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ABSTRACT

This paper presents the wet chemical synthesis of WO$_3$/TiO$_2$ nanocomposites using hydrothermally prepared monoclinic WO$_3$ and anatase TiO$_2$ nanoparticles as composite matrices and filler, respectively. The nanocomposites were prepared in different compositions, i.e. WO$_3$:TiO$_2$ ratio (w/w) of (1:1), (1:3), and (3:1). Physicochemical properties of the resultant WO$_3$/TiO$_2$ nanocomposites were evaluated by X-ray diffraction (XRD), scanning electron microscopy (SEM), BET N$_2$ adsorption-desorption isotherms, UV/vis, and Raman spectroscopy. The resultant nanocomposites exhibited a mesoporous structure with specific surface area of up to 11.5 m$^2$g$^{-1}$, where TiO$_2$ constituent contributed to higher surface area, especially in (1:3) ratio, and higher number of defect sites. While there was no impurities in the investigated nanocomposites as revealed by XRD analysis and Raman spectra, the WO$_3$:TiO$_2$ (1:3) nanocomposites showed the highest light harvesting ability indicated by higher absorption amplitude in both UV and visible regime. Three different kinetic models including pseudo-first-order, pseudo-second order, and Langmuir-Hinshelwood were applied to experimental data. The pseudo-second-order was found to be the best representing model and the adsorption kinetic studies indicated that chemisorption initially dominated the degradation mechanism. Finally, photocatalytic activity of nanocomposites was compared for photodegradation of methylene blue (MB) aqueous medium and in a good agreement with physicochemical properties, WO$_3$:TiO$_2$ (1:3) nanocomposites yielded the highest MB degradation rate ($k$ ~ 0.162 min$^{-1}$) and efficiency (96%) within 120 min under UV irradiation.

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1. INTRODUCTION

A constantly increasing use of synthetic dyes in various industries, such as textiles, rubber, plastics, and food, requires discharging the colored wastewater into the environment [1-3]. Amongst various commercial synthetic dyes, methylene blue (MB) is widely utilized. Long-term exposure to MB can cause formation of Heinz bodies, and tissue necrosis in humans [3]. Therefore, removal of MB from wastewater is very important to reduce the dye hazards to the biotic environment. Currently, there are numerous methods for degradation of synthetic dyes from wastewaters, including physico-chemical and biological techniques [1-3]. However, photocatalytic degradation of MB using semiconductor catalyst has gained a great interest since its catalytic efficiency can be tuned by controlling the microstructural properties of semiconductor [4-7]. Among various n-type semiconductors, TiO$_2$ is widely employed due to its versatile optical properties, low cost, stability against photocorrosion, and large surface area [6, 7]. While the conduction band (CB) and valence band (VB) positions of TiO$_2$ are suitable to drive charge transfer for photooxidation of organic dyes, the large band gap of ~ 3.3 eV ($\lambda_{exc}$ ~ 375 nm) limits its functionality which requires UV irradiation to create exciton.
Another appealing n-type semiconductor is WO₃ which has also been extensively studied for catalysis [8]. Bearing a smaller optical band gap of ~ 2.6 eV (λ_exc ~ 475 nm), WO₃ enables higher light harvesting in visible regime than TiO₂ [8-10]. In spite of photocorrosion issue which leads to the formation of W³⁺ and decreases its catalytic activity, the band position is also favorable for oxidation reactions on the surface [11, 12]. Furthermore, WO₃ surface is characterized by highly negative surface charges and hence, it is advantageous for the adsorption of the cationic dye (MB). To combine the virtue of both TiO₂ and WO₃, recent studies explored WO₃/TiO₂ nanocomposites which exhibit desirable physicochemical properties for the enhanced photocatalytic activity. In addition, the nanocomposites offer other advantages [9-12]. Nanocomposites enable photocatalytic process under visible solar spectrum. The band position of WO₃ is suitable to allow excited state charge transfer to TiO₂ for efficient redox process [13,14]. Moreover, the nanocomposites reduce the synthesis cost as the price of composite material is more affordable than pure WO₃ [12]. Recent studies have reported the synthesis of WO₃/TiO₂ nanocomposites using various methods, such as sol-gel, solvothermal, hydrothermal, and alcoholothermal [10-15]. The morphology of the resulting nanocomposites also differs from unstructured porous network, spherical aggregates and hollow spheres, yielding in a unique physical to microstructural properties with promising catalytic activities [13-15]. However, it should be noted that the above mentioned method is considered time consuming and requires long synthesis routes. In the present work, a facile and low cost approach to synthesize WO₃/TiO₂ nanocomposites by wet chemical synthesis at mild temperature is proposed. The morphology, microstructure, and optical properties of WO₃/TiO₂ nanocomposites with different compositions, i.e. ratio between WO₃ and TiO₂, are characterized and correlated with the photocatalytic activities for the MB degradation. Finally, the mechanism of photocatalytic degradation of MB using the nanocomposites is proposed.

2. EXPERIMENTALS

2. 1. Chemicals, Materials, and Preparation of WO₃/TiO₂ Nanocomposite The chemicals and materials, i.e. Na₂WO₄, 2H₂O (Xiqiao Chemical), CTABr (Himedia, 99%), TiO₂ (MTI, 99%), methanol (96%, UPT, BPPTK LIP1), hydrochloric acid (HCl, smart-lab), and aquadest, were used as received. Hydrothermal synthesis of WO₃ nanoparticle was carried out following the protocol developed in the literature [16] with minor modification, i.e. Na₂WO₄:2H₂O/CTABr molar ratio of 4:1 was used. The WO₃/TiO₂ nanocomposite was prepared using sol-gel method: WO₃ to TiO₂ weight ratio was set to (1:1), (1:3), and (3:1). An amount of TiO₂ was dissolved in 100 mL of aquades and sonicated for 10 min. The TiO₂ solution was then stirred (300 rpm) at 30 °C for 15 min. The powder of WO₃ nanoparticles was added into the TiO₂ solution and stirred for 15 min. The mixture solution was evaporated and dried at 80 °C for 12 h. Eventually, the dried powder was ground resulting in fine powder of WO₃/TiO₂ nanocomposite.

2. 2. Physicochemical Characterization X-ray diffraction (XRD) patterns were recorded using a PAN analytical type X’Pert Pro diﬀractometer with Cu-Kα as the radiation source operated at 40 kV and 40 mA. Diffraction patterns were scanned at angle (2θ) between 10 and 100° with 0.05° resolution. Crystal size was estimated by Scherrer equation. SEM images of WO₃/TiO₂ powder were measured by a scanning electron microscope (SEM, FEI type Inspect 21) operated at 100 kV accelerating voltage. BET N₂ adsorption/desorption isotherms were measured at 77 K (NOVA Touch 4LX). Prior to surface area analysis, the powdered samples were dried at 200 °C in N₂ for 3 h. Raman spectra were collected upon excitation at 785 nm (25 mW) using TEC-HR Raman Spectrometer (Stellarnet Inc) coupled with 180° degree optical probe [17]. Absorption spectra were measured using UV/vis spectrometer (Ocean optics USB4000).

2. 3. Photocatalytic Degradation of Methylene Blue Photocatalytic activity of WO₃/TiO₂ nanocomposites against organic pollutant, i.e. methylene blue (MB), was assessed. Initial test of MB photodegradation was carried out employing WO₃ and TiO₂ nanoparticles and different WO₃/TiO₂ nanocomposites. The photocatalyst was soaked in the aqueous MB solution and then transferred into a custom-built photoreactor and irradiated under UV light for several times, i.e. every 15 min for 2 h. Additionally, the solution was continuously stirred to increase contact between photocatalyst and MB molecules and hence, driving a rapid photodegradation. The reactor was covered to avoid contribution from the ambient light, assuring that the photodegradation was merely driven by UV irradiation. Photodegradation of MB was detected through absorption change at 665 nm measured using UV/vis spectrometer. The decrease of MB optical density was used to determine the decreasing MB concentration due to the catalytic activity of either WO₃ nanoparticles or WO₃/TiO₂ nanocomposite.

3. RESULTS AND DISCUSSION

3. 1. Microstructural and Optical Properties of WO₃/TiO₂ Nanocomposite Morphology of the bare
WO₃ nanoparticles and the resultant WO₃/TiO₂ nanocomposites is shown by SEM micrographs (Figure 1). The morphology of WO₃ (Figure 1a) is characterized by aggregated microstructure with primary crystallites in spherical shape. The average crystallite size is estimated around 47 nm with size distribution as shown in Figure 1b. The histogram of crystallite size distribution further indicates polydispersity of WO₃. For WO₃/TiO₂ nanocomposites, the SEM image depicts the presence of WO₃ (large and aggregated particles in dark) and TiO₂ (tiny particles, white spots). Nonetheless, it can be seen that changing the ratio between WO₃ and TiO₂ alters the surface properties. Even though both nanocomposites showed agglomeration, it is also clear that WO₃/TiO₂ (1:1) nanocomposites revealed a relatively compact surface (Figure 1a) while a fractal surface characterized WO₃/TiO₂ (1:3) nanocomposite. It is worth mentioning that the nanocomposite in this work porous WO₃ was utilized as the composite matrices while smaller TiO₂ nanoparticles were used as composite filler. Hence, the very different surface morphologies can be rationalized with different hydrophilicities between TiO₂ and WO₃ within nanocomposites [18]. TiO₂ is more hydrophilic than WO₃, increasing TiO₂ nanoparticles plausibly leads to a strong aggregation among nanoparticles which eventually breaks the structural integrity of WO₃ porous network as the matrix. As mentioned earlier, porous nanocomposite structure with larger surface area and bigger pore volume is preferred to improve the photocatalytic performance since larger specific surface areas are able to enhance the adsorption of dye molecules. BET surface area and pore structure of different nanocomposites were then investigated by the nitrogen (N₂) adsorption–desorption isotherm (Figure 2). Barret-Joyner-Halenda (BJH) analysis for pore size distribution unravels an average pore size distribution of 4.5 and 9.5 nm for WO₃/TiO₂ (1:1) and (1:3) nanocomposites, respectively, which is in agreement with SEM identification where WO₃/TiO₂ (1:3) nanocomposite shows fractal and larger porous surface. Furthermore, it reveals that both nanocomposite has mesoporosus structures, i.e. pore diameters are below 50 nm. The adsorption-desorption curve is classified as Type IV isotherm curve which also indicates the presence of mesoporous structure. The presence of hysteresis loop and high adsorption at high relative pressure (p/p₀) 0.7 – 1 further indicates the presence of slit-like pores and implies the structure of large mesopores and macropores [19]. Between WO₃/TiO₂ (1:1) and (1:3) nanocomposites, isotherm of (1:3) lifted up due to the higher surface area possessed by (1:3) nanocomposites, i.e. 11.47 m²/g vs. 4.74 m²/g for (1:1). This indicates that TiO₂ constituent possessed larger surface area than WO₃. Such higher specific surface area is beneficial for higher dye adsorption and eventually for photocatalytic degradation process. Nonetheless, the pore volume of both nanocomposites does not differ significantly which spans between 0.20 – 0.22 cm³/g.

A quantitative XRD analysis was carried out to unravel the underlying crystallite structure forming the WO₃/TiO₂ nanocomposites (Figure 3). The diffraction patterns of bare WO₃ and TiO₂ utilized as nanocomposite constituents fit the monoclinic WO₃ crystal and anatase TiO₂ crystal structure according to JCPDS No. 00-043-1035 and JCPDS No. 00-021-1272, respectively. As estimated by Debye-Scherrer formula, the crystallite size of WO₃ and TiO₂ is 46 and 75 nm, respectively. All diffraction patterns of WO₃/TiO₂ (1 :1) nanocomposite show a combination of anatase TiO₂ and WO₃ without...
any impurities. Furthermore, the crystallinity of WO$_3$/TiO$_2$ nanocomposite exceeds 90%, in which the crystallite size of WO$_3$ (51.7 nm) and TiO$_2$ (73.3 nm) is not significantly altered compared with the individual crystallite size.

The purity of WO$_3$/TiO$_2$ nanocomposites was further investigated by Raman spectroscopy (Figure 4). For TiO$_2$, the presence of strong Raman peaks at 150 – 450 cm$^{-1}$ indicates the metal – oxygen bonding in TiO$_2$. Different vibration modes in Raman spectrum of TiO$_2$ characterize E$_g$, B$_g$, and A$_g$, which are due to symmetric stretching vibration, symmetric bending vibration, and antisymmetric bending vibration of TiO$_2$, respectively [19]. In general, the Raman spectrum of TiO$_2$ corroborates the assignment of anatase TiO$_2$ phase in XRD analysis. Raman spectrum is altered as TiO$_2$ is composited with WO$_3$. Additional Raman bands appear at 225, 700 and 805 cm$^{-1}$. The band at 225 cm$^{-1}$ is attributed to v(W–O–W), while the bands at 700 and 805 cm$^{-1}$ are assigned to v(O–W–O) [10].

UV/vis absorption spectra of WO$_3$/TiO$_2$ nano-composites dispersed in water characterize the optical properties, particularly the light harvesting ability (Figure 5). For WO$_3$ constituent, it shows absorption maximum at 350 nm with a broad absorption tail in the visible region. Interestingly, the absorption spectrum of TiO$_2$ shows exceptionally broad and high absorption in visible region (400 – 650 nm) than UV region which is typically associated with band gap absorption. This indicates that the anatase TiO$_2$ used in this study possesses large concentration of defect chemistry inducing strong intraband absorption in visible regime [20]. Nonetheless, it should be noted that defect sites in metal oxide semiconductor are beneficial as these sites provide catalytic center for reduction or oxidation process. When it is composited with WO$_3$, in which WO$_3$ concentration is higher than TiO$_2$ (3:1), the absorption spectrum is dominated by UV absorption. Among three different compositions, WO$_3$/TiO$_2$ (1:1) nanocomposite exhibited both the strong UV-band gap absorption and broad visible absorption due to defects. Therefore, the optical properties of WO$_3$/TiO$_2$ (1:1) nanocomposite features an advantageous character for photocatalyst since it shows band gap absorption for charge separation as well as intraband absorption for catalytic center.

### 3.2. Photocatalytic Degradation of MB and Kinetic Study

The photocatalytic activity of mesoporous WO$_3$/TiO$_2$ nanocomposite was evaluated for the degradation of MB as organic pollutant. Initially, the absorption of MB in aqueous medium is measured as the reference absorption value for undegraded MB (10 ppm). Once the photocatalyst is added into the solution and UV irradiation is applied, the absorption intensity gradually decreases with respect to time, suggesting that the dyes are degraded in the presence of a photocatalyst under direct UV irradiation (Figure 6a). As a control experiment, photocatalytic degradation of MB using bare TiO$_2$ and WO$_3$ is also presented. Under UV irradiation, WO$_3$ catalyst facilitates MB degradation by 68% in 15
min while minimum MB degradation by 42% is yielded when TiO₂ catalyst is employed. These results show the nature of MB adsorption and photocatalytic degradation in both materials. Smaller surface area in WO₃ and its negatively charge surface lead to a more rapid degradation of MB thus saturated adsorption condition is achieved fast, while adsorption of MB using TiO₂ takes longer time and hence, the degradation becomes slower. Nevertheless, the yield of MB degradation after 120 min photocatalyst.

Further catalyst dose dependent study was then carried out. The results imply that increasing dose of WO₃/TiO₂ (1:3) nanocomposites from 90 to 360 mg leads to a more rapid photocatalytic MB degradation (Figure 6b). A quantitative data of temporal MB removal at different doses reveals that 80% of MB is removed using the lowest dose catalyst of 90 mg. A higher dose facilitates very rapid MB degradation within the first 15 min of irradiation, for example 96% of MB is degraded in contact with 360 mg of catalyst, whilst merely 40% of MB is photodegraded using 90 mg of catalyst. A more rapid and higher MB removal at higher dose of photocatalyst is expected since the amount of adsorbed dye on catalyst as well as the light–dye–catalyst interaction increases.

To better understand the photocatalytic degradation mechanism, kinetic analysis of MB degradation using WO₃/TiO₂ nanocomposite under UV irradiation was carried out. The first kinetic model used is the pseudo-first-order model:

$$r = -\frac{dC}{dt} = k_{obs}C$$  \hspace{1cm} (1)

By integrating the above equation, the kinetic model can be expressed as follows:

$$\ln\frac{C_0}{C} = k_{obs}t$$  \hspace{1cm} (2)

where $C_0$ is the initial concentration in the bulk solution, $t$ is the reaction time and $k_{obs}$ is the observed pseudo-first-order rate constant. A plot of $\ln(C/C_0)$ versus $t$ will result in a linear curve, in which the slope equals to the observed rate constant of $k_{obs}$ (Figure 7). For the pseudo-second order kinetic model, two-site-occupancy adsorption is considered. The kinetic model is represented by:

$$\frac{t}{C_e} = \frac{1}{k_2C_e^2} + \left(\frac{1}{C_e}\right)t$$  \hspace{1cm} (3)

The initial sorption rate is defined by the following equation:

$$h = k_2C_e^2$$  \hspace{1cm} (4)

where $C_e$ is dye concentration at equilibrium in solution (mg L⁻¹), $C_i$ is the catalyst uptake capacity at any time (mmol g⁻¹), and $k_2$ is the pseudo-second-order rate constant (g mmol⁻¹ min⁻¹) which is obtained from the offset of the linear plot of $t/C_e$ vs $t$. Kinetic rate constants determined from both kinetic models are summarized in Table 1.

In addition, other kinetic model, i.e. Langmuir-Hinshelwood (L-H), is applied to the data for comparison:

$$\frac{C_e}{q_e} = \frac{1}{K_dG_m} + \frac{C_e}{q_m}$$  \hspace{1cm} (5)

where $q_m$ is the Langmuir constant related to adsorption capacity (mg g⁻¹), $K_d$ is the adsorption rate (mg L⁻¹ h⁻¹), $q_e$ is the dye concentration at equilibrium on WO₃/TiO₂.
nanocomposite (mg L\(^{-1}\)), and \(C_e\) is dye concentration at equilibrium in solution (mg L\(^{-1}\)) (see Table 2).

Kinetic of photocatalytic degradation of MB using WO\(_3\)/TiO\(_2\) nanocomposite is discussed as follows: The variation of catalyst concentration as a function of irradiation time gives a linear fit curve (Figure 7). The results clearly denote that the photocatalytic reaction on WO\(_3\)/TiO\(_2\) nanocomposite is found best to follow the pseudo-second-order kinetic than the pseudo-first-order kinetic model. This indicates that the molecules are mainly chemisorbed rather than physisorbed onto the catalyst surface before photocatalytic degradation occurs. In addition, L-H kinetic model fails to represent the degradation mechanism indicated by lower \(R^2\), and hence, two molecules adsorption at neighboring sites are not the main mechanism on the investigated system here. According to the pseudo-second-order kinetic model the value of \(k\) (0.162 min\(^{-1}\)) is found to be highest for the MB degradation corresponding to the utilization of WO\(_3\)/TiO\(_2\) (1:1) nanocomposite. Compared with other studies, the resulting rate constant for bare anatase TiO\(_2\) \((k \sim 0.024\) min\(^{-1}\)) is comparable and slightly better than that of C-doped TiO\(_2\) \((k \sim 0.01\) min\(^{-1}\)) under UV irradiation [21]. The use of mesoporous WO\(_3\) prepared here also outperforms the photocatalytic degradation rate of MB using other mesoporous WO\(_3\) prepared from ammonium metatungstate [22], i.e. 0.041 vs. 0.0025 min\(^{-1}\). Comparing the degradation rate of MB with WO\(_3\)/TiO\(_2\) hollow spheres [23], i.e. 0.162 vs 0.041 min\(^{-1}\), the present work already exhibits promising materials for photocatalysis.

### 3.3. Photocatalytic Degradation Mechanism of Methylene Blue

The photocatalytic degradation mechanism of MB adsorbed onto WO\(_3\)/TiO\(_2\) nanocomposite surface involves sequential charge separation and charge transfers (Figure 8). Initial charge separation occurs in WO\(_3\)/TiO\(_2\) composite upon UV irradiation. As UV irradiation (\(\lambda \sim 365\) nm, E \(\sim 3.4\) eV) is applied and is higher than the optical band gap of WO\(_3\) (2.7 eV) and TiO\(_2\) (3.2 eV), the absorbed photons creates exciton in both WO\(_3\) and TiO\(_2\), that is a charge separated state between photoexcited electron in the conduction band (CB) and hole in the valence band (VB). Once charge separated state is formed, the possible charge transfers between WO\(_3\) and TiO\(_2\) are as follows: the excited electron is transferred from the CB of TiO\(_2\) to the CB of WO\(_3\), while the hole in VB of WO\(_3\) is injected to the VB of TiO\(_2\). These charge transfer likely occur due to the more positive VB and CB position of TiO\(_2\) than WO\(_3\).

![Figure 8. Proposed photocatalytic degradation mechanism of MB by WO\(_3\)/TiO\(_2\) nanocomposite under UV irradiation](image-url)
WO₃ and hence, the Gibbs free energy (ΔG) indicating the strength of driving force for electron and hole transfer is exergonic (thermodynamically possible). The photoexcited and accumulated electrons and holes eventually react with H₂O₂ and H₂O, respectively, to form OH• radicals (Equations (7)–(9)). However, these charge transfer are not always necessary since the band energy level of both WO₃ and TiO₂ already allows for inducing redox reaction with H₂O₂ and H₂O forming OH• radicals and both are capable of photocatalytically degrading the chemisorbed MB on either WO₃ or TiO₂ part under UV irradiation.

\[ \text{WO}_3\text{TiO}_2 + \text{hv} \rightarrow \text{e}^-\text{CB} + \text{h}^+\text{VB} \]  
\[ \text{h}^+ + \text{H}_2\text{O} \rightarrow \text{H}^+ + \text{OH}^- \]  
\[ \text{h}^+ + \text{OH}^- \rightarrow \text{OH}• \]  
\[ \text{e}^- + \text{H}_2\text{O} \rightarrow \text{OH}^- + \text{OH}^+ \]  

These OH• radicals formation are essential to oxidize and reduce the adsorbed MB molecules yielding the carbon dioxide, H₂O, and other photodegraded molecules. It should be noted that there is a driving force in the surface of photocatalyst enabling the desorption of photodegraded products from the active site to the bulk. This mechanism corresponds to the regeneration of active site in WO₃/TiO₂ associated with the photocatalytic cycle. Furthermore, the reaction between electrons and holes with H₂O₂ and H₂O, respectively, competes with the recombination reaction, which is undesirable as the produced heat considerably reduces the photocatalytic activity.

4. CONCLUSION

Photocatalyst of WO₃/TiO₂ nanocomposites were successfully prepared by wet chemical synthesis. The investigated WO₃/TiO₂ nanocomposites showed highly mesoporous structure and the WO₃:TiO₂ ratio was varied, i.e. (1:1), (1:3), and (3:1) in them. Physical characterization indicated that the higher TiO₂ content in nanocomposites not only contributed to larger surface area but also to more defects site as indicated by intraband visible absorption. WO₃/TiO₂ (1:3) nanocomposite showed the highest degradation rate and MB degradation efficiency (89%) within 120 min. WO₃ constituent played role in the initial adsorption and rapid degradation, whilst TiO₂ nanoparticles were essential to enlarge the adsorption sites and were responsible for slow degradation mechanism. The pseudo-second-order model best fitted the removal of MB in aqueous solution indicating that chemisorption is the initial adsorption mechanism followed by photocatalytic processes. Future study on the improvement of catalytic efficiency of WO₃/TiO₂ photocatalyst and organic dye degradation rate under UV/vis irradiation should be considered for further practical application.

5. REFERENCES


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