A metal-free C\(_{60}\)/CNTs/g-C\(_3\)N\(_4\) nanoheterostructure with excellent visible-light photocatalysis for rhodamine B (Rh B) degradation has been reported. Via a convenient low-temperature solution-phase method, g-C\(_3\)N\(_4\) nanosheets can serve as substrate for dispersion of C\(_{60}\)/CNTs. The loading of C\(_{60}\)/CNTs onto g-C\(_3\)N\(_4\) nanosheets surfaces significantly enhanced visible-light-driven photocatalytic activity of g-C\(_3\)N\(_4\) catalyst, for oxidation of organic pollutant (Rh B, 100%). Excellent photocatalytic properties of C\(_{60}\)/CNTs/g-C\(_3\)N\(_4\) can be predominantly attributed to the intimate interfacial contact among constructing compounds, increased specific surface area and enhanced light adsorption efficiency resulted from C\(_{60}\)/CNTs carbon materials. Particularly, the synergistic heterostructure interaction remarkably hinders the electron–hole pairs recombination, giving rise to significantly enhanced photocatalytic performance of C\(_{60}\)/CNTs/g-C\(_3\)N\(_4\) in comparison with other counterparts.

1. Introduction

Photocatalysis employing semiconductor materials has been considered as a promising method for widespread solar energy utilization and environmental remediation [1–3]. During the past few decades, many research works on photocatalysis field are mainly focused on the development of metal-oxide-based semiconductors, owing to their high thermal and chemical stability [4,5]. Nevertheless, the wide band gap and low quantum efficiency of some metal-oxide-based semiconductors significantly limit their potential applications.
as visible-light photocatalysts. Therefore, it is highly desirable to explore non-metal oxide photocatalysts for a wide range of prospective applications for visible-light photocatalysis [6].

Graphitic carbon nitride (g-C\textsubscript{3}N\textsubscript{4}), a metal-free polymeric photocatalyst, is considered as a multifunctional catalyst for photo/electrocatalytic water splitting, CO\textsubscript{2} reduction and organic pollutant degradation due to its appealing electronic structure, excellent chemical and thermal stabilities, low cost and environment benignity [7-9]. Nevertheless, g-C\textsubscript{3}N\textsubscript{4} has inherently low electronic conductivity and surface area, restricting its potential applications [10]. Construction of heterostructures by combining g-C\textsubscript{3}N\textsubscript{4} with semiconductor materials has been shown to be an efficient method to enhance the photocatalytic efficiency of g-C\textsubscript{3}N\textsubscript{4} [11-13]. Recently, carbonaceous materials such as activated carbon (AC), fullerene (C\textsubscript{60}), carbon nanotubes (CNTs) and graphene (GE) with the superiorities of large surface area, good electrical conductivity and stability, and non-toxicity have shown an ability to enhance the visible-light photocatalytic performance of g-C\textsubscript{3}N\textsubscript{4} [14-16]. For example, fullerene (C\textsubscript{60}) acting as electron acceptor might suppress the recombination of photo-generated electrons and holes in C\textsubscript{60}/g-C\textsubscript{3}N\textsubscript{4} composite, thus improving the photocatalytic efficiency [16]. In addition, Xu et al. have synthesized CNTs modified g-C\textsubscript{3}N\textsubscript{4} (CNTs/g-C\textsubscript{3}N\textsubscript{4}) composites via a hydrothermal method. The as-prepared CNTs/g-C\textsubscript{3}N\textsubscript{4} displayed higher photocatalytic activity for degradation of methylene blue (MB) than that of single g-C\textsubscript{3}N\textsubscript{4} [17]. The aforementioned results suggest that g-C\textsubscript{3}N\textsubscript{4} coupled with carbonaceous materials would greatly enhance its photocatalytic activity. Owing to the excellent light absorption ability and good electron conductivity of carbon materials, it can be speculated that concurrent decoration of g-C\textsubscript{3}N\textsubscript{4} with CNTs and C\textsubscript{60} would significantly enhance the photocatalytic activities of g-C\textsubscript{3}N\textsubscript{4}. However, to the best of our knowledge, there is no reported investigation focused on the photocatalytic activity of C\textsubscript{60}/CNTs/g-C\textsubscript{3}N\textsubscript{4} composite, and systematic study on the photocatalysis mechanism has not yet been reported.

Herein, we developed a homologous, metal-free, g-C\textsubscript{3}N\textsubscript{4}-based catalyst for photocatalytic application. It has been revealed that photocatalytic performance of g-C\textsubscript{3}N\textsubscript{4} can be conspicuously improved by loading C\textsubscript{60}/CNTs towards the degradation of rhodamine B (Rh B) under visible-light illumination. The loading of C\textsubscript{60}/CNTs contributes to the significantly enhanced photocatalytic activity of C\textsubscript{60}/CNTs/g-C\textsubscript{3}N\textsubscript{4} composite, and systematic study on the photocatalysis mechanism has not yet been reported.

### 2. Material and methods

#### 2.1. Preparation of photocatalysts

##### 2.1.1. Preparation of C\textsubscript{60}/CNTs

C\textsubscript{60}-decorated single-walled carbon nanotubes (C\textsubscript{60}/CNTs) was prepared by using the Zhang’ method [16]: SWCNTs (50 mg) and C\textsubscript{60} (50 mg) were dispersed by ultrasonication in toluene (100 ml) for 1 h, and then stirred at room temperature for 12 h. After volatilization of the toluene, the resultant black powder was washed with ethanol several times and dried in vacuum at 80°C for 10 h (denoted as C\textsubscript{60}/CNTs).

##### 2.1.2. Preparation of C\textsubscript{60}/CNTs/g-C\textsubscript{3}N\textsubscript{4}

C\textsubscript{60}/CNTs/g-C\textsubscript{3}N\textsubscript{4} ternary composite was fabricated via a facile hydrothermal method. Especially, C\textsubscript{60}/CNTs (0.05 g), g-C\textsubscript{3}N\textsubscript{4} (0.05 g) powders were added into ethanol solution (15 ml). The suspension was ultrasonicated for 1 h. The mixed solution was then transferred to a 25 ml Teflon-lined stainless steel autoclave reactor and heated at 180°C for 3 h. The C\textsubscript{60}/CNTs/g-C\textsubscript{3}N\textsubscript{4} sample was then collected. The C\textsubscript{60}/g-C\textsubscript{3}N\textsubscript{4} and CNTs/g-C\textsubscript{3}N\textsubscript{4} binary composite was synthesized through a similar method.

##### 2.2. Characterization of photocatalysts

The morphology of the samples was studied by SEM (JSM-7800F) and TEM (JEM-2100F). The crystal structure of the samples was tested with XRD (Rigaku, D/max 2500) equipped with K\textsubscript{α} radiation. Fourier transform infrared (FTIR) spectra of the samples were recorded on infrared spectrophotometer (America Perkin Elmer, Spectrum One). UV–vis diffuse reflection spectroscopy (DRS) was performed on a scan UV–vis spectrophotometer (SHIMADZU, UV-2550). The X-ray photoelectron spectroscopy
C60/CNTs/g-C3N4 composite. As shown in figure 2, g-C3N4 could be fitted into two peaks at 396.7 and 399.0 eV (figure 3). C60, the bands at 526, 575, 1180, 1428 and 1540 cm$^{-1}$ feature typical aromatic C–N heterocycle stretches, and the peaks at 811 cm$^{-1}$ are attributed to the tri-s-triazine units of g-C3N4 [23]. It also can be seen in figure 4(a) that for pure C60, the bands at 526, 575, 1180, 1428 and 1540 cm$^{-1}$ are attributed to the internal modes of the C60 molecule [16]. For C60/CNTs/g-C3N4, both the characteristic peaks of g-C3N4 and C60 can be clearly resolved, identifying the successful incorporation of C60 and g-C3N4 in the ternary composite (figure 4b).

Figure 5 shows the UV–vis diffuse reflectance spectra (DRS) of the as-fabricated samples. As can be seen, g-C3N4 demonstrated strong absorption ability in visible region. Visible-light harvesting ability of C60/g-C3N4 and CNTs/g-C3N4 was substantially enhanced with adsorption edge red shift to 500 and 550 nm, respectively. The result shows that integration of C60 or CNTs in the nanocomposites could improve the light harvesting efficiency of g-C3N4. In addition, the loading of C60/CNTs in the g-C3N4 leads to a further enhancement in the absorption edge (approx. 530 nm), which reveals the key role of CNTs for promoting the light harvesting and thus leading to the excitation of more electrons over the C60/CNTs/g-C3N4 nanocomposite under visible light [23].

The PL spectrum was employed to study the charge-carriers separation/recombination behaviour of photocatalysts. As shown in figure 5b, the PL emission peaks at around 460 nm for the samples reveals that the π-conjugated system of g-C3N4 was not destroyed by the modification treatment [24]. The decrease of PL intensity of C60/CNTs/g-C3N4 ternary nanocomposites in comparison with pure

2.3. Photocatalytic activities studies

Photocatalytic activities of the photocatalysts were evaluated by testing the degradation efficiencies of Rh B, using a Xe lamp (500 W), which was equipped with a UV irradiation source filter ($\lambda > 420$ nm). In a standard photocatalytic experiment, the photocatalyst (50 mg) was dispersed in a mixed aqueous solution of Rh B (50 ml, 0.01 mmol l$^{-1}$), and the suspension was stirred for 40 min in the dark to ensure that it reached the adsorption–desorption equilibrium. Then, the suspension was illuminated by visible light. At 5 min intervals, a 4.0 ml of the suspension was withdrawn, and centrifuged to remove the catalyst. The concentration of Rh B was analysed by UV–vis spectrophotometry.

3. Results and discussion

The phase and structure of photocatalysts were studied by XRD studies. As shown in figure 1, the diffraction peaks at 27.5° for g-C3N4 are detected in the pattern of g-C3N4. The characteristic peaks of the C60/g-C3N4 hybrid were respectively attributed to g-C3N4 (27.5°) [18], and C60 (10.7°, 17.7°, 20.7° and 21.7°), indicating the successful formation of the homogeneous hybrid structure [15]. Alternatively, there is also no difference observed between the C60/g-C3N4 and C60/CNTs/g-C3N4, probably due to low loading amount and the weak diffraction of CNTs [19,20]. Figure 2 depicts the microstructures of C60/CNTs/g-C3N4 composite. As shown in figure 2a, g-C3N4 has layered structure composed of tightly stacked g-C3N4 nanosheets with sizes of the thickness of tens of nanometres. As shown in figure 2bc, some CNTs with an average diameter of around 10 nm can also be observed, which are attached on the sheet-like g-C3N4. C60 molecules with a diameter of approximately 0.7 nm are too small to be directly resolved by TEM observation, which is similar to the previous report [16].

The XPS measurement was performed to determine the chemical composition and valence state of various species. The C 1s (figure 3a) high-resolution spectrum in C60/CNTs/g-C3N4 nanocomposite and g-C3N4 both have two distinct peaks at around 282.6 and 286.2 eV. The peak at 286.2 eV is identified as sp$^2$-bonded carbon (N–C=C). The peak located at 284.6 eV could be assigned to adventitious carbon, C–C bond from the C60 and graphitic carbon [16,21]. The high-resolution XPS spectrum for N 1s in the C60/g-C3N4 hybrid were respectively attributed to g-C3N4 (27.5°) [18], and C60 (10.7°, 17.7°, 20.7° and 21.7°), indicating the successful formation of the homogeneous hybrid structure [15]. Alternatively, there is also no difference observed between the C60/g-C3N4 and C60/CNTs/g-C3N4, probably due to low loading amount and the weak diffraction of CNTs [19,20]. Figure 2 depicts the microstructures of C60/CNTs/g-C3N4 composite. As shown in figure 2a, g-C3N4 has layered structure composed of tightly stacked g-C3N4 nanosheets with sizes of the thickness of tens of nanometres. As shown in figure 2bc, some CNTs with an average diameter of around 10 nm can also be observed, which are attached on the sheet-like g-C3N4. C60 molecules with a diameter of approximately 0.7 nm are too small to be directly resolved by TEM observation, which is similar to the previous report [16].

FTIR spectroscopy was further applied to identify the existence of C60 and g-C3N4 in the C60/CNTs/g-C3N4 composite (figure 4a,b). For pure g-C3N4 (figure 4a), the peaks at 1239, 1318, 1410, 1575 and 1633 cm$^{-1}$ are attributed to the internal modes of the C60/g-C3N4 [23]. It also can be seen in figure 4a that for pure C60, the bands at 526, 575, 1180, 1428 and 1540 cm$^{-1}$ are attributed to the tri-s-triazine units of g-C3N4 [23]. For C60/CNTs/g-C3N4, both the characteristic peaks of g-C3N4 and C60 can be clearly resolved, identifying the successful incorporation of C60 and g-C3N4 in the ternary composite (figure 4b).

Photocatalytic activities of the photocatalysts were evaluated by testing the degradation efficiencies of Rh B, using a Xe lamp (500 W), which was equipped with a UV irradiation source filter ($\lambda > 420$ nm). In a standard photocatalytic experiment, the photocatalyst (50 mg) was dispersed in a mixed aqueous solution of Rh B (50 ml, 0.01 mmol l$^{-1}$), and the suspension was stirred for 40 min in the dark to ensure that it reached the adsorption–desorption equilibrium. Then, the suspension was illuminated by visible light. At 5 min intervals, a 4.0 ml of the suspension was withdrawn, and centrifuged to remove the catalyst. The concentration of Rh B was analysed by UV–vis spectrophotometry.
Figure 1. XRD patterns of the as-prepared samples.

Figure 2. SEM images of the as-prepared samples: (a) g-C$_3$N$_4$, (b) C$_{60}$/CNTs/g-C$_3$N$_4$, (c) TEM image of the C$_{60}$/CNTs/g-C$_3$N$_4$.

g-C$_3$N$_4$ shows that recombination of photo-generated electrons and holes was inhibited with the loading of C$_{60}$/CNTs.

Full nitrogen adsorption isotherms of the as-fabricated samples were studied to gain the information about the specific surface area, as shown in figure 5c. The textural parameters of the photocatalysts are summarized in table 1. As can be seen, the loading of C$_{60}$ onto g-C$_3$N$_4$ or CNTs could lead to a significant
Figure 3. XPS spectra of the as-obtained samples: (a) C 1s and (b) N 1s.

Figure 4. (a, b) FTIR of the as-prepared samples.

decrease in the BET-specific surface, which may be due to the formation of C60 clusters in the mesopores of g-C3N4 or CNTs. It should be noted that the loading of CNTs onto g-C3N4 could lead to a significant increase in the BET-specific surface. In addition, the surface area of C60/CNTs/g-C3N4 composite is obviously smaller than those of CNTs/g-C3N4, which may be due to the addition of C60.

Figure 6a displays the photocatalytic activities of bare g-C3N4, C60, CNTs, C60/CNTs, CNTs/g-C3N4, C60/g-C3N4 and C60/CNTs/g-C3N4 for the degradation of Rh B solution. Only 27.0%, 6.0% and 23.0% of Rh B were removed by pure g-C3N4, C60, CNTs, respectively, after 60 min irradiation, while 41.0%, 55.0% of Rh B by C60/g-C3N4 and CNTs/g-C3N4, approximately. It means that C60 or CNTs loading is beneficial for the improvement of photocatalytic activity of the composite sample. Meanwhile, the photocatalytic efficiency of C60/CNTs also rose to 72.0%. The photocatalytic efficiency of C60/CNTs/g-C3N4 reached 100%, which is remarkably faster than the rate of bare g-C3N4, C60, CNTs, C60/CNTs, CNTs/g-C3N4 and C60/g-C3N4 for the degradation of Rh B solution. Only 27.0%, 6.0% and 23.0% of Rh B were removed by pure g-C3N4, C60, CNTs, respectively, after 60 min irradiation, while 41.0%, 55.0% of Rh B by C60/g-C3N4 and CNTs/g-C3N4, approximately. It means that C60 or CNTs loading is beneficial for the improvement of photocatalytic activity of the composite sample. Meanwhile, the photocatalytic efficiency of C60/CNTs also rose to 72.0%. The photocatalytic efficiency of C60/CNTs/g-C3N4 reached 100%, which is remarkably faster than the rate of bare g-C3N4, C60, CNTs, C60/CNTs, CNTs/g-C3N4 and C60/g-C3N4. It is due to that efficient heterostructure interface among three components that can restrain the recombination of photo-induced charges effectively [25]. In addition, the C60/CNTs coating can improve the visible-light absorption efficiency (figure 5a) and BET-specific surface (table 1) of the photocatalyst, which are both beneficial for the ternary composite to photolyse Rh B. In addition, the effect of C60 amount on the photocatalytic activity of the composites was also investigated (figure 6b). Furthermore, the highest degradation rate was obtained from C60(50 wt%)/CNTs/g-C3N4 sample with almost 100% of Rh B removal. This increase may be attributed to the capturing of electrons by the deposited C60 to hinder the recombination of hole-electron pairs [26,27], whereas the decrease may result from the blocking of visible light by the over-deposited C60. In order to further understand the photocatalytic mechanism of the ternary nanocomposite, the trapping experiment of active species was also investigated. As shown in figure 6c, isopropanol (IPA), triethanolamine (TEOA) and p-benzoquinone (BQ) were, respectively, introduced as the scavengers of hydroxyl radicals (·OH), holes (h+) and superoxide radicals (·O2−) to examine the effects of reactive
Figure 5. (a) DRS of the as-prepared samples. (b) PL spectra of the as-synthesized g-C₃N₄ and C₆₀/CNTs/g-C₃N₄. (c) N₂ adsorption–desorption isotherm curves of the as-prepared samples.

Table 1. BET-specific surface area of the as-prepared samples.

| photocatalysts | BET-specific surface area (m² g⁻¹) | photocatalysts | BET-specific surface area (m² g⁻¹) |
|---------------|-----------------------------------|---------------|-----------------------------------|
| CNTs          | 452.023                           | C₆₀/g-C₃N₄    | 30.231                            |
| C₆₀           | 177.399                           | CNTs/g-C₃N₄   | 254.202                           |
| g-C₃N₄        | 72.984                            | C₆₀/CNTs/g-C₃N₄ | 181.227                           |
| C₆₀/CNTs      | 277.847                           |               |                                   |

species on the photocatalytic degradation of Rh B. It can be seen that BQ and IPA led to a remarkable suppression of the degradation rate of Rh B, whereas EDTA-2Na exhibited a weaker restraining effect on the degradation rate. The results confirmed that ·OH and ·O₂⁻ play a more important role than h⁺ in the photocatalytic degradation of Rh B.

The stability and reusability of C₆₀/CNTs/g-C₃N₄ for photocatalytic degradation of Rh B were evaluated by repeating the photocatalytic experiments under the same conditions for three cycles. It is found that no obvious deactivation in the photocatalytic activity is detected for the C₆₀/CNTs/g-C₃N₄ composite after continuous visible-light irradiation (figure 6d). Figure 7 shows the SEM image of the C₆₀/CNTs/g-C₃N₄ sample after the photocatalytic reaction. After four catalytic runs, the secondary C₆₀/CNTs/g-C₃N₄ nanostructures were still very intact, clearly showing their stabilities. Thus, it represents a promising kind of composite catalyst for Rh B degradation under visible light.

Based on the above analysis, a schematic illustration of the photocatalysis mechanism of the C₆₀/CNTs/g-C₃N₄ is displayed in figure 8. Under visible-light illumination, photo-induced electrons are excited from the valence band (VB) of g-C₃N₄ to the conduction band (CB), resulting in electron–hole pairs. The CB of g-C₃N₄ (~1.12 eV versus NHE) is less negative than that of the CNTs (~0.3 eV versus NHE) or C₆₀ (~−0.2 eV versus NHE) [28,29]; therefore, photoelectrons from the CB of g-C₃N₄ are captured by the CNTs or C₆₀. As a result, the photoactivities of CNTs/g-C₃N₄, C₆₀/g-C₃N₄ have been
enhanced compared with the bare g-C$_3$N$_4$. For the C$_{60}$/CNTs/g-C$_3$N$_4$ nanocomposite, there are two possible ways for the transformation of photo-generated charges: (i) the electrons may firstly transfer to C$_{60}$ and then to CNTs, due to that, as the strong electron acceptor, the fullerenes can capture the electrons, which are spread and moved along the CNTs and fullerenes, thus facilitating the photo-generated carrier separation; (ii) the electrons in the CB of g-C$_3$N$_4$ may firstly transfer to CNTs, and then to C$_{60}$. Consequently, the carrier recombination can be effectively inhibited. In this way, the excited
Figure 8. Schematic diagram of the separation and transfer of photo-generated charges in the composite photocatalyst under visible-light irradiation.

electrons participate in the reduction of O₂ to \( \cdot \)O₂\(^{-}\). Subsequently, the \( \cdot \)O₂\(^{-}\) and \( \cdot \)OH oxidize the Rh B to CO₂ and H₂O.

4. Conclusion

In summary, we have demonstrated that C₆₀/CNTs/g-C₃N₄ composite was synthesized by a simple hydrothermal technology. The results showed that the C₆₀/CNTs/g-C₃N₄ nanocomposite exhibited the best photocatalytic activity under visible light, which is almost five times higher than that of pure g-C₃N₄. It shows that the ternary heterostructure can effectively hinder the recombination of the photo-generated electron–hole pairs, and thus improve the photocatalytic performance of the C₆₀/CNTs/g-C₃N₄ nanocomposite. In this work, the enhancement of photocatalytic activity of C₆₀/CNTs/g-C₃N₄ could be achieved via the loading C₆₀/CNTs, which may provide a promising class of photocatalyst candidates for photocatalytic application.

Data accessibility. The datasets supporting this article have been uploaded as the electronic supplementary material.

Authors’ contributions. Xu.L., R.Z. and Y.X. carried out the laboratory work, participated in data analysis, participated in the design of the study and drafted the manuscript; Xi.L. collected field data; J.S. and N.Y. conceived of the study, designed the study, coordinated the study and helped draft the manuscript. All authors gave final approval for publication.

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