Comparative Structural Properties of Nanosized ZnO/Fe₃O₄ Composites Prepared by Sonochemical and Sol-Gel Methods

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Abstract. In this research, the synthesis of ZnO/Fe₃O₄ nanocomposites was conducted using sonochemical and sol-gel methods as well as natural materials as the primary raw material. The results of X-ray diffraction data analysis showed that the synthesis of nanocomposite used sonochemistry method which resulted in the sample with ZnO and Fe₃O₄ phases without the presence of the others. Interestingly, the sol-gel method resulted in a sample with ZnFe₂O₄ and α-Fe₂O₃ phases without the presence of ZnO. This case happened since Zn formed oxide compound in the form of spinel zinc ferrite. By using the Scherrer equation, the crystallite sizes were of about 7.5 nm for Fe₃O₄, 25.5 nm for ZnO, 13.6 nm for α-Fe₂O₃, and 11.6 nm for ZnFe₂O₄. The elemental compositions of the sample as the result of synthesis using sonochemistry method were 33.08% for Fe and 66.91% for Zn. Meanwhile, the elemental compositions of the sample as the result of synthesis using the sol-gel method were 58.25% or Fe and 41.75% for Zn. Moreover, the infra-red spectrum showed that the functional group of the Fe₃O₄ phase was observed on the wavenumber of 550 cm⁻¹ and 676 cm⁻¹; the functional group of ZnO phase was found on the wavenumber of 433 cm⁻¹; the functional group of α-Fe₂O₃ was detected on the wavenumber of 479 cm⁻¹ and 551 cm⁻¹; and the functional group of ZnFe₂O₄ was recorded on the wavenumber of 526 cm⁻¹. Thereby, the sonochemical method in this study provides a new alternative in the synthesis of ZnO/Fe₃O₄ nanocomposite using natural material as the primary raw material.

Keywords: ZnO, ZnFe₂O₄, Fe₃O₄, α-Fe₂O₃, nanocomposite, sonochemistry, sol-gel.

1. Introduction

In some recent decades, the composite material has been known as an excellent material in many applications. The application of composite material still develops and becomes one of the materials that dominate the scientific technology. Conceptually, the nanocomposite material is the combination of nanomaterial consisting of two or more than two different nanocomposite materials to strengthen (fiber, particle, annular film, and filler) in the matrix (polymer, metal, and ceramics) [1]. Therefore, the choice of nanocomposite synthesis method becomes a significant thing especially in controlling material properties [2].

One of the materials that are exciting to be investigated related to the synthesis as a composite is oxide zinc (ZnO) including the study of its application in various fields. This case is generally associated with the excellence of ZnO properties which are semiconductor material with the high

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stability [3], biocompatibility [4], excitation binding energy of 60 eV [5,6] and categorized into five compounds considered safe by U.S Food and Drug Administration [7,8]. Such excellences expand the application of ZnO in diverse fields as photovoltaic [9], electronic, medical science, cosmetics [10], antibacterial [7] and anti-fungal [11]. However, for the aim of a particular application that needs right magnetic property, ZnO needs to be combined as a composite with the other materials especially magnetic-based nanomaterial.

In recent years, magnetic nanoparticles, especially Fe₃O₄ are one of the nanomaterials playing a significant role in many applications, especially medical application. In general, magnetic nanoparticles have many benefits such as their unique magnetic properties, biocompatible, [12], biodegradable [13], and non-toxic [14]. Therefore, the magnetic nanomaterial is often applied as targeted drug delivery [15], magnetic resonance imaging [16], hyperthermia therapy [17], biosensor [18], antibacterial and antifungal agents [19,20]. Thereby, the excellence of Fe₃O₄ and ZnO properties provides a big chance to be combined in a composite so that it has a more awesome performance for the subsequent application. Theoretically, the combination of both materials to form composite is one of the great strategies that do not disturb each material, but they complement each other and strengthen the properties of both.

In the previous works, the synthesis of ZnO/Fe₃O₄ nanocomposites could be obtained using precipitation, [21], co-precipitation [22], deposition [23], wet milling [24], solvothermal [25], and wet chemical route [26] methods. However, the material as the synthesis results of those methods still revealed agglomeration. That is why, in this research, we proposed the development of synthesis method of ZnO/Fe₃O₄ nanocomposites using sonochemical and sol-gel methods by adding diethylamine surfactant to control the size and distribution of the particles [27]. The choice of the sonochemical method was because this particular method is known as a method that is short time consuming, efficient to gain nano-scale particles with great properties [28]. Besides, the sol-gel method was also selected due to its relatively low-cost, more manageable process, and good control ability [1]. The other benefits in this research are the use of cheap natural material, and it is easily accessed in the form of iron sand as the primary raw material of synthesis using both methods.

### 2. Methods

The materials used were NH₄OH, HCl, zinc acetate dihydrate, natural iron sand of Indonesia, diethylamine, Ethanol, and distilled water. The synthesis of ZnO/Fe₃O₄ nanocomposites using sonochemical method was initiated by reacting Fe₃O₄ powder obtained from natural sand of Indonesia with HCl added with diethylamine. Subsequently, the result of the reaction was dropped with NH₄OH to result in a precipitate followed by a drying process to obtain Fe₃O₄ nanoparticles. Fe₃O₄ nanoparticles were dispersed in distilled water and reacted with zinc acetate dihydrate dissolved while stirred using magnetic stirrer at room temperature. In the stirring process, the mixture was titrated with NH₄OH so that the precipitate was formed. The precipitate was then washed using distilled water until its pH was normal, filtered using filter paper continued by a drying process to attain ZnO/Fe₃O₄ nanocomposites. Meanwhile, the synthesis of ZnO/Fe₃O₄ nanocomposites using sol-gel method began by reacting Fe₃O₄ powder from beach sand with HCl followed by a titration process with NH₄OH for precipitation. The washing process was also carried out to obtain Fe₃O₄ nanoparticles followed by the dispersion process in ethanol. At the same time, the zinc acetate dihydrate solution was also added through a reaction using magnetic stirrer at the room temperature. In this process, the titration with diethylamine and ethanol was done continually so that the precipitate was formed. Lastly, the precipitate was filtered and dried to produce ZnO/Fe₃O₄ nanocomposites. Both samples obtained from both synthesis methods were characterized using instruments in the form of X-ray diffractometer (Philips X’Pert pro). It was undertaken to know the crystal phase and structure using Cu-Kα 1.54060 Å source. Also, to see the composition of the sample elements, the X-ray fluorescence spectroscopy (PANalytical) was executed. Meanwhile, Fourier transform infrared spectroscopy (Shimadzu) was done to identify the functional group of the material. The data analysis was carried out qualitatively and quantitatively using a fitting method.
3. Results and Discussion
Study on X-ray diffraction was carried out using X-ray diffractometer with the radiation of $\lambda = 1.54060$ Å in the range of $20^\circ - 70^\circ$ to determine the crystal structure and phase. Figure 1 shows the diffraction pattern of the X-ray and the fitting analysis result of ZnO/Fe$_3$O$_4$ nanocomposites prepared by sonochemical and sol-gel methods. Fitting results are represented with a red diffraction pattern. Meanwhile, the diffraction data as the experiment results are represented with a black diffraction pattern. Fitting analysis was conducted by refinement technique using the data model of ICSD no 30860 for Fe$_3$O$_4$, AMCSD no 0005203 for ZnO, AMCSD no 0017806 for $\alpha$-Fe$_2$O$_3$, and AMCSD no 9002499 for ZnFe$_2$O$_4$.

Figure 1. X-ray diffraction patterns of ZnO/Fe$_3$O$_4$ nanocomposites synthesized by (a) sonochemical and (b) sol-gel methods

Diffraction pattern model (counting data) has excellent suitability for the diffraction pattern data as the experiment result (measured data) of ZnO/Fe$_3$O$_4$ nanocomposites. The diffraction peaks of ZnO were detected on the angle of $2\theta = 31.7^\circ$, 34.4$^\circ$, 36.2$^\circ$, 47.5$^\circ$, 56.6$^\circ$, 62.8$^\circ$, 66.3$^\circ$, 67.9$^\circ$, and 69.1$^\circ$ that respectively represents the $hkl$ plane of (100), (002), (101), (102), (110), (103), (200), (112), and (201). Meanwhile, the diffraction peaks of Fe$_3$O$_4$ were observed on the angle of $2\theta = 30.3^\circ$, 35.6$^\circ$, 43.3$^\circ$, 53.7$^\circ$, 57.3$^\circ$, and 62.9$^\circ$ with the $hkl$ plane of (022), (113), (004), (224), (115), and (044) [28-30]. These analysis results showed that the sample as the synthesis results had a high crystal phase purity. These results could be compared to the results of the X-ray fluorescence data as presented in Table 1. It is found that the elemental composition of Fe and Zn are 33.08 % and 66.91%, respectively.

The results of data analysis of X-ray diffraction of ZnO/Fe$_3$O$_4$ composites as the synthesis results using sol-gel method showed the form of spinel ferrite phase in the form of ZnFe$_2$O$_4$ and $\alpha$-Fe$_2$O$_3$. Physically, the presence of the ZnFe$_2$O$_4$ phase was caused by Zn atom replacing Fe [31]. Besides, the $\alpha$-Fe$_2$O$_3$ phase was predicted would appear related to the oxidation process of Fe$_3$O$_4$ during the drying process [1]. The diffraction peaks of $\alpha$-Fe$_2$O$_3$ were observed on the angle of $2\theta = 33.2^\circ$, 53.6$^\circ$ and 59.4$^\circ$ with the $hkl$ plane of (110), (224), and (010) [32]. Meanwhile, the diffraction peaks of ZnFe$_2$O$_4$ were observed on the angle of $2\theta = 30.2^\circ$, 35.7$^\circ$, 43.3$^\circ$, 57.3$^\circ$, and 63.0$^\circ$, with the section of $hkl$ (022), (113), (004), (115) and (044) [33]. Regarding this case, the result of the XRF characterization presented in Table 1 also showed that the form of the compound with Fe and Zn elements was about 58.25% and 41.75%. Quantitatively, the analysis result of X-ray diffraction data fitting resulted in some crystal parameters displayed in Table 2.
To ensure that both samples were formed in the nanometric size, this research analyzed the crystallite size using Scherrer equation, primarily related to the highest diffraction peak for each phase. Based on Figure 1, the highest intensity of diffraction peaks of each phase was observed. For example, the diffraction peaks were 35.6°, 36.2°, 33.2°, 35.7° of Fe$_3$O$_4$, ZnO, α-Fe$_2$O$_3$, ZnFe$_2$O$_4$ respectively. Based on the data analysis results, the crystallite sizes resulted in this research were about 7.5 nm for Fe$_3$O$_4$, 25.5 nm for ZnO, 13.6 nm for α-Fe$_2$O$_3$, and 11.6 nm for ZnFe$_2$O$_4$. Thereby, it can be concluded that sonochemical and sol-gel methods were successful in creating nanocomposites based on ferrites using the raw material of iron sand. Nevertheless, in the sol-gel method, the synthesis parameter should be optimized to form ZnO so that the Zn atom did not replace the Fe atom.

**Table 1.** Elemental composition of ZnO/Fe$_3$O$_4$ nanocomposites

| Elemental content | Sonochemical method (%) | Sol-gel method (%) |
|-------------------|-------------------------|--------------------|
| Fe                | 33.08%                  | 58.25%             |
| Zn                | 66.91%                  | 41.75%             |

**Table 2.** The refinement results for X-ray diffraction data of ZnO/Fe$_3$O$_4$ nanocomposites

| Parameter | Sonochemical method | Sol-gel method |
|-----------|---------------------|----------------|
| Space group | ZnO | Fe$_3$O$_4$ | α-Fe$_2$O$_3$ | ZnFe$_2$O$_4$ |
| $a$, $b$, $c$ (Å) | $a = b = 3.250$ | $a = b = c = 8.353$ | $a = b = 5.399$ | $a = b = c = 8.378$ |
| $a$, $b$, $c$ (Å) | $c = 5.208$ | $c = 12.69$ | |
| $α$, $β$, $γ$ | $α = β = 90°$ | $α = β = γ = 90°$ | $α = β = 90°$ | $α = β = γ = 90°$ |
| Crystallite size (nm) | 25.5 | 7.5 | 13.6 | 11.6 |

**Figure 2.** Crystal structures of (a) Fe$_3$O$_4$, (b) ZnO, (c) α-Fe$_2$O$_3$, and (d) ZnFe$_2$O$_4$
Visually, the 3D crystal structures of ZnO/Fe₃O₄ nanocomposites prepared by sonochemical and sol-gel methods are shown in Figure 2. Figure 2 (a) is a crystal structure of Fe₃O₄. This structure informs that the octahedral site was occupied by Fe²⁺ ions and a half of that part was occupied by Fe³⁺ ions, while the tetrahedral part was occupied by Fe³⁺ ions [34]. Figure 2 (b) is a crystal structure of ZnO showing two sublattices related to each other including Zn²⁺ and O²⁻ so that each Zn ion was surrounded by tetrahedral ions and vice versa [35]. Subsequently, Figure 2 (c) is the crystal structure of α-Fe₂O₃. In this crystal structure, Fe³⁺ ion occupied two third of octahedral sites limited by hexagonal close-packed O lattice [36]. Meanwhile, figure 2 (d) identifies the crystal structure of ZnFe₂O₄. In this structure, Zn²⁺ cation occupied tetrahedral sites, and Fe³⁺ cation occupied octahedral sites [37].

Infrared spectra of ZnO/Fe₃O₄ nanocomposites characterized by FTIR spectroscopy in the range of the wavenumber of 400-4000 cm⁻¹ are shown in Figure 3. This characterization was used to investigate functional groups of the ZnO/Fe₃O₄ nanocomposites in both organic and inorganic bonds. The organic bond of the compound of ZnO/Fe₃O₄ nanocomposites is shown by the absorbance peak of 3430, 1647, and 1547 cm⁻¹ that represent vibration mode of O-H bond caused by the vibration of water absorbance and hydroxyl surface. This case is correlated to previous research where the vibration of O-H bond that recorded in the wavenumber of 3500 cm⁻¹ and 1628 cm⁻¹ [5,32]. Moreover, the vibration of H-O-H appeared in the wavenumber of 1630 cm⁻¹ [28]. In the wavenumber of about 1384 cm⁻¹ and 1630 cm⁻¹, there was a vibration of carboxyl bond stretch of symmetric and asymmetric zinc [38,39]. Furthermore, in the area around 1393 cm⁻¹ and 1587 cm⁻¹, the vibration of the carboxyl bond stretch of symmetric and asymmetric iron (C=O) appeared [1,12,40]. The appearance of the stretch vibration of this C=O bond was from reactive carbon during the synthesis process [39]. Furthermore, the vibration peak appeared on the wavenumber of 2376 cm⁻¹ that identified the form of CO₂ bond in the air [41–43].

![Figure 3. FTIR spectra of ZnO/Fe₃O₄ nanocomposites synthesized by (a) sonochemical and (b) sol-gel methods](image)

Characterization of the peak of Fe-O bond vibration is shown in the wavenumber of 550 cm⁻¹ and 676 cm⁻¹ that affirms the form of structure with the type of Fe₂O₄ spinel inverse [1,26,28,41,44,45], in the wavenumber of 465, 551, 420, and 490 cm⁻¹ that strengthens the form of α-Fe₂O₃ [32,46]. Meanwhile, the wavenumber of 526 cm⁻¹ identified the form of ZnFe₂O₄ [37]. The vibration of Zn-O bond was created in the wavenumber of about 426–440 cm⁻¹ and 453 cm⁻¹ [44,47]. The data of the functional groups of ZnO/Fe₃O₄ nanocomposites as the results of the experiment and comparison with many references of the previous research results are presented in Table 3.
4. Conclusion
In this research, the synthesis of ZnO/Fe₃O₄ nanocomposites was successfully conducted using sonochemical method proven by the results of XRD test related to the form of Fe₃O₄ and ZnO phases that had the crystallite size of 7.5 nm and 25.5 nm, respectively. Interestingly, the sample of the results of the synthesis using sol-gel method did not create ZnO/Fe₃O₄ nanocomposites. However, the sample formed α-Fe₂O₃ and ZnFe₂O₄ phases with a size of 13.6 nm and 11.6 nm, respectively, which identified the form of α-Fe₂O₃/ZnFe₂O₄ nanocomposites. Moreover, the results of functional group analysis showed that the Fe-O bond was detected on the wavenumber of about 550 cm⁻¹ and 676 cm⁻¹. Meanwhile, the functional group of Fe-O of α-Fe₂O₃ was observed on the wavenumber of 465, 551, 420, and 490 cm⁻¹. Meanwhile, in the wavenumber of about 426–440 cm⁻¹ and 453 cm⁻¹, the vibration of Zn-O bond was detected and believed originating from the ZnO. Furthermore, in the wavenumber of 526 cm⁻¹, the functional group of Zn-O was identified in ZnFe₂O₄.

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