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The effect of FeMn and FeB additive on hematite (α-Fe₂O₃) and their characterization

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Abstract. In this study, the mineral material addition of FeMn and FeB to the hematite (α-Fe₂O₃) by varying each mass concentration of 0, 4, and 8 wt.% have been prepared by powder metallurgy method. The mixing process of the powder was done by high energy milling (HEM) for 1 h. Then, the powders were calcined at temperature of 1000 ºC for 2 h. The Effect of FeMn and FeB additives on α-Fe₂O₃ causes the increasing of the particles diameter. The increasing of FeMn additive increases the powder density of material, but the addition of FeB decreases it. The material of α-Fe₂O₃ with FeMn additive has dominant phases of α-Fe₂O₃ and the minor phases of MnO₂ and Fe₃O₄. The material of α-Fe₂O₃ with FeB additive has two phases as magnetite (Fe₃O₄) and hematite (α-Fe₂O₃). The magnetic properties of a α-Fe₂O₃ material with FeMn and FeB additives decrease the saturation value and coercivity.

1. Introduction

Material for magnetic is important because it is widely applied in the industries, including automotive. Indonesia has an abundant source of raw materials; one of them is iron sand. According to Widanarto [1] the form of iron sand that naturally occurs in mineral is ferrous ferrite (Fe₃O₄). By the heating processes at elevated temperatures, the resulting of magnetite phase (Fe₃O₄) changed into hematite (α-Fe₂O₃) [2]. This change is followed by the changes in atomic arrangement and its magnetic properties [3]. Hematite (α-Fe₂O₃) materials tend to stable at high temperature in comparison with magnetite (Fe₃O₄) [4]. Therefore hematite is more often used as a raw material for synthesizing ferrite magnets [5]. The ferrite magnet has strong mechanical properties, not easily corrode, high electromagnetic performance, excellent chemical stability, low coercivity, and moderate saturation magnetization [6, 7]. The hematite material (α-Fe₂O₃) is an oxide that is widely used in various applications. Some applications are gas sensors, catalyst agents, lithium ion batteries [8], photoelectrochemical [9], biomedical devices, pigments, [10] and many others. Several methods are used for the preparation of α-Fe₂O₃ such as sol gel, force hydrolysis, microemulsion, precipitation, direct oxidation, thermal decomposition, sonochemical, hydrothermal, solvothermal, and electrochemical [11, 12].

Ferro boron (FeB) is used as an additive for steel and iron because it can improve material mechanical properties and also the price is relatively cheap. Recently, FeB attract attention as a magnetic additive, one of which is the manufacture of NdFeB permanent magnets. FeB is a metal alloy composed of iron and boron with boron content ranging from 10 to 20%. Iron is widely used in the production of permanent magnets due to its high magnetic properties, while boron is hard and diamagnetic.

Ferro manganese (FeMn) has cubic structure and includes as soft magnetic. Manganese has a high melting temperature which is $1244°C$, so it can retain at high temperature of such heat treatment. Therefore, the addition of FeMn into barium hexaferrite can decrease its magnetic properties.

In the previous study, Shen et al. [13] modified the nanocomposite Fe_3O_4-FeB and produced lower magnetic properties than Fe_3O_4. In other research, Lee [14] also synthesized Mn doped Fe_2O_3 and the magnetic properties decrease with increasing Mn. In this preliminary research, we apply FeM and FeB as doping to determine the properties within soft magnetic. FeM and FeB is aplied due to have low magnetic properties. From this study, with addition of FeM and FeB into Fe_2O_3, it is expected to produce nanocomposite Fe_2O_3 with lower magnetic properties. The α-Fe_2O_3 is synthesized into pellet with the addition of iron manganese and iron boron (FeB) was carried out by powder metallurgy method and followed by the calcination process. Powder metallurgy is considered due to its easy and large scale applications.

2. Experimental Methods

FeMn and FeB additives were raw materials which were obtained from the nature with bulk form. Then, it was ground into powders to pass 200 mesh sieve. Furthermore, each of FeMn and FeB powders were added to the main raw material of α-Fe_2O_3 and milled using the high energy milling (HEM) shaker mill PPF-UG for 1 h. The variation of FeMn and FeB additives were 4 and 8 wt% which were added to hematite powder (α-Fe_2O_3). After milling, the powder was measured to find the powder density by pycnometer and its particle distribution was analyzed using the particle size analyzer (PSA). The powders were calcined at $1000°C$ for 2 h. Then the microstructure was analyzed by the X-ray diffraction (XRD - Rigaku SmartLab, Cu-kα 1.5406). While the magnetic properties were analyzed by using the vibrating sample magnetometer (VSM - Electromagnetic, 250).

3. Results and Discussion

The particle size distribution of α-Fe_2O_3 is shown in figure 1. While the distribution analysis and particle size of α-Fe_2O_3 after adding FeMn and FeB additives are shown in figure 2. From figure 2, it is clearly seen that the particle size differences based on the cumulative value diameter at statistical positions 10, 50, and 90%. The particle size distribution obtained is heterogeneous with the presence of peaks formed.
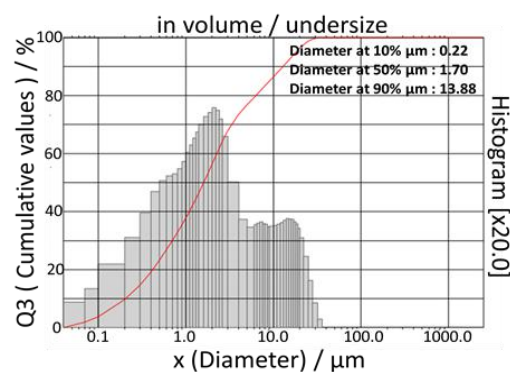

Figure 1. Distribution histogram of mean particle diameter of α-Fe_2O_3.
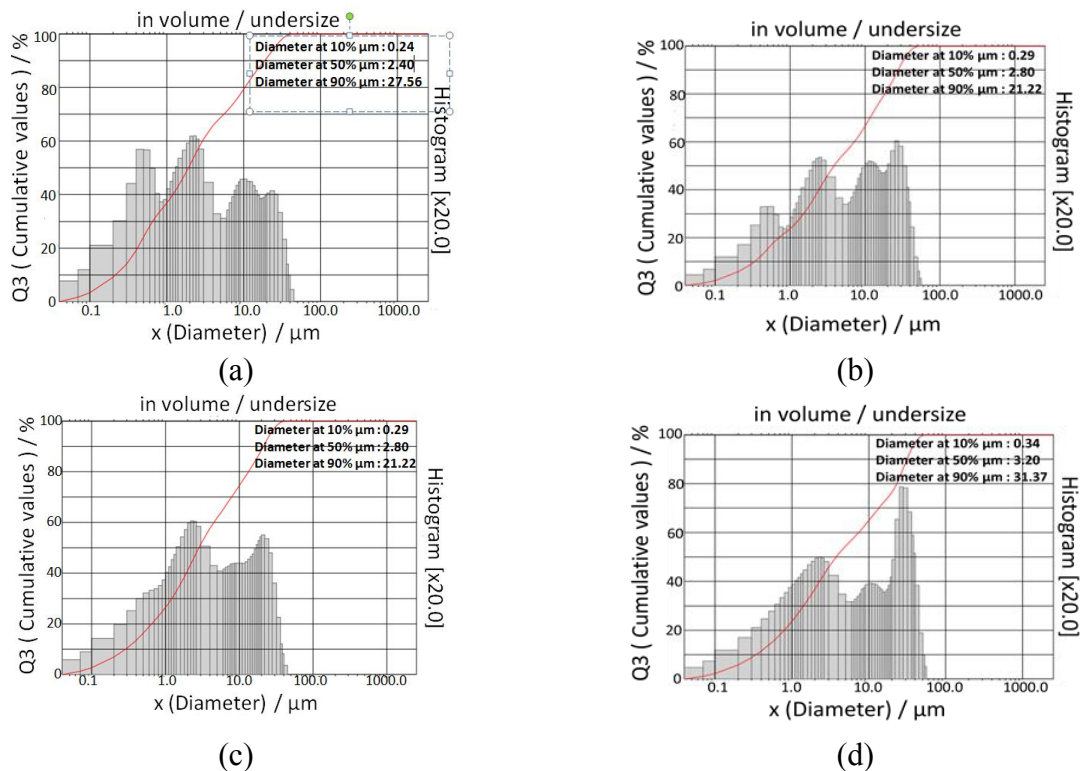
Figure 2. Distribution histogram of mean particle diameter of (a) α-Fe$_2$O$_3$ + 4% FeMn, (b) α-Fe$_2$O$_3$ + 8% FeMn, (c)α-Fe$_2$O$_3$ + 4% FeB, and (d) α-Fe$_2$O$_3$ + 8% FeB.
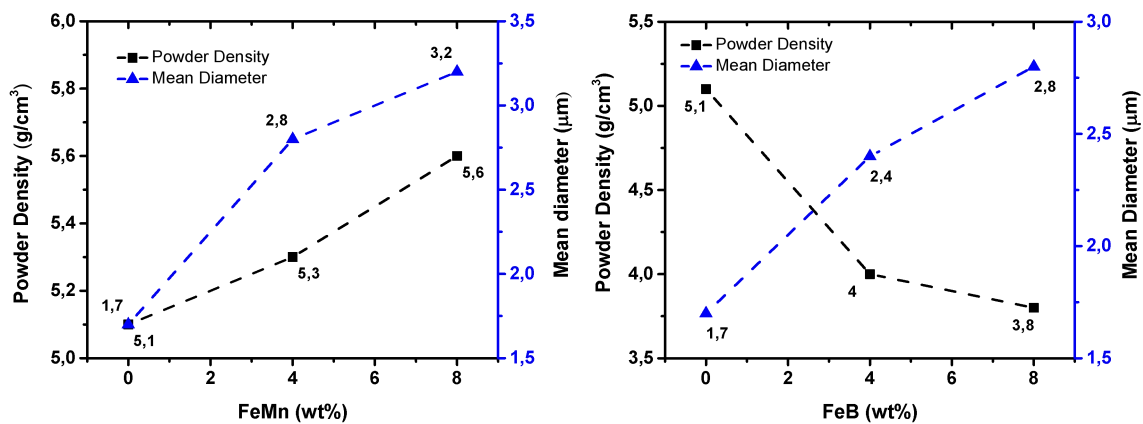

Figure 3. The Correlation of powder density and mean diameter of hematite (α-Fe$_2$O$_3$) with an addition of (a) FeMn, and (b) FeB.

The correlation between powder density and mean diameter of α-Fe$_2$O$_3$ after adding FeMn and FeB are shown in figure 3. From figure 3, it can be seen that the effect of the addition of FeMn and FeB causing the mean diameter of particle tends to increase. It means that the milling effect for 1 h with high energy milling (HEM) has not been able to reduce the particle size of additives, where α-Fe$_2$O$_3$ particle is around 1.7 μm. The powder density values tend to decrease with the addition of FeB additive, while the powder density tends to increase with the addition of FeMn additives.

Thus, it can be seen that the correlation between the powder densities to the mean diameter of FeMn addition is proportional, but inversely proportional for the addition of FeB. The density value of

FeMn, FeB, and α-Fe2O3 is about 7.3, 4, and 5.27 g/cm^3, respectively. From those density values, the FeMn is higher than α-Fe$_2$O$_3$. Thus, density of sample increases with more addition of FeMn. Conversely, the powder density tends to decrease with the addition of FeB. The higher density of FeMn doped α-Fe$_2$O$_3$ than FeB doped α-Fe$_2$O$_3$ can be explained due to the hardness value of FeMn is higher than FeB. From this result, the number of density is strongly influenced by the heat treatment and material composition.

The phase analysis by using XRD of hematite powder (α-Fe$_2$O$_3$) after added by the FeMn and FeB are shown in figure 4. The obtained diffraction pattern are for (a) α-Fe$_2$O$_3$, FeB, and FeMn as raw material, and (b) the mixing of 4% FeMn + α-Fe$_2$O$_3$ and the mixing of 8% FeB + α-Fe$_2$O$_3$ additive after calcination at1000°C for 2 h.

From figure 4 (a) the XRD analysis result of FeMn material shows the major peaks of FeMn phase. FeMn has a cubic structures and lattice parameters of a = 3.668 Å. In the diffraction pattern of FeB, it has single phases with lattice parameter of a = 4.053 Å, b = 5.495 Å, and c = 2.946 Å. While diffraction pattern of the α-Fe$_2$O$_3$ has a single phase which has a rhombohedral structure with lattice parameters of a = b = 5,032 Å and c = 13,733 Å. The diffraction pattern in figure 4 (b) is the diffraction of 4% FeMn + α-Fe$_2$O$_3$ and 8% FeB + α-Fe$_2$O$_3$. The obtained diffraction pattern shows three phases, which has α-Fe$_2$O$_3$ as the dominant phase and the two others phases are MnO$_2$ and Fe$_2$(BO$_3$)O phases.
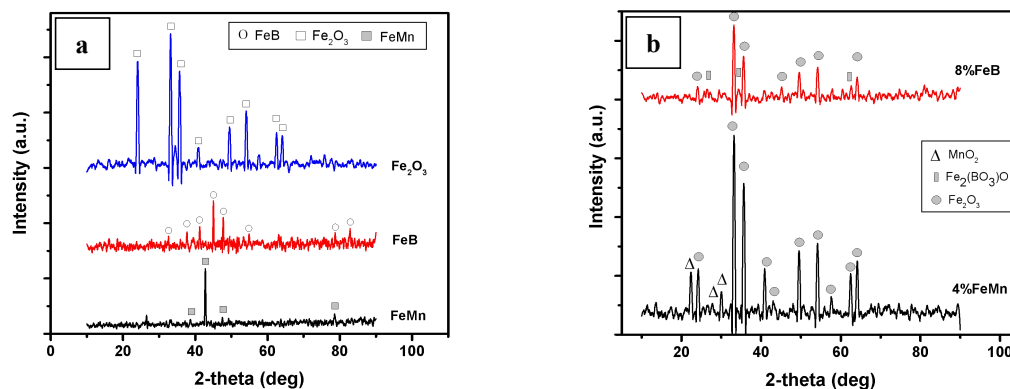

Figure 4. X-ray diffraction pattern of raw materials (a) FeMn, FeB, and α-Fe$_2$O$_3$ and (b) α-Fe$_2$O$_3$ added 4% FeMn and 8% FeB with calcination at 1000 ºC for 2 h.

The magnetic properties of α-Fe$_2$O$_3$with an additive variation of FeMn and FeB before and after calcination is shown in figure 5. In table 1, it can be seen that the value of saturation, remanence magnetization, and coercivity of α-Fe$_2$O$_3$ are 0.68 emu/g, 0.12 emu/g, and 372.31 Oe, respectively. These values are low because the basic properties of the α-Fe$_2$O$_3$ material at room temperature are antiferromagnetic [15], so that this material will have very little response to the magnetic field. While for samples with 4 and 8% FeMn addition, it shown that the value of saturation, remanence magnetization, and coercivity are close resemblance as shown in figure 5 (a). These values are much greater than the value of the α-Fe$_2$O$_3$ material, but for sample with 4 and 8% FeMn addition after calcination at 1000ºC for 2 h, its magnetic properties decrease to a quite low value as shown in figure 5 (b). This can be explained due to the addition of FeMn resulting the MnO$_2$ phase as can be seen from the XRD results. Although the dominant phase in the sample is α-Fe$_2$O$_3$ which has antiferromagnetic properties, the appearance of MnO$_2$ affects significantly its magnetic properties. The magnetic properties of a material are also influenced by other factors such as the degree of crystallinity, particle size and the presence of secondary phase effects [16]. Furthermore, the same phenomena shown on the sample with the addition of 4 and 8% of FeB followed by the calcination at 1000ºC for 2 h. It is also found that due to the addition of FeB, it also forms the new phase of Fe$_2$(BO$_3$) (figure 2 (b)) that decreases the magnetic properties. From the result, prior to calcination, magnetic properties of each

sample is influenced by Fe_2O_3 and the additive, but after the calcination nanocomposite Fe_2O_3-FeMn produced MnO_2 and nanocomposite Fe_2O_3-FeB produced $Fe_2(BO_3)$. Both $Fe_2(BO_3)$ and MnO_2 have antiferromagnetic behavior, therefore their magnetic properties decrease.

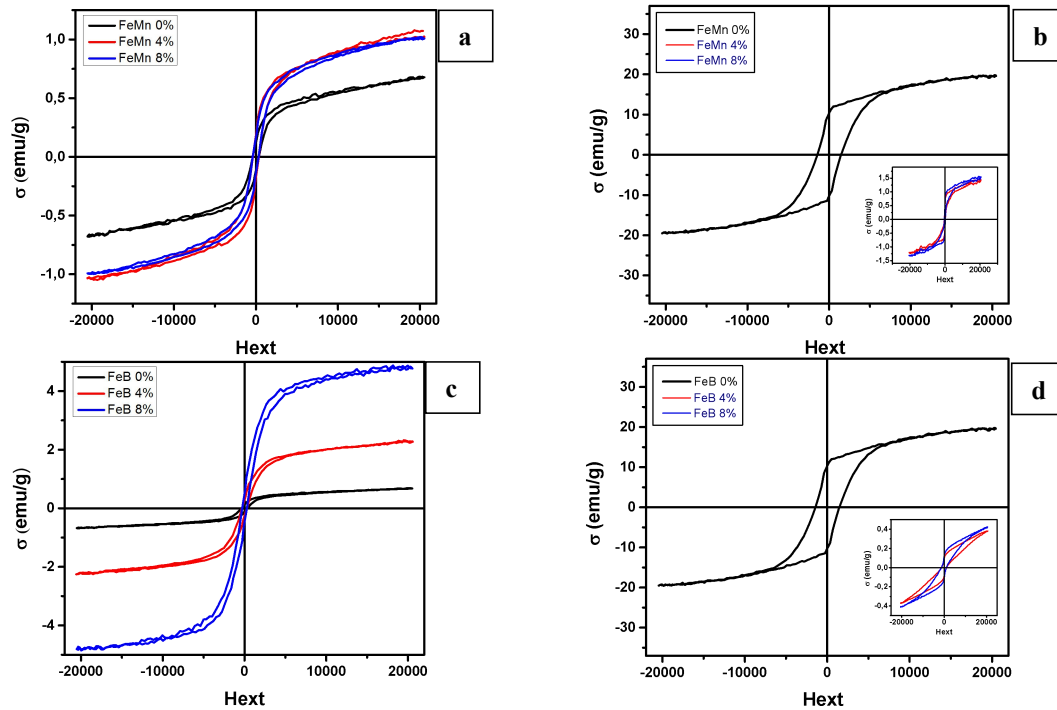**Figure 5**. Magnetic properties of α-Fe_2O_3 with additive variations of FeMn: (a) before and (b) after calcinations; and variations of FeB: (c) before and d) after calcinations.

Table 1. Magnetic properties of α-Fe_2O_3 with additive variations of FeMn, before and after calcined at 1000ºC for 2 h.

Composition of FeMn (wt.%)	Before Calcination			After Calcination		
	σs (emu/g)	σr (emu/g)	jHc (Oe)	σs (emu/g)	Σr (emu/g)	jHc (Oe)
0	0.68	0.12	372.31	19.76	10.31	1430.96
4	1.09	0.18	352.04	1.55	0.48	380.98
8	1.02	0.17	330.94	1.45	0.54	312.91

Table 2. Magnetic properties of α-Fe_2O_3 with an additive variation of FeB, before and after calcined At 1000ºC for 2 h.

Composition of FeB (wt.%)	Before Calcination			After Calcination		
	σs (emu/g)	σr (emu/g)	jHc (Oe)	σs (emu/g)	Σr (emu/g)	jHc (Oe)
0	0.68	0.12	372.31	19.76	10.31	1430.96
4	2.32	0.29	293.92	0.38	0.08	1326.18
8	4.87	0.48	288.50	0.42	0.12	1350.24

4. Conclusions

Powder hematite (α-Fe_2O_3) with the addition of FeMn and FeB additives derived from natural raw materials has been successfully made with powder metallurgy method. Then samples were calcinated at the temperature of 1000ºC for 2 hours. The analysis showed that the powder with the Fe_3Mn_7 addition has a dominant phase α-Fe_2O_3 and two new phases of MnO_2 and Fe_3O_4. The result of powder

density measurement showed that the optimum value was obtained for α-Fe_2O_3 sample with 8% wt Fe_3Mn addition. This material is classified as a hard magnet with magnetization value of saturation, remanent and coercivity of 24.0 emu/g, 10.3 emu/g and 571.8 Oe. The effect of 8 wt.% FeB addition on hematite (α-Fe_2O_3) material before calcination resulting the decrease of the coercivity value from 372.19 Oe to 285.06 Oe. The influence of calcination temperature 1000 ℃ for 2 h causes increasing of the grain growth and magnetic properties. While the effect of FeB addition at 0, 4, and 8 wt.% after calcination tends to convert the sample into soft magnetic with coercivity values   of 1450, 1220 and 1180 Oe

Acknowledgements

References

[1] Widanarto W, Sahar M R, Ghoshal S K, Arifin R, Rohani M S and Hamzah K 2013 *J. Mag. Mag. Mat.* **326** 123

[2] Monazam E R, Breault R W and Siriwardane R 2014 *Ind. Eng. Chem. Res.* **53** 13320

[3] Wang W W, Zhu Y J and Ruan M L 2007 *J. Nanopart. Res.* **9** 419

[4] Darab M I and Shivashankar S A 2014 *RSC Adv.* **4** 4105

[5] Kalendová A, Ryšánek P and Nechvílová K 2015 *Prog. Org. Coatings* **86** 147

[6] Poorbafrani A and Kiani E 2016 *J. Mag. Mag. Mat.* **416** 10

[7] Naseri M G, Saion E B, Hashim M, Shaari A H and Ahangar H A 2011 *Solid State Comm.* **151** 1031

[8] Zhang X, Niu Y, Li Y, Hou X, Wang Y, Bai R and Zao J 2013 *Mater. Lett.* **99** 111

[9] Chirita M and Grozescu T 2009 *Chem. Bull. Polithecnica Univ Timisoara* **54** 1

[10] Kadir I A and Aliyu A B 2013 *African J. Pure Appl. Chem.* **7** 114

[11] Bagheri S, Candrappa K G and Hamid S B A 2013 *Res. J. Chem. Sci.* **3** 62

[12] Kefeni K K, Msagati T A M and Mamba B B 2017 *Mater. Sci. Eng. B* **215** 37

[13] Shen W, Mu Y, Xiao T and Ai Z 2016 *Chem. Eng. J.* **285** 57

[14] Lee J and K S-Y 2018 *ACS Omega* **3** 2634

[15] Suharyadi E, Setiadi E A, Shabrina N, Kato T and Iwata S 2014 *Adv. Mater. Res.* **896** 126

[16] Setiadi E A, Kurniawan C, Sebayang P and Ginting M 2017 *IOP J. Phys.: Conf. Series* **817** 012054

[17] Zuh B, Qiherima Lv L and Wang X 2012 *Key Eng. Mater.* **492** 264

[18] Bell A M T, Rodriguez-Martinez L M, Attfeild J P, Cernik R J, Clarke J F and Perkins D A 1998 *Mater. Sci. Forum* **278-281** 708