



Article Visible Light Photoactivity of g-C₃N₄/MoS₂ Nanocomposites for Water Remediation of Hexavalent Chromium

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Abstract: Water pollution has becoming an increasingly serious issue, and it has attracted a significant amount of attention from scholars. Here, in order remove heavy metal hexavalent chromium (Cr (VI)) from wastewater, graphitic carbon nitride (g-C₃N₄) was modified with molybdenum disulfide (MoS₂) at different mass ratios via an ultrasonic method to synthesize g-C₃N₄/MoS₂ (CNM) nanocomposites as photocatalysts. The nanocomposites displayed efficient photocatalytic removal of toxic hexavalent chromium (Cr (VI)) from water under UV, solar, and visible light irradiation. The CNM composite with a 1:2 g-C₃N₄ to MoS₂ ratio achieved optimal 91% Cr (VI) removal efficiency at an initial 20 mg/L Cr (VI) concentration and pH 3 after 120 min visible light irradiation. The results showed a high pH range and good recycling stability. The g-C₃N₄/MoS₂ nanocomposites exhibited higher performance compared to pure g-C₃N₄ due to the narrowed band gap of the Z-scheme heterojunction structure and effective separation of photo-generated electron–hole pairs, as evidenced by structural and optical characterization. Overall, the ultrasonic synthesis of g-C₃N₄/MoS₂ photocatalysts shows promise as an efficient technique for enhancing heavy metal wastewater remediation under solar and visible light.

Keywords: $g-C_3N_4/MoS_2$ composites; Z-scheme heterojunction; hexavalent chromium Cr (VI); photocatalyst

1. Introduction

Water scarcity and water pollution have long been major global concerns; in recent decades, the rapid development of the economy and industrialization led to increasingly serious environmental pollution problems. Water quality has decreased since a large amount of industrial wastewater, which contained heavy metals, was discharged into the water system [1,2]. Chromium is one of the common sources of heavy metal pollution and mainly exists in Cr (III) and Cr (VI) in water [3]. Cr (III) is one of the essential elements in the human body, which can participate in the metabolism of human fat and is widely used in the adjuvant therapy of diabetes [4]. Cr (VI) poses a lasting threat to the environment and human health and can enter the human body through skin-to-skin contact or breathing. In addition, Cr (VI) has strong oxidation and can oxidize human hemoglobin into methemoglobin, which may cause cancer risk after long-term or short-term exposure [5,6]. Therefore, it is highly important that we find a way to handle Cr (VI) in industrial wastewater economically and efficiently and make it meet the discharge standard.



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Copyright: © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). At present, reducing Cr (VI) to Cr (III) in wastewater is an important way of alleviating chromium pollution in water [7,8], and common methods for treating chromecontaining heavy metal wastewater include the adsorption method [9,10], chemical reduction method [11,12], and biological method [13,14]. In the process of Cr (VI) removal, these methods consume a large amount of power and other resources, have high costs, and may cause other forms of pollution. However, existing treatment methods for the removal of pollutants in the process of treatment have complexity and high costs, and they are prone to secondary pollution and other shortcomings.

Photocatalytic technology is widely used [15–17], which has the advantages of no secondary contamination and strong redox capacity and is widely used in pollutant degradation [18,19], hydrogen generation [20,21] and CO_2 photoreduction [22]. It is one of the best ways to solve future pollution problems [23]. Bi-bridge S-scheme $Bi_2S_3/BiOBr$ heterojunction (Bi₂S₃/Bi/BiOBr), produced by the one-pot solvothermal method, has shown high visible light photocatalytic reduction performance, and the removal efficiency of Cr (VI) was 97% [24]. Graphitic carbon nitride $(g-C_3N_4)$'s band gap is 2.7 eV; it is a common photocatalyst with high stability and environmental friendliness [25–27]. Currently, thermal polycondensation is commonly used in the preparatory work of $g-C_3N_4$, which makes the preparation of $g-C_3N_4$ simple [28–30]. However, due to the rapid recombination rate of photo-generated electrons and holes and the low light absorption range and surface range [31–33], the photocatalytic performance of g-C₃N₄ photocatalysts is low, which makes the use of g-C₃N₄ limited. Numerous researchers have found that the photocatalytic performance of $g-C_3N_4$ could be improved by conducting morphology regulation [34,35], ion doping [36–38], and heterojunction construction [39–41]. Li et al. [42] prepared ultrathin tubular lateral heterostructures (LHSs) of graphitic carbon nitride and carbon dots (CN/C-Dots) by one-step thermal polymerization; they found that the CN/C-Dots LHSs exhibited excellent electrocatalysts for a hydrogen evolution reaction, due to which the charge carriers' transport was enhanced and the specific surface area was increased, meaning more active sites of CN. Renji Rajendran et al. [43] developed a g- $C_3N_4/TiO_2/\alpha$ -Fe₂O₃ ternary magnetic nanocomposite with a Z-scheme by facile calcination and a hydrothermal process. The g- $C_3N_4/TiO_2/\alpha$ -Fe₂O₃ ternary magnetic nanocomposite exhibited excellent photocatalytic performance for the degradation of Rhodamine B (RhB); under visible light exposure, the degradation rate was 95.7%, which was due to the formation of the Z-scheme enhancing the separation and migration of photoexcited electron and hole pairs and the light absorption range. Photocatalysts have been widely used for various purposes. However, photocatalysts still have the shortcomings of low utilization of visible light and high requirements for reaction conditions. MoS₂ has attracted attention as a transition metal dichalcogenide with good chemical stability and adjustable bandwidth [44].

In this work, $g-C_3N_4/MoS_2$ samples with different mass ratios were prepared by the ultrasonic method, and the photocatalytic removal efficiencies of Cr (VI) under different light irradiation sources (ultraviolet light, solar light and visible light) were investigated. $g-C_3N_4/MoS_2$ composites showed strong photocatalytic activity. When the pH value was 3, the initial concentration of Cr (VI) was 20 mg/L, and the photocatalyst demonstrated strong photocatalytic activity. Compared with pure $g-C_3N_4$, the doping of MoS_2 is beneficial for narrowing the band gap and reducing the recombination rate of photo-generated electrons and holes, and the photocatalytic performance of CNM (1:2) increased. The composite photocatalyst has a wide pH range and can still show a high removal rate after multiple reuses, overcoming the shortcomings of existing difficult-to-recover photocatalysts.

2. Results and Discussion

2.1. Characterization

2.1.1. XRD

The X-ray diffraction (XRD) pattern characterization of $g-C_3N_4$, MoS₂, and CNM (1:2) is shown in Figure 1. $g-C_3N_4$ had diffraction peaks at 13.1° and 27.6°, which corresponded to the (100) and (002) planes, respectively [45]. The characteristic diffraction peaks of

13.9°, 32.8° and 58.6° were attributed to the (002), (100), and (110) planes, respectively [46]. Compared with g-C₃N₄ and MoS₂, there is a shift in the peak position of the CNM (1:2) composites, which may be caused by the increased interlayer distance of the defect modified samples [47]. The diffraction peaks of the CNM (1:2) composites were 13.3°, 27.9°, 32.8° and 57.6°, respectively. These were attributed to (100), (002), (100), and (110). The CNM (1:2) composites showed diffraction peaks belonging to g-C₃N₄ and MoS₂, which indicated the MoS₂ had successfully combined with g-C₃N₄.



Figure 1. XRD patterns of g-C₃N₄, MoS₂, and CNM (1:2).

2.1.2. SEM

Scanning electron microscopy (SEM) images of g-C₃N₄ and g-C₃N₄/MoS₂ are shown in Figure 2. Figure 2a,b show SEM images of $g-C_3N_4$ and CNM (1:2), $g-C_3N_4$ had a massive structure, which was combined with a layered structure. CNM (1:2) had a porous structure, the reason for this result may be that during the ultrasonic treatment, the $g-C_3N_4$ was stripped and combined with MoS₂, and a large number of pore structures were formed in the process. Ultrasonic treatment can effectively promote g-C₃N₄ and MoS₂ recombination, effectively accelerate the separation of photo-generated carriers, and improve the photo-generated carrier migration rate, thus improving the photocatalytic activity of composite photocatalytic materials. The CNM (1:2) complex can accelerate the separation of photo-generated carriers and increase the migration rate of photo-generated carriers, thus improving the photocatalytic activity of the composite photocatalyst. Figure 2c-h reveal CNM (1:2) as well as corresponding elemental mapping of C, N, S, and Mo, and EDS of CNM (1:2). The mapping of SEM confirmed that the elements of C, N, S, and Mo exist in CNM (1:2), and it indicated that g-C₃N₄ and MoS₂ had been successfully combined. In the process of MoS_2 doping g-C₃N₄, a g-C₃N₄/MoS₂ photocatalyst with more voids was formed, which increased the area of the photocatalyst in contact with pollutants, and increased the number of active sites on its surface, thus increasing its photocatalytic activity.

2.1.3. XPS

The X-ray photoelectron spectroscopy (XPS) spectra of and CNM (1:2) and used CNM (1:2) are displayed in Figure 3, and the spectra of $g-C_3N_4$ were shown in previous articles [30]. The survey spectra of CNM (1:2) and used CNM (1:2) are shown in Figure 3a, the main elements are C, N, O, S and Mo, indicating $g-C_3N_4$ and MoS₂ were successfully combined. In Figure 3b, the C 1s spectrum of CNM (1:2) has three peaks at 288.2, 286.4, and 284.8 eV; the 288.2 eV is attributed to O-C-N, the 286.4 eV is attributed to C-O, and the 284.8 eV belongs to C-C [48]. In the C 1s spectrum of used CNM (1:2), the peaks shifted

to 288.8, 286.4 and 284.8 eV, respectively [49]. The N 1s of CNM (1:2) has three peaks at 404.3, 400.3 and 398.5 eV, which are attributed to N-H bonds, N-(C)₃, and C = N-C [50]. Additionally, the N 1s peaks of the used CNM (1:2) were shifted to 404.5 eV (N-H), 401.3 eV (C-N-H), and 399.2 eV (N-(C)₃) [51], as shown in Figure 3c. The Mo 3d of CNM (1:2) had four peaks at 235.1, 231.5, 228.1 and 225.4 eV, with the peaks corresponding to Mo⁶⁺, Mo $3d_{3/2}$, Mo $3d_{5/2}$ and S 2s, attributed to the 1T-phase MoS₂ [52,53]. In the Mo 3d spectrum of used CNM (1:2), the peaks shifted to 235.8, 232.3, 228.9, and 226.2 eV, respectively [45]. Figure 3e shows the S 2p spectra of CNM (1:2); it has three peaks at 168.2, 162.2, and 161 eV, which correspond to $S_2^{2^-}$, S $2p_{1/2}$, and S $2p_{3/2}$ [53]. In the S 2p spectrum of used CNM (1:2), those peaks shifted to 169.1, 162.9, and 161.7 eV [45]. In Figure 3f, the presence of Cr was not detected on the surface of the reused photocatalysts, which ensures that the active sites on the surface of the photocatalyst were not covered. The XPS characterization results again confirm that both g-C₃N₄ and MoS₂ have been successfully compounded, and Cr was not detected in the reused CNM (1:2), thus ensuring the excellent reusable performance of the photocatalyst.



Figure 2. SEM images of samples: (a) g-C₃N₄, (b) CNM (1:2), (c) SEM image of CNM (1:2), and corresponding elemental mapping of (d) C, (e) N, (f) S, and (g) Mo. (h) EDS spectrum of CNM (1:2).



Figure 3. XPS spectra of CNM (1:2) and used CNM (1:2): (**a**) Survey spectra, (**b**) C 1s, (**c**) N 1s, (**d**) Mo 3d, (**e**) S 2p, (**f**) Cr 2p.

2.1.4. BET

The Brunauer–Emmett–Teller N₂ adsorption–desorption isotherms of g-C₃N₄, MoS₂ and CNM (1:2) are shown in Figure 4. The specific surface area and pore volumes of g-C₃N₄, MoS₂, and CNM (1:2) are listed in Table 1. The specific surface area of CNM (1:2) (30.7214 m²·g⁻¹) is higher than that of g-C₃N₄ (27.3882 m²·g⁻¹) and MoS₂ (10.5045 m²·g⁻¹). In addition, we found that the pore volumes of g-C₃N₄ (0.2385 cm³·g⁻¹) and MoS₂ (0.0332 cm³·g⁻¹) are lower than that of CNM (1:2) (0.3498 cm³·g⁻¹). The increase in specific surface area and pore volume is beneficial to providing more photocatalytic active sites and improving the activity of CNM (1:2).

Table 1. Specific surface area and pore volumes of g-C₃N₄, MoS₂, and CNM (1:2).

Samples	S_{BET} (m ² ·g ⁻¹)	$V_{Pore} (cm^3 \cdot g^{-1})$
g-C ₃ N ₄	27.3882	0.2385
MoS ₂	10.5045	0.0332
CNM (1:2)	30.7214	0.3498



Figure 4. N₂ adsorption-desorption isotherms of g-C₃N₄, MoS₂ and CNM (1:2).

2.1.5. UV-Vis Diffuse Reflectance Spectra

UV–vis diffuse reflectance spectra images of g-C₃N₄ and CNM (1:2) are displayed in Figure 5a. g-C₃N₄ had an absorption edge at 450 nm, while the absorption edge of CNM (1:2) was redshifted, and the absorption range was 200–800 nm. This indicates that the doping of MoS₂ could effectively improve the utilization ratio of the photocatalyst in visible light, and the light absorption of the photocatalyst was enhanced, thus achieving the purpose of improving the photocatalytic activity of CNM (1:2). The plots of the transformed Kubelka–Munk function versus the photon energy of g-C₃N₄ and CNM (1:2) are shown in Figure 5b. The value of the band gap can be calculated according to the formula $(ahv)^{1/2} = A(hv-Eg)$, $hv = hc/\lambda$; the results show that the band gaps of g-C₃N₄ and CNM (1:2) are 2.72 and 2.31 eV, respectively. The doping of MoS₂ is beneficial to narrowing the band gap and reducing the recombination rate of photo-generated electrons and holes.



Figure 5. (a) UV–vis diffuse reflectance spectra. (b) Plots of the transformed Kubelka–Munk function versus the photon energy of $g-C_3N_4$ and CNM (1:2).

2.1.6. PL Spectra

The separation and transfer ability of photo-excited carriers in the $g-C_3N_4$, MoS₂ and CNM (1:2) are measured with PL spectra. As displayed in Figure 6, the $g-C_3N_4$ and CNM (1:2) exhibited broad peaks at 435 nm. $g-C_3N_4$ showed a stronger fluorescence emission peak intensity, which indicates the higher recombination efficiency of the electron–hole pairs. After adding MoS₂, the CNM (1:2) showed a low PL intensity, the carrier separation efficiency was enhanced, and the electron pairs' separation rate was reduced in the CNM (1:2) nanocomposite. Therefore, the results confirmed the photocatalytic performance of CNM (1:2) increased.



Figure 6. PL spectra of g-C₃N₄, MoS₂, and CNM (1:2).

2.1.7. Transient Photocurrent Responses

The separation and transfer of photo-excited carriers on $g-C_3N_4$ MoS₂ and CNM (1:2) were investigated. The transient photocurrent spectrum of the samples is shown in Figure 7. The CNM (1:2) showed the highest photocurrent density during the samples; it showed that the separation efficiency of the photogenerated electrons and holes is significantly improved after the doping MoS₂. This result confirms that CNM (1:2) can effectively reduce the recombination rate of electrons and holes, thereby increasing the photocatalytic activity of the photocatalysts.



Figure 7. Transient photocurrent responses of $g-C_3N_4$ MoS₂ and CNM (1:2).

2.2. Photocatalysis

2.2.1. Photocatalytic Performance of Different Photocatalysts

The removal rates and the pseudo-first-order reaction kinetics of Cr (VI) by g-C₃N₄, MoS₂, CNM (1:1), CNM (1:2), CNM (1:4), CNM (2:1), and CNM (4:1) were shown in Figure 8a,b. In order to select the optimal ratio of g and m, we prepared photocatalysts with different mass ratios, as shown in Figure 8a, the results show that doping with different MoS₂ masses has a great influence on the photocatalytic activity of the photocatalysts. When the mass ratio of g-C₃N₄ to MoS₂ was 2:1 (CNM (1:2)), it had the best rate of removal of Cr (VI). At the same time, the pseudo-first-order reaction kinetics model shows that the reaction rates of g-C₃N₄, MoS₂, CNM (1:1), CNM (1:2), CNM (1:4), CNM (2:1) and CNM (4:1) are, respectively, 0.0006, 0.0010, 0.0028, 0.0102, 0.0052, 0.0022, and 0.0018 min⁻¹. It was found that when the content of MoS₂ was too high, the removal efficiency of Cr (VI) was decreased. Excessive MoS₂ made the charge transfer rate too fast, which increases the recombination probability of photo-generated electrons and holes; therefore, the photocatalytic activity decreased.



Figure 8. (a) The removal of Cr (VI) by different photocatalysts. (b) Pseudo-first-order reaction kinetics. (c) Photocatalytic removal of Cr (VI) under ultraviolet light. (d) Photocatalytic removal of Cr (VI) under solar light. (e) Effect of pH. (f) Effect of initial concentration. (g) Effect of photocatalytic dosage. (h) Recyclability of CNM (1:2).

In order to investigate the removal ability of photocatalysts, we explored the Cr (VI) removal rate of photocatalysts under different forms of light illumination (ultraviolet light, solar light, and visible light), and the results displayed in Figure 8c,d. The removal rate of Cr (VI) by CNM (1:2) was 91.6% under the illumination of ultraviolet light, and the

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removal rate of Cr (VI) was 91% and 86% under solar light and visible light, respectively. The results revealed that with the different forms of light irradiation, the photocatalytic removal performance of Cr (VI) had a weak effect. At the same time, we investigated the effects of pH value, initial concentration, and dosage on the removal rate (in Figure 8e–g), The experimental results show that with the increase in the pH value, the reducibility of the photocatalyst to Cr (VI) decreases gradually. When the solution is neutral or alkaline, the removal rate of Cr (VI) reduced to 40%, but the removal rate is as high as about 65% under weak acid conditions, which affirms that photocatalysts have a high application range. The stability of the photocatalysts was experimentally assessed three times under visible light (in Figure 8h), and the results showed that the CNM (1:2) photocatalysts still had strong photocatalysts that can only remove Cr (VI) under strong acid conditions and provides data support for the practical engineering application of photocatalysts in the future.

2.2.2. Scavenging Study

In order to investigate the main reactive radicals in the reactions, scavenger tests were performed and the results are displayed in Figure 9a. In this study, ethylenediaminete-traacetic acid disodium salt (EDTA–2Na), potassium persulfate ($K_2S_2O_8$), and ascorbic acid ($C_6H_8O_6$) were used as the scavenger for scavenge holes (h⁺), electrons (e⁻), and superoxide radicals ($\cdot O_2^{-}$), respectively. The test results showed that the removal of Cr (VI) by the CNM (1:2) photocatalyst was significantly decreased after the addition of $K_2S_2O_8$, which implied that the e⁻ played an important role in the removal of Cr (VI), and the results displayed that the addition of ascorbic acid and EDTA-2Na have a slight influence on the removal of Cr (VI), which shows that $\cdot O_2^{-}$ and h⁺ do not play a key role in the reaction.



Figure 9. (a) The effect of reactive scavengers. (b) Variation in Cr valence at different reaction times.

To evaluate the reaction mechanism of CNM (1:2)'s photocatalytic reduction of Cr (VI), an inductively coupled plasma–mass spectrometer (ICP-MS) was used to analyze the concentration of chromium. Ultraviolet-visible spectrophotometry was performed to measure the concentration of Cr (VI), which can determine the valence state change of chromium during the reaction (Figure 9b). The results show that the content of total chromium did not change with the increase in reaction time, but the concentration of Cr (VI.) decreased with the increase in light time. The presence of chromium in aqueous solution mainly includes Cr (III) and Cr (VI), from which it can be inferred that with the increase in reaction time, Cr (VI) in water is reduced to Cr (III). The content of total chromium in the solution remains unchanged, which indicates that the Cr (VI) in the water is reduced to Cr (III) by photocatalysis during the reaction process. Chromium is mainly present in water in the form of minimally toxic Cr (III) instead of being adsorbed on the surface of the photocatalyst; this means it will not form a buildup on the surface of the material and affect the performance of the photocatalyst, which is beneficial for recycling of photocatalysts.

2.2.3. Mechanisms of Photocatalysis

A possible photocatalytic mechanism of CNM (1:2) removal Cr (VI) is shown in Figure 10. Additionally, the figure shows that under the excitation of light, a large number of electron and hole pairs are generated on the surface of photocatalysts, which greatly increases the activity of the photocatalyst for pollutant removal [54]. The band gap of CNM (1:2) was significantly narrower than that of the $g-C_3N_4$ and MoS_2 , which is due to the photoexcited electrons' (e⁻) transition from the conduction band (CB) of $g-C_3N_4$ to the CB of MoS_2 , and the holes' (h⁺) transition from the valence band (VB) of MoS_2 to the VB of $g-C_3N_4$, which is beneficial to the narrowing of the band gap and reduces the recombination rate of photo-generated electrons and holes. The photocatalyst can absorb more energy under the same light conditions and be excited to generate more photo-generated electronhole pairs, thus improving the photocatalytic performance of CNM (1:2) and enhancing the removal rate of Cr (VI) by CNM (1:2). This is due to Z-scheme heterojunction formed between $g-C_3N_4$ and MoS_2 [44]. The reaction equation is shown in Equations (1)–(4).

$$g-C_3N_4 + hv \to g-C_3N_4 (e^- + h^+)$$
 (1)

$$g-C_3N_4 + MoS_2 \rightarrow g-C_3N_4 + MoS_2(e^-)$$
⁽²⁾

$$MoS_2 (e^-) + O_2 \rightarrow MoS_2 + O_2^-$$
(3)

$$\operatorname{Cr}(\operatorname{VI}) + e^{-} \to \operatorname{Cr}(\operatorname{III})$$
 (4)



Figure 10. The mechanism for removal of Cr (VI) by CNM (1:2).

3. Materials and Methods

3.1. Materials

Thiourea (H₂NCSNH₂, AR) was purchased from Hengxing chemical preparation(Tianjin, China), ammonium molybdate ((NH₄)₆Mo₇O₂₄·4H₂O, AR) was acquired from Taishan Chemical Plant (Shandong, China), urea (H₂NCONH₂, AR) was obtained from Tianjin Zhiyuan chemical reagent co(Tianjin, China), ethylenediaminetetraacetic acid disodium salt (EDTA-2Na), potassium persulfate (K₂S₂O₈), ascorbic acid (C₆H₈O₆), hydrochloric acid (HCL), and sodium hydroxide (NaOH) were obtained from Xilong scientific (Guangzhou, China).

3.2. Preparation of Photocatalysts

3.2.1. Preparation of $g-C_3N_4$

 $g-C_3N_4$ was prepared by the thermal polymerization method. Then, 20 g of urea were loaded into a crucible and wrapped in tin foil, with a 5 °C·min⁻¹ heating rate, before being kept at 550 °C for 4 h. After cooling to room temperature, the yellow $g-C_3N_4$ was obtained, using an agate mortar grind to obtain the powdered $g-C_3N_4$.

3.2.2. Preparation of MoS₂

The MoS₂ was prepared by the hydrothermal method. Then, 0.4 g ammonium molybdate ((NH₄)₆Mo₇O₂₄·4H₂O) and 0.8 g thiourea (H₂NCSNH₂) were added to 10 mL of deionized water, stirred for 30 min, and ultrasound-treated for 30 min. The solution was transferred to a hydrothermal reactor and heated for 10 h at 200 °C, after which the solution was cooled to room temperature and strained and washed with deionized water and anhydrous ethanol 3 times, aiming to remove any impurities. The samples were kept at 60 °C for 12 h; the black MoS₂ was obtained using an agate mortar to grind it into a powder (which was bagged for later use).

3.2.3. Preparation of g-C₃N₄/MoS₂ with Different Mass Ratios

 $g-C_3N_4/MoS_2$ with different mass ratios was prepared by the ultrasonic method. The 0.2 g $g-C_3N_4$ and 0.4 g MoS₂ were added to 400 mL of deionized water and ultrasound-treated for 180 min. The $g-C_3N_4/MoS_2$ was obtained and denoted CNM (1:2); the CNM (1:4), CNM (2:1), CNM (4:1), and CNM (1:1) were obtained in the same way, and the different samples were obtained by changing the mass ratios of $g-C_3N_4$ and MoS_2 , as shown in Figure 11.





3.3. Characterization of Photocatalysts

The crystal structures of the photocatalysts were characterized by X-ray diffraction (XRD, Rigaku SmartLab SE, Rigaku Corp., Tokyo, Japan) with Cu-k α radiation. The morphology and structure of the photocatalysts were determined using scanning electron microscopy (SEM, TESCAN MIRA LMS, TESCAN CHINA, Ltd., Shanghai, China). X-ray photoelectron spectroscopy (XPS, Thermo Scientific k-Alpha, Thermo Fisher Scientific Co., Ltd., Shanghai, China) was used to characterize the elemental composition and valence state of the photocatalysts. The optical properties of photocatalysts were measured using a UV–visible spectrophotometer (UV–vis, UV-3600i plus, Shimadzu Corp., Kyoto, Japan). The surface area and pore size of photocatalysts were tested by the Brunauer–Emmett–Teller method (BET, Micromeritics ASAP2460, Micromeritics Corp., Norcross, GA, USA). The steady and transient photoluminescence spectra of photocatalysts were assessed using a fluorescence spectrometer (PL, FLS980, Edinburgh Instruments Ltd., Shanghai, China). The metal element content was tested using an inductively coupled plasma–mass spectrometer (ICP-MS, PerkinElmer NexION 2000, Perkinelmer, MS, USA).

3.4. Photocatalytic Tests

The photocatalytic performance of g-C₃N₄, CNM (1:2), CNM (1:4), CNM (2:1), CNM (4:1) and CNM (1:1) was tested in the photoreactor. A 300 W Xe lamp supported by CEL-LAM with a cutoff filter (λ > 420 nm) was used as the source of solar light and visible light. CEL-LAM 500 was the source of UV light, and the reaction took place at room temperature. A 10 mg photocatalyst was put into the 100 mL Cr (VI) solution (20 mg·L⁻¹), and the pH

value was 3. The photocatalyst and the 20 mg·L⁻¹ Cr (VI) solution were stirred in the dark for 30 min to achieve adsorption–desorption equilibrium, and 2 mL of solution was taken each time to analyze the concentration of Cr (VI). After turning on the light source, samples were taken at 20, 40, 60, 80, 100, and 120 min, respectively. The same 2 mL of solution was taken at a time and filtered using a biofilter membrane (0.45 μ m) to obtain a filtrate without photocatalyst. We then determined the water quality and amount of chromium (VI)-1,5 dtphenylcarbohydrazide using a spectrophotometric method (GB 7467-87) [55] to analyze the concentration of hexavalent chromium. The removal rate (%) was calculated according to Equation (5):

removal rate (%) =
$$\frac{C_0 - C_t}{C_0} \times 100\%$$
 (5)

 C_0 : the initial concentration of Cr (VI); C_t : the concentration of Cr (VI) at the corresponding time.

4. Conclusions

Z-scheme $g-C_3N_4/MoS_2$ nanocomposite heterojunctions were successfully synthesized using an ultrasonic method and demonstrated efficient photocatalytic removal of toxic Cr (VI) from water under UV, visible, and solar light irradiation. The nanocomposites, especially those with the optimized 1:2 $g-C_3N_4/MoS_2$ ratio, exhibited enhanced photoactivity compared to pure $g-C_3N_4$, with over 90% Cr (VI) removal achieved. The superior performance is attributed to the combined effects of the narrowed heterostructure band gap, which enables visible light response, and the effective separation of photo-generated electron-hole pairs at the interfaced junction between the two semiconductors. The results showed that the MoS_2 could be located at the g- C_3N_4 , which was beneficial for the enhancement of photocatalytic activity, owing to the g-C₃N₄/MoS₂ nanocomposites having a broad range of light response and the separation and transfer efficiencies of photo-generated electron-hole pairs being improved. Overall, this work highlights the promise of ultrasonically synthesized $g-C_3N_4/MoS_2$ nanocomposites for tackling the pressing environmental challenge of heavy metal wastewater treatment using solar-driven photocatalysis. Further optimization to translate this efficient lab-scale Cr (VI) remediation to real-world applications should be pursued.

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