Natural hematite properties of Lampakuk, Aceh Besar iron ore extracted by precipitation method

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Abstract. In this paper, the mineral content and its magnetic properties of iron ore from Lampakuk iron ore area, Aceh province were studied. The iron ore was prepared by co-precipitation method. As the results, the main mineral and chemical compositions of samples which were investigated by XRD and XRF analysis tests, showed that the Lampakuk iron ore contain Fe₂O₃ (86.8188%) and some minor impurities, such as SiO₂, MnO, and Al₂O₃ in varying proportions. Compare to XRD results, it was consistent with XRF, the phase compositions of iron ore are mainly hematite (Fe₂O₃). The XRD revealed that hematite is the major mineral components in the Lampakuk iron ores. The magnetic properties of the samples after milling showed there has been increased in the remanent (Br) and coercivity (Hc), while the magnetic saturation (Ms) was decreased. The electron microscope identification inform that the particle were agglomerated.

1. Introduction
Iron ore is one of the important minerals for the raw materials for the manufacturing industry [1-4]. Some properties of oxide among other iron (II) oxide (natural occurring FeO) or iron oxide, powder black oxide, iron (III) oxide (Fe₂O₃) or also known as the iron ore hematite or maghemite. Iron ore is rock containing minerals iron and a number of mineral impurity such as silica, alumina, magnesia and nickel [5,6]. Hematite, as a component in iron ore minerals, can be obtained through an extraction process using certain methods. The iron contained in the rocks can be extracted with the technology existing at the moment and have economic value.

Thus, the extraction of the metals can be meant as a separation of metals from its source, which is usually in the form of the ore. The process of extracting metal from the ore is usually done with some separation techniques, such as pyrometallurgy, hydrometallurgy, and precipitation [3,4]. The making process of precipitation affects some of the properties of the basic material such as iron oxide phase formed, impurities and agglomeration [5,6]. Therefore, a lot of research on synthesis of iron oxide to produce its pure mineral [7,8]. In this work, we introduce the co-precipitation method to extract the pure hematite from local iron ore. The precipitation technique is the most widespread attention because of its simple, easy and cheap process [9]. Here, we introduce the milling technique before the precipitation process starts. Generally, using ball milling technique, the sample can be produced fine powders [10-13].

2. Materials and Method
The iron ore was collected from the Lampakuk mining area, Aceh Besar. The iron ore was washed and dried around 4 hours in the air. Then, to have fine powders, the iron ore was milled for a few hours of 10, 20, 40, 60 hours. The sample was then manually separated by using a ferrite magnet to collect the magnetic powders. The results of the subsequent separation weighed as much as 50 grams. Furthermore, the ore dissolved in HCl. Comparison with 50 grams of iron ore and 280 ml HCl while stirring and heated at a temperature of 145 ºC using a hot plate magnetic stirrer with a speed of 350 rpm. Precipitation with ammonium hydroxide to shed done (NH4OH) 25% into the solution until the pH reaches 6 and formed precipitate. The precipitate was then washed and dried in an oven at a temperature of 150 ºC for 19 hours. Next calcination conducted at temperature 500 ºC during 2 hours using the furnace. Afterwards, the phase composition and crystallite size were identified using X-rays diffractometer (XRD Shimadzu, D6000) and the mineral composition was observed by XRF (Bruecker, S2-Stranger). The data then compared to the database issued by International Crystallography Diffraction Data (ICDD). The crystallite size was calculated by Scherrer formula. The morphology was investigated by a scanning electron microscopy (Philips, XL30). While the magnetic properties observed by using a Permagraph apparatus (Magnet Physik, 2T).

3. Results and Discussion
The sample after milling and precipitation process was firstly observed by X-rays fluorescence (XRF), and the results as shown in Table 1.

| Nr. | Compound       | Percentage (%) |
|-----|----------------|----------------|
| 1.  | Fe2O3          | 86.81          |
| 2.  | SiO2           | 9.14           |
| 3.  | Al2O3          | 2.03           |
| 4.  | CaO            | 0.55           |
| 5.  | MgO            | 0.38           |
| 6.  | SO3            | 0.01           |
| 7.  | K2O            | 0.02           |
| 8.  | Na2O           | 0.01           |
| 9.  | TiO2           | 0.03           |
| 10. | Mn2O3          | 0.17           |
| 11. | LOI            | 0.84           |

The mineral composition of iron ore after separation process, generated by X-ray fluorescence (XRF) as seen in Table 1, indicated that the magnetite (Fe2O3) is dominated by 86.55%, since the minor phases are TiO2, SiO2, Cr2O3, Al2O3 and some others. Compound silicate (SiO2) will be lost if the given substance enhancer (flux) as CaCO3. Next extraction done the process of blast furnace process is intended is the process of ore reduction at high temperatures, this process requires time as well as the amount of solvent that is large enough, but this method has the advantage that the result is more perfect. Furthermore, XRD observations can be seen in Figure 1.

The data results of the x-ray Diffraction (XRD) conducted an analysis of the identification phase is done by observing the angle 2θ, lattice distance factor (d), intensity (I/IO), the phase and the crystal structure. This identification was approached with the value of the angle 2θ according to which certain
minerals listed on JCPDS (Joint Committee for Powder Diffraction Standard). The analysis is performed with the experimental results matching techniques and JCPDS [14].

Analysis of the results of XRD looks that is the dominant phase of Fe₂O₃ and SiO₂ phase followed by a minor. If compared, the diffraction peak profiles XRD test results on the samples of iron ore magnetic separation process visible diffraction peaks are still sharp. Peak (peak) diffraction begins to widen/shrink after chemically (method of precipitation). This indicates that there has been reduction of grain size when the method of precipitation takes place [15, 16].

By doing the calculations in Table 2 shows the size of the crystallites Fe₂O₃ 31 nm for 10 hours of milling and decrease to 26 nm and 27 nm after longer milling time as 20 and 60 hour. This case shows that the method of precipitation (process chemically) have managed to reduce the size of the grains are more suitable [17].

**Figure 1.** XRD pattern of Lampakuk iron ore after milling

**Table 2.** Crystallite size of the milling samples

| Milling Time (hour) | Crystal Plane | Crystal Parameter | Crystallite Size (nm) |
|---------------------|--------------|------------------|-----------------------|
| 10                  | 311          | FWHM (°)         | 2θ (°)                | Size (nm) |
| 10                  | 311          | 0.27730          | 35,5443               | 31        |
| 20                  | 311          | 0.32470          | 35,5284               | 26        |
| 60                  | 311          | 0.31730          | 35,5752               | 27        |

**Figure 2.** SEM image iron ore after 10 hour milling
Figure 3. SEM image iron ore after 60 hour milling

As shown, before milling process, the iron ore sample has a similar grain. Furthermore, after 10 hours and 60 hour milling, the powder surface becomes smaller, but irregular due to cracks during the milling process. This result consistent with the XRD analysis from 10 h to 60 milled samples. From SEM data it is known that the milled sample has a particle size of about 200 µm.

The magnetic properties of the 20-hour milling sample can be seen in the hysteresis loop curve shown in Figure 4.9. From the hysteresis loop curve there is a slight difference with the 10-hour milling sample where there is an increase in the coercivity value (Hc) of the 20-hour milling sample to 9.00 kA / m. The remanent value of the 20 hour sample also increased to 0.071 T, but the magnetic saturation value (Ms) decreased to 0.34 T in the 20 hour milling (see Figure 2 and 3).

Figure 4. Magnetic properties for sample 10 hour milling

Figure 5. Magnetic properties for sample 60 hour milling
In the 60-hour milling iron ore sample, there was a significant increase in the coercivity and remanence values, the coercivity and remanence values were 14.50 kA/m and 0.101 T respectively and the magnetic saturation value continued to decrease, namely 0.32 T. It can be seen that there is an increase in coercivity and remanence values as the milling time increases, but there is a decrease in the magnetic saturation value of the sample [18].

4. Conclusions
From this study, it shows that mineral composition iron ore by magnetic separation contains Fe2O3 of 86.81% in purity and followed by some minor compositions. The results of the phase identification using XRD, the iron ore found in the Lampakuk, Aceh Besar district is dominated by the main phase of hematite (Fe2O3) compound and SiO2 as a minor phase. The grain size was getting smaller after longer milling time. The magnetic properties showed that there is an increase in coercivity (Hc) and remanence (Br) values as the milling time increases, but the magnetic saturation (Ms) value was decreased. The Lampakuk Iron ore can be utilized according to the application of its magnetic properties.

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