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Increasing ferronickel particle size from the carbothermic reduction of saprolitic nickel ore mixtured with iron ore

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ABSTRACT

Saprolite has higher nickel content than limonite. Nevertheless, iron and nickel in saprolite are associated with magnesium silicate structures resulting in lower reducibility, indicated by the presence of small particles of ferronickel in reduced ore. Thus, it results in a low recovery of nickel. Iron and sulfur play an important role in increasing the particle size of ferronickel due to the generation of the liquidus Fe-FeS phase, which could promote the agglomeration of ferronickel particles. Therefore, it is necessary to study the effect of mixing saprolitic nickel ore with iron ore in a selective reduction process to increase the ferronickel particle size and its effect on nickel content and recovery. The reduction process of a pellet composite comprising nickel saprolite, iron ore, coal (as reductant), and sodium sulfate (as additive) was carried out at 1150 °C for 60 minutes in a muffle furnace continued with rapid cooling by water quench. The reduced pellet was ground into less than 74 µm. The magnetic separation was carried out to obtain concentrate/magnetic particles (ferronickel) and tailing/non-magnetic particles (impurities). The research reported that mixing saprolite with iron ore could enlarge the ferronickel particle size in reduced ore due to enhancing the generation of troilite (FeS), which promotes the agglomeration of ferronickel particles. It increased the nickel and iron recovery but reduced the nickel content of ferronickel.

Keywords: saprolite; iron ore; selective reduction; ferronickel.

INTRODUCTION

Nickel is mostly used as an alloying element in ferrous material to improve wear resistance, metal forming, strength, ductility, and weldability (Ma et al., 2022). It is also potentially applied to battery cathodes and superalloys. However, two-thirds of nickel is used for stainless steel production. On Earth, nickel resources are found in laterite and sulfide. The depletion of sulfide, which has higher nickel content, causes some industries to extract nickel from laterite to produce ferronickel, nickel matte, and electrolytic nickel for the stainless steel-making process. Lateritic nickel ore generally consists of two types, namely limonite and saprolite. Limonite is located near the surface, with low nickel, magnesium, and silicon but high iron content. Saprolite is found in the deeper layer and has an opposite chemical composition to limonite (Butt and Cluzel, 2013). Mostly, saprolite is pyrometallurgy processed for producing ferronickel, while limonite is chemically processed to generate nickel sulfate.

The pyrometallurgical process is carried out by heating lateritic nickel ore with some reductants and fluxes. Rotary kiln electric furnace (RKEF) is a well-proven technology for smelting nickel ore (saprolite and sulfide) to produce ferronickel containing 12-25 wt% of nickel and slag (Gleeson et al., 2003; Nurjaman et al., 2021a). This smelting process consists of several stages; there are: drying the nickel ore in a rotary dryer at 100-120 °C to evaporate the moisture, heating the nickel ore in a rotary kiln at 800-900 °C, and smelting in an electric furnace at 1500-1600 °C In the rotary kiln, there is dehydroxylation process of serpentine $(Mg,Fe,Ni)_3Si_2O_5(OH)_4$, as expressed in Equation (1) at 550-850 °C, and partial reduction by adding some limited of coal (as reductant) to reduce the metal oxide into metallic iron and nickel-containing 10% and 80%, respectively (Coronell et al., 2022). Some calcium oxide (as fluxes) also can be added to lower the melting point of the slag.

Specific energy consumption for smelting limonite in RKEF is about 4.5-5 MWh/ton product (Erasmus and Fourie, 2015), which is higher than the ferrosilicon, ferromanganese making process. It also contributes to higher carbon dioxide gas emissions (Haque and Norgate, 2013). The high energy required means expensive in cost production. Therefore, the feasibility of nickel processing in RKEF is limited to high-content nickel ore to produce the costly high-content ferronickel.

Selective reduction is developed as an alternative nickel laterite process (Bahfie et al., 2022). This process consists of carbothermic reduction of nickel laterite with some additive and reductant at temperatures around 1200 °C, continued with grinding and wet magnetic separation process for segregating ferronickel from impurities.

The Reaction (1-5) shows the reduction mechanism of saprolite. The important mechanism in this selective reduction process is how to inhibit the metallization of iron to obtain high-content ferronickel and enlarge the ferronickel particle. The ferronickel particle's size affects nickel recovery from the reduction process. The larger the particle size, the easier it is to separate from impurities in grinding, continuing with the magnetic separation process (Arol and Aydogan, 2004).

$$(Mg,Fe,Ni)_3Si_2O_5(OH)_4 \rightarrow (Mg,Ni)SiO_3 + (Mg,Fe,Ni)_2SiO_4 + H_2O(g) \quad (1)$$

$$FeO + CO \rightarrow Fe + CO_2 \quad (2)$$

$$NiO + CO \rightarrow Ni + CO_2 \quad (3)$$

$$Na_2SO_4 + 4CO \rightarrow Na_2S + 4CO_2 \quad (4)$$

$$Na_2S + FeO + 2SiO_2 \rightarrow FeS + Na_2Si_2O_5 \quad (5)$$

$$Fe + S \rightarrow FeS \quad (6)$$

Reducibility is an important parameter in the selective reduction process, representing its suitability in reducing metal oxide contained in ore (Pal et al., 2016). The reducibility of metal oxide decreases with the increase of bond strength between metal and oxygen (Misono, 2013). In saprolite, iron and nickel oxide are associated with magnesium silicate structures $[(Mg,Fe,Ni)_3Si_2O_5(OH)_4]$, resulting in lower reducibility than limonite [(Fe,Ni) OOH]. It is indicated by the presence of a small particle of ferronickel after the reduction process of saprolite, resulting in low recovery of nickel. Some researchers have observed particle growth of ferronickel in the reduction of nickel laterite. The particle size of ferronickel increases due to the increasing reduction rate of metallic oxide to metallic iron and nickel, along with the increasing temperature and time reduction (Li et al., 2013; Jiang et al., 2020). According to Wang et al. (2017), saprolitic ore's optimal temperature and time reduction is 1350 °C for 2 hours. The solid reductant addition contributed to the growth of ferronickel particles due to the increased reduction atmosphere with CO gas generated. Nevertheless, the more solid reductant added, the more iron oxide will reduce into metallic iron. Thus, suppressing the nickel content in ferronickel (Nurjaman et al., 2021b). The use of limited solid reductant in this selective reduction process should be considered. Iron and sulfur play an important role in increasing ferronickel particle size due to the generation of the liquidus Fe-FeS phase, which could promote the agglomeration of ferronickel particles. Sodium sulfate is a proper sulfur-bearing additive for the selective reduction of nickel laterite (Jiang et al., 2013; Nurjaman et al., 2019). Li et al. (2012) reported that the sodium sulfate addition in nickel laterite reduction could improve the ferronickel particle up to 50 μm compared without the additive, which is about 5-10 μm. Adjusting the slag basicity to enhance the growth of ferronickel particles at low-temperature reduction has been carried out (Nurjaman et al., 2021c). The optimum basicity has been found at 0.6 of ternary basicity with CaO (as fluxing agent) addition which could break the iron and nickel from magnesium silicate structure and promote the low melting point phase, such as olivine. However, increasing temperature and time reduction, the addition of additives, and flux will increase the production cost.

Improving the reducibility of saprolite by mixing with limonite also has been carried out by (Rao et al., 2016), which reported the presence of iron oxide and metallic iron from limonitic ore could lower the reduction temperature of saprolite. However, the investigation has been limited only to the nickel content and recovery. Iron oxide is the main chemical compound in iron ore, and its reserve is abundant worldwide. Therefore, in this work, the iron oxide addition from iron ore was blended with saprolitic nickel ore to investigate its effect on growth particle size, phase transformation, nickel content, and recovery.

MATERIALS AND METHODS

Materials

The nickel and iron ore were from Indonesia. Its chemical composition was analyzed by using XRF, as listed in Table 1. The nickel ore is classified as low-grade saprolitic ore with nickel and iron contents are 1.73% and 12.68%, respectively. While iron ore is a high-grade with 64.34% of iron content. From XRD analysis, as illustrated in Figure 1, the iron in iron ore was found as Fe_xO_y, which is hematite (Fe_2O_3) and magnetite (Fe_3O_4). Contrary different with saprolite, iron and nickel are found as substitute elements of magnesium in lizardite $[(Fe,Ni,Mg)_3Si_2O_5(OH)_4]$, forsterite $[(Fe,Mg,Ni)_2SiO_4]$, and enstatite $[(Fe,Ni,Mg)SiO_3]$. Both iron ore and saprolite contain quartz (SiO_2) as impurities.

This process uses bituminous coal as a reductant and sodium sulfate as an additive. The proximate analysis of the reductant is listed in Table 2.

Table 1. Chemical composition of saprolite (wt%).

Ores	Elements (wt%)							
	Fe	Ni	Si	Al	Ca	Cr	Mn	S
Saprolite	12.68	1.73	8.30	1.03	0.53	0.41	0.24	0.09
Iron ore	64.34	-	3.93	2.11	0.23	-	0.28	0.31

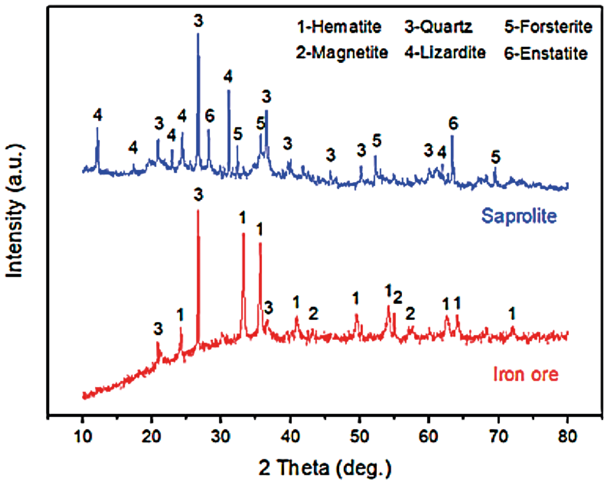

Figure 1. XRD analysis of saprolite and iron ore.

Table 2. Proximate analysis of reductant (wt%).

Fixed Carbon	Sulphur	Moisture	Volatile Matter	Ash
35.47	0.71	9.44	47.72	7.37

In this work, the effect of reductant dosage, the mixture of nickel laterite with iron ore, and sodium sulfate addition in the selective reduction process were investigated clearly on metal content and recovery, phase transformation, and particle growth of ferronickel. The composition of the samples is listed in Table 3. There is no repetition in data acquisition for each sample. However, the sample was carefully treated to minimize the data error.

Table 3. Composition of samples.

Variable	Ore	Reductant	Sodium Sulfate
Reductant	Saprolite	0.1- 1 stoichiometric	10 wt% of ore both for saprolite and iron ore
	Iron ore	0.1-0.5 stoichiometric	
The mixture of saprolite with iron ore	0-100% (of mass weight)	Optimum stoichiometric (0.2)	10 wt% of mixed ore
Sodium sulfate	Optimum mixture	Optimum stoichiometric (0.2)	10-30 wt% of mixed ore

Methods

A 50 gram of ore (saprolite and iron ore), some bituminous coal, and sodium sulfate (as listed in Table 3) were prepared into less than 149 µm and mixed homogenously prior to agglomerating into a 10-15 mm diameter of pellet. As shown in Figure 2, the green pellets were reduced in a muffle furnace at 1150 °C for 60 minutes in a closed-graphite crucible. It's quenched with water after the reduction process to prevent ferronickel's re-oxidization. The reduced ore was heated at 120 °C for 4 hours for drying and then crushed into less than 74 µm prior to the wet magnetic separation process using 500 Gausses of a magnetic field for obtaining the concentrates of magnetic ferronickel and the non-magnetic impurities.

RESULTS AND DISCUSSIONS

The Effect of Reductant Dosage

The effect of the reductant amount on the reduction process of saprolite and iron ore is shown in Figure 3. The increasing of metal content and recovery is found in a certain reductant stoichiometric addition. In iron ore, the optimum condition is found at 0.4 stoichiometry with iron content and recovery is 86.4% and 84.8%, respectively.

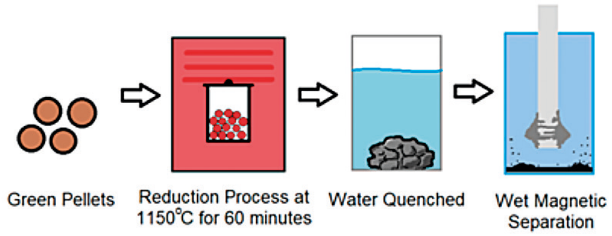

Figure 2. Schematic of work-flow diagram.

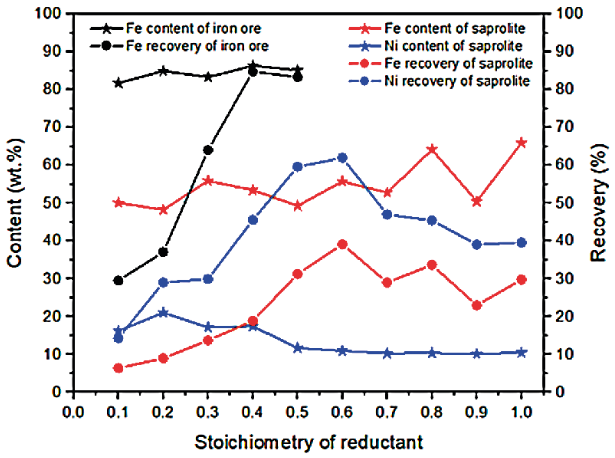

Figure 3. Effects of modified amount reductant on Fe-Ni content and recovery.

In saprolite, the optimum condition is obtained at 0.2 stoichiometry with nickel content and recovery at 21% and 29%, while iron content and recovery at 48.3 and 9.0%, respectively. The lower metal recovery in saprolite indicates that the metal compounds in saprolite are more difficult to reduce than iron oxide. It is due to the complex structure of metallic elements with magnesium silicate oxide in saprolite compared with the simple oxide structure in iron ore (Nurjaman et al., 2021d).

From XRD analysis in Figure 4, the metallic iron (Fe), fayalite (Fe_2SiO_4), wustite (FeO) troilite (FeS), and quartz (SiO_2) are observed in reduced ore with various reductant addition. At stoichiometry 0.2, the presence of wustite indicates that the reductant is insufficient to transform the iron oxide into metallic iron. The more reductants are added, the more metallic iron is generated. The transformation of wustite into metallic iron is expressed in Equation (2). Troilite, which is generated from the sulfidation mechanism, as described in Equations (5 and 6), also increases with the increasing of reductant addition. Fayalite is observed at a stoichiometric of 0.3, which is generated from the reaction of iron oxide and quartz (Jiang et al., 2013). At stoichiometry of 0.5, no-wustite and fayalite are found, which means all of them have been reduced completely, transforming into troilite and metallic iron.

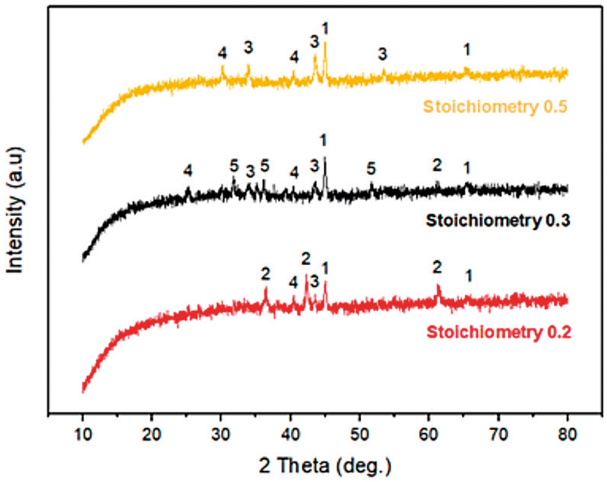

Figure 4. The XRD pattern of reduced iron ore with various reductant addition. 1-iron (Fe), 2-wustite (FeO), 3-troilite (FeS), 4-Quartz (SiO_2), 5-fayalite (Fe_2SiO_4).

Forsterite, ferronickel, and diopside are found in reduced saprolitic nickel ore with various reductants, as illustrated by the XRD analysis in Figure 5. Similar to the reduction of iron ore, the intensity of the metallic phase (ferronickel) increases with the increasing addition of reductants, followed by the slightly decreasing intensity of forsterite. Increasing the reductive atmosphere can reduce the potential of forsterite formation. The iron and nickel

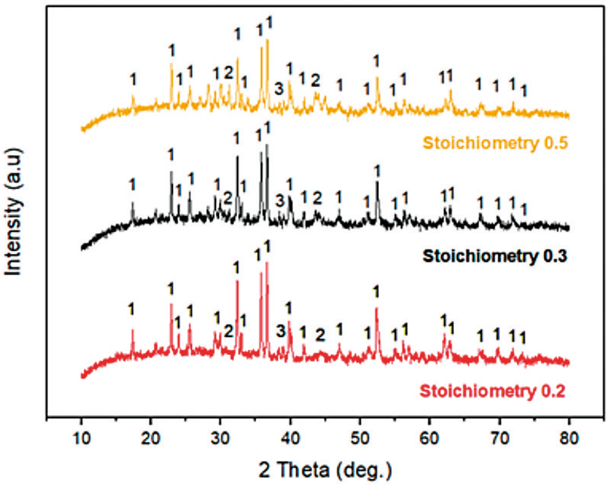

Figure 5. The XRD pattern of reduced saprolitic nickel ore modified amount reductant. 1-forsterite; 2-ferronickel; 3-diopside.

also will be released from its magnesium silicate structure of forsterite to form ferronickel. Nevertheless, the intensity of diopside, which is another complex of magnesium calcium silicate structure, is not changed with the increase of reductant. It indicates that the iron and nickel, which can also substitute the magnesium in diopside, are still trapped in their atomic structure (Kloprogge et al., 2017). Therefore, the presence of forsterite and diopside in various stoichiometry is associated with the low recovery of iron and nickel in the reduction process of saprolite, as shown in Figure 3.

The microstructure analysis of reduced iron ore is shown in Figure 6 (a-c). The particle size of metallic iron (white color) increases with the increasing reductant dosage. It is followed by the increasing volume of troilite (gray color) covering the spherical iron particle. Iron-troilite (Fe-FeS) eutectic has a lower melting point, about 980 °C. Thus, the reduction above this temperature will promote the liquidus phase of Fe-FeS, which is affected to the increase of diffusion rate and mass transfer for agglomerating the metallic iron particle. The large particle will be positively affected on the deliberation of the metallic phase from impurities by grinding and followed by a magnetic separation process. Nevertheless, the more formation of troilite will lower the recovery of iron. It is also illustrated in Figure 3, where the recovery of iron decreases from a stoichiometry of 0.4 to 0.5. Figure 6 (d) shows the saprolitic reduced ore microstructure analysis at a stoichiometry of 0.2. The particle size of metallic ferronickel (white color) is much smaller than metallic iron, as shown in Figure 7, which indicates that the metallic compound in saprolite is more difficult to reduce than iron ore.

Mixing of Saprolite with Iron Ore

In this section, the experiment was carried out to study the effect of mixing saprolite with iron ore in a selective reduction process on metal content and recovery, phase transformation, and particle growth of the metallic phase. The composition of the sample is listed in Table 3. From Figure 8, the nickel and iron recovery improves significantly as the iron ore is added increasingly into the saprolite. It also increases the iron content but decreasing the nickel content of ferronickel. The increase of iron content and recovery reaches the optimum at the addition of 60 wt% of iron ore. It is not changed with further addition of iron ore, which indicates that the iron oxide has

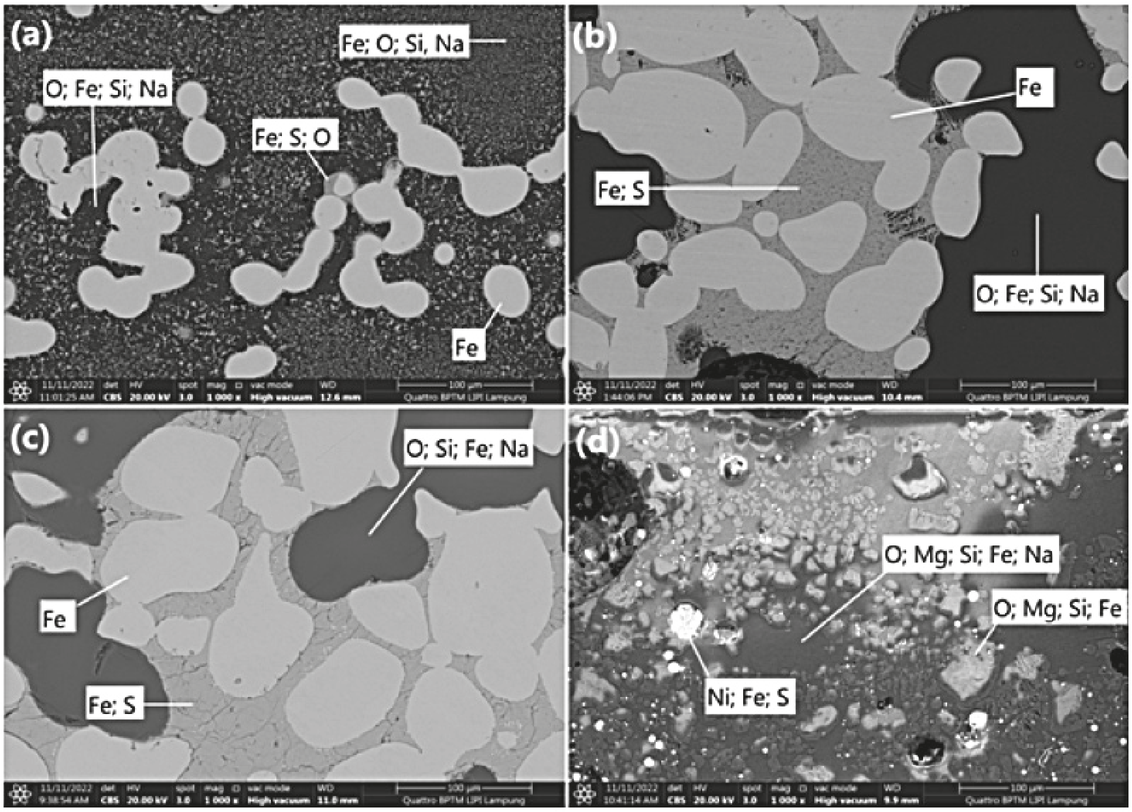

Figure 6. SEM-EDS analysis of reduced iron ore in various stoichiometries of (a) 0.2, (b) 0.3, (c) 0.5, and saprolite in the stoichiometry of: (d) 0.2.

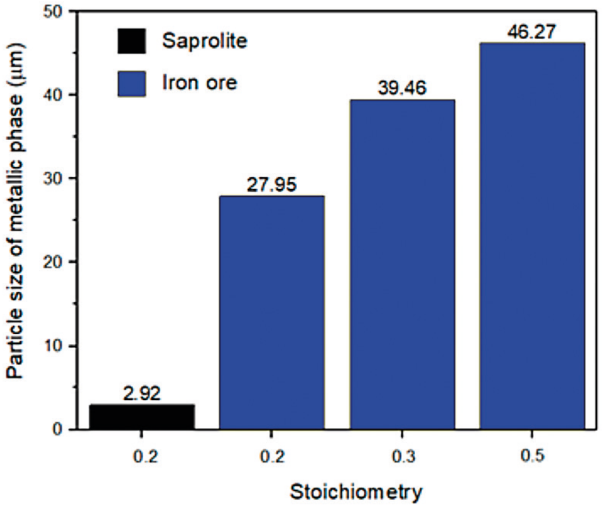

Figure 7. The particle size of the metallic phase of reduced saprolite and iron ore.

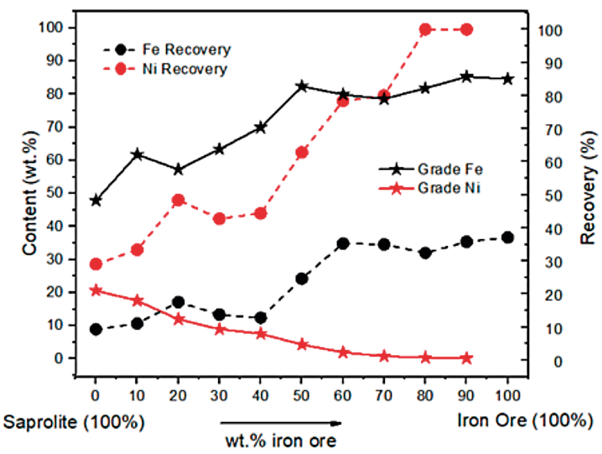

Figure 8. Effect of mixing saprolite with iron ore on iron-nickel content and recovery.

approached the maximum reduction limit transforming into a metallic phase. The highest nickel recovery is obtained by adding 80 wt% of iron ore. Nevertheless, the nickel content is very low. The more iron ore added, the more troilite (FeS) formed (Febriana et al., 2020). More liquidus phase will be generated when the reduction process is carried out above the Fe-FeS eutectic temperature, i.e., 980 °C. This large volume of liquidus phase in reduced mixed ore could promote the breakage of iron and nickel from the magnesium silicate structure in saprolite and dilutes into the metallic iron phase (Peng et al., 2019). Nevertheless, the presence of metallic iron will suppress the nickel content in metallic products (of ferronickel). It is explained in Figure 8 that increasing iron recovery is followed by decreasing nickel content.

The forsterite, ferronickel, diopside, and troilite phases are found in the XRD analysis of mixture-reduced ore, in which the composition of saprolite and iron ore (in wt%) is 80-20, 50-50, and 10-90, as shown in Figure 9. The reduction of reduced ore comprised of 100% saprolite is dominated by forsterite. The intensity of forsterite and diopside seems to decrease, while ferronickel increases with the increasing addition of iron ore in mixed ore. Nevertheless, the troilite intensity is not changed due to the presence of a limited reductant, which is not only used in the reduction of metal oxide but also in the decomposition of sodium sulfate for generating the troilite. At 10-90 of the saprolite-iron ore mixture, the nickel and iron recovery is 100% and 35.77%, respectively. It means that the presence of forsterite in this mixture contains no nickel. More iron oxide in a mixture could replace the nickel in a magnesium silicate structure with iron. Nevertheless, the more iron, the less nickel content in the concentrate is resulted. From Figure 8, the recovery of iron in 10% saprolite-90% is much lower than 100% iron ore. It seems that the presence of saprolite could inhibit the reduction of iron oxide into metallic iron. In Figure 9, the troilite seems to increase with the increase of iron ore in the mixed reduced ore due to the presence of more iron oxide, which reacted with sulfur. Based on XRD results (Figure 9), mixing saprolite with iron ore has been affected on reducing the presence of complex silicate phases such as forsterite and diopside in saprolitic nickel ore.

Figure 10 illustrates the microstructure of ferronickel in reduced ore. The particle size of ferronickel (white

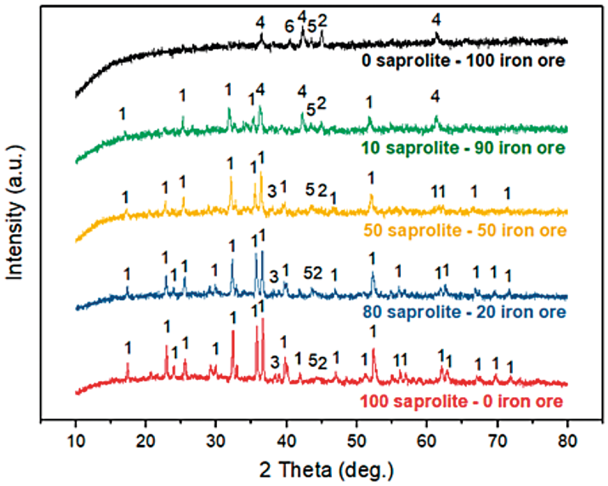

Figure 9. XRD pattern of reduced mixed ore modified amount iron ore. 1- forsterite; 2-iron/ferronickel; 3- diopside; 4- wustite; 5-troilite; 6-quartz.

color) increases with the increasing iron ore addition in saprolite. The sphere ferronickel is observed at 0, 20, and 50% of iron ore addition. At 90% addition of iron ore, the sphere of ferronickel agglomerated forms large-irregular ferronickel. The presence of iron ore could trigger troilite formation, which could increase the size of ferronickel particles by forming a liquid eutectic Fe-FeS phase, which could increase the mass transfer rate of ferronickel particles to agglomerate (Rao et al., 2016; Elliot et al., 2017).

In this experiment, mixing saprolitic nickel ore with iron ore has increased the particle size of ferronickel up to 24.24 µm at 10%-90% of saprolite-iron ore, as described in Figure 11. Nevertheless, the low nickel content of ferronickel results from this mixture of ore due to the presence of more metallic iron.

The Effect of Sodium Sulfate Addition

The various sodium sulfate was added to the 50% saprolite-50% iron ore mixture. The reduction process was carried out at 1150 °C for 60 minutes to investigate the effect of sodium sulfate addition on the selective reduction of mixed ore (saprolite and iron ore). Figure 12 shows that the nickel recovery and iron recovery increase with the increasing of sodium sulfate addition. However, it decreases the nickel content. At 30% of sodium sulfate addition, the iron and nickel recovery seems to decrease due to the presence of more sulfur which could generate more troilite (FeS) and nickel sulfide (NiS) (Liu et al., 2015).

From XRD analysis (Figure 13), the forsterite, ferronickel, diopside, troilite, and quartz phases are observed from mixed-reduced ore with various sodium sulfate addition. The peak intensity of the forsterite decreases with the increasing of sodium sulfate. It indicates that the presence of sodium sulfate can reduce the forsterite phase and partially change its composition to troilite and quartz, where the intensity of these two compounds increases. Meanwhile, the intensity of diopside and ferronickel is not changed significantly.

The addition of sodium sulfate in mixed ore of saprolite-iron ore has a role in increasing nickel content and nickel recovery through the formation of troilite, which can be affected on the enlargement of ferronickel particles. In this experiment, it was found that the addition of sodium sulfate above 25 wt% can reduce nickel content due to the effect of the increasing in iron recovery. Besides that, according to Elliot et al. (2017), the decrease in nickel content could be due to the presence of a small amount of nickel sulfide,

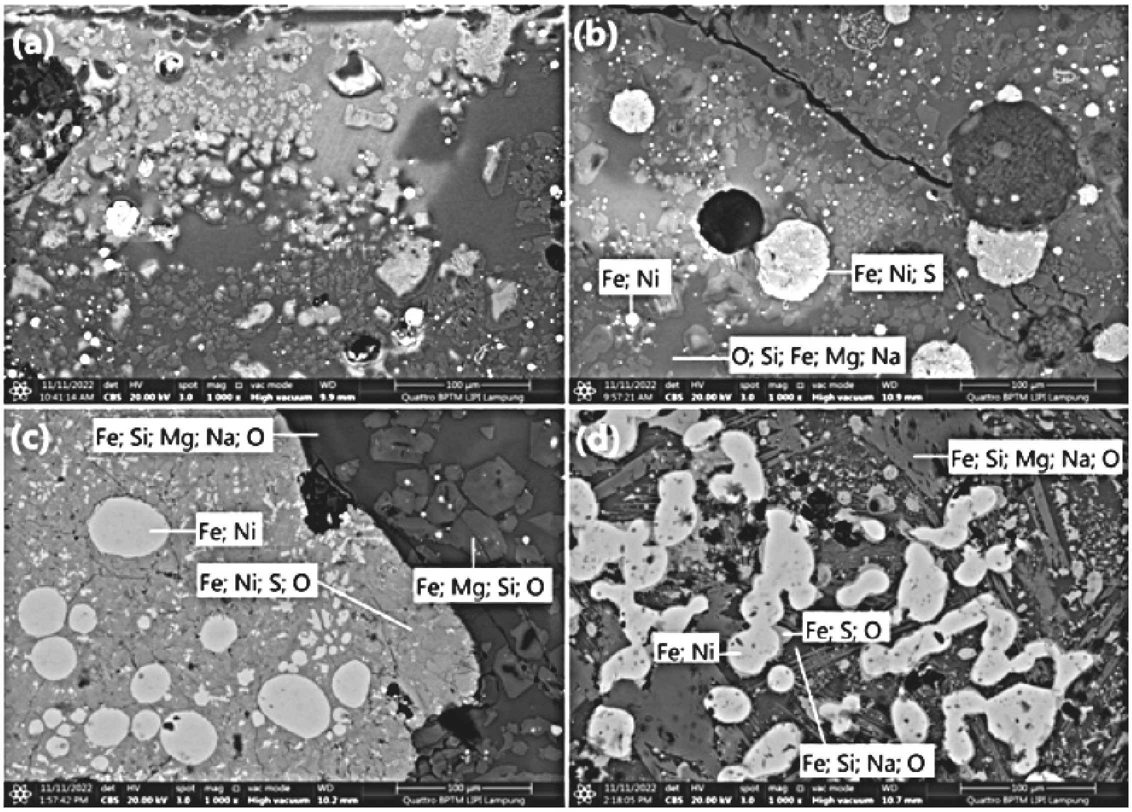

Figure 10. SEM-EDS analysis of reduced mixed ore with the composition of saprolite-iron ore: (a) 100-0; (b) 80-20; (c) 50-50; (d) 10-90.

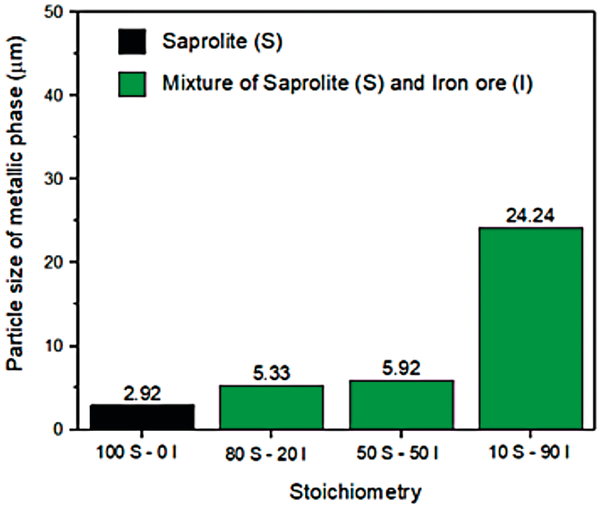

Figure 11. Ferronickel particle average size and percentage.

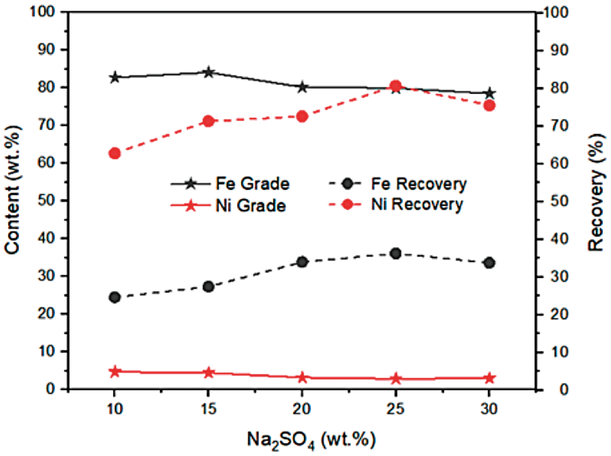

Figure 12. Effects of modified amount additive on Fe-Ni recovery and content.

which could form by the excess amount of sulfur, where the chemical reaction is similar to Equation (6). Nickel sulfide will begin to form above 1075 °C to form a submicron-sized Fe-Ni-S structure (Harris et al., 2013).

From this experiment, the particle size of ferronickel in reduced ore was successfully increased with the addition of iron ore into saprolitic nickel ore, which is affected by the increasing nickel recovery. Nevertheless, the reduction of iron oxide into metallic iron from iron ore would suppress the nickel content in ferronickel. The addition of sodium sulfate to inhibit the metallization of iron could not solve the problem significantly in this reduction of saprolitic nickel ore, which is contrary to the reduction of limonitic nickel ore. Other additives should be used in the next experiment to obtain the optimum result.

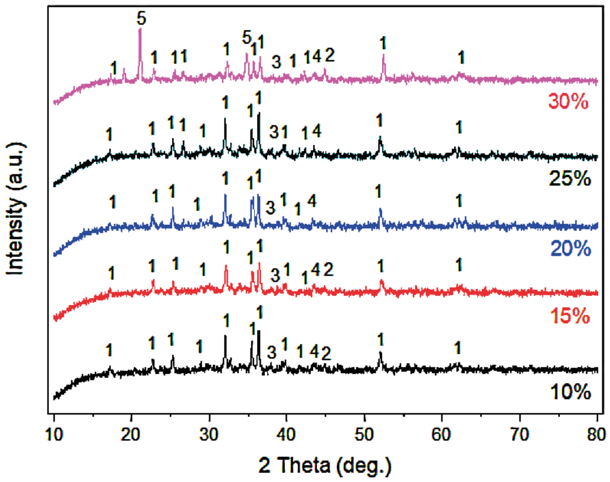

Figure 13. XRD pattern of reduced mixed ore modified sodium sulfate1-Forsterite; 2- erronickel; 3-diopside; 4-pyrrhotite; 5-quartz.

CONCLUSION

Iron oxide has higher reducibility than iron and nickel in magnesium silicate structure, shown by the higher recovery of metallic phase and larger metallic particle size in iron ore than saprolite. Both for iron ore and saprolite, the recovery of metallic element (iron or/and nickel) and metallic particle size increase with the increase of reductant dosage. The addition of iron ore in saprolite can increase the iron and nickel recovery, iron content, and ferronickel particle size due to the formation of pyrrhotite which could promote agglomeration ferronickel by the presence of liquidus phase. Nevertheless, the increase in iron recovery suppresses the nickel content. A 100% nickel recovery was obtained by adding 80% iron ore into saprolite. It indicates that the presence of the liquidus phase could release iron and nickel in the magnesium silicate structure. Adding up to 25% sodium sulfate in the saprolite and iron ore mixture can increase the recovery of iron-nickel and nickel content. Above 30%, the nickel content and recovery decrease due to the formation of nickel sulfide.

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