

Research Article

Anatase TiO₂ Nanospindle/Activated Carbon (AC) Composite Photocatalysts with Enhanced Activity in Removal of Organic Contaminant

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This paper embarks upon the three levels of analysis ranging from nanoscale materials synthesis to combination and functionality. Firstly, we have prepared anatase TiO₂ nanospindles with an even length of about 200 nm and a central width of about 25 nm by hydrothermal synthesis method at 100°C for 6 h. Secondly, we have dispersed TiO₂ nanospindles on the surface of activated carbon (AC) and fabricated TiO₂/AC composite *via* a dip-coating method. Thirdly, the TiO₂/AC composite has been studied as the photocatalyst to remove the organic contaminants in the waste water and exhibits excellent degradation rate in comparison with pure anatase TiO₂ nanospindles.

1. Introduction

Up to date, titanium dioxide (TiO₂) has attracted much attention as heterogeneous photocatalyst and has been widely applied in the environment science owing to its significant advantages such as nontoxicity, low cost, chemical stability, and superior photoactivity over other semiconductors that have been investigated [1–5]. People have fabricated a variety of geometric structures of TiO₂ nanoparticles that comprises zero dimensional (0D) structure such as spheric nanoparticle [6, 7] and one-dimensional (1D) structure as nanowire [8, 9], nanorod [10], nanobelt [11, 12], or nanotubes [13, 14]. The later is particularly useful in dealing with waste water owing to the large surface to volume ratio which further improves the photocatalytic activity comparing to spheric particles under the irradiation of visible light. So far, numerous efforts have been developed to the fabrication of TiO₂ nanoscale materials with special morphologies by some traditional methods such as, sol-gel, micelle, and hydrothermal or solvothermal methods [15]. However, few studies reported the synthesis of anatase TiO₂ nanospindles by these methods [16, 17]. Qiu et al. [18] had fabricated anatase TiO₂ nanospindles by a hydrothermal treatment method and found they provided a good solution to the problems

of poor electron transport and severe aggregation of TiO₂ nanoparticles.

In general, one main drawback of the TiO₂ nanostructures, when used in the practical application, comes from their easy loss during the process of water treatment, resulting in low utilization rate and high cost, which limits their widespread use. Some attempts have been employed to improve the reuse efficiency of TiO₂, for example, the immobilization of TiO₂ nanoparticles onto some supports such as carbon nanotube [19, 20], glass [21], ceramic [22], and activated carbon [23–27]. However, the current immobilization techniques are still not stable enough to improve the reaction efficiency for the reducing of TiO₂ dispersion and introducing transfer limit of the mass. It has also been observed that the lixiviation of TiO₂ particles from the supports occurs after running a period. However, among these supports, the activated carbon (AC) particles were chosen broadly as the support to increase remarkably the photoactivity of TiO₂ because of the high adsorption capability of the AC [25, 28–30], which was expected to solve above-mentioned problem.

In the present work, we are planning to report a novel TiO₂/AC composite photocatalyst by the coating of anatase TiO₂ nanospindles onto the surface of columniform AC particles, which showed a promising photocatalytic activity than

pure TiO₂ due to the synergistic effect between supporting materials and TiO₂ nanospindles. Most importantly, this composite photocatalysts effectively reduced the lixiviation of TiO₂ from the surface of AC.

2. Experimental Procedure

2.1. Preparation of Anatase TiO₂ Nanospindles. Firstly, TiOSO₄ powders (93 wt%, Liaoning Dandong Chemical Company) with a weight of 3.00 g were dissolved into 350 mL deionized water by a vigorous stirring for 0.5 h, and then aqueous solution of NH₃·H₂O (28%, Nanjing Chemical Company) with a concentration of 10 wt% was added dropwise into the above solution to adjust the pH value to 6.5. After a continuous stirring of 0.5 h, the white precipitation was obtained by a centrifugal separation. Secondly, the obtained precipitation was mixed with 250 mL deionized water with a vigorous stirring again. After that, a mixture solution involving 2 g of sodium oxalate and 150 mL deionized water was added slowly into the above solution. After a vigorous stirring for 0.5 h, the precipitation was separated by a centrifuge. Finally, the mixture including 4 g of H₂O₂ (30%, Guangdong Zhongcheng Chemical Ltd.) and 250 mL deionized water was used as the react reagent, which was reacted with the obtained products from step 2 for 12 h until a brown transparent solution was produced finally, and then it was kept heating at 100°C for 6 h. The large scale of TiO₂ nanospindles was formed and uniformly distributed in the water.

2.2. Preparation of TiO₂/AC Composite Photocatalyst. In a typical procedure, 1 g of commercial granular AC particles with an average diameter of 4 mm (Guangzhou Wuhuan Activated Carbon Company, China) was suspended in the above TiO₂ suspension by continuous slow stirring for 1 h and then kept at room temperature for 10 h. Thereafter, the AC granular particles with the TiO₂ coating were obtained after a simple vacuum filtration process and then dried at 70°C for 12 h. Finally, the prepared *x*TiO₂/AC composite photocatalysts with various content of TiO₂ were formed by repeating above experimental procedure, where *x* is 0, 0.5, 1, 1.5, and 2, representing the wt% of TiO₂.

2.3. Characterization. The obtained TiO₂ nanospindles were characterized by XRD on a D/MAX-RC diffractometer operated at 30 kV and 100 mA with CuK_α radiation. The anatase crystallite size was obtained from the line broadening of the 200 diffraction peak and calculated by the Scherrer equation, $D = K\lambda/\beta \cos \theta$, where θ is a shape factor ($K = 0.9$ in this work), λ is the wavelength of the incident X-rays, and β is the broadening of the peak at half the maximum height (full width at half-maximum) measured in radians. The surface morphology and particle size of TiO₂ nanospindles were examined by means of transmission electronic microscopy (TEM) on an FEI-XL30 transmission. The morphology of the TiO₂/AC composite was observed by a scanning electronic microscope (SEM, Hitachi S-2300) equipped with an energy dispersive spectroscopy using an accelerating voltage

of 200 kV. To obtain the cross section of the samples, we broke the sample after being frozen in liquid nitrogen for 10 minutes. FTIR measurement of the sample was performed on an Avatar FTIR equipment. The contact angle between water and the external surface of AC was measured to evaluate the hydrophilicity property using a contact angle meter (Motic Images plus 2.0). The specific surface area of the samples obtained was measured in a Gemini VII 2390 Series Surface Area Analyzer (from Micromeritics) with the BET method.

2.4. Evaluation of Photocatalytic Activity. The photocatalytic degradation experiments were carried out using a cylindrical batch reactor opened at the air with 250 mL of capacity. A 175 W 365 nm commercial halogen lamp was used as the visible irradiation source. The total UV intensity was 2.1–2.2 mW/cm² by turning on all the UV light. The UV emission of the lamp was filtered to close to sunlight while avoiding its variation with time and cloudiness [25]. Methylene blue (MB) purchased from Aldrich was chosen as a model molecule for the photocatalytic test. Each catalyst of 50 mg/L was suspended in MB aqueous solution (0.05 mol·L) followed by sonication for 15 minutes. Before the irradiation, the suspension was maintained in the dark for 1 h to reach complete equilibrium adsorption. After being irradiated under the visible light, 2 mL of suspension was withdrawn periodically and immediately centrifuged at 5000 rpm for five times, and its absorbance at 664 nm was measured by UV-Vis spectroscopy (1501, Shimadzu).

3. Results and Discussion

3.1. Morphology and Structure of TiO₂ Nanospindles. In general, TiO₂ nanospindle was prepared by hydrothermal treatment of Ti source precursor with NaOH solution or water/alcohol mixture at a higher temperature (160°C) [24]. However, herein we fabricated successfully the TiO₂ nanospindle particles after a desirable heating treatment at 100°C. TEM images (Figures 1(a) and 1(b)) clearly indicated the unique morphologies of the synthesized TiO₂ nanospindles, which have an even length of about 200 nm and a central width of about 25 nm. XRD pattern in Figure 1(c) shows clearly that the TiO₂ nanospindle particles are anatase crystal structure (JCPDS card no. 21-1272), and their crystal size of TiO₂ is about 6.5 nm, which is calculated according to the Sherrer equation. This revealed that anatase TiO₂ nanospindles were directly produced after the continuous heating treatment processing. It is suggested that the heating temperature of the solution at 100°C is feasible enough for the nucleation of anatase from the amorphous crystal. In general, to obtain anatase TiO₂ nanospindle prepared by hydrothermal method, the temperature is higher than 160°C [28]. Therefore, it is more practical to fabricate large-scale anatase TiO₂ nanospindles in the current work for the easily synthetic process and lower cost. FTIR spectra of nanospindle of TiO₂ are shown in Figure 1(d). The main absorption peaks were located at 3420.7, 1622.3, and 520.1 cm⁻¹. The band at 3420.7 cm⁻¹ was assigned to –OH stretching of surface hydroxyl group and at 1622.3 cm⁻¹ to –OH vibration of

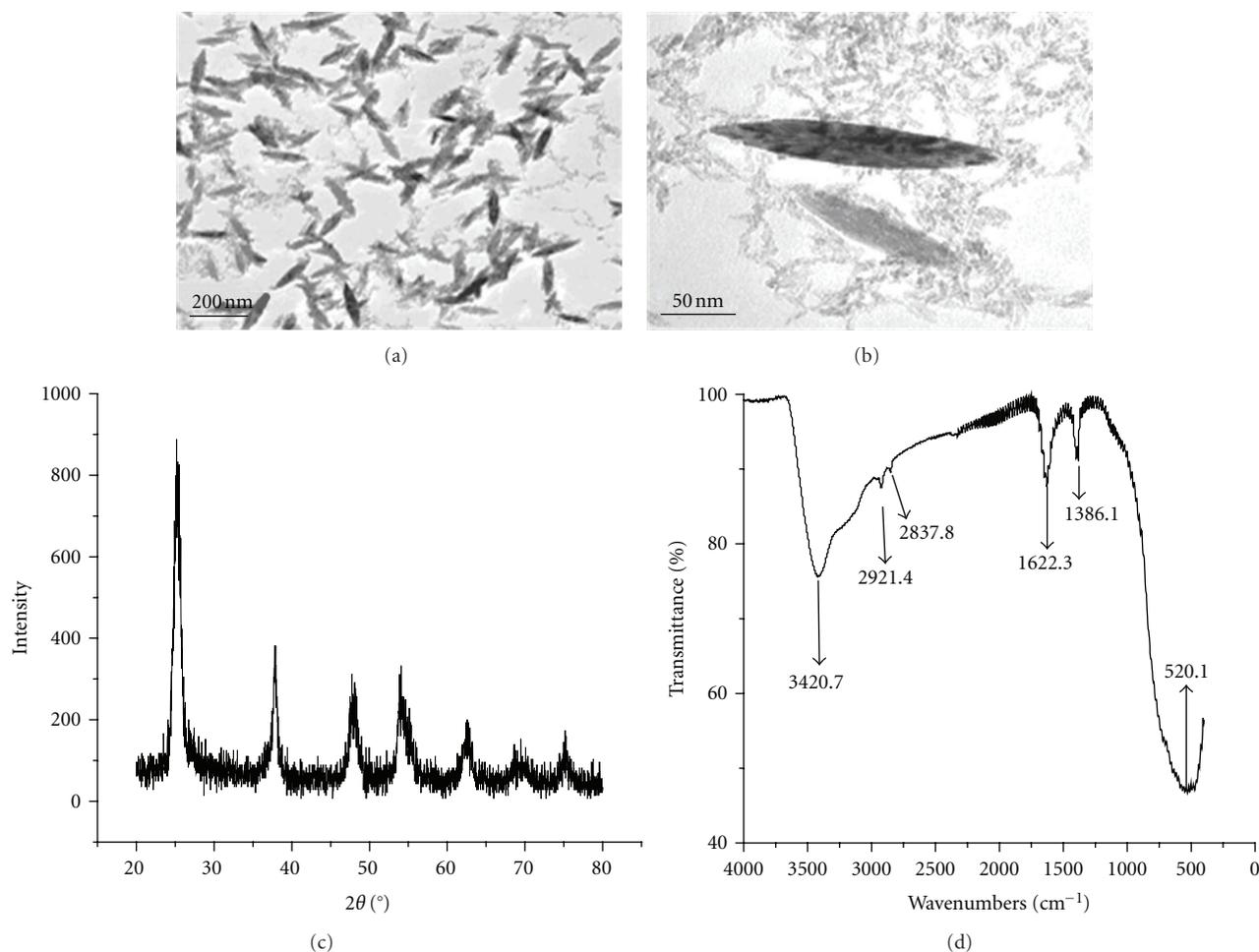


FIGURE 1: TEM images of the as-prepared large TiO_2 nanospindles (a, b). XRD diffraction of as-prepared TiO_2 nanospindles (c). FT-IR spectrum of as-prepared TiO_2 nanospindles (d).

physically adsorbed water. The band at 520.1 cm^{-1} was ascribed to the Ti–O stretching vibration [31]. Weak bands at 2837.8 to 2921.4 cm^{-1} can be assigned to the O–H stretching of H_2O_2 at the TiO_2 surface.

3.2. Physical Properties of Nanospindles TiO_2/AC Composite Photocatalyst. The surface morphologies of TiO_2/AC composite catalysts were characterized by SEM with the result shown in Figure 2. It is clearly found in Figure 2(a) that the pure AC without TiO_2 coating has a very rough surface structure with plenty of small pores. The average size of the pores was as large as approximately $3.5\text{ }\mu\text{m}$, which has enough space to accommodate TiO_2 nanoparticles. With the increasing content of TiO_2 coating (see Figures 2(b) and 2(c)), the surface of AC became more and more smooth and expressly the sample with 1 wt% TiO_2 coating indicated a flattest surface structure. This indicated that this concentration of TiO_2 nanospindles coating on the surface of AC produced an optimal layer. Most of TiO_2 nanospindles were entirely filled into the pores of AC, resulting in the formation of glossy surfaces. However, it was discovered that there were still a few pores on the surface which was

able to absorb some organic compounds. Further increase of TiO_2 coating such as 2 wt% TiO_2 content, most of TiO_2 nanospindles were scattered on the top layer of AC resulting in the formation of incompact and rougher external structure on the surface. The result of increasing size of TiO_2 was from the interaction between TiO_2 and AC. It could be found in Figure 2(e) that plenty of TiO_2 nanospindles laid on the surface of AC and produced a tight surface morphology as the further increase of TiO_2 . Moreover, the BET surface area of 1.5 wt% TiO_2/AC , 2.0 wt% TiO_2/AC composite, and pure AC particles were 299.22 , 250.01 and $372.02\text{ m}^2\text{ g}^{-1}$, respectively. The pore volumes of them were 0.15 , 0.10 , and $0.20\text{ cm}^3/\text{g}$, respectively, which indicated that the surface area of TiO_2/AC was less than that of pure AC due to that the coated TiO_2 particles occupied the pores of granular AC. Moreover, with the increasing of TiO_2 nanospindles, the surface area and pore volume of the catalysts decreased gradually. This suggested that most of TiO_2 nanospindles were entirely filled into the AC pore as with the increasing content of TiO_2 coating and decreased the surface area. The EDS analysis confirmed that the Ti elements with a concentration of 19.74% were existed on the surface of AC,

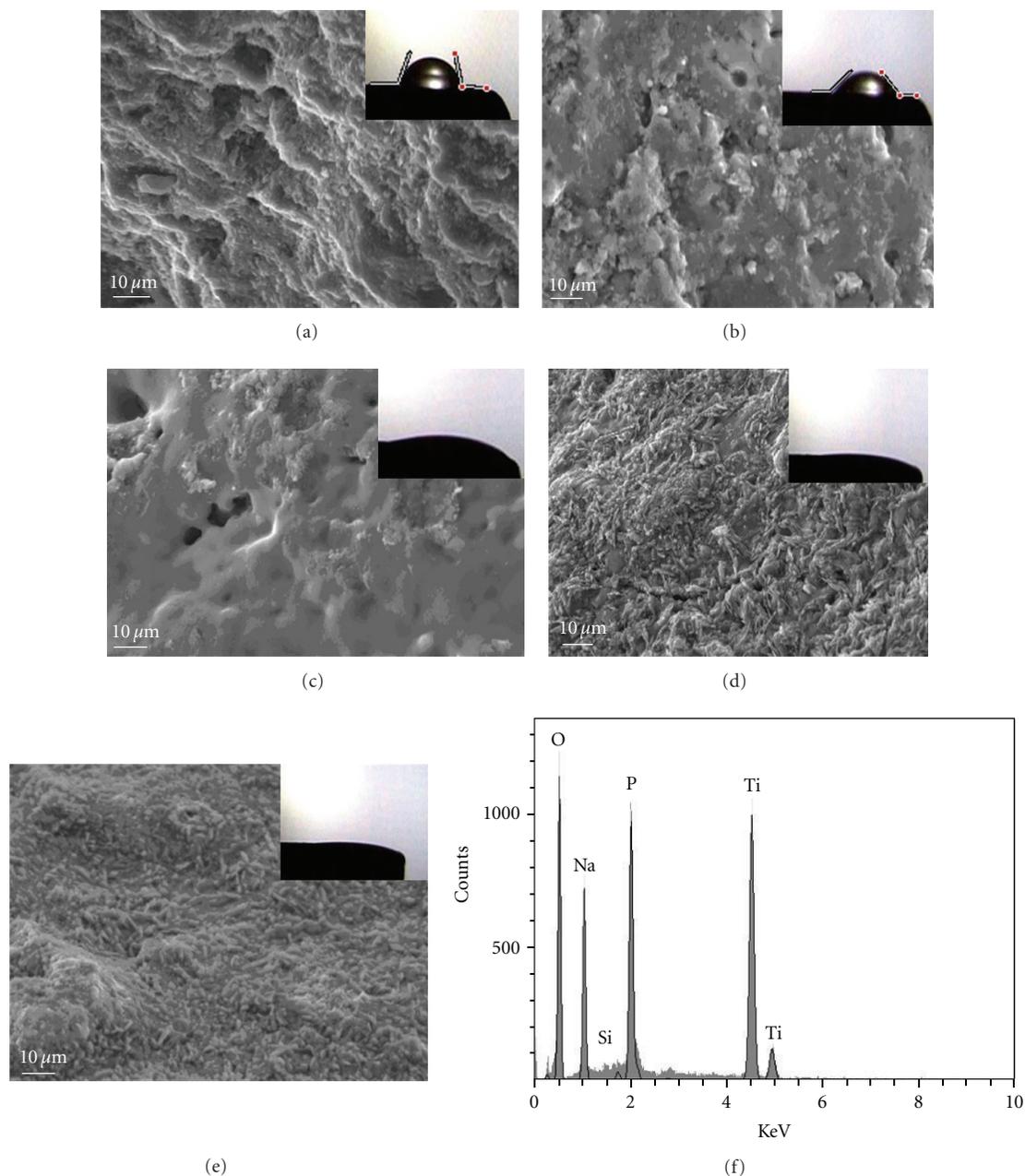


FIGURE 2: SEM image of AC without coating (a), with 0.5 wt% TiO₂ coating (b), with 1 wt% TiO₂ coating (c), with 1.5 wt% TiO₂ coating (d), with 2 wt% TiO₂ coating (e), and EDS analysis of the sample with 1.5 wt% TiO₂ coating (f).

but carbon element is almost not detected. This result further revealed that the top layer on the surface of AC has been occupied completely by TiO₂. The contact angle data of TiO₂/AC composite catalyst is shown in the inset image of Figure 2. The naked AC showed a high water contact angle of $115 \pm 2.5^\circ$, which indicated that the pure AC had a superior hydrophobicity. However, the contact angle of the surface of the TiO₂/AC composite catalyst was enormously decreased compared to the naked AC. It was almost tiled when the water droplet was contacted with the surface of the sample with 1.5 wt% of TiO₂ coating, suggesting that the TiO₂ nanoparticles changed significantly the polarity

of AC surface from hydrophobicity to hydrophilicity. The images in Figure 3 exhibit the cross-sectional structures of TiO₂/AC composites photocatalysts. It was clearly observed that the sample with 1.5 wt% of TiO₂ was covered by a thin layer of TiO₂ coating with the thickness of about 5 μm when compared to the naked AC.

The surface hydrophilicity of activated carbon plays an essential role in degradation process of organic compounds. In general, the higher hydrophilicity of surface is able to induce stronger capability in absorbing more aqueous organic molecules. This will be beneficial for the degradation process of organic contaminants.

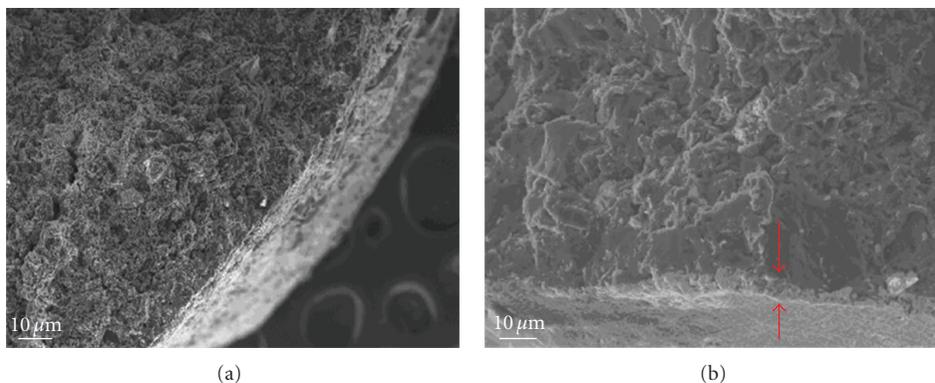


FIGURE 3: SEM image of cross-sectional area of the samples ((a), naked AC; (b), 1.5wt% TiO₂/AC).

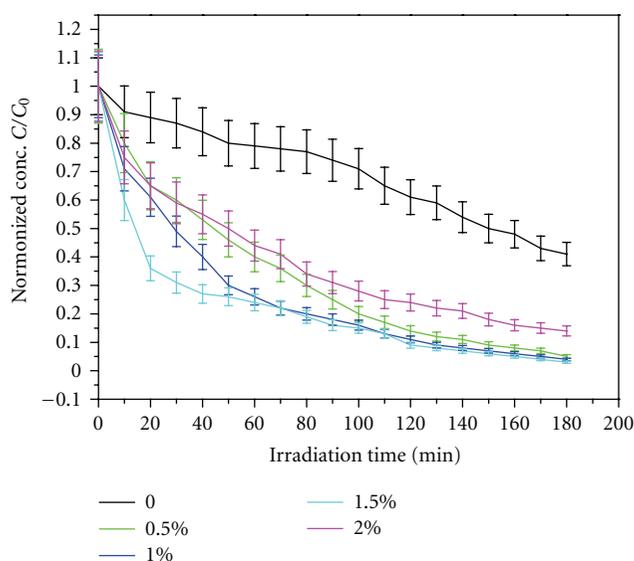


FIGURE 4: Photocatalytic degradation of methylene blue over TiO₂ and x TiO₂/AC ([MB] = 0.05 mol L⁻¹; reaction time t = 180 min).

3.3. Photocatalytic Activity. To assess the photocatalytic activity of the TiO₂/AC samples, methylene blue (MB) was used as a model organic substrate in the suspended TiO₂/AC reaction system under visible light irradiation as probe reaction. As we know, the MB (initial absorbance 0.8–1.0) owns two special absorption peaks separately located at 290 nm and 665 nm. The results of degradation experiment displayed in Figure 4 show that the MB solution with 1.5% TiO₂/AC catalysts changed colour from blueness to almost colorlessness within 180 minutes of irradiation. The UV-Vis absorption spectra curves of MB studied at different times of irradiation are detailedly showed in Figure 5, where the photocatalytic degradation rate of MB on naked TiO₂ in this work was obviously lower than x TiO₂/AC composite. However, for these composite catalysts with various content of TiO₂, they showed the sharp reduce of degradation rate in the initial 30 minutes which mainly resulted from the adsorption of AC. However, after 30 minutes of irradiation, these samples displayed gradual tendency of decreasing rate

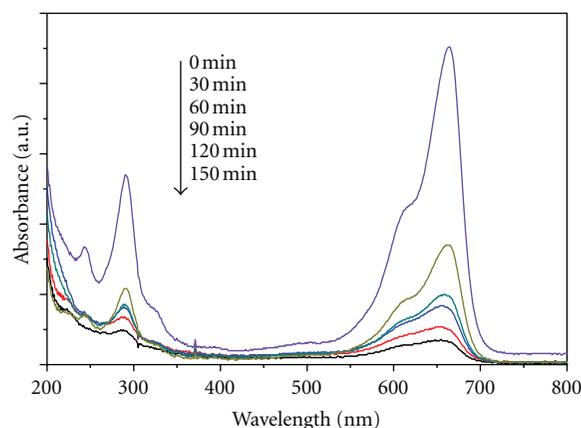


FIGURE 5: UV spectra of 1.5 wt% TiO₂/AC sample suspended in methylene blue solution ([MB] = 0.05 mol L⁻¹).

which mainly caused by the photocatalytic degradation of TiO₂ catalysts. It is widely accepted that AC is able to adsorb a wide range of synthetic organic compounds due to its high porosity. Therefore, the harmful organic compounds in the waster water will be moved easily to the surface of the catalysts. The TiO₂ nanospindle coating on the surface of AC indicated excellent capability in photocatalytic degrading organic compounds. These TiO₂ nanospindles are capable of prolonging the separation lifetime of photogenerated e⁻/h⁺, resulting in the increasing rate of •OH radical generation by the photocatalyst. Therefore, the synergistic effect between AC and TiO₂ nanospindles indicates greater degradation rate than pure TiO₂ nanospindles. Considering the immobilization of TiO₂ onto support, it is necessary to characterize the reuse efficiency of these composite catalysts. The results of the repetitive test on this sample with 1.5 wt% TiO₂ showed that degradation rate on MB is still greater than 90% after the reuse of 15 times.

4. Summary

In this work, anatase TiO₂ nanospindles with an even length of about 200 nm and a central width of about 25 nm were

fabricated by a simple hydrothermal treatment method. This method is more available to produce large-scale anatase TiO₂ nanospindles for the easy process and lower cost. We also prepared TiO₂/AC composite catalysts by the dip coating technique, where all the samples with TiO₂ coating indicated much better photocatalytic activity than naked AC. The photocatalytic activity experiments discovered that the sample with 1.5% TiO₂ coating exhibited the highest photocatalytic activity for the synergistic effect between AC and TiO₂ nanospindles.

Acknowledgments

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