Nanopowder and Magnetic Fluid Synthesis of Zn$_{0.2}$Fe$_{2.8}$O$_4$ Particles and Their Structural and Magnetic Behaviors

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Abstract: In this paper, the authors report the fabrication of Zn$_{0.2}$Fe$_{2.8}$O$_4$ particles from Indonesian sand in the forms of nanopowder and magnetic fluid. The fabrication was conducted by employing a combined coprecipitation-sonochemical method. The analysis of the XRD data presented that the Zn$_{0.2}$Fe$_{2.8}$O$_4$ was crystallized in a spinel structure with a lattice parameter of about 8.378 Å. The data analysis of the TEM image showed the Zn$_{0.2}$Fe$_{2.8}$O$_4$ particles distributed in nanometric size with a spherical shape. The magnetic investigation showed that the magnetic nanopowder and fluid both had superparamagnetic character. Moreover, the band gap of the Zn$_{0.2}$Fe$_{2.8}$O$_4$ particles was about 2.28 eV. These characters open a significant opportunity for the prepared samples to be applied for sensory and antibacterial applications.

Keywords: nanopowder, magnetic fluid, Zn$_{0.2}$Fe$_{2.8}$O$_4$, spinel, superparamagnetic.

1. Introduction

Nanotechnology research using the material of iron oxide has frequently been performed for the past few years to produce a material with an optimum magnetic response. One example material of iron oxide is magnetite (Fe$_3$O$_4$) nanoparticle that has been developed for many applications due to its excellent magnetic response showing superparamagnetic properties [1]. However, unfortunately, nanometric Fe$_3$O$_4$ particles have a lower saturation magnetization compared to bulk Fe$_3$O$_4$ [2]. Therefore, the magnetization of Fe$_3$O$_4$ particles could be improved by performing a transitional metal substitution forming a (M,Fe$_{1.2}$)$_{3-x}$(Fe$_{2.8}$)$_x$O$_4$ system where M could be Cu [3], Mn-Co [4], Ni-Zn [5], and Mg [6] with $x$ as substitutional fraction notation.

Zink (Zn) as one of the metal substitutions is superior in improving the performance of the Fe$_3$O$_4$ particles. In the form of Zn-ferrites (Zn$_x$Fe$_{3-x}$O$_4$), the material is biocompatible with a low level of toxicity [7], having relatively high capacitance [8], transmission and band gap [9], and showing a paramagnetic property at room temperature [10]. The previous report showed that the substantial fraction of $x = 0 - 0.4$ for the Zn$_{x}$Fe$_{3-x}$O$_4$ nanoparticle could maintain the inverse spinel distribution [11] and its superparamagnetic properties [3]. Other reports presented that the highest saturation magnetization was obtained at $x = 0.2$ forming a Zn$_{0.2}$Fe$_{2.8}$O$_4$ system in the forms of nanopowder and ferrofluid.
Its excellent magnetic property makes ferrofluid become one of the critical materials to be developed, especially to vary its applications. The most recent report shows that ferrofluid could be applied as a hyperthermia treatment agent [7], solar cell photovoltaic system [12], energy harvester [13] and even as an electromagnetic hybrid generator [14]. In this research, the Zn$_{0.2}$Fe$_{2.8}$O$_4$ ferrofluid was fabricated from Zn$_{0.2}$Fe$_{2.8}$O$_4$ nanoparticles dispersed in water (H$_2$O) as a carrier medium and TMAH (C$_4$H$_{13}$NO) as a surfactant. Interestingly, the natural Fe$_3$O$_4$ from Indonesian iron sand was employed as a main source. Such nanomagnetic particle is layered by the surfactant to result in an active surface that could efficiently bind the surface of the dispersant medium in producing the homogeneous colloidal suspension with good superparamagnetic characteristics.

2. Experimental Method
The purified iron sand was dissolved in HCl using a magnetic stirrer at ambient temperature to prepare the Zn$_{0.2}$Fe$_{2.8}$O$_4$ particles. In order to separate the non-dissolved solid after the chemical reaction, the chloride iron solution was filtered and then added with ZnCl$_2$ that has been diluted by the stoichiometry to produce the Zn$_{0.2}$Fe$_{2.8}$O$_4$ particles. Such mixture was then reacted with NH$_4$OH via a coprecipitation process under the effect of ultrasonic radiation. The reaction of the Zn$_{0.2}$Fe$_{2.8}$O$_4$ particles fabrication is shown in Equation 1.

$$2\text{FeCl}_3 + \text{FeCl}_2 + 0.21\text{ZnCl}_2 + 8.42\text{NH}_4\text{OH} \rightarrow 1.07\text{Zn}_{0.2}\text{Fe}_{2.8}\text{O}_4 + 8.43\text{NH}_4\text{Cl} + 8.21\text{H}_2\text{O}$$

The sediment of the Zn$_{0.2}$Fe$_{2.8}$O$_4$ particles was then neutralized through washing technique. The sediment was then calcined to produce the sample in nanopowders. In general, the synthesis procedure was maintained by following our previous work [15]. The fabrication of sample in the form of Zn$_{0.2}$Fe$_{2.8}$O$_4$ ferrofluid was performed by dispersing in the water the Zn$_{0.2}$Fe$_{2.8}$O$_4$ particle sediment in C$_4$H$_{13}$NO. Subsequently, the sample was characterized using XRD, SEM, FTIR, and VSM.

3. Results and Discussion
In Fig. 1, the TEM image exhibits that the Zn$_{0.2}$Fe$_{2.8}$O$_4$ particle has a spherical shape with a mean particle size of 12.6 ± 0.4 nm with an inhomogeneity level of 3.4 %. Another research group reported that the magnetic nanoparticles prepared by a chemical combustion method and using Tween 80 as a surfactant produced the particle size of 36.5 nm with an inhomogeneity level of 52 % [16]. It means that this work has successfully provided the magnetic nanoparticles in powders. It also indicates that without a surfactant, the ultrasonic irradiation during sonochemical process successfully formed magnetic particles with a narrower size and high homogeneity. The smaller particle size of the magnetic particles was resulted by the micro-emission process of the ultrasonic field during the sonication process that produced local heat and bubble cavitation in the material. Therefore, the obtained smaller particles were originated from the cavitation process and the local heat that increases the crystal growth of the magnetic particles [17]. Interestingly, the prepared Zn$_{0.2}$Fe$_{2.8}$O$_4$ particles in this work, produced by a combination sonochemical-coprecipitation method have a particle size that was smaller than that of the Zn$_{0.2}$Fe$_{2.8}$O$_4$ produced by a coprecipitation method [18].
The formation of the Zn$_{0.2}$Fe$_{2.8}$O$_4$ particles was analyzed based on the FT-IR graphic pattern shown in Figure 2. The FT-IR spectrum at wavenumbers of ~1635 cm$^{-1}$ and 3441 cm$^{-1}$ representing the vibration of the hydroxyl functional group for symmetrical hydroxyl bond [19] and asymmetrical hydroxyl bond [20] while the weak ~2350 cm$^{-1}$ spectrum was CO$_2$ binding group [21]. The transmittance peak at a wavenumber of ~402 cm$^{-1}$ was the vibration of the metal-oxygen (M-O) bond at an octahedral position [22]. Further, the Zn-O-Fe bond at the tetrahedral position was broken into two transmittance peaks, namely ~655 cm$^{-1}$ and ~1340 cm$^{-1}$ that indicated a successful substitution of Fe ion by Zn ion occupying a tetrahedral position. Therefore, it shows that the Zn ion substitution in Fe$_3$O$_4$ that formed the Zn$_{0.2}$Fe$_{2.8}$O$_4$ system still maintain its inverse spinel crystal structure.

Figure 2. FTIR spectrum of Zn$_{0.2}$Fe$_{2.8}$O$_4$ nanoparticles

In this work, a further structural characterization was conducted via XRD using Cu-K$\alpha$ ($\lambda = 0.15406$ nm). The diffraction pattern of the Zn$_{0.2}$Fe$_{2.8}$O$_4$ nanoparticles is shown in Figure 3a, and its crystal structure is shown in Figure 3b. The figure presents the $hkl$ planes of (220), (311), (400), (422), (511), and (440) showing a cubic structure with the space group $F d \cdot 3 m$ [23]. The refinement analysis via
Rietveld method using AMCSD 7766 as a model data presented that the \( \text{Zn}_x\text{Fe}_{3-x}\text{O}_4 \) nanoparticles have an inverse cubic spinel structure with an ionic distribution that is expressed in the system of \([\text{Zn}_{1-x}^{2+}\text{Fe}^{3+}_x]_A[\text{Fe}_{2-x}^{3+}]_B\text{O}_4\). The A symbol represents the tetrahedral position, and the B symbol represents the octahedral position. The XRD data analysis coincides with the analysis of the FT-IR data as discussed above. Another investigation to analyze the elemental composition of the sample was done using XRF. The data presented that the sample has elemental contents of Zn: Fe of about 0.6:1. A further calculation of the mass fraction of Zn and Fe in the sample was 1/3 for \([\text{Zn}]/[\text{Zn}+\text{Fe}]\) and 2/3 for \([\text{Fe}]/[\text{Zn}+\text{Fe}]\). Thus, the distribution of the inverse spinel can be expressed by \([\text{Zn}_{1/3}\text{Fe}_{2/3}]_A[\text{Fe}_{5/3}]_B\text{O}_4\).

\[
\frac{k\lambda}{\beta \cos \theta} = D
\]

Where \( k = \) lattice constant (0.98 for cubic cell), \( \lambda = \) wavelength (0.5406 nm), \( \beta = \) full width half at the maximum intensity/FWHM (rad.), and \( \theta = \) Bragg’s peak angle [24].

**TABLE 1. Lattice parameters and particle size of the \( \text{Zn}_{0.2}\text{Fe}_{2.8}\text{O}_4 \) nanoparticle**

| Data       | Particle size (nm) | Unit cell volume (Å³) |
|------------|--------------------|-----------------------|
| \( \text{Zn}_{0.2}\text{Fe}_{2.8}\text{O}_4 \) | \( 8.378 \) 12.65 | \( 11.95 \) 588.13 |

The UV-Vis data of the \( \text{Zn}_{0.2}\text{Fe}_{2.8}\text{O}_4 \) particle that were analyzed by using a Tauc Plot are shown in Figure 4. The Tauc Plot exhibits information of optical parameter of the sample associating with optical band gap. The value of energy band gap depends on crystal structure, thermal effects, and also defects.
Zn doping substitution could cause defects in the crystal due to the radius difference between Zn and Fe. A Zn substitution in Fe$_3$O$_4$ formed Zn$_{0.2}$Fe$_{2.8}$O$_4$ nanoparticles that produced an energy band of 2.28 eV of which value was higher than the Fe$_3$O$_4$ nanoparticle, namely 2.2 eV [26]. This phenomenon is caused by the Zn substitution that has electron affinity (15 Kj/mol) lower than that of Fe (0 Kj/mol), which lead to locating the energy level in the bottom of the conduction band [9]. Based on the quantum mechanics, the extra energy is required by valence electrons leading to an increase in the energy band gap to excite to higher energy states in the conduction band. Another group reported that the magnetic nanoparticles with the energy of 2.2 ± 0.2 eV could be used as photocatalytic agent producing photocatalytic activity of up to 96.7 % [27]. Therefore, the sample in this work that has energy band gap of 2.28 eV opens a significant potential for the application as a photocatalytic agent in the visible light spectrum.

The hysteresis (M-H) curves are presented in Figure 5. The figures show the respective circles and solid lines representing the experimental data and fitting model using Langevin’s equation as shown in Equation 3 [28]. The data analysis presents the saturation magnetization ($M_s$) values of the Zn$_{0.2}$Fe$_{2.8}$O$_4$ powder and ferrofluid are 33.530 ± 0.040 and 1.263 ± 0.003 emu/g, respectively. Figure 5 also presents the S shape of the Zn$_{0.2}$Fe$_{2.8}$O$_4$ powder and ferrofluid that represent the superparamagnetic characters of the samples [29].

$$L(x) = M_s \left( \coth \left( \frac{Bx}{k_B} \right) - \frac{1}{Bx} \right)$$

(3)

Where $B = \mu/k_B T$, $x = H$, and $M_s$ is saturation magnetization.

Theoretically, the Zn$^{2+}$ substitution changes the ionic distribution in the spinel structure of Fe$_3$O$_4$ and leads to a change in the magnetic behaviors of the samples. In the system of Zn$_{0.2}$Fe$_{2.8}$O$_4$, the Zn$^{2+}$ ions may replace the Fe$^{3+}$ ions in tetrahedral position. As a consequence, the Fe$^{3+}$ ions in octahedral changed to Fe$^{3+}$ ions. The magnetic moments in the octahedral site are antiparallel with the tetrahedral site. The imbalance metal ions between the octahedral and tetrahedral sites resulted in some unpaired magnetic spins producing net magnetic moments that can be used to calculate the saturation magnetization of the spinel system [30]. Our previous works presented that the magnetic behaviors of the magnetic nanoparticles in the powders and ferrofluids are also affected by the metal ion composition, primary and secondary particle sizes, fractal structures, and nanoclusters of the samples [29,31].
FIGURE 5. Hysteresis curve of superparamagnetic Zn$_{0.2}$Fe$_{2.8}$O$_4$ in (a) nanopowder (b) ferrofluid

The characteristic of ferrofluids is a combination of liquid character and magnetic nanoparticles that are easy to be controlled by an external magnetic field. In the magnetic fluids, the saturation magnetization is lower than that of nanopowders originating from the effect of viscosity of the fluids. On powders, the magnetic particles did not have any liquid carriers that differ from the ferrofluids coated by C$_4$H$_{13}$NO dispersed in H$_2$O. The effect of magneto-viscous explains that the high viscosity of the ferrofluids leads to a reduction in the thermal effect [32]. Under external magnetic field, the low thermal energy decreases the Brownian motion of the particles in the ferrofluids and reduces the magnetic response. The low saturation magnetization of the ferrofluids is also affected by the chemical reaction between the surfactant and magnetic particles. Moreover, the spin canting in the surface and pinned spins that were close to the surface also provided a further explanation of why the saturation magnetization of the ferrofluids was lower than that of the nanopowders [33].

The higher magnetic moment of ferrofluids leads to an increase in the aggregation as the effect of van der Waals forces and dipoles interaction [34]. Using a small-angle neutron scattering, the ferrofluids form an aggregation in a one dimension chain-like structure as reported in the previous work [35]. Under an external magnetic field, the chain-like structure builds pre-existing chains. Despite resulting from a single domain particle, the saturation magnetization of ferrofluids also results from the magnetization of the pre-existing chain particles [36]. Magnetic moments of the ferrofluids build a symmetric fan arrangement along the chain axis. Therefore, the magnetizing and demagnetizing processes in the M-H curve of the ferrofluids showed the quasi-magnetic hysteresis that differs from the M-H curve of the nanopowders as shown in Figure 5.

4. Conclusion
The Zn$_{0.2}$Fe$_{2.8}$O$_4$ particles form an inverse cubic spinel structure in a nanometric size and a spherical shape. The combined sonochemical-coprecipitation method has succeeded in producing the Zn$_{0.2}$Fe$_{2.8}$O$_4$ particles with high homogeneity. Both of Zn$_{0.2}$Fe$_{2.8}$O$_4$ nanopowder and ferrofluid exhibit superparamagnetic phenomena with a saturation magnetization of 33.530 ± 0.040 emu/g, and 1.263 ± 0.003 emu/g, respectively. All samples exhibited superparamagnetic behaviors at room temperature. Furthermore, the prepared samples with energy band gap of 2.28 eV open a significant potential application as a photocatalytic agent.

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