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Volume 342

2018

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International Conference on Innovative Technology, Engineering and Sciences 2018 (iCITES 2018)
1–2 March 2018, Universiti Malaysia Pahang (UMP) Pekan Campus Library, Malaysia

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Accepted papers received: 19 March 2018
Published online: 6 April 2018

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To cite this article: H Purwanto *et al* 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* **342** 012054

IOP Conf. Series: Materials Science and Engineering **342** (2018) 012054 doi:10.1088/1757-899X/342/1/012054

Phase change of iron ore reduction process using EFB as reducing agent at 900-1200ºC

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Abstract. Treatment of low grade iron ore involved reduction of oxygen in iron oxide by using reductant such as carbon monoxide or hydrogen gas. Presently, carboneous materials such as coke/coal are widely used as a source to provide reducing gas, but some problem arises from this material as the gas can harm the environments. Therefore, empty fruit bunch biomass from oil palm becomes an alternative to replace the usage of coke/coal as their major composition is carbon and hydrogen. The idea of replacing coke with biomass will reduce the amount of carbon dioxide release as biomass is a carbon neutral and renewable source, and at the same time abundance of waste from oil palm industries can be overcome. Therefore, the aim of this research is to upgrade the low grade iron with reducibility more than 50% being used in iron and steel making. In this research, low grade iron ore are mixed together with EFB then is making into composite pellet before being reduced at certain parameter chosen. The variables involved in this research is composition EFB (10%, 30% and 50%), temperature (1000ºC, 1100ºC and 1200ºC) and reduction time is fixed with 30 minutes. From the experiment conducted, the highest reducibility achieved is 76.37% at temperature 1200ºC. While XRD analysis shows the existence of metallic iron phase started to form at 1000ºC with composition of 30% of EFB. Meanwhile, from magnetization test show that at 1200ºC the highest magnetic susceptibility is achieved as the dominance phase at 1200ºC is metallic phase. Therefore it is an interesting alternative to replace coke with biomass for reducing agent in upgrading low grade iron into workable ores.

1. Introduction

Malaysia should upgrade low grade iron as limited sources of workable iron ore is available; hence treatment of low grade iron ore is a good alternative which simultaneously can reduce production cost and also the depletion of high grade iron ore. Malaysia iron ore is impossible to be used in domestic iron and steel making as the ore contain high amount of combined water and impurities [5]. Therefore, the proposed idea to improve the iron content of local iron ore into acceptance level of iron ore is an attractive alternative. The local iron ore is deposited from various mining area in Malaysia and total iron ore deposited is estimated about 9.6 Mt in 2014 as stated in minerals yearbook [1]. The ores are mainly used by the domestic cement, and also iron and steel industries.

Present process of treating the local iron ore with coke have contributed to high amount of carbon dioxide emission and this situation is worrying for the environment. International Energy Statistics estimated about 59 million metric tonnes of CO_2 are given off from the total coal used in Malaysia until 2013 [2]. Since Malaysia is one of the largest palm oil industry in the world, utilizing biomass especially from palm oil industry in the meantime prompt to lower need of fossil fuels which is contributed in reducing carbon dioxide emission to the environment. Hence, alternative ways have been taken to replace the coke as reducing agent with biomass waste. As one of the largest palm oil exporters, Malaysia has abundance of oil palm waste which is not being utilized [7]. Therefore, the chosen of EFB from the palm oil waste as a reducing agent is an interesting alternative as if the waste is not being utilized; the cost to eliminate the waste is high.

Hence in this recent study, the oil pam EFB is used as a reductant in order to reduce the iron oxide. Previous research have been focus at low temperature studies, therefore in this study high temperature has been chosen in order to study the effect of the temperature on the composite pellet of iron oxide with EFB. The aim of this research is to determine the suitable and optimum parameters in order to produce metallic iron phase and to study the reduction mechanism by using EFB as the reducing agent.

2. Experimental Procedures

2.1 Raw Materials

The raw materials used in this experiments were obtained from different area which is iron ore was taken from Bahau while the oil palm Empty Fruit Bunch (EFB) is taken from nearby palm oil mill (Mempaga-Bentong,Pahang). Table 1 shown the chemical analysis of original iron ore and both proximate and ultimate analysis of the EFB was provided in Table 2. Before crushing process, the raw materials were dried in the oven to remove moisture for 24h at 110ºC. The iron ore was crushed to get coarse powder up to 50 micrometer and then was sieved to get finer iron powder to be observed under XRD, XRF and TG-DTA machine. The fine iron ore and empty fruit bunch was subsequently mixed with chosen mass ratio and was made into pellet by hand rolling. The pellet was compacted to produce pellet with 10mm-20mm in diameter.

Table 1. Chemical analysis of iron ore (Bahau iron ore)

Elements	Fe	Al_2O_3	MgO	SiO_2	K_2O	TiO_2	Cr_2O_3	MnO	Fe_2O_3
Percentage (%)	51.92	9.20	0.44	14.87	0.06	0.52	0.06	0.23	74.23

Table 2. Properties of Empty Fruit Bunch

Proximate Analysis (wt.%)				Ultimate Analysis (wt.%)			
Moisture	Volatile	Fixed	Ash	Carbon	Hydrogen	Oxygen	Nitrogen
6.8	83.94	8.98	7.08	45.64	6.19	48.17	0.35

2.2 Methods

Reduction test is conducted using vertical electric tube furnace with a reactor quartz tube in the middle. The pellets are put in the reactor and are heated at temperature of 1000ºC, 1100ºC, and 1200ºC. Before sample was inserted into the furnace, the argon gas was kept flow into the furnace the make the condition inside in inert state. Heating rate was set at 10ºC/min and the temperature was maintained for 30 min for the reduction process to be completed. This experiment was conducted in inert condition with the absence of air or other impurities. During cooling process, argon gas was kept flow in order to avoid oxidation occurred to the composite pellet during reaction inside the furnace.

2.3 Estimation of Reduction Degree

In reduction reaction, the reaction is completed when minerals transformation from hematite had completely changed into metallic iron. Several phase changes during minerals transformation are:

i. **Hematite** to **Magnetite**: $3Fe_2O_3 + CO \longrightarrow 2Fe_3O_4 + CO_2$

ii. **Magnetite** to **Wustite** : $Fe_3O_4 + CO \longrightarrow 3FeO + CO_2$

iii. **Wustite** to **Iron** : $FeO + CO \longrightarrow Fe + CO_2$

By using molecular weight equation, the weight loss for each reduction reaction due to oxygen removal was calculated. From the molecular weight equation, weight for each compound is known. Similar to iron ore from other places, the major elements in Bahau iron ore are geothite and hematite. For every phase transformation involved in reduction reaction the oxygen removed is estimated as shown in Table 3 and calculation of reducibility shown in equation (2.1).

Table 3. Estimation of oxygen removal

Reaction	Reductant	Oxygen removal (%)
$FeO(OH) \rightarrow Fe_2O_3$	CO / H_2	2.5
$Fe_2O_3 \rightarrow Fe_3O_4$		3.33
$Fe_3O_4 \rightarrow FeO$		6.89
$FeO \rightarrow Fe$		22.22

$$\text{Reducibility} = \frac{Reduction\ degree}{Theoretical\ oxygen\ removal} \quad (1)$$

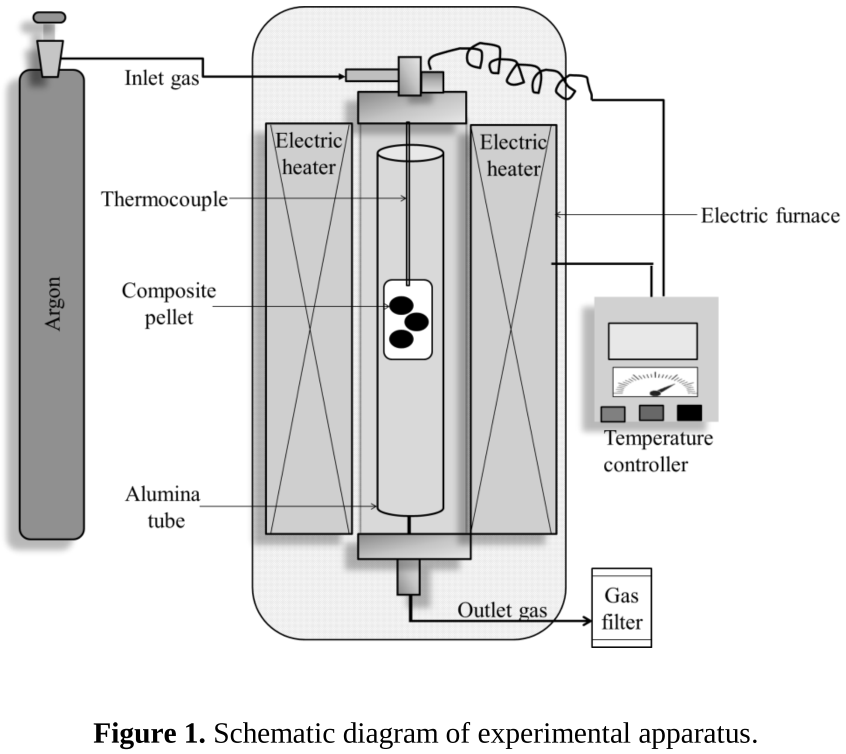

Figure 1. Schematic diagram of experimental apparatus.

3. Results and Discussion

3.1 Reducibility behavior of composite pellet after reduction process

The composite pellet with different composition of EFB was reduced at temperature 1000°C to 1200°C. The reducibility curve of iron ore at different temperature are shown in Figure 2. The graph shows increasing trend as the temperature increase and the reducibility reach the highest value of 78.22% at 1200^{o}C. As there was reaction between the ore particles and the EFB, the reduction degree is corresponding with the increasing temperature. Reduction degree was calculated based on the oxygen removal from iron oxide in the iron ore as shown in equation (2.1). This condition will then contribute to phase change in the iron oxide as the carbon in the EFB act as a solid reductant during reduction reaction.

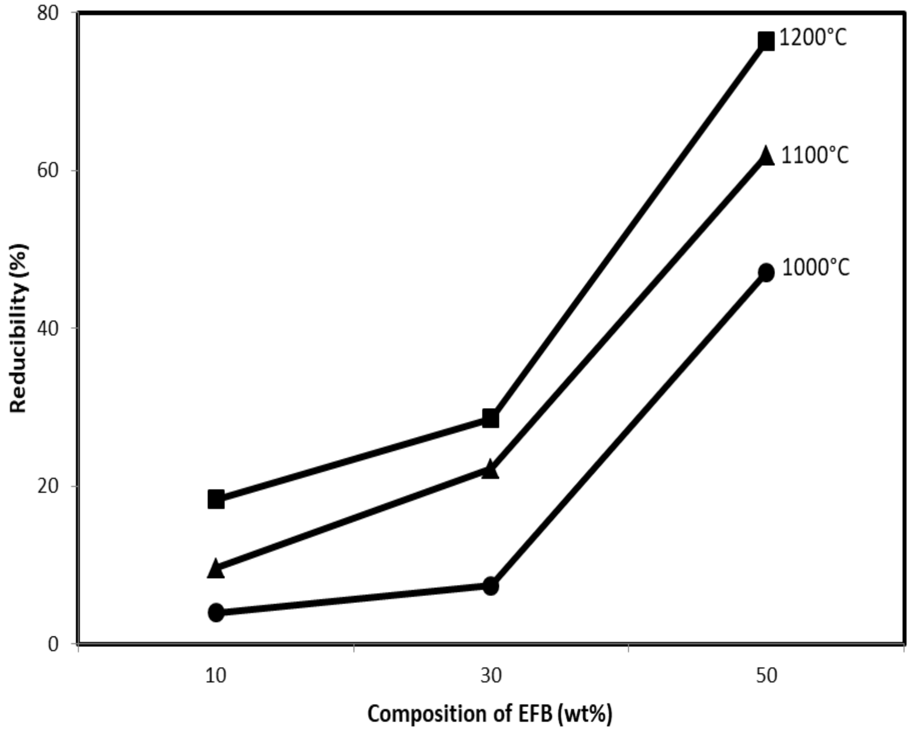

Figure 2. Reduction curves of reduced composite pellet with different composition of biomass and temperature.

3.2 Characterization of reduced iron oxide – EFB composite pellet by XRD

Figure 3 shows XRD pattern of an original iron ore and composite pellet after reduction process at 1200^{o}C with various composition. The pattern of original iron ore is clearly indicated the major compositions are geothite ($Fe_2O_3.H_2O$) and hematite (Fe_2O_3) together with some quartz (SiO_2) as the impurity. The composite pellets of all compositions on reduction at 1000^{o}C shown mainly wustite and iron phase. However, metallic phase started to become a dominance phase when 50% of EFB used compared to a temperature of 1200^{o}C metallic phase is the dominance at lower EFB content. It may be further noted that at higher temperature the reduction is better with lower content of reductant agent. This condition is consistent with the higher reduction degree achieved at 1200^{o}C for all compositions. The reaction between the iron ore and EFB was step up as the temperature increase and the oxygen was removed expeditiously compared to the oxygen released during reduction at lower temperature. Additional heat was required during the reaction since the EFB was in solid form hence more heat was required to vaporize the EFB into gas phase. The EFB then act as reducing agent in the form of carbon monoxide gas to ensure the reaction was proceed.

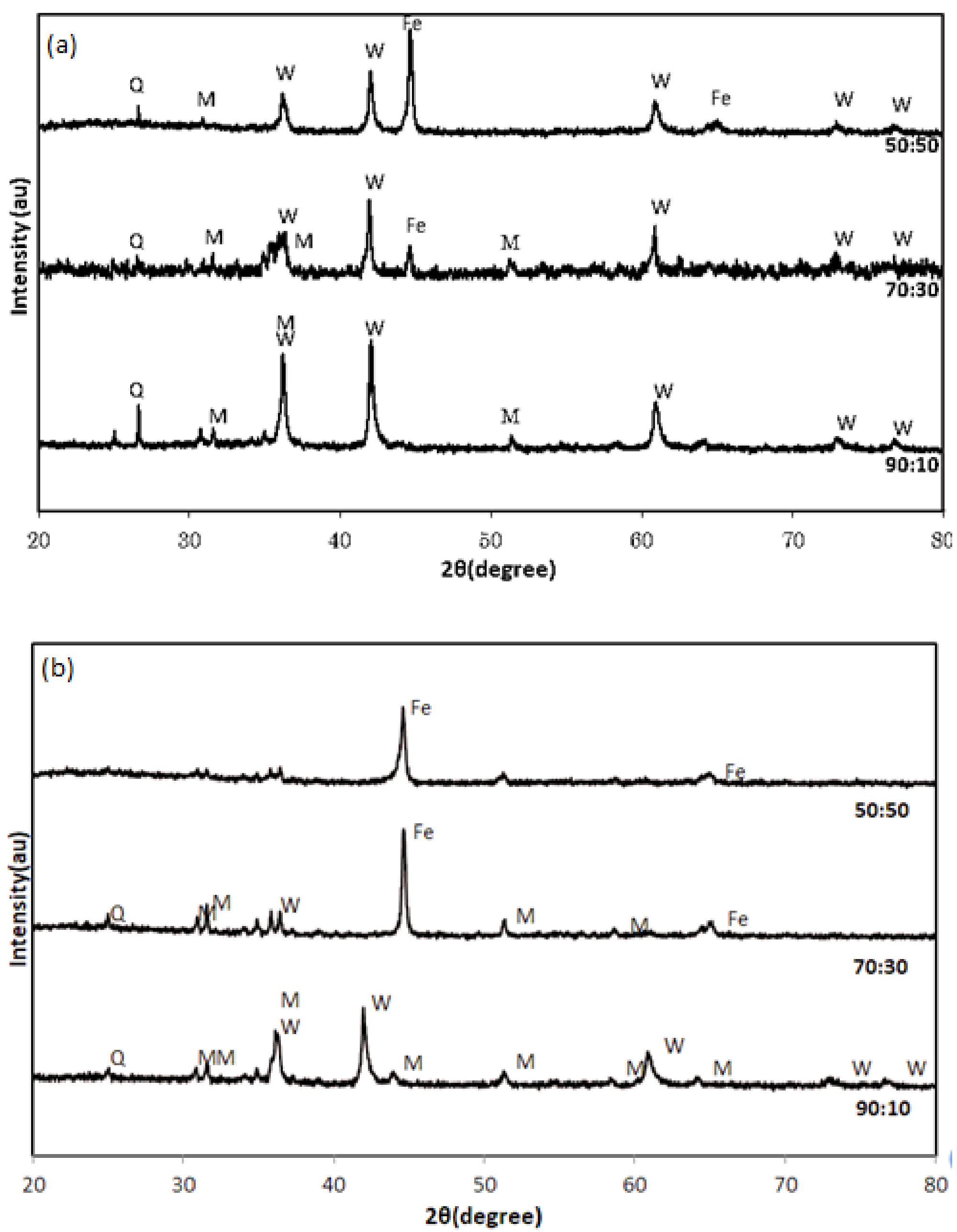

Figure 3. XRD pattern of composite pellet samples after reduction with different EFB composition a. 1000ºC; b. 1200ºC.

3.3 Analysis of magnetic properties of reduced pellets

Figure 4 shows the magnetization curve of the reduced composite pellet containing 30mass% of EFB at different temperatures. Generally, increasing temperature is enhanced the magnetization of the reduced pellet and this condition can be determined by the magnetic susceptibility and magnetic saturation parameters. Magnetic susceptibility is determined from the slope of the magnetization curve and commonly high in the presence of ferromagnetic materials and low for paramagnetic and diamagnetic materials. The magnetic saturation is measured from the point where the minerals started to reach its saturated state. Purwanto et al.[5] stated that the magnetic saturation for the untreated laterite ore is 3.38emu/g, and the value is quiet low due to the main mineral in the ore are geothite which is considered as weak paramagnetic mineral. Reducing the composite pellet at 1000°C is enhanced both the magnetic saturation and magnetic susceptibility simultaneously. Reducing composite pellet at 1200°C clearly improved magnetic properties as the value for magnetic saturation is comparably higher than original iron ore as reported. At 1000°C, the value of the saturated magnetization is 36.5 emu/g which is relatively higher compared to the original iron ore and this result correspond to the existence of magnetite phase that exhibit ferromagnetic properties or known as strong magnetic minerals. As further reduced up to 1200°C, the magnetization is slightly increased as the iron phase start to become a dominance peak in the reduced composite pellets. The magnetization at 1000°C is lower because of the formation of wustite phase cause the magnetization to decrease because wustite is a weak magnetic phase. As reported, the magnetic susceptibility of original iron ore is low as the main phase in the ore is geothite which attribute properties of weak magnetic phase. Therefore, similar to the magnetic saturation, the susceptibility of the magnetic properties is lower at the presence of wustite or other weak magnetic phase. The magnetic susceptibility at high temperature is in the range of 36-45emu/g, exhibit a degree of susceptibility similar to natural magnetite which is in the range of 35.98-45.14 emu/g [12].

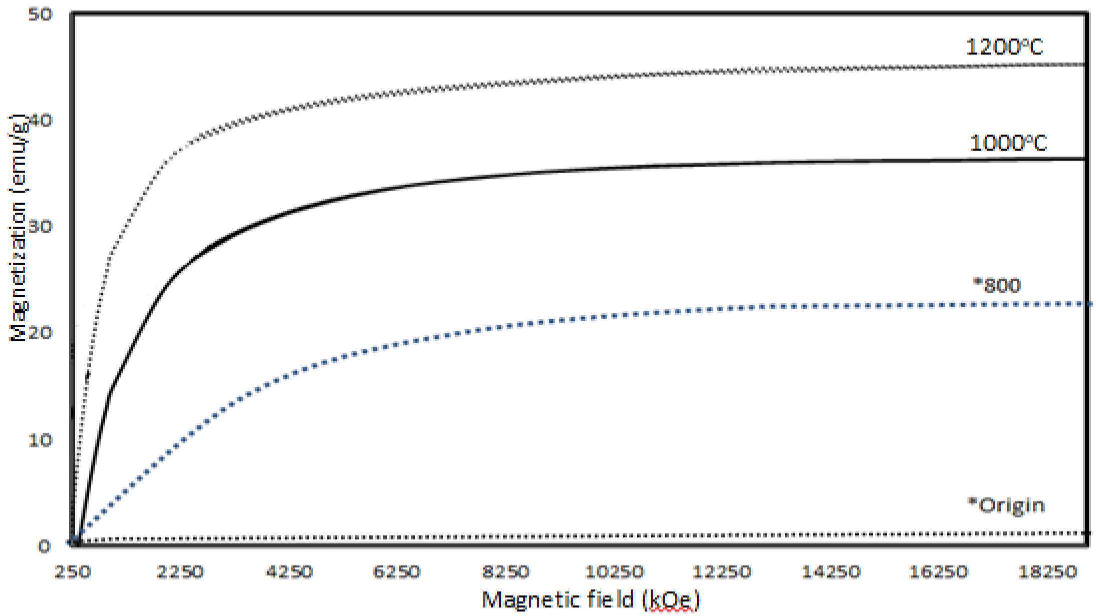

Figure 4. Magnetization curves of composite pellet with 30% EFB content at different temperature.

4. Conclusion

Reduction test of iron ore with EFB at high temperature of 1000°C, 1100°C and 1200°C have been conducted and the following conclusions were achieved. The extent of reduction was higher at maximum temperature compared to a lower temperature. Reduction reaction of iron oxide was rapidly reduced in biomass-iron oxide composite pellet. The highest reduction degree of 38.22% was achieved at 1200°C shown that the reduction degree increase with increasing temperature. Metallic iron phase was existed as the reaction proceeds at higher temperature as indicated in XRD patterns of the composite pellet. At temperature of 1000°C metallic iron phase started to appear in the composite pellet with the right composition of biomass-iron oxide. The magnetic test for different temperature of reduction reaction have revealed that iron phase have higher magnetic properties compared to wustite as iron phase exhibits ferromagnetic properties. The magnetization of reduced pellets at 1200^{o} C was 44.6emu/g which is higher compared to at 1000^{o}C. This can be concluded that iron phase have better magnetic properties compared to other phase and this is one of the preferred condition in reduction reaction process.

References

[1] Takahashi, R., 1990, *Fundamentals of Metallurgical Reaction Kinetics, Group Training Course on Mineral Processing and Metallurgy in the Research Institute of Mineral Dressing and Metallurgy (SENKEN).*

[2] Habermann, A.,Winter, F., Hofbauer, H., Zirngast, J., and Schenk, J. L, 2000, *The Iron and Steel Institute of Japan (ISIJ) International*, **40** 10 p 935-942.

[3] Wei, R., Cang, D., Bai, Y., Huang, D. and Liu, X, 2016, *Ironmaking & Steelmaking*, **43** 2 p 144-152.

[4] Muwanguzi, A., Karasev, A., Byaruhanga, J. and Jönsson, P, 2012, *ISRN Materials Science*, p 1-9.

[5] Abd Rashid, R. Z., Mohd. Salleh, H., Ani, M. H., Yunus, N. A., Akiyama, T., & Purwanto, H, 2013, *Advanced Materials Research*, **701** p 28-31.

[6] Abd Rashid, R. Z., Mohd. Salleh, H., Ani, M. H., Yunus, N. A., Akiyama, T., & Purwanto, H., 2014, *Renewable Energy*, **63** p 617–623.

[7] Choy, Y. K., 2005, *Energy Sources*, **27** 7 p 589–596.

[8] Guo, D., Hu, M., Pu, C., Xiao, B., Hu, Z., Liu, S., Zhu, X, 2015, *International Journal of Hydrogen Energy*, **40** 14 p 4733–4740.

[9] N. F. M. Yunos, N. A. Aziz, A. N. Ismail, M. A. Idris, 2015, *Materials Science Forum*, **819** p 31-36.

[10] Sahajwalla, V., Dubikova, M., & Khanna, R., 2004, *Transformation through Technology*, p 351-362.

[11] Srivastava, U., Kawatra, S. K., & Eisele, T. C., 2013, *International Journal of Mineral Processing*, **119** p 51–57.

[12] Yunus, N. A., Ani, M. H., Salleh, H. M., Rashid, R. Z., Akiyama, T., Purwanto, H., & Othman, N. E., 2014, *International Journal of Minerals, Metallurgy, and Materials*, **21** 4 p 326-330.

[13] Takyu, Y., Murakami, T., Son, S. H., & Kasai, E., 2015, *ISIJ International*, **55** 6 p 1188–1196.