



Photocatalytic Performance of a $\text{WO}_3/\text{TiO}_2/\text{SiO}_2$ Composite for Rhodamine B Degradation Under Different Light Conditions

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ARTICLE INFO

Keywords : $\text{WO}_3/\text{TiO}_2/\text{SiO}_2$ Composite, Photocatalysis, Rhodamine B, Visible Light Degradation

Received : 2 July

Revised : 20 August

Accepted: 20 September

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ABSTRACT

A $\text{WO}_3/\text{TiO}_2/\text{SiO}_2$ composite was synthesized and evaluated as a photocatalyst for the degradation of Rhodamine B. The composite was characterized by scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDX), powder X-ray diffraction (XRD), and Fourier-transform infrared spectroscopy (FTIR). SEM analysis revealed an irregular and heterogeneous surface morphology with pronounced particle agglomeration. The XRD pattern indicated the coexistence of monoclinic WO_3 and anatase TiO_2 , while SiO_2 exhibited a predominantly amorphous character. EDX analysis showed 7.60% WO_3 , 21.80% TiO_2 , and 64.50% SiO_2 , together with 6.12% minor oxide components. Under the contact-time experiment, 89.03% Rhodamine B degradation was obtained after 75 min using 0.15 g of composite and 10 ppm dye under visible-light irradiation. In the initial-concentration experiment, the highest observed degradation was 86.40% at an initial Rhodamine B concentration of 10 ppm. Among the irradiation conditions evaluated, visible light produced the highest degradation (89.30%), compared with UV irradiation (69.30%) and the dark condition (67.90%). These results indicate that the $\text{WO}_3/\text{TiO}_2/\text{SiO}_2$ composite exhibited the highest Rhodamine B removal under the tested visible-light conditions, with adsorption and non-photochemical processes also contributing to dye removal

INTRODUCTION

Rhodamine B is a synthetic dye that is widely used in various industrial applications and is frequently used as a model pollutant in photocatalytic degradation studies. Its persistence in aqueous systems and potential environmental impact have encouraged the development of effective treatment methods for dye-containing wastewater. Photocatalytic degradation using semiconductor materials is considered a promising approach because reactive species generated during irradiation can promote the decomposition of organic pollutants.

A preliminary study by the authors investigated the effect of catalyst loading on Rhodamine B photodegradation using a $\text{WO}_3/\text{TiO}_2/\text{SiO}_2$ composite. The study identified 0.15 g as the optimum catalyst mass, giving 85.10% Rhodamine B degradation under visible-light irradiation for 75 min (Adhaina et al., 2020).

However, the previous study was primarily focused on catalyst loading and did not systematically evaluate the effects of contact time, initial Rhodamine B concentration, or different irradiation conditions. In addition, the structural characteristics of the composite were not comprehensively investigated. Therefore, the present work extends the previous study by further characterizing the $\text{WO}_3/\text{TiO}_2/\text{SiO}_2$ composite and systematically evaluating the effects of contact time, initial Rhodamine B concentration, and irradiation conditions on its photocatalytic performance.

The objective of this study was to determine the optimum reaction conditions for Rhodamine B degradation and to provide a more comprehensive evaluation of the photocatalytic performance of the $\text{WO}_3/\text{TiO}_2/\text{SiO}_2$ composite under dark, UV, and visible-light conditions.

LITERATURE RIVIEW

TiO_2 is one of the most widely studied semiconductor photocatalysts because of its chemical stability and relatively low toxicity. However, its wide band gap limits its ability to utilize visible light efficiently. The incorporation of SiO_2 can improve the dispersion of TiO_2 and provide a supporting surface, while WO_3 is a semiconductor capable of responding to visible light. The combination of WO_3 and TiO_2 may facilitate interfacial charge transfer and improve charge separation, while SiO_2 can contribute to surface adsorption and dispersion of the active semiconductor phases (Lettieri et al., 2021; Haghighi et al., 2022).

METHODOLOGY

A. Materials and Instrumentation

Silicon dioxide (SiO₂), titanium dioxide (TiO₂), tungsten trioxide (WO₃), ethanol (99%), ammonium hydroxide (NH₄OH), and Rhodamine B were used as the principal materials. The visible-light experiments used a 10 W pink-blue Grow Light LED with predominantly blue and red emission at approximately 450 and 660 nm, respectively. The composite was characterized using an X-ray diffractometer (XRD), scanning electron microscope equipped with energy-dispersive X-ray spectroscopy (SEM-EDX), and Fourier-transform infrared spectrometer (FTIR).

B. Preparation and Characterisation of WO₃/TiO₂/SiO₂ Composite

The WO₃/TiO₂/SiO₂ composite was prepared using a sequential wet impregnation procedure. First, TiO₂ was incorporated onto SiO₂ to form the TiO₂/SiO₂ support. A 15 wt% TiO₂ loading relative to SiO₂ was used, corresponding to 2.70 g of TiO₂ for 18.00 g of SiO₂. TiO₂ was dispersed in 99% ethanol and mixed with SiO₂ in a round-bottom flask. The mixture was refluxed at 60–70 °C for 5 h, followed by centrifugation and drying at 120 °C for 5 h.

The resulting TiO₂/SiO₂ support was subsequently impregnated with WO₃. A 10 wt% WO₃ loading relative to the TiO₂/SiO₂ support was used, corresponding to 2.12 g of WO₃. Because WO₃ was used as a powder, it was not described as being dissolved. Instead, the WO₃ powder was dispersed in a sufficient volume of 25 wt% aqueous NH₄OH to provide an alkaline impregnation medium and form a workable suspension. The WO₃-containing suspension was then contacted with the TiO₂/SiO₂ support and mixed to facilitate wet impregnation. The impregnated material was subsequently dried and calcined at 500 °C for 2 h to obtain the WO₃/TiO₂/SiO₂ composite (Haghighi et al., 2022; Jiménez et al., 2026; Marceau et al., 2021; Yuan et al., 2025). The sequential preparation was used to form the TiO₂/SiO₂ support before the subsequent incorporation of WO₃, while the alkaline medium was intended to facilitate precursor-support interactions during impregnation (Jiménez et al., 2026; Marceau et al., 2021). The SEM-EDX characterization data for the WO₃/TiO₂/SiO₂ composite were previously reported by the authors (Adhaina et al., 2020). In the present study, these data are retained as supporting morphological and compositional information and are discussed together with the additional XRD and FTIR characterization.

C. Photocatalytic Test for Rhodamine B Degradation

1. Effect of Contact Time on Rhodamine B Degradation

A 0.15 g portion of WO₃/TiO₂/SiO₂ composite was added to 10 mL of Rhodamine B solution at the desired initial concentration. The suspension was irradiated in a closed reactor under dark, UV, or visible-light conditions. The visible-light source was a 10 W Grow Light LED, while the UV source was operated at a wavelength of 365 nm. Contact times of 30, 60, 75, and 90 min were investigated. After irradiation, the catalyst was separated by filtration, and the filtrate was analyzed using UV-Vis spectrophotometry at the maximum absorption wavelength of Rhodamine B (λ_{max} = 554 nm). For UV-Vis measurement, the filtrate was diluted 50-fold before analysis. Each experimental

condition was evaluated using three independent replicates, and the results were reported as mean $\pm$ standard deviation. The percentage degradation of RhB was calculated as: Degradation (%) = $[(A_0 - A_t)/A_0] \times 100$, where A_0 and A_t are the absorbances of the diluted RhB solution before irradiation and after irradiation for time t , respectively. The optimum contact time was selected as the condition giving the highest percentage degradation.

2. Effect of Initial Rhodamine B Concentration on Degradation

The $\text{WO}_3/\text{TiO}_2/\text{SiO}_2$ composite (0.15 g) was added to 10 mL of Rhodamine B solution at initial concentrations of 5, 10, 15, 20, and 25 ppm. The reactions were conducted for 75 min, corresponding to the selected optimum contact time, under visible-light irradiation using the 10 W pink-blue Grow Light LED in a closed reactor. After irradiation, the catalyst was removed by filtration, and the filtrate was diluted 50-fold with distilled water prior to UV-Vis analysis. The absorbance of RhB was measured at 554 nm (Adhaina et al., 2020). Each experiment was performed in three independent replicates ($n = 3$), and the results were expressed as mean $\pm$ standard deviation. The percentage degradation was calculated using the equation described above. The optimum initial concentration was defined as the concentration giving the highest percentage degradation under the tested conditions.

A 0.15 g portion of $\text{WO}_3/\text{TiO}_2/\text{SiO}_2$ composite was added to 10 mL of 10 mg L^{-1} Rhodamine B (RhB) solution and irradiated for 75 min. The photocatalytic experiments were conducted in a closed reactor under UV, visible-light, and dark conditions. For the UV irradiation experiment, a 365 nm UVA lamp was used. The visible-light condition used the 10 W pink-blue Grow Light LED with predominant emission at approximately 450 and 660 nm. After irradiation, the catalyst was removed by filtration, and the filtrate was analyzed using UV-Vis spectrophotometry. The absorbance of RhB was measured at 554 nm, corresponding to its maximum absorption wavelength (λ_{max}) in aqueous solution (Adhaina et al., 2020). Prior to UV-Vis analysis, the filtrates were diluted 50-fold with distilled water. Each photocatalytic experiment was performed in three independent replicates ($n = 3$), and the results were expressed as mean $\pm$ standard deviation. The percentage degradation of RhB was calculated using the equation described above. The optimum irradiation condition was defined as the condition giving the highest percentage degradation after 75 min.

RESULT AND DISCUSSION

A. Characterisation of the WO₃/TiO₂/SiO₂ Composite

The WO₃/TiO₂/SiO₂ composite was prepared through a sequential wet-impregnation approach, in which the TiO₂/SiO₂ component was first formed before the subsequent incorporation of WO₃. This sequence was selected to facilitate the initial dispersion of TiO₂ within the SiO₂ support, followed by the incorporation of WO₃ into the preformed TiO₂/SiO₂ material. The resulting material was obtained as a fine grayish-white powder. The crystalline phases and structural characteristics of the starting materials and resulting composite were subsequently investigated by X-ray diffraction (XRD).

Figure 1(a) shows the XRD pattern of the SiO₂ starting material. A broad diffraction feature centered at approximately 22° is observed, indicating the predominantly amorphous nature of SiO₂. The observed pattern was compared with the JCPDS/ICDD reference data (Card No. 46-1045). The absence of sharp and intense crystalline reflections further supports the predominantly amorphous character of the SiO₂ material.

The XRD pattern of WO₃ is presented in Figure 1(b). Several distinct diffraction reflections are observed at 2θ values of 23.13°, 23.61°, 24.37°, 28.66°, 28.93°, 33.29°, 33.61°, 34.18°, 41.51°, 41.93°, 47.28°, 48.30°, 49.97°, 50.35°, 50.75°, 53.51°, 54.22°, 54.81°, and 56.12°. The characteristic reflections in the range of approximately 23–25° are consistent with monoclinic WO₃ and were assigned by comparison with the JCPDS/ICDD reference pattern (Card No. 43-1035). The phase identification was based on the overall agreement between the observed diffraction pattern and the standard reference pattern rather than on any single diffraction reflection.

Figure 1(c) presents the XRD pattern of TiO₂. The major reflections observed at approximately 25.3°, 37.8°, 48.0°, 53.9°, 55.1°, and 62.7° are characteristic of the anatase phase of TiO₂ and are consistent with JCPDS/ICDD Card No. 21-1272. The intense reflection near 25.3° is attributed to the (101) plane, while the other characteristic reflections correspond to the anatase TiO₂ structure. The XRD pattern of the WO₃/TiO₂/SiO₂ composite is shown in Figure 1(d). The composite pattern contains characteristic reflections attributable to both anatase TiO₂ and monoclinic WO₃, whereas the SiO₂ component mainly contributes to the broad amorphous background. The persistence of the characteristic TiO₂ and WO₃ reflections after the sequential impregnation process indicates that the crystalline phases of these oxide components were retained during composite preparation. The absence of a distinct crystalline SiO₂ reflection is consistent with the predominantly amorphous nature of the SiO₂ starting material. Overall, the XRD results are consistent with the formation of a multiphase WO₃/TiO₂/SiO₂ composite containing anatase TiO₂ and monoclinic WO₃ supported on predominantly amorphous SiO₂, in agreement with the characteristics reported for WO₃/TiO₂/SiO₂ photocatalytic composites (Haghighi et al., 2022).

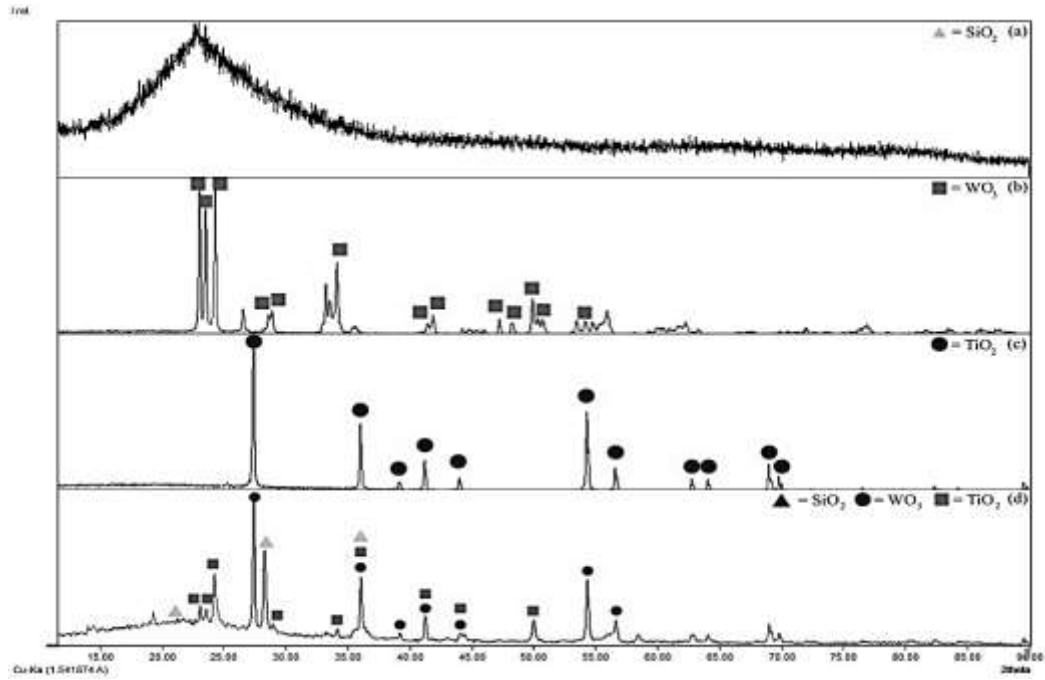


Figure 1. XRD Patterns of (A) SiO_2 , (B) WO_3 , (C) TiO_2 , and (D) $\text{WO}_3/\text{TiO}_2/\text{SiO}_2$ Composite. the Observed Diffraction Patterns Were Compared With JCPDS/ICDD Reference Data for SiO_2 (Card No. 46-1045), Monoclinic WO_3 (Card No. 43-1035), and Anatase TiO_2 (Card No. 21-1272)

The SEM image presented in Figure X was obtained at 3000 $\times$ magnification, which differs from the magnification used in the authors' previous report (Adhaina et al., 2020) (Figure 2). At this magnification, the composite exhibits an irregular and heterogeneous surface morphology, with particle agglomeration and non-uniform surface features. The observed heterogeneity is consistent with the multiphase nature of the $\text{WO}_3/\text{TiO}_2/\text{SiO}_2$ composite. The SEM-EDX characterization of the same composite system was previously reported by the authors (Adhaina et al., 2020); therefore, the present SEM observation is interpreted as complementary morphological information rather than as an entirely independent material characterization.

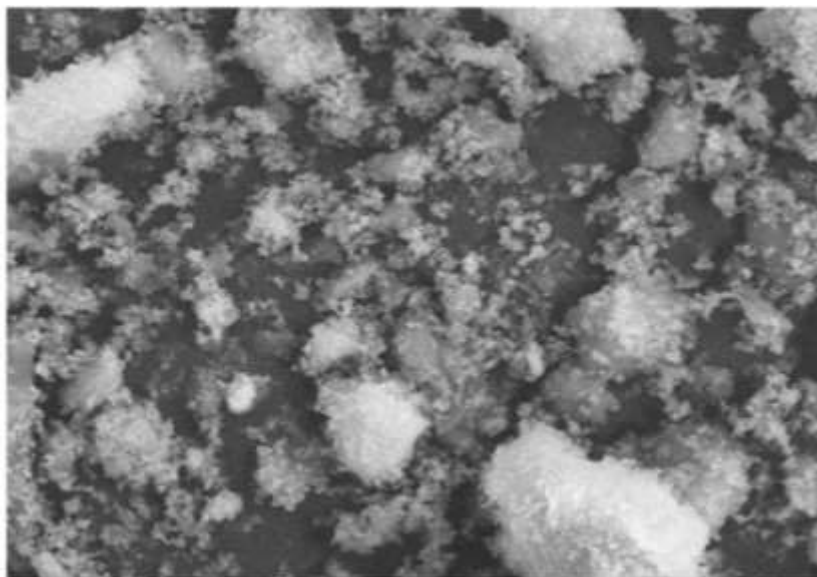


Figure 2. SEM Micrograph of the WO₃/TiO₂/SiO₂ Composite At 3,000× Magnification Showing the Irregular, Heterogeneous, and Agglomerated Surface Morphology

The EDX analysis provides complementary compositional information for the composite. As presented in Table 1, the detected composition consisted mainly of SiO₂ (64.50%), TiO₂ (21.80%), and WO₃ (7.60%), together with minor oxide components of NaO (3.04%) and Al₂O₃ (3.08%). The simultaneous detection of Si, Ti, W, and O supports the presence of the three intended oxide components in the composite. The relatively high SiO₂ content is consistent with its role as the supporting component, while the detected TiO₂ and WO₃ indicate their incorporation into the SiO₂-based material.

The EDX-derived composition differs from the nominal composition used during synthesis, particularly for TiO₂ and WO₃. This difference is reasonable because EDX provides semi-quantitative compositional information from the specific region analyzed rather than a direct measurement of the bulk composition. The measured values may be influenced by local compositional heterogeneity, surface morphology, and the interaction volume of the electron beam. Therefore, the EDX results should be interpreted as representative of the analyzed region rather than as the exact bulk composition of the entire composite.

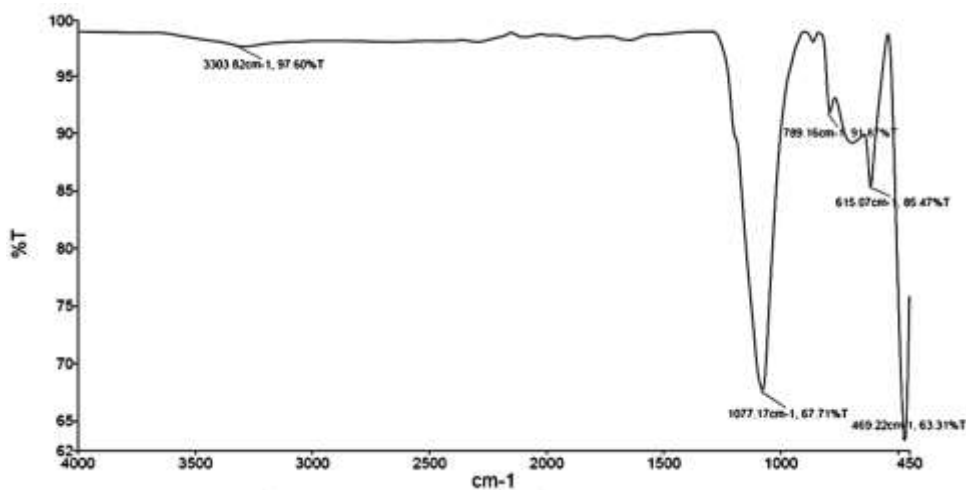
Overall, the irregular, heterogeneous, and agglomerated morphology observed by SEM, together with the EDX composition and the crystalline-phase information obtained from XRD, provides complementary evidence supporting the formation of the WO₃/TiO₂/SiO₂ composite.

Table 1. EDX-Derived Oxide Composition of the WO₃/TiO₂/SiO₂ Composite

No	Component	Composition (%)
1.	TiO ₂	21.80
2.	WO ₃	7.60
3.	SiO ₂	64.50
4.	NaO	3.04
5.	Al ₂ O ₃	3.08

FTIR characterization was performed to identify the characteristic vibrational bands associated with the oxide components in the WO₃/TiO₂/SiO₂ composite (Figure 3). The spectrum shows a broad absorption band at 3303.82 cm⁻¹, which can be attributed to O-H stretching vibrations associated with surface hydroxyl groups and/or adsorbed water. A strong band at 1077.17 cm⁻¹ is assigned to the asymmetric stretching vibration of Si-O-Si, while the band at 469.22 cm⁻¹ is attributed to Si-O-Si bending vibrations, consistent with characteristic silica features (Yuan et al., 2025; Jin et al., 2024).

The absorption bands in the lower-wavenumber region are associated with metal-oxygen vibrations. The band at 615.07 cm⁻¹ can be attributed to metal-oxygen vibrations associated with the Ti-O-Ti and/or W-O-W framework, while the band at 789.16 cm⁻¹ may involve W-O-related vibrations and Ti-O-Si interactions. These assignments should be considered in the context of overlapping vibrations because Ti-O-Ti and W-O-W stretching modes occur within similar spectral regions. Recent FTIR studies of WO₃-containing materials report W-O/W-O-W vibrations broadly in the 450–900 cm⁻¹ region, while Ti-O vibrations occur in the lower-wavenumber region (Jin et al., 2024; Kaitheri et al., 2026).

Figure 3. FTIR spectra of WO₃/TiO₂/SiO₂ Composite

Overall, the FTIR spectrum exhibits characteristic vibrational features attributable to Si–O–Si, metal–oxygen, and surface hydroxyl groups, supporting the presence of the SiO₂, TiO₂, and WO₃ components in the composite. The FTIR results are consistent with the XRD and EDX analyses, which independently confirm the presence of the corresponding oxide phases and constituent elements.

B. Photocatalytic Degradation Test of Rhodamine B Using the WO₃/TiO₂/SiO₂ Composite

1. Effect of Contact Time on Rhodamine B Degradation

Figure 5 shows that the highest Rhodamine B degradation was obtained at a contact time of 75 min, reaching $89.03 \pm 0.08\%$. The degradation values at 30, 60, and 90 min were 60.60%, 61.80%, and 83.90%, respectively. The increase in degradation from 30 to 75 min can be attributed to the longer interaction time between the photocatalyst, light, and Rhodamine B, which provides more opportunity for adsorption and photocatalytic reactions to occur. At 90 min, the degradation slightly decreased compared with that at 75 min. This decrease may be associated with the establishment of adsorption–desorption equilibrium and/or reduced photocatalytic efficiency as the reaction progresses. Therefore, 75 min was selected as the optimum contact time based on the highest degradation obtained under the tested conditions.

The slight decrease in degradation from 89.03% at 75 min to 83.90% at 90 min may indicate that the photocatalytic system approached a limiting condition, in which the available Rhodamine B molecules and accessible surface sites became less favorable for further degradation. Adsorption–desorption processes at the catalyst surface may also contribute to the observed change. Therefore, 75 min was selected as the optimum contact time based on the highest observed degradation percentage. The error bars represent the variation among the replicate measurements.

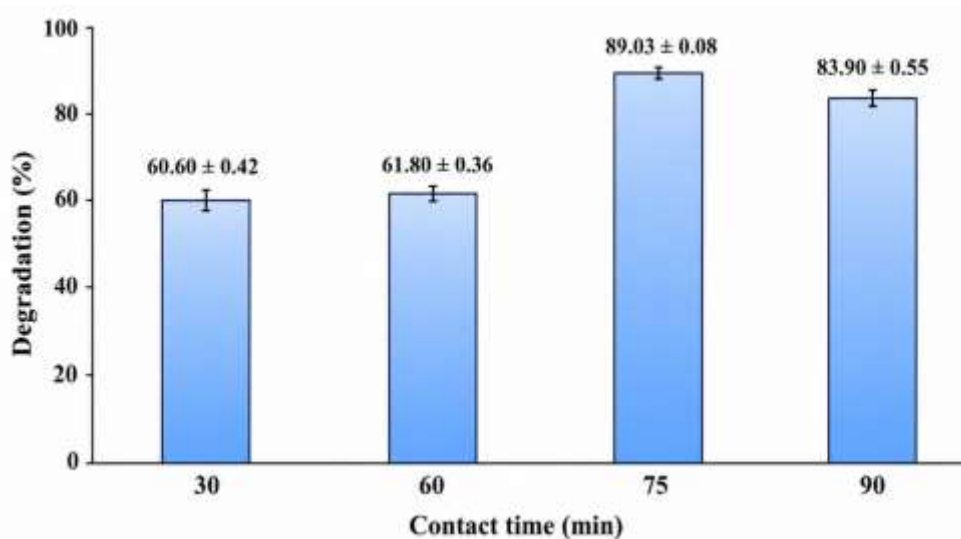


Figure 4. Effect of Contact Time on Rhodamine B Degradation Using the WO₃/TiO₂/SiO₂ Composite Under Visible-Light Irradiation. Data Are Presented as Mean ± Standard Deviation (N = 3)

2. Effect of Initial Rhodamine B Concentration on Degradation

Figure 5 shows the effect of the initial Rhodamine B concentration on its photocatalytic degradation using the $\text{WO}_3/\text{TiO}_2/\text{SiO}_2$ composite. The degradation efficiency increased from 72.80% at 5 ppm to the highest observed value of 86.40% at 10 ppm. At higher initial concentrations, the degradation efficiencies were 79.49%, 85.20%, and 84.20% at 15, 20, and 25 ppm, respectively. Although the degradation efficiency fluctuated at concentrations above 10 ppm, none of the tested concentrations produced a higher degradation value than that obtained at 10 ppm. Therefore, 10 ppm was selected as the optimum initial Rhodamine B concentration under the tested conditions.

The increase in degradation efficiency from 5 to 10 ppm may indicate that, within this concentration range, the amount of Rhodamine B available for interaction with the photocatalyst was favorable for the photocatalytic process. At higher dye concentrations, the degradation efficiency may be influenced by the increasing number of dye molecules relative to the available active sites on the catalyst surface and by attenuation of light penetration through the solution. A more concentrated Rhodamine B solution can absorb or scatter a greater fraction of the incident light, thereby reducing the light reaching the catalyst surface and potentially limiting the generation and utilization of photogenerated reactive species. The variations observed at 15–25 ppm may reflect the combined effects of surface interaction, light attenuation, and experimental variation. Based on the highest observed degradation efficiency, 10 ppm was selected as the optimum initial Rhodamine B concentration for subsequent photocatalytic experiments.

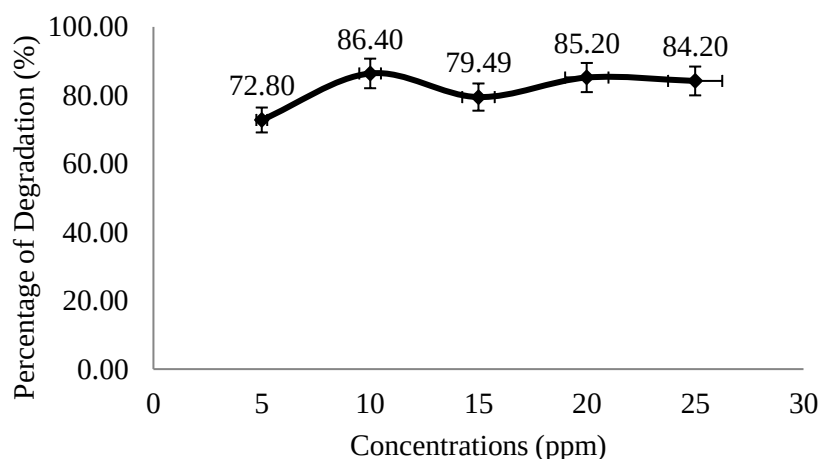


Figure 5. Effect of Initial Rhodamine B Concentration on Degradation Using the $\text{WO}_3/\text{TiO}_2/\text{SiO}_2$ Composite. Data Are Presented as Mean $\pm$ Standard Deviation (N = 3)

3. Effect of Light Source on WO₃/TiO₂/SiO₂ Photocatalysis

Figure 6 shows the effect of the irradiation condition on Rhodamine B degradation using the WO₃/TiO₂/SiO₂ composite. The highest observed degradation was obtained under visible-light irradiation, reaching 89.30%, followed by UV irradiation at 69.30% and the dark condition at 67.90%. The relatively high degradation under dark conditions indicates that adsorption and other non-photochemical processes contributed substantially to the removal of Rhodamine B. Irradiation resulted in additional degradation, with the visible-light condition producing the highest overall degradation among the tested conditions.

The higher degradation observed under visible-light irradiation can be related to the combined properties of the components in the WO₃/TiO₂/SiO₂ composite. SiO₂ primarily functions as a support and provides a surface for interaction with Rhodamine B, while TiO₂ acts as a semiconductor component. WO₃ may facilitate interfacial charge transfer with TiO₂ and improve charge separation, thereby reducing electron-hole recombination (Wang et al., 2021; Zhao et al., 2021; Falletta et al., 2022). The photogenerated charge carriers can subsequently participate in reactions leading to the formation of reactive oxygen species, which contribute to Rhodamine B degradation (Haghighi et al., 2022; Adhaina et al., 2020; Falletta et al., 2022).

The difference between the visible-light and dark conditions corresponds to an increase of 17.20 percentage points, whereas the difference between the UV and dark conditions is only 1.40 percentage points. This result suggests that, under the experimental conditions employed, visible-light irradiation provided a more favorable condition for Rhodamine B degradation than UV irradiation. However, the observed degradation cannot be attributed solely to photocatalytic activity because substantial dye removal also occurred under dark conditions. Moreover, the result does not by itself establish that the band-gap characteristics of WO₃ are the sole factor responsible for the higher degradation under visible light. Further optical and mechanistic analyses would be required to establish the contribution of light absorption and interfacial charge transfer.

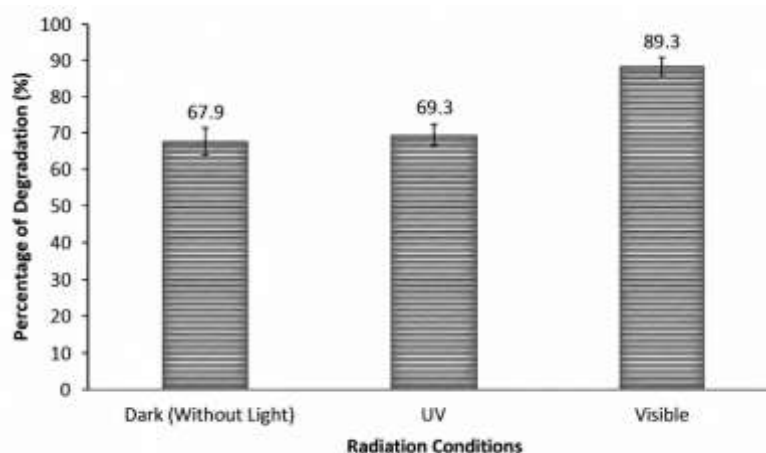


Figure 6. Effect of Dark, UV, and Visible-Light Irradiation Conditions on Rhodamine B Degradation Using the WO₃/TiO₂/SiO₂ Composite

Under visible-light irradiation, SiO₂ primarily provides a support and adsorption surface, whereas TiO₂ acts as a semiconductor. WO₃ can participate in interfacial charge transfer with TiO₂ and may facilitate charge separation, thereby reducing electron-hole recombination. The resulting charge carriers can participate in the formation of reactive oxygen species that contribute to Rhodamine B degradation (Wang et al., 2021; Haghighi et al., 2022; Falletta et al., 2022). The higher degradation observed under visible light in this study indicates that the combined WO₃/TiO₂/SiO₂ system was more effective under the tested visible-light condition than under the tested UV condition; however, the result alone does not establish that the band gap of WO₃ is the sole reason for the difference.

CONCLUSIONS AND RECOMMENDATIONS

The WO₃/TiO₂/SiO₂ composite contained monoclinic WO₃ and anatase TiO₂ phases, while the SiO₂ component exhibited a predominantly amorphous character according to XRD analysis. SEM revealed an irregular and heterogeneous surface morphology with pronounced particle agglomeration. EDX analysis indicated 7.60% WO₃, 21.80% TiO₂, and 64.50% SiO₂, together with minor oxide components consisting of 3.04% NaO and 3.08% Al₂O₃. Because no purification step was documented, the possible contribution of these minor components to the photocatalytic performance cannot be excluded. In the contact-time experiment, 0.15 g of the composite achieved 85.10% Rhodamine B degradation from a 10 ppm solution after 75 min under visible-light irradiation. In the initial-concentration experiment, the highest observed degradation was 86.40% at 10 ppm. Under the irradiation-condition experiment, the degradation percentages were 67.90% under dark conditions, 69.30% under UV irradiation, and 89.30% under visible-light irradiation. These results indicate that, under the experimental conditions investigated, the WO₃/TiO₂/SiO₂ composite showed the highest Rhodamine B removal under visible-light irradiation, although the relatively high degradation under dark conditions indicates that adsorption and other non-photochemical processes also contributed substantially to dye removal.

FURTHER STUDY

This study still has limitations, so further research on the topic of the Photocatalytic Performance of a WO₃/TiO₂/SiO₂ Composite for Rhodamine B Degradation Under Different Light Conditions is needed to improve this research and broaden the understanding for both the author and the readers.

ACKNOWLEDGMENT

This research was supported by the Ministry of Research and Technology and Higher Education of Indonesia and Faculty of Mathematic and Natural Science, Mulawarman University.

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