



MAGNETIC BEHAVIOR IDENTIFICATION OF HIGH-PURITY HEMATITE (Fe_2O_3) EXTRACTED FROM IRON ORE BY CO-PRECIPITATION METHOD

Fauzi^{*1}, Zulfalina¹, Malahayati¹, M. Nizar Machmud², and Zulkarnain Jalil^{1,3}

¹ Department of Physics, Syiah Kuala University, Banda Aceh 23111, Indonesia

² Department of Mechanical Engineering, Syiah Kuala University, Banda Aceh 23111, Indonesia

³ Graduate School of Physics, Syiah Kuala University, Banda Aceh 23111, Indonesia

*fauzi@usk.ac.id

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ABSTRACT

Magnetic mineral identification was successfully carried out on the iron ore from Manggamat in South Aceh Regency, Indonesia. This initial study was conducted in an attempt to increase the added value of local minerals. This study was performed by using co-precipitation method combined with mechanical milling and analyzed using X-ray diffraction and fluorescence (XRD, XRF) and the magnetic properties evaluated by Permagraph. These investigations indicate that the dominant phase in the iron ore is hematite (Fe_2O_3), showed a content of 85.31%, followed by SiO_2 compounds (11.01%), and the lowest concentration was SnO_2 (0.02 %). Furthermore, observation of the magnetic properties showed that the magnetic saturation (M_s) of 0.1 Tesla, then residual magnetization or remanent (B_r) of 0.017 Tesla, and a reverse magnetic field or coercivity (H_c) of 12.39 kA/m. As the milling time increased, the magnetic saturation and remanent field decreased, whereas the coercive field increased. This characteristic is known as the superparamagnetic behavior which widely used in biomedical application.

Keywords: Iron ore; hematite; magnetic properties; co-precipitation; milling; Aceh Province

INTRODUCTION

The world's hematite industry is developing rapidly due to the increasing demand from various industries of daily life; electronics, chemicals, foundry, ceramics, paints, sand paper and magnetic materials industries. Hematite (Fe_2O_3) is one of the compounds present in iron ores. It has a heavy and relatively hard oxide mineral that constitutes the most important iron ore owing to its high iron content and abundance ^[1].

In Indonesia, Aceh Province is known as one of the areas which has large deposits of iron ore minerals. It is scattered across several places such as Aceh Besar, Pidie, Southwest Aceh, Subulussalam, Gayo Lues, East Aceh, and South Aceh with a total deposit reached 92.3 million tons ^[2]. Some are still in the form of rocks and some more in the form of placer deposits in rivers or in an estuary. However, its use is still not optimal due to the lack of intensive research in this area; therefore, the iron ore in Aceh is currently being exploited without detailed investigative effort, implying a lack of improvement in the added value of the

mineral itself. To date, studies on Aceh's iron ore mineral content and further purification efforts are still very rare.

In this study, hematite purification was carried out using a co-precipitation method. Co-precipitation is known to be the most efficient method for increasing the percentage of Fe_2O_3 and removing metal impurities^[3-4]. It involves removal of harmful solids and impurities using physical and chemical treatment methods. Furthermore, the magnetic properties will be evaluated with a focus on the superparamagnetic behavior. This finding is interesting, given the widely recognized applications of superparamagnetic materials. Thus, the results of Aceh's hematite iron ore study will be used by policy makers for further utilization as an industrial raw material that can add value to the regional economy.

MATERIALS AND METHOD

Sampling was conducted in the iron ore mining area of Manggamat, South Aceh Regency, Indonesia. The iron ore stone was manually broken into small chunks using a hammer. The chunks were washed to remove adhering impurities. The samples were then crushed with a mortar and pestle and sieved (sieving). Then, the iron ore was dissolved in HCl and heated to 145 °C using a magnetic stirrer at 350 rpm. Precipitation was performed by dripping 25% NH_4OH into the solution until a precipitate formed. The precipitate was then washed and dried in an oven at 150 °C for 19 h. The steps are illustrated in Fig. 1.

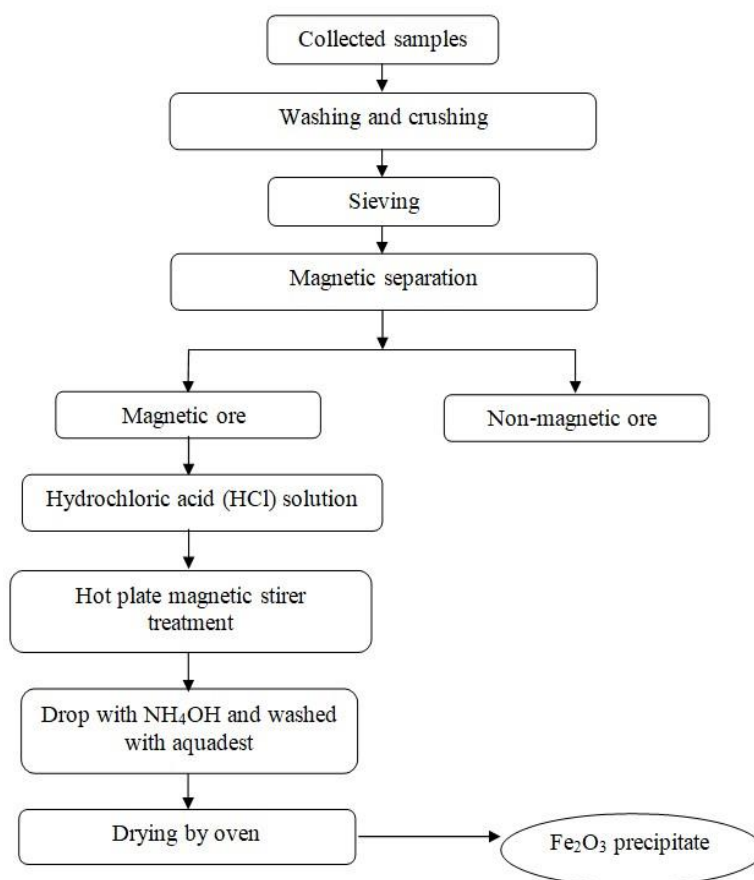


Figure 1. Hematite extraction process of Manggamat iron ore mineral

Furthermore, calcination is carried out at 500°C (2 hours). To obtain a fine powder, mechanical alloying techniques were performed using a high energy ball mill (Fritsch, P6). The ball-to-powder ratio (BPR) was 10:1, and the powder was milled for 20 h at a rotational speed of 450 rpm. Phase composition studies were performed using an X-ray diffractometer (Shimadzu D-6000, Cu-K α radiation). The elemental content was determined using X-ray Fluorescence (XRF, Brucker S Ranger). The particle size analyzer (PSA) was obtained to find the distribution of the iron ore after milling. Furthermore, the powder morphology was examined using a scanning electron microscope (SEM, JEOL JSM-5310LV). Finally, the magnetic characteristics were analyzed using a Permagraph (MagnetPhysik, 2T). For the magnetic test, the iron ore powder was first compacted into a pellet form using a hydraulic press. The magnetic properties observed were the magnetic saturation (Ms), remanence (Br), and coercivity (Hc). For hydrogen storage performance test was using the differential scanning calorimetry (DSC, Shimadzu-60).

RESULTS AND DISCUSSION

The XRD observation was using a XRD Shimadzu D6000 with Cu-K α radiation ($\lambda = 1.54060$ Å) at angle of 2θ from 20°-80°. XRD test aims to identify the phase composition minerals in Manggamat iron ore after milling process. The diffraction pattern is as shown in Figure 1. From the results, there are several dominant peaks (Figure 2). By comparing to the database (JCPDS, PDF # 840311) identified that the main phases were Fe₂O₃ (hematite), Fe₃O₄ (magnetite) and SiO₂ as minor phase. If refer to the literature, this hematite phase is the dominant phase inside iron ore rock^[5-6].

The qualitative analysis of the Manggamat iron ore shown that there are two phase dominantly, hematite (Fe₂O₃) dan magnetite (Fe₃O₄). This informs that the resulting material will be magnetic ^[7]. The measurement results of the particle size analyzer (PSA) obtained the distribution of the iron ore particle size to the volume fraction for the iron ore material shown in the following Figure 2a. It can be seen that visually the particle size morphology corresponds to the SEM results, which is in the range of less than 20 μ m.

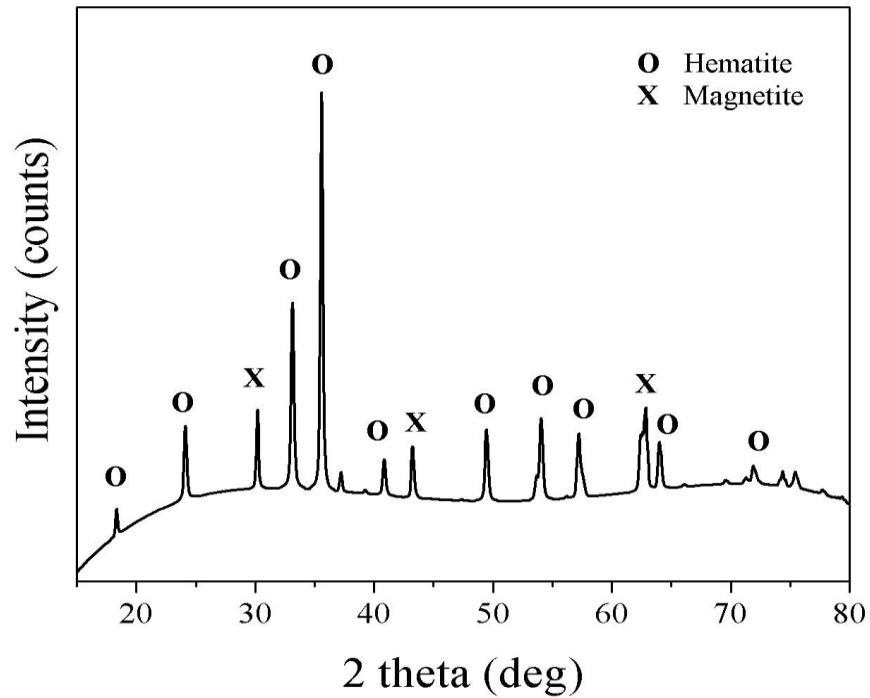


Figure 2. XRD profile of Manggamat Iron Ore

The following statistical data processing of iron composite particle size after milling shows that the mean particle size is $8.177 \mu\text{m}$. The results of this measurement also inform that the largest particle size distribution occurs at particle sizes less than $10 \mu\text{m}$, namely 71% for the volume fraction of the total (Figure 3). Meanwhile, the particle size less than $1 \mu\text{m}$ was obtained 13.8%. These results confirm that the measured iron ore is still on the micrometer scale [8].

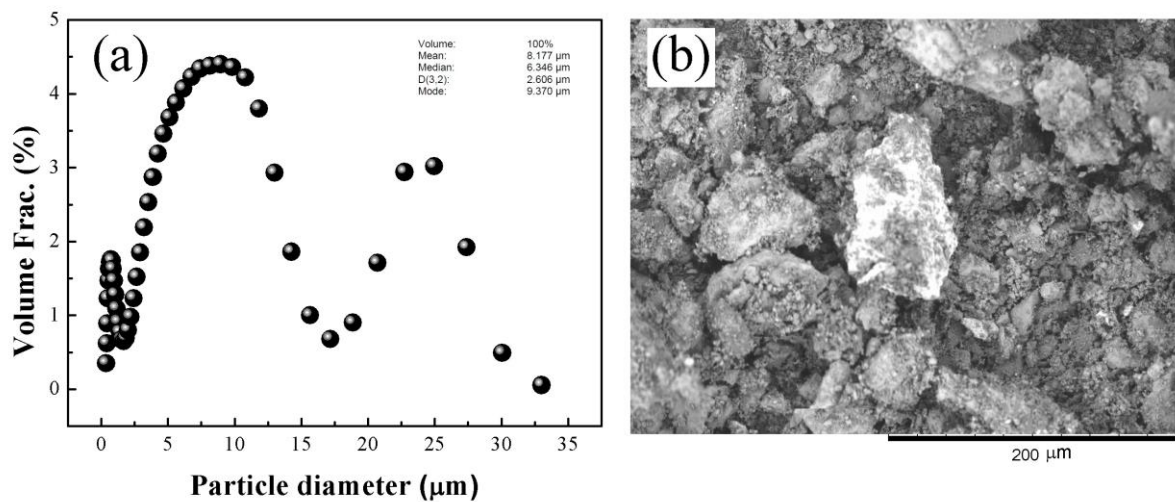


Figure 3. (a) Particle size distribution and (b) SEM photo of iron sand material

Characterization by X-ray method fluorescence (XRF) was performed for mineral content observation of the iron ore rocks. The results of XRF characterization of ore samples shows the several elements contained inside it. It found that the highest concentration was hematite (Fe_2O_3 , 85.31%), then followed by the silica (SiO_2 , 11.01%) and the lowest concentration is SnO_2 is 0.02%.

The magnetic properties characterization of iron ore after milling was done using Permagraph with an external magnetic field equal to 2 Tesla. The data obtained is in the form of hysteresis loop curve (Figure 4). From the curve is obtained by several magnitudes magnetic matters, including magnetic saturation (M_s), residual or remanent magnetization (B_r), and reverse magnetic field or coercivity (H_c). These results indicate that iron ore Manggamat, which has the majority phase Fe_2O_3 , has a magnetic phase with a value certain. This is evidenced from the results measurement of magnetic properties where obtained saturation magnetization (M_s) of 0.1 Tesla, then residual magnetization or remanent (B_r) = 0.017 T, and coercivity (H_c) of 12.39 kA/m.

The Manggamat iron ore shows the prospect and possibility to be as future materials for magnetic applications in the large scale. This can be applied after doing some more extended separation and purification route by using a comprehensive and integrated works [9-10]. The hysteresis loops of the samples show that both samples have magnetic properties. However, the sample results after magnetic separation had a higher magnetic saturation value (M_s), and the remnant values (B_r) and coercivity (H_c) tended to remain constant. This was caused by the increase in the mass fraction of the magnetic phase, thereby increasing the value of M_s [11-12].

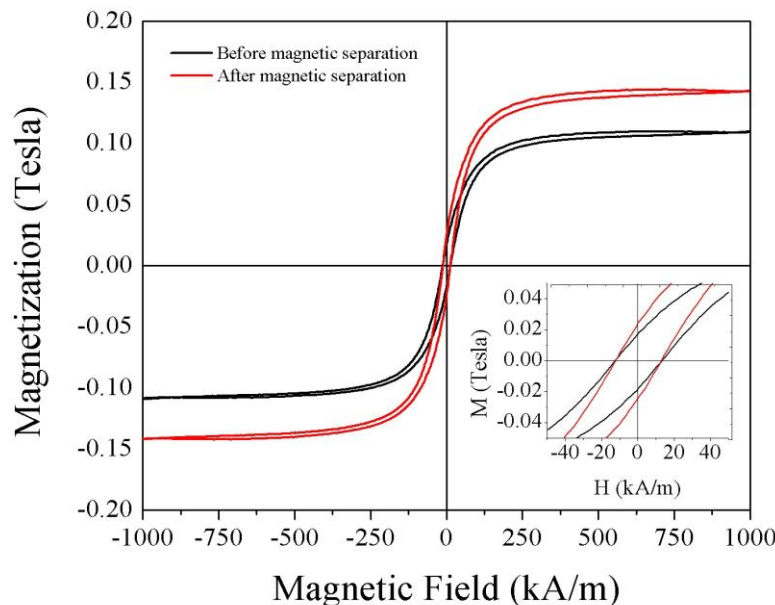


Figure 4. Hysteresis loop of iron ore before (-) and after magnetic separation (-).

These properties are known as nanomaterial characteristics [13-14] as the sample reaches a scale of ≤ 100 nm. On the large value of iron ore, their respective magnets magnetic saturation (M_s) is 0.1 tesla, then the remanent is 0.026 tesla, and has a slightly higher coercivity which is 15.7

kA / m. To obtain better results, it is necessary to carry out further studies, especially the iron ore powder preparation technique. The simplest method involves magnetic separation, and the process is followed by roasting at a certain temperature.

The magnetic property observations (summarized in Table 1) show the role of high-energy planetary milling in the experiment. As the milling time increased, the magnetic saturation and remanent field decreased, whereas the coercive field increased. This characteristic is known as super-paramagnetic behavior^[15-16].

Table 1. Magnetic properties of hematite after milling process

Treatment	Magnetic Properties		
	Ms (T)	Hc (kA/m)	Br (T)
Before separation	0.016	12.39	0.017
After separation	0.022	12.23	0.024

The magnetization of the small particles was found to be slightly larger than the bulk value. This difference was caused by an uncompensated magnetic moment. The presence of two superparamagnetic relaxation modes would be expected for hematite nanoparticles if their magnetic structure is similar to that of bulk hematite^[17-18]. The using of ferric salt solutions is sensitive for small changes in the reaction condition, which results in large variations of the average particle size and shape. As normal for hematite, the behavior of the produced sub-rounded hematite particles changed from antiferromagnetic to paramagnetic, when the particle size decreased to the nanometer scale^[19-22].

The mechanical milling method itself is a more practical method for producing nano-sized materials that can be developed on a large scale. Almost all types of metal and ceramic materials can be refined using the milling method^[23-24]. Apart from reducing the particle size, also improves the surface structure. During the high-energy ball milling process, the powder particles were subjected to a high-energy impact. The fine mixing of the powders has decreased the diffusion space to the micrometer range^[25-27]. Finally, the microstructure of the particles appeared to be more homogenous at the microscopic scale than at the initial stage.

CONCLUSION

The iron ore located in the South Aceh district, Indonesia, is dominated by hematite (Fe_2O_3) as the main phase and SiO_2 as the minor phase. Mineral identification using XRF revealed that hematite was dominant with a content of 85.31%, followed by SiO_2 (11.01 %) and SnO_2 (0.02 %). The magnetic properties obtained its magnetic magnitude, that is, saturation magnetization (Ms) of 0.1 Tesla, then residual or remanent magnetization (Br) of 0.017 Tesla, and magnetic field reverse or coercivity (Hc) of 12.39 kA/m. As the milling time increased, the magnetic saturation and remanent field decreased, whereas the coercive field increased. This characteristic is known as super-paramagnetic behavior.

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