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## Effect of reduction temperature and sodium-based additives on nickel upgrading process of laterites ores

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**Abstract.** In this present work, the selective reduction of nickel laterite was conducted by using 10 wt. % of sodium carbonate, sodium chloride and sodium sulphate as additives. The 5 wt. % of palm shell charcoal was used as the reductant. The selective reduction process for nickel laterite was carried out from 950°C to 1150°C for 60 minutes followed by wet magnetic separation using 500 Gauss permanent magnet. The characterization of reduced ore was performed by X-Ray Diffraction, optical microscope and Scanning Electron Microscope with Energy Dispersive X-Ray Spectroscopy, while the chemical composition of concentrate or ferronickel was identified by X-Ray Fluorescence. The results showed that the nickel grade was increased with the increasing of reduction temperature for nickel ore with sodium sulfate additive. Nevertheless, it was relatively constant for sodium carbonate and sodium chloride additive. Nickel recoveries found increased with the increasing of reduction temperatures of nickel ore with sodium-based additives. The ferronickel particle was agglomerated and growth with the increasing of reduction temperature, thus it provided favourable conditions for the separation of ferronickel from impurities in the magnetic separation process.

### 1. Introduction

Nickel laterite is closely associated with iron oxide and silicate minerals as an isomorphous substitution for iron and magnesium in the lattice. Laterite ores are not able to process by physical beneficiation methods because of their complex mineralogy. The inherent difficulty of the beneficiation of laterite ores leads to their higher relative processing costs [1-2]. Pyrometallurgical or hydrometallurgical methods are usually applied in nickel laterite processing. Hydrometallurgical processes, including high-pressure acid leaching (HPAL), atmospheric acid leaching (AL) and Caron process [3]. The pyrometallurgical method has been applied commercially to extract nickel from the nickel laterite ore such as blast furnace (BF) smelting, rotary kiln-electric furnace (RKEF) process, Krupp-Renn (Oheyama) process [4].

The BF and RKEF process is considered to be energy intensive for producing ferronickel from laterite ores as it involves several high-temperature steps, especially when laterite ores with low nickel content are treated [5]. As for the Oheyama process, it is less energy consumable compared to the RKEF process but problem encountered by the Oheyama process is the ringing of the reactor wall caused by the hot-sticky agglomerates [4,6].



A new method for producing ferronickel from nickel laterite ores by reducing metal oxides at moderate temperature and followed by magnetic separation process was called selective reduction process. By controlling the reduction conditions, it should be possible to obtain reasonable enrichment and high nickel recoveries while minimizing the reduction of the iron oxides.

Several investigations on the reduction roasting of nickel laterite ore using coal-based reductant followed by magnetic separation to produce ferronickel concentrate had been performed. A variety of reducing conditions and chemical additives in selective reduction process had been used to increase the nickel grade and recovery in the ferronickel alloy. According to Li et al., a concentrate of ferronickel containing 9.48% Ni with a nickel recovery of 83.01% was obtained from the nickel laterite ore containing 1.91% Ni. The reduction was carried out at 1100°C for 60 minutes with the addition 20 wt. % of sodium sulfate [7]. Jiang et al. (2013) reported that the addition of 10 wt. % of sodium sulfate and 2 wt.% of coal in nickel laterite ore (1.49% Ni and 34.69% Fe) processing by reduction process at 1200°C for 50 minutes which reported a nickel grade of 10.86% and recovery of 88.56% [8].

However, there is still less information in the selective reduction of nickel laterite processing using other sodium-based additives. In this present work, the extracting ferronickel concentrate from nickel laterite ore was carried out with added some sodium-based additives such as sodium carbonate, sodium chloride and sodium sulfate in the reduction process continued with the magnetic separation processes. The effects of various additives and reduction temperature on this selective reduction process were investigated clearly.

## 2. Materials and Methods

### 2.1. Materials

The nickel laterite ore was from South East Sulawesi, Indonesia. The X-ray fluorescence (XRF) was performed to determine the chemical compositions of the nickel ore, as showed in table 1.

**Table 1.** Chemical analysis of laterite ores.

Element	Fe	Ni	Al	Si	Ca	Mg	Cr	Mn	Co
wt. %	50.5	1.4	4.86	6.5	0.18	1.81	2.68	0.85	0.07

A 10 wt. % of palm shell charcoal was used as the reductant. The composition of the reductant is listed in table 2. The sodium-based additives which were used in this reduction process were sodium carbonate, sodium chloride, and sodium sulfate.

**Table 2.** Chemical analysis of palm kernell shell charcoal.

Chemical analysis of ash content (wt. %)						Proximate analysis (wt. %)				S	Calories (cal/g)
SiO <sub>2</sub>	CaO	MgO	Al <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	Fe <sub>2</sub> O <sub>3</sub>	Moisture	Volatile content	Ash content	Fixed carbon content		
54.09	20.71	3.37	3.12	1.6	6.72	0.43	22.57	21	77	0.01	6.244

### 2.2. Reduction and Magnetic Separation Process

The nickel laterite ore and palm shell charcoal were crushed and ground to less than 149 µm. The fine nickel laterite and 5 wt. % palm shell charcoal was thoroughly mixed with different additives, i.e. sodium carbonate, sodium chloride and sodium sulfate with 5 to 15 wt. %. The mixture was pelletized into 10-15 mm diameter and dried in an oven at 100°C for 4 hours. The 60 gram of pellets were placed in a graphite crucible and the reduction process was carried out in the laboratory using muffle furnace. It was heated using various reduction temperatures, i.e. 950°C, 1050°C and 1150°C for 60 minutes. After the reduction process, the reduced pellets were quenched into water and dried at 100°C for 4 hours

followed by grinding into less than 74  $\mu\text{m}$ . Then, the wet magnetic separation process was performed to the fine-reduced ore using 500 Gauss permanent magnet. The magnetic particle of reduced ore or concentrate was rich in ferronickel, while non-magnetic or tailing was rich in impurities/slag.

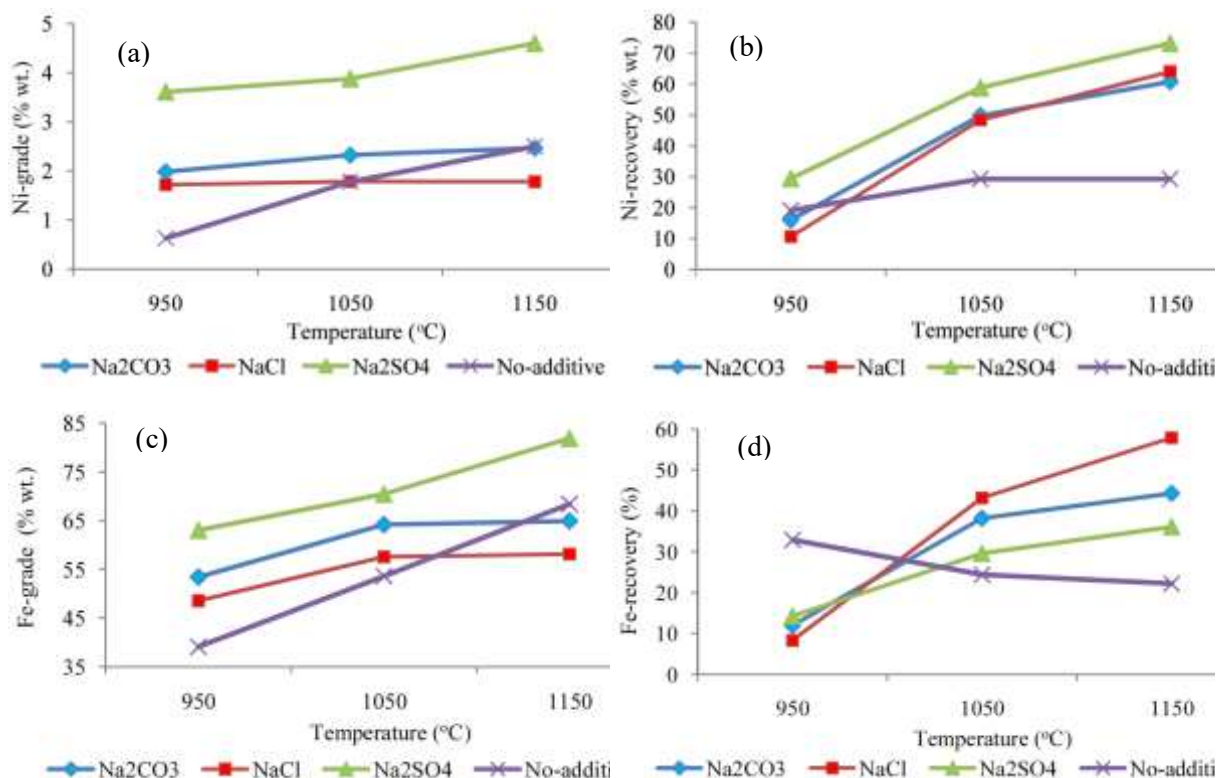
### 2.3. Characterization

The characterization of concentrate was identified by X-ray Fluorescence (XRF). Polished sections were prepared to observe the morphological changes and analyzed the element content of the reduced ore using Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS). X-ray Diffraction (XRD) analysis was used to determine the different mineralogical phases present in the reduced ore.

## 3. Results and Discussion

### 3.1. Effect of reduction temperature

The effect of reduction temperatures of nickel laterite ore on iron-nickel grade and recovery using various additives is given on figure 1(a-d). The 10 wt. % of additives and 5 wt. % of palm shell charcoal reductants were used in this experiment. The increasing of reduction temperature increased the nickel grade in nickel ore concentrate, as shown in figure 1(a). The optimum temperature reduction was 1150°C produced 4.6 wt. % of nickel grade using sodium sulfate additive. The additives addition in selective reduction process promoted the recovery of nickel, as shown in figure 1b. Nickel recovery also increased along with the increased temperature from 950°C to 1150°C. The optimum nickel recovery, i.e. 73.2%, was resulted from the reduction of nickel ore at temperature 1150°C using sodium sulfate additive.



**Figure 1.** Effect of reduction temperature on iron-nickel grade and recovery using 10 wt. % additives.

The increase of iron grade was found in with and without additives during the increasing of reduction temperature, as shown in figure 1(c). The optimum iron grade in this selective reduction process, i.e.

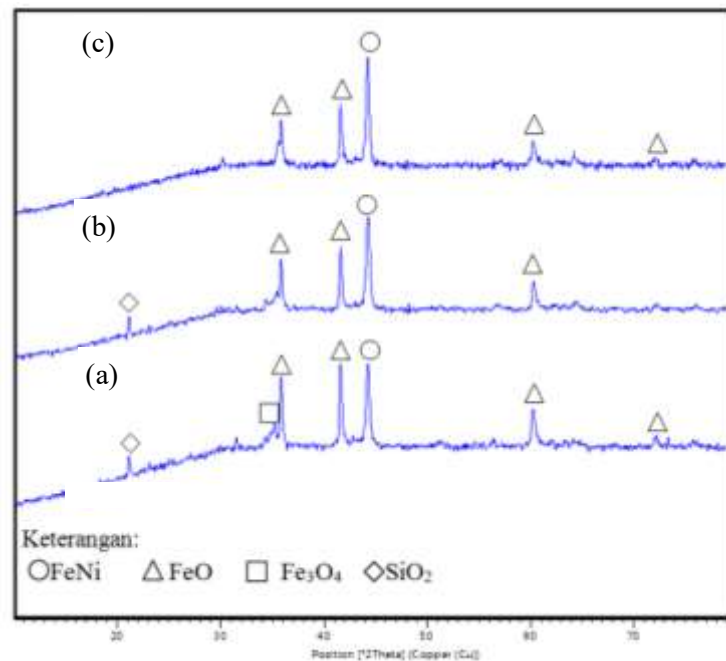
81.9 wt. %, was resulted from the reduction process at temperature 1150°C using sodium sulfate additive. In the addition of sodium chloride and sodium carbonate, the iron grade in concentrate was relatively constant after the reduction process at temperature 1050°C. Nevertheless, at temperature reduction 1150°C, the iron grade was lower than no-additive addition.

The iron recovery was decreased for nickel ore reduction without additives by the increasing of reduction temperature, as shown in figure 1(d). By the addition of the additives, the iron recovery was increased with the increasing reduction temperature. The addition of sodium chloride additive in nickel ore promoted a significant increase of iron recovery due to the pyro-hydrolysis reaction of sodium chloride which was rising along with the increase of temperature [9]. The increased temperature up to 1150°C should increase the nickel-iron grade and recovery with additives. The increase of temperature should improve the degree of metallization on nickel and iron [10].

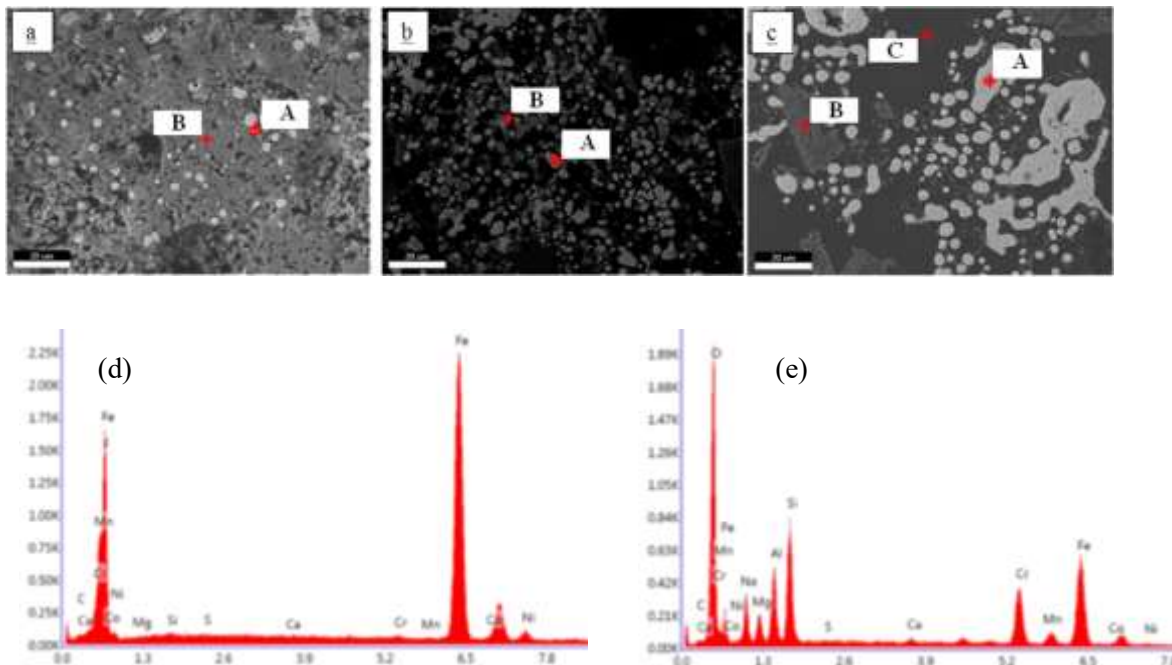
From figures 1(b) and (d), the iron and nickel recovery of reduced ore containing sodium carbonate was increased with the increasing of reduction temperature. From XRD analysis, as shown in figure 2, the peak of ferronickel diffraction increased along with the increasing of reduction temperature up to 1150°C. Nevertheless, from figure 1(a), the optimum nickel grade of reduced ore with sodium carbonate additive was only 2.5%. The low nickel grade in this reduced ore due to the high of iron recovery resulted from the reduction process. The decomposition of sodium carbonate produced carbon dioxide (CO<sub>2</sub>) gas, as expressed in equation (1) [11], promotes the formation of wustite (FeO) due to the Boudouard reaction by producing carbon monoxide (CO) gas to enhance the reduction process. Furthermore, the wustite will reduce into iron. As shown in figure 2, The wustite was decreased with the increase of temperature reduction due to the more carbon monoxide resulted from decomposition of sodium carbonate. The peak of quartz (SiO<sub>2</sub>) was decreased along with the increase of temperature and it was disappeared at the temperature of 1150°C. The quartz was decreased with the increasing temperature reduction also indicated that the decomposition rate of sodium carbonate enhanced by the increasing of reduction temperature.



From SEM and EDAX analysis, as shown in figure 3, the sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) which resulted from the decomposition of sodium carbonate was found as dark area (point C) with the appearance element of Na, Si, and O, while the ferronickel was found as light-white area which was indicated by the element of Fe and Ni. Sodium silicate has a low melting temperature, i.e. 1089°C. Thus, it gives a beneficial condition for migrations and aggregations of metallic iron and nickel in liquidus state. As shown in figure 3, a less amount of ferronickel particle was found in reduction process at temperature 950°C and it was found the more and larger ferronickel particle at temperature 1150°C.



**Figure 2.** XRD pattern of the reduced nickel laterite with 10 wt. % additive sodium carbonate at various temperature (a) 950°C, (b) 1050°C (c) 1150°C.



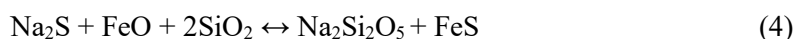
**Figure 3.** SEM analysis of reduced laterite in presence of 10 wt. % sodium carbonate with reduction temperature: (a) 950°C, (b) 1050°C and (c) 1150°C; EDAX analysis of point: (d) B, (e) A

Figure 4(a-c) presented the XRD analysis of reduced nickel laterite at various reduction temperatures with sodium chloride additive. Along with the increase of reduction temperature, the peak of fayalite ((Fe, Mg)<sub>2</sub>SiO<sub>4</sub>) decreased due to the reaction of fayalite with carbon monoxide forming magnetite.

There was no-wustite, such as in sodium carbonate, due to the limited of CO gas sources. As shown in figure 4, magnetite was formed at 1050°C followed by reducing the peak of fayalite. At 1150°C, the peak of magnetite was reduced due to the transformation of metallic iron from Fe<sub>3</sub>O<sub>4</sub> by the reduction process, thus the peak of ferronickel increased. Hydrochloride gas was formed by the pyro-hydrolysis of sodium chloride. Nevertheless, the hydrochloride gas tended to be difficult to directly chlorinate iron oxides during the process of chlorination in this reduction process, thus iron chloride was not found in reduced nickel ore, as shown in XRD pattern (figure 4). It was also agreed with Zhou et al., that the chlorination of iron oxide was not occurred even at 1150°C of reduction temperature [9]. In this reduction process, the metallic iron was resulted from the reduction mechanism of fayalite which transform into magnetite, then reduced into metallic iron.

The increasing of reduction temperature of nickel ore with sodium chloride additive promoted the growth of ferronickel particle in reduced nickel ore, as shown in figure 5. From SEM and EDAX observation, it was found that fayalite as the main gangue in the reduced ore was found as a grey area (point B) which indicated by the element Fe, Mg, and Si, as shown in figure 5(d), while ferronickel shown as white area (point A) which indicated by element Fe and Ni. Sodium chloride addition promoted the aggregation of fayalite during the process of reduction, shown in figure 5. Therefore, it had an effect on the increase of iron grade and recovery in the concentrates through the reduction of fayalite into metallic iron.

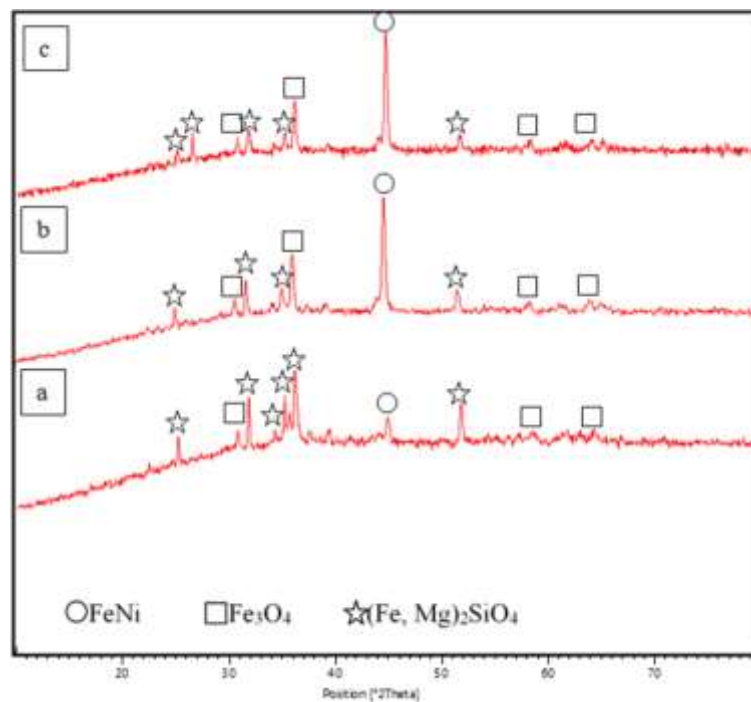
The result of XRD testing at various temperatures with additive sodium sulfate is shown in figure 6(a-c). The peak of wustite (FeO) increased along with the increase of temperatures due to the reduction of hematite to wustite. However, most of the wustite was not transform into metallic iron during the increasing of temperature in this reduction process. As expressed in equation (3), the sodium sulfate was reduced into Na<sub>2</sub>S. It reacts with wustite to form non-magnetic troilite (FeS), as expressed in equation (4), thus it inhibits the reduction of wustite into metallic iron. Therefore, it suppressed the iron recovery and resulted in low-grade iron in concentrate. The peak of ferronickel on XRD patterns decreased at the increasing temperature up to 1150°C. Nevertheless, the nickel grade was increased as given on figure 1(a-b). This indicates that there is more nickel than iron content in the concentrates.



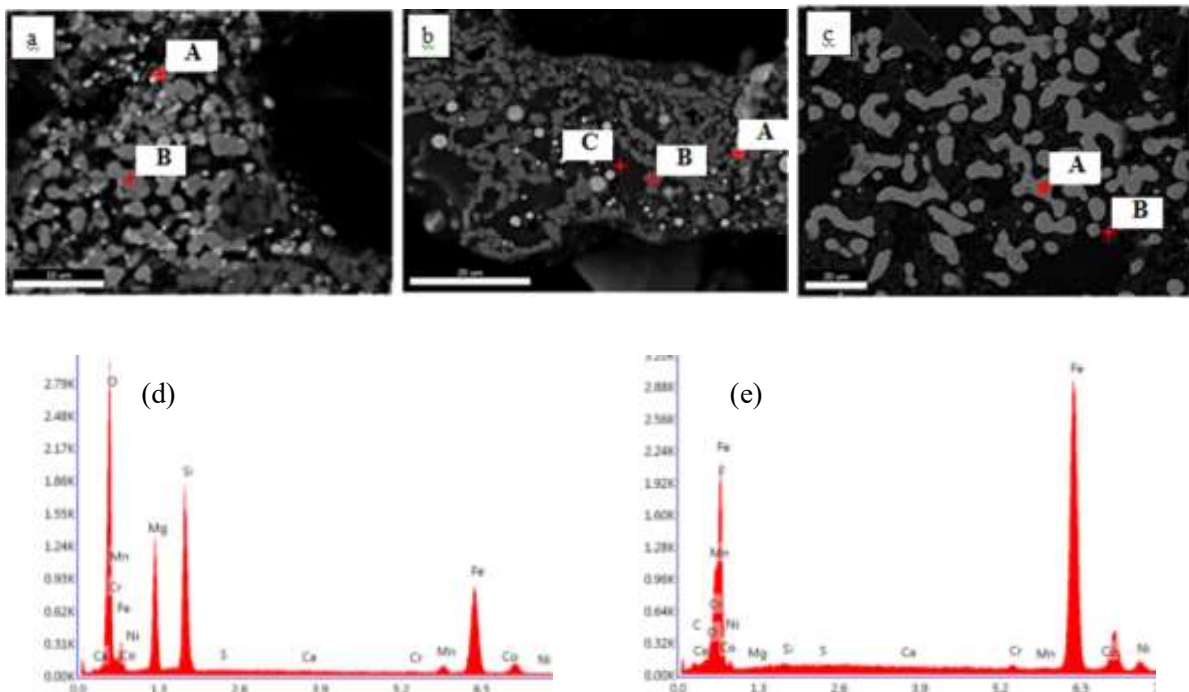
The growth of ferronickel size in reduced nickel laterite at temperatures 950°C to 1150°C using sodium sulfate additive was observed and presented on figure 7 (a-c). Fine ferronickel particles were found at reduction temperature 950°C and distributed uniformly. The ferronickel particles (point A) were agglomerated and grown larger during the increasing temperature. The diameter of particle size was 30.6 µm surrounded by troilite (point B) at reduction temperature 1150°C, as shown in figure 7. From XRD analysis, the troilite was also found in the reduction of nickel laterite with sodium sulfate additive, as shown in figure 6. At temperature 1050°C, the metallization occurred in liquid condition and it promoted the reduction rate of nickel and iron oxide into metallic phase due to the low eutectic temperature of troilite, which was 985°C [12].

Therefore, the nickel and iron grade of reduced ore with sodium sulfate additive was higher than the one with no-additive, even with sodium carbonate and sodium chloride additives. The decomposition rate of (Fe,Mg)<sub>2</sub>SiO<sub>4</sub> into wustite and quartz was also increased in liquidus state. Thus, wustite was increased along the increasing of reduction temperature, while quartz was found at reduction temperature of 1150°C.

The investigation of ferronickel size in nickel ore concentrate toward reduction temperature was reported in this study. The growth of ferronickel size was increased by the increasing of reduction temperature from 950°C to 1150°C, as shown in figure 8. The particles size was 30.6, 12.8, and 0.8 µm at temperature 1150°C for reduction nickel ore with the addition 10 wt. % of sodium sulfate, sodium carbonate, and sodium chloride additives, respectively. The addition of sodium sulfate additives produced ferronickel size three times larger than the one with sodium carbonate and sodium chloride.

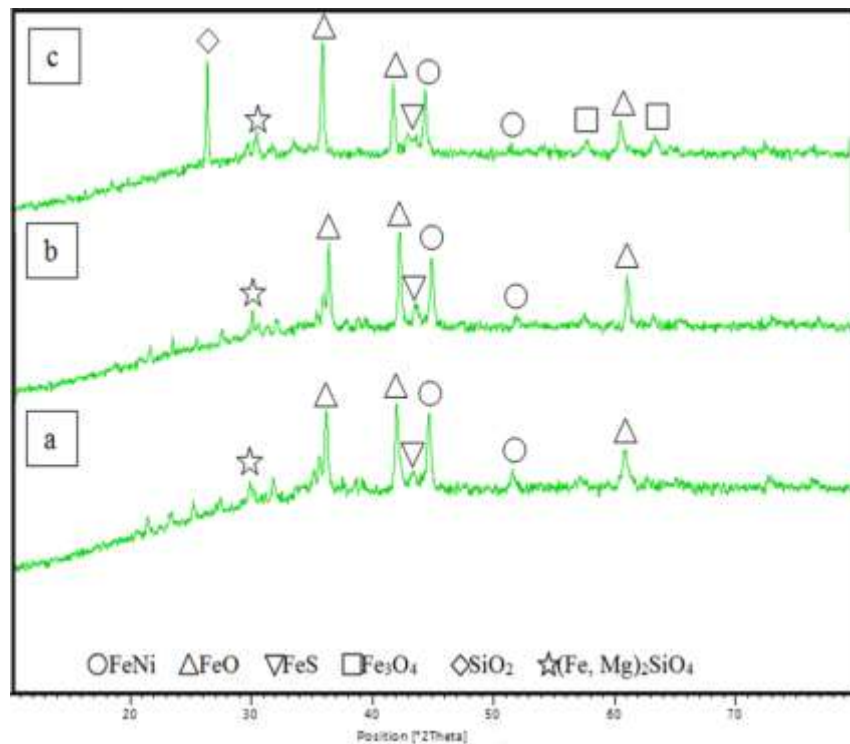


**Figure 4.** XRD pattern of the reduced laterite with 10% wt. additive sodium chloride and various temperature: (a) 950°C, (b) 1050°C (c) 1150°C.

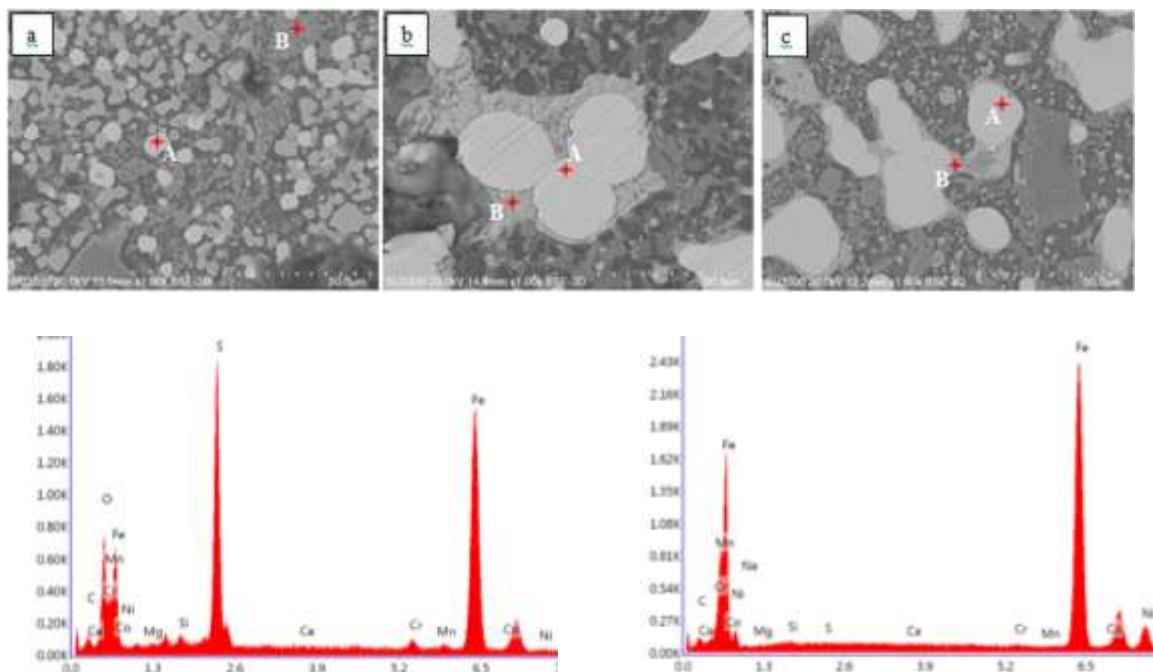


**Figure 5.** SEM analysis of reduced laterite in the presence of 10 wt. % sodium chloride with reduction temperature at: (a) 950°C, (b) 1050°C and (c) 1150°C; and the EDAX analysis of point: (d) B, (e) A.

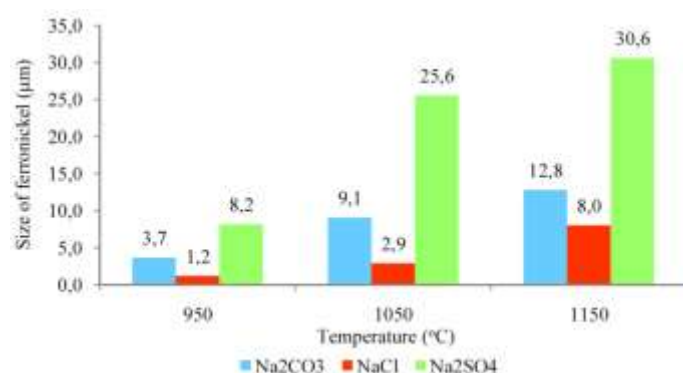




**Figure 6.** XRD pattern of the reduced laterite with the additive sodium sulphate and various temperature (a)950°C, (b)1050°C, (c)1150°C.



**Figure 7.** SEM analysis of reduced laterite in the presence 10 wt. % of sodium sulphate with reduction temperature at: (a) 950°C, (b) 1050°C and (c) 1150°C; and EDAX analysis of point: (d) B, (e) A.



**Figure 8.** Ferronickel particles size of reduced nickel ore at various temperatures for 60 min with 10 wt. % additives.

#### 4. Conclusion

The addition of sodium sulfate additive in the reduction of nickel ore containing 1.4%Ni-50.5% Fe resulted in higher nickel grade and recovery than sodium carbonate and sodium chloride additives, due to the troilite (FeS) formation for inhibiting the metallization of iron oxide, thus lowering the iron recovery resulted in high nickel grade in concentrate. The nickel grade was increased with the increasing reduction temperature for nickel ore with sodium sulfate additive. Nevertheless, it was relatively constant for sodium carbonate and chloride additives, due to the high iron recovery was resulted. The growth of ferronickel particles in reduced ore was increased with the increasing of reduction temperature, thus it gave the beneficial condition for nickel and iron separation through the magnetic separation process.

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