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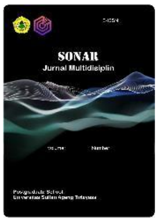

Crystal Structure Analysis of Magnetite (Fe_3O_4) Nanoparticles By Green Synthesis Method Using Moringa Oleifera Extracts

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ABSTRACT

A green synthesis obtained from several basic materials such as $FeCl_3.6H_2O$, $FeSO_4.7H_2O$, NH_4OH, and moringa oleifera extract using the coprecipitation method has been successfully carried out by obtaining Fe_3O_4 nanoparticle material. Analysis of the crystal structure of Fe_3O_4 nanoparticles can be seen from the results of characterization using X-Ray Diffraction (XRD) which shows diffraction peaks namely (220), (311), (400), (422), (511), (440), and (533) with the main peak at index (311). The Fe_3O_4 nanoparticle sample using the natural reducing agent moringa oleifera, was able to eliminate the Fe_2O_3 diffraction peak and decrease the crystallite size of Fe_3O_4 nanoparticles. The disappearance of the diffraction peak and the decrease in crystallite size are due to the effect of the addition of moringa oleifera reducing agent which stabilizes during the reaction process.

Keywords: crystal structure, moringa oleifera, nanoparticles Fe_3O_4

INTRODUCTION

Over the past few years, magnetic nanoparticles (MNPs) have gained more attention due to their great potential for applications in various fields such as engineering, energy, biomedicine, and the environment. Some magnetic nanoparticles have been studied such as nickel, cobalt, and iron because they have unique characteristics (Ali et al., 2021). Magnetite (Fe_3O_4) nanoparticles are the most commonly used magnetic nanoparticles due to their biocompatibility, low toxicity, and superparamagnetic properties that can be used in various applications such as magnetic labels, biosensors, catalysts, magnetic hyperthermia, and drug delivery (Saputra et al., 2024; Stan et al., 2017).

The success of applying Fe_3O_4 nanoparticles in various fields depends on the synthesis method and crystal size control. This is an alternative way to overcome the shortcomings of Fe_3O_4 nanoparticles, which are easy to agglomerate and unstable in acidic conditions, becoming a crucial problem because it can cause oxidation, reduce the magnetic properties, and stability of Fe_3O_4 nanoparticles (Liu et al., 2020; Riyanti et al., 2023). Crystal size is thought to determine the magnetic properties of magnetic materials (Kiswanto et al., 2021). The smaller the crystal size, the better the magnetic responsiveness of Fe_3O_4 nanoparticles, in other words, it is easy to respond when exposed to an external magnetic field (Rampengan & Polii, 2019). Magnetic nanoparticles are often produced using chemical and physical processes, but these conventional methods require high costs, high energy, and hazardous compounds (Wang et al., 2022). To overcome this problem, recent research has found an alternative through an easy biological pathway in producing NPMs using natural ingredients such as microorganisms and plant extracts known as the "green synthesis" method (Akhtar et al., 2013; Bindhu et al., 2020). Low toxicity, low cost, and environmentally friendly are some of the advantages of MNPs synthesis process mediated by plant extracts (Mabarroh et al., 2022).

Plant extracts can be used as natural reducing agents and stabilizers during the synthesis process because they contain phytochemical substances or proteins such as phenolics and flavonoids, several studies have reported that there are various plant extracts that are widely used in green synthesis of Fe_3O_4, such as Chromolaena odorata, Archidendron pauciflorum, and Pomegranate granatum (Saputra et al., 2024; Sari et al., 2023). One of the plants commonly used as a natural reducing agent is moringa oleifera (MO), high phytochemical substances such as saponins, flavonoids, and phenolic acids, contained in MO that can function as reducing agents and stabilizers during the green synthesis process (Yoga Darmawan et al., 2023). Based on the advantages of these natural reducing agents, this study aims to analyze the crystal structure of Fe_3O_4 nanoparticles with reducing agent (40 mL NH_4OH 10%) and Fe_3O_4 nanoparticles synthesized using moringa oleifera extract with reducing agent (35 mL NH_4OH 10% and 5 mL MO).

RESEARCH METHODS

Materials. The basic materials used in this research are chemicals, namely ferric chloride hexahydrate ($FeCl_3.6H_2O$), ferrous sulfate heptahydrate ($FeSO_4.7H_2O$), distilled water, ammonium hydroxide (NH_4OH), and moringa oleifera (MO) powder obtained from Safiya.

Preparation of moringa oleifera (mo) extract. MO extract was made in a simple way as follows; mix MO powder and distilled water (5 g / 60 ml) stirred using a magnetic stirrer with a stirring temperature concentration of 60 °C and a stirring time of 60 minutes. Next, filtering was carried out using Whatman paper to obtain MO extract.

Green synthesis Fe_3O_4 nanoparticles. Fe_3O_4 nanoparticles were obtained through the coprecipitation method, weighing $FeCl_3.6H_2O$ (4.054 g) and $FeSO_4.7H_2O$ (2.086 g) using digital scales. Then each was dissolved with 7.5 ml of distilled water and stirred for 15 minutes. Then, 5 ml of MO extract was added to the mixture. Add NH_4OH 10% solution (35 ml), dripped dropwise into the mixture. After 90 minutes, the precipitate was separated and washed using distilled water. After that, the black precipitate was dried for 2 hours at 100 °C until dry. For samples that fully use chemical reducing agents are labeled (Fe_3O_4), while for samples that use the addition of MO extract are labeled (GS-Fe_3O_4).

Characterization. Crystal structure analysis was performed using Bruker D8 Advance x-ray diffraction (XRD), the X-ray wavelength used was 1.5406 Å and 2θ = 20-80°.

RESULTS AND DISCUSSION

The x-ray diffraction (XRD) characterization results for Fe_3O_4 and GS-Fe_3O_4 samples are shown in Figure 1.

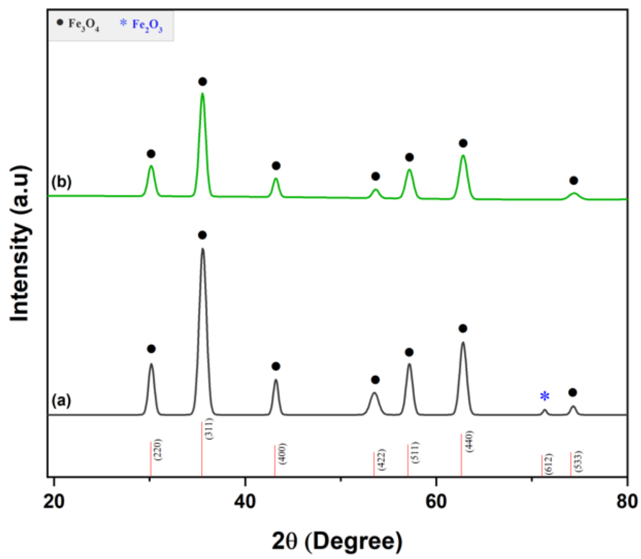

Figure 1. XRD pattern of (a) Fe_3O_4 nanoparticle sample, (b) GS-Fe_3O_4 nanoparticle sample

Figure 1 shows the diffraction pattern of Fe_3O_4 nanoparticles and Fe_3O_4 nanoparticles synthesized using moringa oleifera (MO) extract characterized as GS-Fe_3O_4. The crystal structure of Fe_3O_4 nanoparticles is indicated by the appearance of diffraction peaks around 2θ including; 30.2°, 35.5°, 43.2°, 53.5°, 57.2°, 62.8°, and 74.3° corresponding to miller planes (220), (311), (400), (422), (511), (440), and (533) respectively with the main peak at index (311). The diffraction peak data analysis was matched with XRD crystallography open database (COD) pattern No. 1010369. Oxidation occurred on Fe_3O_4 thus indicating the formation of Fe_2O_3 phase (COD. No. 4002383) in both samples.

For further analysis, this study also estimated the crystallite size of the nanoparticles based on the Debye-Scherrer equation, as shown in Equation 1 (Ghoohestani et al., 2024).

$$D = \frac{k\lambda}{\beta \cos\theta} \quad (1)$$

where D, k, λ, and β are the nanoparticle crystal size, Scherrer constant, X-ray beam wavelength, and full width at half maximum (FWHM), respectively.

Table 1. Grain size of Fe_3O_4 nanoparticles and GS-Fe_3O_4 nanoparticles

Sample	**Average Crystal Size (nm)**	**Phase Composition (%)**	
		Fe_3O_4	**Fe_2O_3**
Fe_3O_4	8,4 ± 0,4	92,98	7,02
GS-Fe_3O_4	7,9 ± 0,4	100	0

Table 1 shows that after adding MO as a natural reducing agent, the average crystallite sizes of Fe_3O_4 and GS-Fe_3O_4 are 8.4 ± 0.4 nm and 7.9 ± 0.4 nm, respectively. There is a decrease in the crystallite size. This indicates that the use of MO as a natural reducing agent in Fe_3O_4 nanoparticles can maintain the stability of the nanoparticles (Latifa et al., 2024).

After adding MO as a natural reducing agent, the Fe_2O_3 phase decreased from 7.02% to 0%. In contrast, there was an increase in phase in the Fe_3O_4 sample from 92.98% to 100% as shown in Table 1. This indicates the use of MO as a natural reducing agent can reduce the oxidation of Fe_3O_4 nanoparticles. The oxidation process can be reduced because MO contains abundant phytochemical substances with strong antioxidant properties (Jiananda et al., 2023; Mallenakuppe et al., 2019).

CONCLUSION

Based on the research that has been done, it can be concluded that green synthesis obtained from several basic ingredients such as $FeCl_3.6H_2O$, $FeSO_4.7H_2O$, NH_4OH, and moringa oleifera (MO) extract using the coprecipitation method was successfully carried out by obtaining Fe_3O_4 nanoparticle material. The results of characterization of Fe_3O_4 nanoparticles using x-ray diffraction (XRD) showed diffraction peaks of (220), (311), (400), (422), (511), (440), and (533) with the main peak at index (311). The Fe_3O_4 nanoparticle sample using the natural reducing agent MO, was able to eliminate the Fe_2O_3 diffraction peak and decrease the crystallite size of

Fe_3O_4 nanoparticles. The disappearance of the diffraction peak and the decrease in crystallite size are due to the effect of the addition of MO reducing agent which stabilizes during the reaction process.

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