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SYNTHESIS AND CHARACTERIZATION ANALYSIS OF FE_3O_4/SiO_2 CORE SHELL

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ABSTRACT

Fe_3O_4/SiO_2 core shell synthesis has been carried out, and its characteristics has been analyzed to be used as a photocatalyst using co-precipitation and sol-gel method. Fe_3O_4 is used as the core material to eliminate the difficulty of separating the photocatalyst powder from the solution medium, SiO_2 is used to avoid oxidation by preventing the Fe_3O_4 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 SiO_2 succeeded in reducing the value of the magnetization of Fe_3O_4, which proves that Fe_3O_4 has been successfully coated by SiO_2.

Keywords: core shell, stober, crystal structure, magnetization

INTRODUCTION

Fe_3O_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_3O_4 nanoparticles possess magnetic, optical, and catalytic properties, making them suitable for catalytic applications [1-2]. Fe_3O_4 particles are superior particles used for catalytic removal of toxic elements from industrial waste products [3-5].

Exposed Fe_3O_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_3O_4 to form a core shell structure, so as to improve the stability, dispersion, and biocompatibility of Fe_3O_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_3O_4 nanoparticles, which can improve the performance of Fe_3O_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_3O_4 particles [8].

In this case, the core-shell Fe_3O_4/SiO_2 is used as a composite photocatalyst. Thus, Fe_3O_4 is used as the core material to eliminate the difficulty in separating the photocatalyst powder from the solution medium because Fe_3O_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_3O_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_3O_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_3O_4

Synthesis of Fe_3O_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 $FeSO_4.7H_2O$ into 500 ml of deionized water. The solution was then stirred using a magnetic stirrer for 2 hours while 80 ml of NH_4OH 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_3O_4 powder.

Synthesis of Fe_3O_4/SiO_2

The synthesis of Fe_3O_4/SiO_2 was carried out using the Stober method by mixing 1 gram of Fe_3O_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_3O_4/ SiO_2 powder.

Characterization of Fe_3O_4/SiO_2

The characterization of Fe_3O_4/SiO_2 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 Fe_3O_4 and SiO_2, as well as the crystal structure of the Fe_3O_4 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 Fe_3O_4 after being coated with SiO_2. This characterization provides insight into the crystal structure, functional groups, and magnetic properties of Fe_3O_4/SiO_2 core shell composites, confirming the successful synthesis and coating of core materials with SiO_2.

RESULT AND DISCUSSION

FIGURE 1 shows the XRD patterns of Fe_3O_4, Fe_3O_4/SiO_2. 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 Fe_3O_4 as reported in previous studies [10]. These specific peaks are consistent with the crystallographic patterns associated with Fe_3O_4, confirming the presence of this phase within the sample. The XRD pattern distinctly showcased a single-phase structure dominated by Fe_3O_4, while the peaks corresponding to SiO_2 were notably absent. The absence of SiO_2 peaks can be attributed to its amorphous nature, where in the lack of a regular crystalline structure makes it indistinguishable in the XRD pattern.

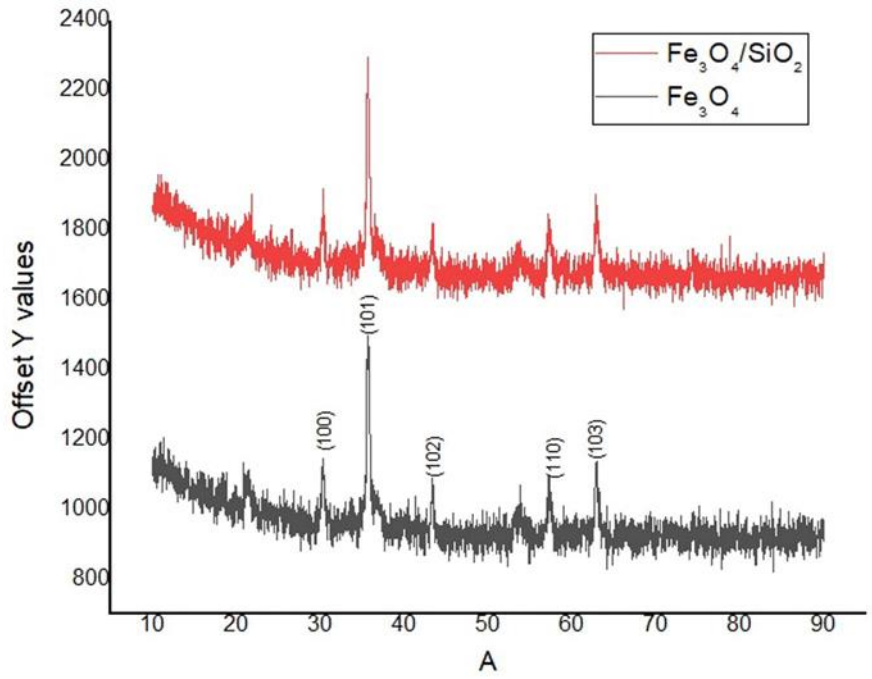

FIGURE 1. Diffraction Patterns of Fe_3O_4, Fe_3O_4/SiO_2

Furthermore, the absence of discernible peaks for SiO_2 in the XRD pattern underscores its amorphous nature, as opposed to the crystalline Fe_3O_4 phase. The amorphous characteristics of SiO_2 prevent the identification of specific crystallographic orientations or phases using XRD. This observation aligns with the intrinsic properties of SiO_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_3O_4 and the amorphous nature of SiO2 within the sample, providing valuable insights into its structural composition and phase distribution.

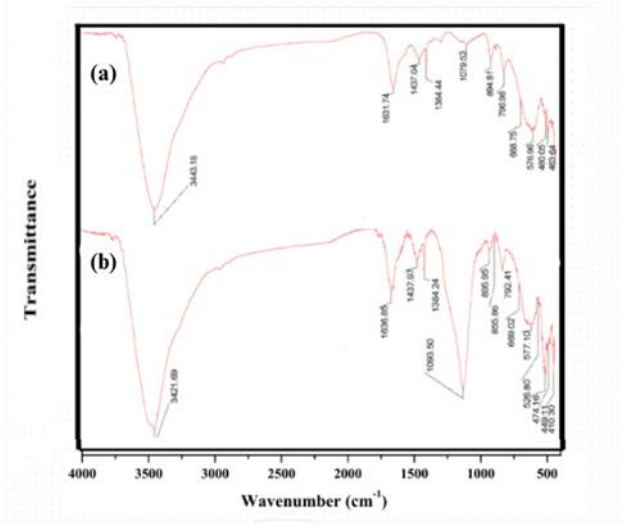

FIGURE 2. Graphics FTIR of (a) Fe_3O_4, (b) Fe_3O_4/SiO_2

The results of Fourier Transform Infrared (FTIR) spectroscopy testing on Fe3O4 nanoparticle samples and core-shell Fe3O4/SiO2 nanocomposites are shown in FIGURE 2. The FTIR spectra, which cover a spectral range from 400 to 4000 cm-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 Fe3O4 nanoparticle samples and Fe3O4/SiO2 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 Fe3O4/SiO2 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 1. Functional Group References

Functional Group	Wavenumber (cm^{-1})	Reference Wavenumber (cm^{-1})	Reference
Fe-O-Fe	576	572-587	[12]
Si-O-Si	1093	1091	[13]
O-H	1637	1637	[14]

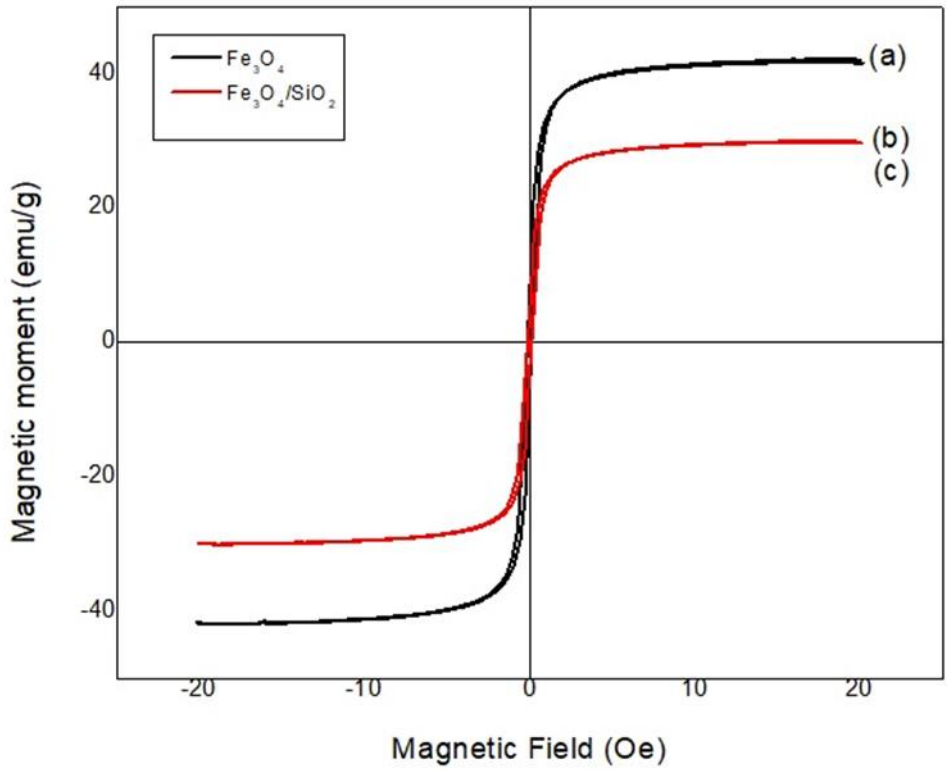

FIGURE 3. Magnetization Curves of (a) Fe_3O_4, (b) Fe_3O_4 /SiO_2

FIGURE 3 shows the Vibrating Sample Magnometer (VSM) of Fe_3O_4 nanoparticles and Fe_3O_4/SiO_2 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 Fe_3O_4 nanoparticles with SiO_2. Specifically, the reduction in magnetization value for the Fe_3O_4/SiO_2 nanocomposites compared to pure Fe_3O_4 nanoparticles suggests an alteration in the magnetic properties, indicating the incorporation of SiO_2 onto the Fe_3O_4 surface. Furthermore, the degradation in the Fe_3O_4 magnetization value further substantiates the successful coating of Fe_3O_4 nanoparticles by SiO_2 [15].

The decrease in VSM results can be attributed to the successful coating of Fe_3O_4 with SiO_2. This reduction in magnetization indicates that the core material, Fe_3O_4, was effectively shielded by the SiO_2 shell, confirming the formation of the core-shell structure. The SiO_2 coating prevents direct contact of Fe_3O_4 with the solution, there by reducing magnetization. This finding supports the intended purpose of SiO_2 as a protective coating for Fe_3O_4, preventing oxidation and ensuring the stability of the core-shell structure.

CONCLUSION

Core shell Fe_3O_4/SiO_2 has been successfully synthesized by co-precipitation and Stober methods and characterized Fe_3O_4/SiO_2 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 SiO_2 effectively reduces the magnetization of Fe_3O_4.

This confirms that SiO_2 is suitability for coating Fe_3O_4, highlighting the successful formation of core-shell structures for potential photocatalytic applications.

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