.25 stoichiometric on pellet type B.

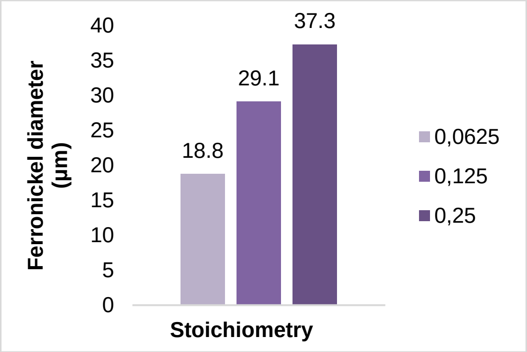

Figure 10. The average ferronickel size after reduction with various reductant addition on pellet type B.

## Effect of Sulphur Content in Reducing Agent to Ferronickel

Two different anthracite coals were used to investigate the effect of sulfur in coal on the selective reduction process, i.e., anthracite types A and B containing 2.68% S and 5% S, respectively, as listed in Table 2. The 0.25 of stoichiometry coal addition and 10 wt% of sodium sulfate were added to nickel ore. The reduction process was carried out at 1150°C for 60 min.

Figure 11 shows that the nickel grade and recovery of concentrate with 5% S are larger than 2.68% S. The iron recovery is lower. It indicates the sulfur content in the reductant influence the selective reduction process of nickel laterite by promoting the sulfidation mechanism.

According to Calkins (1994), most of sulfur in coal is found as a pyrite ($FeS_2$). It will react with $CO_2$ gas, which generate from Reaction (4-7), to produce FeS, CO and $SO_2$, as expressed in Reaction (11) (Lv *et al.*, 2015). Thus, more troilite will be produced from coal type B with higher sulfur content. As previously explained, troilite plays an important role in selective reduction process of nickel laterite. The CO gas produced from Reaction (11) will also promotes the reduction rate of metallic oxides.

$$FeS_2 + 2CO_2 \rightarrow FeS + 2CO + SO_2 \text{ .........(11)}$$

From the XRD analysis, shown in Figure 12, iron sulfide is found in both reduced pellets with 2.68% S and 5% S. Nevertheless, reduced pellets with 5% S have higher wustite and ferronickel peaks.

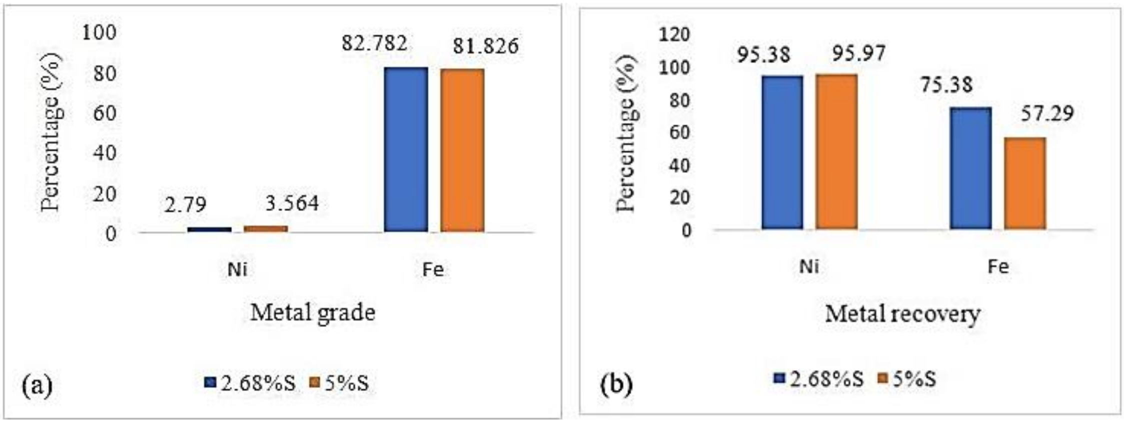

Figure 11. Metal grade (a) and recovery (b) with different sulphur contents in reductant

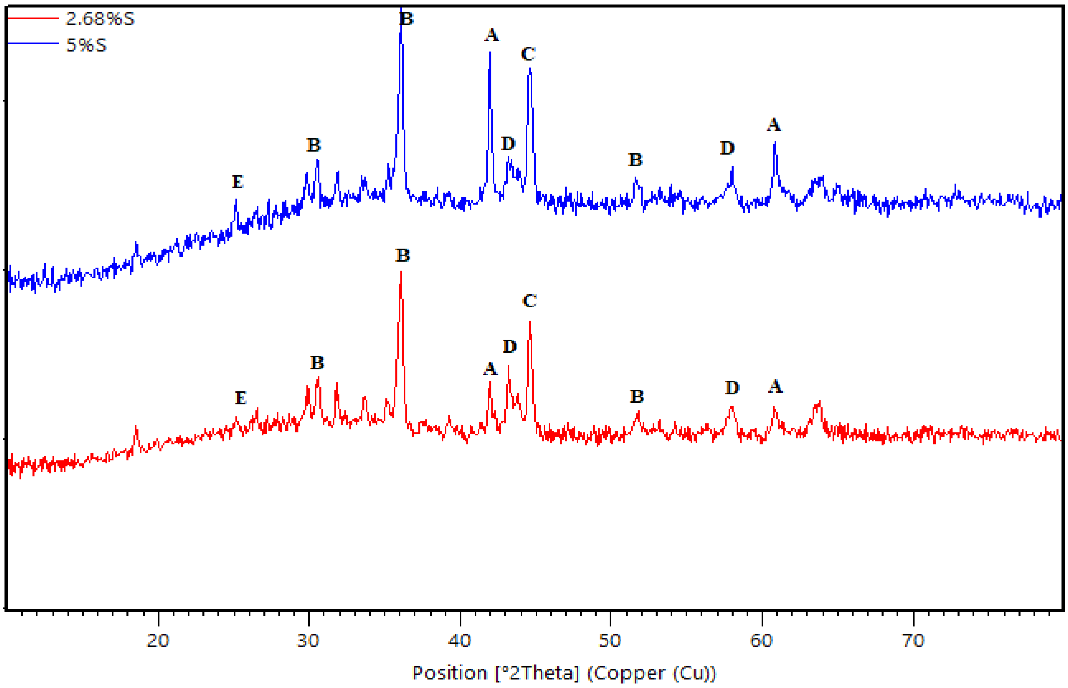

Figure 12. XRD pattern of the reduced pellets with different sulphur contents in reductant. (A–Wustite (FeO); B – Olivine ($FeMgSiO_4$) ; C–Iron Nickel (FeNi); D–Iron Sulfide (FeS); E–Quartz ($SiO_2$)).

Iron sulfide is found from the microstructure analysis of reduced pellet with 5% S, as shown in Figure 13(b). It has the largest ferronickel size, as shown in Figure 14. From the SEM-EDS picture, iron sulfide covers the ferronickel particle. According to Zhu *et al.* (2019), the liquidus iron sulfide could lower the surface tension of ferronickel particles, promoting the agglomeration of ferronickel particles. From Figure 13(b), more wustite is also found than that of Figure 13(a). It indicated that the sulfur content in the reductant also could inhibit the metallization of iron oxide. Thus, it increased the nickel grade in concentrate. Therefore, it seems the sulfur content in reductant could replace the use of external additives in the selective reduction process of nickel laterite, which lowers the cost of production in this nickel ore processing.

## CONCLUSION

Reduction temperature and amount of reductant are important parameters in the reduction of nickel laterite ore. The nickel grade and recovery increased with the increase of temperature. A similar result was also obtained with the increase in reductant addition. It was due to the increased reduction in the atmosphere, which could enhance the reduction rate of metallic oxide.

Sulfur is also very important in the selective reduction process of nickel laterite, which could inhibit iron oxide's metallization and promote the agglomeration of ferronickel size. Thus, it could increase the nickel grade and recovery in concentrate. The sulfur content in the reductant has shown a similar role with the addition of sulfate/elemental sulfur additive. In this work, a higher sulfur (5% S) in reductant has resulted in a higher nickel grade and recovery of ferronickel concentrate, i.e., 3.564 and 95.97%, respectively. Therefore, sulfur in a reductant could substitute the addition of sulfate (additive) in selective reduction of lateritic nickel ore.

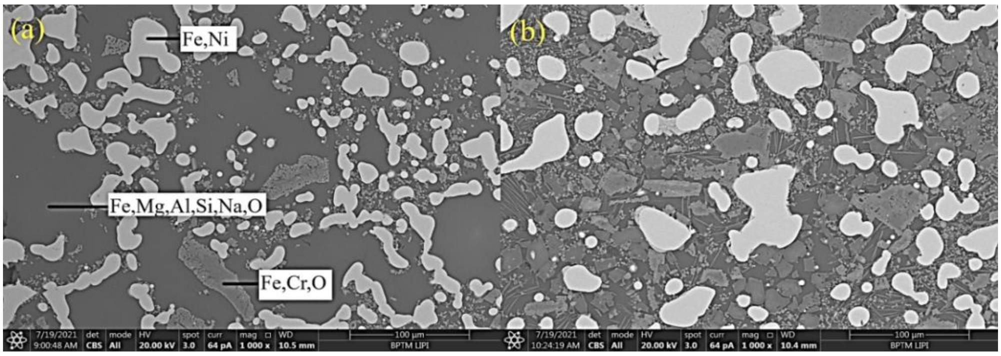

Figure 13. Microstructures of reduced pellets with sulphur content of (a) 2.68% and (b) 5% with 0.25 stoichiometric reduction at 1150ºC for 60 min.

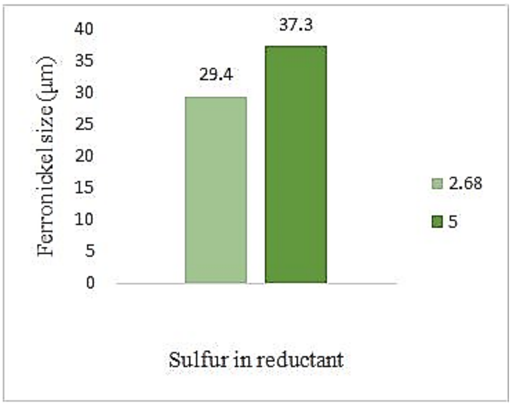

Figure14. The average ferronickel size of reduced pellets with different reductant types.

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