

Engineering $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ and $\text{ZnO}/\text{g-C}_3\text{N}_4$ Heterojunctions for Efficient Visible-Light-Driven Photocatalytic Degradation of Organic Pollutants

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Abstract

The development of efficient photocatalysts for wastewater treatment remains a major challenge due to the persistence of dye pollutants in aquatic environments. In this study, g-C₃N₄-based composites, Bi₂O₃/g-C₃N₄ and ZnO/g-C₃N₄, were successfully synthesized and evaluated for the photocatalytic degradation of methyl orange (MO) and methylene blue (MB) under UV–visible light irradiation. Structural and optical properties were characterized using FTIR, UV–Vis diffuse reflectance spectroscopy, and photoluminescence analysis. The incorporation of Bi₂O₃ and ZnO enhanced visible-light absorption and suppressed charge carrier recombination, resulting in improved photocatalytic activity compared to pristine g-C₃N₄. The optimized 18wt% Bi₂O₃/g-C₃N₄ composite achieved the highest MO degradation efficiency of 68.82%, while 18wt% ZnO/g-C₃N₄ exhibited 68.67% degradation of MB. Process parameter studies revealed that a catalyst dosage of 1.0 g/L, initial dye concentration of 10 ppm, and near-neutral pH conditions were optimal for both systems. The enhanced performance is attributed to the synergistic interaction between g-C₃N₄ and the metal oxides, which improves light harvesting and charge utilization. These findings highlight the potential of g-C₃N₄-based composites as efficient photocatalysts for dye removal in wastewater treatment applications.

Introduction

The rapid growth of industrial activities has resulted in the extensive release of organic pollutants into aquatic environments. Among these pollutants, synthetic dyes represent one of the most significant classes of contaminants due to their widespread use in textile, printing, leather, pharmaceutical, and cosmetic industries. It is estimated that approximately 10–15% of dyes used during industrial processing are discharged into wastewater streams [1], [2], [3]. The presence of dye pollutants in water bodies can significantly reduce light penetration, inhibit photosynthetic activity, and pose serious risks to aquatic organisms and human health due to their toxic and potentially carcinogenic properties. Among the numerous dyes reported in industrial wastewater, methyl orange (MO) and methylene blue (MB) are commonly used as model pollutants for photocatalytic degradation studies [4], [5], [6]. Methyl orange is an anionic azo dye characterized by a complex aromatic structure that provides high chemical stability and resistance to biodegradation. Similarly, methylene blue is a cationic dye that is widely used in textile dyeing and biological staining applications. Both dyes exhibit high persistence in aquatic environments, making their removal through conventional treatment methods such as adsorption, coagulation, and biological treatment challenging.

In recent years, semiconductor photocatalysis has emerged as a promising and environmentally friendly approach for the degradation of organic pollutants in wastewater. Photocatalysis involves the activation of semiconductor materials by light irradiation to generate electron–hole pairs, which subsequently participate in redox reactions to produce reactive oxygen species such as hydroxyl radicals ($\cdot\text{OH}$) and superoxide radicals ($\cdot\text{O}_2^-$). These highly reactive species are capable of degrading organic pollutants into harmless products such as carbon dioxide and water [7], [8]. Compared to conventional treatment

technologies, photocatalysis offers several advantages including high efficiency, low energy consumption, and the potential for complete mineralization of pollutants [9], [10].

Among various semiconductor photocatalysts investigated, graphitic carbon nitride ($\text{g-C}_3\text{N}_4$) has attracted significant attention due to its unique physicochemical properties. $\text{g-C}_3\text{N}_4$ is a metal-free polymeric semiconductor composed of tri-s-triazine units connected by planar amino groups. It possesses a moderate band gap energy of approximately 2.7–2.8 eV [11], [12], enabling visible-light absorption. In addition, $\text{g-C}_3\text{N}_4$ exhibits high thermal and chemical stability, low toxicity, and can be synthesized easily from inexpensive nitrogen-rich precursors such as melamine, urea, and dicyandiamide through thermal polymerization [13].

Despite these advantages, pristine $\text{g-C}_3\text{N}_4$ suffers from several intrinsic limitations that hinder its photocatalytic performance. These include a relatively low specific surface area, limited visible-light absorption range, and rapid recombination of photogenerated electron–hole pairs [14], [15]. The fast recombination rate of charge carriers significantly reduces the number of active radicals generated during photocatalytic reactions, thereby limiting the overall degradation efficiency. To overcome these limitations, several modification strategies have been proposed to enhance the photocatalytic activity of $\text{g-C}_3\text{N}_4$. These strategies include elemental doping [12], [16], morphology engineering [10], and the construction of heterojunction structures with other semiconductor materials [11], [14]. Among these approaches, the formation of semiconductor heterojunctions has proven to be particularly effective for improving photocatalytic performance. Heterojunction structures facilitate the separation of photogenerated charge carriers by creating an internal electric field at the interface between two semiconductors, thereby suppressing electron–hole recombination and enhancing photocatalytic efficiency.

Metal oxide semiconductors such as bismuth oxide (Bi_2O_3) and zinc oxide (ZnO) have been widely studied due to their favorable electronic structures and strong photocatalytic properties. Bi_2O_3 is a p-type semiconductor with strong visible-light absorption and excellent oxidation capability [17], while ZnO is an n-type semiconductor known for its high electron mobility, chemical stability, and strong oxidation potential [18], [19], [20]. Coupling these metal oxides with $\text{g-C}_3\text{N}_4$ can form heterojunction structures that enhance light absorption and promote efficient charge transfer between the semiconductor components. In particular, the integration of Bi_2O_3 with $\text{g-C}_3\text{N}_4$ can form a Z-scheme heterojunction, which facilitates charge separation through the formation of an internal electric field at the interface. On the other hand, coupling ZnO with $\text{g-C}_3\text{N}_4$ can create a Type II heterojunction, which can also improve charge transfer and enhance photocatalytic performance.

In this study, two $\text{g-C}_3\text{N}_4$ -based composite photocatalysts, namely $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ and $\text{ZnO}/\text{g-C}_3\text{N}_4$, were synthesized and investigated for the photocatalytic degradation of organic dyes in aqueous solution. The photocatalytic performance of the composites was evaluated using methyl orange and methylene blue as model pollutants under UV–visible light irradiation. The structural and optical properties of the

synthesized materials were characterized using FTIR, UV–Vis diffuse reflectance spectroscopy, and photoluminescence spectroscopy. Furthermore, the relationship between heterojunction formation, charge separation efficiency, and photocatalytic activity was systematically analyzed. The objective of this work is to provide a comparative evaluation of two g-C₃N₄-based heterojunction photocatalysts and to elucidate the mechanisms responsible for their enhanced photocatalytic performance in the degradation of both anionic and cationic dye pollutants.

Experimental Section

2.1 Materials

Melamine (C₃H₆N₆), bismuth oxide (Bi₂O₃), zinc oxide (ZnO), methanol, methyl orange (MO), methylene blue (MB), hydrochloric acid, sodium hydroxide, and distilled water were used in this study. All reagents were of analytical grade and used without further purification.

2.2 Synthesis of g-C₃N₄

g-C₃N₄ was synthesized through thermal polymerization of melamine. Approximately 4 g of melamine powder was placed in a covered ceramic crucible and heated in a muffle furnace at 550°C for 2 h with a heating rate of 5°C/min. After cooling to room temperature, the obtained yellow product was ground into fine powder and stored for further use.

2.3 Preparation of Bi₂O₃/g-C₃N₄ Composite

Bi₂O₃/g-C₃N₄ composites were prepared using the wet impregnation method. A predetermined amount of Bi₂O₃ was dispersed in methanol and mixed with g-C₃N₄ powder. The suspension was stirred continuously for several hours to ensure uniform mixing. The mixture was then filtered and dried at 80°C overnight. The dried powder was calcined at 500°C for 2 h to obtain the final Bi₂O₃/g-C₃N₄ composite photocatalysts.

2.4 Preparation of ZnO/g-C₃N₄ Composite

ZnO/g-C₃N₄ composites were prepared using a similar procedure. ZnO powder was dispersed in methanol and mixed with g-C₃N₄. The mixture was stirred to obtain a homogeneous suspension, followed by filtration and drying. The dried sample was calcined to produce the ZnO/g-C₃N₄ composite photocatalysts.

2.5 Characterization

The structural and optical properties of the synthesized photocatalysts were analyzed using the several techniques including of Fourier Transform Infrared Spectroscopy (FTIR), UV–Visible Diffuse Reflectance Spectroscopy (UV–Vis DRS) and Photoluminescence (PL) Spectroscopy.

2.6 Photocatalytic Degradation Experiments

Photocatalytic activity was evaluated by monitoring the degradation of methyl orange and methylene blue under UV–visible light irradiation. A known amount of photocatalyst was dispersed in dye solution and stirred in the dark for 30 minutes to establish adsorption–desorption equilibrium before illumination. During irradiation, aliquots of the solution were collected at regular intervals and analyzed using UV–Vis spectroscopy to determine the concentration of the dye.

Results and Discussion

3.1 FTIR Analysis

The FTIR spectra presented in Fig. 2 reveal the characteristic functional groups of pristine g-C₃N₄ as well as the Bi₂O₃/g-C₃N₄ and ZnO/g-C₃N₄ composites. For pristine g-C₃N₄, a prominent absorption band observed at approximately 804 cm⁻¹ corresponds to the breathing mode of the triazine units, which is a typical structural feature of g-C₃N₄. In addition, strong absorption bands located in the 1500–1600 cm⁻¹ region are attributed to the stretching vibrations of C = N bonds within the aromatic heterocyclic framework [21], [22]. Meanwhile, the peaks observed between 1200 and 1400 cm⁻¹ are associated with the stretching vibrations of C–N bonds in the heterocyclic ring structure, further confirming the successful formation of the polymeric g-C₃N₄ network [23].

Upon incorporation of Bi₂O₃ into the g-C₃N₄ matrix, an additional absorption band emerges in the range of 604–646 cm⁻¹, which can be assigned to the Bi–O bending vibration, indicating the successful introduction of Bi₂O₃ into the composite structure. Despite the appearance of this new band, the overall spectral profile of the Bi₂O₃/g-C₃N₄ composites remains largely similar to that of pristine g-C₃N₄, suggesting that the fundamental framework of g-C₃N₄ is preserved after composite formation. Furthermore, the intensity of the Bi–O vibration becomes more pronounced with increasing Bi₂O₃ loading from 6wt% to 24wt%, while the characteristic peaks of g-C₃N₄ remain nearly unchanged. These observations indicate that the incorporation of Bi₂O₃ does not significantly alter the intrinsic structure of the g-C₃N₄ framework.

Similarly, in the ZnO/g-C₃N₄ composites, the presence of ZnO can be identified by an absorption band in the low wavenumber region of 500–550 cm⁻¹, which is attributed to the Zn–O stretching vibration [24]. The ATR-FTIR spectra show that the intensity of this Zn–O band gradually increases with increasing ZnO loading, particularly for the 12wt%, 18wt%, 24wt%, and 30wt% ZnO/g-C₃N₄ samples. At lower ZnO contents, the Zn–O band exhibits relatively weak intensity due to the smaller amount of ZnO present in the composite. As the ZnO loading increases, the higher concentration of Zn–O bonds results in a more distinguishable absorption band in this region.

Overall, the characteristic functional groups of both g-C₃N₄ and the respective metal oxides are clearly observed in the composite spectra, confirming the successful synthesis of the Bi₂O₃/g-C₃N₄ and ZnO/g-

C₃N₄ photocatalysts. The coexistence of the characteristic peaks of g-C₃N₄ with the Bi–O and Zn–O vibrations further indicates that the metal oxides were successfully incorporated into the g-C₃N₄ matrix without disrupting its fundamental structure.

3.2 Optical Properties and Band Gap Analysis

UV–Vis Diffuse Reflectance Spectroscopy (UV–Vis DRS) was employed to investigate the optical absorption properties of the synthesized photocatalysts. The UV–Vis DRS spectra of pristine g-C₃N₄, pure metal oxides, and their respective composites are shown in Fig. 3.

For the Bi₂O₃/g-C₃N₄ system, pristine g-C₃N₄ exhibits an absorption edge at approximately 480 nm, indicating its ability to absorb visible light due to its moderate band gap. In comparison, pure Bi₂O₃ shows an absorption edge around 460 nm. After incorporating Bi₂O₃ into the g-C₃N₄ matrix, the Bi₂O₃/g-C₃N₄ composites exhibit a noticeable red shift in the absorption edge toward the visible-light region compared with pristine g-C₃N₄. This shift suggests that the electronic interaction between Bi₂O₃ and g-C₃N₄ enhances the light-harvesting capability of the composite photocatalysts. Such behavior indicates a synergistic effect resulting from the coupling of the two semiconductor components.

Among the investigated samples, the 18wt% Bi₂O₃/g-C₃N₄ composite exhibits the most pronounced red shift, with the absorption edge extending to approximately 560 nm. This result suggests that this loading level provides an optimal interaction between Bi₂O₃ and g-C₃N₄, thereby enhancing visible-light absorption. In contrast, the 24 wt% Bi₂O₃/g-C₃N₄ composite shows a slightly lower absorption edge at approximately 550 nm. The relatively smaller shift may be attributed to excessive Bi₂O₃ content, which could partially block active sites or alter the interfacial interaction between the two components, thereby reducing the efficiency of light absorption [10], [25]. Overall, these observations indicate that the incorporation of Bi₂O₃ significantly improves the optical response of g-C₃N₄, particularly at an intermediate loading level.

Similarly, the UV–Vis DRS spectra of pure g-C₃N₄, pure ZnO, and ZnO/g-C₃N₄ composites reveal notable changes in optical absorption behavior. As summarized in Table 4.1, pristine g-C₃N₄ exhibits an absorption edge at approximately 460 nm, corresponding to a band gap of 2.80 eV, which enables visible-light absorption. In contrast, pure ZnO shows an absorption edge at around 400 nm with a wider band gap of 3.20 eV, indicating that ZnO primarily absorbs light in the ultraviolet region.

Table 1
Photocatalysts and their light absorption edges.

Photocatalyst	Absorption Edge (nm)	Band Gap (eV)
Pure g-C ₃ N ₄	460	2.80
Pure Bi ₂ O ₃	460	2.80
18wt% Bi ₂ O ₃ /g-C ₃ N ₄	560	2.25
24wt% Bi ₂ O ₃ /g-C ₃ N ₄	550	2.30
Pure ZnO	400	3.20
18wt% ZnO/g-C ₃ N ₄	485	2.73
24wt% ZnO/g-C ₃ N ₄	480	2.78

After coupling ZnO with g-C₃N₄, the ZnO/g-C₃N₄ composites display a distinct red shift in their absorption edges compared with the individual components. This shift suggests that the interaction between ZnO and g-C₃N₄ enhances visible-light absorption, likely due to the formation of an effective heterojunction structure. Among the synthesized samples, the 18wt% ZnO/g-C₃N₄ composite exhibits the most significant red shift, with the absorption edge extending to approximately 485 nm and a reduced band gap of 2.73 eV. The narrowing of the band gap indicates enhanced visible-light utilization, which can be attributed to strong interfacial interaction and efficient heterojunction formation between ZnO and g-C₃N₄. Meanwhile, the 24wt% ZnO/g-C₃N₄ composite also exhibits a red shift but with a slightly lower absorption edge at approximately 480 nm and a band gap of 2.78 eV compared with the 18wt% composite. This result suggests that excessive ZnO loading may weaken the synergistic interaction between the two components, thereby slightly reducing the visible-light absorption capability of the composite photocatalyst [26].

Overall, the UV–Vis DRS results demonstrate that coupling g-C₃N₄ with Bi₂O₃ and ZnO effectively extends light absorption into the visible region. In particular, the 18wt% loading for both Bi₂O₃/g-C₃N₄ and ZnO/g-C₃N₄ composites exhibits the most pronounced red shift, indicating optimized heterojunction interaction. The enhanced visible-light absorption is expected to contribute to improved photocatalytic performance, which will be discussed in the following section.

3.3 Photoluminescence (PL) Spectroscopy

PL spectroscopy was performed to investigate the recombination behavior of photogenerated charge carriers in the synthesized photocatalysts. The PL spectra of pristine g-C₃N₄, pure metal oxides, and their respective composites are presented in Fig. 4.

PL emission occurs when photogenerated electrons recombine with holes and release energy in the form of light. Therefore, a higher PL intensity indicates a higher recombination rate of electron–hole pairs, while a lower PL intensity suggests more efficient charge separation, which is beneficial for photocatalytic activity [21], [27].

For the $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ system, pristine $\text{g-C}_3\text{N}_4$ exhibits the strongest PL emission intensity, indicating severe recombination of photogenerated charge carriers. After incorporating Bi_2O_3 into the $\text{g-C}_3\text{N}_4$ matrix, the PL intensity of all $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ composites decreases significantly, suggesting that coupling with Bi_2O_3 effectively suppresses electron–hole recombination and enhances charge separation. Among the investigated composites, the 18wt% $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ sample exhibits slightly higher PL intensity compared to the 24wt% $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ composite, indicating that charge recombination is somewhat more pronounced at the lower loading. In contrast, the 24wt% $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ composite shows a lower PL intensity, suggesting more efficient suppression of electron–hole recombination. This improvement may be attributed to stronger interfacial interaction and more effective heterojunction formation between Bi_2O_3 and $\text{g-C}_3\text{N}_4$ at higher loading levels [28]. Meanwhile, pure Bi_2O_3 exhibits the lowest PL intensity, reflecting its intrinsically efficient charge-separation capability.

A similar trend is observed in the $\text{ZnO}/\text{g-C}_3\text{N}_4$ composites. As shown in Fig. 4.3, pristine $\text{g-C}_3\text{N}_4$ again exhibits the highest PL intensity, indicating the most severe electron–hole recombination. In contrast, pure ZnO shows much lower PL intensity, suggesting more efficient charge separation. After coupling ZnO with $\text{g-C}_3\text{N}_4$, both 18wt% and 24wt% $\text{ZnO}/\text{g-C}_3\text{N}_4$ composites exhibit significantly reduced PL intensity compared with pristine $\text{g-C}_3\text{N}_4$, confirming that heterojunction formation between ZnO and $\text{g-C}_3\text{N}_4$ effectively suppresses charge-carrier recombination. Between the two composites, the 18wt% $\text{ZnO}/\text{g-C}_3\text{N}_4$ sample shows slightly higher PL intensity than the 24wt% composite, indicating that some degree of recombination still occurs at this loading level. This behavior may be related to incomplete surface coverage of ZnO on the $\text{g-C}_3\text{N}_4$ framework, leaving certain regions where recombination can still occur [29]. Despite the slightly higher PL intensity, the UV–Vis DRS results demonstrate that the 18wt% $\text{ZnO}/\text{g-C}_3\text{N}_4$ composite exhibits superior visible-light absorption, which contributes to its enhanced photocatalytic activity.

Overall, the PL results indicate that the formation of $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ and $\text{ZnO}/\text{g-C}_3\text{N}_4$ heterojunction structures effectively suppresses electron–hole recombination, thereby improving charge separation efficiency. This enhanced charge-carrier dynamics is expected to play a crucial role in improving the photocatalytic degradation performance of the composites.

3.4 Photocatalytic Degradation of Dyes

The photocatalytic performance of the synthesized materials was evaluated by monitoring the degradation of dye pollutants under UV–visible light irradiation. Methyl orange (MO) and methylene blue (MB) were selected as model dyes representing anionic and cationic organic pollutants, respectively.

3.4.1 Methyl Orange Degradation

A series of photocatalytic experiments were conducted to evaluate the performance of pristine g-C₃N₄, Bi₂O₃, and Bi₂O₃/g-C₃N₄ composites with varying Bi₂O₃ loadings (6, 12, 18, and 24wt%). All experiments were carried out under identical conditions, using an initial methyl orange (MO) concentration of 10 ppm, natural solution pH (6.7), and a fixed photocatalyst dosage of 1 g/L. The photocatalytic degradation profiles of MO are presented in Fig. 5, while the corresponding degradation efficiencies are summarized in Table 2.

Prior to light irradiation, adsorption–desorption equilibrium was established under dark conditions. The optimized composite exhibited a relatively high MO removal of 58.39% in the dark, indicating that a significant portion of the dye molecules was adsorbed onto the catalyst surface rather than degraded [30], [31]. In contrast, the photolysis experiment (without catalyst) resulted in a negative degradation value (–72.17%), suggesting an apparent increase in absorbance. This behavior may be attributed to the inherent photostability of MO or experimental factors such as re-dispersion of previously adsorbed dye molecules and changes in the absorption spectrum due to partial photochemical transformation [32], [33].

Under light irradiation, pristine g-C₃N₄ exhibited the lowest photocatalytic activity, with a negative degradation percentage (–32.91%), indicating its limited capability for MO degradation. This poor performance can be attributed to its rapid electron–hole recombination rate and relatively limited visible-light absorption, as confirmed by the PL and UV–Vis DRS analyses. In comparison, pure Bi₂O₃ showed a slightly improved degradation efficiency of 5.11%, suggesting some intrinsic photocatalytic activity. However, its performance remains limited, likely due to insufficient charge separation efficiency and suboptimal light absorption characteristics.

Table 2
Photocatalytic activity of prepared photocatalysts.

Catalyst	Degradation efficiency (%)
g-C ₃ N ₄	-32.91
Bi ₂ O ₃	5.11
6wt% Bi ₂ O ₃ /g-C ₃ N ₄	7.55
12wt% Bi ₂ O ₃ /g-C ₃ N ₄	47.36
18wt% Bi ₂ O ₃ /g-C ₃ N ₄	68.82
24wt% Bi ₂ O ₃ /g-C ₃ N ₄	23.32

The formation of $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ composites significantly enhanced the photocatalytic performance. The degradation efficiency increased progressively from 7.55% for 6wt% $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ to a maximum of 68.82% for 18wt% $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$, demonstrating the beneficial effect of incorporating Bi_2O_3 into the $\text{g-C}_3\text{N}_4$ matrix. This enhancement can be attributed to the formation of a Z-scheme heterojunction, which promotes efficient separation of photogenerated charge carriers, as evidenced by the reduced PL intensity. In addition, UV–Vis DRS results indicate that the 18wt% composite exhibits the strongest visible-light absorption, further contributing to its superior photocatalytic activity. However, further increasing the Bi_2O_3 loading to 24wt% resulted in a significant decrease in degradation efficiency to 23.32%. This decline may be attributed to excessive Bi_2O_3 coverage on the $\text{g-C}_3\text{N}_4$ surface, which can block active sites, hinder light penetration, and disrupt effective interfacial charge transfer [23], [34]. Moreover, the UV–Vis DRS analysis shows that the 24wt% composite exhibits slightly reduced visible-light absorption compared to the 18wt% sample, supporting the observed decline in photocatalytic performance.

Overall, these results demonstrate that Bi_2O_3 loading plays a critical role in determining photocatalytic efficiency, with the 18wt% $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ composite identified as the optimal composition. The enhanced performance is attributed to the synergistic effects of improved visible-light absorption and suppressed electron–hole recombination, highlighting the importance of heterojunction engineering in optimizing photocatalytic systems.

3.4.2 Methylene Blue Degradation

To evaluate the photocatalytic performance of the synthesized materials, a series of comparative experiments were conducted using $\text{ZnO}/\text{g-C}_3\text{N}_4$ composites with varying ZnO loadings (6, 12, 18, 24, and 30wt%), alongside pristine $\text{g-C}_3\text{N}_4$ and pure ZnO . The photocatalytic degradation of methylene blue (MB) was carried out under identical conditions, with a catalyst dosage of 1 g/L, an initial dye concentration of 10 ppm, and a solution pH of 5.11. The degradation profiles are presented in Fig. 6, while the corresponding degradation efficiencies are summarized in Table 3.

Prior to illumination, adsorption–desorption equilibrium was established under dark conditions. A relatively low MB removal of 33.73% was observed, indicating that the removal process in the absence of light is primarily governed by physical adsorption of dye molecules onto the catalyst surface. This behavior is expected, as the absence of light prevents the generation of electron–hole pairs and, consequently, the formation of reactive oxygen species required for photocatalytic degradation [27]. In contrast, the photolysis experiment (without catalyst) resulted in a comparatively high removal percentage ($\sim 60.78\%$), suggesting that MB undergoes partial self-degradation under light irradiation. This phenomenon can be attributed to the photosensitive nature of methylene blue, where direct light exposure can excite MB molecules, leading to partial structural degradation even in the absence of a photocatalyst [35]. However, this process remains inefficient for complete mineralization due to the lack of reactive radical species typically generated in photocatalytic systems.

Under photocatalytic conditions, pristine g-C₃N₄ exhibited moderate degradation efficiency (47.01%), which is higher than that of pure ZnO (34.30%). The relatively better performance of g-C₃N₄ can be attributed to its ability to absorb visible light, whereas ZnO, with its wider band gap, is mainly active under UV irradiation. The formation of ZnO/g-C₃N₄ heterojunction composites significantly enhanced the photocatalytic performance. Increasing the ZnO loading to 12wt% improved the degradation efficiency to 51.20%, indicating that the heterojunction structure begins to effectively facilitate charge separation and enhance light absorption at this composition.

Table 3
Photocatalytic activity of prepared photocatalysts.

Photocatalyst	Degradation efficiency (%)
Pure g-C ₃ N ₄	47.01
Pure ZnO	34.30
6wt% ZnO/g-C ₃ N ₄	33.68
12wt% ZnO/g-C ₃ N ₄	51.20
18wt% ZnO/g-C ₃ N ₄	68.67
24wt% ZnO/g-C ₃ N ₄	24.75
30wt% ZnO/g-C ₃ N ₄	23.41

The optimal photocatalytic performance was achieved with the 18wt% ZnO/g-C₃N₄ composite, which exhibited the highest degradation efficiency of 68.67%. This significant enhancement can be attributed to the formation of an efficient heterojunction interface that promotes effective charge separation, reduces electron–hole recombination, and improves visible-light absorption. These findings are consistent with the UV–Vis DRS results, which show the most pronounced red shift and band gap narrowing, as well as the PL analysis, which confirms suppressed recombination at this composition. However, further increasing the ZnO loading to 24wt% and 30wt% resulted in a sharp decline in degradation efficiency to 24.75% and 23.41%, respectively. This decrease may be attributed to excessive ZnO coverage on the g-C₃N₄ surface, which can block active sites, hinder light penetration, and disrupt effective interfacial charge transfer. In addition, agglomeration of ZnO particles at higher loadings may reduce the effective contact area between the two components, thereby weakening the heterojunction effect [36].

Overall, these results demonstrate that ZnO loading plays a crucial role in determining photocatalytic performance, with the 18wt% ZnO/g-C₃N₄ composite identified as the optimal composition. The enhanced activity is primarily attributed to the synergistic effects of improved visible-light absorption

and efficient suppression of electron–hole recombination, highlighting the importance of optimizing heterojunction composition for high-performance photocatalytic systems.

3.5 Process Parameter Studies

Process parameter studies were conducted using the optimized 18wt% Bi₂O₃/g-C₃N₄ and 18wt% ZnO/g-C₃N₄ composites to determine the key factors influencing photocatalytic degradation performance. The effects of photocatalyst dosage, initial dye concentration, and solution pH were systematically investigated for the degradation of methyl orange (MO) and methylene blue (MB), respectively.

3.5.1 Effect of Photocatalyst Dosage

The influence of photocatalyst dosage (0.5, 1.0, and 1.5 g/L) on dye degradation was examined under constant conditions ([dye] = 10 ppm; pH = 6.7 for MO and 5.11 for MB) are shown in Figs. 7(a) and (b). In both systems, increasing the catalyst dosage from 0.5 to 1.0 g/L led to a significant enhancement in photocatalytic performance. Specifically, MO degradation increased from – 66.07% to 68.82%, while MB degradation improved from 15.50% to 68.67%, indicating that a higher number of active sites and greater photon absorption promote the generation of reactive oxygen species. However, a further increase in catalyst dosage to 1.5 g/L resulted in a noticeable decline in degradation efficiency (MO: 37.33%; MB: 42.04%). This reduction can be attributed to light scattering and shielding effects, where excessive catalyst particles hinder light penetration into the suspension, thereby limiting photon utilization [35], [37]. Additionally, particle agglomeration at higher loadings reduces the effective surface area and the number of accessible active sites [23]. These results demonstrate that 1.0 g/L is the optimal catalyst dosage for both systems, providing a balance between sufficient active sites and effective light penetration.

3.5.2 Effect of Initial Dye Concentration

The effect of initial dye concentration on photocatalytic performance was evaluated by varying MO (5–20 ppm) and MB (10–25 ppm) concentrations while maintaining a constant catalyst dosage (1.0 g/L) illustrated in Figs. 7(c) and (d). For both systems, the highest degradation efficiency was observed at 10 ppm, with values of 68.82% (MO) and 68.67% (MB). At lower concentration (e.g., MO at 5 ppm), slight fluctuations and even negative degradation values were observed, which may be attributed to measurement limitations and reduced adsorption driving force at very low dye concentrations. In contrast, increasing the dye concentration beyond 10 ppm resulted in a significant decline in degradation efficiency (e.g., MO: down to – 27.59% at 20 ppm; MB: down to ~ 10–18% at ≥ 15 ppm). This behavior can be explained by several factors. First, higher dye concentrations increase competition for active sites, limiting adsorption and slowing the reaction rate [28]. Second, the solution becomes more optically dense, reducing light penetration and photon absorption by the photocatalyst [38]. Third, the formation of intermediate by-products during degradation may block active sites and inhibit further reaction [9], [39]. Thus, an initial dye concentration of 10 ppm is identified as the optimal condition for achieving maximum photocatalytic efficiency in both systems.

3.5.3 Effect of Solution pH

The effect of solution pH on photocatalytic performance was investigated at pH 3, natural pH (6.7 for MO; 5.11 for MB), and alkaline conditions (pH 9–10) are shown in Figs. 7(e) and (f). For the $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ system (MO degradation), the highest degradation efficiency (68.82%) was achieved at near-neutral pH (6.7). Under acidic conditions (pH 3), the degradation efficiency decreased (31.79%), although adsorption remained relatively high due to electrostatic attraction. This can be explained by the protonation of the catalyst surface, where excess H^+ ions compete with MO molecules for active sites, limiting photocatalytic reactions (Hanafi & Sapawe, 2020). In alkaline conditions, the catalyst surface becomes negatively charged, leading to electrostatic repulsion with anionic MO molecules, resulting in a drastic decline in degradation efficiency. For the $\text{ZnO}/\text{g-C}_3\text{N}_4$ system (MB degradation), the optimal performance (68.67%) was observed at the natural pH (5.11). At pH 3, the degradation efficiency decreased sharply (11.96%) due to electrostatic repulsion between the positively charged catalyst surface and cationic MB molecules, which inhibits adsorption. At pH 9, the degradation efficiency improved moderately (40.37%) due to enhanced electrostatic attraction; however, excessive OH^- ions compete with MB molecules for active sites and may also affect the structural stability of ZnO, thereby limiting overall performance [40]. Overall, these findings indicate that near-neutral pH conditions provide the most favorable environment for photocatalytic degradation, as they promote balanced electrostatic interactions, efficient adsorption, and enhanced generation of reactive species.

3.6 Photocatalytic Mechanism for $\text{g-C}_3\text{N}_4$ -based heterojunctions

The photocatalytic mechanisms of the $\text{ZnO}/\text{g-C}_3\text{N}_4$ and $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ composites are illustrated in Fig. 8. The two systems exhibit distinct charge transfer pathways due to differences in their band structures and interfacial interactions.

For the $\text{ZnO}/\text{g-C}_3\text{N}_4$ composite in Fig. 8(a), a conventional Type II heterojunction is formed due to the staggered alignment of the conduction band (CB) and valence band (VB) edges between ZnO and $\text{g-C}_3\text{N}_4$. Upon UV–visible light irradiation, both semiconductors are photoexcited, generating electron–hole (e^-/h^+) pairs. Because the CB of $\text{g-C}_3\text{N}_4$ is more negative than that of ZnO, photogenerated electrons migrate from the CB of $\text{g-C}_3\text{N}_4$ to the CB of ZnO. Simultaneously, holes transfer from the VB of ZnO to the VB of $\text{g-C}_3\text{N}_4$. This spatial separation of charge carriers effectively suppresses recombination. The accumulated electrons in ZnO participate in reduction reactions, converting dissolved oxygen into superoxide radicals ($\cdot\text{O}_2^-$), while the holes in $\text{g-C}_3\text{N}_4$ oxidize water or hydroxide ions to generate hydroxyl radicals ($\cdot\text{OH}$). These reactive oxygen species subsequently degrade methylene blue (MB). However, the Type II mechanism may partially reduce the redox ability due to the migration of charges to less energetic band positions.

In contrast, the $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ composite follows a direct Z-scheme mechanism, which preserves strong redox potentials (Fig. 9(b)). Upon irradiation, both Bi_2O_3 and $\text{g-C}_3\text{N}_4$ generate electron–hole pairs. Due to the internal electric field and band alignment, electrons in the CB of Bi_2O_3 recombine with holes in the VB of $\text{g-C}_3\text{N}_4$ at the interface. This selective recombination pathway retains highly energetic electrons in the CB of $\text{g-C}_3\text{N}_4$ and strong oxidative holes in the VB of Bi_2O_3 . As a result, electrons in the CB of $\text{g-C}_3\text{N}_4$ reduce O_2 to form $\text{O}_2^{\bullet-}$ and holes in the VB of Bi_2O_3 oxidize H_2O or OH^- to generate $\bullet\text{OH}$. This Z-scheme configuration maintains high redox capability while simultaneously suppressing electron–hole recombination, leading to enhanced photocatalytic degradation of methyl orange (MO).

Conclusion

In this study, $\text{g-C}_3\text{N}_4$ -based heterojunction photocatalysts were successfully synthesized by coupling $\text{g-C}_3\text{N}_4$ with Bi_2O_3 and ZnO . Characterization results confirmed the formation of composite structures with enhanced optical properties and improved charge carrier separation. Photocatalytic experiments demonstrated that the composites exhibited superior dye degradation performance compared with the individual components. The $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ composite showed the highest activity for methyl orange degradation, while $\text{ZnO}/\text{g-C}_3\text{N}_4$ demonstrated effective degradation of methylene blue. The improved photocatalytic performance is attributed to the synergistic interaction between $\text{g-C}_3\text{N}_4$ and metal oxide semiconductors, which promotes efficient charge transfer and generation of reactive oxygen species. These findings highlight the potential of $\text{g-C}_3\text{N}_4$ -based heterojunction photocatalysts for wastewater treatment applications.

Declarations

Author Contribution

R.A.M. wrote the main manuscript text and prepared figure 8 A.A.H.N. and F.N.H.F. prepared figures 1-7 S.S and A.R. M. provided facilities and equipments A.N.S. proofread, conceptualization and idea and supervision All authors reviewed the manuscript

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Figures

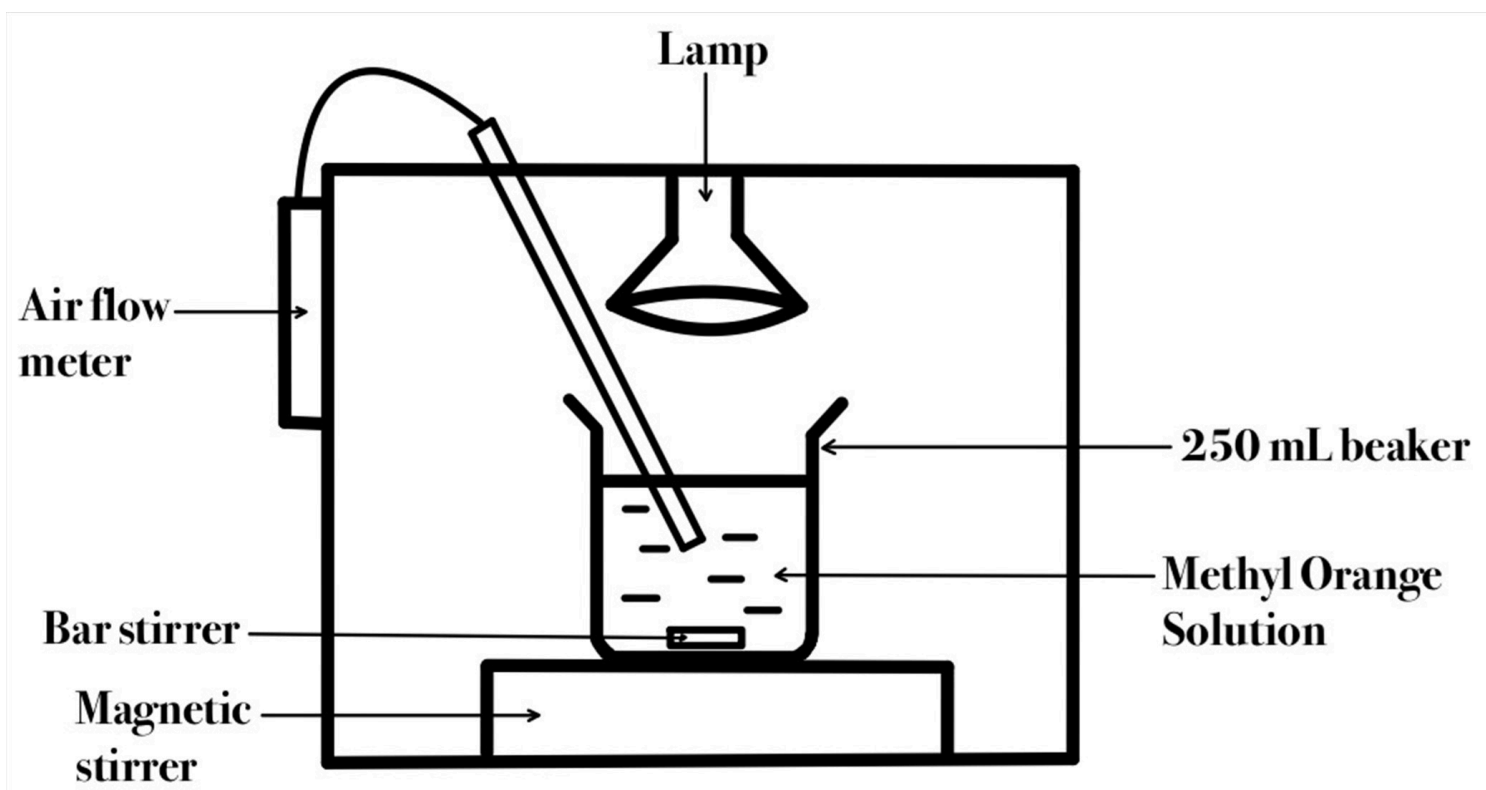


Figure 1

Experimental setup schematic diagram

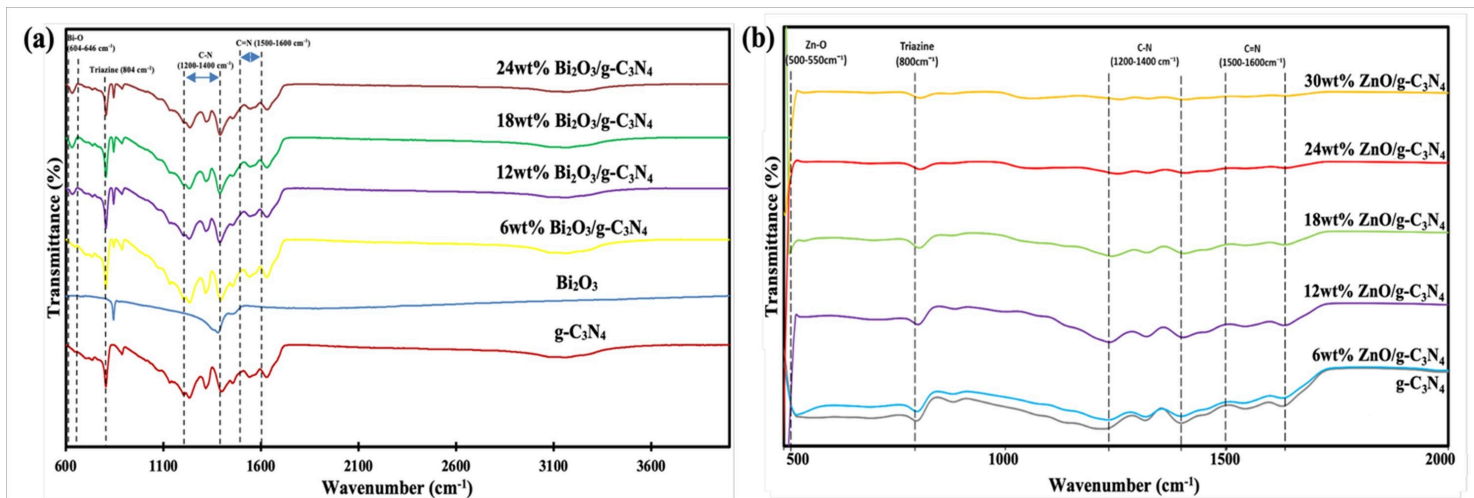


Figure 2

FTIR spectrum for (a) Bi₂O₃/g-C₃N₄ and (b) ZnO/g-C₃N₄ samples.

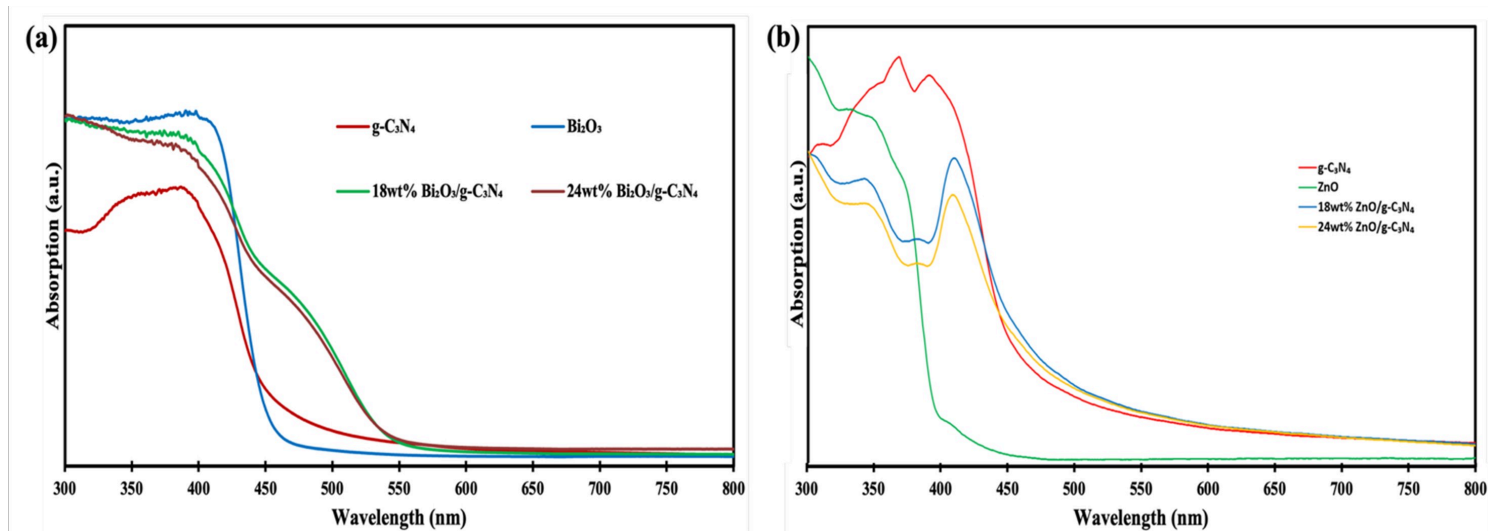


Figure 3

UV-Vis DRS of (a) $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ and (b) $\text{ZnO}/\text{g-C}_3\text{N}_4$ samples.

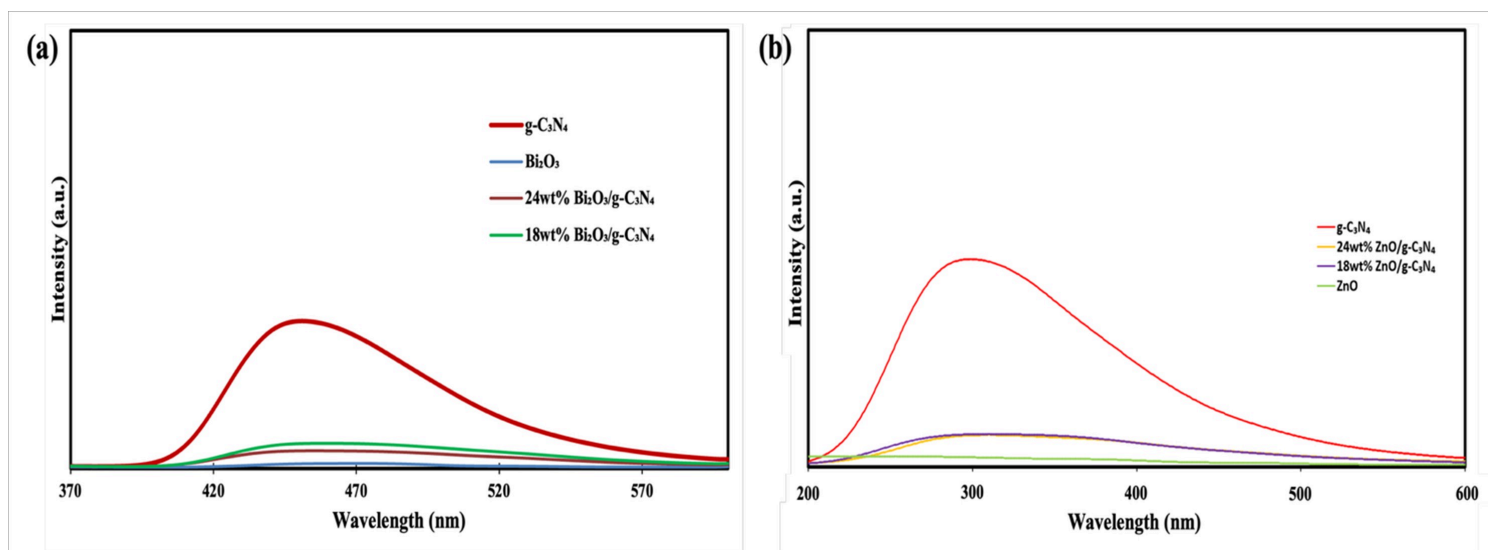


Figure 4

PL patterns for (a) $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ and (b) $\text{ZnO}/\text{g-C}_3\text{N}_4$ samples

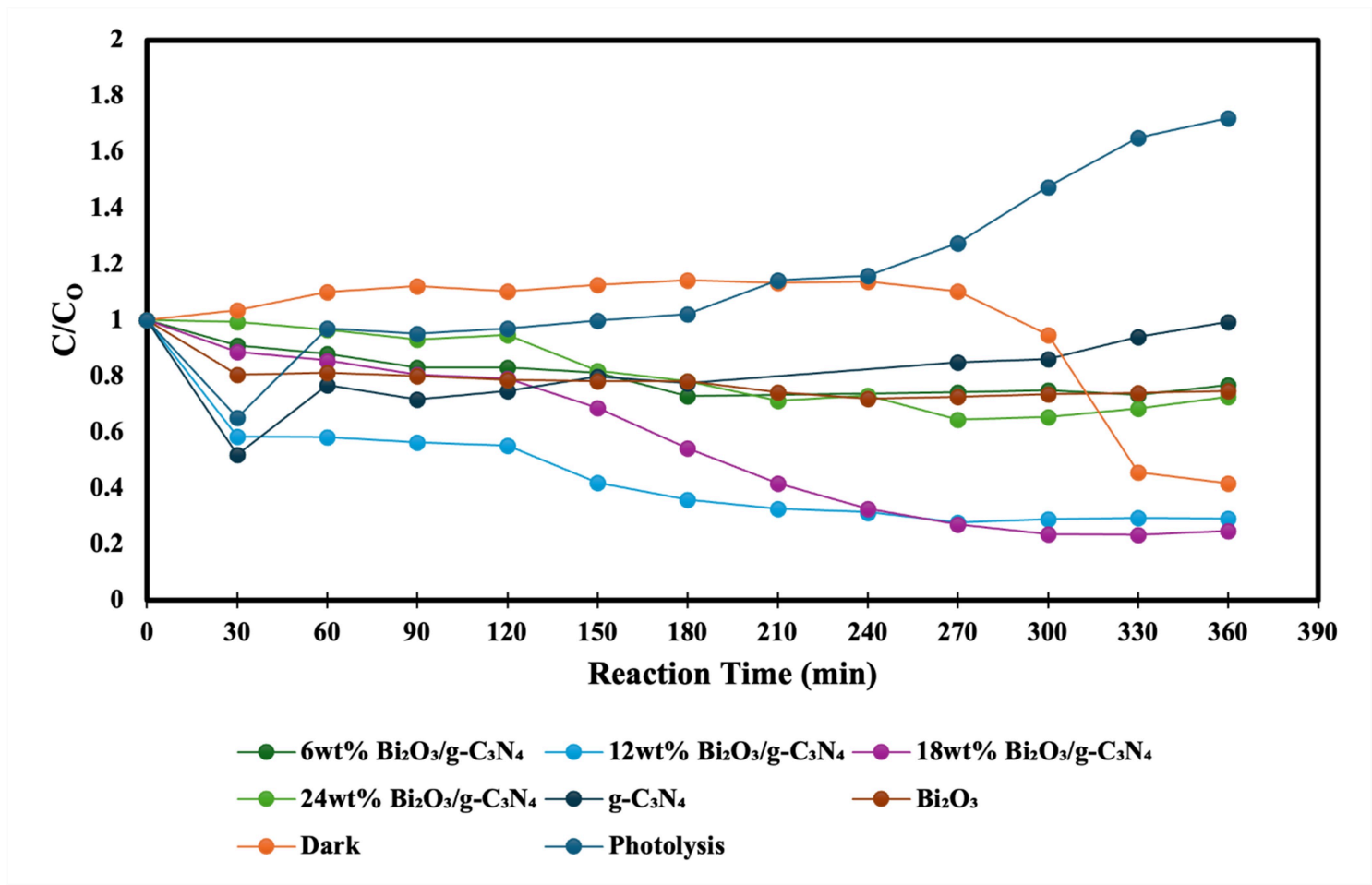


Figure 5

MO degradation using $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ samples with different amount of Bi_2O_3 .

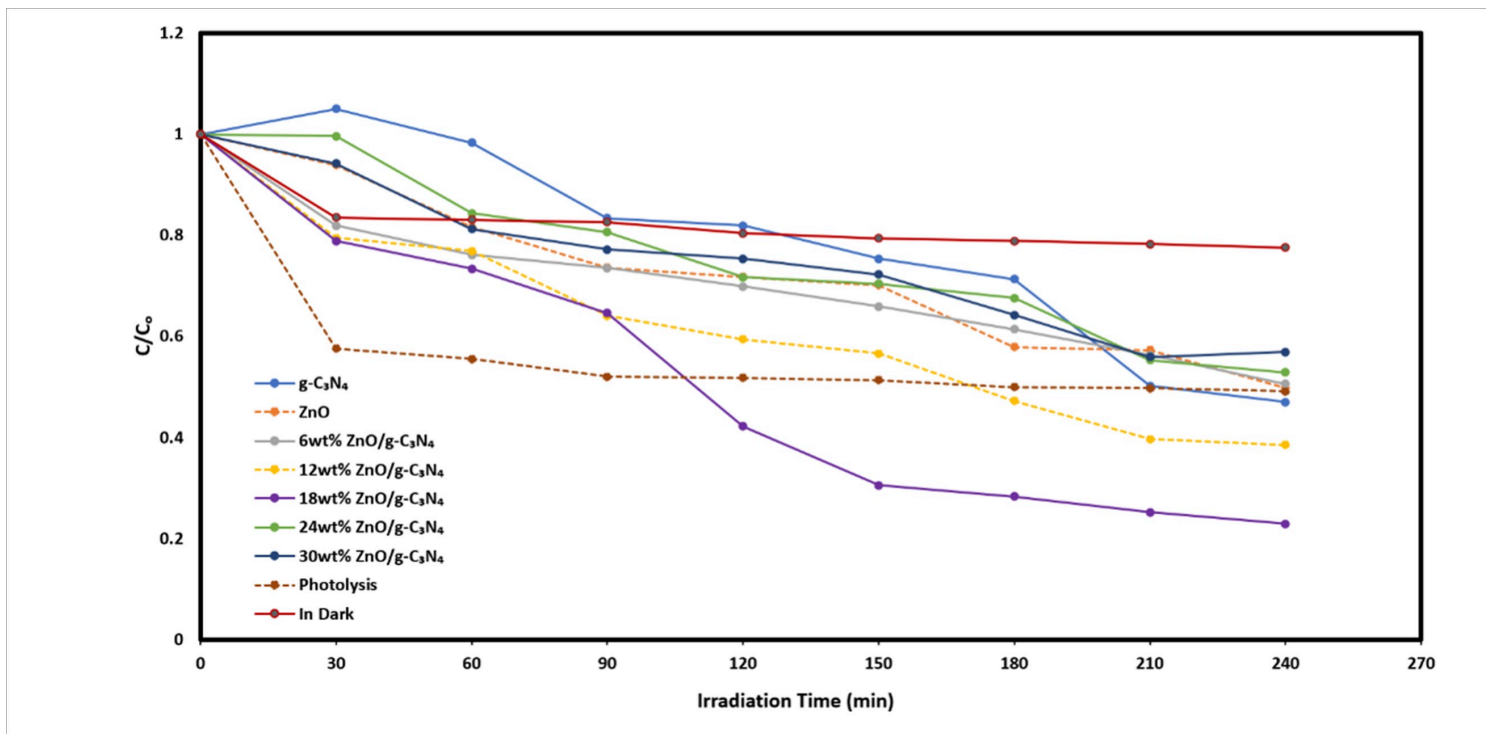


Figure 6

MB degradation using ZnO/g-C₃N₄ samples with different amount of ZnO.

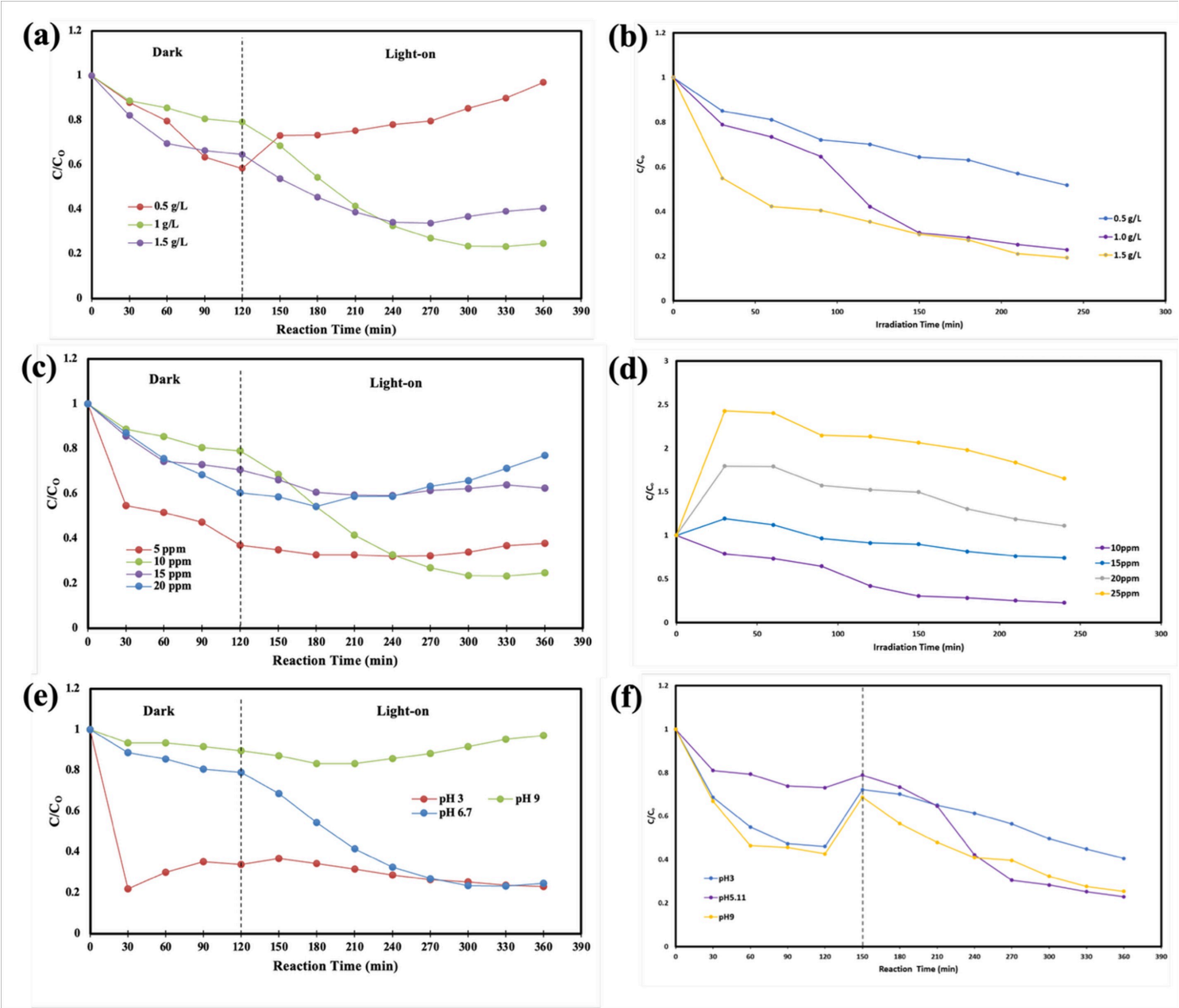


Figure 7

Effect of different (a) and (b) photocatalyst's amount, (c) and (d) initial concentration of dye and (e) and (f) pH of dye solution. The left and right panels are for MO and MB dyes, respectively.

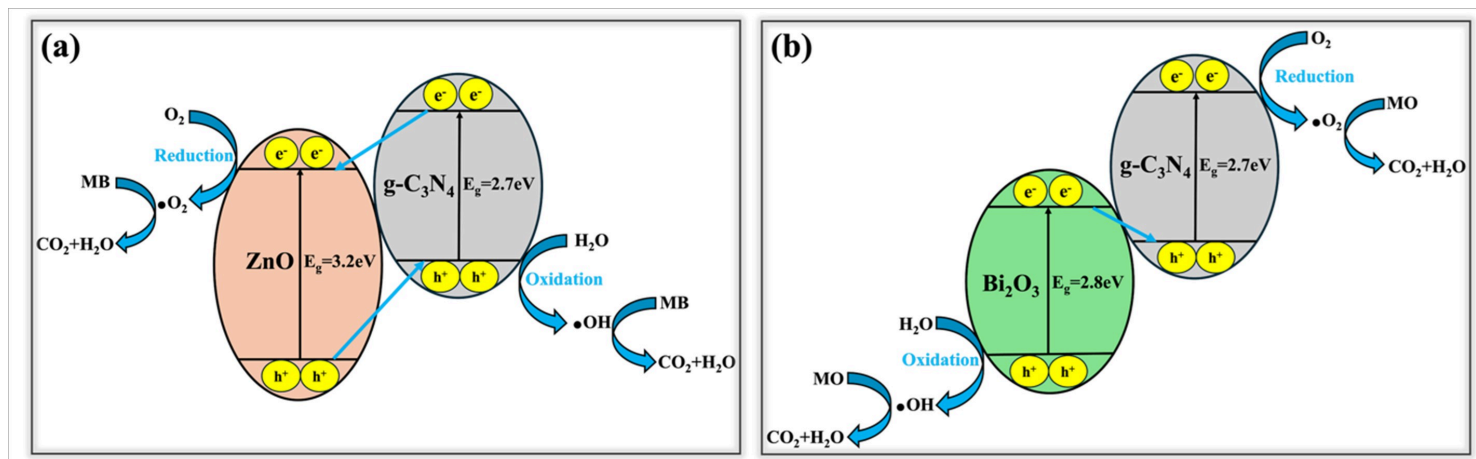


Figure 8

Charge transfer mechanisms of (a) Type II ZnO/g-C₃N₄ and (b) Z-scheme Bi₂O₃/g-C₃N₄ heterojunctions under light irradiation for photocatalytic dye degradation.