

Study of Microstructure and Optical Properties of Fe₂O₃/TiO₂ Composites as Functional Materials

Studi Mikrostruktur dan Sifat Optik Komposit Fe₂O₃/TiO₂ sebagai Material Fungsional

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ABSTRACT

Semiconductors have conductivity levels between insulators and conductors which can be applied in various fields such as photocatalytic, adsorption, and Dye Sensitizer Solar Cell (DSSC). However, some semiconductors are only active under ultraviolet light, therefore to improve their utilization, modifications are made by producing a hybrid combination of two or more materials or doping materials. This study aims to obtain a semiconductor material with a low band gap energy from the Fe₂O₃/TiO₂ composite material. Fe₂O₃/TiO₂ composites were synthesized by the solid-state method and characterized by XRD, SEM-EDX, and UV-Vis. The characterization using XRD showed the peak intensity of TiO₂ and Fe₂O₃. The morphology of the material obtained using SEM-EDX showed an even distribution of particle size, as well as the distribution of Ti, Fe, and O elements. The optical properties of the composite showed strong absorbance in the UV region for higher TiO₂ compositions. On the other hand, composite materials with a higher Fe₂O₃ composition showed stronger absorbance in the visible light region.

Keywords: Fe₂O₃, microstructure, optic, TiO₂.

ABSTRAK

Semikonduktor adalah material dengan tingkat konduktivitas di antara material isolator dan konduktor yang dapat diaplikasikan dalam berbagai bidang seperti fotokatalitik, adsorpsi, Dye Sensitizer Solar Cell (DSSC) dan masih banyak lagi. Namun, beberapa semikonduktor hanya aktif dibawah sinar ultraviolet, sehingga untuk meningkatkan kinerja material semikonduktor dalam pemanfaatannya, umumnya dilakukan modifikasi berupa pembuatan komposit yakni kombinasi dua atau lebih material maupun material doping. Penelitian ini bertujuan untuk membuat material komposit Fe₂O₃/TiO₂ untuk mendapatkan material semikonduktor dengan energi celah pita yang rendah. Material komposit Fe₂O₃/TiO₂ disintesis dengan metode solid state yang kemudian dikarakterisasi dengan XRD, SEM-EDX dan UV-Vis. Hasil karakterisasi menggunakan XRD menunjukkan adanya intensitas puncak dari TiO₂ dan Fe₂O₃. Morfologi material yang diperoleh menggunakan SEM-EDX menunjukkan distribusi ukuran partikel yang merata, serta sebaran unsur Ti, Fe dan O. Sifat optik dari komposit menunjukkan absorbansi yang kuat di daerah sinar UV untuk komposisi TiO₂ yang lebih tinggi. Sebaliknya, material komposit dengan komposisi Fe₂O₃ yang lebih tinggi menunjukkan absorbansi yang lebih kuat di daerah sinar tampak.

Keywords: Fe₂O₃, mikrostruktur, optik, TiO₂.

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1. INTRODUCTION

Based on its ability to conduct electric current, a material can be divided into three types: conductors, insulators, and semiconductors. The structure of their band gap energies reveals the difference between the three materials. Semiconductors are substances with electrical conductivity between insulators and conductors and an energy gap between 0 and 4 eV. (Aminullah et al. 2019). Some semiconductor materials are metal oxides such as TiO₂, SnO₂, CuO, CaO, Fe₂O₃, and Fe₃O₄.

TiO₂ and Fe₂O₃ are semiconductor materials that are commonly found and used in various fields. TiO₂ (titanium dioxide) is a semiconductor material that is widely studied because of its good optical and electronic properties. In addition, this material has long-term stability, strong oxidation strength, low cost, and non-toxicity. Structurally, TiO₂ has three crystalline phases: anatase, rutile, and brookite. Anatase and rutile have a tetragonal crystal structure, while brookite has an orthorhombic crystal structure. The anatase and brookite phases are metastable phases that easily change to the rutile phase when heated. The band gap energy value of TiO₂ in the anatase phase is 3.2 eV, while in the rutile phase, it is 3.0 eV (Listanti et al. 2018). Some applications of TiO₂ include sensors, solar cells, photocatalysts, and health products.

Fe₂O₃ is known as hematite with a rhombohedral crystal structure. Fe₂O₃ is resistant to chemical reactions and temperature, environmentally friendly, widely used in semiconductor applications, and can absorb light (Nurrahmawati 2019). Fe₂O₃ can be used as a catalyst, gas sensor, solar cell, pigment, and lithium-ion battery.

The application of semiconductor materials such as photocatalytic generally requires specific criteria to achieve the optimal activity, both from the band gap energy factor or others. Researchers often combine two or more materials by doping, composite manufacturing, and carriers to achieve this goal. Composite manufacturing is easier to conduct because it does not damage the basic structure of the material and is produced through physical reactions (Iqbal et al. 2020). The synthesized Fe₂O₃/TiO₂ composite was

carried out to obtain optimal band gap energy to achieve the good photocatalytic activity, including degrading indigo carmine dye waste (Lubis et al. 2019) and methyl orange (Koohestani 2019).

In addition to optical properties, microstructural characteristics are important indicators of the material's identification. Microstructural characteristics can be in the form of diffraction patterns, elemental mapping, morphology, and topography. These aspects determine the physical characteristics of the material (Hasa 2007). Therefore, in this study, an assessment of the microstructure and optical properties of Fe₂O₃/TiO₂ composites was carried out.

2. MATERIALS AND METHODS

2.1. Materials

The materials used were TiO₂ (Merck, 99%), Fe₂O₃ (Merck, 99%), and methanol (Aldrich, 99%). The tools used were glassware, magnetic stirrer, oven, furnace, Panalytical XRD, SEM-EDX, and UV-Vis Diffuse Reflectance spectrophotometer Shimadzu-2450.

2.2. Methods

Fe₂O₃ and TiO₂ were mixed in a beaker with a ratio of 1, 2, 3, and 4. About 50 mL of methanol was added and stirred for 2-3 hours until homogeneous. The homogeneous mixture was allowed to stand at room temperature to obtain a Fe₂O₃/TiO₂ composite precipitate. The precipitate was separated, then dried in an oven at 90°C for 1 hour to evaporate the remaining methanol. Then it was calcined at 500°C for 1 hour. The obtained solids were characterized by XRD, SEM-EDX, and UV-Vis.

3. RESULTS AND DISCUSSION

The microstructure study of the Fe₂O₃/TiO₂ composite with mass ratios of 1, 2, 3, and 4 (Fig. 1) shows the diffraction pattern of the material according to JCPDS No. 21-1272 for TiO₂ and JCPDS No. 39-1346 for Fe₂O₃. The diffractogram identified peaks appearing at 25°, 32°, 47°, 53°, and 63° as TiO₂ peaks, and Fe₂O₃ peaks at 33°, 35°, 40°, 49°, and 62°. The combination of peaks that appear in one diffraction pattern indicates that the material

synthesis was successful. The mass ratio also affects the peak intensity. As shown in Fig. 1, the

peak intensity of TiO_2 decreases as the amount of Fe_2O_3 increases.

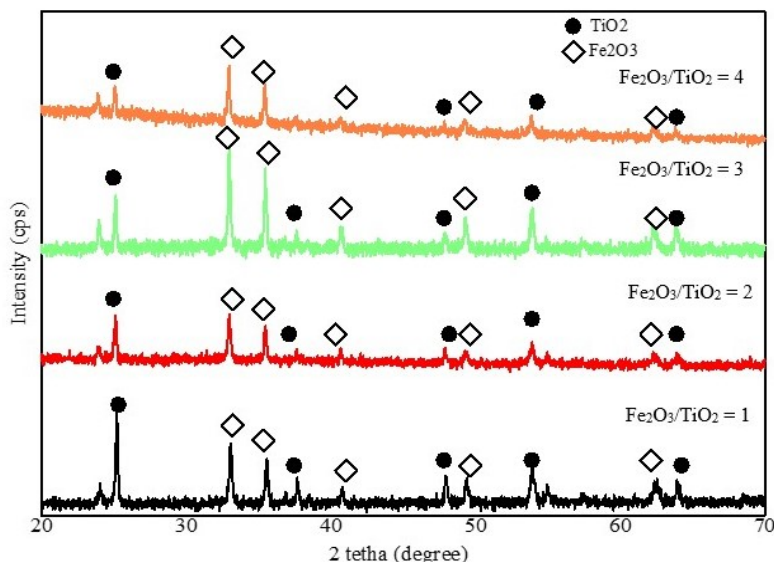


Figure 1. Diffraction pattern of $\text{Fe}_2\text{O}_3/\text{TiO}_2$ composite

The microstructural characteristics of the material are also supported by SEM-EDX data which shows the morphology and distribution of $\text{Fe}_2\text{O}_3/\text{TiO}_2$ composite elements (Fig. 2 and Fig. 3). Fig. 2(a) shows the composite morphology of $\text{Fe}_2\text{O}_3/\text{TiO}_2$ with a ratio of 1, and Fig. 2(b) with a ratio of 3. Both samples showed a uniform morphology. It indicates that the material synthesis was proceeding smoothly. Fig. 3 shows the distribution of Ti, Fe, and O elements of the $\text{Fe}_2\text{O}_3/\text{TiO}_2$ composite. Element O is indicated by the intensity of the bright blue color, which is relatively evenly distributed, but there are dark areas that are poor in oxygen. Fe elements are identified by red color and Ti by green color. It indicates a good distribution of the two elements.

The $\text{Fe}_2\text{O}_3/\text{TiO}_2$ composite's optical characteristics were identified by a UV-Vis instrument. Fig. 4 shows the difference in absorbance of $\text{Fe}_2\text{O}_3/\text{TiO}_2$ composite materials to light wavelengths at different ratios. $\text{Fe}_2\text{O}_3/\text{TiO}_2$ composites with a ratio of 1 had absorbance at a

wavelength of around 300-600 nm. The $\text{Fe}_2\text{O}_3/\text{TiO}_2$ composite with a ratio of 4 experienced a shift in absorbance to around 350-650 nm. It can occur because Fe_2O_3 has a band gap energy of 2.2 eV, which is active under visible light, so the combination of Fe_2O_3 and TiO_2 materials can reduce the bandgap energy of TiO_2 (Iqbal et al. 2020).

The increasing amount of Fe_2O_3 in the $\text{Fe}_2\text{O}_3/\text{TiO}_2$ composite shifts the material's absorbance toward the visible light wavelength region. The absorbance at a given wavelength is inversely proportional to the band gap energy. Visible light has a longer wavelength than UV light, so materials with high absorbance in the visible light wavelength area have a smaller band gap energy value. Therefore, the $\text{Fe}_2\text{O}_3/\text{TiO}_2$ composite with a ratio of 4 has a smaller band gap energy value than the $\text{Fe}_2\text{O}_3/\text{TiO}_2$ composite with a ratio of 1 (Iqbal et al. 2020). It is supported by the reflectance data shown in Fig. 5.

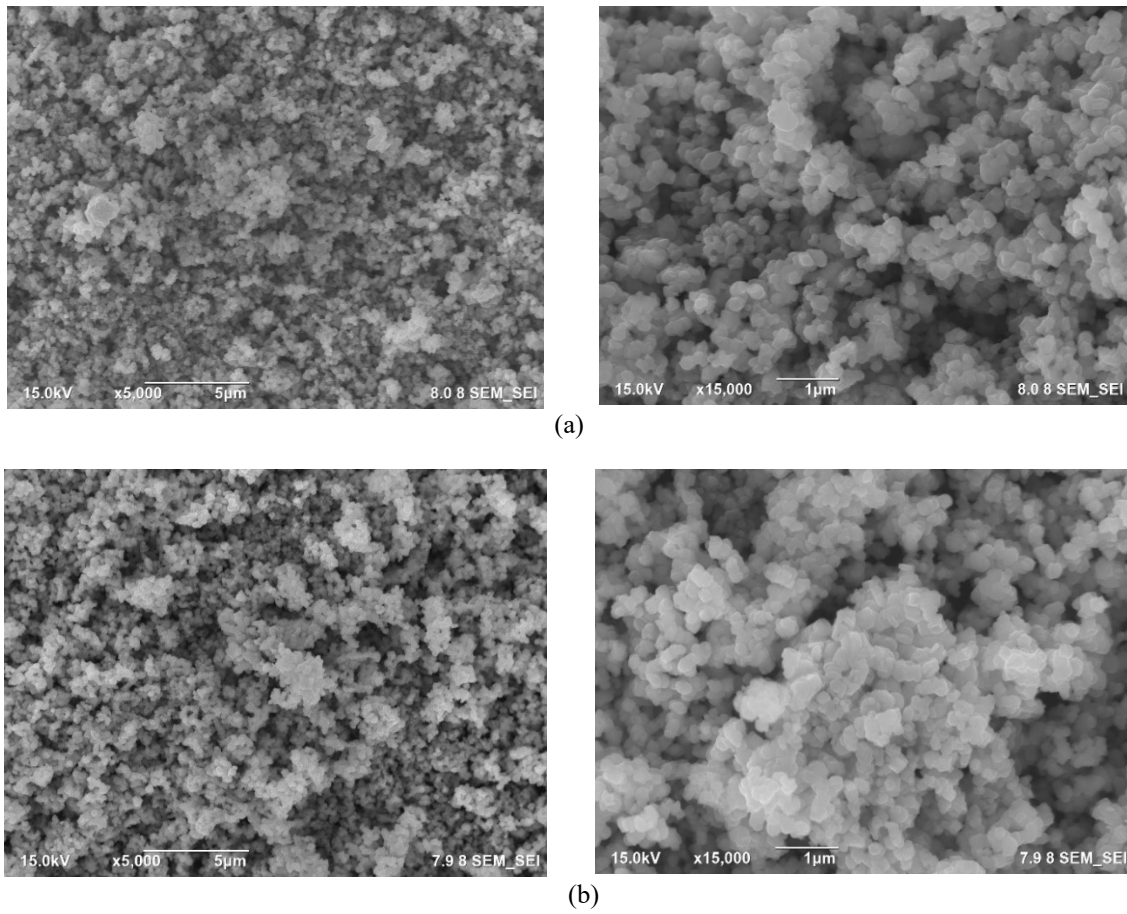


Figure 2. Characterization of $\text{Fe}_2\text{O}_3/\text{TiO}_2$ composites using SEM with mass ratios (a) 1 and (b) 3

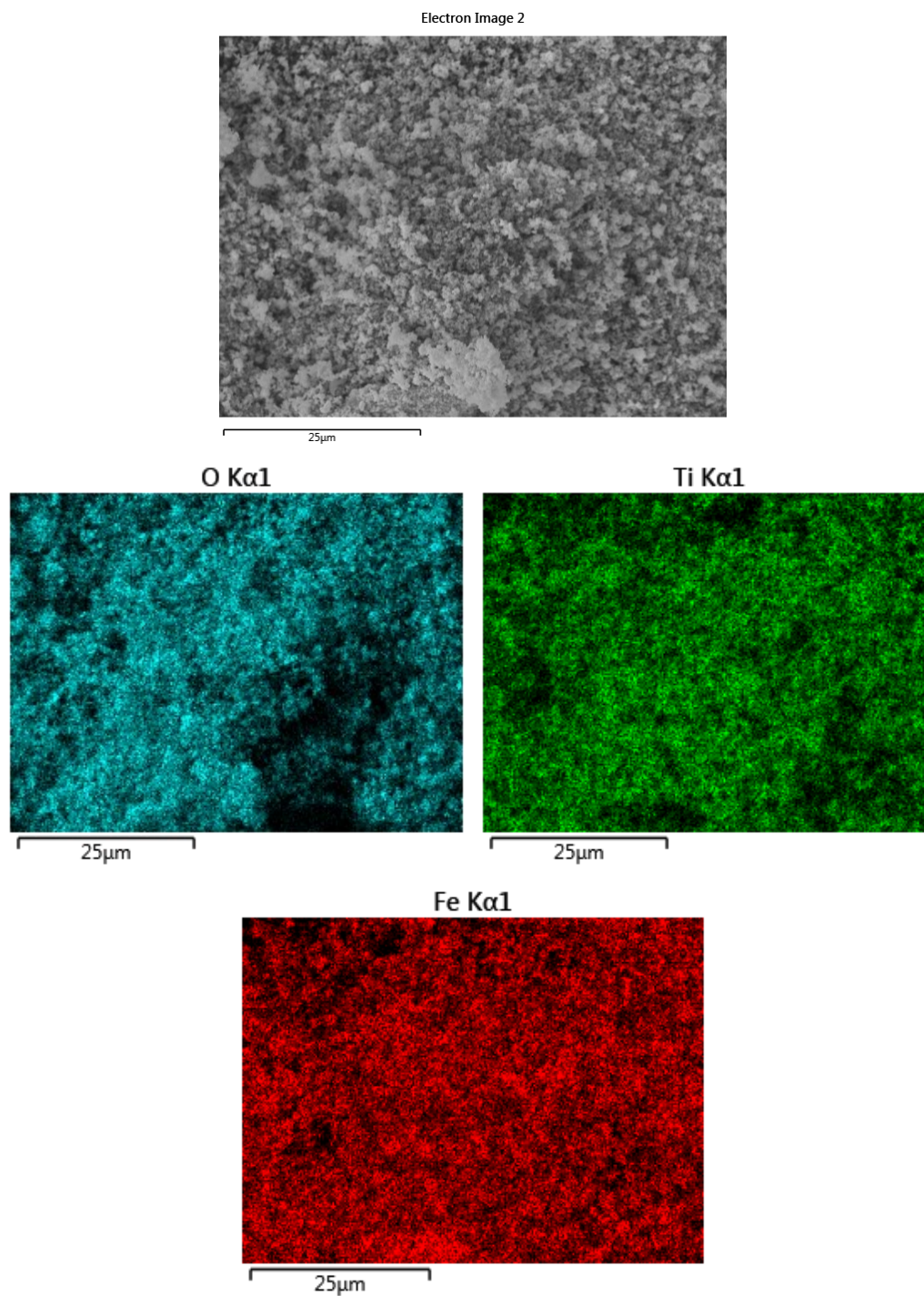


Figure 3. Distribution of Ti, Fe, and O elements in $\text{Fe}_2\text{O}_3/\text{TiO}_2$ composites

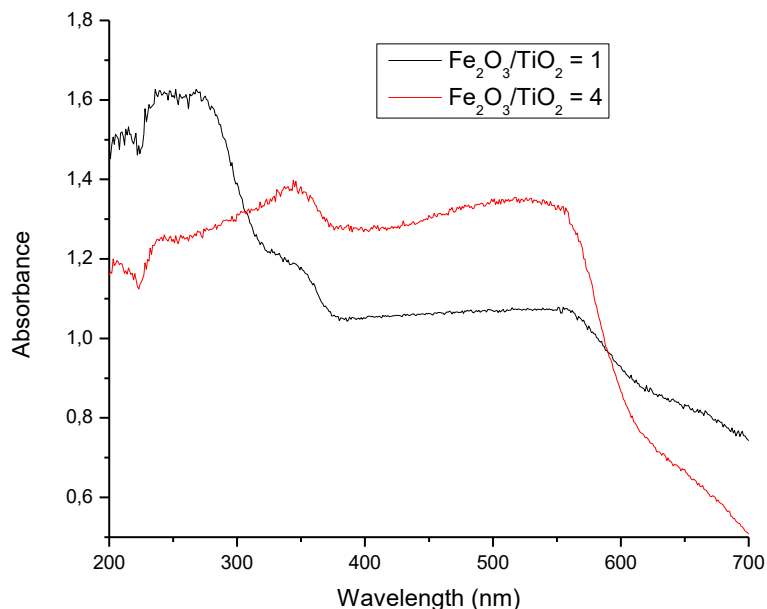


Figure 4. Absorbance spectra to the wavelength of $\text{Fe}_2\text{O}_3/\text{TiO}_2$ composites

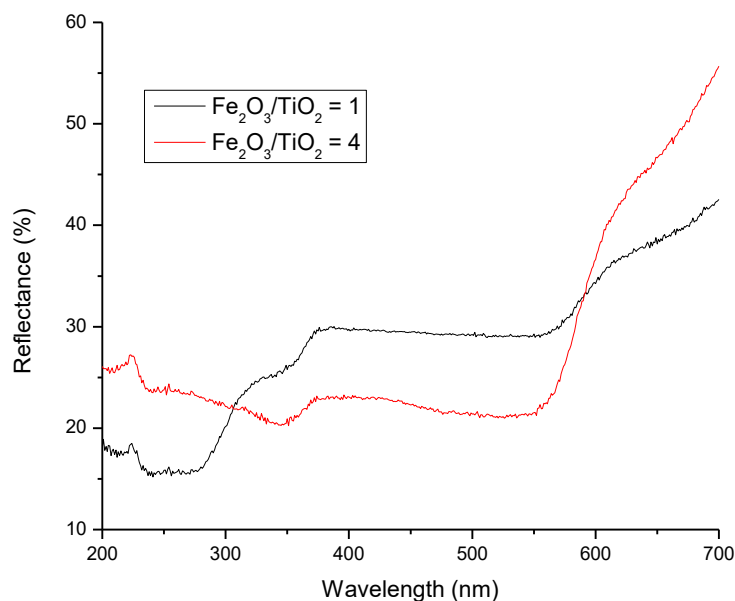


Figure 5. Reflectance spectra to the wavelength of $\text{Fe}_2\text{O}_3/\text{TiO}_2$ composites

4. CONCLUSIONS

The $\text{Fe}_2\text{O}_3/\text{TiO}_2$ composite had good microstructural characteristics based on the diffraction pattern, which showed conformity with JCPDS No. 21-1272 for TiO_2 and JCPDS No. 39-1346 for Fe_2O_3 . Characterization using SEM-EDX showed good particle uniformity and elemental distribution. In addition, the optical characteristics of $\text{Fe}_2\text{O}_3/\text{TiO}_2$ composites indicate the absorbance of the material visible

light wavelengths as the amount of Fe_2O_3 increases.

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