Analysis of the effect of temperature on the reduction roasting process of Lampung manganese ore using palm kernel shell charcoal

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Abstract. Lampung Province has many manganese ore resources that have not been fully utilized. According to Indonesian Minister of Energy and Mineral Resources regulation no. 5 of 2017, manganese ore is prohibited from being exported before the ore is processed into a concentrate with 49% manganese content. The type of mineral contained in manganese ore used in this experiment is pyrolusite (MnO2). The method used to improve the Mn content used in this experiment is to reduce roasting. The reducing agent in this experiment is 25% palm kernel. The roasting process is carried out at 1000 °C, 1100 °C and 1200 °C. Meanwhile, the holding time varies from 30 to 150 minutes. The results obtained from baking are characterized using XRF to determine the final product content. XRD and SEM-EDS are used for the result identification phase. The highest content of manganese was 41.28% by weight obtained after baked at 1100 °C for 120 minutes with the formed phase being Mn2SiO4.

Keywords: Manganese ore; pyrolusite; palm kernel shell charcoal; reduction roasting

1. Introduction
Lampung Province has manganese ore resources with 30-35% by weight of manganese content. Based on the Regulation of the Minister of Energy and Mineral Resources no. 05 of 2017, there is a ban on the export of manganese ore before it is processed into concentrates with a minimum content of 49%. Research on manganese ore needs to be done at a laboratory scale before being used at a factory scale to obtain optimal data.

The roasting reduction method is used to increase the levels of manganese in the ore. Research on the reduction of metal oxides with carbon reductors has been carried out and in general, the process that occurs is when a mixture of ore and reducing agent is heated under inert conditions, the reduction process will take place from the reaction of CO reductant gas with ores [1]. According to Welham's research, in the Mn-OC balance system, MnO2 will react above 510 °C, and then Mn2O3 will react above 915 °C [2]. In contrast to Yi et al., Which shows that there is still a Mn2O3 phase that has not reacted to roasting for 10 minutes, then after baking for 30 minutes, there are only MnO and Fe phases [3]. The reactions that occur in the process of reducing the manganese ore roasting according to Yi et al. [3], like the following equations (1), (2), and (3):
Based on this statement it can be said that temperature and time are variables that affect the yield of manganese ore roasting.

Research on the reduction of manganese ore has been carried out by several researchers. Kononov and Ostrovsky [4] conducted research with three different types of manganese ore and compared the reduction reactions that occur under argon, helium, and hydrogen conditions and obtained the fastest reaction result is using hydrogen gas [4]. Qiu Ye [5] examined low level manganese ore using microwave heaters at temperatures of 300 °C to 800 °C and found that there was a phase change with increasing temperature, but above 750 °C there was only thickening of Mn₂SiO₄ phase on the roasting results. Gao et al. [6] in his research pre-reducing manganese ore using CO gas reducing agents, then doing kinetic analysis and obtaining the reduction rate of manganese ore with CO gas reducing agents increases with increasing temperature and CO levels in reducing gas and energy activation of 66 kJ/mol.

This research uses alternative reductant derived from oil palm shells. Palm oil shells in Indonesia have the potential to become alternative reductors after the torrefaction process is carried out to form palm shell charcoal. A projection of oil palm plantations in Indonesia in 2020 according to Miettinen et al. is reaching 13 million hectares [7]. Aisha Noor et al. [8] state that oil palm shells have a fixed carbon content of 50.23 % by weight. Palm shells have low emissions when burning because they only have ash content of around 2-8 %, and have low sulphur content [9]. This study has a focus on the effect of temperature on the roasting process of manganese ore originating from Lampung Province using coconut shell charcoal reductors.

2. Method

The study was conducted in the research unit for mineral technology - LIPI using a muffle furnace with a maximum temperature of 1200 °C. This research was carried out using manganese ore formed by pellets together with palm shell charcoal as much as 25 % by weight of the manganese ore used. The results of XRD characterization and mineralography of manganese ore can be seen in Figure 1. Manganese ore used is manganese ore from Way Kanan, Lampung, Indonesia. Manganese ore and coconut shell charcoal used is -100 # + 120 #. The composition of coconut shell charcoal and manganese ore used can be seen in Tables 1 and 2.

The formed pellet is then heated in a muffle furnace at a temperature of 100-120 °C for 4 hours to remove the moisture content that is on the surface of the pellet. The study was conducted using temperature variations of 1000, 1100, and 1200 °C and holding time for 30, 60, 90, 120, and 150 minutes. The results of the study were characterized using XRF, XRD and SEM-EDS tests.

Table 1. Proximate analysis of palm shell charcoal.

| Parameter       | %    |
|-----------------|------|
| Volatile Matter | 20.47|
| Ash             | 2.1  |
| Fixed Carbon    | 77   |
| Moisture        | 0.43 |
Table 2. Results of XRF manganese ore characterization.

| Element | %   |
|---------|-----|
| LE      | 54.153 |
| Mn      | 35.464 |
| Fe      | 3.827  |
| Si      | 4.960  |
| Pb      | 0.508  |
| Zn      | 0.427  |
| Cu      | 0.158  |
| S.      | 0.083  |
| Ni      | 0.059  |
| P.      | 0.034  |
| Sb      | 0.025  |
| Zr      | 0.01   |
| Others  | 0.292  |
| Total   | 100   |

Figure 1. Characterization results of manganese ore (a) XRD pattern; (b) Mineralography.
Note: Pyrl = pyrolusite, Cryp = Cryptomelane

3. Results and Discussions
Figure 2 shows a graph of mass loss percentage from roasting. At a temperature of 1000 °C, the greatest mass loss was found in a 120 minute holding time variation of 23.49 %. At 1100 °C, the biggest mass loss was also found at a detention time of 120 minutes, which is 25.74 %, however at a temperature of 1200 °C, the greatest mass loss was at a holding time of 90 minutes, which is 22.27 %. Based on Figure 2, at temperatures of 1000 °C to 1100 °C mass loss percentage tends to increase, however at a holding time of 30 minutes and 150 minutes, mass loss has decreased. The reduction is assumed due to the high silica ore content for low-grade manganese ore, which is 4.96 % by weight. According to research
Kumar et al. [10], silica level affects the rate of burn reaction of manganese ore. Manganese ore with high silica content has a slower reaction rate compared to silica ore with high iron oxide levels [4].

Figure 3 shows a graph of manganese ore roasting. The highest levels of manganese were obtained during 120 minutes roasting at a temperature of 1100 °C at 41.28 wt %. These results are consistent with Yi's research which obtained the highest levels of manganese at a temperature of 1100°C, but Yi got the highest levels at a faster holding time of 100 minutes [3]. Based on Figure 3 it can be seen that in roasting with a holding time of 90 minutes, manganese levels have decreased. This is caused by the inhomogeneity of the sample of manganese ore pellets used.

The SEM-EDS results of roasting at a holding time of 120 minutes and temperatures of 1000 °C, 1100 °C, and 1200 °C are shown in Figure 4. Based on the results of SEM-EDS characterization, it can be assumed that the phases formed on the results of manganese ore roasting between other Fe metal phases, and manganese.

Figure 2. Percentage for the mass loss resulted from manganese ore roasting

Figure 3. Mn content of manganese ore roasting results

Figure 5 shows the variations in temperature and time tend not to affect the formation of the manganese ore roasting phase because the majority of the formed phases are silicate Mn$_2$SiO$_4$ phases.
without the oxide or carbide phases. This is because the composition of the constituent elements of manganese ore used has low levels of Mn and Fe as shown in Table 2. This results differ from the manganese ore used in the study of Yi et al., that use total Fe content of 21-28 wt %, so that in that research the Mn oxide (MnO) phase was formed [3].

![SEM-EDS characterization samples](image)

**Figure 4.** Results of SEM-EDS characterization in samples with a hold time of 120 minutes (a) Temperature of 1000 °C; (b) Temperature of 1100 °C; (c) Temperature of 1200 °C
4. Conclusions and Recommendations

Based on the results and discussion it can be concluded that the temperature affects the levels of manganese linearly, but for 90 minutes roasting the manganese levels have decreased which is assumed due to differences in the initial composition of the ore. This might be caused by not doing the sampling method when taking manganese ore before making pellets. Percent loss of mass also tends to increase with increasing temperature, but the samples that are roasted for 30 minutes and 150 minutes have decreased which is assumed due to the kinetic factors influence caused by high silica levels. Manganese formed from the results of roasting is Mn$_2$SiO$_4$. To obtain high levels of manganese, further research can be done using an inert environment to obtain consistent results, using sampling methods to ensure homogeneous samples, as well as silica separation before the roasting process to maximize the reduction process and also minimize the formation of the Mn$_2$SiO$_4$ phase.

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