

Characterization of Magnetic Composite with Filler East Lombok Iron Sand and Polyvinyl Alcohol Matrix

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Abstract: In the current study it has been successfully fabricated and magnetically characterized Fe_3O_4 composite with polyvinyl alcohol (PVA) matrix. Fe_3O_4 is obtained from the iron sands of East Lombok. Morphology and elements East Lombok iron sand analysis results from Scanning Electron Microscope Energy Dispersion X-Ray (SEM-EDX) shows that iron sand tends to be spherical and the elements contained in it are Fe (ferum), O (oxygen), Ti (titanium), Al (aluminum), Si (silica), Mg (magnesium), and V (vanadium) and the average diameter of the Particle Size Analyzer (PSA) analysis was 165.76 m. Characterization includes remanent magnetization value (M_r), coercivity (H_c) and maximum energy product value (BH)_{max}. In this study, Fe_3O_4 was mixed with a PVA matrix with a percentage variation of 70% : 30%; 60% : 40%; 50% : 50% of the total mass of 10 grams. Based on the data obtained from this research, the addition of Fe_3O_4 causes an increase in the density, remanent magnetization, coercivity, maximum energy product. However, the resulting compressive strength is directly proportional to the concentration of the matrix added.

Keywords: Magnetically characterized, Remanent magnetization, Coercivity, Maximum energy product

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I. INTRODUCTION

The use of permanent magnets is increasing, especially in the electronics and automotive industries, causing Indonesia to become the second largest permanent magnet market in the world. As for meeting these needs, 100% of the permanent magnets on the market are imported from Japan and China. In fact, Indonesia has the potential to produce itself without must be imported, because the availability of raw materials for permanent magnets is very abundant in the country, especially iron sand (Rahayu et al., 2018). Iron sand is the main ingredient in the manufacture of ferrite-based permanent magnets. The type of ferrite magnet that is commonly used is barium ferrite (BaFe) because it has strong mechanical properties and is not easily corroded (Jafirin et al., 2014). However, ferrite type magnets have a weakness, namely low magnetic properties. The remanence (B_r) of these magnets is only 1/3 of that of rare earth magnets (NdFeB) (Sunaryono et al., 2013).

Iron sand is one of the basic ingredients in the manufacture of magnets. In general, iron sand has the main composition of iron oxides Fe_3O_4 (magnetite) and Fe_2O_3 (hematite), silicon oxide (SiO_2), as well as other compounds, namely Fe, Ni, and Zn with smaller levels (Indrayana, 2019). Iron sand is a natural raw material whose utilization is not widely known because it requires a touch of technology in its utilization. The existence of iron sand which is widely distributed and abundant in Indonesia, especially in East Lombok is an economic attraction to be developed into a more valuable and efficient product (Idayanti et al., 2018).

Soft ferrite has the formula MFe_2O_4 where M is Cu, Zn, Ni, Co, Fe, Mn, Mg with a crystal structure like spinel minerals. The properties of this material have high permeability and specific resistance and low coercivity reported in their research that the magnetic susceptibility of $MnFe_2O_4$ (manganese ferrite) is higher than other ferrites such as Fe_3O_4 , $CoFe_2O_4$ and $NiFe_2O_4$ with a magnetic spin of 5b (Baker, 2018). In addition, its much lower resistivity than $CoFe_2O_4$ and $NiFe_2O_4$, high biocompatibility compared to Fe_3O_4 , $-Fe_2O_3$, $CoFe_2O_4$, and $NiFe_2O_4$ when applied for magnetic resonance imaging (MRI). In addition, $MnFe_2O_4$ at room temperature (20°C) has a low anisotropy energy (Zuo et al., 2005), this condition will cause thermal energy at room temperature to prevent the anisotropic energy from returning to its lowest state. This phenomenon then gives rise to superparamagnetic properties in single nanoparticles. New facts continue to emerge from the study of manganese ferrite using various methods, the results obtained are variations in grain size from the smallest (4 nm) at a temperature of 320 °C (Matzen et al., 2011), to the largest (154.1 nm) at 420°C (Lakshman et al., 2002).

From the description of the research results above, the use of Fe_3O_4 , as a magnetic material has not been carried out. This study used Fe_3O_4 as a filler in composites with a polyvinyl alcohol (PVA) binding matrix. The method of making composites with the vacuum infusion method. Vacuum infusion is one method of

making composite materials with the working principle of utilizing atmospheric pressure as a means of suppression. Vacuum Infusion has inlet (in late) and outlet (out late) with the amount as needed. The in late channel is used as the resin inflow, while the late out channel is used as the resin outflow. So that no air is trapped in the PVA matrix fluid, resulting in reduced voids in the composite.

This study aims to determine Characterization of mechanical and magnetic properties, composite magnetic material with Fe₃O₄ filler, and PVA matrix. The mechanical properties to be tested are density and compressive strength. The magnetic properties to be tested are remanent magnetization value (Mr), coercivity (Hc) and maximum energy product value (BH)max.

In the current study, the effects of the effect of weight variation (%Wt) of Fe₃O₄ filler and PVA binding matrix on the characteristics of mechanical and magnetic properties, composite magnetic. In this work, the matrix used is polyvinyl alcohol (PVA) because the adhesive matrix does not require high heating temperatures during the mixing process. The average temperature for dissolving the matrix is less than 25 C. In addition, the matrix has good durability and chemical resistance (Cahya Rahayu et al., 2018).

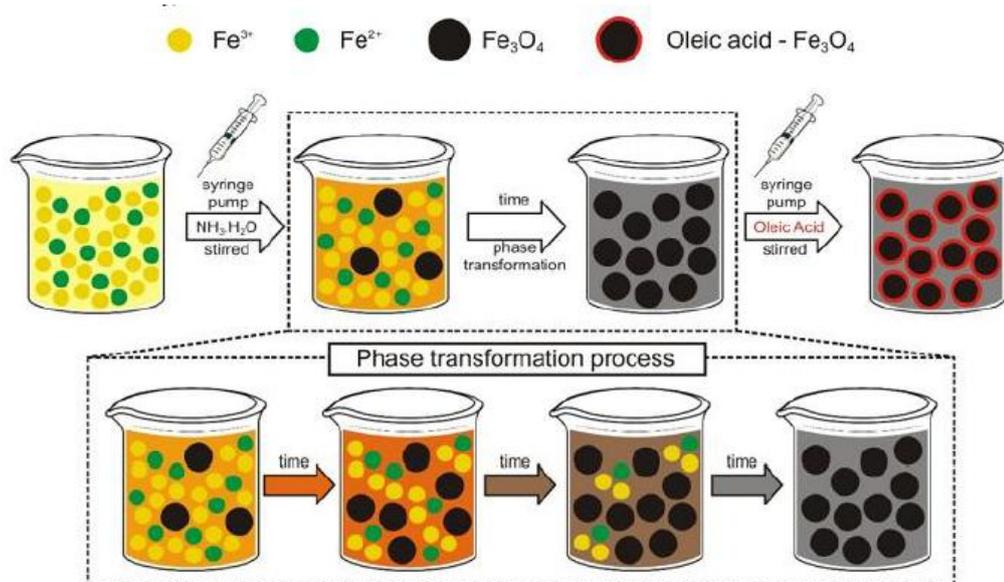
II. EXPERIMENTAL PROCEDURE

1.1 Preparation of Fe₃O₄ particle.

The iron sand available in Poh Gading village, East Lombok district, Indonesia. Coprecipitation method is one type of wet chemical method used to synthesize Fe₃O₄. In the synthesis process, precursors in the form of anhydrous metal salts are used as a source of metal ions and basic hydroxide compounds such as NaOH and KOH act as coprecipitant. Coprecipitation method is a synthesis method which has the simplest synthesis step and does not require high temperature treatment ($T < 120^{\circ} \text{C}$) for the synthesis of Fe₃O₄ samples. There are 3 main stages of the coprecipitation synthesis method. First, the preparation of the precursor metal salt solution. If the source of Fe ions is iron sand, the anhydrous precursors FeCl₃·6H₂O and FeCl₂·4H₂O are not used. Making a solution of Fe²⁺/Fe³⁺ from iron sand is by dissolving iron sand with 12M HCl at a temperature of $\pm 70^{\circ} \text{C}$. Second, the reaction for the formation of Fe₃O₄ through an alkaline reaction. The Fe solution was dropped into the coprecipitant solution while being stirred at a T-synthesis temperature $< 120^{\circ} \text{C}$. Third, the process of washing Fe₃O₄ nanoparticle slurry and then oven at 90°C for 4 hours.

The control carried out on the synthesis parameters, such as synthesis temperature, stirring rate, and coprecipitant concentration, will affect the particle size of Fe₃O₄. In general, the mass of Fe₃O₄ nanoparticles produced is the largest compared to other synthesis methods. The weakness of the coprecipitation method is that the grain size distribution of the nanoparticles tends to be large and the polydispersivity of the particles is small. Nanoparticles easily agglomerate. The solution is to functionalize the surface of the nanoparticles by adding a capping agent in the form of oleic acid, as illustrated in Figure 1.

Figure1. The synthesis process of Fe₃O₄ by coprecipitation method with capping agent oleic acid



1.2 Magnetic composite material preparation.

The process of making magnetic composite materials in this study was carried out using the vacuum infusion method. Vacuum infusion is one method of making composite materials with the working principle of

utilizing atmospheric pressure as a means of suppression. Vacuum Infusion has inlet (in late) and outlet (out late) with the amount as needed. The in late channel is used as the resin inflow, while the late out channel is used as the resin outflow. So that no air is trapped in the PVA matrix fluid, resulting in reduced voids in the composite. The stage preparation of pulverizing Fe₃O₄ up to a size of 70 m using a planetary ball mill. Fe₃O₄ particles mixed with Polyvinyl Alcohol (PVA) binder matrix with variation of composition percentage by weight (% Wt) : 70% : 30%; 60% : 40%; 50% : 50% of the total mass of 10 grams. The mixing process with the wet mixing method uses aquades. The sample mixture that has dried is then compacted (dry pressing) at a pressure of 120 kg/cm² using a compacting machinea digital and magnetized solenoid using an NdFeB permanent magnet with a flux density of 1 Tesla to orient the spin orientation. After that, the sample was allowed to stand for 24 hours for the maturation process of the bond structure (age hardening) to obtain a hard and strong sample.

1.3 Procedure for characterization of composite magnetic materials.

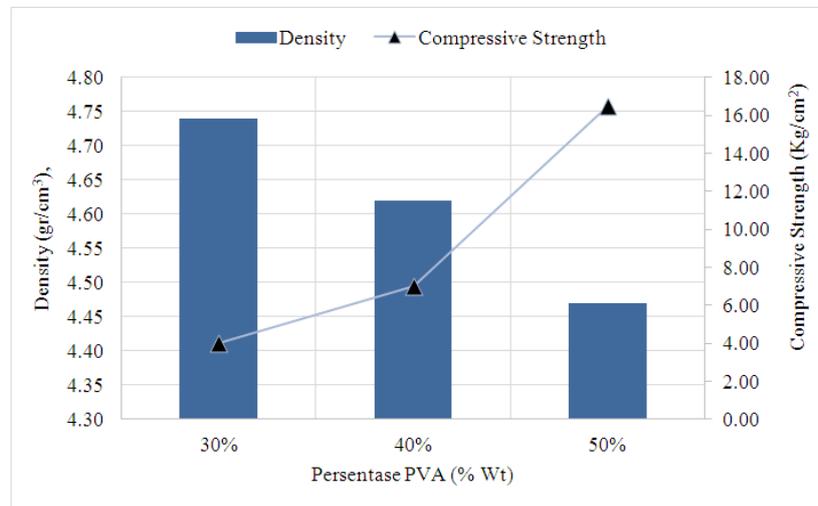
In this study, the weight percentage of Fe₃O₄ and the PVA binder matrix was varied, so that the mechanical and magnetic characteristics of the magnetic composite material being studied were varied. Testing of mechanical properties in the form of density measurement, compressive strength and observations with a scanning electron microscope (SEM) microstructure, as well as the distribution of Fe₃O₄ particles in the composite. Meanwhile, magnetic testing was carried out using a permagraph to determine the value of the magnetic parameters which included: remanence (Br), coercivity (Hc) and maximum energy product (BHmax). Then an analysis of the mechanical and magnetic property trend data was carried out as a function of weight percentage variation (% Wt) of the PVA matrix.

III. RESULTS AND DISCUSSION

3.1. Characteristics mechanical properties of material magnet composite Fe₃O₄

The percentage variation (%wt.) of PVA used in the manufacture of Fe₃O₄ magnetic composites affects the mechanical properties. Particle distribution image results show different results for each variation percentage weight (% Wt) of PVA. The image of the distribution of Fe₃O₄ particles with variations in PVA concentration in Figure 4. shows results that are not much different from one another. The PVA matrix appears to be evenly distributed over the entire sample surface, although at higher matrix concentrations more pores/cavities are found. The figure shows that the PVA function as a binder shows good results because it does not agglomerate. The density shown also shows results that are not much different. Sample variation with PVA percentage 30% wt., 40% wt., and 50% wt. respectively, showed density values of 4.74, 4.62, and 4.47 g/cm³. The density of the composite is influenced by the density of the Fe₃O₄ particles. The density of Fe₃O₄ particles is greater than the density of the polyvinyl alcohol (PVA) matrix. The higher the percentage of PVA weight (the smaller the percentage of Fe₃O₄ particle weight) then the density of the specimen will decrease. However, the opposite trend was shown by the compressive strength. The compressive strength actually increased with the addition of PVA concentration with the results of 4, 7, and 16.5 Kg/cm², as shown in Figure 2. The PVA matrix in the composite magnetic material acts as a binder for Fe₃O₄ particles, reinforcement, the secondary part that holds the load, so that the size of the compressive strength of the composite material is highly dependent on the percentage of weight of the matrix that forms it.

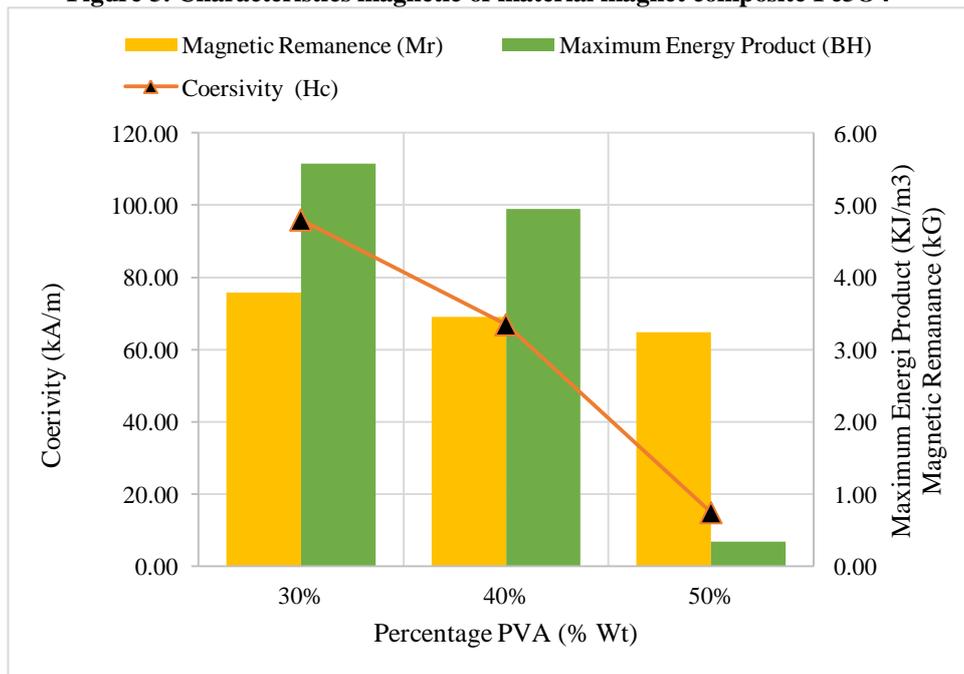
Figure 2. Characteristics mechanic of material magnetcomposite Fe₃O₄



3.2. Characteristics magnetic properties of material magnet composite Fe₃O₄

The tendency to decrease the magnetic properties (magnetic remanence (Mr), coersivity (Hc) and maximum energy product (BH)) is influenced by the variation in the percentage (% Wt) of the PVA matrix. Based on SEM observations, the PVA matrix agglomerates and has more pores along with the subtraction of particle Fe₃O₄ weight percentage (%Wt). Changes in the characteristics of magnetic properties due to changes in the percentage weight of the PVA matrix are shown in Figure 2. The magnetic remanence (Mr) of the specimen is: 3.79, 3.45, 3.25 kG, coersivity (Hc) is : 96, 67, 15 kA/m and the maximum energy product (BH) is : 5.57, 4.95, 0.34 KJ/m³, at weight percentage of PVA: 30%, 40% and 50% Wt, respectively. The characteristics of the magnetic properties of the specimen are influenced by the percentage of Fe₃O₄ particle weight. The greater the percentage of weight, the characteristics of the magnetic properties also increase, and vice versa. Because Fe₃O₄ particles are ferromagnetic.

Figure 3. Characteristics magnetic of material magnet composite Fe₃O₄



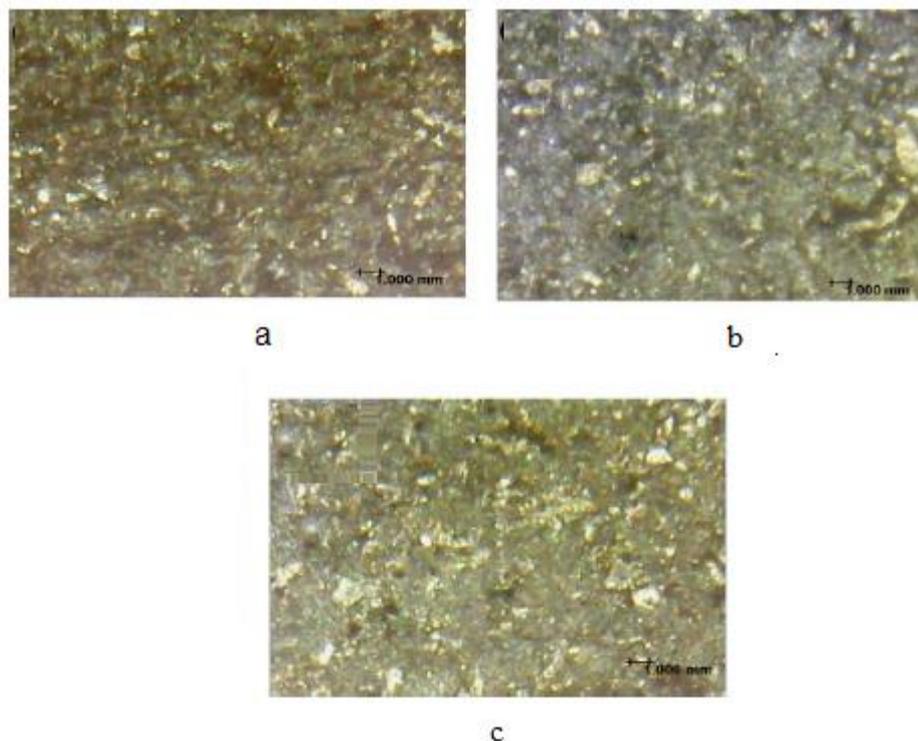
3.3. Surface morphology of material magnet composite Fe₃O₄ with matrix PVA

Surface morphology of material composite magnet Fe₃O₄ with matrix PVA, as shown in Figure 4. They were examined by using SEM for different modes of the distribution of Fe₃O₄ particles mechanism occurred in magnet composite with matrix PVA. SEM image clearly in Figure 4. the white PVA matrix particles are seen to be evenly distributed over the entire surface of the sample and no matrix agglomeration is seen. Furthermore, the results of the measurement of the dimensions and density of the variation of the sample show that the addition of the percentage of PVA weight in the sample is inversely proportional to the density of the specimen. This is because PVA has a lower density (3.28 g/cm³) compared to Fe₃O₄ particles, which is 7.57 g/cm³. The same result is also shown by the compressive strength value. The compressive strength increased along with the addition of PVA weight percentage as shown in Figure 2. The PVA matrix appears to be evenly distributed over the entire sample surface, although at higher matrix concentrations more pores/cavities are found.

The Figure 4. shows that the PVA function as a binder shows good results because it does not agglomerate.

In Figure 4. it can be seen that the pores or cavities indicated by the black color are getting bigger and more numerous with the addition of %wt. PVA matrix. Therefore, the resulting magnetic density and remanence trend is also significantly reduced. The characteristic magnetic properties of the magnet composite material samples are as follows : the magnetic remanence (Mr) of the specimen is: 3.79, 3.45, 3.25 kG, coersivity (Hc) is : 96, 67, 15 kA/m and the maximum energy product (BH) is : 5.57, 4.95, 0.34 KJ/m³, at respectively. with PVA weight percentage is 30 % wt., 40 % wt. and 50 % wt. The characteristic mechanical properties, density respectively were of 4.74, 4.62, and 4.47 g/cm³, and compressive strength were of 4, 7, and 16.5 Kg/cm².

Figure 4. SEM image of of magnet composite a. PVA 30%, b PVA 40%, c. PVA 50%



IV. CONCLUSION

Based on the results of mechanical testing and SEM observations, concluded that the distribution of Fe_3O_4 particles affects the density of magnetic composite materials with PVA matrix. Matrix PVA has the ability to bind materials magnets well as indicated by the PVA particles that are evenly distributed so that the trend of mechanical and magnetic tend to be more stable, while the particle distribution image, matrix CMC agglomerate so that the resulting density trend is more fluctuating. Addition the composition of Fe_3O_4 particles in the composite sample causes an increase in density, compressive strength, and magnetic properties (remanence). While the addition of the percentage by weight (%Wt.) of the PVA matrix in the magnetic composite sample caused a decrease in density and magnetic properties, but the compressive strength produced was directly proportional to the concentration of the PVA matrix.

Conflict of interest

There is no conflict to disclose.

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