Lowering of Grinding Energy and Enhancement of Agglomerate Strength by Dehydration of Indonesian Laterite Ore

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In order to utilize the iron-rich laterite as raw material for ironmaking process, the effect of dehydration on physical properties of its agglomerate was investigated experimentally. The laterite used in this experiment was produced in Sebuku Island (Indonesia) containing 50.88 mass% of iron. The laterite ore was analyzed by X-ray diffraction for its mineral composition. Differential Thermal Analysis was employed for the thermal analysis and High Temperature microscopic observation was conducted to clarify the behavior of dehydration during the thermal treatment. Agglomeration was carried out in the form of briquette and the strength was measured for laterite ore before and after preheating. Furthermore, Grinding Work Index was measured to evaluate the energy consumption for grinding operation.

X-ray diffractometry and Differential Thermal Analysis revealed that the iron mainly formed iron oxide hydrate (goethite), and it decomposed at the range of 530–673 K. High Temperature Microscopic observation explained that the dehydration resulted in crack generation. The crushing strength was measured for the briquettes prepared from the laterite with or without preheating. The improvement of crushing strength was obtained by preheating the laterite ore at 673 K for 1 h. The grinding work index for preheated laterite was obtained about 35% lower than that of no preheated original ore. Generally, the experimental results showed that preheating treatment promoted in increasing the crushing strength of briquette and decreasing energy consumption for the grinding of laterite ore.

KEY WORDS: laterite; preheating; dehydration; grinding energy; ironmaking.

1. Introduction

Laterite ore is a natural resource containing several elements such as iron, silicon, aluminum, chromium, nickel, cobalt, and so on. It is widely and abundantly distributed in the equatorial region, however, the effective utilization has never been so advanced. This is caused by the quite low purity when it is used as a raw material for the individual metal resources. In Indonesia, the laterite ore is also abundant and is mainly used as a nickel resource, while the usage is limited to the laterite having Ni content more than 2 mass%. At present, the low-grade Ni laterite ore (normal-ly, lower Ni content than 2 mass%) has never been in effective use in spite of the huge deposit. Although the Ni content is not enough in Ni production industries, around 50 mass% Fe is sometimes included in the low-grade Ni laterite ore. As Indonesia has no domestic deposit of hematite ore, the relatively high-grade Fe laterite is quite attractive as a domestic iron resource.

In ironmaking process, iron ore is supplied in forms of agglomerate such as sinter, pellet or briquette. One of the requirements for the agglomerate is to have enough strength which is an important property during the transportation, heating, reduction and so on. In literature, there are some reports concerning processes to prepare iron ores classified to limonite group in ironmaking process. Imanishi et al.¹¹ studied pelletizing of the residual solid after Ni extraction from a high-grade Ni laterite ore. They revealed that the strength of the green pellets was not enough for use in ironmaking process but can be increased by adding limestone or bentonite as a binder. The report suggested that the low strength of the green pellets was caused by the large porosity of the residue due to the goethite decomposition during the Ni leaching. Sayama et al.²³ investigated the strength of the briquette produced from the limonitic iron ore. They reported that the higher pressure in briquetting process resulted in the higher strength of the briquette. They also indicated that the higher strength was obtained for the briquette fired at the higher temperature. Some studies reported elsewhere that combined water influences the properties of agglomerates produced from iron ore containing goethite.³⁻⁷

In contrast, a process has never been studied to prepare the high-grade Fe but low Ni content laterite for the ironmaking industry. Therefore, it is necessary to investigate the possibility of the high-grade Fe laterite as a resource of ironmaking processing. The laterite contains amount of crystalline water. This water is mostly combined with iron oxide in a form of iron oxide hydrate. The dehydration of the combined water during high temperature process would rise a structure change which was a large influence on the strength of agglomerates made by laterite ore. Hence, this study aims to clarify the effects of the pretreatment, in par-
ticular, the dehydration on the grinding energy and the strength of agglomerate after firing. Briquettes were employed for the agglomerate since its production was relatively independent of the particle size distribution of ore.

2. Experiment

2.1. Laterite Sample

Indonesian laterite ore taken from Sebuku (Kalimantan) was employed as a sample. Chemical composition of the sample, which was measured by Inductively Coupled Plasma (ICP) device, is listed in Table 1. The total Fe content is 50.88 mass%, whereas the impurities, such as nickel, chromium, aluminum and so on, are also included. The amount of the impurities is larger than that in hematite ore used in ironmaking industry. Mineral composition of the sample was determined by X-ray diffraction method. Figure 1 shows the X-ray diffractogram. It is clear that the iron exists mainly in the form of goethite (Fe$_2$O$_3$ ·H$_2$O). The peaks of hematite (Fe$_2$O$_3$) are also detected but have low intensity. The other strong peaks stem from the existence of quartz (SiO$_2$).

2.2. Sample Preparation

For the estimation of the crushing strength, the laterite sample was formed in a cylindrical briquette. The laterite ore was dried at 413 K in an oven for 1 day, and subsequently ground with a ball mill (volume: 3.50 /$m^3$) at a rotating speed of 60 rpm for 7.2 ks. After grinding, 70 mass% of the sample has a size under 44 mm, and almost 100 mass% has that under 150 mm. About 2 g of the ground sample was then subjected to produce a briquette by the mechanical compression at 300 MPa. The size of the briquette formed was 11 /$m^3$ in diameter and 81 /$m^3$ in height. Three kinds of briquettes were produced with different methods according to Fig. 2. The first method is the briquette produced by the laterite sample without heating, but only with grinding (briquette A). Another is that with heating at 673 K for 1 h after the grinding (briquette B), and the other is that with the heat treatment prior to the grinding (briquette C). For each run, the briquette was completely dried at 373 K in an oven for 36 ks. Firing of the briquette was conducted in a muffle furnace under air flowing at 8.33 × 10$^{-5}$ Nm$^3$/s. The briquettes were heated at the rate of 0.16 K/s up to a prescribed temperature range from 1 498 to 1 623 K, where the temperature was kept for 7.2 ks.

2.3. Procedure

The strength of the briquette before and/or after firing was measured by compression-testing machine as shown in Fig. 3. A briquette was laid on a table below a load cell. The table was elevated by hydraulic compression at a rate of 2.16 × 10$^{-4}$ m/s. The maximum load needed to break the briquette was defined as the crushing strength.

In-situ observation of the surface morphology for the laterite sample during heating was performed by using High Temperature Microscope (OLYMPUS BH2–UMA). A bit of the laterite ore was put in an alumina crucible of 3 /$m^3$ in diameter and 3 /$m^3$ in height. The laterite was heated at the rate of 8.33 × 10$^{-2}$ K/s up to 1 073 K and its surface was simultaneously monitored by the CCD camera connected to the microscope. All the images were stored in a video tape. To avoid deposition of evaporated gas from the sample, argon gas was passed through the furnace chamber at the rate of 3.33 × 10$^{-7}$ Nm$^3$/s. Differential Temperature Analysis (TA instrument SDT 2960 series) was conducted at the heating rate of 0.16 K/s up to 1 273 K under the Ar atmosphere. Porosity of the briquette was determined with mercury-porosimeter.

Grinding Work Index of the laterite ore was determined according to the Japanese Industrial Standard (JIS M 4002). The ball mill used in the test has a size of 0.305 m in inner diameter and 0.305 m in length, and was set at the rotating speed of 70 rpm. For the first operation, about 7 × 10$^{-4}$ m$^3$ of the sample was introduced into the ball mill, and was ground for 100 rotations. The sieving of the ground sample

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Table 1. Chemical composition of laterite ore.

<table>
<thead>
<tr>
<th>Element</th>
<th>Fe$_{tot}$</th>
<th>NiO</th>
<th>Cr$_2$O$_3$</th>
<th>Cr$_2$O$_4$</th>
<th>CaO</th>
<th>SiO$_2$</th>
<th>Al$_2$O$_3$</th>
<th>MgO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight</td>
<td>50.88</td>
<td>0.38</td>
<td>0.03</td>
<td>5.90</td>
<td>0.03</td>
<td>8.66</td>
<td>8.84</td>
<td>0.31</td>
</tr>
</tbody>
</table>

*Laterite after heating at 1073 K, weight loss -11.0%*

Fig. 1. XRD pattern of original ore.

Fig. 2. Flow diagram for pretreatment methods of laterite ore.

Fig. 3. Schematic diagram of compression testing machine.
was subsequently carried out and the weight of the sample over the size of 250 μm was measured. From the result, the weight of the sample under the sieve produced by one rotation \( G_{bp} \) was determined. The next grinding was operated for the sample over the sieve with addition of original sample. The amount of this addition refers to the weight of the sample under 250 μm. The grinding was repeated until the \( G_{bp} \) become constant. The grinding work index \( W_i \) was defined according to the Eq. (1),

\[
W_i = \frac{44.5}{P_1^{0.25} \times G_{bp}^{0.82} \times \left( \frac{10}{\sqrt{P}} - \frac{10}{\sqrt{F}} \right)} \times 1.10 \quad \text{(kWh/t)}
\]

where, \( P_1 (\mu m) \) is the size of the sieve employed, \( G_{bp} (g) \) is the average of \( G_{bp} (g) \) for the last three operations, \( P (\mu m) \) is the 80 mass%-particle size of the sample under the sieve employed, and \( F (\mu m) \) is the 80 mass%-particle size of the original sample.

3. Results and Discussion

3.1. Thermal Analyses of Laterite Ore

Figure 4 shows a DTA curve for the laterite sample. There is a large endothermic peak from around 530 to 673 K. Daenuwy and Dalvi\(^9\) reported that the goethite in the Indonesian laterite decomposed in the temperature range of 493 to 623 K, showing an endothermic peak in a DTA curve. The temperature necessary to decompose the goethite is different to the ores, as reported elsewhere\(^9-11\) As shown in Fig. 1, the laterite sample used contains hydrous ferric oxide of goethite. Figure 5 depicts the X-ray diffractogram of the laterite sample after heating at 673 K for 3.6 ks. Comparing the diffractogram with that before the heat treatment shown in Fig. 1, the peaks of goethite disappeared by the heating at 673 K. Instead of the disappearance, several peaks arising from hematite formation were strongly detected. This fact clearly indicates that the goethite in the sample used decomposed by the heat treatment. Therefore, the endothermic peak in the DTA curve shown in Fig. 4 also attributed to the decomposition of the goethite, that is, the release of the chemically combined water from the laterite sample.

3.2. Effect of Dehydration on the Strength of Fired Briquettes

Based on the above-mentioned results, the crushing strength was compared for the laterite briquettes prepared in the three different pretreatment methods as shown in Fig. 2. Here, in the briquette (A), goethite exists without the decomposition and in the briquettes (B) and (C), it never exists. In Fig. 6, the crushing strengths for the briquettes (A), (B) and (C) are plotted against the temperature where the briquettes were fired. The crushing strength for all the briquettes gradually increases with increasing the firing temperature up to 1 598 K. At 1 623 K, the strength for the briquettes (B) and (C) further increases whereas that for the briquette (A) decreases. It may be caused by the increase of the briquette porosity as shown in Fig. 8. For all the firing temperatures employed, the briquette (C) shows the largest strength in the three briquettes. The strength for the briquette (A) is smaller than that for the briquette (B), and almost half of that for the briquette (C). These differences in the crushing strength among the briquettes are well explained from pore generation caused by the decomposition of goethite as explained later.

3.3. Direct Observation of Dehydration Behavior

Figure 7 shows the images for the surface morphology of the laterite sample obtained from the high temperature microscopic (HTM) observation. The heating condition is also given in the same figure. The image No. 1 was taken at the heating stage lower than 673 K. In this stage, any crack generation was never observed, and the laterite sample kept the surface morphology before the heating. During holding temperature at 673 K, small crack became visible at the surface as shown in the image No. 2. This pore generation ap-
parently indicates to initiate the release of the combined water caused by the goethite decomposition. Thus, the pores are not uniformly distributed, matching the goethite distribution in the laterite ore sample. In the heating stage over 673 K, cracks were formed by connecting the pores. The cracks further grew up with increasing the temperature like a root with many branches. The images No. 3 and No. 4 represent these phenomena. As mentioned above, the briquette (A) was produced by the laterite sample without the heat treatment at 673 K. Therefore, the crack generation by the decomposition of goethite occurred in the firing process. Although sintering of the material proceeded in the firing process, the crack generated still remained in the briquette. And then the crack brought about the lower crushing strength than that for the other briquettes. For both the briquettes (B) and (C), the laterite sample subjected to the heat treatment was employed. This material is also porous due to the crack generation during the heat treatment. However, the porous particles were compacted in the briquetting process by the compressing pressure of 300 MPa. Therefore the crushing strengths for the briquettes (B) and (C) are larger than that for the briquette (A). Moreover, for the briquette (C), the grinding after the heat treatment broke the pores resulting from the crack generation. This fact gave rise to the further crushing strength than the briquette (B) as shown in Fig. 6. In Fig. 8, the crushing strength and porosity of the briquettes after firing processes are compared between the briquettes (A) and (C). It is obvious that the lower porosity resulted in larger crushing strength.

3.4. Effect of Dehydration on Grinding Energy

In addition to the improvement of the briquette strength, the heat treatment of the original laterite before the grinding process decreased energy consumption for grinding. Figure 9 shows Rosin–Rammler distribution for the ground original laterite and that after the heat treatment at 673 K for 3.6 ks. The figure was obtained by plotting the data according to the Rosin–Rammler equation as follows12):

\[
R_{D_p} = 100 \exp \left(-\left(\frac{D_p}{D_e}\right)^n\right)
\]

where, \(R_{D_p}\) is over sieve sample (%), \(n\) is distribution constant (→), and \(D_e\) is absolute size constant (μm). Table 2
lists \( D_e \) and \( n \) for the samples obtained from the Fig. 9. The median diameter \( (D_{\text{me}} \text{ (\mu m)}) \) calculated from Eq. (3) is also listed in the same table.

\[
D_{\text{me}} = D_e^{n \times 0.6935} \quad \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (3)
\]

For the original laterite, the grinding for 3.6 ks produced the particles having a median diameter of 139 \( \mu m \), whereas the further grinding for 3.6 ks resulted in the diameter of 26 \( \mu m \). In contrast, for the laterite after the heat treatment, the grinding for 3.6 ks generated the particles of 28 \( \mu m \) in median diameter. This is only half of the time to obtain the similar size for the original laterite. These results are well reflected to the changes in the grinding work index by the heat treatment.

Table 3 compares the grinding work indexes for the laterite sample with or without the heat treatment. In the same table, the index for hematite and bauxite\(^\text{13)}\) are also listed. The grinding work index obtained for the original laterite ore is 9.7 kWh/t. This value is very close to that for bauxite, but lower than that for hematite. The grinding work index for the laterite after the heat treatment is 6.3 kWh/t, which is about 35\% lower than that for the original laterite. This means that the hardness of the laterite was lowered by the heat treatment. This deterioration was clearly caused by the pore formation by the dehydration of the goethite in the laterite sample. According to the thermal analysis as shown in Fig. 3, the thermal decomposition energy for goethite was measured as the value of 1.46 kWh/t. Therefore, the total energy for grinding of laterite ore including preheating treatment saved about 20\% comparing with total grinding energy for no preheating original ore.

### 4. Conclusion

In order to clarify the possibility of the laterite ore produced in Indonesia as a resource for ironmaking process, the effects of preheating treatment on the grinding energy and the strength of agglomerates from laterite ore were examined by X-ray diffraction, high temperature microscope, Differential Thermal Analysis and measurement of Grinding Work Index. The results obtained are summarized as follows:

1. The goethite in the laterite used decomposed at the temperature range from 530 to 673 K.
2. Small crack caused by the goethite decomposition generated at 673 K. This crack grew up like a root (of tree) with increasing the temperature.
3. Crushing strength of fired briquette remarkably increased by the preheating treatment before grinding.
4. The grinding work index for preheated laterite ore is about 35\% lower than that for the original one.
5. The preheating treatment promoted not only the lowering of energy consumption for grinding but also the increasing of the crushing strength of laterite briquette.

### REFERENCES