

Research Article

Fabrication of Magnetite/Silica/Titania Core-Shell Nanoparticles

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$\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles were synthesized via a sol-gel method with the aid of sonication. Fe_3O_4 nanoparticles were being encapsulated within discrete silica nanospheres, and a layer of TiO_2 shell was then coated directly onto each silica nanosphere. As-synthesized $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles showed enhanced photocatalytic properties as evidenced by the enhanced photodegradation of methylene blue under UV light irradiation.

1. Introduction

Over the past decades, titanium dioxide (TiO_2) nanoparticles have gained much attention as a photocatalyst and catalyst support [1, 2]. TiO_2 nanoparticles have many advantages as compared to other photocatalysts, which include excellent high stability against chemical and photonic corrosion and high photocatalytic activity [3]. TiO_2 nanoparticles of small mean particle sizes possess high surface area and photocatalytic activity. However, TiO_2 nanoparticles of high surface area are thermally unstable and lose their surface area readily [3]. Therefore, much effort has been focused on coating of TiO_2 on high surface area supports such as silica or alumina in order to stabilize TiO_2 nanoparticles.

TiO_2 nanoparticles could be difficult to recover and lost readily upon being dispersed into wastewater. One of the ways to overcome this problem is to coat TiO_2 onto magnetite (Fe_3O_4) cores and the resulting $\text{Fe}_3\text{O}_4/\text{TiO}_2$ core-shell nanoparticles can be recovered easily through manipulation by external magnetic field. Li et al. synthesized $\text{Fe}_3\text{O}_4/\text{TiO}_2$ nanocomposite photocatalyst using a sol-gel method [3]. However, it was difficult to achieve complete coating of Fe_3O_4 nanoparticles with TiO_2 at nanometer scale using the sol-gel method. Besides, TiO_2 would oxidize Fe_3O_4 nanoparticles and lead to a reduction of magnetic moment. Some researchers had attempted to coat a thin layer of SiO_2 between Fe_3O_4 nanoparticles and TiO_2 shell. The presence of a SiO_2 layer between TiO_2 shell and Fe_3O_4 nanoparticles

could increase the lifetime of photogenerated holes which in turn, resulted in increased photoreactivity [3, 4]. This is attributed to the SiO_2 layer which serves as an insulating layer between Fe_3O_4 nanoparticles (hole-electron trap center) and the TiO_2 shell.

However, there are currently very few literature which report on the synthesis of $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles and their photocatalytic properties. Besides, the reported synthesis methods for $\text{Fe}_3\text{O}_4/\text{SiO}_2$ nanoparticles were generally complicated and time consuming. Besides, the coating of Fe_3O_4 with SiO_2 using TEOS was a very slow process which required 12 to 48 hours of mechanical stirring at room temperature [5]. Santra et al. used the microemulsion method for the preparation of $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles which was a lengthy process and involved the use of several types of surfactants [6]. Gad-Allah et al. and Watson et al. reported the preparation of $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ nanocomposites [4, 7]. However, $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles prepared by them were in the form of patches and not discrete nanoparticles. As such, these core-shell nanoparticles exhibited in reduction on their surface area and photocatalytic properties. Song and Gao reported the use of the sol-gel process to synthesize $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ nanoparticles but the particles synthesized were very big size of about 500 nm [8].

Herein, we have reported a facile and efficient synthesis approach for the fabrication of $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ discrete core-shell nanoparticles by the sol-gel method with the aid

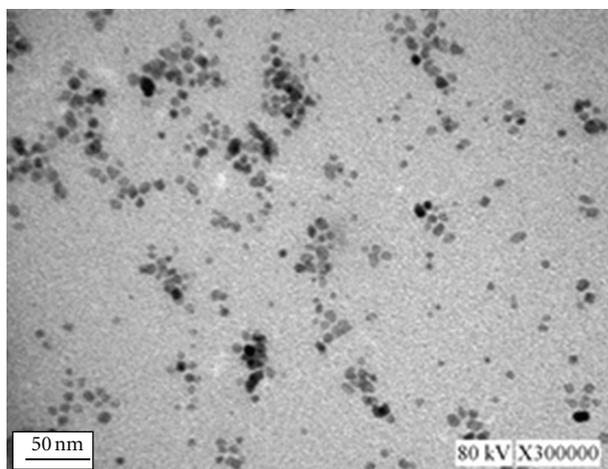


FIGURE 1: Fe_3O_4 nanoparticles were used as seeds for coating of SiO_2 and TiO_2 shells.

of sonication. Fe_3O_4 nanoparticles were being encapsulated inside discrete SiO_2 nanospheres within 90 minutes, and a TiO_2 layer was then coated directly onto each SiO_2 nanosphere via the sol-gel method. The photocatalyst properties of as-synthesized $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles were evaluated by the photodegradation of methylene blue (MB) with or without UV light irradiation.

2. Materials and Methods

2.1. Materials. Iron (II) chloride tetrahydrate, $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ (Merck); Iron (III) chloride 6-hydrate, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (AnalaR); tetraorthosilicate, TEOS (99.3%, J.T. Baker); absolute ethanol, EtOH (99.0%, HmbG Chemicals); hydrochloric acid, HCl (37%, HmbG Chemicals); ammonia solution, NH_4OH (28%, R&M Chemicals); titanium (IV) isopropoxide, TIPP (97%, Aldrich); and Milli-Q water ($18.2 \text{ M}\Omega \text{ cm}^{-1}$) were used throughout the experiment. All chemicals were used as received without further purification.

2.2. Preparation of Fe_3O_4 Nanoparticles. Fe_3O_4 nanoparticles were prepared using a simple chemical coprecipitation method [8]. Typically, 0.15 mol of $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ and 0.30 mol of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ were freshly prepared in aqueous HCl (2 M), respectively. Both $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ aqueous solution were then added rapidly to 20 mL of de-aerated Milli-Q water under nitrogen flow at 80°C with the mixture being continuously stirred under nitrogen. Upon adding an aqueous NH_4OH solution (28%, 4 mL), a distinctive black precipitate of Fe_3O_4 nanoparticles was formed immediately. Fe_3O_4 nanoparticles were isolated and purified by centrifugation and then washed with Milli-Q water three to four times to remove excess NH_4OH solution.

2.3. Preparation of Magnetite/Silica ($\text{Fe}_3\text{O}_4/\text{SiO}_2$) Core-Shell Nanoparticles. A modified Stöber method was used to coat Fe_3O_4 nanoparticles with SiO_2 shell [9]. About 30 mg of freshly prepared Fe_3O_4 nanoparticles were dispersed in a mixture of 30 mL of ethanol and 6 mL water as seeds.

The dispersion was homogenized by sonication for about 10 minutes. 3.3 mmol of TEOS was then added into the mixture and sonicated for another 20 minutes. Finally, 30 mmol of aqueous ammonia was added and the mixture was sonicated for 60 minutes. $\text{Fe}_3\text{O}_4/\text{SiO}_2$ core-shell nanoparticles were isolated by magnetically separation and then washed with ultrapure water.

2.4. Preparation of $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ Core-Shell Nanoparticles. A layer of TiO_2 shell was coated directly onto $\text{Fe}_3\text{O}_4/\text{SiO}_2$ nanoparticles via the hydrolysis and condensation of TIPP in the presence of $\text{Fe}_3\text{O}_4/\text{SiO}_2$ nanoparticles as seeds. 0.36 mL of TIPP was added into $\text{Fe}_3\text{O}_4/\text{SiO}_2$ suspension and stirred continuously for 18 hours at room temperature. The resulting $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles were dried in an oven and finally calcined at 450°C for 3 hours to convert the TiO_2 outer shell from amorphous phase to photocatalytically active crystalline anatase phase [10].

2.5. Photocatalytic Activity Evaluation. The photocatalytic activity of $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles was investigated by measuring the photodegradation rate of an aqueous solution of MB in the presence of $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles and under UV irradiation. 0.02 mmol MB solution (25 mL) and a measured amount of $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles were placed inside a glass vial. A 6 W UV tube with a wavelength of 254 nm was used as the irradiation light source. During the photocatalytic reaction, the core-shell nanoparticles were being well dispersed by stirring the suspension continuously. At predetermined intervals of UV irradiation, a subsample of the suspension was collected and analyzed by UV-Vis spectrophotometer at its characteristics absorption wavelength ($\lambda_{\text{max}} = 642 \text{ nm}$) of MB.

2.6. Morphological Characterization. The morphologies of all samples were examined using a scanning electron microscopy (SEM) (JEOL Model JSM-5300LV) and a transmission electron microscopy (TEM) (JEOL JSM-6710F). The elemental composition of the core-shell nanoparticles were analysed by SEM associated energy-dispersed X-ray microanalysis (EDX) operated with the beam energy of 20 kV. The BET surface area analysis was conducted using the nitrogen absorption-desorption method at 77.30 K (Micromeritics ASAP 2010). The phase of the $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles was identified using an X-ray diffractometer (XRD) (RIGAKU, Getgerflex D/MAX-1C).

3. Results and Discussion

3.1. Preparation of $\text{Fe}_3\text{O}_4/\text{SiO}_2$ Nanoparticles. In this study, Fe_3O_4 nanoparticles with mean diameter of approximately 10 nm (Figure 1) were prepared by a chemical coprecipitation method [11]. These Fe_3O_4 nanoparticles were subsequently used as seeds for coating of SiO_2 shell.

Fe_3O_4 nanoparticles were being encapsulated within the SiO_2 shells upon the hydrolysis and condensation of TEOS as new bonds of Fe–O–Si were formed between the interface of Fe_3O_4 and TEOS. Ultrasonication was used

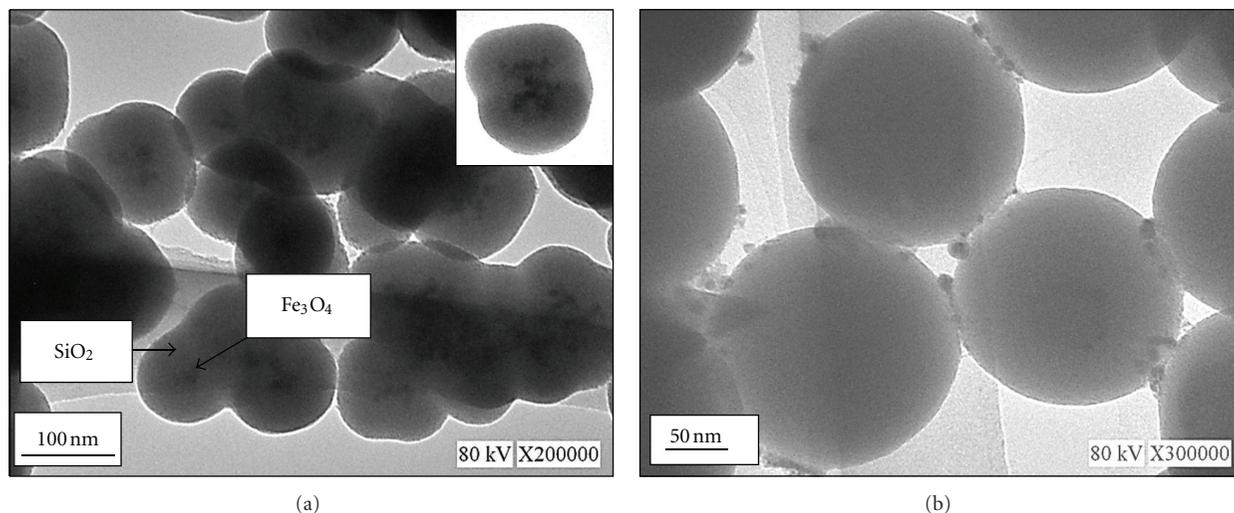


FIGURE 2: TEM micrographs of (a) $\text{Fe}_3\text{O}_4/\text{SiO}_2$ nanoparticles prepared by sonication; inset shows an individual $\text{Fe}_3\text{O}_4/\text{SiO}_2$ nanoparticles and (b) $\text{Fe}_3\text{O}_4/\text{SiO}_2$ nanoparticles prepared by the stirring method.

to accelerate the hydrolysis of TEOS. This was followed by lateral polymerization, and the formation of a three-dimensional network via siloxane formation (Si–O–Si), to produce a homogenous SiO_2 coating [4]. TEM micrographs as shown in Figure 2(a) show that Fe_3O_4 nanoparticles were fully encapsulated within the SiO_2 shell using the sonication method. The mean diameters of $\text{Fe}_3\text{O}_4/\text{SiO}_2$ core-shell nanoparticles were observed to be approximately 120 nm.

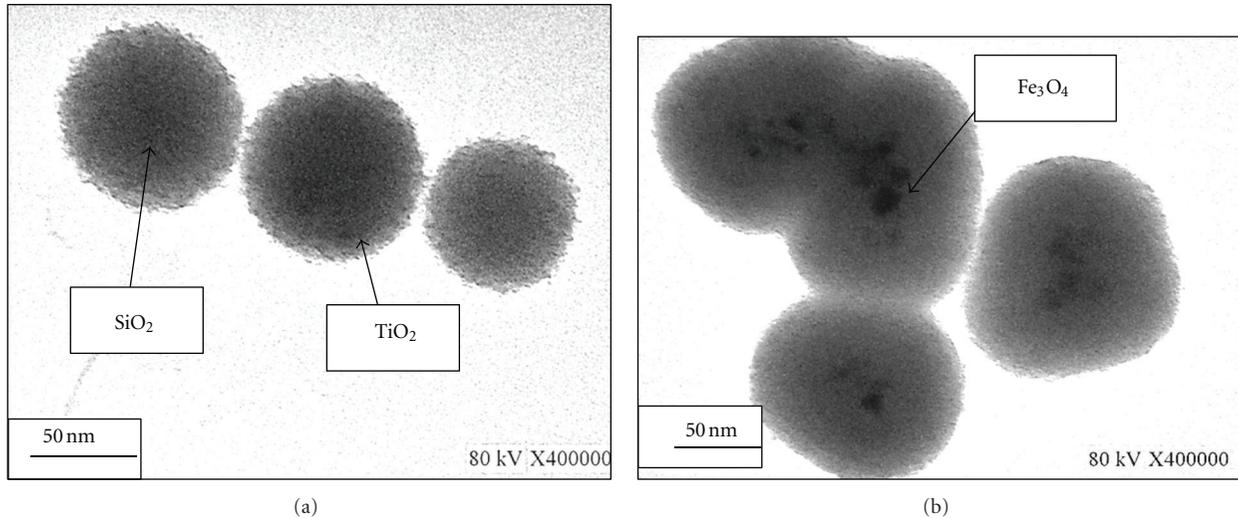
Fe_3O_4 nanoparticles were observed to have dispersed uniformly within the SiO_2 matrix. However, $\text{Fe}_3\text{O}_4/\text{SiO}_2$ core-shell nanoparticles obtained in this study were not spherical in shape after the inclusion of Fe_3O_4 nanoparticles. This could be due to the dispersing status of magnetic nanoparticles which was related to their surface charge density and in turn directed the formation of $\text{Fe}_3\text{O}_4/\text{SiO}_2$ core-shell nanoparticles of various morphologies and structures [12].

Besides the sonication method, the stirring method was also being used for coating Fe_3O_4 nanoparticles with SiO_2 shell. However, this approach had failed to encapsulate all the Fe_3O_4 nanoparticles within the SiO_2 shells, as shown in Figure 2(b). This shows that sonication is an effective way for rapid coating of Fe_3O_4 nanoparticles core with the SiO_2 shells. Besides, the high speed of coating by the sonication method had prevented the oxidation and aggregation of Fe_3O_4 nanoparticles. $\text{Fe}_3\text{O}_4/\text{SiO}_2$ core-shell nanoparticles prepared in this study were more discrete and uniform in size as compared to that reported by Morel et al. who had also used the sonication method for coating Fe_3O_4 nanoparticles with SiO_2 shell [5]. Deng et al. had reported on the effect of reaction parameters such as the types of alcohol, the volume ratio of alcohol to water, the amount of catalyst, and the amount of precursor on the formation of $\text{Fe}_3\text{O}_4/\text{SiO}_2$ nanoparticles [13]. Although they were able to prepare nanoparticles of spherical shape, their preparation method was lengthy and required 12 hours of stirring. In this

study, no surfactant was necessary during the formation of $\text{Fe}_3\text{O}_4/\text{SiO}_2$ nanoparticles. Stjern Dahl et al. reported that Triton-100 was used in the emulsion method for preparing $\text{Fe}_3\text{O}_4/\text{SiO}_2$ core-shell nanoparticles [14]. Kobayashi et al. also reported on the need to modify the surfaces of Fe_3O_4 nanoparticles with silane coupling agent before the preparation of $\text{Fe}_3\text{O}_4/\text{SiO}_2$ nanoparticles [15].

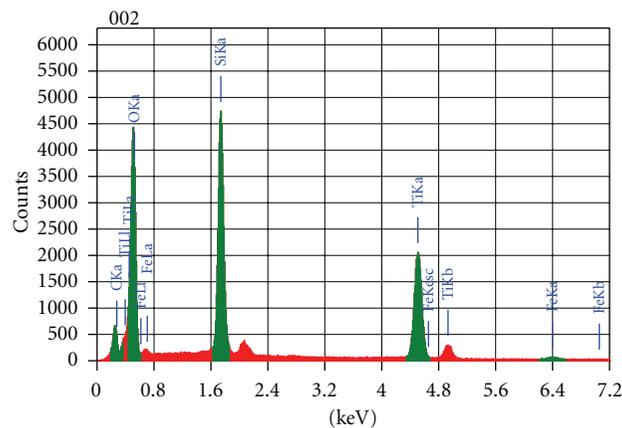
3.2. Preparation of $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ Core-Shell Nanoparticles. TiO_2 was deposited on SiO_2 nanoparticles by the hydrolysis of TIPP precursor. Figure 3(a) shows the TEM micrograph of $\text{SiO}_2/\text{TiO}_2$ nanoparticles without inclusion of Fe_3O_4 nanoparticles. All of these $\text{SiO}_2/\text{TiO}_2$ nanoparticles were spherical in shape with rough surfaces. The direct coating of TiO_2 onto surfaces of $\text{Fe}_3\text{O}_4/\text{SiO}_2$ nanoparticles resulted in the formation of core-shell type structures with Fe_3O_4 nanoparticles being the cores and SiO_2 and TiO_2 are the shells (Figure 3(b)). The EDX spectrum as shown in Figure 3(c) revealed the presence of four types of elements (Si, Fe, O, and Ti). This suggested that TiO_2 was being coated onto the surfaces of $\text{Fe}_3\text{O}_4/\text{SiO}_2$ nanoparticles. On the basis of the above analysis and observations from TEM images, we could conclude that a thin layer of TiO_2 layer of approximately 20–30 nm in thickness had been coated onto the surfaces of $\text{Fe}_3\text{O}_4/\text{SiO}_2$ nanoparticles (Figure 3(b)). The overall mean diameters of $\text{Fe}_2\text{O}_3/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles were approximately 140 nm.

Figure 4 shows the XRD pattern of $\text{Fe}_2\text{O}_3/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles after calcination at 450°C in air. The broad peaks were characteristic of the SiO_2 matrix [15]. The XRD patterns also demonstrated that the apparently amorphous nature of TiO_2 coated on the surface of $\text{Fe}_2\text{O}_3/\text{SiO}_2$ nanoparticles. In this case, the anatase phase of TiO_2 could have been formed after heat treatment at 450°C for 3 hours [3, 8]. The BET specific surface area of $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles prepared in this study was $138\text{ m}^2/\text{g}$, and this value was substantially



(a)

(b)



(c)

FIGURE 3: TEM micrograph of (a) $\text{SiO}_2/\text{TiO}_2$ nanoparticles, (b) $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ nanoparticles, and (c) EDX spectra of $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles.

higher than that of $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles reported by Gad-Allah et al. at $21\text{--}54\text{ m}^2/\text{g}$ [4].

Figure 5 presents a photograph of the $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ aqueous dispersion before and after a magnet was being attached to the outside of the sample vial. $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles were observed to be attracted to the magnet being attached outside of the sample vial. The result indicated that even after coating of both SiO_2 and TiO_2 layers unto Fe_3O_4 nanoparticles, their magnetic property had remained intact. As such, these $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles could be easily recovered after their application in the aqueous medium.

3.3. Photocatalytic Properties of $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ Core-Shell Nanoparticles. Figure 6 shows the degradation of MB dye with and without $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles added at various duration with and without UV irradiation. It was observed that without UV irradiation, the

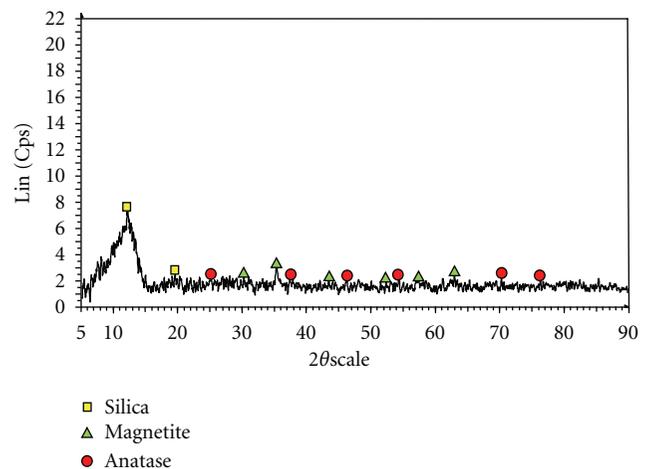


FIGURE 4: XRD pattern of $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles after being calcined at 450°C in air.

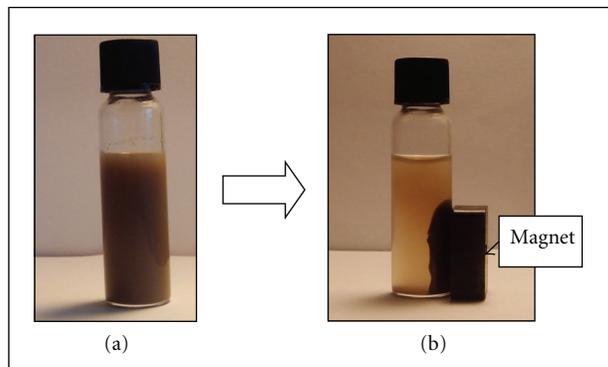


FIGURE 5: Photographs of a vial containing (a) $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell aqueous dispersion and (b) when a magnet was attached to the outside of the sample vial.

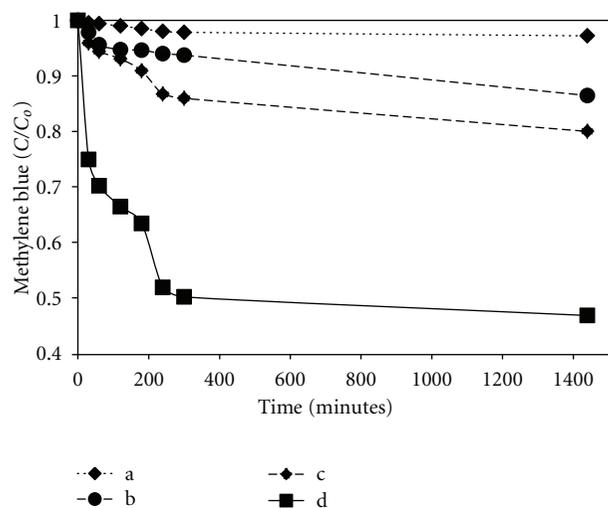


FIGURE 6: Changes in concentration of MB dye (a) alone, no UV irradiation, (b) alone, UV irradiation, (c) with $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles, no UV Irradiation, and (d) with $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles, UV irradiation.

concentration of MB dye alone remained almost constant after 24 hours. However, its concentration decreased by about 20% with UV irradiation. The degradation of MB dye was substantially enhanced by the addition of $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles with its concentration being degraded by up to 70% with UV irradiation (Figure 6(d)). We can therefore conclude that the degradation of MB dye was attributed to the photocatalytic activities of the TiO_2 layer on the core-shell nanoparticles.

The photocatalytic decomposition of MB dye catalyzed by $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles was further evidenced in Figure 7, which shows UV spectra of photocatalytic decomposition of MB dye with and without addition of $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles, as well as with and without UV irradiation. The intensity of absorption at around 650 nm (λ_{max}) was observed to decrease gradually over the one-hour duration of UV irradiation in the presence

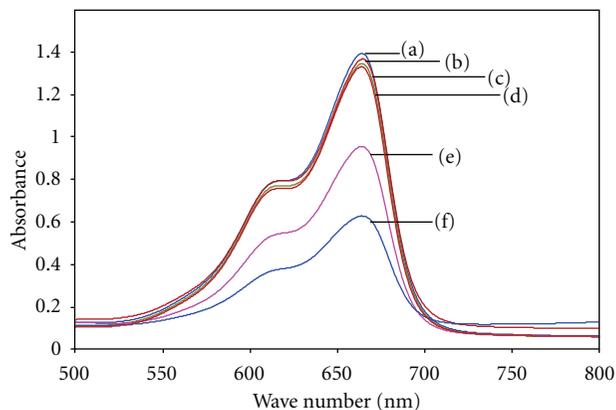


FIGURE 7: Absorption of MB dye solution (10^{-5} M, 20 mL), (a) alone, no UV irradiation, (b) alone, UV irradiation, (c) with $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles (5 mg) added, no UV irradiation, (30 mg) (d) $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles (30 mg) added, no UV irradiation, (e) with $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles (5 mg) added, UV irradiation, and (f) with $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles (30 mg) added, UV irradiation.

of $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles. The intensity of absorption of MB dye had remained the same without UV irradiation.

4. Conclusion

In this study, a simple and facile synthesis approach was developed for the preparation of a magnetically separable photocatalyst consisting of an Fe_3O_4 core, an SiO_2 intermediate layer, and a photocatalytically active TiO_2 shell. This synthesis method was rapid and did not require the addition of any surfactant to direct the formation of SiO_2 or TiO_2 shells. The photocatalytic activity of TiO_2 surface shell was not affected by the intermediate SiO_2 layer and Fe_3O_4 core. The $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ core-shell nanoparticles possessed high specific surface area of $138 \text{ m}^2/\text{g}$ and exhibited a good photocatalytic activity for the photodegradation of MB dye in aqueous solution.

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