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Production of pig iron nugget from low-grade iron ore and pyrolyzed oil-palm-empty-fruit-bunch composites

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Abstract. Utilization of low-grade iron ore and pyrolyzed oil palm empty fruit bunch (EFB) is an effective way as the solution to deal with the depletion of iron reserves and administer of empty fruit bunch waste. This study was aimed to analyze the pig iron that is produced by carbothermic reduction of low-grade iron ore with pyrolyzed palm oil EFB as a reductant. The ore from Lampung was reduced at temperature 1400°C and 1450°C for holding time 40 minutes with inert condition (N₂ atmosphere). The structures of pig iron were studied by using X-Ray diffraction (XRD), and the microstructures and chemical composition were analyzed by using Scanning Electron Microscopy-Energy Dispersive X-Ray Spectroscopy (SEM-EDS). It was found that iron nugget that was reduced at 1450°C for 40 minutes that was wholly separated from its slag, with reduction degree 98.5%, metallization degree 99.2%. The microstructure has metallic iron as a base with some pores. On the other hand, the other iron nuggets that are not separated from its slag consist of two prominent phases, metallic iron and lamellar fayalite (Fe₂SiO₄).

1. Introduction

Iron ore is the main ingredient for the steelmaking process, besides scrap metal. Based on the iron content, iron ore is classified into high-grade and low-grade iron ore. The iron mineral that is mostly mined is high-grade iron ore to produce high-grade iron and steel due to its excellent process efficiency. However, its resources are limited in availability [1]. Therefore, one way to deal with the shrinkage of iron reserves is by developing a process to utilize low-grade iron ore. A study by Cahyono et al. investigated the reduction of low-grade iron ore which has the high content of combined water requires less temperature compared to the reduction of high-grade iron ore [2].

In recent years, several researchers have observed the use of various types of biomass as a reduction agent in the process of reducing low-grade iron ore. The wastes from palm oil are already well-known as biomass. Apart from palm oil, common biomasses as iron ore reductant are also produced from wood, sugar pine sawdust, and rice husk [3]. A study by Yunus et al. studied the reduction of iron using palm oil empty fruit bunch (EFB) can increase the iron content up to 62.7 wt%. Thus, it will be beneficial for low-grade iron [4]. Also, in the other work, it was reported that reduction process using some types of biomasses as a reductant at a temperature around 1400°C would transform iron ore into metallic iron and produce pig iron that is distinctly separated with slag [5].

In this paper, the carbothermic reduction process of low-grade iron ore with pyrolyzed oil palm empty fruit bunches as a reductant was investigated. Reduction degree, metallization degree at



temperature 1400°C and 1450°C for holding time 40 minutes were calculated based on their chemical composition after the carbothermic reaction.

2. Experimental method

2.1 Materials

The iron ore was obtained from Lampung, Indonesia. The characteristic of the ore was hard, it was found in the form of a rock, and the color was reddish. Iron ore was crushed, grinded, and sieved (140 mesh) to reduce the size. Table 1 shows the elemental composition of iron. The predominant element in the ore is Fe, accounting to 48.4 wt% of the ore. It is followed by other elements such as Si, Al, Mg, Ca, P, S, K, Cr and Ti that are considered as impurities. Figure 1 shows that the ore mainly consists of hematite (Fe_2O_3) and quartz (SiO_2). The result is by the chemical composition test. It has a relatively enormous amount of silica. The presence of hematite is already predicted because the ore has the reddish color and as hard as a rock. Using Rietveld Refinement, the percentage of hematite is 61.9%, and quartz is 38.1%.

Table 1. Chemical composition of low-grade iron ore from Lampung.

Element	Fe	Si	Al	Mg	Ca	P	S	K	Cr	Ti
Weight (%)	48.4	20	2.94	0.326	0.223	0.107	0.0989	0.0234	110 ppm	ND

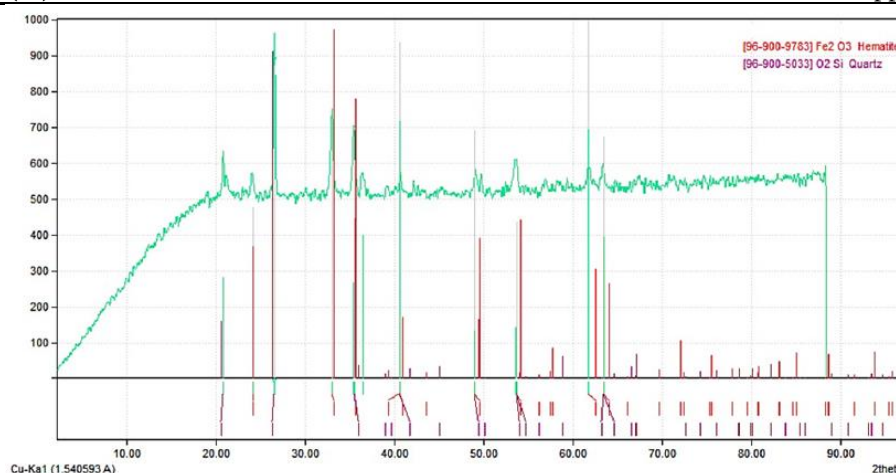


Figure 1. XRD pattern of low-grade iron ore from Lampung.

Table 2. Elemental composition of pyrolyzed EFB.

Analysis Parameters	Quantity (%)
Proximate:	
Moisture in air-dried sample	5.76
Ash	20.35
Volatile Matter	21.64
Fixed Carbon	52.25
Ultimate:	
Carbon	54.56
Hydrogen	3.5
Nitrogen	1.63
Total Sulfur	0.18
Oxygen	21.77

The reductant was pyrolyzed palm oil empty fruit bunch. The empty fruit bunch was pyrolyzed at 400°C for one hour in a furnace after previously dried for 24 hours. The char collected then underwent

a crushing and sieving process (140 mesh). Table 2 represents the ultimate and proximate analyses for pyrolyzed EFB. Pyrolyzed EFB composed of the high amount of carbon by ultimate analysis (54.56 wt%) and 52.25 wt% of the fixed carbon from the Proximate analysis. The ultimate analysis also indicated the presence of 21.77 wt% oxygen as well as the insignificant amount of hydrogen, nitrogen, and sulfur in EFB biomass. The proximate analysis of pyrolyzed EFB also revealed the significant presence of volatile matter: 21.64 wt%, ash: 20.35 wt% and moisture: 5.76 wt%.

2.2 Reduction process

The ratio of the mixture was 82.75 wt% Fe of iron ore and 17.25 wt% C of char that were mixed with water as the binder. The mixture then shaped into round pellets (diameter 2 cm) and dried. The samples were reduced at temperature 1400°C and 1450°C for holding time 40 minutes in an electric tube furnace. In this study, there were two types of samples namely, sample A (for 1400°C and 40 minutes) and sample B (for 1450°C and 40 minutes).

2.3 Characterisation

The samples were characterized by using XRD (Rigaku MiniFlex 600), and the microstructures and chemical composition were analyzed by using SEM-EDS (INSPECT F50). Reduction degree and metallization degree were calculated based on their chemical composition after the carbothermic reaction.

3. Result and discussion

3.1 Phase formation of reduction products

X-ray powder diffraction patterns of iron nuggets reduced at various temperatures and holding time are shown in Figure 2. At temperature 1400°C, the sample produced Fe and Fe₂SiO₄ (fayalite) phases with different levels of crystallinity. Based on the XRD pattern of sample A, fayalite phase has higher crystallinity compared than Fe phase. In previous studies, it was reported the rate of growth of the fayalite with increasing temperature and decreasing oxygen fugacity where FeO (wustite) is formed at around 900°C and starts to react with quartz to form some low melting point minerals at around 900°C-1000°C, such as fayalite [6][7][8][9]. This result has implications for the high Si content in iron ore causing the formation of the fayalite phase in the pig iron produced. This is supported by quantitative XRD using X'Pert High Score which is already equipped with Rietveld data in Table 3, where the amount of fayalite is 74%. Based on the XRD data above, it can be concluded that a temperature of 1400°C with a holding time of 40 minutes has not provided a reductive environment sufficient for the perfect carbothermic reduction process due to fayalite being the dominant phase.

Generally, there is a significant difference between this sample and the previous one. It was clear that at a temperature of 1450°C with a holding time 40 minutes it managed to provide a reductive environment so that the Fe phase became dominant at 74%. The fayalite phase that was previously found as the most dominant phase in this condition was no longer detected. The other phases found in this condition were iron oxide phase with 26%, based on quantitative data XRD in Table 3. Also, Fe crystallinity increase with increasing temperature. It is seen that the peak of phase Fe increases at an angle of 45. In previous studies, it was reported that with the increase in temperature and holding time also decreases the presence of fayalite [5][9].

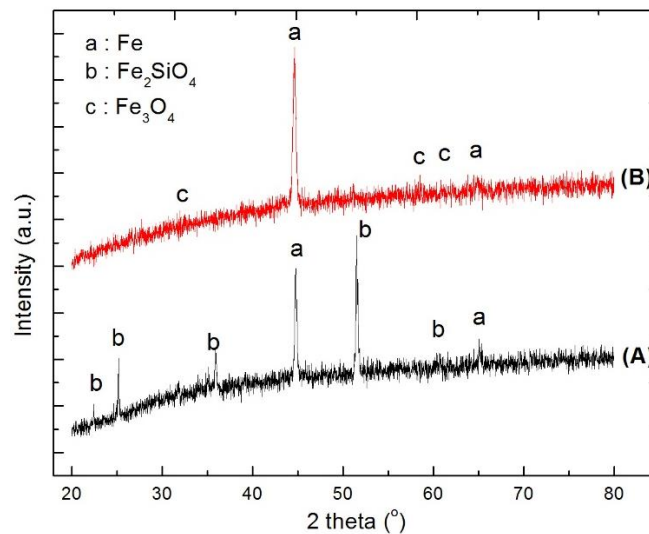


Figure 2. XRD patterns of the iron nugget.
(A) Sample A (for 1400°C) and (B) Sample B (for 1450°C).

Table 3. Quantitative data analysis of the samples based on Rietveld calculation.

Sample	Phase amount (%)		
	Iron (Fe)	Fayalite (Fe ₂ SiO ₄)	Iron Oxide (Fe ₃ O ₄)
A	26	74	-
B	74	-	26

3.2 Macro and micro structure

It can be seen that sample A did not melt as shown in Figure 3 but the pellets stick to each other on the side. When the magnet touched it, it is known that the iron nugget is magnetic. The iron nuggets cut into four parts to see the cross-section inside the pellet. As shown in Figure 4, the inside of those the pellets is shiny and metallic, with some dark spots scattered on them, high porosity in the middle, and some small pores dispersed on the cross-section. The significant porosity contains slags, which is glassy and has black color. Fayalite hindered reduction of FeO and formation of fayalite was inhibited with increasing CO concentration [9]. Some slags were lost during the cutting process. The yellowish part may be corrosion due to the usage of water during the cutting process.

This reduction process at 1450°C for 40 minutes is the only one that produced separated pig iron and slag. The pig iron was hidden under the slag. The slag has black color with some reddish mark, a phenomenon that has been found on the surface of almost all iron nuggets that was reduced with pyrolyzed palm oil empty fruit bunch during this study.

When the slag was taken, surprisingly there is an iron nugget under the slag. The iron nugget is metallic and shiny. Unlike the previous iron nuggets, this iron nugget is wholly separated from its slag. It also has a relatively smooth surface compared to the last iron nuggets that have the rough surface. Based on eye observation, the iron nugget was found to have no porosity and no slag. It was completely separated from the slag. The big piece of slag is black. When the slag was flipped, the bottom part has the gray color. Both of the slag and iron nugget was tested with the magnet in contrast to the slag that did not react. Iron nugget showed a response. The iron nugget then cut into two pieces. The cross-section is found to be mirror-like and reflective.

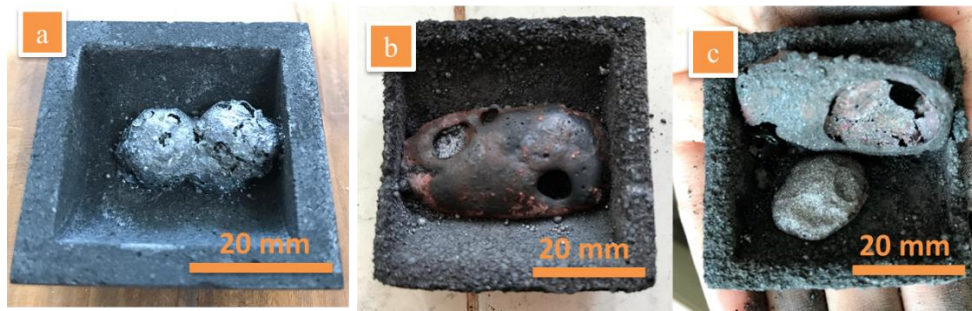


Figure 3. Iron Nugget after Reduction.

(a) Sample A (for 1400°C), (b) Sample B (for 1450°C), and (c) Sample B after it was flipped.

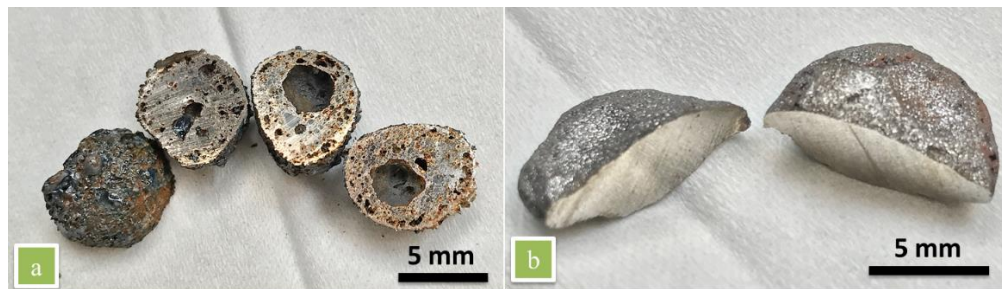


Figure 4. Cross-Section of Iron Nugget after Cutting.

(a) Sample A (for 1400°C) and (b) Sample B (for 1450°C).

Selected samples were examined by SEM-EDS to know the morphological changes during the reduction process, and the results are given in Figure 5 and Table 4. Using the backscattered electrons detector to illustrate the difference in molecular weight of the atoms that make up the surface high molecular weight samples and atoms will be brighter in color than low molecular weight atoms. It is clear that Figure 5 (a) shows a significantly different brightness level and there are several pores. In this figure, the metallic iron phase has a rather bright color while the gray one is a non-metal phase or slag which is a lamellar fayalite phase. Also, there are several pores between the Fe phase and slag. The EDS results show the constituent oxides in samples A is Si, Ca, F, K, and C. The most typical types of slag found in iron nuggets are FeO, SiO₂, CaO, MgO, and MnO. Those oxides may easily diffuse into eutectic phase such as fayalite. The lamellar area is found to be mainly composed of: ferrous, silicon, and oxygen. The big difference between ferrous quantity between the darker and lighter regions inside the lamellar can be seen in the phase diagram of FeO and SiO₂ [10]. Above the temperature of 1300°C, fayalite will be the formation of liquids (slag) because it has a low melting point [7]. In the reduction of iron oxide, any unreacted iron usually ends up in slag. The presence of iron in slag indicates an incomplete reduction reaction and is considered iron loss [5].

Different results can be seen in Figure 5 (b), it seems clear that the slag size is much smaller than other samples and there are no pores. In this condition, the metallic iron phase becomes the dominant phase as evidenced by the EDS results result which is 91.53%. It can be concluded that the size of the slag is strongly influenced by the increase in temperature and holding time. Also, the biomass of oil palm empty fruit bunch as a reductant successfully reduces low-grade iron ore.

3.3 Metallization Degree

The reduction process causes notable changes in the degree of reduction and degree of metallization shown in Table 5. When sample A was burned at 1400°C for 40 min, the degree of metallization was 97.70%. However, it becomes 99.20% when sample B reduced at 1450°C. The results indicate that the increasing temperature and holding time increases the degree of metallization, as well as the degree of reduction. These results are consistent with previous studies reported by Srivastava et al. [5], Cahyono et al. [2] and Yunus et al. [4]. This increase shows that oxygen in the oxide phase decreases continuously and phase transformation occurs during the reduction process and causes the appearance

of the dominant Fe phase. Besides in Table 3, it is clear that the effect of temperature rise is directly proportional to the degree of reduction in the reduction results.

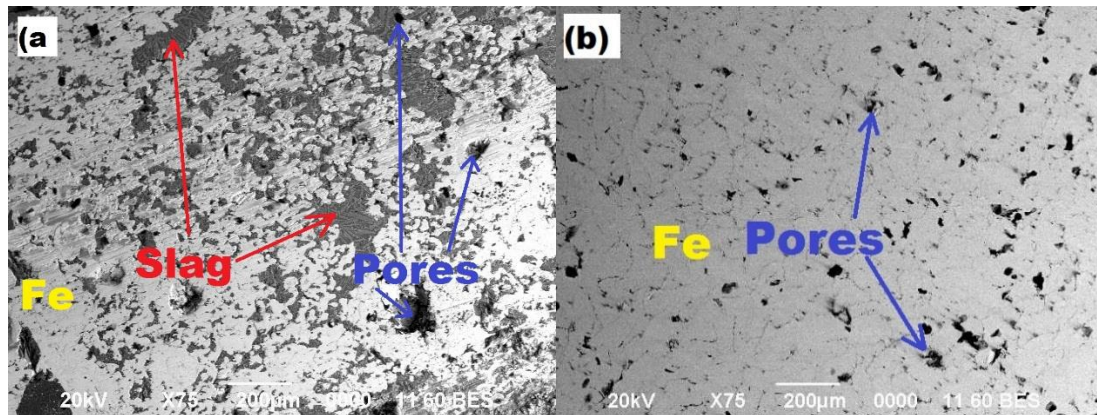


Figure 5. Back Scattered Microstructures Image of The Carbothermic Products.

(a) Sample A (for 1400°C) and (b) Sample B (for 1450°C).

Table 4. EDS data of pig iron nugget product.

Sample	Weight (%)						
	C	O	F	Si	K	Ca	Fe
A	7.64	6.68	1.55	5.01	1.05	0.62	77.45
B	5.75	0.45	2.27	-	-	-	91.53

Table 5. Reduction Degree and Metallization Degree.

Samples	Reduction Degree (%)	Metallization Degree (%)
A	74.34	97.70
B	98.50	99.20

Where the lowest degree reduction was obtained in sample A which was reduced at 1400°C for 40 minutes, and the highest degree of reduction in sample B was reduced at 1450°C for 40 minutes. At high temperatures, pyrolysis product resulted in a more massive amount of gas products; hence, the degree of reduction of iron ore increased at the higher temperature. The amount of CO is sufficient to reduce hematite to become magnetite and wustite [2].

Table 6 summarizes several studies on pig iron nugget production using biomass as a reducing agent. Metallization degree in this research is highest compared to Srivastava et al. [5] and Fu et al. [11] research, which is up to ~99.20%. Although the carbon content obtained from biomass in Fu et al. [11] research was quite high compared to this study and Srivastava et al. [5]. Also, there is an initial treatment given to biomass in this study and Fu et al. [11]. Based on the content of Fe (wt%), the present study was the lowest, i.e., 48.8 compared to Srivastava et al. (63.27) [5] and Fu et al. (65.89) [11] and the use of binders using only water in this investigation. Metallization degree in Srivastava et al. research was obtained up to ~98.8% with drying at 150°C for 1 day before reduction process [5]. However, in this study, no initial treatment was carried out before the reduction process and obtained metallization degree up to ~99.20% even though the reduction temperature was the same as Srivastava et al. research.

Table 6. Comparison of Reduction of Iron Ore with Biomass between previous and present study.

References	Srivastava et al [5]	Fu et al [11]	Present study
Biomass type	Fix. Carbon, (wt%) Fine wood ; (47.60)	Rice crust char, Bamboo char, Coconut crust char ; (44.67-92.79)	Empty fruit Bunch ; (52.25)
Preparation	-	Crushing, Grinding, Drying at 105°C	Drying 24 h & Pyrolysis 400°C 1 h
Iron Ore Type (Total Fe, wt%)	Magnetite concentrate (63.17)	65.89	Hematite (48.8)
Binder & Flux	Limestone	Bentonite, CaO	Water
Temp. (°C)	1425-1472	1250, 1300, & 1350	1400 & 1450
Reduction Time (min)	7-50	15-25	30 & 40
Condition Atmosphere	Air	Argon	Nitrogen
Preparation	Drying 150°C for 24 h	-	-
Metallization Degree (%)	up to ~98.8%	~95.56%	up to ~99.20%

4. Conclusion

- The iron nugget that was reduced at 1400°C is dominated by phase fayalite than metallic iron. On the other hand, the iron nugget that was reduced at 1450°C has 74% Fe, and the rest is ferrous oxide.
- The iron nugget that was reduced at 1450°C for 40 minutes was separated from its slag. Meanwhile, the other iron nuggets are only patched with each other on the side, without completely melting.
- When the iron nuggets that are not separated were cut in the cross-section, it is found that there is a big hole in the middle which is filled with slag. It indicates that the slag is formed in the center of the pellet. When the microstructure is observed, it is found that there are two prominent phases: metallic iron and lamellar fayalite.
- The iron nugget that was produced from reduction at 1450°C is found to be metallic and shiny. The mass percentage of ferrous is as high as 91.53 wt%. The microstructure mainly consists of metallic iron with some pores.
- The degree of reduction and the degree of metallization increases with the rising temperature at the time of reduction at the temperature range of 1400°C-1450°C.

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