Bonding analysis of magnetic nanoparticles (MNPS) zinc ferrite (ZnFe$_2$O$_4$) encapsulated with silica (SiO$_2$)

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Abstract. Magnetic nanoparticles have synthesized by method ZnFe$_2$O$_4$ coprecipitation with mixing ZnSO$_4$.7H$_2$O and FeCl$_3$.6H$_2$O at 90°C, stirring speed with 1000 rpm during 1 hour and NaOH as copresicipitan. FeCl$_3$.6H$_2$O is Ferric chloride hexahydrate or also called iron (III) chloride hexahydrate, or iron (III) chloride hexahydrate is a chemical compound in the form of solid and brownish-yellow or orange. Then encapsulated to modify the surface ZnFe$_2$O$_4$ with silica and considering the concentration of variation at 50%, 30%, 20%, 15%, 10%, and 5% after while encapsulated of silica tested using X-Ray Diffraction (XRD). XRD is an analysis method used to identify the crystalline phase in the material by determining the parameters of the lattice structure and to get particle size. XRD tested resulted, indicating a change of normal spinel structure turned into Hexagonal Close-Packed (HCP). FTIR is one of the tools used to analyze chemical compounds. FTIR Test results are used for functional group analysis and identifying absorption peaks. Test analysis of functional groups using a Fourier Transformation Infra-Red Spectroscopy (FTIR) identifies peak absorption, while the characteristic functional groups of ZnFe$_2$O$_4$ that are M-O, Zn-Otet, Fe-Oct, and Zn-O-Fe shifting towards wavenumber more high after encapsulated with silica due to lower vibrational energy due to absorption by the silica.

1. Introduction

The magnetic research nanoparticles currently being studied are ferrite nanoparticles. This is because ferrite nanoparticles have advantages compared to other materials. The benefits of ferrite nanoparticles include magnetization, low coercivity, and high anisotropic energy. Ferrite nanoparticles play an essential role in the fields of biotechnology and biomedicine. Nano ferrite particles are also used for imaging in vivo and hyperthermia therapy [1].

The type of magnetic nanoparticles attracted many researchers were zinc ferrite (ZnFe2O4). This is because ZnFe$_2$O$_4$ has soft-magnetic properties, has a normal spinel structure with Zn$^{2+}$ ions occupying the tetrahedral part, and Fe$^{3+}$ ions occupying the octahedral part [2]. Bulk ZnFe$_2$O$_4$ at 10$^5$K showed antiferromagnetic properties. Bulk-sized ZnFe$_2$O$_4$ will be paramagnetic at higher temperatures. Nano-sized ZnFe$_2$O$_4$ has superparamagnetic properties. ZnFe2O4 nanoparticles have the potential to be used in the medical field based on their properties as radiosensitizers in cancer therapy and are capable of destroying cancer cells by using external magnetic fields after
radiotherapy [3]. The fabrication process used to control particle size by synthesizing nanoparticles through the coprecipitation method. The coprecipitation method is a relatively simple procedure method and produces a relatively small grain size distribution. In addition, this method can also be done under ambient temperature conditions [4]. ZnFe$_2$O$_4$ nanoparticles have disadvantages including being easily oxidized and easily agglomerated. The process of magnetic nanoparticle encapsulation uses inorganic/organic, silica, and polymer molecules as changes due to its incompatibility properties [5]. The effect of ZnFe$_2$O$_4$ encapsulated with silica can reduced agglomeration, with a deficiency of TEOS (tetraethoxysilane), which is toxic, not environmentally friendly, and expensive [6]. In this study, ZnFe$_2$O$_4$ nanoparticles use different silica casters namely Na$_2$SiO$_3$ (sodium silicate solution) which has advantages such as not toxic, environmentally friendly, and economical. To see the bond between Na$_2$SiO$_3$ and ZnFe$_2$O$_4$ by analyzing its functional group. The benefit of this study informs that coastal communities of ZnFe$_2$O$_4$ nanoparticles can be used as an alternative drug for skin cancer therapy and silica encapsulation process carried out so that nanoparticles can be stored for a long time.

2. Research Methodology

The method used in this study is the coprecipitation method, which consists of 2 stages, including:

2.1 Synthesis of ZnFe$_2$O$_4$ Nano-Particles

The main material for the synthesis of ZnFe$_2$O$_4$ nanoparticles is ZnSO$_4$.7H$_2$O and FeCl$_3$.6H$_2$O as providers of Zn$^{2+}$ and Fe$^{3+}$ ions with a ratio of 1:2 mole fraction.

\[
\text{Zn}^{2+} + 2\text{Fe}^{3+} + 8\text{OH}^- \rightarrow \text{ZnFe}2\text{O}4 + 4\text{H2O} \quad (1)
\]

Synthesis by coprecipitation method by mixing; 1,149 grams of ZnSO$_4$.7H$_2$O with 20 ml distilled water distiller for 2 minutes until homogeneous, do the same for 2,161 grams of FeCl$_3$.6H$_2$O. Mix the two solutions and add 3.57 ml of HCL distiller for 5 minutes. Then mix the solution into the NaOH solution gradually while stirring using a magnetic stirrer for 1 hour at a speed of 1000 rpm at 90°C. Next to the deposition stage to get a sample, the solution is deposited with the help of an external field (permanent magnet) to make the settler faster. Then Deposition of washing six times using distilled water so that salts are in a mixture of dissolved take part so that a pure sample of ZnFe$_2$O$_4$ is obtained. ZnFe$_2$O$_4$ is then dried with a furnace at 90°C. The dried samples obtained were then encapsulated using silica.

2.2 The Process Of Encapsulating ZnFe$_2$O$_4$ Nano Particles Using Silica

ZnFe$_2$O$_4$ nanoparticles are encapsulated into 15 ml silica, which has different concentrations according to table 1. Silica solution is formed from sodium silicate which is added with distilled water than in the stirrer for 30 minutes at room temperature. ZnFe2O4 nanoparticles in solution included 0.6 grams of silica and distiller for 5 hours at room temperature so mixed, and there is a bond between ZnFe2O4 with silica nanoparticles. The deposition stage is carried out with an external magnetic field (permanent magnet). Then the sediment was washed five times. The drying stage is left at room temperature to dry. The sample then test the crystal structure, grain size, and functional group using XRD and FTIR.

Table 1. Identification of samples with varying concentrations of silica

| Percentage (%) | Comparison ZnFe2O4: Solution |
|---------------|------------------------------|
| 50            | 0.6:50%SS (15 ml SS+15 ml aquades) |
| 30            | 0.6:30%SS (9 ml SS+21 ml aquades) |
| 20            | 0.6:20%SS (6 ml SS+24 ml aquades) |
| 15            | 0.6:15%SS (4.5 ml SS+25.5 ml aquades) |
| 10            | 0.6:10%SS (3 ml SS+27 ml aquades) |
| 5             | 0.6: 5%SS (1.5 ml SS+28.5 ml aquades) |
3. Results And Discussion

3.1 The process fabricating results of the nanoparticles ZnFe₂O₄ before and after encapsulated using by silica.

In this study the synthesis of nanoparticles in the form of brownish deposits shown in Figure 1.a. These deposits are then dried using a furnace to obtain a slightly brownish-colored powder which is the typical color of ZnFe₂O₄ shown in figure 1.b:

![Figure 1](image_url)

**Figure 1.** a) The precipitation process ZnFe₂O₄ solution, b) Powder ZnFe₂O₄, c) deposition process ZnFe₂O₄ encapsulated solution with silica and d) ZnFe₂O₄ powder encapsulated with silica

Followed by the deposition process by washing five times, as shown in figure 1.c drying process at room temperature in figure 1.d.

3.2 Characterization ZnFe₂O₄ and ZnFe₂O₄ Encapsulated Silica Using XRD.

The following is a characterization of ZnFe₂O₄ and ZnFe₂O₄ encapsulated with silica using XRD shown in the following figure:

![Figure 2](image_url)

**Figure 2.** XRD pattern of the sample spectrum (a) ZnFe₂O₄ and (b) ZnFe₂O₄ encapsulated with 50% silica

The XRD ZnFe₂O₄ spectrum pattern before and after encapsulation with silica in figure 2. The results of ZnFe₂O₄ synthesis in figure 2.a are identified as peaks (220), (311), (511) and (440) which are in area 2θ around 31°, 35°, 6°, 37°, 9°, 43°, 7°, 57°, 8° and 62,9° as characteristics of the phase ZnFe₂O₄ have a normal spinel structure. After the encapsulation process with 50% silica concentration, diffraction of the new field appears. Other peaks (100), (101), (110), (102), (111), (003) and (103) are phase characteristics of silica which have a hexagonal Close-Packed (HCP) crystal structure.
Table 2. The results of distance analysis between fields and lattice parameters of ZnFe$_2$O$_4$ magnetic nanoparticles and encapsulation of ZnFe$_2$O$_4$ compounds using silica

| Sample Name         | d(Å) | α +Δα(Å) |
|---------------------|------|----------|
| ZnFe$_2$O$_4$       | 2.56 | 8.35±0.01|
| ZnFe$_2$O$_4$ + silica | 2.44 | 8.08±0.01|

Table 2 According to table 2 lattice parameters of nano scale magnetic particles before and after encapsulated ZnFe$_2$O$_4$ decreased, i.e. = 8.35 ± 0.01 Å be 8.08 ± 0.01 Å, appears to shift the main peak (311) due to the addition of silica nano particles into ZnFe2O4, causing electrostatic interactions [7]. The crystal grain size of nanoparticles using the Scherrer equation. Table 3 illustrates the size of pure ZnFe$_2$O$_4$ and encapsulated ZnFe$_2$O$_4$

Table 3. The results of crystal grain size analysis of pure ZnFe$_2$O$_4$ magnetic particles and encapsulation of ZnFe$_2$O$_4$ with silica

| Sample Name | Grain Size (nm) | Phase Ratio | Phase Ratio | Phase Ratio | Phase Ratio | Phase Ratio |
|-------------|-----------------|-------------|-------------|-------------|-------------|-------------|
|             |                 | ferrite     | α-Fe$_2$O$_3$ | γ-FeOOH | α-FeOOH | Fe$_2$O$_3$ |
| ZnFe$_2$O$_4$ | 8,33±0.01       | 82,49       | 17,51       | 0           | 0           | 0           |
| ZnFe$_2$O$_4$ + Silica | 23,51±0.02 | 52,45       | 7,16        | 17,83       | 8,15        | 9,53        | 57,33

Table 3 shows that the grain size of the particles after encapsulation increased from 8.33 ± 0.01 nm to 23.51 ± 0.02 nm. This increase occurs because silica is a template when it is encapsulated with ZnFe2O4 so that the particle size of the particles enlarges. Illustration of the silica encapsulation process in figure 3 below.

Figure 3. Silica encapsulation process

After ZnFe$_2$O$_4$ magnetic nanoparticles were encapsulated with silica, the main peak height (311) increased. So that the degree of crystallinity also decreases due to the presence of phase α-Fe$_2$O$_3$ which is a mineral hemaide, γ-FeOOH (lepidocrocite) and α-FeOOH (geotide) which are considered as impurity phases of nano particles ZnFe$_2$O$_4$ because they are antiferromagnetic [8]. As well as ferrimagnetic phase γ-Fe$_3$O$_4$ (maghemite) but because it is dominated by the antiferromagnetic phase FeOOH and α-Fe$_2$O$_3$ and para-magnetic silica phase so that the sample magnetic response decreases. The appearance of the FeOOH phase is caused by a reaction between hydrogen oxide and magnetic ions when surface adsorption occurs, which leads to a balance between complexes of complexion and coprecipitation.

3.3 Bonding analysis on ZnFe$_2$O$_4$, which has been functionalized with silica using FTIR.

Analysis of functional groups contained in the material was tested using FTIR. Figure 4 and Figure 6 shows the presence of absorption between ZnFe$_2$O$_4$, encapsulated ZnFe$_2$O$_4$, and pure silica.
Figure 4. FTIR (a) Silica spectrum, (b) sample (ZnFe₂O₄ coated 5% silica) (c) sample (ZnFe₂O₄ coated with 15% silica), (d) sample (ZnFe₂O₄ coated with 10% silica), (e) sample (ZnFe₂O₄).

Based on graph four can be interpreted the achievement of wave numbers and functional groups formed by each absorption that can be seen in the following table.

Table 4. ZnFe₂O₄ which is encapsulated with silica in a concentration variation of 5%, 10%, and 15%

| Functional Group | ZnFe₂O₄ | Silica | ZnFe₂O₄ : Silica |
|------------------|---------|--------|------------------|
|                  | Wave Number | 5% | 10% | 15% |
| O-H              | 3425.58 | 3410.15 | 3425.58 | 3433.29 |
| C-H              | 2931.8 | - | 2924.09 | 2924.04 |
| C-H              | 2368.59 | - | 2368.59 | 2337.72 |
| Si-O-Si          | - | 2276 | 2291.43 | - |
| H-O-H            | 1635.64 | 1651.07 | 1635.64 | 1635.64 | 1643.35 |
| C-O              | 1481.33 | - | 1481.33 | 1481.33 | 1489.05 |
| C-O              | 1350.17 | - | 1381.03 | 1381.03 | 1381.03 |
| Si-O-Si          | - | 1087.95 | 1049.28 | 1049.28 | 1049.28 |
| Si-OH            | 904.41 | - | - | - |
| Si-O-Si          | - | 794.67 | - | 779.24 | 779.24 |
| Zn-O tet         | 555.49 | - | 586.36 | 570.93 | 686.66 |
| Fe-O oct         | 424.34 | - | 455.2 | 455.2 | 455.2 |
| M-O              | 347.19 | - | 354.9 | 362.62 | 378.05 |

Based on the results of the 4th image FTIR spectrum and 10 ZnFe₂O₄ samples obtained the absorption peak with wave number 347.19 cm⁻¹; 393.19 cm⁻¹; 424.34 cm⁻¹; 555.49 cm⁻¹; 1350.17 cm⁻¹; 1635.64 cm⁻¹; 2368.59 cm⁻¹; 2931.8 cm⁻¹ and 3425.58 cm⁻¹. At wave number 3425.58 cm⁻¹ stretching vibrations occur absorption of O-H group (hydroxide) which is indicated as absorption of water vapor molecules. The presence of these absorption groups when nano particles have a high ratio of surface area and volume causes iron atoms as Lewis acids to tend to form covalent bonds with surrounding water molecules. At wave number 2368.59 cm⁻¹ and 2931.8, C-H absorption occurs with symmetrical stretching vibrations and stretching asymmetry indicated as carbon and hydrogen uptake due to the presence of impurity agents during the synthesis process [9] so that it causes carbon and hydrogen bonds in the sample. Regular with the C = O bond type vibration stretching wave number 1172.72 cm⁻¹ which is the impurity agent of the bond between carbon and oxygen. At wave number 1635.64 cm⁻¹, the absorption of the H-O-H group with bending vibration is indicated as water absorption caused by the condition of the sample that is not completely dry. Wave number
1350.17 cm\(^{-1}\) occurs in the absorption of the Zn-O-Fe group with a stretching vibration, which is the highest energy of the two elements between Zn and Fe \([10]\). At wave number 555.49 cm\(^{-1}\), there is absorption of the Zn-O tet (Zinc oxide) group stretching vibration with the tetrahedral position \([3]\). For wave number 424.34 cm\(^{-1}\) to 393.19 cm\(^{-1}\) there is absorption of Fe-O oct (Ferrite oxide) group with vibration stretching octetraheral position and wave number 347.19 cm\(^{-1}\) bending vibration occurs with M-O group absorption (methal oxide).

Based on figures 4 and 10 show that pure silica experiences vibrations at wave number transition 794.67 cm\(^{-1}\); 964.41 cm\(^{-1}\); 1087.85 cm\(^{-1}\); 1651.07 cm\(^{-1}\); 2337.72 cm\(^{-1}\) and 3487.3 cm\(^{-1}\). At wave number 794.67 cm\(^{-1}\) with stretching vibrations asymmetry is assumed to occur absorption of Si-O-Si functional groups. For wave number 964.41 cm\(^{-1}\) there is a contribution of O-H so that the group becomes Si-OH with bending vibrations. This absorption group is often called the silanol group because of the bond between Si and OH. Wave number 1651.07 cm\(^{-1}\) occurs Si-O-Si uptake with stretching vibrations asymmetry, namely siloxane groups with bonds between two silica and oxygen elements which can be seen in Figure 5 below.

Wave number 2337.72 cm\(^{-1}\) occurs in the absorption of Si-O-Si groups with vibration stretching symmetry. Whereas at wave number 3487.3 cm\(^{-1}\) there is O-H uptake with stretching vibration which is water absorption contained in silica material.

![Scheme II. Figure 5 luster silanol \([11]\)](image)

Wave number 3425.58 cm\(^{-1}\) to 3487.3 cm\(^{-1}\) occurs in the absorption of the Zn-O-Fe group with a stretching vibration, which is the highest energy of the two elements between Zn and Fe \([10]\). At wave number 2931.8 cm\(^{-1}\), there is absorption of the Zn-O tet (Zinc oxide) group stretching vibration with the tetrahedral position \([3]\). For wave number 2337.72 cm\(^{-1}\) to 2368.59 cm\(^{-1}\) there is absorption of Fe-O oct (Ferrite oxide) group with vibration stretching octetraheral position and wave number 2924.04 cm\(^{-1}\) bending vibration occurs with M-O group absorption (methal oxide).

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Table 5. ZnFe\(_2\)O\(_4\) which is encapsulated with silica at various concentrations of 20%, 30% and 50%.

| Functional Group | Wave Number (cm\(^{-1}\)) | Vibration Type |
|------------------|--------------------------|----------------|
| ZnFe\(_2\)O\(_4\) | 3425.58, 3487.3          | Stretching(H-bonded) |
| Silica 20%      | 2931.8                   | Stretching Symmetry |
| 3425.58, 3487.3 | 2931.8                   | Stretching Symmetry |
| 3425.58, 3487.3 | 2924.04                  | Stretching Symmetry |
| ZnFe\(_2\)O\(_4\) | 2337.72, 2368.59         | Stretching         |
| Silica 30%      | 2931.8                   | Stretching Symmetry |
| 2337.72, 2368.59| 2924.04                  | Stretching Symmetry |
| ZnFe\(_2\)O\(_4\) | 2337.72, 2368.59         | Stretching         |
| Silica 50%      | 2931.8                   | Stretching Symmetry |
| 2337.72, 2368.59| 2924.04                  | Stretching Symmetry |
The M-O function group with bending vibration occurs at wave number 354.9 cm\(^{-1}\); 308.61 cm\(^{-1}\); 362.62 cm\(^{-1}\); 378.05 cm\(^{-1}\); 354.9 cm\(^{-1}\); 316.9 cm\(^{-1}\); and 308.61 cm\(^{-1}\) seems to see a decrease in absorption peak from the six ingredients. This absorption peak shift is due to the vibrational energy of the material molecules tends to decrease each addition of concentration. In the Fe-Ooct functional group with stretching vibrations from the six concentrations, the absorption peak of the wave number is the same, namely 455.2 cm\(^{-1}\), it can be assumed that there is no new absorption because the vibrational energy tends to be constant. The Zn-Otet function with stretching vibrations in the six concentrations showed new uptake at the constructs of 5% and 15% with wave numbers 586.36 cm\(^{-1}\) and 686.66 cm\(^{-1}\). So that it is indicated that at this concentration the vibration energy increases but tends to be stable at the wave number 570.93 cm\(^{-1}\). Silica material uptake occurred at wave numbers 794.67 cm\(^{-1}\) to 1087.85 cm\(^{-1}\) and 2276 cm\(^{-1}\) when silica is used as ZnFe\(_2\)O\(_4\) encapsulation, the absorption changes. Si-O-Si function groups with stretching symmetry vibrations at concentrations of 10%, 15%, 20%, 30%, and 50% with the absorption of wave numbers 779.24 cm\(^{-1}\) while at 5% concentration there was no absorption. This is due to the low levels of silica contained in these concentrations. In the absorption of wave numbers, 902.6 cm\(^{-1}\) bending occurs with a Si-OH functional group which is a group of silanol and is only absorbed in a concentration of 20%. The absorption of Si-O-Si functional groups with asymmetric vibration stretchings at wave numbers 1049.28 cm\(^{-1}\) from the six concentrations, their absorption tends to be constant which can be assumed that the vibrational energy is also constant at the wave number. The type of stretching vibration with a constant wave number but its energy tends to decrease with an increase in wave number if compared to the time before encapsulation, but at a concentration of 20%, no absorption occurs. In functional groups of absorption of H-O-H bending vibrations with wave numbers, 1635.64 cm\(^{-1}\) vibrational energy at low concentrations tends to be constant at concentrations of 5% and 10%. While at high concentrations labeled five there appears a new wave number of 1643.35 cm\(^{-1}\) concentrations of 30% and 50% and 1651.07 cm\(^{-1}\) at a concentration of 20% due to the difference in absorption indicated that the silica and ZnFe\(_2\)O\(_4\) materials are completely encapsulated which is characterized by a difference in absorption of these wave numbers. The Si-O-Si function group of symmetrical vibrations in the wave number 2276 cm\(^{-1}\) after encapsulation occurred absorption of new wave numbers at concentrations of 5%, 20%, and 50% while concentrating 30%, 15% and 10% of the table absence of absorption. This can be indicated that at a concentration of 5%, 20% and 50% of the material ZnFe\(_2\)O\(_4\) is completely encapsulated in this functional group, which is characterized by the presence of new wave numbers. The vibration energy tends to decrease with increasing wave numbers at concentrations of 5%, 20%, and 50%.

4. Conclusion
Based on the results of the study conducted, the following findings are obtained that M-O functional group bonds occur at a wavelength of 300 cm\(^{-1}\); Fe-Ooct occurs on the wavelength 424.34 cm\(^{-1}\); Zn-Otet with wave number 555.49 cm\(^{-1}\) with the highest absorption of metal with
the Zn-O-group. Fe wave number 1350.17 cm\(^{-1}\). After the encapsulation process is carried out, an increase in the absorption of wave numbers at several concentrations is caused by the vibration energy decreasing when the concentration of silica is added.

5. References

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