SYNTHESIS AND CHARACTERIZATION OF HEMATITE (Fe$_2$O$_3$) OF IRON ORE AND MAGNETITE (Fe$_3$O$_4$) FROM IRON SAND THROUGH PRECIPITATION METHOD FOR INDUSTRIAL RAW MATERIALS

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ABSTRACT

Indonesia is a country that has enormous iron ore and iron sand mine that can be utilized for various industrial purposes. This research has been successfully conducted synthesis and characterization of hematite iron ore and magnetite from iron sand. Iron sand and iron ore that has been crushed manually repaired with a magnet was carried out with the HCl and NH$_4$OH then dried in the temperature of 150 °C and calcinated at a temperature of 500 °C. Characterization was carried out using X-ray diffraction (XRD) and X-ray fluorescence (XRF), where the preliminary information obtained from XRF results in an iron ore sample by manual separation have 95.99% of Fe$_3$O$_4$ and followed by compounds SiO$_2$ (2.10%). While the iron sand contains 81.42% of Fe$_3$O$_4$ and 2.5% of SiO$_2$. After the precipitation process, Fe$_2$O$_3$ compounds contained in iron ore has a content of 96.58% and Fe$_3$O$_4$ compounds contained in iron sand (86.73%). The results of XRD indicate the dominant primary phase in iron ore is hematite or Fe$_2$O$_3$, and in iron sand is magnetite Fe$_3$O$_4$. Before the extraction process, Fe$_2$O$_3$ was 58.009 μm in size and after the process of extracting the particles was reduced to 20.950 μm. While the Fe$_3$O$_4$, prior to the extract, has a grain size of 59.009 μm, and after an extraction process, the grain size reduced into 25.950 μm. The calculation results indicate there is a slight size difference between the grain size of iron sand and iron ore.

Keywords: Iron Ore; Hematite; Iron Sand; Magnetite; Precipitation Method

Introduction

Indonesia is rich in mineral resources as we often encounter coal, nickel ore, iron ore, iron sand, and others. In iron ore, there are lots of mixtures of FeO (wustite), Fe$_3$O$_4$ (magnetite), and Fe$_2$O$_3$ (hematite), as well as several other impurities compounds such as Al$_2$O$_3$, MgO, SiO$_2$ which are used as minor components. Iron ore itself contains many oxide compounds which have high values with different levels in each region. Iron ore originating from Karnataka, India has a chemical composition with levels such as Fe $63.84%$, SiO$_2$ $2.64%$, Al$_2$O$_3$ $3.98%$, CaO $0.14%$, and MgO $0.08%$.\(^1\)

In the Trenggalek Regency, East Java Province has iron ore containing Fe with a total content of 22.28 to 51.26%; SiO$_2$: 8.02 to 44.18%; TiO$_2$: 3.8 to 14.76%.\(^2\) West Sumatra itself has a composition of iron content with high levels reaching 62%. In each region, the oxide content in each iron ore has a difference. This causes iron ore to be utilized directly according to its content, for example, iron ore with Fe content of 57.69-70% can be used as raw material for

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cement, and iron ore containing more than 70% can be used in the manufacture of steel. 

Figure 1. (a) Fe₂O₃ Powder, (b) The structure of Magnetite Fe₂O₃

Iron sand is a process of sand deposition containing iron particles (magnetite), which are scattered in various beaches, which are formed due to the process of destruction by weather, surface water and waves of origin rocks containing iron minerals such as magnetite, ilmenite, iron oxide, then accumulates and washed away by waves of seawater. This iron sand is usually dark gray or black, which consists of opaque minerals mixed with granules and also contains some of the most dominant compounds such as Fe₃O₄ which is 86% and other compounds including TiO₂, SiO₂, Al₂O₃ and several compounds another minor.

Figure 2. (a) Fe₃O₄ Powder, (b) The structure of Magnetite Fe₃O₄

Method

The basic material of Fe₂O₃ powder is iron ore taken from Lhoong, Aceh Besar, and for metal oxide Fe₃O₄ is the result of extraction from the iron sands of the Lampanah coast, Aceh Besar. The next step is the iron ore is cleaned and crushed, and then filtered using a filter size of 80 mesh so that the size obtained is equal. However, the iron sand is first sun-dried for 4 hours in the open air at a solar temperature of around 30,6°C. After the drying process is carried out, the next step is the manual separation process for iron ore and iron sand samples using magnetic (separation magnetic).

The next process is the precipitation process using HCL acid and base solution (NH₄OH) until it reaches pH 6, then stirring on a hot plate magnetic stirrer at 145°C at 350 rpm speed. The resulting sludge is then washed using distilled water until clean and then dried using an oven at a temperature of 150°C for 19 hours and then calcined at a temperature of 500°C for 2 hours.

The chemical treatment used in this study (precipitation method) is very important in order to improve the quality of iron ore and iron sand, especially in terms of reducing the gross elements present in both materials, and also can make grain sizes from the material becomes smaller. With the reduced grain size, this iron ore and iron sand can become one of the industrial raw materials that can be applied in the industrial fields.

The characteristics performed with XRF are used to determine the percentage of mineral content in the material. The sample used in the form of powder. For XRF testing itself, the sample does not need special treatment but can be tested directly by placing the sample in the sample holder and using argon gas for operational media.
The data obtained was weight percent (wt%), which shows the percentage of mineral content in iron ore and iron sand. Then the characteristics performed with XRD to determine the crystal structure and can inform the grain size. For the data collection process itself, X-ray Diffractometer (Shimadzu) is used.

**Result and Discussion**

**Identification Results from XRF**

The process of analyzing this method is carried out on iron ore samples by magnetic separation and chemical separation (precipitation method). The results of the composition of the iron ore using XRF are shown in Table 1.

| Compound | Magnetic separation (%) | Precipitation Methods (%) |
|----------|-------------------------|---------------------------|
| Fe₂O₃    | 95.99                   | 96.58                     |
| SiO₂     | 2.10                    | 2.10                      |
| CuO      | 0.55                    | -                         |
| Br       | 0.37                    | 0.36                      |
| CaO      | 0.24                    | 0.19                      |

**Table 2. Results Identification of iron sand samples**

| Compound | Magnetic separation (%) | Precipitation Methods (%) |
|----------|-------------------------|---------------------------|
| Fe₃O₄    | 81.42                   | 86.73                     |
| SiO₂     | 4.4                     | 2.5                       |
| Al₂O₃    | 3                      | -                         |
| P₂O₅     | 0.41                    | 0.34                      |
| K₂O      | 0.11                    | 0.075                     |

From the XRF table test results above, it can be seen that the compound with the highest content contained in iron sand after manual separation is Fe₃O₄ with a composition percentage of 81.42%. Further then followed by the TiO₂ compound, which has a percentage of composition of 7.61%. The lowest concentration compound is ZnO of 0.06%. Then the results of X-Ray Fluorescence after extracting using the Fe₃O₄ phase precipitation method showed an increase where the content of the most dominant element compared to other identified elements was Fe₃O₄ with a percentage of 86.73%. The percentage of Fe₃O₄ identified was almost 90%, indicating that the natural material from the iron sand used had a magnetite content that was more dominant than other compounds.
A comparison of XRF test results between Fe$_3$O$_4$ and Fe$_2$O$_3$ shows that the percentage level of purity of iron ore natural material has a higher percentage than the percentage of purity of iron sand.

**Identification Results from XRD**

The results of observations using XRD on iron ore samples can be seen in Figure 1. Data from the X-Ray Diffraction (XRD) is done by analyzing the phase identification with an angle of $2\theta$, lattice distance ($d$), intensity ($I/I_0$), phase, and crystal structure. This identification is with an angle of $2\theta$ certain mineral values, as stated in the JCPDS (Joint Committee for Powder Diffraction Standard). The analysis was carried out using the matching technique of experimental results and JCPDS. XRD analysis results show that the dominant phase is Fe$_2$O$_3$ and followed by the SiO$_2$ minor phase. If the diffraction peak profile is compared, the XRD test results on the iron ore samples, which are defined by the magnetic separation process, show that the diffraction peaks are still sharp.

Phase analysis using X-Ray Diffractometer (XRD) is also carried out to determine what phases are contained in Fe$_3$O$_4$ magnetite derived from iron sand. Data of XRD observations for iron sand samples can be seen in Figure 4.

In the XRD results of iron sand before experiencing peak acid-base solution still looks sharp, the phases that are present are the most dominant and have the highest intensity found in Fe$_3$O$_4$ compounds, namely at $2\theta=35.5020$, $2\theta=30.0809$, and $2\theta=56.9852$. In addition, other phases are also still visible, namely TiO$_2$ at an angle of $2\theta=73.8103$ and the SiO$_2$ phase at an angle of $2\theta=43.1163$. On the other hand, the extracted iron sand has a widening peak, which identifies that the particle size of the Fe$_3$O$_4$ compound has begun to be reduced. From the picture also visible phases are lost after the sand is extracted using the precipitation method. Moreover, the phase that arises remains dominated by the compound Fe$_3$O$_4$ at an angle of $2\theta=35.4853$.

![Figure 3. XRD results for iron ore magnetic separation and precipitation methods](image-url)
Figure 4. XRD graph results on iron sand samples before and after extraction

($\text{Fe}_3\text{O}_4$, $\text{Fe}_2\text{O}_3$, $\text{TiO}_2$, $\text{SiO}_2$).

Sample Size of $\text{Fe}_2\text{O}_3$ and $\text{Fe}_3\text{O}_4$

The precipitation method can produce a smaller grain size than the previous sizes. The average grain size from the XRD result can be defined using the Scherrer equation.\(^1\)

Calculation results for the calculation of sample grain size can be seen in table 3, wherein the table shows the size of the crystallite $\text{Fe}_2\text{O}_3$ magnetic separation process ($58.009 \mu m$) and the method of precipitation size ($20.950 \mu m$).

The results of grain size reduction for iron sand samples can be seen in table 4, where $\text{Fe}_3\text{O}_4$ in the magnetic separation process ($59.009 \mu m$) and in the precipitation, process decreased ($25.950 \mu m$).

From the calculation results of grain size measurements of iron sand and iron ore shows that there is a slight difference in grain size between the two. This indicates that there has been a reduction in grain size during the precipitation method.

Reducing the grain size that occurs in iron ore and iron sand is very useful where this material can be used as a catalyst in the application of hydrogen storage tubes, this application will be further investigated by the author. So from the results of this study, we can find out that so far, iron sand is not only one of the materials for making cement, but can be used as one of the industrial raw materials that are needed in a variety of applications.

| Table 3. Calculation of $\text{Fe}_2\text{O}_3$ crystallite size |
|-------------------------|---------------------|---------------|
| $\text{Fe}_2\text{O}_3$ Sample | Parameters |              |
|                          | FWHM(°) | θ (°) | Size (µm) |
| separation               | 0.15180 | 17.72285 | 58.009    |
| precipitation            | 0.42040 | 17.7665  | 20.950    |
Table 4. Calculation of Fe$_3$O$_4$ crystallite size

| Sample      | FWHM(°) | θ (°)  | Size (µm) |
|-------------|---------|--------|-----------|
| Separation  | 0.14680 | 17.71285 | 59.009    |
| Precipitation | 0.40040 | 17.7565 | 25.950    |

Conclusion

The conclusions obtained from this research are: Identification of minerals using XRF iron ore samples of magnetic separation process containing Fe$_2$O$_3$ (hematite) of 95.99% and the precipitation methods containing Fe$_2$O$_3$ (hematite) of 96.58%. Whereas for iron sand samples containing Fe$_3$O$_4$ with a percentage of composition 81.42% and Fe$_3$O$_4$ precipitation method with a percentage of 86.73%.

Results of phase identification using X-Ray, iron ore found in Lhoong, Regency Aceh Besar is dominated by the compound Fe$_2$O$_3$ as the main phase and SiO2 as the minor phase. Likewise, with iron sand samples, the most dominant main phase is Fe$_3$O$_4$ The grain size is getting smaller after the precipitation process.

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