Green synthesis and characterization of Zinc Ferrite (ZnFe\(_2\)O\(_4\)) nanocomposite via *Tristaniopsis merguensis* Griff. natural extract

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**Abstract.** Green synthesis and characterization of zinc ferrite (ZnFe\(_2\)O\(_4\)) nanocomposite via *Tristaniopsis merguensis* natural extract was performed. The precursors used in this analysis were Zn(NO\(_3\))\(_2\)·6H\(_2\)O and Fe(NO\(_3\))\(_3\)·9H\(_2\)O, it was known that the extracts of *Tristaniopsis merguensis* leaves had high total phenolic content. Synthesis is carried out by comparing the temperature without annealed and annealed at 700 °C. The presence of phenolic compounds in the extract of *Tristaniopsis merguensis* functions as a bioreduct agent and chelating agent in the formation of zinc ferrite material. The XRD analysis showed the diffraction peaks typical of ZnFe\(_2\)O\(_4\) at 29.98°, 35.85°, 42.75°, 56.51° and 62.09° at 700 °C annealed, indicating that the ZnFe\(_2\)O\(_4\) structure had a face center cubic structure with miller index (220), (311), (400), (400) and (400). The average particle size of ZnFe\(_2\)O\(_4\) is 9.0414 nm based on Scherrer's calculations. FTIR analysis shows the presence of phenolic compounds in *Tristaniopsis merguensis* leaves extracts interacting with Zn\(^{2+}\) and Fe\(^{3+}\) ions producing sharp absorption at wave number 543.80 cm\(^{-1}\) corresponding to the vibrational mode of the metal oxygen bond (Zn-O-Fe).

**1. Introduction**

Magnetic nanoparticles have been the most challenging and interesting scientific issues in recent years due to their magnetic, optical, electrical, photochemical and photocatalytic properties [1-8]. Nanosized spinel ferrites have been fascinated by attention and efforts from both scientific and technological research points of views. One magnetic spinel-ferrites material, iron oxide nanoparticles with MFe\(_2\)O\(_4\) formula (M = divalent metal cations, such as Zn, Mn, Co, etc.) usually used as magnetic carriers because they are ferromagnetic. Among zinc ferrites (ZnFe\(_2\)O\(_4\)) is very interesting to be applied to photocatalysis technology with visible light, because it has good photochemical stability. Various methods, such as hydrothermal [9-12], electrospinning [13] and combustion method [14] have been used to synthesize ZnFe\(_2\)O\(_4\) nanoparticles with various microstructures and study their photocatalytic
activity to degrade dyes. At present, methods for the synthesis of iron oxide material begin to be directed towards environmentally friendly synthesis based on plant extracts, Matinise et al [15] has successfully synthesized ZnFe₂O₄ using Moringa oleifera extract, it is known that plant extracts have high chemical reactivity and function as bioreductors and chelating agents in the synthesis of spinel-ferrites material [16],[17].

Tristaniopsis merguensis Griff is one of the potential local plants to be developed at Bangka Belitung. People in Bangka Belitung know Tristaniopsis merguensis leaves very well as they are mostly used as traditional medicine and tea products. Tristaniopsis merguensis leaves are known to have high total phenolic content [18]. The content has the potential to be used as chelating agents and bioreductors in the zinc ferrite (ZnFe₂O₄) synthesis. This research will examine the synthesis and characterization of zinc ferrite (ZnFe₂O₄) nanocomposites using Tristaniopsis merguensis Griff natural extract.

2. Material and Methods

2.1 Materials

Zinc nitrate hexahydrate (Zn(NO₃)₂.6H₂O) and Iron (III) nitrate nonahydrate Fe(NO₃)₃.9H₂O were produced by Sigma Aldrich and Merck. The leaves of Tristaniopsis merguensis were obtained from Bangka Island (Indonesia).

2.2 Tristaniopsis merguensis extract

30 g of Tristaniopsis merguensis leaves were dipped in 300 ml of distilled water for 2 hours under a magnetic stirrer (70 °C). After cooling naturally to room temperature, the solution was centrifuged and stored at 4 °C in a refrigerated place for further study.

2.3 Synthesis of Zinc Ferrite (ZnFe₂O₄)

For 1 h of magnetic stirring, 50 ml of Tristaniopsis merguensis extract was used to dissolve 5 g of Zn(NO₃)₂.6H₂O and Fe(NO₃)₃.9H₂O. After cooling to room temperature, the mixture was coated with foil and kept at room temperature for 18 h. The mixture was dried in a standard oven at 100 °C after 18 h, then washed with distilled water several times and annealed at 700 °C for 2 h.

2.4 Characterization of the material

The crystalline structure was characterized by X-ray diffraction (X-ray diffraction pattern Rigaku Miniflex600 D/tex ultra 1D) with λCuKα1= 1.5406 Å radiation. The chemical bonding was obtained using the absorption spectrometer of Fourier transform-infrared (FT-IR) (Bruker Alpha with ZnSe beam splitter) within 500-4000 cm⁻¹ range.

3. Result and Discussion

3.1 X-ray diffraction analysis

Synthesis of zinc ferrite (ZnFe₂O₄) nanocomposites was carried out through biosynthesis methods using Tristaniopsis merguensis extract. The purity phase of ZnFe₂O₄ was studied by x-ray diffraction (XRD) analysis (Figure 1), in this study, ZnFe₂O₄ samples were performed without anneal/non anneal (room temperature) and annealed at 700 °C. The annealed temperature of 700 °C in the formation of zinc ferrite (ZnFe₂O₄) using plant extracts is considered to be the optimum annealed temperature [15]. It is known, based on the results of the XRD analysis, that the non-annale sample shows an amorphous phase which is influenced by the natural content of the Tristaniopsis merguensis extract. The presence of crystalline phase and pure structure of ZnFe₂O₄ was demonstrated in the annealed sample 700°C.
Figure 1. XRD analysis for (a) ZnFe$_2$O$_4$ annealed (700 °C) and (b) ZnFe$_2$O$_4$ non-annealed (R.T)

The peaks of the ZnFe$_2$O$_4$ nanocomposite diffraction pattern are clearly shown at values of 2θ namely 29.98°, 35.85°, 42.75°, 56.51° and 62.09° with FWHM (Full Width at Half Maximum) values of 0.0139, 0.0289, 0.0122, 0.0169 and 0.0191, respectively. All diffraction peaks indicate that the structure of the ZnFe$_2$O$_4$ nanocomposite has a face centered cubic structure (JCPDS No: 00-022-1012 with a = 8.441 Å) which corresponds to the Miller index (220), (311), (400), (511) and (440). The XRD analysis results are used not only to determine the crystal structure in the sample, but can also be used to determine the size of zinc ferrite nanocomposite crystals. Based on the calculation using the Scherrer formula, it is obtained the size of zinc ferrite (ZnFe$_2$O$_4$) nanocomposite in this study, which is 9.0414 nm.

$$D = \frac{K\lambda}{\beta \cos \theta}$$

Table 1. XRD data analysis of ZnFe$_2$O$_4$ nanocomposite particle size

| hkl | 2θ (deg) | θ (rad) | FWHM (rad) | D (nm) |
|-----|----------|---------|------------|-------|
| 220 | 29.98    | 0.2616  | 0.0139     | 10.2761 |
| 311 | 35.85    | 0.3128  | 0.0289     | 5.0279  |
| 400 | 42.75    | 0.3730  | 0.0122     | 12.182  |
| 511 | 56.51    | 0.4931  | 0.0169     | 9.2942  |
| 440 | 62.09    | 0.5418  | 0.0191     | 8.4262  |

Average particle size (D) 9.0414

3.2 FTIR analysis

Fourier Transform Infra Red (FTIR) then analyzed nanocomposite samples of zinc ferrite (ZnFe$_2$O$_4$) to present the chemical structures of the functional groups of zinc ferrite (ZnFe$_2$O$_4$). FTIR analysis was performed on ZnFe$_2$O$_4$ non-anneal (room temperature) and annealed at 700 °C. Based on the analysis,
there are a number of absorption bands showing the formation of Zinc Ferrite (ZnFe₂O₄) nanocomposite.

**Figure 2.** FTIR spectra of ZnFe₂O₄ nanocomposite non annealed (R.T) and annealed at 700 °C

Figure 2 shows the main absorption band of ZnFe₂O₄ has a high intensity and a sharp shift to the wavenumber 543.80 cm⁻¹ corresponding to the vibrational mode of the metal oxygen bond (Zn-O-Fe) [19–23]. This wavenumber shift caused by high temperatures in the annealing process. However, the broad intense band at 3400-3419 cm⁻¹ in the sample showed the existence of -OH stretching vibration (hydroxyl / phenol) generated by the phenol group, which was confirmed by the presence of 768.55 cm⁻¹ in the non-annal sample. This absorption band then does not appear in the annealed sample due to the formation of metal bonds with oxygen in the phenol group previously formed zinc ferrite material. At wavenumber 1715.25 cm⁻¹ shows the presence of vibration C=O stretching and this absorption band does not appear in the annealed sample 700 °C, this is because the bond is broken due to increasing temperatures. The absorption band at 1585–1626, 1384-1455, 1113-1229 cm⁻¹ respectively showed C=C, C-H and C-O vibrations which confirmed that the content of Tristaniopsis merguensis extract contained compounds with phenolic structures, based on previous research that Tristaniopsis merguensis had a high total phenolic content of 86.724 ± 1.83 mg EAG / g [18]. The mechanism proposed for the formation of ZnFe₂O₄ nanocomposites is shown in Figure 3.
Figure 3. Proposed mechanism for forming ZnFe$_2$O$_4$ nanocomposites

4. Conclusion
In this study, green synthesis of Zinc Ferrite (ZnFe$_2$O$_4$) nanocomposite via Tristaniopsis merguensis Griff natural extract was successfully carried out. XRD data shows that ZnFe$_2$O$_4$ nanocomposite has a crystalline phase ZnFe$_2$O$_4$. All diffraction peaks indicate that the structure of the ZnFe$_2$O$_4$ nanocomposite has a face centered cubic structure with an average particle size of 9.0414 nm. The proposed mechanism for forming zinc ferrite nanocomposites was developed from an FTIR study to understand the interaction of Zn$^{2+}$ and Fe$^{3+}$ ions via chemical compounds of the Tristaniopsis merguensis extract.

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