



CHARACTERISTICS OF NATURAL MAGNETITE (Fe_3O_4) FROM BEACH SAND AS CATALYST APPLICATION IN MATERIALS INDUSTRY

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Abstract. An identification of magnetite nanoparticles synthesized from natural iron sand using co-precipitation method has been conducted. The treatment was undertaken at room temperature and the heating used a pair of acid-base compounds, namely HCL as a solvent and NH_4OH as a precipitate. Crystal structures, percentages of elements, particle sizes and magnetic characteristics of the materials were characterized by testing XRF, XRD and Permagraph. The results were then compared with the commercial material purchased from Aldrich (with 97% purity). From the results, it was found that the percentage value of the purity of Fe_3O_4 derived from natural sand before the extraction was 81.42%, and after the extraction it increased to 86.73%. Furthermore, the saturation magnetization (M_s) value for Fe_3O_4 ferrite from iron sand was 0.29 T, the residual magnetization or Remanen (Br) was 0.081 T, and Coersivity (Hc) was 1.82 kA/m.

Keywords: Natural Magnetite, Fe_3O_4 , nanoparticles, beach sand, catalyst

I. INTRODUCTION

Aceh is one of the provinces in Indonesia that has large natural resources. There are abundant natural resources and important natural materials in Aceh such as gold, coal, iron ore, iron sand and so on. Generally, the natural resources are still in raw form and most of them have not been found and the deposits are unknown. Along with the development of industry, it automatically increases the demand of certain raw materials for certain products. The synthesis of Fe_3O_4 nanoparticles in the past few years has been carried out by researchers using different methods, for example the one with a co-precipitation method [3]. The same method has also been developed as stated in Ref. [8]. Among the synthesis methods, the co-precipitation method is the simplest method because the treatment process is easier to do and requires a low reaction temperature ($<100^\circ\text{C}$). Some information that can be collected from surveys on various process conditions used to control the size of Fe_3O_4 particles is shown in Table 1. The most widely used iron salts are FeCl_2 and FeCl_3 , and the

precipitate agents in general are NaOH or NH_4OH , but there are also ones using Tetramethyl Ammonium Hydroxide (TMAOH). The reaction temperatures are ranging from room temperature to 100°C with the diameter of nanoparticles of 2 to 51 nm [5]. In this study, the structures and magnetic characteristics of an iron sand are studied. The material was collected from the coastal area of Syiah Kuala, Banda Aceh. It then will be compared with the synthetic material of Aldrich Fe_3O_4 . The information relating with the results will be useful to enhance its economic values and for additional database information for the local area.

II. METHODOLOGY

The materials in this study consisted of main material of pure magnetite Fe_3O_4 (97%, size 50-100 nm, Aldrich sigma production) and Fe_3O_4 iron oxide powder from iron sand collected from Syiah Kuala beach in Aceh. The iron oxide powder had previously undergone a good extraction through magnetite separation and chemical treatment using the co-precipitation

method. Characteristics of the material were determined by using XRF (type S2 Bruker, Stranger). Iron sand for testing the samples did not need undergo a special treatment so it could be tested directly by placing the sample in the sample holder. Argon gas was used for operation medium. Data was obtained in the form of weight percentage (wt%) which informs the percentage of mineral contents within iron sand.

Table 1. The results of the synthesis of Fe_3O_4 by co-precipitation method [5].

Source Fe^{2+}	Source Fe^{3+}	Precipitation	Temp. ($^{\circ}C$)	Diam. (nm)
$FeCl_2$	$FeCl_3$	NaOH	RT and 80	6 and 12
$FeCl_2$	$FeCl_3$	Na_4OH	RT	12
$FeCl_2$	$FeCl_3$	Na_4OH	80	3 to15
$FeCl_2$	$FeCl_3$	NaOH	ns	8.5
$FeCl_2$	Ns	NaOH	88	7
$FeSO_4$	$FeCl_3$	Na_4OH	ns	7.5
ns	Ns	NaOH	80	5.5 and 12.5
$FeCl_2$	$FeCl_3$	Na_4OH	RT	3 to11
$FeCl_2$	$FeCl_3$	Na_4OH	30-90	8.4 to 51
$FeSO_4$	$FeCl_3$	NaOH	HT	11.8
$FeCl_2$	$FeCl_3$	NaOH	70	Ns
$FeCl_2$	Ns	NaOH	25 and 45	2 to 12.5
		Na_4OH		
		$N(CH_3)_4$		
		OH		

RT= Room Temperature, ns = not specified, HT = heated up

To determine the composition of phase percentages, qualitative identification was carried out using X-Ray diffraction (XRD) (Philips PW 3710 diffractometer, Co- $K\alpha$ radiation ($\alpha = 1.78896 \text{ \AA}$)). The equipment to characterize magnetic properties was Permagraph (Magnet Physik, Germany) with external magnetic field 2 T. The main materials in this research were the iron sand from Syiah Kuala beach, HCL, NaOH, and distilled water. Iron sand was dissolved in HCL 100 ml and it was filtered through filter

paper. NaOH 75 ml was slowly added in to the filtered solution through titration process. Separated deposits with the remainder of the titration with distilled water and the sediment were drained using a magnetic stirrer. Afterward, it was dried in the Magnetite sintering at temperature of $70^{\circ}C$ for 5 h.

III. RESULTS AND DISCUSSIONS

X-Ray Fluorescence result after extraction using Fe_3O_4 phase co-precipitation method showed an increase with the most dominant element content compared to other identified elements was Fe_3O_4 with percentage of $86.73 \pm 0.35\%$. The percentage of Fe_3O_4 that was identified was almost 90%. It showed that the natural material from iron sand had more dominant magnetite content than other compounds.

Table 2. Results of identification of XRF iron sand

Magnetic Separation method		Co-precipitation method	
Compound	(%)	Compound	(%)
Fe_3O_4	81.42 ± 1.23	Fe_3O_4	86.73 ± 0.35
SiO_2	4.4 ± 0.2	SiO_2	2.5 ± 0.02
Al_2O_3	3 ± 0.9	Al_2O_3	0
P_2O_5	0.41 ± 0.05	P_2O_5	0.34 ± 0.08
K_2O	0.11 ± 0.005	K_2O	0.075 ± 0.004
CaO	1.04 ± 0.0095	CaO	0.72 ± 0.01
TiO_2	7.61 ± 0.100	TiO_2	8.93 ± 0.02
V_2O_5	0.57 ± 0.004	V_2O_5	0.57 ± 0.007
Cr_2O_3	0.19 ± 0.005	Cr_2O_3	0.22 ± 0.006
ZnO	0.06 ± 0.003	ZnO	0.06 ± 0.003
Bi_2O_3	0.61 ± 0.01	Bi_2O_3	0.23 ± 0.0003
Re_2O_7	0.3 ± 0.02	Re_2O_7	0.2 ± 0.01
Eu_2O_3	0.55 ± 0.03	Eu_2O_3	0.60 ± 0.05

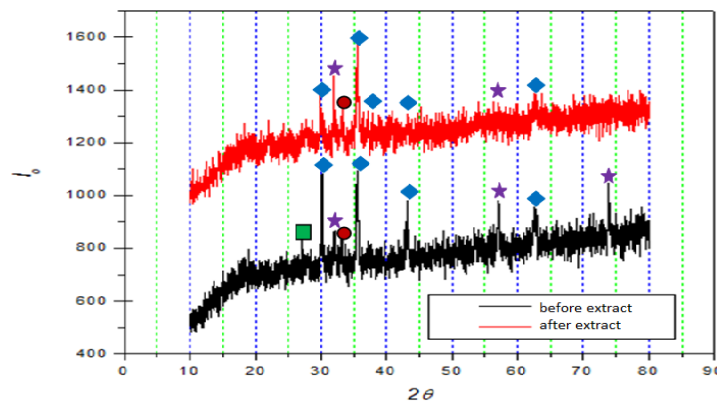


Figure 1. Graph of Xrd Fe_3O_4 from iron sand (◆ = Fe_3O_4 ● = Fe_2O_3 ☆ = TiO_2 ■ = SiO_2)

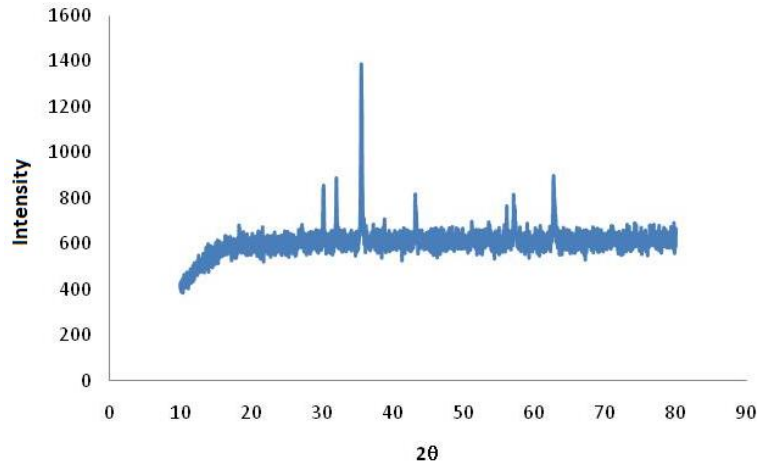


Figure 2. Chart of XRD for Fe_3O_4 from Aldrich

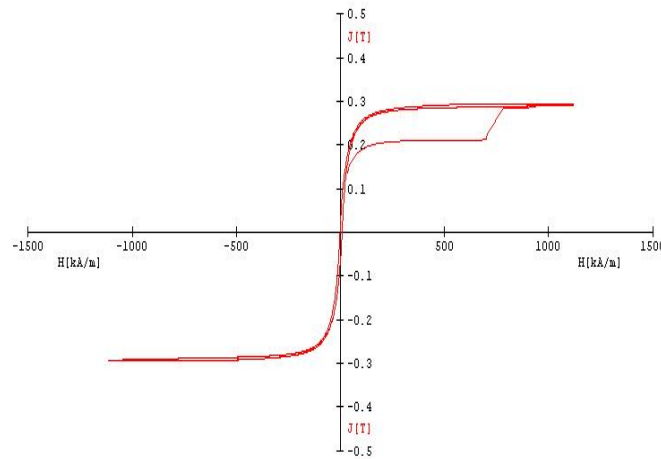


Figure 3. The hysteresis loop curve samples of iron sand

The percentage of Aldrich Fe_3O_4 which was used as the comparison was 97%. These two samples would be tested with X-rays and Permagraphs to distinguish the characteristics between Aldrich Fe_3O_4 and Fe_3O_4 from natural sand. Widths of the peak of each sample graph (Figure 1) shows the number of X-rays scattered in the same field of dhkl. It represents that the higher the diffraction peak is, the more the X-rays scattered in the same field of dhkl. The width and height of the XRD chart identify the particle size of the samples. This can be seen from XRD on Fe_3O_4 from iron sand which had not been synthesized in order to experience widening of the graph after synthesizing. The same thing happened to the Fe_3O_4 from Aldrich which had not been synthesized, it had larger FWHM than the synthesized one. Thus, the materials which had been synthesized by the co-precipitation method had more nanocrystalline characteristics than those which had not been synthesized. The X-ray

result of Aldrich Fe_3O_4 in Figure 2 shows that there was a Fe_3O_4 phase and no other phase was found. The suitable data for search was JCPDS from Fe_3O_4 (#19-0629). All peaks of the diffraction patterns for the sample were identified as Fe_3O_4 phases with cubic structures with a lattice constant of 8.335 Å, which was close to ICSD data No. 82237 ($\alpha = 8, 3873$ Å). The results of magnetic characteristics analysis for Fe_3O_4 ferrite from iron sand included the Saturation Magnetization (Ms) value of 0.29 T, residual magnetization or Remanent (Br) of 0.081 T, and Coersivity (Hc) of 1.82 kA/m. In Figure 3, it shows that the curve formed is a type of soft magnetic (soft magnet). Weak saturation magnetization was caused by non-uniformity in particle size and uneven distribution which was clumping or agglomeration. The size of the coercive field was affected by the particle size and agglomeration which means that the larger the particle size, the greater the coercive size.

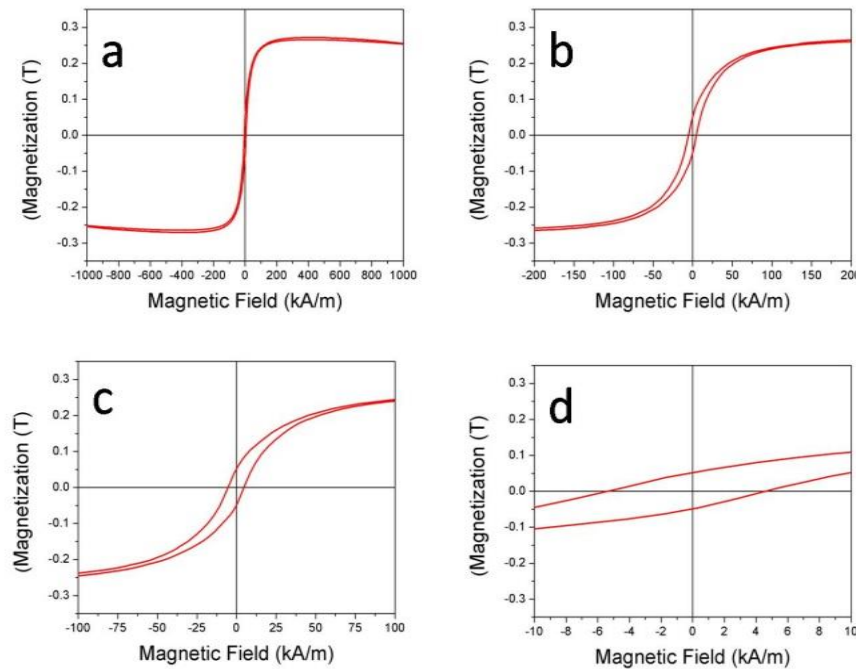


Figure 4. Hysteresis curve of Aldrich Fe_3O_4 powder (a) shows the overall results of the curve hysteresis, (b) and (c) show change in the shape of the curve hysteresis with shrunk scale on the X-axis as the outer magnetic field value (H_c), and (d) shows the value of (M_r).

The results of measurements of Fe_3O_4 can be observed in Figure 4. Figure 4(a) shows the overall results of the curve hysteresis of Aldrich Fe_3O_4 sample. The hysteresis curve shows known properties of the saturation magnetization (M_s), magnetization time (M_r), and the terrain coersivity (H_c). From the figure 4(a) can be observed that the value of M_s is around 2.5 T. Figure 4(b) and 4(c) shows changes in the shape of the hysteresis with shrunk scale on the X-axis i.e. outer magnetic field value (H_c). However, from the second image, it is not yet determined the value of M_r and H_c . By shrinking the scale of the X-axis as in the Figure 4(d), it can be observed that the value of M_r amounting about 0.75 T and H_c is 4.5 kA/m, respectively.

CONCLUSION

From the analysis of the obtained results, the percentage value of the purity of Fe_3O_4 derived from natural sand before extraction was 81.42%, and after the extraction it was increased to 86.73%. The purity of Aldrich Fe_3O_4 material was 97%. Meanwhile the analysis of magnetic characteristics of Fe_3O_4 ferrite from iron sand gave the Saturation Magnetization (M_s) value of 0.29 T, residual magnetization or Remanen (Br)

of 0.081 T, and Coersivity (H_c) of 1.82 kA/m. For Aldrich Fe_3O_4 ferrite, it was found that the M_s value was around 2.5 T and M_r value was about 0.75 T and H_c 4.5 kA/m.

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