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Characterization of magnetic properties of ferric oxide from Sekotong beach iron sand West Lombok Indonesia

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Abstract

In the present study preparation and characterization of ferric oxide (Fe3O4) with ball milling has been done, for enhancing magnetic properties. Fe3O4 is obtained from the iron sands Sekotong Beach, West Lombok, Indonesia. The Fe3O4 has gained its importance due to its low cost, substitute for pure BaFe. Nano-sized Fe3O4 has applications in industrial fields such as ceramics, catalysts, energy storage, magnetic data storage, ferrofluids, as well as in medical diagnosis. Characterization of iron sand samples using Scanning Electron Microscopy with Energy Dispersive X-ray (SEM-EDX), and Permagraph Magnet Physik Germany, at BRIN LIPI Bandung. The result of characterization using SEM-EDX shows that iron sand samples contain Fe and O elements derived from Phase Magnetite (Fe3O4). The magnetic properties: Magnetic remanence (Mr), coersivity (Hc), maximum energy product (BH)) of magnetic, 3,01 kG, 4,97 kOe, 3,72 MGOe respectively. A small coercivity value is one of the characteristics of super paramagnetic materials.

Keywords: Ferric Oxide (Fe3O4); Iron Sand; Magnetic Properties; Phase Magnetite

1. Introduction

The island of Lombok, Indonesia has natural resources in the form of iron sand which is very potential. Iron sand is a raw material for the manufacture of ferrite-based permanent magnets with various applications [1]. The high use of permanent magnets in Indonesia, especially in the electronics and automotive industries, has made Indonesia 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 its own without having to import, because the availability of raw materials for permanent magnets is very abundant in the country, especially iron sand [2].

Indonesia has abundant natural resources in the form of iron sand. Sand is commonly found on beaches such as the west coast of Sumatra, the south coast of Java, Kalimantan, Sulawesi, Nusa Tenggara and the Maluku islands [3]. Iron sand is usually used as an additive to cement and steel, in the development of material technology, iron sand can be used in the field of nanomaterial technology. Nanotechnology is a technology that involves atoms and molecules with a size smaller than 1,000 nanometers. One of the most widely developed types of nanomaterials is Fe3O4 nanoparticles. Iron sand can be a source of Fe3O4 nanoparticles, because iron sand contains basic magnetic materials such as magnetite (Fe3O4), hematite (α -Fe2O3) and maghemite (γ -Fe2O3) [4]. To produce a homogeneous and fine grain size of nanoparticles, Fe3O4 can be synthesized in several ways, namely by using coprecipitation methods, sol-gel, solid state, hydrothermal, sonochemical, molten salts, and others [5] [6]. The Fe₃O₄ has gained its importance due to its low cost, substitute for pure BaFe. Nano-sized Fe3O4 has applications in industrial fields such as ceramics, catalysts, energy storage, magnetic data storage, ferrofluids, as well as in medical diagnosis [6].

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Studies on the synthesis and modification of magnetite (Fe3o4) nanoparticles continue to be improved, because magnetic nanoparticles are materials that have superior optical, magnetic, and chemical properties. The advantages of magnetic nanoparticles are most prominent for very small particles, around 10-20 nm, and in general. These unique properties are lost for magnetic sizes up to 40-50 nm [7]. Due to their unique properties, magnetic nanoparticles can be used in various fields. The study [8] Fe3O4 magnetic nanoparticles were used as filler for ferrogel composites. The contribution of Fe3O4 magnetic filler on ferrogel composites can be seen in the magneto-elasticity characterization. Ferogel composite with Fe3O4 particle filler is more sensitive to the influence of magnetic field changes than Fe2Cl3 particle filler. This is because the remanent magnet Fe3O4 (8,233 emu/gr) is larger than Fe2Cl3 nanoparticles (7.695 emu/gr) [9]. Research on composites with polyvinyl alcohol and Fe3O4 has been carried out by [10]. The research developed is to synthesize artificial muscles with experimental approaches ranging from those similar to robotic locomotion to more sophisticated soft locomotion. They combined the elastic properties of the PVA gel and the magnetic properties of the micrometer-sized Fe3O4 particles.

Research on the synthesis and characterization of Fe3O4 nanoparticles generally uses a source of Fe, from expensive commercial iron salts, such as: FeCl3, FeCl2, FeSO4, Fe(NO3)3, Fe(ClO4)3.6H2O. [Fe(CO)5], [Fe{N- (SiMe3)22] and La0.5Sr0.5FeO3 [4-7]. For this reason, efforts to obtain materials containing magnetic materials continue to be carried out, especially in finding sources of Fe that are available in nature, at lower prices.

Several studies have been carried out to use Java iron sand as a material to synthesize magnetic nanoparticles (Fe3O4). The desired iron sand as raw material for the synthesis of nanoparticles is easier to separate from the impurities so that it has a high Fe content. However, no one has reported the synthesis of magnetic nanoparticles (Fe3O4) from Lombok's iron sands.

The method used to synthesize magnetite nanoparticles in this study is the coprecipitation method. The coprecipitation method is a method that is quite effective and quite simple when compared to other methods. This method is based on precipitation through the organic compounds used, so that this method becomes simpler, produces a grain size distribution that is relatively the same and can be carried out at different temperatures.

Therefore, in this study, a hybrid between Fe3O4 (Ferric Oxide) and NdFeB (Neodyum Iron Boron) was carried out to improve the mechanical and magnetic properties of magnetic composite materials. To simplify the manufacturing process to make it easier, the method used is bonded magnet with the addition of a polyvinyl alcohol (PVA) matrix / binder. The average temperature for dissolving the matrix is less than 25 °C. In addition, the PVA matrix has good durability and chemical resistance [11]. In this research, Fe3O4 and NdFeB were varied at a certain composition, so that a trend was obtained from the mechanical and magnetic properties of the hybrid magnetic composite material being studied. The testing of the mechanical properties of magnets includes observation of particle distribution images, density measurements, and compressive strength. The magnetic testing was carried out using a permagraph to determine the value of the magnetic parameters which included remanence (Mr), coercivity (Hc) and maximum energy product (BHmax). Furthermore, it will be studied and analyzed the resulting mechanical and magnetic properties trends as a function of Fe3O4 variations.

Thus, it should be noted that there is no data found in the literature that would explain the possibility of using Fe3O4 from iron sands in Lombok Island as a magnetic composite filler, so that the mechanical and magnetic properties are better, for industrial use. This study aims to determine the mechanical and magnetic properties of the Fe3O4 - NdFeB hybrid magnetic composite with PVA matrix which is very much needed by the industry, as an alternative solution to the more expensive rare earth magnetic composite material.

2. Material and methods

2.1. Materials

The main material of this research is iron sand from Sekotong Beach, West Lombok, as shown in Figure 1. This is based on a previous study that used iron sand from Telindung Beach, East Lombok to be synthesized into Fe3O4 magnetite and characterized [8]. The iron sand of Sekotong Beach, West Lombok is first extracted using a permanent magnet to separate the iron sand from its impurities. Then, the Fe3O4 nanoparticle synthesis process was carried out using the coprecipitation method. The research supporting tools used include a beaker glass, magnetic stirrer, rotary ball mill, spatula, stainless steel mold, solenoid press, technical scale, SEM-EDX, and Permagraph Magnet Physik German.



Figure 1 The iron sand from Sekotong Beach, West Lombok

2.2. Methods

Method of synthesis of Fe3O4 sample in this study carried out in several stages: Iron sand extraction at Sekotong beach, West Lombok is carried out using permanent magnets. in order to separate themselves from impurities. Refining the size of the iron sand by using ball milling, then sifting with a size of 200 mesh to get a uniform size of iron sand. Dissolve 40 grams of iron sand into 38 ml of HCl (12M) at 55°C and stir for about 60 minutes using a magnetic stirrer. Reaction equation of dissolved iron sand in to HCl:

 $Fe_{3}O_{4}(s) + 8HCl(l) \longrightarrow 2Fe_{3}Cl_{3}(l) + Fe_{3}Cl_{2}(l) + 3Fe_{3}O_{2}(s) + 3H_{2}O(l) + H_{2}(g)$ (1)

Precipitation by coprecipitation method. The solution that has been formed is then filtered using filter paper, the result is a filtrate. To assist in the precipitation of the solution, 73 ml of NH4OH was added and then allowed to stand for 1 hour to obtain a precipitate. The result of iron sand sediment (ilrat) which is black in color is then washed with distilled water 7 times [4].

Reaction equation of precipitation:

$$2 \text{FeCl}_{3} (l) + 2 \text{FeCl}_{2} (l) + 3 \text{H}_{2} \text{O} (l) +$$

8NH4OH (l) \longrightarrow Fe₃O₄(s) + 8NH4Cl (l) + 5H₂O (l) (2)

3. Results and discussion

3.1. The magnetic properties of Ferric Oxide from Sekotong Beach Iron Sand West Lombok Indonesia

The magnetic properties of ferric oxide (Fe3O4) were tested using Permagraph Magnet Physik Germany, at BRIN LIPI Bandung. Characterization of magnetic properties studied include: Magnetic remanence (Mr), coercivity (Hc), maximum energy product (BH)). The results of the Fe3O4 permagraph test contained in the iron sand of Sekotong beach are shown in Figure 2.

The results of the magnetic properties test are illustrated in the form of a hysteresis curve, shown in Figure 2, which is the data from the test using a permagraph. Based on the hysteresis curve, magnetic properties: Magnetic remanence (Mr), coersivity (Hc), maximum energy product (BH)) of magnetic, 3, 01 kG, 4,97 kOe, 3,72 MGOe respectively. A small coercivity value is one of the characteristics of super paramagnetic materials. Then based on the coercivity value, Fe3O4 is classified as a soft magnet [10].

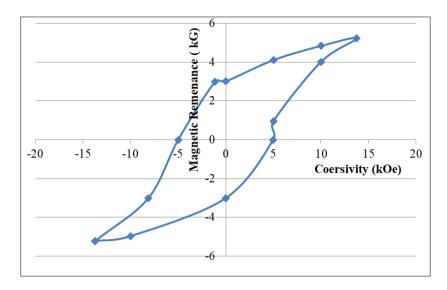


Figure 2 Hysteresis curves of Fe3O4 (from Sekotong beach iron sand)

3.2. The ferric oxide microstructure of Sekotong beach iron sand

Observation of the microstructure aims to determine the composition of the elements contained in the iron sand of Sekotong beach and the morphology of the microstructure. Based on the observation of the microstructure with Scanning Electron Microscopy with Energy Dispersive X-ray with a magnification range of 500 times, as shown in Figure 5. In the photo of the microstructure, the phase of Magnetite (ferric oxide) (Fe3O4) is clearly visible, under a digital microscope with a scale of 500 times indicated by Particulate images are solid black in color, phase maghemite (γ -Fe2O3), white color and phase hematite (α -Fe2O3) are shown with colors in between. Based on the morphology of the microstructure, the maghemite and hematite phases are in the form of agglomeration. According to research [12], the characteristics of magnetite ferric oxide (Fe3O4) are difficult to reduce, and the magnetic properties are very strong.

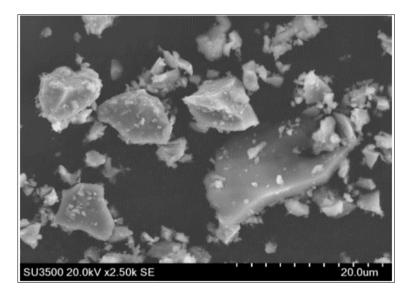


Figure 3 Morphology of microstructure iron sand from Sekotong Beach (sample)

Based on the morphological structure of the iron sand samples from Sekotong beach, West Lomok as shown in Figure 1. it is shown that there is a particle agglomeration process with particle sizes varying from 2-5 mm. The results of the iron sand sample composition test using SEM-EDX, Sekotong beach iron sand, West Lomok contain elements of C, Al, Si, Mn, Ca, Fe, and O as shown in Table 1.

Based on Table 1. it is shown that the main elements of iron sand samples are Fe, Ca and O. (higher percentage than other elements). While some other elements such as K, C, Na, Mg, Al, Si, and Ca with content less than 10%. The content

of Fe and O comes from the Magnetite (Fe3O4), maghemite (γ -Fe2O3), and hematite (α -Fe2O3) phases which are iron oxides that are widely found in iron sands, according to study results [1].

No	Elements	Sample (%wt)
1	Fe	57.12
2	С	2.94
3	0	21.11
4	Al	0.37
5	Si	8.17
6	Са	9.02
7	Mn	1.36

Differences in the physical characteristics of sand mineral content such as Fe, Al, Ca, and Si are caused by differences in the location of the sediment. The elemental Fe content in iron sands found on Sekotong Beach, West Lombok is quite high, namely 55.15%, compared to the Fe content in iron sands found in Padang Freshwater Beach which is 41.32% [12]. Apart from being a source of Fe3O4 iron sand is also one of the main raw materials in the steel industry and other heavy equipment, so its presence plays a very important role in Indonesia and even internationally [6].

4. Conclusion

The addition of Fe3O4 causes a decrease in mechanical properties (density, compressive strength), on the other hand, the addition of Fe3O4 causes an increase in the magnetic properties (Magnetic remanence (Mr), coersivity (Hc), maximum energy product (BH)) of the Fe3O4 - NdFeB hybrid magnetic composite material, with PVA binding matrices. The decrease in density is caused by the density of Fe3O4 compared to the density of NdFeB. The addition of Fe3O4 filler to the magnetic composite with a constant matrix condition causes a decrease in compressive strength. On the other hand, the addition of Fe3O4 filler causes an increase in the magnetic moment, so that the magnetic properties of the specimen increase.

Compliance with ethical standards

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Disclosure of conflict of interest

No conflict of interest.

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