SYNTHESIS AND CHARACTERIZATION OF HEMATITE (Fe₂O₃) OF IRON ORE AND MAGNETITE (Fe₃O₄) 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 NH₄OH then dried in the temperature of 150 ºC and calcinated at a temperature of 500 ºC. 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 Fe₂O₃ and followed by compounds SiO₂ (2.10%). While the iron sand contains 81.42% of Fe₃O₄ and 2.5% of SiO₂. After the precipitation process, Fe₂O₃ compounds contained in iron ore has a content of 96.58% and Fe₃O₄ compounds contained in iron sand (86.73%). The results of XRD indicate the dominant primary phase in iron ore is hematite or Fe₂O₃, and in iron, sand is magnetite Fe₃O₄, Before the extraction process, Fe₂O₃ was 58.009 μm in size and after the process of extracting the particles was reduced to 20.950 μm. While the Fe₃O₄, 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), Fe₃O₄ (magnetite), and Fe₂O₃ (hematite), as well as several other impurities compounds such as Al₂O₃, MgO, SiO₂ 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%; SiO₂ 2.64%; Al₂O₄ 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%; SiO₂: 8.02 to 44.18%; TiO₂: 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.[3]

(a)   (b)
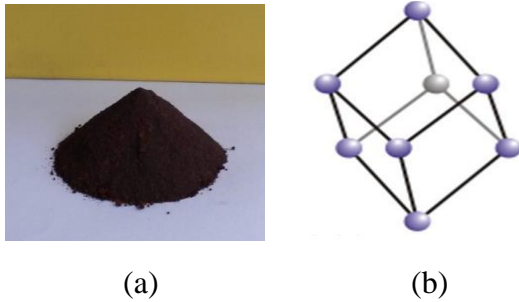
Figure 1.(a) Fe_2O_3 Powder, (b) The structure of Magnetite Fe_2O_3

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 Fe_3O_4, which is 86% and other compounds including TiO_2, SiO_2, Al_2O_3 and several compounds another minor.

(a)   (b)
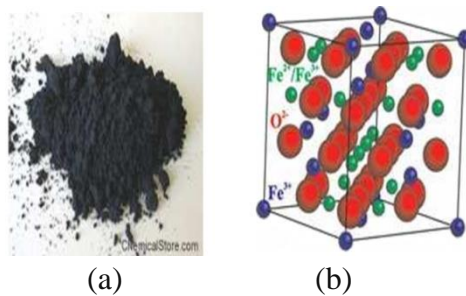
Figure 2.(a) Fe_3O_4 Powder, (b) The structure of Magnetite Fe_3O_4

Exploration of natural sand related to research on magnetite nanoparticles (Fe_3O_4) is still very little compared to iron sand exploration for raw materials; this can be used as a material for research consideration in the field of nanomaterials. In recent years magnetite nanoparticles (Fe_3O_4) have been of particular concern to experts. This happens, because the opportunities of Fe_3O_4 nanoparticles are very wide. It was reported that magnetite (Fe_3O_4) had been widely used in industrial fields such as magnetic recording media, high-density digital recording disks, magnetic fluids, data storage, MRI, Drug Delivery System, SPR Biosensors, microwave devices, magnetic sensing.[4]

Method

The basic material of Fe_2O_3 powder is iron ore taken from Lhoong, Aceh Besar, and for metal oxide Fe_3O_4 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 30,6ºC. 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 (NH_4OH) until it reaches pH 6, then stirring on a hot plate magnetic stirrer at 145ºC 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 150ºC for 19 hours and then calcined at a temperature of 500ºC 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.

The compound with the highest content contained in iron ore in the magnetic separation process was Fe_2O_3, with a composition percentage of 95.99%. They were then followed by the SiO_2 compound, which has a composition percentage of 2.10%. Then the chemical separation process (precipitation method), the results of the quantitative analysis showed that the compound of Fe_2O_3 increased to 96.58%, while the SiO_2 compound still showed the same percentage as the magnetic separation process.

The analysis process was also carried out on iron sand samples by magnetic separation and chemical separation (precipitation methods). The results of the composition of the iron sand using XRF will be shown in Table 2 below.

Table 1. Results Identification of iron ore samples

Compound	Magnetic separation (%)	Precipitation Methods (%)
Fe_2O_3	95.99	96.58
SiO_2	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 (%)
Fe_3O_4	81.42	86.73
SiO_2	4.4	2.5
Al_2O_3	3	-
P_2O_5	0.41	0.34
K_2O	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 Fe_3O_4, with a composition percentage of 81.42%. Further then followed by the TiO_2 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 Fe_3O_4 phase precipitation method showed an increase where the content of the most dominant element compared to other identified elements was Fe_3O_4 with a percentage of 86.73%. The percentage of Fe_3O_4 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_3O_4 and Fe_2O_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 2θ, lattice distance (d), intensity (I/I0), phase, and crystal structure. This identification is with an angle of 2θ 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_2O_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_3O_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_3O_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_3O_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_3O_4 at an angle of $2\theta=35.4853$.
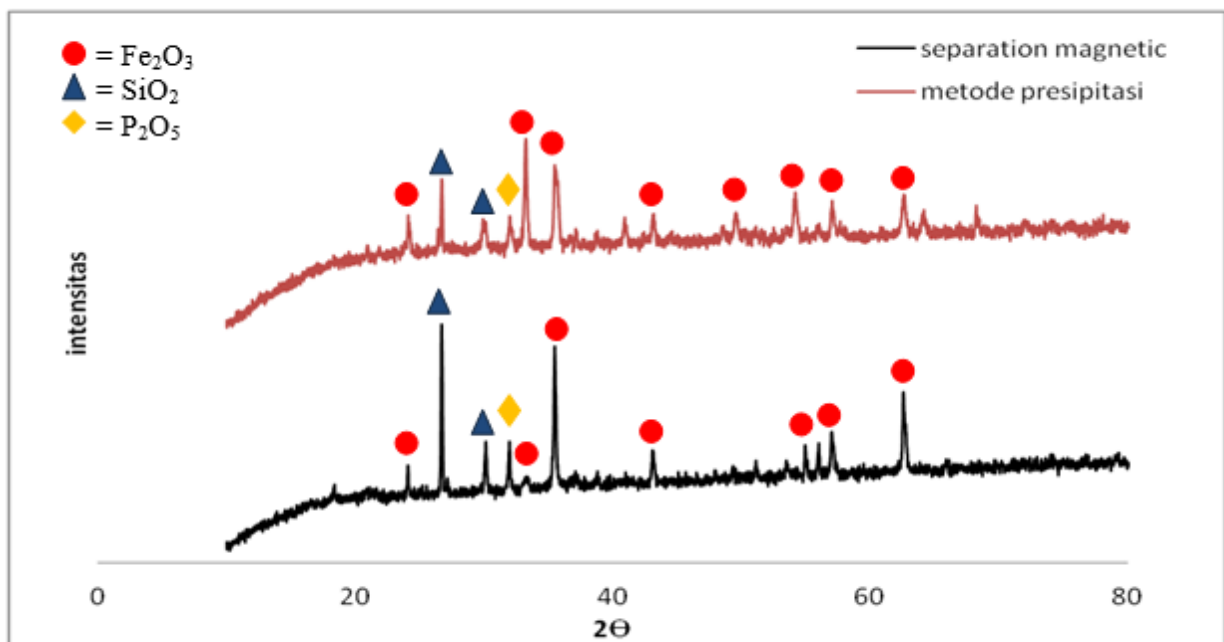
Figure 3. XRD results for iron ore magnetic separation and precipitation methods
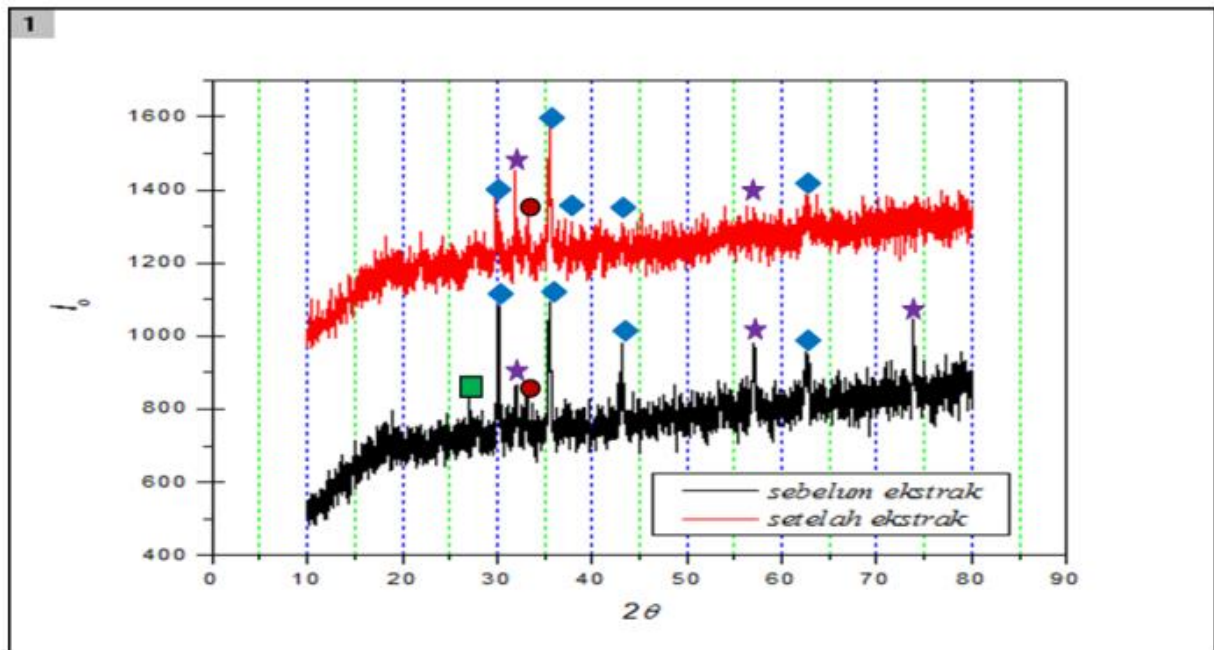
Figure 4. XRD graph results on iron sand samples before and after extraction

($\blacklozenge$ =Fe$_3$O$_4$, $\bullet$ =Fe$_2$O$_3$, $\star$ =TiO$_2$, $\blacksquare$ =SiO$_2$).[4]

Sample Size of Fe$_2$O$_3$ and Fe$_3$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 Fe$_2$O$_3$ magnetic separation process (58.009 µm) and the method of precipitation size (20,950) µm.

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

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 Fe$_2$O$_3$ crystallite size

Fe$_2$O$_3$ Sample	Parameters		
	FWHM(º)	θ (º)	Size (µm)
separation	0.15180	17.72285	58.009
precipitation	0.42040	17.7665	20.950

Table 4. Calculation of Fe_3O_4 crystallite size

Fe_3O_4 Sample	Parameters		
	FWHM(^{o})	θ (^{o})	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_2O_3 (hematite) of 95.99% and the precipitation methods containing Fe_2O_3 (hematite) of 96.58%. Whereas for iron sand samples containing Fe_3O_4 with a percentage of composition 81.42% and Fe_3O_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_2O_3 as the main phase and SiO2 as the minor phase. Likewise, with iron sand samples, the most dominant main phase is Fe_3O_4 The grain size is getting smaller after the precipitation process.

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