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Facile synthesis of C–TiO₂–MoS₂ nanocomposite based on commercial TiO₂ nanopowder for photodegradation of methylene blue†

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We present a facile synthesis method for a C–TiO₂–MoS₂-based photocatalytic nanocomposite designed for the efficient degradation of pollutant dyes, specifically methylene blue (MB) and rhodamine B (RhB). Few-layered MoS₂ nanosheets were exfoliated from natural bulk MoS₂ via urea-assisted ball milling. These nanosheets were then mixed with TiO₂ nanopowder and sodium deoxycholate (SDC) surfactant, followed by thermal annealing at 600 °C in argon (Ar) gas. The resulting TiO₂–MoS₂ mixture was found to be coated and interconnected by a carbon layer, which acted as an electron-conducting bridge and simultaneously prevented the aggregation of TiO₂ nanoparticles and MoS₂ nanosheets. The photocatalytic performance of the nanocomposite was evaluated through the degradation of MB and RhB. Remarkably, the nanocomposite exhibited excellent degradation of a high-concentration MB (RhB) solution (10^{−4} M), achieving near-complete degradation within 45 minutes under UV light and 50 minutes under visible light. Furthermore, under UV irradiation, the nanocomposite reached a degradation efficiency of 99.6% with a rate constant of 0.128 min^{−1}, while slightly lower values were observed under visible light. A possible mechanism responsible for the enhanced MB degradation was also proposed. This strategy provides a promising pathway for the development of effective photocatalysts for practical photocatalytic applications.

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1. Introduction

In recent years, various water treatment technologies, including adsorption, photocatalysis, and microwave-assisted catalysis, have been widely studied for removing organic pollutants from wastewater.^{1–3} Adsorption is a cost-effective and simple method with high pollutant removal efficiency, it only transfers contaminants onto solid surfaces, requiring subsequent disposal or regeneration. However, there are still several obstacles, including limited adsorption capacity, ready

inactivity, secondary pollutants, and high energy consumption.⁴ Microwave-assisted catalysis, a newer technique, enhances reaction rates through rapid and uniform heating at the molecular level. It offers reduced processing time and energy input, but the stability of catalysts under microwave conditions and scalability remain concerns.⁴ Thus, these considerations support the need for developing efficient and sustainable photodegradation systems. Photocatalytic degradation of organic contaminants has emerged as an environmentally sustainable approach for wastewater treatment.^{5,6} Titanium dioxide (TiO₂), available in various nanostructured morphologies, has gained significant attention as a promising photocatalyst for decomposing harmful dyes due to its high photocatalytic efficiency, excellent photostability, and non-toxic nature. Additionally, TiO₂ nanoparticles are readily available on the market. However, two primary limitations hinder its practical application in photocatalysis: (1) its absorption is limited to the ultraviolet region, which accounts for only about 4% of the solar spectrum, and (2) the rapid recombination of photogenerated charge carriers significantly reduces its photocatalytic efficiency.

To overcome these issues, many research groups have developed many strategies to improve further the photocatalytic

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performance of TiO_2 , such as doping with non-metallic ions, metal ions,^{7–10} depositing noble metals.^{11–13} Recently, a heterostructure formation of TiO_2 with carbonaceous nanomaterials or narrow bandgap-semiconductors is an effective approach. Numerous previous studies have reported that combining TiO_2 with carbonaceous nanomaterials, for instance, carbon nanotubes,^{14,15} C60,^{16,17} graphene oxide,^{18–20} and carbon dots^{21,22} can enhance the light absorption of TiO_2 across the entire visible spectrum, compared to bare TiO_2 . The enhanced light absorption was ascribed to the formation of chemical bonds (Ti–O–C) between TiO_2 and the carbon-based materials.²³ Crucially, carbonaceous materials act as effective electron acceptors, facilitating the transfer of electrons from the photogenerated electron–hole pairs in TiO_2 , thereby enhancing charge separation efficiency.^{24,25} However, the low visible light utilization still restricts their wide applications in environmental management.

Different from carbonaceous materials, molybdenum sulfide (MoS_2), a member of transition metal dichalcogenides (TMDCs), is a semiconductor with a narrow indirect bandgap of about 1.3 eV in bulk, while MoS_2 monolayer possesses a large direct bandgap of 1.9 eV.^{26,27} This allows MoS_2 in the nanocomposite acts as an effective photo-absorber over the solar spectrum. Moreover, the establishment of a type-II band alignment between MoS_2 and TiO_2 promotes efficient separation of photoinduced electron–hole pairs. Consequently, $\text{TiO}_2/\text{MoS}_2$ composites have garnered growing attention for a wide range of catalytic applications. For instance, Wang *et al.* developed a $\text{MoS}_2/\text{TiO}_2$ (P25) composite photocatalyst that demonstrated enhanced photocatalytic performance in degrading methylene blue (MB) and rhodamine B (RhB) under simulated sunlight.²⁸ By using a two-step hydrothermal process, Chandrasekhar *et al.* created $\text{MoS}_2/\text{TiO}_2$ heterostructure nanocomposites, which demonstrated high photocatalytic activity in textile dye contaminants.²⁹ Tang *et al.* used a straightforward and scalable one-step sol–gel process to successfully create brown- TiO_2 @ MoS_2 heterostructures using 2D MoS_2 nanosheets exfoliated from natural bulk molybdenite. Under visible light irradiation, the maximum degradation efficiency of 94.80% and the maximum degradation rate of 0.01764 min^{-1} were obtained using the heterostructure catalyst. According to experimental findings, as compared to pure brown- TiO_2 , the creation of brown- TiO_2 and 2D MoS_2 heterostructures can improve visible light absorption, extra active sites, and the separation and transfer of photogenerated charge.³⁰

Recently, Nguyen *et al.* synthesized a Z-scheme C- $\text{MoS}_2/\text{TiO}_2$ heterostructure *via* a hydrothermal method, utilizing a carbon layer as a connecting bridge. The resulting catalyst achieved 99% degradation of methylene blue within 60 minutes under visible light irradiation.³¹ Ali *et al.* reported a simple one-step solvothermal synthesis of a C- TiO_2 - MoS_2 heterostructure with visible light activity, utilizing a novel MoS_2 cluster compound using a novel MoS_2 cluster compound $[(\text{NH}_4)_2 \text{Mo}_3\text{S}_{13} \cdot 2\text{H}_2\text{O}]$ as the precursor for the synthesis of MoS_2 . The composite demonstrates superior photocatalytic activity under visible light for the degradation of RhB and 4-ATP compared to pure TiO_2 . However, most MoS_2 samples used in these studies were

synthesized from costly molybdenum and sulfur compounds, which require strict reaction conditions.³²

Inspired by the above research, this work aims to develop new photocatalyst material based on C- TiO_2 - MoS_2 nanocomposite. The proposed nanocomposite was prepared using a simple method by mixing TiO_2 and lab-made MoS_2 nanosheets with SDC solution and followed by the thermal annealing process at 600 °C in Ar gas. The resulting microstructure of the nanocomposite exhibited several key advantages for photocatalytic activity, including enhanced charge carrier separation and reduced aggregation of TiO_2 and MoS_2 . The nanocomposite demonstrated effective photodegradation of high-concentration solutions of the pollutant dyes under both UV and visible light irradiation.

2. Experiment

2.1 The preparation of MoS_2 nanosheets

The exfoliation of bulk MoS_2 (99.5% with a lateral size of 2–3 μm , purchased from Macklin) was carried out by using the ball milling method. First, a powder mixture consisting of bulk MoS_2 and urea (in a weight ratio of 1 : 30) was weighed and added to the milling jars. Next, a specific amount of ZrO_2 milling balls with various diameters is added to the jars. In this experiment, the weight ratio of the powder mixture to the ZrO_2 balls was maintained at 1 : 30. The grinding process was then conducted at a rotational speed of 400 rpm for 20 hours to facilitate exfoliation. After grinding, the resulting mixture was dissolved in deionized (DI) water and ultrasonicated for 30 min to achieve a uniform solution. The solution was then filtered several times using vacuum filtration to remove urea residuals. Finally, the cleaned MoS_2 nanosheets were dispersed in DI water or other suitable solvents (Fig. S1†).

2.2 Preparation of C- TiO_2 - MoS_2 nanocomposite

TiO_2 nanopowder (99.9%, anatase phase with average size $\sim 25 \text{ nm}$) was sourced from Nanoshel. To prepare the mixture, 10 g of TiO_2 was dispersed in 300 mL of ethanol. Next, 100 mg of MoS_2 nanosheets was then added to the TiO_2 suspension. In this work, a weight ratio of MoS_2 and TiO_2 (1% w/w) was adopted from previous work.³⁰ The mixture was treated by ultrasonication for 60 min, followed by magnetic stirring for 30 minutes. The TiO_2 - MoS_2 mixture was then obtained by employing a vacuum pneumatic system to dry the solution at 80 °C. Next, the TiO_2 - MoS_2 mixture was immersed in ethanol with SDC surfactant and magnetically stirred for 60 min. The sample was then transferred to the vacuum pneumatic system for drying. Finally, the sample was annealed at 600 °C in a flow of Ar gas for 30 min to facilitate the carbonization of the SDC surfactant to obtain C- TiO_2 - MoS_2 composite, as shown in Fig. S2†

2.3 Characterization methods

The morphologies of the as-prepared samples were examined by a field-emission scanning electron microscope (FE-SEM, Hitachi S-4800) at an operating voltage of 5 kV. High-resolution

transmission electron microscopy (HRTEM) analysis of samples were acquired by a JEOL JEM2100F microscope. The thickness of as-exfoliated MoS_2 was measured by an atomic force microscope (AFM, XE-100 from Park Systems). The crystallinity of the as-exfoliated sample was investigated by X-ray diffraction (XRD, D8 Advance, Bruker, Germany). Raman spectroscopy with an excitation wavelength of 532 nm was used to confirm the formation of the as-prepared samples. The distribution of elements in the nanocomposite was measured by energy dispersive spectroscopy (EDS) mapping (Ultim Max 65& JSM – IT800SHL). N_2 adsorption–desorption isotherms were analyzed using the BET method (TriStar II 3020, USA), while the pore volume distribution was determined from the adsorption branch of the isotherms based on the Barrett–Joyner–Halenda (BJH) model.

2.4 Photocatalytic activity measurement of the samples

The photocatalytic performance of the as-synthesized samples was assessed through the degradation of MB and RhB in aqueous solutions under UV light ($\lambda = 365$ nm, 3 W) and visible light (Xenon lamp, $\lambda = 450$ nm, 3 W). In every experiment, 20 mg of the sample was dispersed in 50 mL of an aqueous methylene blue (MB) solution ($100 \text{ mg L}^{-1} \sim 10^{-4} \text{ M}$), and the suspension was magnetically stirred in the dark for 30 minutes to achieve adsorption–desorption equilibrium of MB molecules on the catalyst surface. Subsequently, the suspension was exposed to UV and visible irradiation individually. The distance between

the light source and the surface of the solution was 25 cm. The photodegradation of the dyes was monitored at 5 minute intervals. After each interval, the catalyst was removed by centrifugation, and the dye concentration in the solution was determined by measuring the absorbance at 664 nm for MB and 553 nm for RhB using a UV-vis spectrophotometer (Shimadzu UV-vis (UV – 2450)).

3. Results and discussion

3.1 Characterization of as-exfoliated MoS_2 nanosheets

Fig. 1a shows the SEM image of bulk MoS_2 , which has a stacked structure of multiple layers. After exfoliation, the bulk MoS_2 was converted into nanosheets as shown in Fig. 1b. To confirm, the AFM image revealed MoS_2 flakes with a typical height of ~ 3.7 nm, indicating few-layer MoS_2 (Fig. 1c). The Raman spectra of bulk and as-exfoliated MoS_2 , presented in Fig. 1d, show two key peaks (named E_{2g} and A_{1g}), linked to the in-plane vibrations of Mo and S atoms and the out-of-plane vibrations of S atoms, respectively. Notably, the distance between these peaks (Δk) may be utilized to roughly estimate the number of layers of MoS_2 . Fig. 1d indicates that Δk (22.8 cm^{-1}) of the as-exfoliated MoS_2 is lower compared to that of bulk MoS_2 (25.6 cm^{-1}), suggesting the presence of few-layer structures. Fig. 1e displays the XRD patterns for both bulk and exfoliated MoS_2 . The bulk MoS_2 (black curve) presents a distinctive diffraction pattern with peaks at $2\theta = 14.4^\circ$ (002), 29° (004), 32.7° (100), 33.4° (101), 35.8° (102), 39.6° (103), 44.2° (006), 49.8° (105), 58.4° (110), and

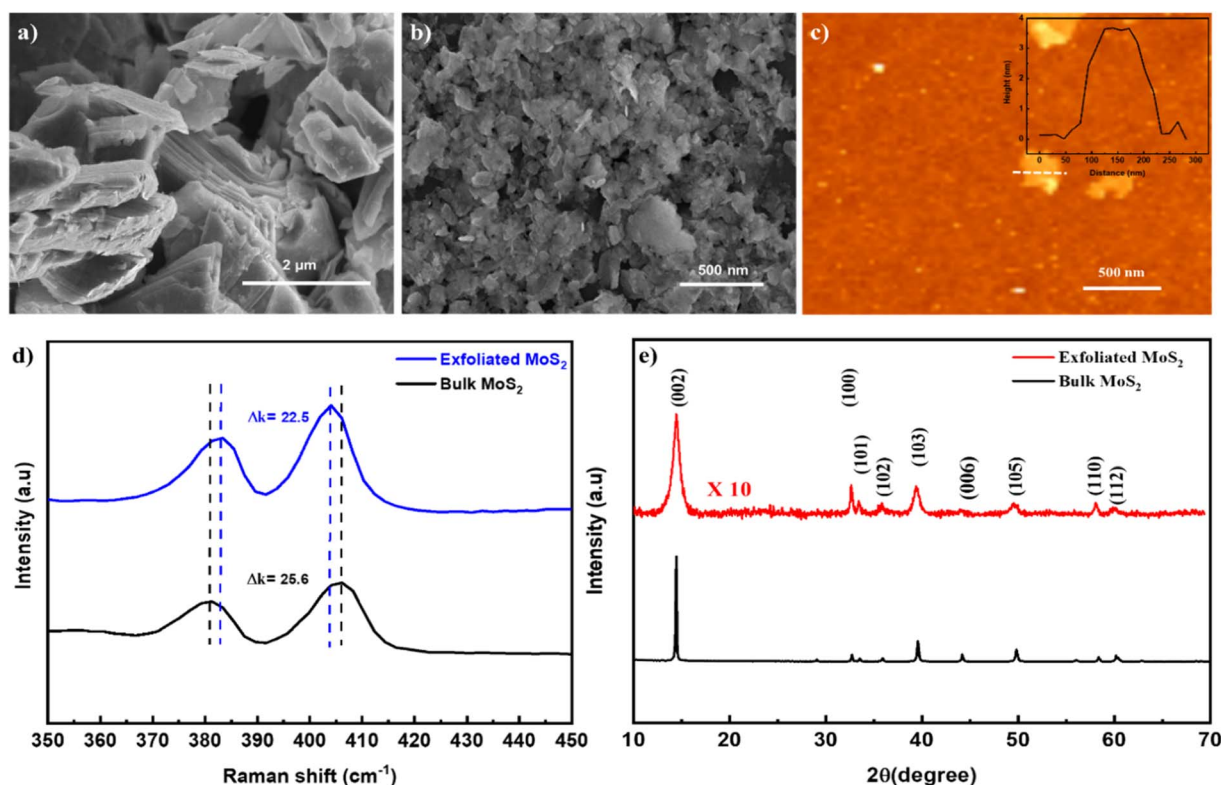


Fig. 1 (a) SEM image of bulk MoS_2 , (b) SEM image of as-exfoliated MoS_2 nanosheets, (c–e) AFM image, Raman spectra, and XRD patterns of bulk MoS_2 and as-exfoliated MoS_2 . Inset is the height profile of as-exfoliated MoS_2 in (c).



60.3° (112) (JCPDF #96-900-7661). As bulk MoS₂ was thinned-down to nanosheets, there was a noticeable decrease in the intensity of the peaks, along with a corresponding peak broadening, as illustrated by the red curve in Fig. 1e. The broadening and the reduction in intensity of the peak at 14.4° (002) serve as definitive indicators of the presence of thin MoS₂ nanosheets. Furthermore, the exfoliated MoS₂ nanosheets also demonstrated excellent dispersibility in various solvents, including deionized water, isopropanol, and ethanol (Fig. S1†). This enhanced dispersibility was attributed to the formation of NH₂ groups at the edges of the nanosheets, as confirmed by FTIR analysis (Fig. S1†).

3.2 Characterizations of as-prepared C-TiO₂-MoS₂ nanocomposite

The morphology of the C-TiO₂-MoS₂ nanocomposite was illustrated in Fig. 2a. However, it was difficult to distinguish TiO₂ nanoparticles and MoS₂ nanosheets due to their sizes being tiny. For clear observation, HR-TEM measurement was conducted and revealed in Fig. 2b. The HRTEM image showed that the TiO₂-MoS₂ mixture was covered and linked to each other by a carbon layer. Fig. 2b exhibits that C-TiO₂-MoS₂ nanocomposites with the lattice spacing of 0.352 nm corresponds to the (101) plane of TiO₂, while the lattice spacing of 0.62 nm is assigned to the (002) plane of MoS₂, respectively.

XRD analysis was performed to determine the crystal structure and phase composition of the synthesized samples. As shown in Fig. 2c, the XRD pattern of TiO₂ (red curve) exhibits distinct diffraction peaks at 25.3° (101), 37.8° (004), and 48.0° (200), which are characteristic of anatase TiO₂ (JCPDS #97-002-4276). The pattern of exfoliated MoS₂ is also included for comparison. In the case of the C-TiO₂-MoS₂ nanocomposites, the characteristic peaks corresponding to both anatase TiO₂ and exfoliated MoS₂ were clearly observed, while diffraction peaks of carbon layer could not be detected. In addition, weak diffraction peaks corresponding to rutile TiO₂ were detected, which can be attributed to the partial phase transformation of anatase to rutile induced by thermal annealing at 600 °C. To further confirm the formation of the C-TiO₂-MoS₂ nanocomposite, Raman measurements also were conducted. As shown in Fig. 2d, we can identify Raman active modes for the anatase phase of TiO₂ (black curve) at 145.1 cm⁻¹ (E_g), 400 cm⁻¹ (B_{1g}), 517.3 cm⁻¹ (A_{1g}), and 638.0 cm⁻¹ (E_g), as well as the prominent E_{1g} and A_{1g} peaks of thin MoS₂, located at 381.6 cm⁻¹ and 405.4 cm⁻¹, respectively. Additionally, the C-TiO₂-MoS₂ nanocomposite exhibited a Raman peak at 448.3 cm⁻¹, characteristic of the rutile phase of TiO₂, which is attributed to thermal annealing at 600 °C.³³ Additionally, peaks at 1346 cm⁻¹ and 1597 cm⁻¹ correspond to the D and G bands of the carbon layer (red curve) were observed.³⁴ These results demonstrated the coexistence of TiO₂, carbon, and MoS₂ within the C-TiO₂-MoS₂ nanocomposite.

The EDS mapping of the C-TiO₂-MoS₂ nanocomposites showed that the distribution of the carbon (C), titanium (Ti), oxygen (O), molybdenum (Mo), and sulfur (S) elements was highly uniform (Fig. 2e).

The N₂ adsorption-desorption isotherms and pore size distribution curves of MoS₂, TiO₂, and C-TiO₂-MoS₂ were shown in Fig. S3 (ESI†). As shown in Fig. S3,† all the samples exhibited typical type IV isotherm features and H3 hysteresis loops, confirming the presence of mesoporous structures.³⁵ Notably, the C-TiO₂-MoS₂ nanocomposite demonstrated the significantly higher BET surface area of 62.48 m² g⁻¹ than that of bare TiO₂ (16.3 m² g⁻¹) and MoS₂ nanosheets (6.7 m² g⁻¹). The pore volumes are 0.8, 0.36, and 0.018 cm³ g⁻¹ for C-TiO₂-MoS₂ nanocomposite, TiO₂ nanopowder, and MoS₂ nanosheets, respectively. These results confirm that the C-TiO₂-MoS₂ nanocomposite has a more developed mesoporous network, which is advantageous for applications requiring high surface area and accessible porosity.

3.3 Evaluation of photocatalytic performance

The photocatalytic performance of the samples was evaluated by monitoring the decomposition of MB under UV irradiation, focusing on the absorption peak at 664 nm. The typical absorption peak intensity at 664 nm decreased with prolonged irradiation. Fig. 3a-e illustrates the time-dependent absorption spectra of MB degradation by bare TiO₂, bare MoS₂, C-TiO₂, TiO₂-MoS₂, and C-TiO₂-MoS₂ nanocomposite, respectively. As the irradiation time increased, the intensity of the absorption peak at 664 nm speedily diminished, and nearly vanished after 120 minutes for bare MoS₂ and bare TiO₂ (Fig. S4 in ESI†). Compared to only UV without catalyst (Fig. S4c†), bare MoS₂, TiO₂, and C-TiO₂, TiO₂-MoS₂ composite showed a faster degradation of MB after 45 min. In C-TiO₂-MoS₂ nanocomposite case, MB molecules can be degraded completely within 45 min.

The degradation percentage and degradation rate of MB are two important parameters to estimate the photocatalytic efficiency of the photocatalyst. The decline in intensity of the distinctive absorption peak at 664 nm was used to calculate the degradation percentage.

$$D(\%) = \frac{C_0 - C_t}{C_0} \times 100$$

where, C₀ and C_t are the concentrations of MB at time 0 and *t* (min), respectively. Where *t* is the irradiation time. As shown in Fig. 3f, degradation percentage (*D*%) of the samples was calculated to be 33.8%, 35.8%, 43.7%, 88.9%, and 99.6% for bare MoS₂, bare TiO₂, C-TiO₂, TiO₂-MoS₂, and C-TiO₂-MoS₂ nanocomposite after 45 min under UV irradiation, respectively.

To determine the degradation rate of MB on the samples, a pseudo-first-order kinetic model was used in this study. The rate of degradation (*k*) of MB was derived from the slope of the linear transform ln (C_t/C₀), where C_t and C₀ are the intensity values of the peak at 664 nm at time *t* and 0, respectively. As shown in Fig. 3g, the C-TiO₂-MoS₂ nanocomposite shows the highest *k* value (0.128 min⁻¹), followed by TiO₂-MoS₂ (0.05 min⁻¹), C-TiO₂ (0.013 min⁻¹), bare TiO₂ (0.011 min⁻¹), and MoS₂ (0.0096 min⁻¹). The obtained results showed that the photodegradation efficiencies of the TiO₂-MoS₂ and C-TiO₂-MoS₂ samples are significantly higher than those of bare TiO₂



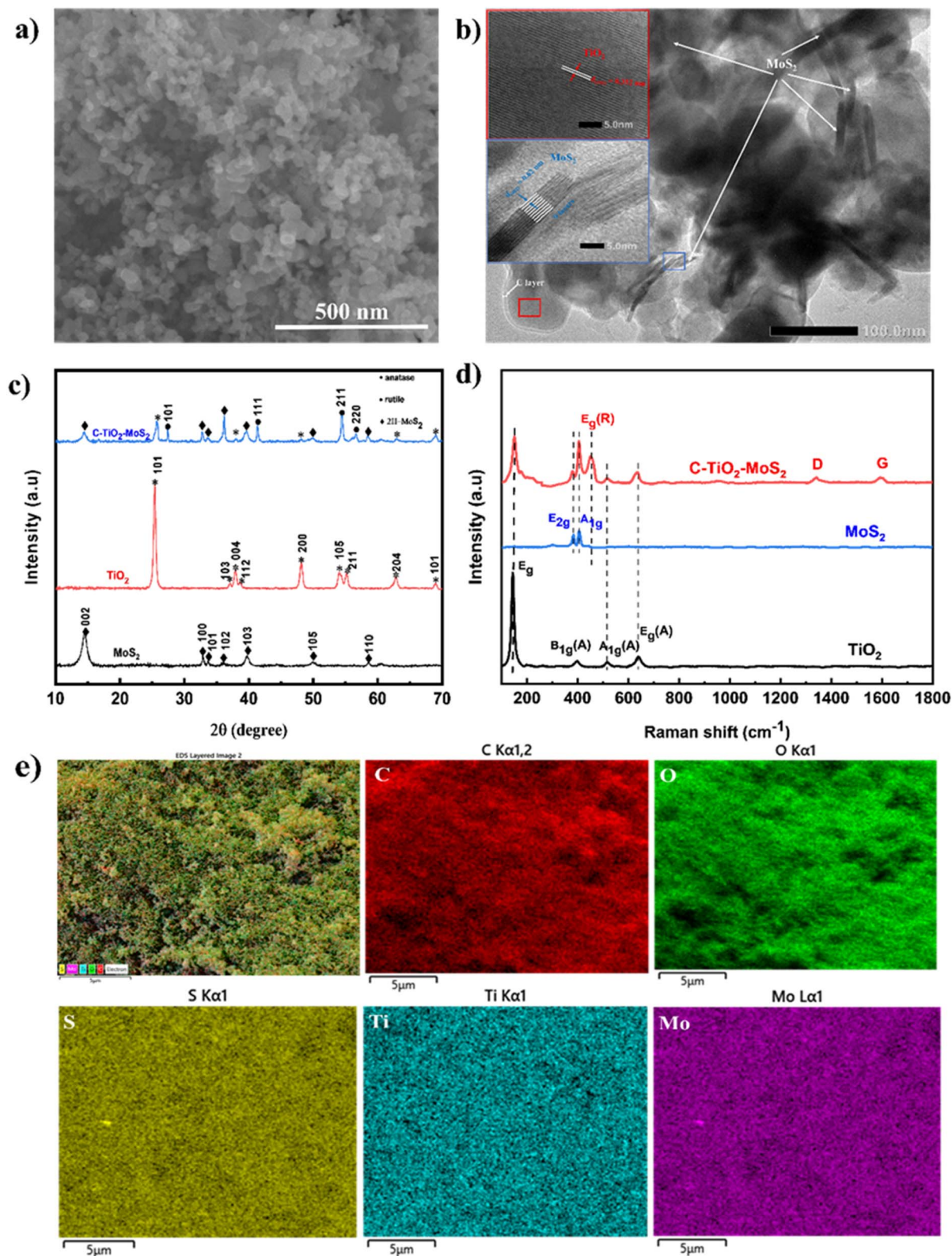


Fig. 2 (a) SEM, (b) HRTEM image, (c) XRD pattern, (d) Raman spectra, and (e) EDS elemental mapping of C-TiO₂-MoS₂ nanocomposite.

and bare MoS₂, C-TiO₂. It was evident that the combination of TiO₂-MoS₂ can provide more active sites and enhance the separation of photogenerated charge carriers due to the

formation of type II band alignment between MoS₂ and TiO₂. Moreover, the introduction of the outer carbon layer created an effective medium for the transport of charge carriers, thereby



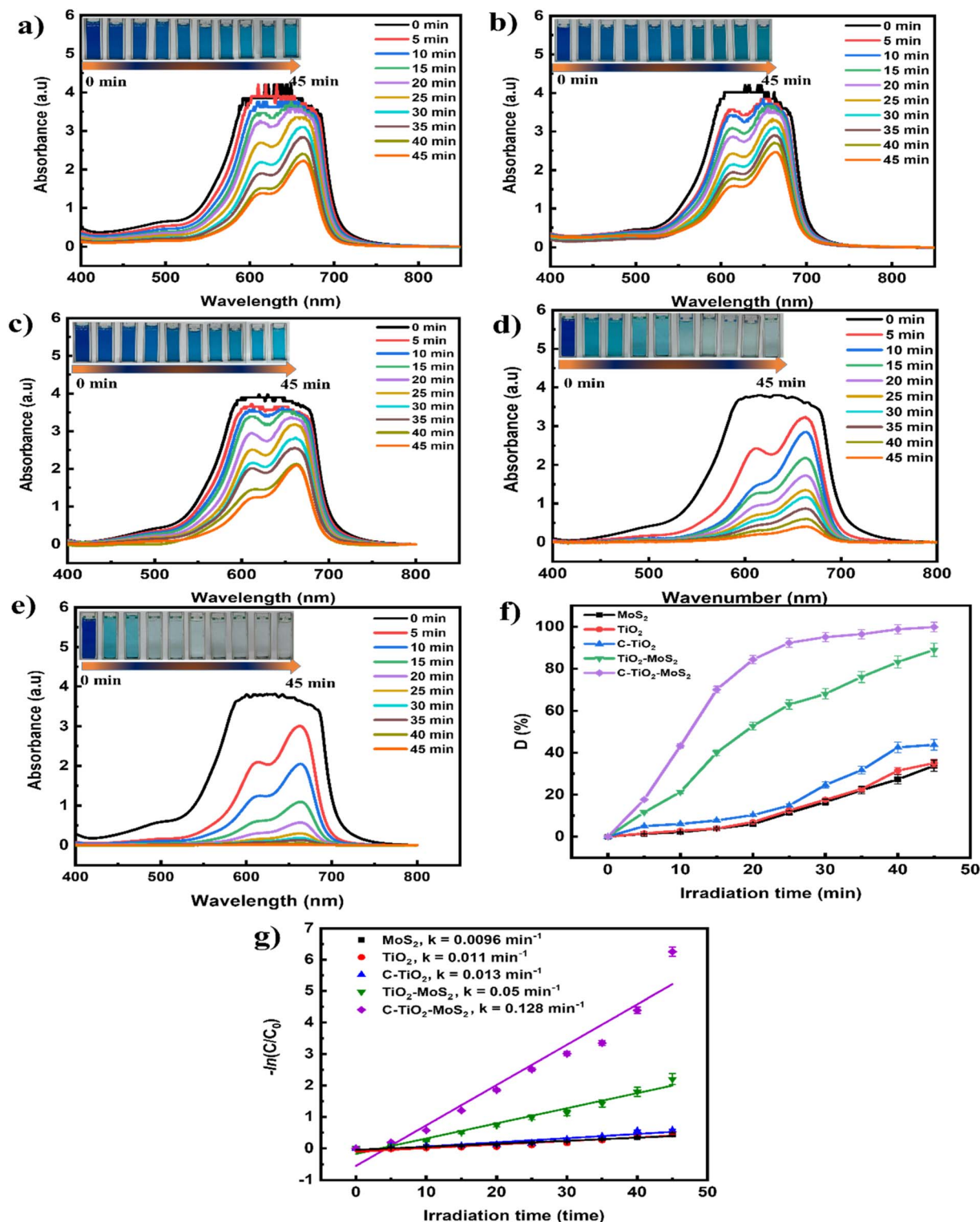


Fig. 3 Time-dependent UV-vis absorption spectra of MB (10^{-4} M) degradation in the presence of (a) bare TiO₂, (b) bare MoS₂, (c) C-TiO₂, (d) TiO₂-MoS₂ and (e) C-TiO₂-MoS₂ under UV irradiation. (f) and (g) are MB degradation percentage and degradation rate of the samples, respectively.

improving the photocatalytic activity of C-TiO₂-MoS₂ nanocomposite. More discussion will be presented in the mechanism section.

The stability of the C-TiO₂-MoS₂ nanocomposite as a photocatalyst under UV irradiation was also thoroughly assessed over five cycles as seen in Fig. 4. The C-TiO₂-MoS₂ catalyst

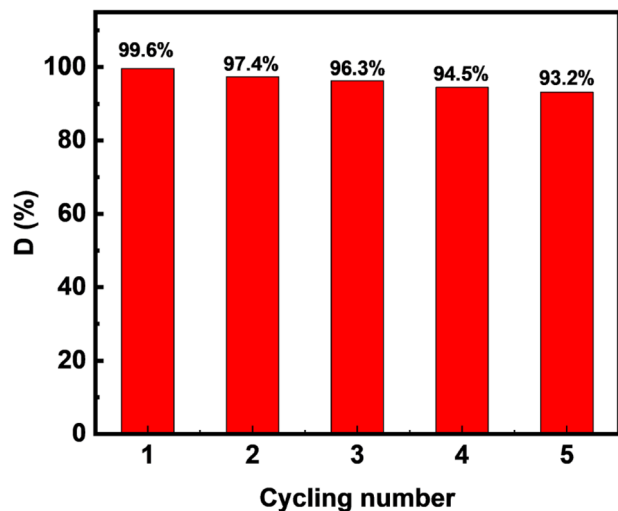


Fig. 4 Five consecutive cycles for the MB photodegradation using C-TiO₂-MoS₂ nanocomposite.

showed degradation percentages of approximately 99.6%, 97.4%, 96.3%, 94.5%, and 93.2% for MB molecules over the 1st to 5th cycles, respectively. After five cycles, the value of 93.2% was retained, indicating the good stability of C-TiO₂-MoS₂ nanocomposite.

The photocatalytic performance of the nanocomposite was also systematically evaluated by varying the catalyst dosage,

initial pH. When the amount of catalyst was increased from 10 mg to 20 mg/50 mL of MB solution, the degradation efficiency improved as shown in Fig. 5. This is due to the increased number of active sites. However, increasing the nanocomposite loading to 30 mg resulted in a plateau in photocatalytic efficiency, which is attributed to excessive turbidity that hindered light penetration and limited the activation of deeper catalyst layers.

Fig. 6 shows that the initial pH of the solution notably influenced the photocatalytic efficiency, which is attributed to favorable electrostatic interactions between the photocatalyst surface and pollutant molecules. Under acidic condition (pH = 5), the photocatalytic efficiency was decreased while that increased at basic condition (pH = 11), due to catalyst surface charge repulsion or inhibition of reactive species generation.

For real applications, the C-MoS₂-TiO₂ nanocomposite was also expected to achieve a high degradation efficiency under visible light. As shown in Fig. 7, the MB solution was mostly degraded after 50 min, achieving a degradation efficiency of 99.4% with a corresponding rate constant of $k = 0.091 \text{ min}^{-1}$. These values are relatively close to those of the nanocomposite under UV irradiation. This implies that the nanocomposite can operate with high efficiency under both UV and visible light. We made a summary of the photodegradation efficiency of our samples and those of previously reported TiO₂-based composites and MoS₂-based composites, as shown in Table 1. It should be noted that direct quantitative comparisons are challenging,

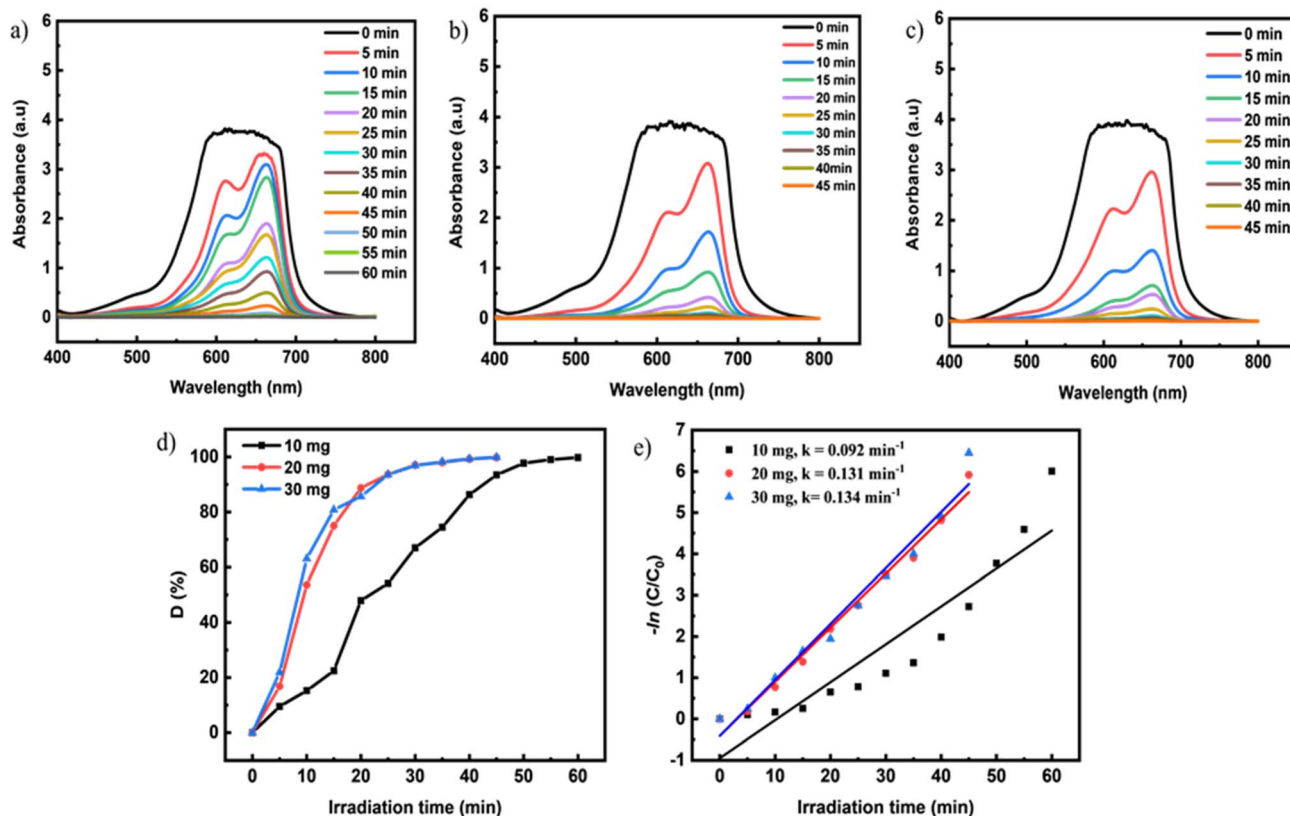


Fig. 5 Time-dependent UV-vis absorbance spectra of MB degradation with varying the amount of the nanocomposite (10, 20, and 30 mg) (a–c). Photodegradation efficiency and constant are presented in (d) and (e), respectively.



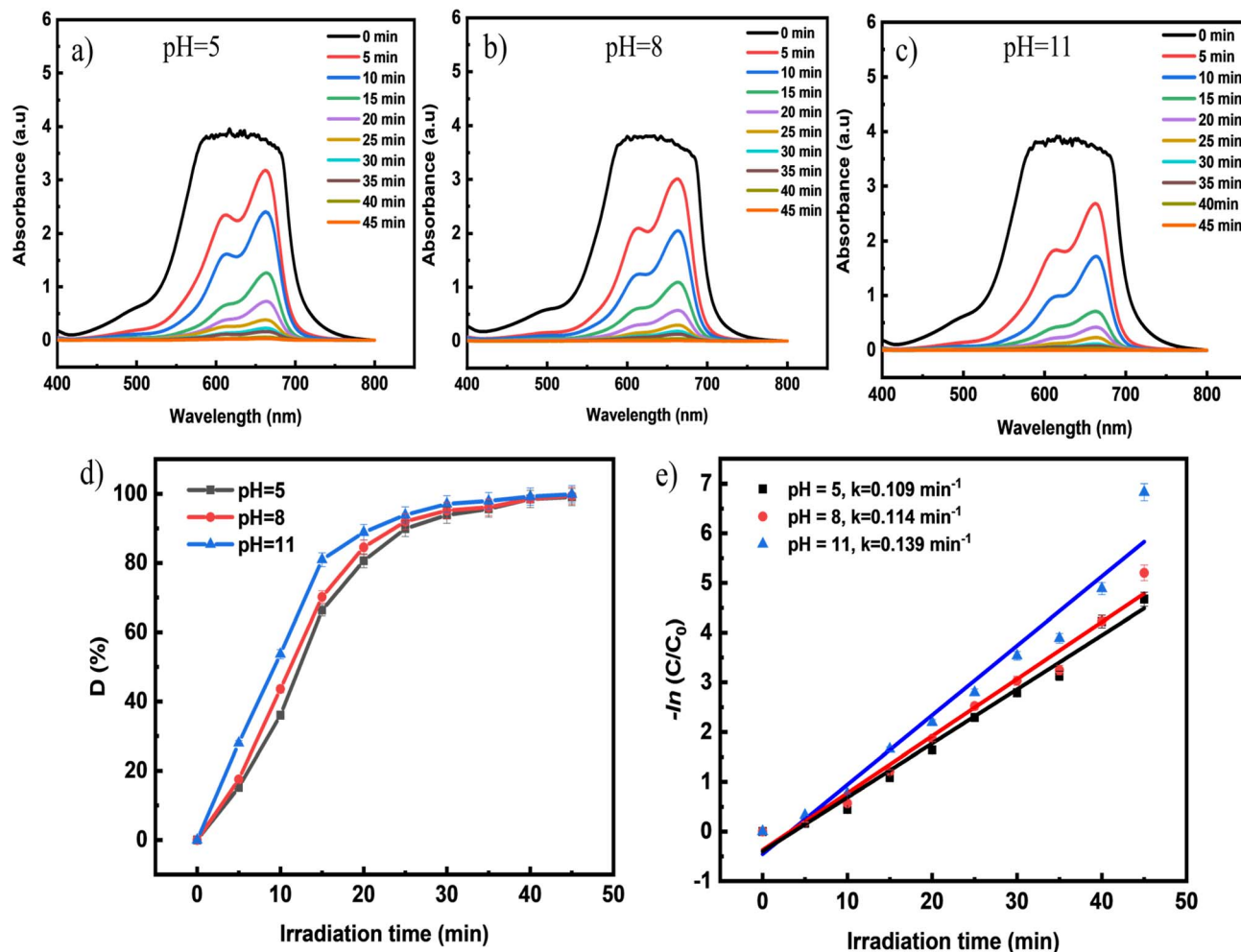


Fig. 6 The effect of initial pH value on photodegradation efficiency. (a–c) Time-dependent UV-vis absorbance spectra of MB degradation with varying the pH of MB solution (5, 8, and 11), respectively. (d and e) Are photodegradation efficiency and constant are presented, respectively.

as experimental conditions such as dye concentration and light source intensity vary considerably among different studies. In this study, thus, we emphasize the ability of the synthesized photocatalyst to effectively degrade methylene blue at significantly higher concentrations (10^{-4} M) than those typically reported (often $\sim 10^{-5} \text{ M}$). This demonstrates the robustness and high catalytic capacity of the material under more practical and challenging conditions. While a full comparative study under identical experimental settings would provide further validation, the current findings highlight the uniqueness of our system in handling high dye concentration, which is relevant for real wastewater treatment applications.

To check photocatalytic activity of the nanocomposite with another pollutant, the C-TiO₂-MoS₂ nanocomposite was further applied to the decomposition of RhB under the same conditions. The same trends were recorded and shown in Fig. S6.†

We try to explain the mechanism behind the enhanced degradation efficiency of the pollutant dyes using our sample under both UV and visible light, we propose the following explanation, illustrated in Fig. 8a. Under UV irradiation, where

the photon energy exceeds the bandgap energies of both MoS₂ and TiO₂, electron-hole pairs are generated in both semiconductors. A significant fraction of the photogenerated electrons from the conduction bands (CB) of MoS₂ and TiO₂ are rapidly transferred to the carbon layer, which acts as a conductive pathway. This facilitates electron migration and enhances their interaction with surface-adsorbed oxygen molecules. Simultaneously, some electrons from the CB of MoS₂ transfer to the CB of TiO₂ due to the formation of type-II band alignment. This transfer minimizes electron-hole recombination. The photoinduced holes interact with water molecules or hydroxyl ions on the catalyst surface, leading to the generation of hydroxyl radicals ($\cdot\text{OH}$), while the photogenerated electrons reduce molecular oxygen (O₂) to form superoxide radicals ($\cdot\text{O}_2^-$). Consequently, both $\cdot\text{OH}$ and $\cdot\text{O}_2^-$ radicals decompose pollutant dyes into carbon dioxide (CO₂) and H₂O and intermediates. Under visible light irradiation, electron-hole pairs are primarily generated in the MoS₂, while minor electrons are produced in the TiO₂ due to the carbon impurity level.⁴¹ This explains why the photodegradation efficiency of the nanocomposite catalyst under visible light is slightly lower than that

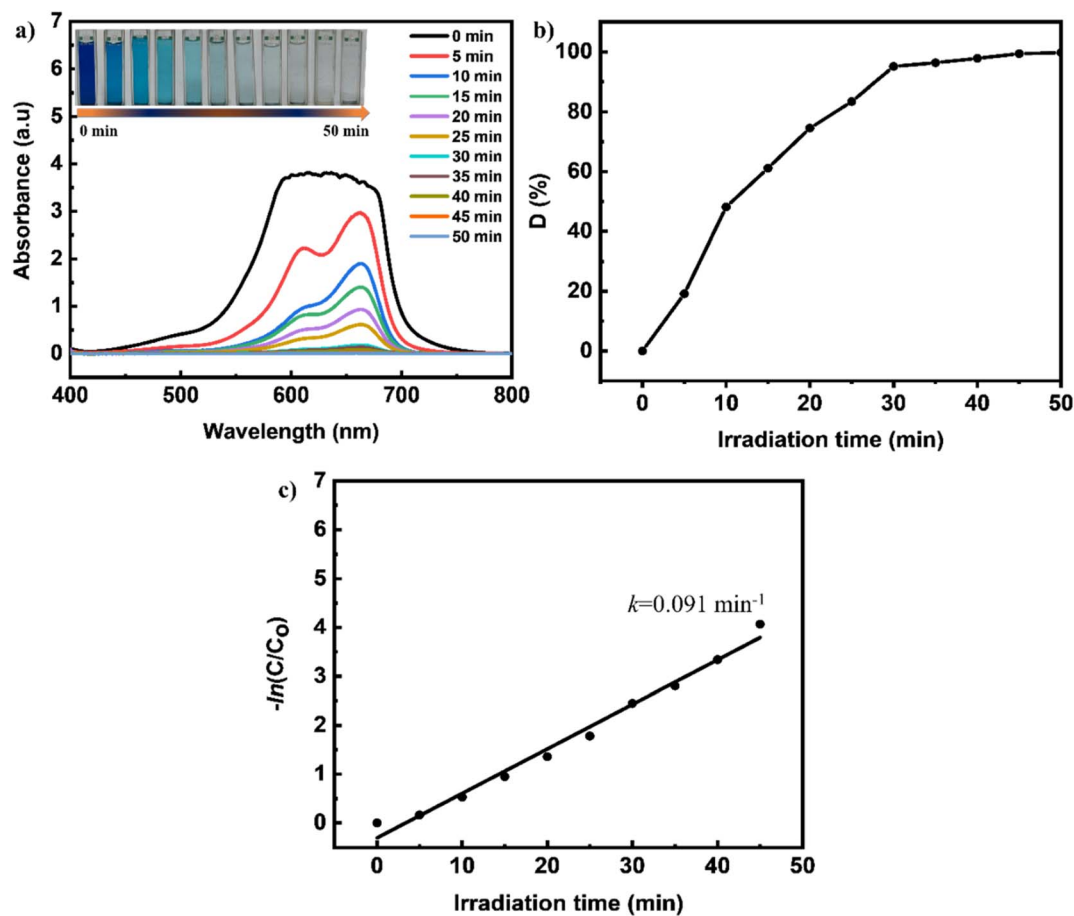


Fig. 7 (a) Time-dependent UV-vis absorption spectra of 10^{-4} M MB degradation in the presence of C-TiO₂-MoS₂ under visible light, (b) MB degradation percentage and (c) degradation rate, respectively.

Table 1 Comparison table of photocatalytic activities of catalysts based on MoS₂ and TiO₂

Photocatalyst	Dyes	Concentration (mg L ⁻¹)	Catalyst loading (g L ⁻¹)	Light source	Time (min)	Degradation efficiency (%)	Ref.
MoS ₂	MB	20	0.15	200 W Xe lamp [$\lambda > 420$ nm]	120	5.9	36
TiO ₂	RhB	5	2	500 W Xe lamp [$\lambda > 420$ nm]	180	19	37
C-TiO ₂	MB	5	0.1	Philips household CFL, 85W, visible light	120	94	21
MoS ₂ /TiO ₂	MB	10	1	500 W, Xenon lamp	90	93.8	30
MoS ₂ /TiO ₂ (P25)	MB	8	0.35	300 W, Tungsten lamp, ($\lambda = 400$ –660 nm)	240	32	29
MoS ₂ /TiO ₂	RhB	10	—	230 W UV lamp, [$\lambda = 365$ nm]	150	85.3	38
MoS ₂ /TiO ₂	MB	20	0.33	UV lamp ($\lambda = 360$ nm)	65	100	39
MoS ₂ /graphene/TiO ₂ (P25)	RhB	10	0.5	150 W solar simulator	80	80	40
C-MoS ₂ /TiO ₂	MB	100	0.2	150 W Xe lamp [$\lambda = 300$ –1900 nm]	60	99	31
MoS ₂	MB	100	0.2	3 W, UV lamp ($\lambda = 365$ nm)	120	94.7	This work
TiO ₂	MB	100	0.2	3 W, UV lamp ($\lambda = 365$ nm)	120	92.8	
C-TiO ₂ -MoS ₂	MB	100	0.2	3 W, UV lamp ($\lambda = 365$ nm)	45	99.6	
				3 W, Xenon lamp ($\lambda = 450$ nm)	50	99.4	
				3 W, UV lamp ($\lambda = 365$ nm)	45	99.5	
	RhB	100	0.2	3 W, Xenon lamp ($\lambda = 450$ nm)	50	99.1	



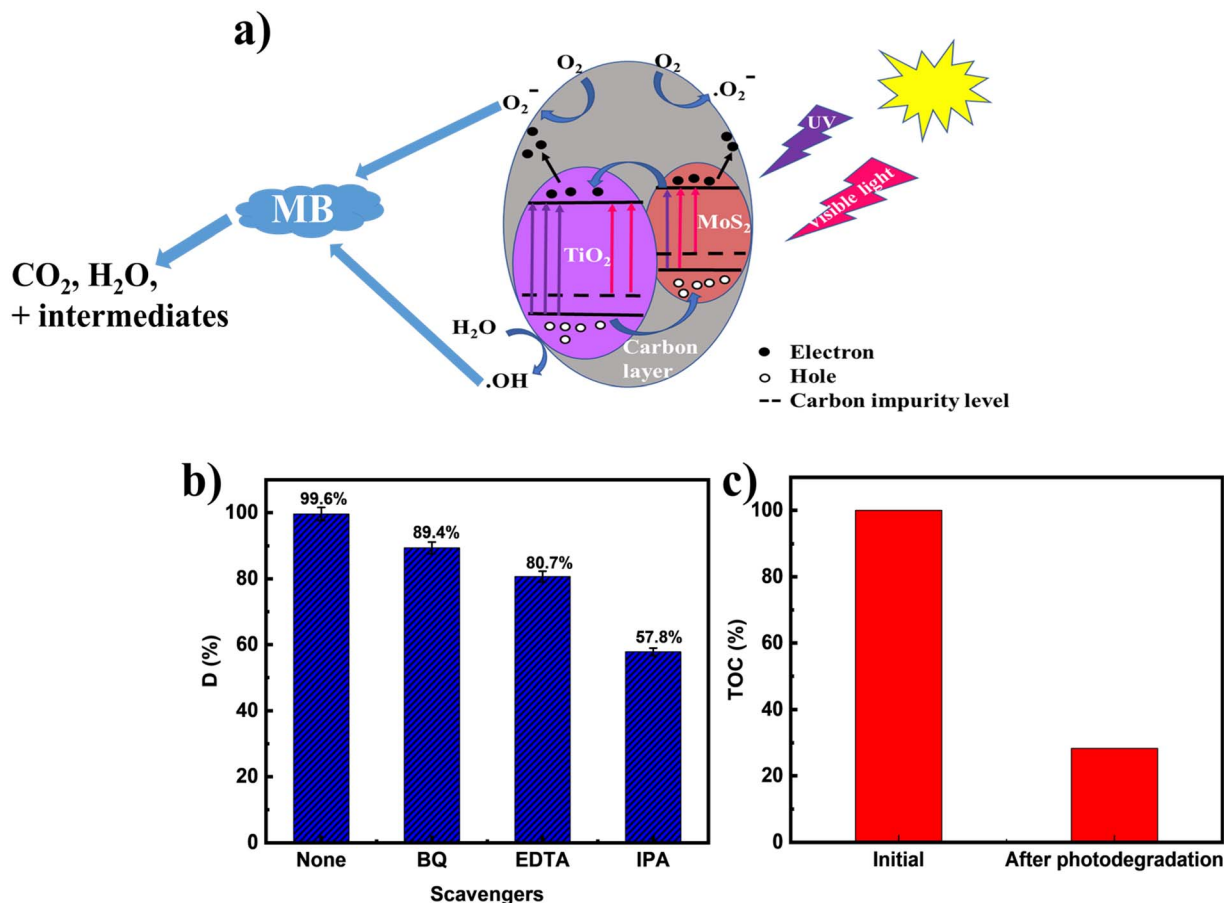


Fig. 8 (a) A proposed mechanism for the degradation of the pollutant dyes by the prepared nanocomposites under light irradiation, (b) reactive species trapping tests, (c) removal of TOC (%) before and after photodegradation.

under UV light. To assess the key roles of the active species involved in the photocatalytic reaction, three distinct scavengers—namely isopropyl alcohol (IPA), ethylenediaminetetraacetic acid disodium salt (EDTA-2Na), and benzoquinone (BQ)—were introduced into the reaction system. These agents were employed to selectively quench hydroxyl radicals ($\cdot\text{OH}$), photogenerated holes (h^+), and superoxide radicals ($\cdot\text{O}_2^-$), respectively.^{42,43} As illustrated in Fig. 8b, the degradation efficiency of methylene blue (MB) under UV irradiation decreased from 99.6% to 89.4%, 80.7%, and 57.8% in the presence of BQ, EDTA-2Na, and IPA, respectively. This result indicates that hydroxyl radicals ($\cdot\text{OH}$) are the dominant reactive species responsible for MB degradation, whereas holes (h^+) and superoxide radicals ($\cdot\text{O}_2^-$) contribute to a lesser extent. In addition, the total organic carbon (TOC) analysis was also conducted to evaluate the extent of methylene blue (MB) degradation and its conversion into inorganic end-products. As shown in Fig. 8c, the TOC measurements were used to assess the degree of MB mineralization. The C-TiO₂-MoS₂ nanocomposite achieved a TOC removal efficiency of approximately 72.7%, indicating that a substantial portion of the MB dye was mineralized into CO₂ and H₂O under UV irradiation. These results demonstrate the strong mineralization capability of the C-TiO₂-MoS₂ photocatalyst.

4. Conclusions

In summary, the C-TiO₂-MoS₂ nanocomposite, incorporating 2D MoS₂ exfoliated from bulk MoS₂, was successfully synthesized by mixing TiO₂-MoS₂ nanopowder with the SDC surfactant, followed by thermal annealing at 600 °C in Ar gas. The photocatalytic performance of the nanocomposite was evaluated through the degradation of MB under both UV and visible light irradiation. The results demonstrated excellent degradation efficiency, achieving 99.6% degradation of MB under UV light within 45 minutes and 99.4% under visible light within 50 minutes. The nanocomposite exhibited similar photocatalytic performance when applied to the degradation of high-concentration RhB. These findings highlight a promising strategy for the development of high-performance materials for practical photocatalytic applications.

Data availability

The datasets used and/or analyzed during the current study are available from the corresponding author upon reasonable request. In addition, all the data generated or analyzed during this study are included in this article.



Author contributions

Thi Huyen Nguyen: conceptualization, methodology, investigation, formal analysis. Tien Dung Cao: formal analysis, investigation. Thi Phuong Mai: formal analysis, investigation. Van Hau Tran: formal analysis, investigation. Van Trinh Pham: formal analysis, investigation. Van Chuc Nguyen: formal analysis, investigation, project administration. Van Nhat Pham: formal analysis, investigation. Van Tiep Phung: formal analysis, investigation. Ngoc Minh Phan: formal analysis, investigation. Van Tan Tran: investigation. Van Hao Nguyen: formal analysis, investigation. Van Tu Nguyen: supervision, conceptualization, methodology, formal analysis, writing – original draft preparation, validation, writing – reviewing and editing. All authors discussed and approved the paper.

Conflicts of interest

There are no conflicts to declare.

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References

- 1 Y. Q. Yang, Z. H. Zheng, D. F. Zhang and X. D. Zhang, *Environ. Sci.:Water Res. Technol.*, 2020, **6**, 2454–2464.
- 2 W. T. Xu, J. N. Chen, Y. Qiu, W. Peng, N. Shi and J. C. Zhou, *Separ. Purif. Technol.*, 2019, **213**, 426–436.
- 3 T. H. Nguyen, A. S. S. Tran, T. T. Cao, V. Tu Nguyen, V. T. Pham, V. T. Tran, T. Q. X. Le, H. T. Bui, V. H. Tran, T. Do, D. L. Pham, N. M. Phan, H. Abe and V. C. Nguyen, *RSC Adv.*, 2025, **15**(14), 10754–10762.
- 4 H. Kumari, S. Suman, R. Ranga, S. Chahal, S. Devi, S. Sharma, S. Kumar, P. Kumar, S. Kumar, A. Kumar and R. Parmar, *Water Air Soil Pollut.*, 2023, **234**, 349.
- 5 M. Mehta, A. P. Singh, S. Kumar, S. Krishnamurthy, B. Wickman and S. Basu, *Vacuum*, 2018, **155**, 675–681.
- 6 K. Li, Y. Zhong, S. Luo and W. Deng, *Appl. Catal. B Environ.*, 2020, **278**, 119313.
- 7 F. Bi, Y. Zhang, Z. Zhou, L. Guo, Z. Zhu, B. Liu and X. Zhang, *Molecules*, 2025, **30**(3), 697.
- 8 Q. Chen, A. Ozkan, B. Chattopadhyay, K. Baert, C. Poleunis, A. Tromont, R. Snyders, A. Delcorte, H. Terry and M.-P. Delplancke-Ogletree, *Langmuir*, 2019, **35**, 7161–7168.
- 9 S. Liu, and J. Yu, in *Nanostructured Photocatalysts*, ed. H. Yamashita, H. Li, Springer, Cham, Switzerland, 2016, ch. 10, pp. 187–200.
- 10 H. Lee, I.-S. Park, H.-J. Bang, Y.-K. Park, H. Kim, H.-H. Ha, B.-J. Kim and S.-C. Jung, *Appl. Surf. Sci.*, 2019, **471**, 893–899.
- 11 S. A. Ansari, M. M. Khan, M. O. Ansari and M. H. Cho, *Sol. Energy Mater. Sol. Cells*, 2015, **141**, 162–170.
- 12 A. Zielińska-Jurek, E. Kowalska, J. W. Sobczak, W. Lisowski, B. Ohtani and A. Zaleska, *Appl. Catal. B Environ.*, 2011, **101**, 504–514.
- 13 S. Sakthivel, M. Shankar, M. Palanichamy, B. Arabindoo, D. Bahnemann and V. Murugesan, *Water Res.*, 2004, **38**, 3001–3008.
- 14 P. Akhter, F. Ali, A. Ali and M. Hussain, *Diamond Relat. Mater.*, 2024, **141**, 110702.
- 15 N.-B. Trinh, T. A. Nguyen, S. Van Vu, H.-G. T. Vo, T. N. H. Lo, I. Park and K. Q. Vo, *RSC Adv.*, 2024, **14**, 34037–34050.
- 16 J. Huang, X. Feng, F. Bi, G. Huang, R. Rao, R. Qiao and X. Zhang, *Sep. Purif. Technol.*, 2025, **359**, 130804.
- 17 J. Huang, J. Wei, F. Tian, F. Bi, R. Rao, Y. Wang, H. Tao, N. Liu and X. Zhang, *Mater. Today Chem.*, 2024, **41**, 102292.
- 18 Y. Zhang, Z. Zhou, T. Chen, H. Wang and W. Lu, *J. Environ. Sci.*, 2014, **26**, 2114–2122.
- 19 A. Purabgola, N. Mayilswamy and B. Kandasubramanian, *Environ. Sci. Pollut. Res.*, 2022, **29**, 32305–32325.
- 20 Á. Tolosana-Moranchel, A. Manassero, M. L. Satuf, O. M. Alfano, J. A. Casas and A. Bahamonde, *Appl. Catal. B Environ.*, 2019, **246**, 1–11.
- 21 S. Shen, K. Chen, H. Wang and J. Fu, *Diamond Relat. Mater.*, 2022, **124**, 108896.
- 22 R. M. Sendão, M. Algarra, E. Ribeiro, M. Pereira, A. Gil, N. Vale, J. C. Esteves da Silva and L. Pinto da Silva, *Adv. Sustainable Syst.*, 2024, **8**, 2300317.
- 23 W. Ren, Z. Ai, F. Jia, L. Zhang, X. Fan and Z. Zou, *Appl. Catal. B Environ.*, 2007, **69**, 138–144.
- 24 H. Park, H.-I. Kim, G.-H. Moon and W. Choi, *Energy Environ. Sci.*, 2016, **9**, 411–433.
- 25 E. Bu, X. Chen, C. López-Cartes, F. Cazana, A. Monzón, J. Martínez-López and J. J. Delgado, *Catal. Today*, 2023, **422**, 114220.
- 26 H. Dong, D. Chen, K. Wang and R. Zhang, *Nanoscale Res. Lett.*, 2016, **11**, 1–14.
- 27 K. F. Mak, C. Lee, J. Hone, J. Shan and T. F. Heinz, *Phys. Rev. Lett.*, 2010, **105**, 136805.
- 28 D. Wang, Y. Xu, F. Sun, Q. Zhang, P. Wang and X. Wang, *Appl. Surf. Sci.*, 2016, **377**, 221–227.
- 29 G. Chandrabose, A. Dey, S. S. Gaur, S. Pitchaimuthu, H. Jagadeesan, N. S. J. Braithwaite, V. Selvaraj, V. Kumar and S. Krishnamurthy, *Chemosphere*, 2021, **279**, 130467.
- 30 Z. Tang, L. Xu, K. Shu, J. Yang and H. Tang, *Colloids Surf. A Physicochem. Eng. Asp.*, 2022, **642**, 128686.
- 31 A. H. Mady, M. A. Mahadadalkar, M. L. Baynosa, D. R. Kumar, A. M. Rabie, J. Lee, W. K. Kim and J.-J. Shim, *J. Colloid Interface Sci.*, 2022, **606**, 337–352.
- 32 M. M. Ali and K. N. Y. Sandhya, *New J. Chem.*, 2016, **40**, 8123–8130.
- 33 G. Cao and N. Yi, *ChemistrySelect*, 2020, **5**, 11530–11533.
- 34 R. Yuan, Y. Guo, I. Gurgan, N. Siddique, Y.-S. Li, S. Jang, G. A. Noh and S. H. Kim, *Carbon*, 2025, **238**, 120214.
- 35 X. Han, Z. Zhang, Y. Dong, J. Zhao, G. Sun, J. Hu, Q. Xu, X. Zhang, L. Li, T. Toyao, K. Shimizu and N. Zhang, *Chem. Eng. J.*, 2025, **510**, 161609.
- 36 X. Ni, C. Chen, Q. Wang and Z. Li, *Chem. Phys.*, 2019, **525**, 110398.
- 37 L. Zhang, Y. He, Y. Wu and T. Wu, *Mater. Sci. Eng. B*, 2011, **176**, 1497–1504.



- 38 L. Zheng, S. Han, H. Liu, P. Yu and X. Fang, *Small*, 2016, **12**, 1527–1536.
- 39 I. Tacchini, E. Terrado, A. Anson and M. T. Martínez, *Micro Nano Lett.*, 2011, **6**, 932–936.
- 40 W. Gao, M. Wang, C. Ran and L. Li, *Chem. Commun.*, 2015, **51**, 1709–1712.
- 41 Y. Zhang, M. Gong, Xi. Liu, L. Ji, Z. Yang and X. Zhu, *J. Mater. Sci.*, 2019, **54**, 2975–2989.
- 42 Y. Zhu, J. Xue, T. Xu, G. He and H. Chen, *J. Mater. Sci. Mater. Electron.*, 2017, **28**, 8519.
- 43 A. Mancuso, S. Mottola, O. Sacco, V. Vaiano and I. De Marco, *Nanomaterials*, 2023, **13**, 3130.

