Magnetic properties of Fe$_3$O$_4$ nanoparticles synthesized from natural iron sand via ball-milling process

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Abstract. Magnetite (Fe$_3$O$_4$) nanoparticles show physical and chemical properties that have a great opportunity in biotechnology applications. In nature, magnetite can be found in iron sand. This article related to the synthesis of Fe$_3$O$_4$ nanoparticle from iron sand by a physical route. The iron sand used is obtained from the Tiram beach, West Sumatra. After purification using a permanent magnet, then made in the size of nanoparticles using high-energy milling. The milling time is varied 10, 15, 20, 25 and 30 hours. The Fe$_3$O$_4$ purity as increasing of milling time and homogenous Fe$_3$O$_4$ phase reached at 30 h milling time. All samples were characterized by X ray diffraction (XRD), scanning electron microscopy (SEM), and vibrating sample magnetometer (VSM) at room temperature. All of the samples have been found possess ferrimagnetic property and saturation magnetization ($M_S$) of Fe$_3$O$_4$ nanoparticle are influenced by milling time.

1. Introduction

Material research at the nanometer scale is very rapidly carried out throughout the world today. The end result of this research is to change the existing technology which is generally based on micrometer scale material into technology based on the nanometer scale. Entering the 21st century, new discoveries in the field of nanomaterials appear almost every week and new applications begin to appear in various fields such as electronics, energy, chemistry, health and medicine, the environment, and so on [1].

Nanomaterials are very interesting studies, because nanoscale materials usually have properties which include physical, chemical, and biological properties that change dramatically when the material dimension enters the nanometer scale. These properties are superior to the bulk material, thus giving more application opportunities. Through the mechanism of controlling size, chemical composition, surface modification and controlling the interactions between particles, these properties can be changed [2-4].

Magnetite (Fe$_3$O$_4$) nanomaterial is a material that has various advantages, including: superparamagnetic and high magnetic saturation. The size reduction of particles can make nanomaterial products have different properties, so as to improve the quality of the material. Magnetites that are on a nanometer scale have properties that depend on their size. Therefore, how to synthesize uniform nanoparticles by adjusting their size is one of the key problems in the scope of nanoparticle synthesis [5].

The particle size determines the magnetic properties, where the smaller the grain size, Fe$_3$O$_4$ nanoparticles will have a high magnetic responsibility (easily magnetized by external magnetic fields). In other words, the superparamagnetic effect will be more dominant along with the smaller diameter of Fe$_3$O$_4$ nanoparticles [6].
Fe$_3$O$_4$ is one phase of iron oxide, which is amphoteric and has high absorption. Fe$_3$O$_4$ (FeO.Fe$_2$O$_3$) have spinel inversion structures and contain Fe$^{2+}$ and Fe$^{3+}$ ions [7]. In its structure, oxygen ions share with metal ions. The large distance between metal ions causes an exchange between metal ions through oxygen ions. The transfer of electrons between Fe$^{2+}$ and Fe$^{3+}$ at the tetrahedral and octahedral sites in Fe$_3$O$_4$ causes it to have unique electrical and magnetic properties. Fe$_3$O$_4$ is abundant in iron sand in West Sumatra, Indonesia and has been processed into Fe$_3$O$_4$ thin film [8-9]. In this article, we discuss the magnetic properties of Fe$_3$O$_4$ nanoparticles that have been synthesized from iron sand minerals in West Sumatra.

2. Experimental
Iron sand that has been taken from the location of Tiram beach, West Sumatra, then separated from the rubbish and other impurities. The iron sand that has been cleaned is then crushed by lumping for 3 hours. The iron sand that has been finely then sifted, aims to get the same grain size. Purification of iron sand is carried out in a physical route for the synthesis of Fe$_3$O$_4$ nanoparticle as reported in [8, 9]. The iron sand samples that have been separated from the residue are then milled using High Energy Milling with the ratio of balls to iron sand is 1:10. The milling time is varied 10, 15, 20, 25 and 30 hours. All samples were characterized by X-ray diffraction (XRD, type X’PERT POWDER PW 30/40, CuK$\alpha$), scanning electron microscopy (Hitachi SEM model TM-3000) and vibrating sample magnetometer (VSM, type VSM250).

3. Result and discussion
Formation of Fe$_3$O$_4$ nanoparticles from natural iron sand using the milling method is known from XRD and SEM characterization. Figure 1 shows the XRD pattern and SEM image of iron sand samples after milled for 30 hours. It is seen that after milling time 30 hours there is only Fe$_3$O$_4$ phase. The loss of the hematite phase is caused by a high-energy milling process that can increase the reaction kinetics associated with magnetite formation from hematite [10]. The crystalline size of Fe$_3$O$_4$ nanoparticles for each variation of milling time 30 hours is 20.5 nm. The particle size of Fe$_3$O$_4$ nanoparticles is 1.47 $\mu$m.

Figure 2 presents the magnetization hysteresis loops of Fe$_3$O$_4$ nanoparticle at room temperature. The saturation magnetization ($M_s$) values of Fe$_3$O$_4$ nanoparticle are influenced by the milling time. Samples milled for 10, 15, 20, 25 and 30 hours showed saturation magnetization of 33.85, 32.12, 30.19, 27.89, and 25.10 emu/g, respectively.

It appears from figure 2, the milling time increases causing saturation magnetization to decrease. These results are consistent with the studies reported in [10], which use coarse metallic Fe particles as starting material. The tendency to decrease in saturation magnetization is due to a decrease in the number of Fe phases and changes in particle size as a result of an effect of milling time [10].
Figure 2. Hysteresis loop for Fe$_3$O$_4$ nanoparticle for different milling time.

4. Conclusion
Fe$_3$O$_4$ nanoparticles have been synthesized from natural iron sand as raw material. Through a 30-hour milling process, Fe$_3$O$_4$ nanoparticles with crystal sizes of around 25.5 nm have been obtained. It was found that the milling time influenced the magnetization of Fe$_3$O$_4$ nanoparticles. The saturation magnetization of the samples for 10, 15, 20, 25 and 30 hours are 33.85, 32.12, 30.19, 27.89, and 25.10 emu/g.

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