

The effect of silica mass ratio on pore structure and magnetic characteristics of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ core-shell nanoparticles

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Tetra-Ethyl Ortho-Silicate
(TEOS);
Magnetic nanoparticle.

Abstract

The fabrication of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ core-shell was prepared from natural iron sand as Fe_3O_4 core resource and in situ SiO_2 -coating method. $\text{Fe}_3\text{O}_4@\text{SiO}_2$ was synthesized via an ultrasonic route with various ratios of Tetra-Ethyl Ortho-Silicate (TEOS) to evaluate the morphological, pore structure, and magnetic properties of the core-shell. X-Ray Diffraction (XRD) and Fourier Transform Infra-Red (FTIR) were used to characterize the prepared Fe_3O_4 and $\text{Fe}_3\text{O}_4@\text{SiO}_2$. Scanning Electron Microscope (SEM) was used to analyze the effect of TEOS on the particle size, and Transmission Electron Microscopy (TEM) was used to observe the morphology of core-shell and shell thickness. The Brunauer-Emmett-Teller (BET) data revealed that $\text{Fe}_3\text{O}_4@\text{SiO}_2$ (65) exhibited a larger pore diameter of 88.17 nm and eight-times higher BET surface area (80.23 m^2/g) than $\text{Fe}_3\text{O}_4@\text{SiO}_2$ (55) and $\text{Fe}_3\text{O}_4@\text{SiO}_2$ (45) (27.69 nm; 10.5 m^2/g). The Vibrating Sample Magnetometer (VSM) data indicated that increased TEOS addition on $\text{Fe}_3\text{O}_4@\text{SiO}_2$ caused a decrease in the magnetization value but still gave good magnetic properties from 95.32 emu/g for $\text{Fe}_3\text{O}_4@\text{SiO}_2$ (45) to 17.02 emu/g for $\text{Fe}_3\text{O}_4@\text{SiO}_2$ (65). The study found that the higher SiO_2 content reduced the agglomeration of the Fe_3O_4 core, as indicated by no hysteresis loop on the gas of Nitrogen (N_2) adsorption-desorption curve of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ (65), resulting in core-shell material with better properties in higher specific surface area, average pore size and volume for further application.

1. Introduction

Recently, Fe_3O_4 nanoparticles have provided unique material properties and have a significant role in various applications due to their essential superparamagnetic properties, which show high saturation magnetization but remnant magnetism and low coercivity [1]. For example, the Fe_3O_4 magnetic feature eases the separation of material from the mixed system, such as for the adsorption application [2], catalyst in the reaction [3], and drug delivery system [4]. Indonesia has abundant iron sand that can be used as a Fe_3O_4 source. This iron sand has been reported to produce Fe_3O_4 , replacing the Fe_3O_4 synthetics route from expensive FeCl_3 material [5,6]. Many research attempts are related to studying chemical stability, structure, magnetic properties, and synthesis methods

to support its Fe_3O_4 magnetic application [7,8]. The problem with using Fe_3O_4 in the direct application is that it is chemically unstable due to the high surface energy of Fe_3O_4 nanoparticles, causing particle agglomeration [9]. One of the most effective methods to cope with the problem is to incorporate/combine amorphous silica material with Fe_3O_4 Nanoparticles (NPs). Amorphous mesoporous silica material has a non-toxic nature, a highly specific surface, an ordered mesoporous structure, tunable pore size, and volumes with abundant Si-OH bonds on the pore surface [10].

Core-shell nanomaterials with magnetic Fe_3O_4 nanoparticles as the core have been successfully synthesized previously, such as $\text{Fe}_3\text{O}_4@\text{TiO}_2$ [11], $\text{Fe}_3\text{O}_4@\text{Ag}$ [12], and

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$\text{Fe}_3\text{O}_4@\text{ZnO}$ [13] using hydrothermal techniques; $\text{Fe}_3\text{O}_4@\text{SiO}_2$ was carried out using the Stöber method [14]. The core-shell nanoparticles showed superparamagnetic behavior at room temperature, and their toxicity was evaluated using hepatocellular carcinoma liver tumor cells. Furthermore, it has potential applications in biomedical and pharmaceutical fields thanks to its excellent properties, such as increased thermal and chemical stability, dispersibility, low toxicity [6,15], pH-responsive drug release, and biocompatibility [15,16]. Core-shell $\text{Fe}_3\text{O}_4@\text{SiO}_2$ and its modification with Ag doping, $\text{Fe}_3\text{O}_4@\text{SiO}_2\text{-Ag}$ is very effective as an anti-bacterial agent (*E. Coli* and *S. Aureus*) and an anticancer drug carrier [17], also helpful in designing thermal seeds in magnetic hyperthermia therapy [18], and for environmental applications [15–22].

Modifications of the Fe_3O_4 magnetic surface with SiO_2 enable selective targeting by forming functional groups on the surface through chemical bonds (covalent, ionic, or hydrogen). Due to their hydrophilic properties, this silica coating feature possesses excellent dispersibility and chemical stability in aqueous solutions [23]. However, the fabrication of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ core-shell is still challenging due to the aggregation of multiple Fe_3O_4 magnets during surface coating. The aggregation of the Fe_3O_4 core impacts the decrease of the surface area and limits their applications, such as in adsorption and bio immobilization [24]. $\text{Fe}_3\text{O}_4@\text{SiO}_2$, as a drug carrier or adsorbent, needs a high Surface-to-Volume (S/V) ratio, which can lead to more robustness in their application [25,26]. The surface-to-volume ratio of nanomaterial plays a vital role because the high S/V increases the biomolecule or dye molecule density that can be adsorbed in the $\text{Fe}_3\text{O}_4@\text{SiO}_2$ surface [27]. Therefore, the additional silica ratio and the thickness of the silica shell on the $\text{Fe}_3\text{O}_4@\text{SiO}_2$ significantly affect the aggregation of Fe_3O_4 , the pore structure, and the characteristics of the core shell [26,28]. The Fe_3O_4 with uniform and controlled thickness silica coating is still being challenged. Therefore, adjusting silica content is needed to tailor the end properties of the material.

In this study, we report the facile synthesis of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ with the in-situ method. The Fe_3O_4 core was prepared from iron sand by the co-precipitation method. The incorporation of SiO_2 to Fe_3O_4 was adjusted according to the silica ratio on the $\text{Fe}_3\text{O}_4@\text{SiO}_2$. Silica coating parameters are used to tune this silica layer's thickness and minimize the Fe_3O_4 particle agglomeration. The magnetic incorporation of SiO_2 into Fe_3O_4 is tailored to particle size, pore, surface area, and magnetic properties, which are discussed in this paper. By adjusting the silica ratio on the $\text{Fe}_3\text{O}_4@\text{SiO}_2$, the thickness of the $\text{Fe}_3\text{O}_4@\text{SiO}_2$ shell can be controlled and significantly impact the increment of the core shell's surface area and pore diameter. This feature of silica microspheres with magnetic properties is an available prospect for the wide potential application of iron sand as raw material for better uses.

2. Materials and methods

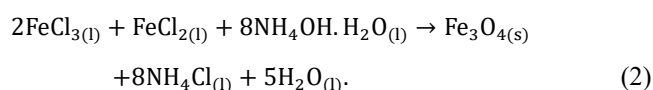
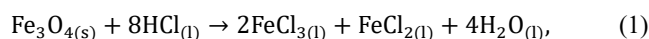
2.1. Materials

All chemicals were in analytical grade and used without any further purification. Iron sand (98% Fe_3O_4), Hydrochloric Acid (HCl, Pro analysis (37%)), Ammonium Hydroxide (NH_4OH), Ethanol ($\text{C}_2\text{H}_5\text{OH}$, 96%), Ammonia ($\text{NH}_3\cdot\text{H}_2\text{O}$, 25%), Tetraethyl Orthosilicate (TEOS, 97%) were purchased from Sigma Aldrich, Distilled water, and Filter paper (Whatman, 60 mm).

2.2. Synthesis method

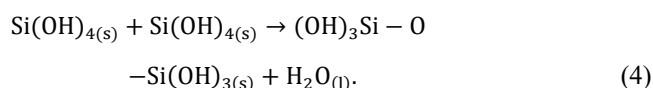
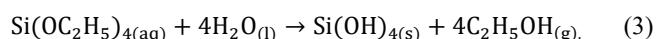
2.2.1. Synthesis of Fe_3O_4 magnetic

The synthesis of Fe_3O_4 nanoparticles was prepared and extracted from iron sand using the co-precipitation method, as done in the previous research [29]. A mass of 20 g of iron sand was dissolved in 38 mL of HCl (2M) and stirred for 10–12 min at 60°C. When the color changed to yellow, 24 mL of NH_4OH (6M) was added and stirred until the color became dark black. The solution was separated and washed with distilled water several times. The Fe_3O_4 was dried in an oven at 70°C for 12 h. The chemical reaction proceeds as follows [16–18]:



2.2.2. Synthesis of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ core-shell

The synthesis process of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ core-shell was carried out using the in-situ coating method as done according to previous reports with a little change [19]. A mass of 1 g of Fe_3O_4 nanoparticle powder was mixed with 24 mL distilled water and dispersed in ultra-sonification for 30 min. Furthermore, 240 mL of ethanol was added to the mixture while stirring until it was interpreted as evenly mixed. Next, 4 mL of ammonia and a variation of mass ratio TEOS addition (4.5, 5.5, and 6.5 mL) were added. The mass variation of TEOS addition in Fe_3O_4 were subsequently denoted as $\text{Fe}_3\text{O}_4@\text{SiO}_2(45)$, as $\text{Fe}_3\text{O}_4@\text{SiO}_2(55)$, and as $\text{Fe}_3\text{O}_4@\text{SiO}_2(65)$. The mixture was stirred for eight hours at room temperature. The resulting solution was brownish-black and washed with distilled water until it reached a pH of 7. The final stage of the resulting precipitate was dried at 60°C for 24 h; these are the chemical reactions of the prepared SiO_2 shell from TEOS:



2.2.3. Mechanism of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ core-shell formation

Figure 1 shows the mechanism for the formation of core-shell $\text{Fe}_3\text{O}_4@\text{SiO}_2$. Tetraethyl Orthosilicate (TEOS) was used as a silica precursor to make a SiO_2 layer on the surface of Fe_3O_4 nanoparticles. This reaction proceeds through

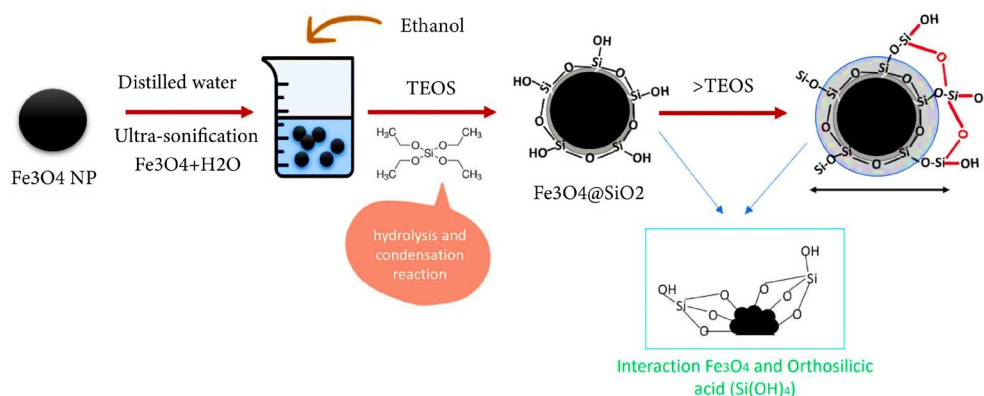


Figure 1. A schematic illustration of the fabrication of a core-shell structure Fe₃O₄@SiO₂ NPs [10,15].

hydrolysis and condensation reaction through the ionization of Si (OC₂H₅)₄. Si (OC₂H₅)₄ reacts with H₂O and is converted through ionization to form ortho silicic acid Si (OH)₄, which can be used as a silicon oxide precursor and ethanol gas (C₂H₅OH). The ortho silicic acid formed binds to the OH group on the surface of the Fe₃O₄ nanoparticles, and the condensation reaction is then repeated to form a SiO₂ layer while forming a network structure around the particles. The dense silica coating factors affecting the mesoporous shell formation are ethanol and ammonia concentrations. The modification of the surface of Fe₃O₄ nanoparticles can be conducted with appropriate ligands or functional groups. This modification is believed to increase its stability and prevent agglomeration. Silane coupling agents, for example, can be used to modify the surface chemistry of Fe₃O₄ nanoparticles. The use of ultrasonication or other dispersion techniques during the synthesis process is to break up lumps. However, be careful in increasing the temperature during ultrasonication, as excessive heat can cause further caking. Optimizing the strength and duration of ultrasonication and mechanical stirring during ultrasonication can help distribute the heat generated by ultrasonication and prevent local overheating.

2.3. Characterizations

All materials preparation was characterized by X-ray diffraction (Paan Analytical, Type: Expert Pro) and scanned at a range of 5°C–75°C. Fourier transform infrared spectroscopy (FTIR, Shimadzu, Tpe: IRPrestige) analyzed functional groups at 400 to 4000 cm⁻¹ wavenumbers. The material morphological structure and particle size were conducted using Scanning Electron Microscopy (SEM, Zeiss Evo MA 10), and the formation of the core-shell structure was monitored using a transmission electron microscope (Hitachi HT7700, USA). The magnetic properties were recorded at room temperature using a Vibrating Sample Magnetometer (VSM; 7404, Lakeshore, USA).

3. Results and discussion

3.1. Structure analysis of Fe₃O₄@SiO₂ magnetic

The X-Ray Diffraction (XRD) patterns of Fe₃O₄, TEOS, and Fe₃O₄@SiO₂ with different amounts of TEOS are shown in

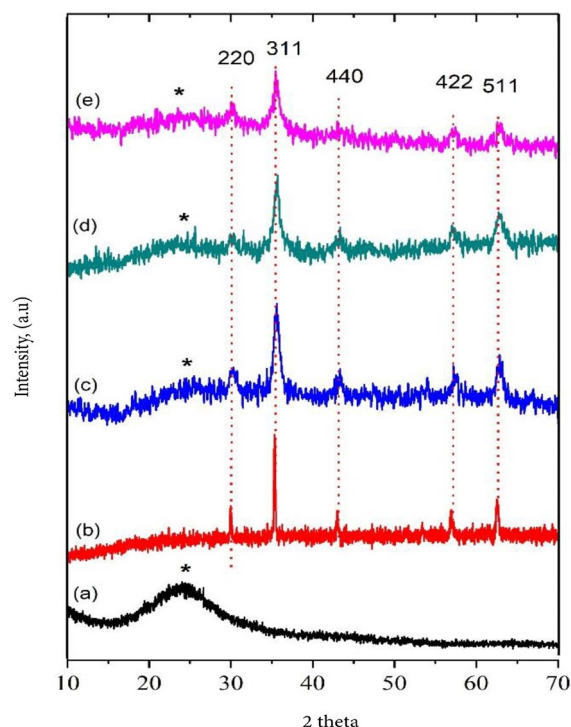
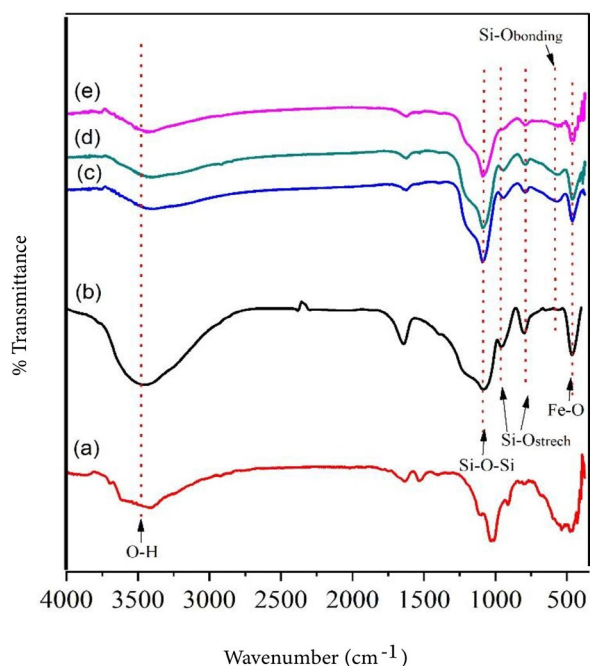


Figure 2. Pattern diffraction of: (a) SiO₂, (b) Fe₃O₄, and (c)-(e) Fe₃O₄@SiO₂ core-shell: 45, 55, and 65.

Figure 2. In all synthesized Fe₃O₄@SiO₂ core-shell, five major 2θ diffraction peaks at 30.2°, 35.5°, 43.1°, 57.1°, and 62.8° observed, which represent (220), (311), (400), (422) and (511) planes of standard Fe₃O₄ magnetic pattern, respectively [29]. The characteristic of amorphous silica SiO₂ was showed in broad diffraction peak at 24°C. This peak of silica is weak in all synthesized materials, which might be due to a small signal-to-noise ratio [5]. The obtained Fe₃O₄@SiO₂ XRD patterns showed that the addition of SiO₂ did not significantly change the structure of Fe₃O₄ and revealed that after coating silica, the core still persevered its crystallinity. Furthermore, as the loading amount of SiO₂ increased, the intensity peak at 35.5°C was decreased, revealing the reduction of Fe₃O₄ crystallinity. In addition, the peak width at 35°C of all synthesized material is broader than that of Fe₃O₄ magnetic. This is because the Fe₃O₄ magnetic surface was under stress between the Fe₃O₄ core and SiO₂ shell

Table 1. Pattern X-ray of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ core-shell nanoparticles.

Peak (2 θ), Samples of ($\text{Fe}_3\text{O}_4@\text{SiO}_2$) Core-Shell			Crystal field (hkl)	Average crystallite size (Debye Scherrer), hkl (311) $D = (0.9\lambda / \beta \cos\theta)$ nm			Ref.
45	55	65		45	55	65	
29.98°	29.07°	30.01°	(220)				[19]
35.39°	35.34°	35.35°	(311)	8.47	14.12	22.59	[7]
43.02°	42.99°	43.00°	(400)				[30]
53.51°	53.44°	53.47°	(422)				[31]
56.96°	56.89°	56.95°	(511)				[6]
62.49°	62.46°	62.49°	(440)				[32]

**Figure 3.** The infrared spectra of (a) Fe_3O_4 , (b) SiO_2 , $\text{Fe}_3\text{O}_4@\text{SiO}_2$ were synthesized by in-situ method with a ratio of (c) TEOS 45, (d) 55, (e) 65.

due to the slight increment of inter-planar distance [10]. The crystallite size of the Fe_3O_4 cores calculated by the Debye Scherrer equation reveals 1.6 nm while the $\text{Fe}_3\text{O}_4@\text{SiO}_2(45)$, $\text{Fe}_3\text{O}_4@\text{SiO}_2(55)$, $\text{Fe}_3\text{O}_4@\text{SiO}_2(65)$ have 8.47, 14.12, and 22.59 nm, respectively. These results reveal that adding silica content increased the crystallite size of the $\text{Fe}_3\text{O}_4@\text{SiO}_2$. This examination of XRD results verifies that the $\text{Fe}_3\text{O}_4@\text{SiO}_2$ are constructed of Fe_3O_4 and SiO_2 (shown in Table 1) [30–32].

3.2. Functional analysis by FTIR of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ magnetic

The characterization of the synthesized $\text{Fe}_3\text{O}_4@\text{SiO}_2$ core-shell with mass ratio variation of SiO_2 is obtained through the Fourier Transform Infra-Red (FTIR) spectrum, represented in Figure 3. The Fe_3O_4 magnetic have adsorption characteristic peaks at 582 and 456 cm^{-1} assigned to the stretching vibration of Fe in the tetrahedral and octahedral region [30], in which these peaks also appear in the magnetic nanoparticles spectrums after TEOS addition. The FTIR spectrum of TEOS showed the adsorption band of Si-O-Si asymmetric vibration, Si-O stretching vibration, and Si-O-Si

Table 2. Magnetic data of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ core-shell nanoparticles by Vibrating Sample Magnetometer (VSM).

Samples	Crystal Size, D (nm)	M_s (emu/g)	H_c (Tesla)	M_r (emu/g)
Fe_3O_4	1.6	95.01	0.067	19.86
$\text{Fe}_3\text{O}_4@\text{SiO}_2(45)$	8.47	80.14	0.040	18.67
$\text{Fe}_3\text{O}_4@\text{SiO}_2(55)$	14.12	23.73	0.014	7.226
$\text{Fe}_3\text{O}_4@\text{SiO}_2(65)$	22.59	17.13	0.013	4.27

symmetric at 1130, 960, and 800 cm^{-1} , respectively [28,32]. This absorption peak is present in all synthesized $\text{Fe}_3\text{O}_4@\text{SiO}_2$ core-shell materials, and Si-O-Si asymmetric vibration intensity increases with increasing silica ratio (TEOS) of Fe_3O_4 magnetic particles. Moreover, the Silanol group (Si-OH) on the surface of SiO_2 at 1629 cm^{-1} [14] is present in all synthesized materials. This confirms that the raw material's functional groups and chemical bondings indicate that the SiO_2 (the shell) was successfully incorporated in Fe_3O_4 (the core).

The remanent magnetization (M_r), coercivity magnetic field (H_c), and saturation (M_s) data obtained by the magnetic hysteresis loop are essential in determining the magnetic properties of the material. The saturation magnetic is a state when the maximal of the external magnetic field can be applied to the material. The coercivity is a magnetic field described as material's resistance to make zero magnetism. This coercivity value determined the nature of paramagnetic. The higher H_c value indicates stronger paramagnetic nature properties [33]. The remanent magnetization is the magnetization residue after removing the external magnetic field. Table 2 compares the magnetic properties of the Fe_3O_4 and Fe_3O_4 after TEOS coating. Fe_3O_4 has the highest H_c value, 0.067 tesla, which exhibited a superparamagnetic nature. The addition of TEOS contributes to lowering the H_c and remanent values along with the larger crystal size obtained by the Debye Scherrer formula (D) from XRD data. A detailed investigation by Li et al. for Fe_3O_4 NPs envisages a decrease of both remanent magnetization and coercivity with an increase in size even when the saturation magnetization increases [34]. This data revealed that the smaller crystal size tended to have a higher value of H_c and M_r . The coercivity field was further discussed in Figure 4. The coercive field of Fe_3O_4 was smaller than the others. $\text{Fe}_3\text{O}_4@\text{SiO}_2(45)$ has a coercivity field similar to Fe_3O_4 . After the addition of more TEOS content on the Fe_3O_4 , the

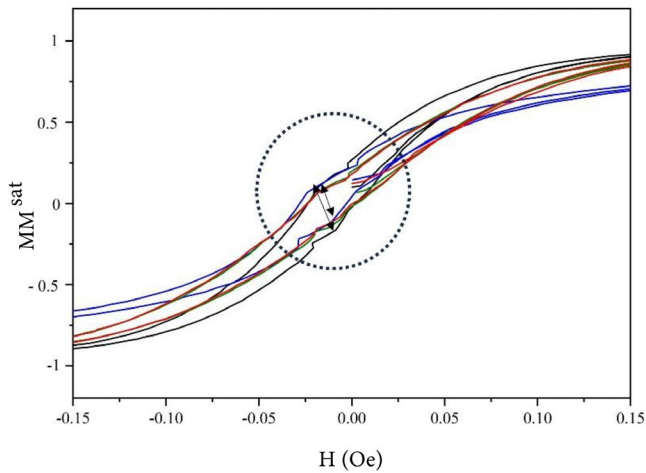


Figure 4. The coercivity field of (a) Fe_3O_4 (red line), (b) $\text{Fe}_3\text{O}_4@\text{SiO}_2$ with TEOS ratio 45 (black line), (c) TEOS ratio 55 (green line), and (d) TEOS ratio 65 (blue line).

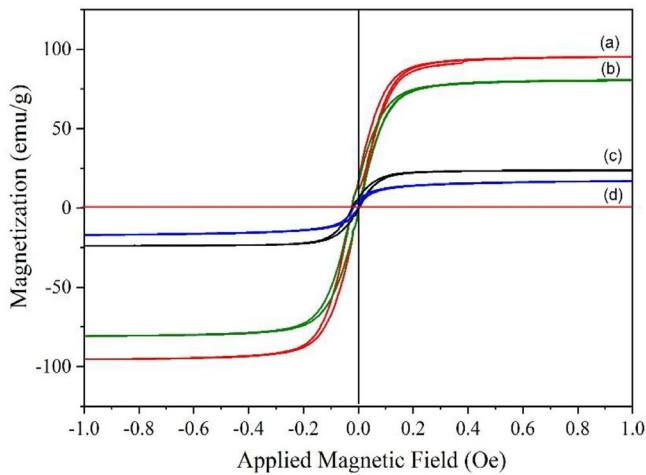


Figure 5. The curve of hysteresis of (a) Fe_3O_4 (red line), (b) $\text{Fe}_3\text{O}_4@\text{SiO}_2$ with TEOS ratio 45 (black line), (c) TEOS ratio 55 (green line), and (d) TEOS ratio 65 (blue line).

coercivity became larger. By designing the $\text{Fe}_3\text{O}_4@\text{SiO}_2$ core-shell structure, the Fe_3O_4 core acts as a soft magnetic (weak magnetic anisotropy which easily magnetized and demagnetized), and the SiO_2 acts as the shell of a hard magnetic (large magnetic anisotropy) [35]. The M_s value, obtained by applying the law of approach to saturation [36], increases with increasing crystal size for all samples. Meanwhile, H_c and remanent magnetization (M_r) are also affected by crystallite size. The high H_c value may be caused by the strong spin interactions in the highly crystalline Fe_3O_4 NPs during spin alignment. The crystalline properties of Fe_3O_4 NPs affect their magnetic properties, especially H_c . This trend is consistent with that found in the previous work [38].

In Figure 5, the results of the measurements of the hysteresis curve for the sample $\text{Fe}_3\text{O}_4@\text{SiO}_2$ core-shell, prepared by the in-situ method, show a trend of decreasing magnetization value with the addition of layers of SiO_2 particle as a covering for the Fe_3O_4 particles. The magnetization saturation values of Fe_3O_4 , $\text{Fe}_3\text{O}_4@\text{SiO}_2$ (45, 55, and 65) were 95.32, 80.58, 23.62, and 17.02 emu/g [29,37]. The

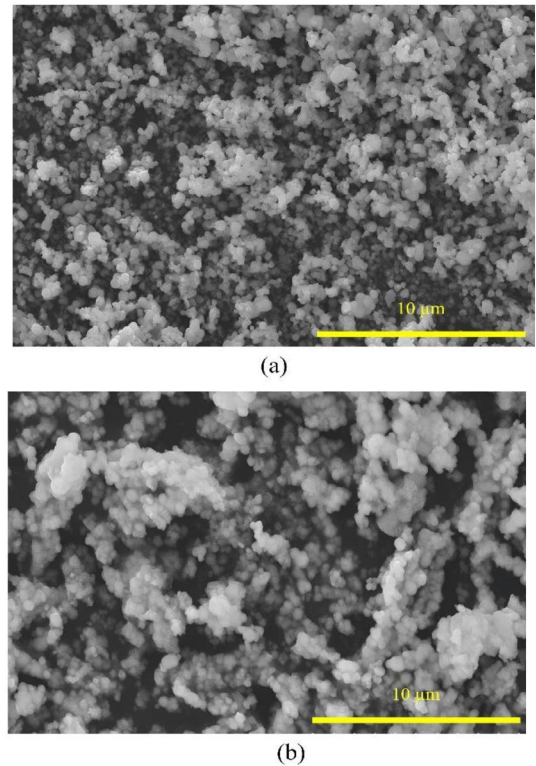


Figure 6. SEM Images of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ core-shells, for TEOS ratio 45 (a) and 65 (b).

magnitude of magnetization decreased from approximately 95.32 emu/g to 17.02 emu/g, approximately 95.32 emu/g to 17.02 emu/g, along with an increase in the ratio mass of TEOS (46, 55, and 65) (see Figure 5(a)-(d)), and the shell thicknesses increased from 142 to 161 nm.

3.3. $\text{Fe}_3\text{O}_4@\text{SiO}_2$ magnetic

The morphology of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ with different Silica ratios was investigated by Scanning Electron Microscope (SEM) image. Figure 6 shows the spherical-like morphology of both synthesized $\text{Fe}_3\text{O}_4@\text{SiO}_2$ materials. The $\text{Fe}_3\text{O}_4@\text{SiO}_2$ with an addition ratio of 45 of TEOS has a slightly rough surface, whereas the surface of the $\text{Fe}_3\text{O}_4@\text{SiO}_2$ with an addition ratio of 65 of TEOS appears to be relatively smooth. The Transmission Electron Microscopy (TEM) images of Fe_3O_4 and synthesized $\text{Fe}_3\text{O}_4@\text{SiO}_2$ are presented in Figure 7. The Fe_3O_4 nanoparticles have spherical shapes and showed an increment of particle size after coating with SiO_2 . The particle size was estimated at 150 nm and 161 nm for $\text{Fe}_3\text{O}_4@\text{SiO}_2$ (45) and $\text{Fe}_3\text{O}_4@\text{SiO}_2$ (65). Meanwhile, the Fe_3O_4 , before coating, has an estimated particle size of 120 nm. Meanwhile, the average particle size observed from SEM and TEM indicates the size of the Fe_3O_4 NPs. The size of the Fe_3O_4 core we obtained exceeds 100 nm because it is probably caused by the Fe_3O_4 NPs synthesis method we used. As reported, the formation of Fe_3O_4 follows the nucleation growth mechanism via the oxidation-precipitation method. Nucleation of magnetite particles occurs when the molar ratio of $\text{Fe}(\text{OH})_2/\text{Fe}(\text{OH})_3$ approaches 1:2, a characteristic value of magnetite structure. Then, Fe_3O_4 nuclei begin to grow.

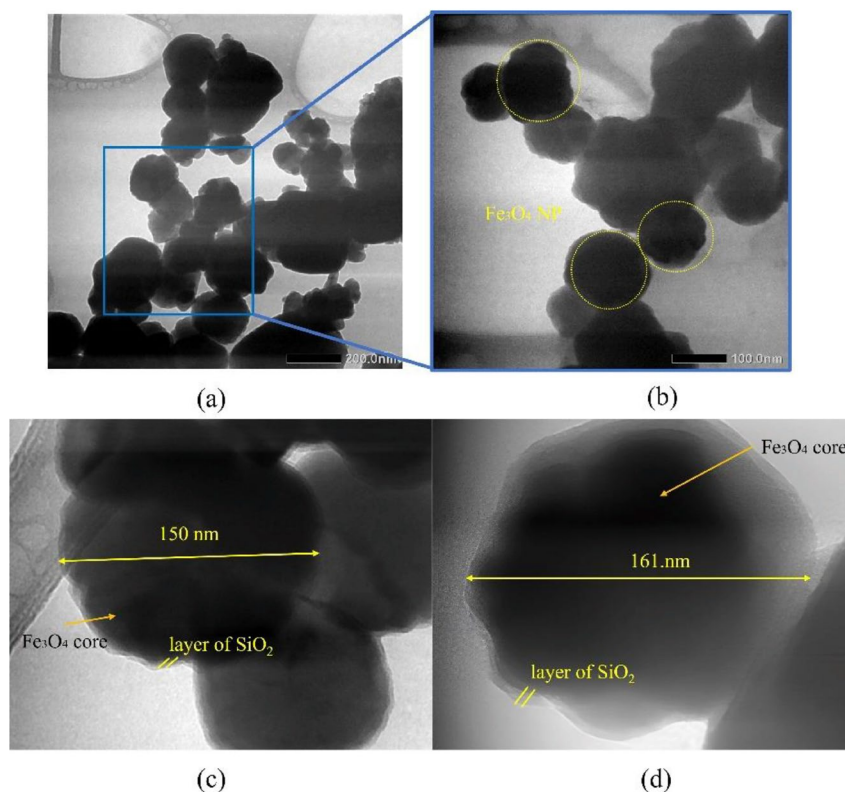


Figure 7. TEM images of Fe₃O₄ and Fe₃O₄@SiO₂ (core-shell): Fe₃O₄ particles (a)-(b) and SiO₂ shell thickness for core-shell with TEOS ratios of (c) 45 and (d) 65.

Therefore, the product size highly depends on reaction conditions such as reaction time, molar ratio of FeSO₄ and oxidizing agent, and concentration of FeSO₄, etc. [21,38]

From the TEM image, it can be seen that the core-shell formation is identified by two different contrasts observed: the dark contrast of the Fe₃O₄ core and the light contrast of the SiO₂ shell. The layer of SiO₂ was estimated to be around 3 nm for Fe₃O₄@SiO₂(45) and 6 nm for Fe₃O₄@SiO₂(65). The existence of SiO₂ in Fe₃O₄ aligns with previous FTIR and XRD results. Furthermore, this SEM and TEM result showed that the additional SiO₂ ratio exhibits different layers, surface roughness, and particle size. The more TEOS addition results in a thicker and smoother layer of SiO₂ and larger particles of Fe₃O₄@SiO₂ [39,40].

The more TEOS addition results in a thicker and smoother layer of SiO₂ and larger particles of Fe₃O₄@SiO₂ [9]. TEOS acts as a silica layer maker on the Fe₃O₄@SiO₂. It started from the corporation of the SiO₂ layer via Fe-O-Si chemical bonding on the surface Fe₃O₄. The addition of TEOS subsequently will construct Si-O-Si silica chain polymers that grow layer by layer, corresponding to shell thickness increment of Fe₃O₄@SiO₂. These chemical bonds were identified in previous FTIR data. Adding TEOS to Fe₃O₄ contributes to the size increment of nanoparticles and the core shell. This finding also aligns with the research result of Prado et al., (2012), which revealed that the higher TEOS/Fe₃O₄ molar ratio would lead to a thicker silica shell [35].

The Fe₃O₄ nanoparticles obtained by co-precipitation due to the large surface-to-volume ratio, high surface energy, and

magnetic dipole-dipole attractions between the particles, and magnetic nanostructures are highly prone to aggregation. Therefore, controlling the core size is necessary by choosing the correct method, solvent, and precursor to obtain the desired core size. To synthesize well-dispersed silica-coated Fe₃O₄ NPs, the sol-gel method, the Stöber method, and microemulsion are the most common methods for coating the surface of Fe₃O₄ nanoparticles with silica. Silica coating on the surface of Fe₃O₄ with various layers cannot completely change the size of the morphology formed.

Energy Dispersive X-ray (EDX) analysis results obtained for Fe₃O₄@SiO₂-core-shell showed that the elemental composition of Si and Fe with the corresponding mass percentage of 4.0% and 51.0% (Fe₃O₄@SiO₂(45)) then 26.0% and 28.0% (Fe₃O₄@SiO₂(65)), respectively. The elemental mapping Figure 8 of Fe₃O₄@SiO₂-core-shell morphology shows that Si is more dominant on the surface than Fe with an increase in the ratio mass of TEOS. This indicates the successful preparation of Fe₃O₄@SiO₂-core-shell in the ratio mass of TEOS, as evidenced by other characterizations.

3.4. N₂ Adsorption-desorption of Fe₃O₄@SiO₂ magnetic

The type of porous structure of synthesized Fe₃O₄@SiO₂ was further analyzed by the N₂ adsorption-desorption experiment in Figure 9 and summarized in Table 3. The hysteresis loop of obtained synthesized material was classified as a type IV pattern for all materials microspheres, which show mesoporous characteristics [40]. The isotherm of Fe₃O₄ with a lower Si amount (ratio 45) showed a notable hysteresis loop

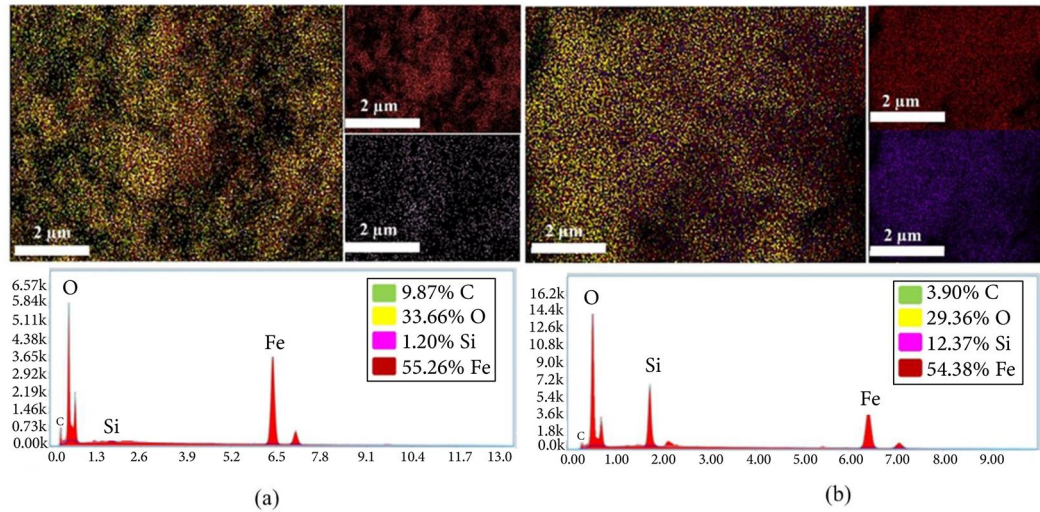


Figure 8. EDX mapping of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ core-shells nanoparticles with TEOS ratio: (a) 45 and (b) 65.

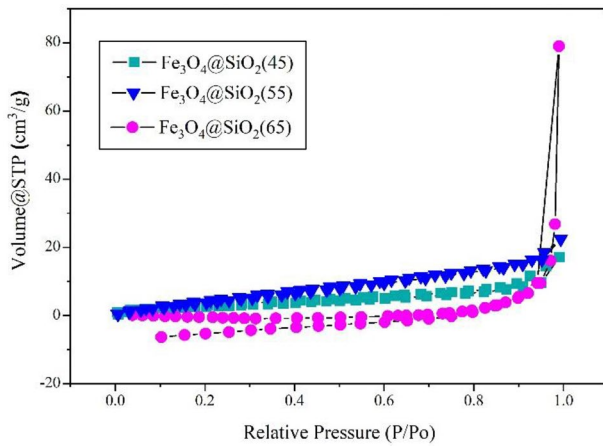


Figure 9. N_2 Adsorption-desorption of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ -core-shells ratio TEOS: (a) 45, (b) 55, and (c) 65.

Table 3. Surface area, pore volume, and pore diameter of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ -core-shell nanoparticles.

Materials	S_{BET} (m^2/g)	Pore volume (cc/g)	Pore diameter ($\text{\AA}$)
$\text{Fe}_3\text{O}_4@\text{SiO}_2(45)$	10.50	0.02	27.69
$\text{Fe}_3\text{O}_4@\text{SiO}_2(55)$	21.04	0.03	33.04
$\text{Fe}_3\text{O}_4@\text{SiO}_2(65)$	80.23		88.17

in the relative pressure range of 0.3-0.9, which indicates the mesopores formation due to agglomeration [41].

Furthermore, the agglomeration of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ with lower Si content caused the smaller specific surface area, average pore size, and volume. It can be seen that the histogram of the $\text{Fe}_3\text{O}_4@\text{SiO}_2$ composite with Si content between the two other materials has a pattern that is almost similar to 45.

The average pore diameter of $\text{Fe}_3\text{O}_4@\text{SiO}_2(45)$, $\text{Fe}_3\text{O}_4@\text{SiO}_2(55)$, and $\text{Fe}_3\text{O}_4@\text{SiO}_2(65)$ were 27.69, 33.04, and 88.17 nm, respectively. In addition, the Fe_3O_4 with a ratio of 65 of TEOS addition has a BET surface area of $80.23 \text{ m}^2/\text{g}$, which is eight times higher than the Fe_3O_4 with a ratio of 45 of TEOS addition ($10.5 \text{ m}^2/\text{g}$). Interestingly, from Figure 9, it was seen that Fe_3O_4 with a higher Si amount

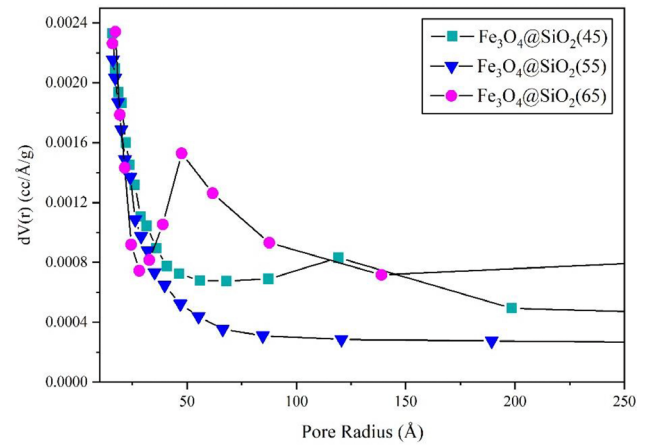


Figure 10. Pore size distribution by DFT method of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ -core-shells ratio TEOS: (a) 45, (b) 55, and (c) 65.

(ratio 65) has no hysteresis loop. This result indicates that more content of the SiO_2 layer helps reduce the agglomeration, which releases the void space among particles and reflects in higher specific surface area, average pore size, and volume.

The porosity analysis was done by simulating the adsorption curve using Density Functional Theory (DFT). The obtained porosity curve is shown in Figure 10. $\text{Fe}_3\text{O}_4@\text{SiO}_2(45)$ has a pores size of $27.691 \text{ \AA}$ (2.7691 nm), $\text{Fe}_3\text{O}_4@\text{SiO}_2(55)$ has a pores size of $33.041 \text{ \AA}$ (3.3041 nm), and $\text{Fe}_3\text{O}_4@\text{SiO}_2(65)$ is $88.171 \text{ \AA}$ (8.8171 nm). The results indicated that adding SiO_2 to the surface of the Fe_3O_4 nanoparticles would make the pores more mesoporous.

In the core-shell synthesis, $\text{Fe}_3\text{O}_4@\text{SiO}_2$ may be linked to Janus nanofluids through the formation of a unique nanoparticle structure that combines the different properties of the core (Fe_3O_4 nanoparticles) and the shell (SiO_2) [42]. The agglomeration process of core-shell particles with TEOS precursor media can be related to nanofluids, and it can be considered whether the core-shell structure influences the agglomeration of these particles and the morphology of the aggregates formed [42,43].

For further studies, the influence of aggregation morphology on thermal conductivity provides a basis for understanding how Janus nanofluid aggregates are structured, which may be influenced by core-shell synthesis. Moreover, the image analysis modeling approach on Janus nanofluids can provide a deep understanding of particle morphology. It is necessary to consider whether this modeling approach can be applied to nanofluids resulting from core-shell synthesis to describe and predict the distribution of particle morphology in more detail [42–45].

4. Conclusion

This research successfully synthesized the $\text{Fe}_3\text{O}_4@\text{SiO}_2$ core-shell from natural iron sand using the in-situ SiO_2 coating method. The silica thickness ranging from 3 to 6 nm could be controlled by changing the Tetra-Ethyl Ortho-Silicate (TEOS) mass ratio from 5.5 mL to 6.5 mL. The surface area tended to follow the same pattern as the thickness increased. Increasing the percentage of TEOS resulted in an increase in surface area and a decrease in magnetization of $\text{Fe}_3\text{O}_4@\text{SiO}_2$. The study found that adding TEOS content (SiO_2) reduced the agglomeration of Fe_3O_4 -core. This $\text{Fe}_3\text{O}_4@\text{SiO}_2$ magnetic material with higher specific surface area, average pore size, volume, and good magnetic properties could make promising materials for further application.

Nomenclature

SiO_2	Silicon dioxide (or silica)
Fe_3O_4	Iron (II) oxide or magnetite
NPs	Nanoparticles
TEOS	Tetra Ethyl Ortho-Silicate
XRD	X-Ray Diffraction
FTIR	Fourier Transform Infra-Red
M_r	Remanent magnetic field
H_c	Coercivity magnetic field
M_s	Saturation magnetic field
EDX	Energy Dispersive X-ray
SEM	Scanning Electron Microscope
TEM	Transmission Electron Microscopy
nm	Nanometer
BET	Brunauer-Emmett-Teller
S_{BET}	Specific surface area (of porous material by BET method)
VSM	Vibrating Sample Magnetometer
N_2	Gas of Nitrogen
DFT	Density Functional Theory
D	Crystal size (by Debye Scherrer formula)
β	Full width at half maximum of peak X-ray diffraction
λ	X-ray wavelength (Cu K_α)
Å	Angstrom (1 Å = 10^{-10} meter = 10 nm)

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Authors contribution statement

First author: Conceptualization; Supervision; Methodology; Writing—original draft, and Writing – review and editing.

Second author: Resources, Investigation, Investigation and Software.

Third author: Methodology, Data curation, Formal analysis, Visualization, Software and Writing - original draft.

Forth author: Writing – review and editing and did proofread the manuscript.

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