High purity Fe₃O₄ from Local Iron Sand Extraction

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Abstract. Indonesia has a long coastline and is rich with iron sand. The iron sand is generally rich in various elements such as iron and titanium. One of the products processing of the iron sand mineral is iron (II) (III) oxide (magnetite Fe₃O₄). The stages of purification process to extracting magnetite phase and discarding the other phases has been performed. Magnetite phase analysis of iron sand extraction retrieved from Indonesia have been investigated. The result of analysis element of iron sand shows that it consists of majority Fe around 65 wt%. However, there are still 17 impurities such as Ti, Al, Ce, Co, Cr, Eu, La, Mg, Mn, Na, Sc, Sm, Th, V, Yb, and Zn. After extraction process, Fe element content increases up to 94%. The iron sand powder after milling for 10 hours and separating using a magnetic separator, the iron sand powders are dissolved in acid chloride solution to form a solution of iron chloride, and this solution is sprinkled with sodium hydroxide to obtain fine powders of Fe₃O₄. The fine powders which formed were washed with de-mineralization water. The X-ray diffraction pattern shows that the fine powders have a single phase of Fe₃O₄. The analysis result shows that the sample has the chemical formula: Fe₃O₄ with a cubic crystal system, space group: Fd-3m and lattice parameters: a = b = c = 8.3681 (1) Å, α = β = γ = 90°. The microstructure analysis shows that the particle of Fe₃O₄ homogeneously shaped like spherical. The magnetic properties using vibrating sample magnetometer shows that Fe₃O₄ obtained have ferromagnetic behavior with soft magnetic characteristics. We concluded that this purification of iron sand had been successfully performed to obtain fine powders of Fe₃O₄ with high purity.

Keywords: Iron sand, local resources, purifying, magnetic, Fe₃O₄, and high purity

1. Introduction
In Indonesia, approximately 2 billion tons natural iron sand resources are spread along the southern coast of Java, Sumatra, West Nusa Tenggara, and so on with the main content of iron oxide, titania, silica and alumina [1]. In Indonesia, iron sand is the source of raw material of iron/steel industry [1]
and cement [2]. Also, the product of iron sand processing can be Fe$_3$O$_4$. By processing the natural resources of iron sand into Fe$_3$O$_4$, the added value can be increased several number fold and can be used for import substitute products to enhance the competitiveness of the national industry. Magnetite (Fe$_3$O$_4$) is an interesting material to develop because it is very applicable so that many researchers are interested in developing it [3-9]. Some of the treatment methods used are milling and precipitation [3], coprecipitation [4,7], precipitation and sol-gel [5], mechanical vibration [6], coprecipitation with polyethylene [8], mechanical alloying [9]. Through the Fe$_3$O$_4$ calcination process, barium hexaferrite may be produced as a microwave absorber [3], for biomedical [10], and others.

In this paper, we will report Fe$_3$O$_4$ synthesis results from the natural iron sand of the titanomagnetite type. The iron sand powder after milling for 10 hours and separating using a magnetic separator, the iron sand powders are dissolved in acid chloride solution to form a solution of iron chloride, and this solution is sprinkled with sodium hydroxide to obtain fine powders of Fe$_3$O$_4$. Therefore this study aims to obtain nanoparticle of Fe$_3$O$_4$ through the iron sand extraction.

2. Experiment

The iron sand of titanomagnetite is retrieved from Lampung, Indonesia. The purifying process of iron sand into iron oxide product in the form of high purity Fe$_3$O$_4$ is shown in Figure 1.

![Figure 1. The purifying process of iron sand into iron oxide product](image)

The iron sand of titanomagnetite was milled by using high energy milling (HEM), then dissolved in chloride acid (HCl) technical quality with the ratio of 250 g/L HCl to solve iron chloride. 5 M sodium hydroxide technical quality was added to a solution of iron chloride at a temperature of 80 °C for 1 hour. The precipitate was washed with demineralization water until pH of 7 with the purpose of disappearing the residual salts, then the precipitate was separated from the solvent using a permanent magnet and after that dried in an oven at a temperature of 100 °C. The dry powder sample was re-milled and then heated at a temperature of 400 °C in a furnace for 5 hours, so that obtained a Fe$_3$O$_4$ powder.
The Fe$_3$O$_4$ powder was analyzed by X-ray diffractometer (XRD) of Pan Analytical product with CuK$\alpha$ radiation ($\lambda = 1.5406 \ \text{Å}$). The qualitative and quantitative analysis of phase was carried out by GSAS program. The particle morphology of the Fe$_3$O$_4$ powders was observed by the scanning electron microscope (SEM) using JED 2300 JEOL. The elemental composition was analyzed by using energy dispersive spectroscopy (EDS). Finally, the magnetic properties were measured by using vibrating sample magnetometer (VSM) of Oxford product.

3. Result and discussion

Figure 2 shows the result of microstructure observation on iron sands sample of titanomagnetite type extracted by using scanning electron microscope.

![Figure 2. The surface morphology of iron sand samples of titanomagnetite type results from the extraction process.](image1)

![Figure 3. The elementary analysis using energy dispersive spectroscopy.](image2)

In Figure 2 it appears that the surface morphological observations show that the samples have the same form and color, although there is still a wide particle of about 1.8-10 $\mu$m. It is assumed that the sample has a homogeneous phase. However, photographs of these surface morphologies also cannot determine the phases contained in the titanomagnetite iron sand samples of these extractions. For the analysis of the existing phases in the iron sand, x-ray diffraction is used, while for the analysis of the constituent elements, energy dispersive spectroscopy is used. Figure 3 shows the result of elementary analysis using energy dispersive spectroscopy on titanomagnetite iron sand sample. The resulting energy spectrum as shown in Fig. 3 shows that the most dominant elements are oxygen (O) and iron (Fe) which are consecutively at 0.525 keV and 6.398 keV energy with K-wavelength. This means that the extraction of titanomagnetite iron sand sample has successfully separated the iron (Fe) element from other impurities. In detail, the element content present in the iron sands of this type of titanomagnetite is shown in Table 1.

In Table 1 it appears that the Fe content of total iron sand samples of titanomagnetite type of this extraction is 79.12%. These results indicate that the iron content in the titanomagnetite iron sand sample has been successfully performed.
**Table 1.** The result of elementary analysis using energy dispersive spectroscopy

| No. | Element    | Content (% weight) |
|-----|------------|--------------------|
| 1.  | Iron (Fe)  | 79.12 ± 0.21       |
| 2.  | Titanium (Ti) | 1.32 ± 0.11       |
| 3.  | Magnesium (Mg) | 0.66 ± 0.11       |
| 4.  | Aluminum (Al) | 1.10 ± 0.09       |
| 5.  | Carbon (C)  | 1.62 ± 0.06       |
| 6.  | Oxygen (O)  | 16.18 ± 0.11      |

Figure 4 shows the results of the measurement and structural analysis that can identify the phase of the x-ray diffraction pattern of titanomagnetite iron sand samples extracted.

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**Figure 4.** The X-ray diffraction pattern shows that the fine powders have a single phase of Fe$_3$O$_4$.

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Figure 4 shows that there have been formed Bragg diffraction peaks which are suspected to have been single phase. The phase identification refers to JCPDS number 19-629, i.e. the magnetite phase with the cubic crystal structure and lattice parameters: $a = b = c = 8.3681$ (1) Å. Similar results were obtained by Sunaryono et al. [4] and also Rusianto et al. [6]. Thus, based on the results of the analysis of the x-ray diffraction pattern, the amount of phase content contained in the iron titanomagnetite iron extraction is shown in Table 2.

**Table 2.** The phase mass fraction contained in the iron sands sample of the titanomagnetite type of extraction

| No. | Mineral     | Empirical formula | Mass fraction (%) |
|-----|-------------|-------------------|------------------|
| 1.  | Magnetite   | Fe$_3$O$_4$       | 100              |
Figure 5 shows the magnetic properties of iron sand samples of titanomagnetite extracted species. The relationship between $M$ and $H$ is not linear. This indicates that the sample of titanomagnetite iron sand of this extract has ferromagnetic properties. When the magnetic field intensity of $H$ is increased to reach $H = 1$ Tesla, the value of $M$ has a saturation point called saturation magnetization $Ms$ around 43 emu/g; then when the magnetic field $H$ is lowered, the curve path does not return through the original curve. At the price of $H = 0$, the magnetization of $M$ has 18.5 emu/g. This condition is called remanent magnetization $Mr$: Furthermore, the intensity value of the magnetic field $H$ is lowered (negative value), the $M$ curve will intersect the axis of 226 Oe magnetic field denoted as $Hc$. This $Hc$ intensity is what is required to make the flux density $B = 0$ or eliminate the flux in the material. The intensity of the $Hc$ magnet is called the coercivity of the material or the coercivity field. Based on this hysteresis curve, the titanomagnetite iron sand sample of this extraction is a magnetic material.

4. Conclusion
The samples $\text{Fe}_3\text{O}_4$ have been successfully extracted by coprecipitation method from local iron sand. The sample has a homogeneous phase. However, photographs of these surface morphologies also can not determine the phases contained in the titanomagnetite iron sand samples of these extractions. The resulting EDS shows that the most dominant elements are oxygen (O) and iron (Fe). The XRD data show that the sample has a single phase with the structure of spinel ferrite particles. The functional group's analysis by using FTIR shows that transmittance peaks of the iron sand extraction appeared at wave numbers around 600 cm$^{-1}$ that indicated the presence of the Fe–O bond vibrations. The hysteresis loop shows that all of the samples have the ferromagnetic behavior with a relatively small of coercivity value.

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