SYNTHESIS AND CHARACTERIZATION ANALYSIS OF Fe₃O₄/SiO₂ CORE SHELL

Divas Cassia Mayora¹,*, Iwan Sugihartono¹, Isnaeni², Haswi Purwandanu Soewoto³, Akhmad Futukhillah Fataba Alaih⁴, Djoko Triyono⁴, Rahmat Setiawan Mohar², Tan Swee Tiam⁵

¹Department of Physics, Faculty of Mathematics and Natural Sciences, Universitas Negeri Jakarta, Jl. Rawamangun Muka No. 01, Rawamangun 13220, Indonesia
²Center for Photonics Research. National Research and Innovation Agency (BRIN), KST B.J. Habibie, South Tangerang, Banten 15314, Indonesia
³Center for Mining Technology, National Research and Innovation Agency (BRIN), KST B.J. Habibie, South Tangerang, Banten 15314, Indonesia
⁴Department of Physics, Faculty of Mathematics and Natural Sciences, Universitas Indonesia, New Campus Universitas Indonesia, Depok, West Java 16426, Indonesia
⁵School of Energy and Chemical Engineering, Xiamen University Malaysia, Selangor Darul Ehsan 43900, Malaysia

*Corresponding Author Email: dcmayora@gmail.com

ABSTRACT

Fe₃O₄/SiO₂ core shell synthesis has been carried out, and its characteristics have been analyzed to be used as a photocatalyst using co-precipitation and sol-gel method. Fe₃O₄ is used as the core material to eliminate the difficulty of separating the photocatalyst powder from the solution medium, SiO₂ is used to avoid oxidation by preventing the Fe₃O₄ core from coming into direct contact with the solution. The samples were then characterized using X-Ray Diffraction (XRD), Fourier Transform Infra-Red (FTIR), and Vibrating Sample Magnetometer (VSM). The XRD and FTIR results showed that the crystal structure and movement of the sample molecules matched the standard characteristics from previous studies. The VSM characterization results show that SiO₂ succeeded in reducing the value of the magnetization of Fe₃O₄, which proves that Fe₃O₄ has been successfully coated by SiO₂.

Keywords: core shell, stober, crystal structure, magnetization
INTRODUCTION

Fe$_3$O$_4$ is utilized as the core material to facilitate the separation of the photocatalyst powder from the solution medium, addressing the difficulty of separation. Additionally, Fe$_3$O$_4$ nanoparticles possess magnetic, optical, and catalytic properties, making them suitable for catalytic applications [1-2]. Fe$_3$O$_4$ particles are superior particles used for catalytic removal of toxic elements from industrial waste products [3-5]. Exposed Fe$_3$O$_4$ has unstable chemical properties and easily agglomerates. Therefore, many substances such as SiO$_2$, TiO$_2$, Ag, and graphene have been used to coat Fe$_3$O$_4$ to form a core shell structure, so as to improve the stability, dispersion, and biocompatibility of Fe$_3$O$_4$ [6]. In this study, Silica (SiO$_2$) was used as a protective shell to prevent oxidation and to improve the stability, dispersion, and biocompatibility of Fe$_3$O$_4$ nanoparticles, which can improve the performance of Fe$_3$O$_4$ nanoparticles in the attachment of organic molecules to the nanoparticle surface [7]. However, SiO$_2$ coating can reduce agglomeration as well as uneven dispersion of Fe$_3$O$_4$ particles [8]. In this case, the core-shell Fe$_3$O$_4$/SiO$_2$ is used as a composite photocatalyst. Thus, Fe$_3$O$_4$ is used as the core material to eliminate the difficulty in separating the photocatalyst powder from the solution medium because Fe$_3$O$_4$ excels at removing toxic elements from industrial waste products [9]. In the interim, SiO$_2$ is used to avoid oxidation by preventing the Fe$_3$O$_4$ core from coming into direct contact with the solution. It’s good at absorbing water, stays stable, and can be used by living things. After forming the Fe$_3$O$_4$/SiO$_2$ core shell, characterization testing was carried out on the morphology of the layer using the structure and size of crystals using X-Ray Diffraction (XRD), molecular movement using Fourier Transform Infra-Red (FTIR), and Vibrating Sample Magnetometer (VSM).

METHOD

Synthesis of Fe$_3$O$_4$

Synthesis of Fe$_3$O$_4$ was carried out using the co-precipitation method by mixing 40.55 g of 0.5 M FeCl$_3$ with 34.75 g of 0.25 M FeSO$_4$·7H$_2$O into 500 ml of deionized water. The solution was then stirred using a magnetic stirrer for 2 hours while 80 ml of NH$_4$OH was slowly added dropwise. The solution was subsequently left to precipitate overnight, washed ten times, and then calcined at 50°C for 2 hours to produce Fe$_3$O$_4$ powder.

Synthesis of Fe$_3$O$_4$/SiO$_2$

The synthesis of Fe$_3$O$_4$/SiO$_2$ was carried out using the Stober method by mixing 1 gram of Fe$_3$O$_4$ sample into a mixture of 400 ml ethanol and 100 ml deionized water. The solution was then subjected to ultrasonic vibration at 80 kHz for 15 minutes. Subsequently, the solution was mixed with 25 ml of 25% NH$_3$ and subjected to ultrasonic vibration at 80 kHz for 5 minutes. The solution was then dropwise added with 10 ml of TEOS while being subjected to ultrasonic vibration at 80 kHz for 2 hours. The solution was then washed and left overnight until dry to produce Fe$_3$O$_4$/SiO$_2$ powder.
Characterization of Fe₃O₄/SiO₂

The characterization of Fe₃O₄/SiO₂ is conducted by utilizing three distinct characterization techniques. Initially, X-Ray Diffraction (XRD) was employed to characterize the crystal structure of the sample. The results demonstrate the presence of Fe₃O₄ and SiO₂, as well as the crystal structure of the Fe₃O₄ nucleus. Subsequently, analyze the Fourier Transform Infra-Red (FTIR) to identify functional groups present in the sample, such as Fe-O and Si-O bonds. Lastly, the characterization of the Vibrating Sample Magnetometer (VSM) to assess the magnetization of the sample, showing a reduction in magnetization of Fe₃O₄ after being coated with SiO₂. This characterization provides insight into the crystal structure, functional groups, and magnetic properties of Fe₃O₄/SiO₂ core shell composites, confirming the successful synthesis and coating of core materials with SiO₂.

RESULT AND DISCUSSION

FIGURE 1 shows the XRD patterns of Fe₃O₄, Fe₃O₄/SiO₂. Based on figure, XRD characterization results revealed distinct peaks at 2θ values of 30.90°, 35.67°, 47.20°, 54.83°, and 62.84°, which correspond to the characteristic peaks of Fe₃O₄ as reported in previous studies [10]. These specific peaks are consistent with the crystallographic patterns associated with Fe₃O₄, confirming the presence of this phase within the sample. The XRD pattern distinctly showcased a single-phase structure dominated by Fe₃O₄, while the peaks corresponding to SiO₂ were notably absent. The absence of SiO₂ peaks can be attributed to its amorphous nature, where in the lack of a regular crystalline structure makes it indistinguishable in the XRD pattern.
Furthermore, the absence of discernible peaks for SiO\textsubscript{2} in the XRD pattern underscores its amorphous nature, as opposed to the crystalline Fe\textsubscript{3}O\textsubscript{4} phase. The amorphous characteristics of SiO\textsubscript{2} prevent the identification of specific crystallographic orientations or phases using XRD. This observation aligns with the intrinsic properties of SiO\textsubscript{2}, where its lack of long-range order results in a diffuse scattering pattern rather than distinct peaks. Consequently, the comprehensive XRD characterization elucidates the predominant presence of Fe\textsubscript{3}O\textsubscript{4} and the amorphous nature of SiO\textsubscript{2} within the sample, providing valuable insights into its structural composition and phase distribution.

FIGURE 2. Graphics FTIR of (a) Fe\textsubscript{3}O\textsubscript{4}, (b) Fe\textsubscript{3}O\textsubscript{4}/SiO\textsubscript{2}

The results of Fourier Transform Infrared (FTIR) spectroscopy testing on Fe\textsubscript{3}O\textsubscript{4} nanoparticle samples and core-shell Fe\textsubscript{3}O\textsubscript{4}/SiO\textsubscript{2} nanocomposites are shown in FIGURE 2. The FTIR spectra, which cover a spectral range from 400 to 4000 cm\textsuperscript{-1}, provide important insights into the vibrational frequencies associated with specific functional groups that are present in the samples. It is possible to identify and characterize the distinctions in the samples by examining the matching peaks in the FTIR spectra.

Additionally, a thorough understanding of the chemical bonding and interactions inside the Fe\textsubscript{3}O\textsubscript{4} nanoparticle samples and Fe\textsubscript{3}O\textsubscript{4}/SiO\textsubscript{2} core-shell nanocomposites is made possible by the identification of particular functional groups using FTIR research. It is possible to determine with certainty which functional categories correspond to the observed peaks by comparing them with previous investigations and published literature [11]. The generated Fe\textsubscript{3}O\textsubscript{4}/SiO\textsubscript{2} nanocomposites' composition and structure are validated by this comparative analysis, which also makes it easier to understand their possible uses and characteristics based on the detected functional groups.

<table>
<thead>
<tr>
<th>Functional Group</th>
<th>Wavenumber (cm\textsuperscript{-1})</th>
<th>Reference Wavenumber (cm\textsuperscript{-1})</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-O-Fe</td>
<td>576</td>
<td>572-587</td>
<td>[12]</td>
</tr>
<tr>
<td>Si-O-Si</td>
<td>1093</td>
<td>1091</td>
<td>[13]</td>
</tr>
<tr>
<td>O-H</td>
<td>1637</td>
<td>1637</td>
<td>[14]</td>
</tr>
</tbody>
</table>
FIGURE 3 shows the Vibrating Sample Magnometer (VSM) of Fe₃O₄ nanoparticles and Fe₃O₄/SiO₂ nanocomposites. Based on the figure, the magnetic curves were determined to be 42.18 emu/g and 30.41 emu/g, respectively. This observed difference in magnetization values provides significant insights into the coating process of Fe₃O₄ nanoparticles with SiO₂. Specifically, the reduction in magnetization value for the Fe₃O₄/SiO₂ nanocomposites compared to pure Fe₃O₄ nanoparticles suggests an alteration in the magnetic properties, indicating the incorporation of SiO₂ onto the Fe₃O₄ surface. Furthermore, the degradation in the Fe₃O₄ magnetization value further substantiates the successful coating of Fe₃O₄ nanoparticles by SiO₂ [15].

The decrease in VSM results can be attributed to the successful coating of Fe₃O₄ with SiO₂. This reduction in magnetization indicates that the core material, Fe₃O₄, was effectively shielded by the SiO₂ shell, confirming the formation of the core-shell structure. The SiO₂ coating prevents direct contact of Fe₃O₄ with the solution, thereby reducing magnetization. This finding supports the intended purpose of SiO₂ as a protective coating for Fe₃O₄, preventing oxidation and ensuring the stability of the core-shell structure.

CONCLUSION

Core shell Fe₃O₄/SiO₂ has been successfully synthesized by co-precipitation and Stober methods and characterized Fe₃O₄/SiO₂ core-shell nanoparticles for use as photocatalysts. The results showed that the crystal structure and movement of the sample molecules are aligned with the standard characteristics, and SiO₂ effectively reduces the magnetization of Fe₃O₄.
This confirms that SiO\(_2\) is suitability for coating Fe\(_3\)O\(_4\), highlighting the successful formation of core-shell structures for potential photocatalytic applications.

**REFERENCES**


