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Synthesis and Characterization of TiO_2 /Fe_3O_4 Composites Using Sonication Method

Elsa Avrilia Putriani[1], Noor Hindryawati[2*], Hajar Anuar[3]
Mulawarman University
Corresponding Author: Noor Hindryawati hindryawati@gmail.com

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A B S T R A C T

TiO_2/Fe_3O_4 composite was synthesized by sonication method using mass ratio of 10:1 then calcined at 400ºC for 2 hours. Characterization of TiO_2/Fe_3O_4 composite was carried out using X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscope (SEM) instruments. The results of characterization of TiO_2/Fe_3O_4 composite using XRD showed diffractogram results with a collection of Fe_3O_4 phases and TiO_2 phases with a crystallinity value of 64.7%. FTIR analysis showed Ti-O-Ti vibrations appeared in the range of 700-900 cm-1 and absorption bands from Fe-O vibrations appeared at a peak around 600 cm-1. SEM analysis showed the morphology of TiO_2 /Fe_3O_4 composite with Fe_3O_4 particles attached to the surface of TiO_2 particles and forming a rough surface

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INTRODUCTION

Inorganic semiconductor materials are now increasingly being developed in various industrial fields (Wang et al., 2024). One of the widely used semiconductor materials is Titanium Dioxide (TiO_2). Titanium Dioxide (TiO_2) is one of the semiconductor materials that is widely found in nature and is used in various industries (Haidry et al., 2024). For example, in *photocatalytic technology*, biomaterials, capacitors, solar cells, *drug delivery*, gas sensors, *self-cleaning*, waste degradation, antibacterial, anticancer therapy, health products, biological and chemical sensors, corrosion protection, cosmetic materials, air purifiers, water purification, hospital cleaning equipment and hospital sterilization (Kustiningsih et al., 2022; Sri Kunarti et al., 2018; Sunaryono et al., 2020).

The use of TiO_2 is very diverse because TiO_2 has various advantages such as economical cost, has high decomposition performance, is abundant in nature, is non-toxic, has stable chemical properties, has good optical characteristics, strong oxidation ability, biocompatibility, high crystallinity, is insoluble in water and has a high specific surface area (AlMohamadi et al., 2024; Behravesh et al., 2024; Moosavi et al., 2020; Sunaryono et al., 2020). However, TiO_2 apparently has several weaknesses including having a large band gap of 3.0-3.2 eV, is not easy to separate, recycle and recover after treatment, tends to clump and has a low adsorption capacity in its application (Li et al., 2024; Moosavi et al., 2020; Sri Kunarti et al., 2018). Therefore, modification of TiO_2 compounds is needed to increase the efficiency of using TiO_2 compounds, one of which is by compositing Fe_3O_4 in TiO_2 compounds.

Composite is a new material produced by combining two or more materials to produce a new material with different characteristics from the constituent materials and superior properties compared to the original material (Krauklis et al., 2021; Reknosari et al., 2021). In this case, Fe_3O_4 is used to be composited with TiO_2 because Fe_3O_4 has several advantages including being easy to synthesize, economical, non-toxic, biocompatible (Rizky Pradipta et al., 2021)and having supermagnetic properties with a large response to magnetic fields, making it easier to separate and reuse (Nee Koo et al., 2019). In addition, the use of Fe3O4 in the manufacture of composites aims to facilitate access to the separation of dissolved substances and test solutions by using an external magnetic field (Dirgayanti et al., 2021). In its manufacture, TiO_2/Fe_3O_4 composites can be made using various methods. One of the methods that can be used is the sonication method.

Sonication is a method of making compounds using ultrasonic waves that will convert electrical signals into physical vibrations, resulting in the breakdown of molecules in the solution (Putri et al., 2021). This method was chosen because of its advantages, including an efficient and simple working process, can use room temperature, can produce particles with uniform sizes (Dwi et al., 2020), prevent agglomeration, can produce a large surface area for the composite (Putri et al., 2021), can minimize contaminants (Elgarahy et al., 2024)and reduce aggregation of the modified composite (Deepa et al., 2020).

The purpose of this study is to determine the characteristics of TiO_2, Fe_3O_4 and TiO_2/Fe_3O_4 composite materials based on characterization using an X-Ray

Diffraction *(XRD)* instrument , to determine the characteristics of TiO_2/Fe_3O_4 composite materials based on characterization using a Fourier Transform Infrared Spectroscopy (FTIR) instrument and to determine the characteristics of TiO_2 , Fe_3O_4 and TiO_2/Fe_3O_4 composite materials based on characterization using a Scanning Electron Microscope (SEM) instrument.

RESEARCH METHODS

1. Tools and materials

The tools used in this study were analytical balance, spatula, watch glass, stirring rod, spray bottle, stirring rod, beaker glass , measuring cup, measuring pipette, bulpfiller , dropping pipette, external magnet, watch glass, spatula, petri dish, set of reflux apparatus, stative pole, clamp, three-neck round bottom flask, thermometer, hot plate, magnetic stirrer, measuring flask, ointment bottle, *centrifuge* , *centrifuge* tube , Memmert oven, Kalstein + Furnace , sonication, Match! application, Origin application, X-Ray Diffraction (XRD) Brucker (8D Advance Eco), Fourier Transform Infrared Spectroscopy (SEM) and Scanning Electron Microscope (SEM).

The materials used in this study were TiO_2 Merck, $FeCl_3.6H_2O$ Merck, $FeSO_4.7H_2O$ Merck, aqueous solution, NH_4OH 25% Merck, $(NH_4)_2SO_4$ Merck, vaseline , aluminum foil and plastic wrap.

2. Research Procedures

a. Synthesis of Fe_3O_4

A total of 12g of $FeCl_3.6H_2O$ and 8 g of $FeSO_4.7H_2O$ (mole ratio 2: 1) were dissolved in 50 mL of distilled water . Then the two solutions were mixed and stirred using a magnetic stirrer until the solution looked clear while heating at a temperature of 70ºC. Then 100 mL of 25% NH_4OH was added dropwise (maintained at a temperature of 70ºC) for 3 hours. Next, the filtrate and residue were separated with the help of an external magnet. After that, it was dried in an oven at a temperature of 100ºC for 3 hours and cooled in a desiccator for 15 minutes. Finally, the Fe_3O_4 was weighed (Reknosari et al., 2021).

b. Synthesis of TiO_2/Fe_3O_4 Composite

A total of 100 mg of Fe_3O_4 was dissolved in 200 mL of distilled water and 1 g of TiO_2 was dissolved in 100 mL of $(NH_4)_2SO_4$ 0.02 M. Then the two solutions were mixed and sonicated for 30 minutes at room temperature. Then centrifuged at 4000 rpm for 15 minutes to obtain a solid. After that, the obtained solid was dried in an oven at 100ºC for 18 hours. Finally, calcination was carried out using *a furnace* at 400ºC for 2 hours and the TiO_2/Fe_3O_4 composite was ready to be characterized (Tajareh et al., 2019).

3. Characterization Test

a. X-Ray Diffraction (XRD)

Sample preparation for XRD analysis is carried out by preparing 0.5 g of sample (in powder form, size below 40 microns) and placing it in a test sample holder with a minimum volume of 2 mL. The sample is then placed into the instrument in the "sample stage" position and is ready for analysis.

b. Fourier Transform Infrared Spectroscopy (FTIR)

Sample preparation for FTIR analysis uses the KBr Pellet method. A 1-10 mg sample powder is carefully ground with 100 mg KBr using a mortar and pestle and then pressed into a mold to form a thin disc. The sample spectrum is then read using FTIR in the range of 400-4000 cm $^{-1}$.

c. Scanning Electron Microscope (SEM)

Sample preparation for SEM analysis is done by sticking or spreading 0.1 g of sample on the surface of carbon tape, then placing the sample on the sample holder and the sample is ready to be analyzed.

RESULTS AND DISCUSSION

1. Synthesis of Fe_3O_4

In the synthesis of Fe_3O_4, 8.416 g of Fe_3O_4 material was obtained with a yield of 42.08%. Based on **Figure 1 ,** Fe_3O_4 is a blackish brown solid with a strong magnetic force. It can be seen that when Fe_3O_4 is brought close to an external magnetic field, the Fe_3O_4 particles are completely attracted to the external magnetic field. This indicates that the synthesis of Fe_3O_4 was successful and the formation of Fe_3O_4 occurred optimally. The reactions that occur in this synthesis are as follows.

$$2FeCl_{3(aq)} + FeSO_{4(aq)} + 8NH_4OH_{(aq)} \rightarrow \underset{\text{endapan hitam}}{Fe_3O_{4(s)}\downarrow} + 6NH_4Cl_{(aq)} + (NH_4)_2SO_{4(aq)} + 4H_2O_{(l)}$$

Based on the reaction, the stoichiometric reaction shows that Fe_3O_4 can be synthesized maximally with a mole ratio of 2:1 at pH 9-14 (Rahmayanti, 2020). Therefore, in this study, the synthesis of Fe_3O_4 used a mole ratio of 2:1 while maintaining pH 9-10 to maximize the formation of Fe_3O_4 .

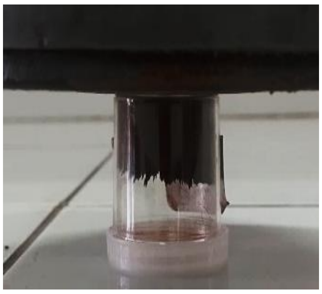


Figure 1. Results of Fe_3O_4 Synthesis

2. Synthesis of TiO_2/Fe_3O_4 Composite

In the synthesis of TiO_2/Fe_3O_4 composite, the mass of TiO_2/Fe_3O_4 composite was obtained as much as 0.866 grams with a yield value of 78.72% and a crystallinity value of 64.7%. Based on Figure 2 , the TiO_2/Fe_3O_4 composite is a light brown solid and indicates the presence of magnetic properties in the material. It can be seen that when the TiO_2/Fe_3O_4 composite is brought close to an external magnetic field, some of the solids are attracted to the magnetic field, while others are not attracted. This is influenced by the mass ratio of TiO_2 which is 10 times greater than Fe_3O_4 so that only some of the composite is attracted to the external magnetic field.

Figure 2. Results of TiO_2/Fe_3O_4 Composite Synthesis

3. *X-Ray Diffraction* (XRD) Analysis Results

XRD analysis was carried out to determine the crystal phase and crystallinity value of TiO_2 , Fe_3O_4 and TiO_2/Fe_3O_4 composite materials. The following are the results of the identification of the diffraction peaks obtained.

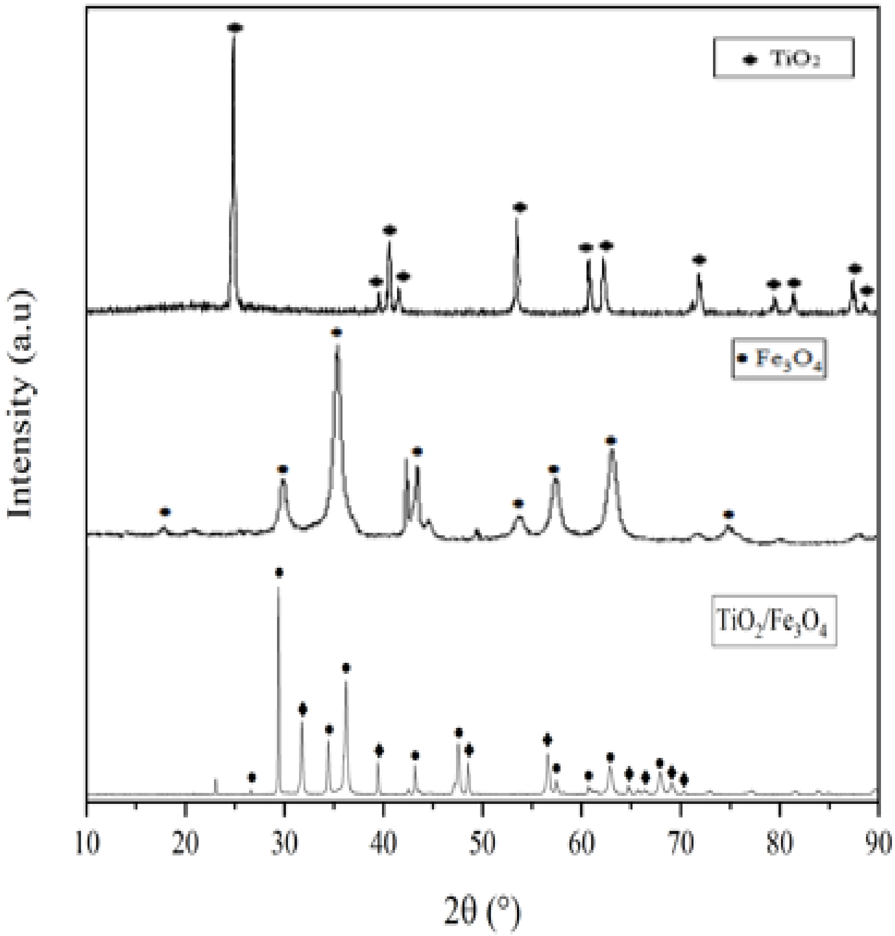


Figure 3. Difactogram of TiO_2, Fe_3O_4 and TiO_2/Fe_3O_4 Composite (♦TiO_2 and ●Fe_3O_4)

Based on Figure 3, the results of the diffraction peak analysis using the Match! application are obtained. In the TiO_2 material, there are sharp peaks indicating the anatase phase of TiO_2 . Diffraction peaks appear at angles of 25.5°, 37.1°, 38.0°, 38.7°, 48.2°, 54.1°, 55.2°, 62.2°, 62.8°, 68.9°, 70.4°, 75.2° and 76.2° which are reflections of the Miller indices [101], [103], [004], [112], [200], [105], [211], [213], [204], [116], [220], [215] and [301] which correspond to the angles of TiO_2 in ICCD: 10-71-1169.

In the Fe_3O_4 material there are sharp peaks indicating the Fe_3O_4 magnetite phase. Diffraction peaks appear at angles of 18.4°, 30.3°, 35.5°, 43.6°, 53.6°, 57.2°, 62.9° and 74.4° which are reflections of the miller index [111], [220], [311], [400], [422], [511], [440] and [533]. According to (Khalid et al., 2023)the diffraction peaks, it shows the cubic plane of Fe_3O_4 according to JCPDS: No. 01-82-1533.

TiO_2/Fe_3O_4 composite there are sharp peaks indicating the Fe_3O_4 phase, TiO_2 phase. The Fe_3O_4 magnetite diffraction peaks appear at angles of 26.5°, 29.4°, 34.4°, 43.2°, 47.5°, 57.4°, 60.7°, 62.8° and 67.8° which are reflections of the Miller index [310], [220], [311], [400], [331], [511], [440], [440] and [422]. According to (John Prabhahar et al., 2019; Khalid et al., 2023; Sriram et al., 2019; Takai et al., 2019)these angles correspond to the crystal plane of pure Fe_3O_4 , the cubic plane of Fe_3O_4 and the diffraction pattern matches the inverse spinel crystal structure of Fe_3O_4 based on JCPDS: 98-3969, JCPDS: No. 01-82-1533 and JCPDS: No. 65-3107. The TiO_2 phase appears at angles of 31.7°, 36.2°, 39.4°, 48.5°, 56.5°, 64.6°, 66.3°, 69.0° and 70.2° which are reflections of the miller indices [121], [101], [200], [200], [220], [310], [204], [301] and [220]. According to (Cimen et al., 2024; Du et al., 2021; Ngo et al., 2022; Rathore et al., 2023)the diffraction peaks, it corresponds to the tetragonal crystal structure based on JCPDS: No. 21-1276 and JCPDS: No. 21-1272.

4. *Fourier Transform Infrared Spectroscopy* (FTIR) Analysis Results

In this study, the characterization of TiO_2/Fe_3O_4 composites was carried out using FTIR to identify the functional groups contained in the TiO_2/Fe_3O_4 composites. The analysis results were carried out by interpreting the absorption peaks from the IR spectrum (Fadillah et al., 2017). The following are the results of the FTIR spectrum identification of the TiO_2/Fe_3O_4 composites.

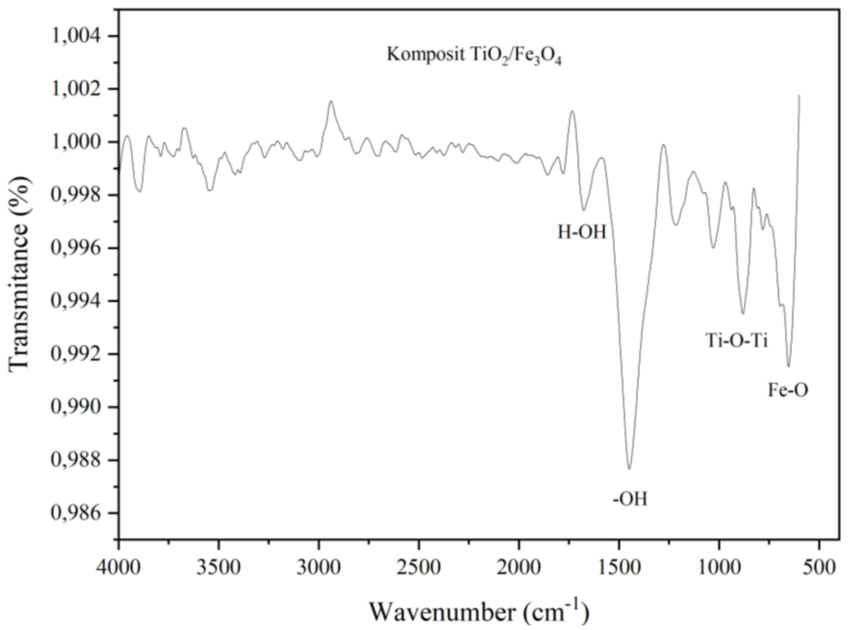
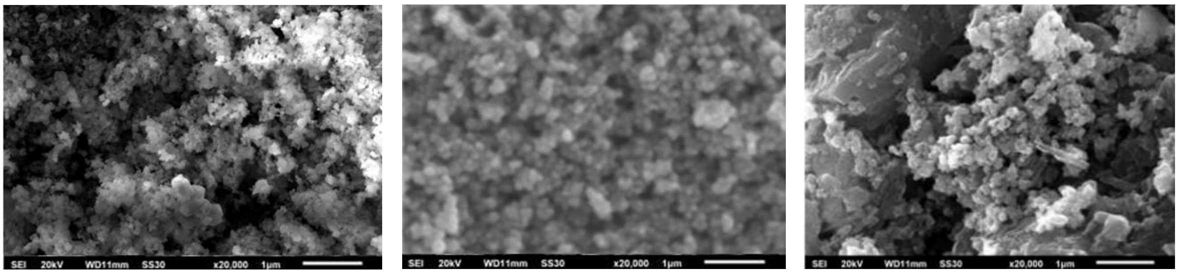


Figure 4. FTIR Spectrum of TiO_2/Fe_3O_4 Composite

Based on Figure 4, the FTIR spectrum results on the TiO_2/Fe_3O_4 composite show a broad absorption in the range of 1200-1600 cm $^{-1}$ which indicates the presence of -OH stretching vibrations from surface hydroxide species. The peak in the spectrum around 1680 cm $^{-1}$ indicates the occurrence of H-OH bending from water. The spectrum showing the characteristic peak of Ti-O-Ti vibrations appears in the range of 700-900 cm $^{-1}$ and the absorption band of Fe-O vibrations appears at a peak around 600 cm $^{-1}$ (Tedsree et al., 2017).

5. ***Scanning Electron Microscope*** **(SEM) Analysis Results**

SEM analysis was performed to observe the surface of the TiO_2/Fe_3O_4 composite with high image resolution. The following are images of the surface of TiO_2, Fe_3O_4 and the TiO_2/Fe_3O_4 composite with a magnification of 20,000 times.



(a) (b) (c)

Figure 5. SEM images of (a) TiO_2, (b) Fe_3O_4 and (c) TiO_2/Fe_3O_4 Composite

Based on **Figure 5 (a)**, SEM results show that TiO_2 particles are spherical and stick together to form a dense aggregate (Krakowiak et al., 2022). **Figure 5 (b)** , SEM results show that Fe_3O_4 particles are spherical with the formation of relatively small particles and a homogeneous size distribution (Aguinaco et al., 2022). **Figure 5 (c)** , SEM results show that the TiO_2/Fe_3O_4 composite is spherical and joins to form a large aggregate and it can be seen that the Fe_3O_4 particles stick to the surface of the TiO_2 particles to form a rough surface (Kustiningsih et al., 2022).

CONCLUSION

Based on the results of the synthesis of TiO_2/Fe_3O_4 composites using the sonication method, the TiO_2/Fe_3O_4 composite was obtained in the form of a light brown solid with a mass of 0.866 grams and a yield value of 78.72%. The results of the characterization of the TiO_2/Fe_3O_4 composite using the XRD instrument showed that the diffraction peaks of the Fe_3O_4 magnetite phase appeared at angles of 26.5°, 29.4°, 34.4°, 43.2°, 47.5°, 57.4°, 60.7°, 62.8° and 67.8° while the TiO_2 phase appeared at angles of 31.7°, 36.2°, 39.4°, 48.5°, 56.5°, 64.6°, 66.3°, 69.0° and 70.2° with a crystallinity value of 64.7%. The FTIR spectrum shows that the absorption band of Ti-O-Ti vibration appears in the range of 700-900 cm $^{-1}$ and the absorption band of Fe-O vibration appears at a peak around 600 cm $^{-1}$. SEM characterization shows that Fe_3O_4 particles adhere to the surface of TiO_2 particles forming a rough surface.

FURTHER STUDY

This research still has limitations so that further research is needed related to the topic of Synthesis and Characterization of TiO2 / Fe3O4 Composites Using Sonication Method to perfect this research and increase insight for readers.

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