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Facile in-situ synthesis of 2D/3D g-C₃N₄/Cu₂O heterojunction for high-performance photocatalytic dye degradation

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Abstract

Constructing heterojunction is a promising method to enhance visible-light utility and promote photogenerated e⁻−h⁺ pairs separation of g-C₃N₄. Herein, the present work reported a g-C₃N₄/Cu₂O heterojunction by a facile in situ synthetic strategy. The structure, morphology, optical, electrochemical and photocatalytic properties were investigated. The results showed the combination of g-C₃N₄ nanosheets and Cu₂O nanospheres resulted in the close contact interface and wide visible-light response range, and fast separation of the photo-generated e⁻−h⁺ pairs. The pristine g-C₃N₄ showed weak photocatalytic performance towards methyl orange (MO) under visible-light irradiation, compared with pristine g-C₃N₄, the photocatalytic degradation rate of optimized g-C₃N₄/Cu₂O composite demonstrates the substantially enhanced photocatalytic activity, which showed no obvious loss after five repeated use. The enhanced performance was ascribed to the synergistic effect of good visible-light harvesting and enhanced separation rate of the charge carriers.

1. Introduction

The past few decades have witnessed increasing attention to address the global issues of environmental pollution, owing to its negative impact on the sustainable development of human society [1–3]. However, the limitations (especially the potential secondary pollution and high cost) for dye treatment impede the widespread applications of conventional strategies, for example of chemical oxidation [4], physical adsorption [5], biological treatments [6]. Compared with these traditional strategies, semiconductor-based photocatalysis is regarded as the state-of-the-art technology for environmental remediation owing to its eco-friendly features, recyclability and efficient [7, 8]. Although great progress has been made in photocatalysis technology, the fast recombination rate of photo-generated charge carriers and narrow sunlight response range are still regarded as hot research spots in the field.

To expand the sunlight response range, researchers are eagerly sought photocatalysts with narrow bandgap. Recently, two-dimensional (2D) g-C₃N₄ nanosheet with layered structure has earned great attention because of its easy syntheses, 2D layered structure, controllable electronic energy structure, suitable bandgap of 2.7 eV and high stability [9]. Nonetheless, as a photocatalyst, the practical use and photocatalytic performance of pure g-C₃N₄ still suffered from fast recombination rate of photogenerated e⁻−h⁺ pairs and relatively narrow visible light response range. Therefore, continuous efforts has been underway to enhance the photocatalytic efficiency of g-C₃N₄, for example, incorporating another semiconductor with matched bandgap structure [10], metal or non-metal doping [11, 12] and noble metal deposition etc [13].

Generally, constructing heterojunctions by integrating g-C₃N₄ with other narrow bandgap semiconductors is regarded as the most promising method to increase its visible light response range, promote the photogenerated e⁻−h⁺ pairs separation as well as to extend their lifetime, and thus leading to improved photocatalytic activity [14, 15]. Among suitable semiconductors for constructing heterojunctions with g-C₃N₄, Cu₂O has been widely considered as an efficient cocatalyst coupled with semiconductors with wide band

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structure due to its direct band gap of 2.0 eV, abundance, non-toxicity and low cost \cite{16-19}. For pristine g-C₃N₄, the inherent drawback of fast photogenerated $e^–h^+$ pairs recombination rate can be overcome by coupling with Cu₂O. Besides, modifying and fabricating the suitable interface is also an important measure to enhance photocatalytic activity. Recently, 2D nanosheets and 3D nanospheres have earned much attention for their specific configuration, large specific surface area and high photocatalytic activity \cite{20, 21}. Moreover, integrating 2D and 3D can be used as a way to increase the contact interaction of miscellaneous nanomaterials \cite{22}. Therefore, it is a crucial procedure to fabricate the composite photocatalyst of 2D g-C₃N₄ nanosheets and 3D Cu₂O nanospheres. Until now, the 2D/3D g-C₃N₄/Cu₂O heterojunction with relatively evident benefits has been successfully reported \cite{23}. However, a high efficient method in constructing the heterojunction is still needed.

Herein, we propose a facile and in situ strategy to engineer heterojunction of 2D g-C₃N₄ nanosheets and 3D Cu₂O nanospheres by a two-step thermal-etching process following with hydrothermal synthesis method. The synthesized g-C₃N₄/Cu₂O photocatalysts exhibit a narrow bandgap of 2.03 eV and significantly improved photocatalytic activity for removing MO in aqueous solution under visible-light irradiation, which can be attributed to the synergistic effect of good visible-light harvesting and enhanced separation rate of the photo-generated charge carriers.

2. Experimental details

2.1. Materials
Melamine was obtained from Macklin Chemical Reagent Co., Ltd (Shanghai, China). Copper acetate monohydrate (Cu(C₂H₃O₂)₂·H₂O), Methyl orange (MO) were purchased from Aladdin Chemical Reagent Co., Ltd (Shanghai, China). Ethanol (EtOH), sodium hydroxide (NaOH), glucose, triethanolamine (TEA), tertiary alcohol (t-BuOH), 1,4-benzoquinone (BQ) and triethanolamine (TEOA) and polyvinylpyrrolidone (PVP, K-30, Mₐ = 30 000) were bought from Sinopharm Chemical Reagent Co. Ltd. All the chemical reagents employed in this study were of analytical grade and used as received without further purification. Deionized water (18.2 MΩ·cm) was used throughout the experimental process.

2.2. Sample preparation

2.2.1. Synthesis of 2D g-C₃N₄ nanosheets

2D g-C₃N₄ nanosheets were synthesized according to \cite{24}. Typically, a yellow product of bulk g-C₃N₄ was synthesized by simple heating ten grams of melamine in the muffle furnace at 550 °C for 2 h. Subsequently, the as-prepared and bulk g-C₃N₄ was further processed under the similar conditions at a relatively lower heating rate. The temperature was cooled down to room temperature and then a white product of g-C₃N₄ nanosheets was obtained, finally, the product was ground to a fine powder.

2.2.2. Synthesis of g-C₃N₄/Cu₂O composite photocatalysts

The g-C₃N₄/Cu₂O composites photocatalysts were obtained via hydrothermal synthesis method. Briefly, 0.24 g Cu(C₂H₃O₂)₂·H₂O and 1.49 g TEA were dissolved in the mixed solution of 24 ml EtOH and 36 ml water to obtain a green solution, and then calculated amount of g-C₃N₄ nanosheets were dispersed in the system by ultrasonication for 1 h. 0.02 g NaOH and 0.8 g PVP were subsequently added under continuous stirring in the suspension. Thereafter, 2 ml of 1 M glucose solution was slowly dropped into this solution as the reducing reagent. The mixture was immediately transferred to a 100 ml teflon autoclave, and heated at 90 °C in an oven for 6 h, when cooled down, the target products were obtained by centrifugation, washing repeatedly with water and EtOH, and then drying at 60 °C under vacuum. The as-synthesized products were labeled as g-C₃N₄/Cu₂O-X, where X corresponds to the weight rate of g-C₃N₄ and Cu₂O in composites (0.5:1, 1:1, 2:1, 3:1, 5:1 and 10:1). Furthermore, the Cu₂O spheres were also prepared via the same procedure without adding g-C₃N₄ nanosheets.

2.3. Characterization

The phase structure, compositions, and crystal planes were investigated by a XRD-6100 x-ray diffractometer (XRD, Shimadzu, Japan) at room temperature. The surface chemical composition and states were further revealed by x-ray photoelectron spectroscopy (XPS, ThermoFisher K-Alpha, USA). Morphology was observed from SEM and TEM images using scanning electron microscope (SEM, Zeiss, Germany) and transmission electron microscopy (TEM, Tecnai G220 S-TWIN, USA). UV–vis diffuse reflectance spectroscopy (UV–vis DRS, PerkinElmer, USA) was recorded at 200 ~ 800 nm on a lambda 750 spectrophotometer using BaSO₄ as the reference. The photoluminescence spectra (PL, Zolix, China) were measured at room temperature excited at 325 nm using a ZLX-FS fluorescence spectrometer. Electrochemical impedance spectroscopy (EIS) was carried
out on an electrochemical station (CHI 660E, Chenhua, China). The photocurrent measurement was performed on the same electrochemical workstation with a light on/off cycle of 30 s.

2.4. Photodegradation test
The photocatalytic performances were tested by the decomposition of MO under simulated visible light irradiation ($\lambda > 420$ nm). Visible irradiation was provided by a 500 W xenon lamp (Xujiang Electromechanical Plant, Nanjing, China) with a 420 nm cutoff filter. In every experiment, 0.03 g photocatalyst was totally added in 50 ml of MO solution (10 mg l$^{-1}$). Prior to irradiation, the suspensions were magnetically stirred in dark for 60 min to establish absorption–desorption equilibrium between the surface of photocatalyst and MO. At given time intervals, 4 ml aliquots were taken out and centrifuged to remove the photocatalyst. The concentration of the residual MO was researched using a UV–vis spectrophotometer at 464.1 nm of maximal absorption peak (Lambda 35, PerkinElmer).

3. Results

3.1. Structure and morphology characterization
The phase structure of $g$-$C_3N_4$, Cu$_2$O, and the $g$-$C_3N_4$/Cu$_2$O samples with the different Cu$_2$O loading amount was characterized by XRD (figure 1). The Bragg reflection located at $2\theta = 27.5^\circ$ in the XRD pattern can be observed for pure $g$-$C_3N_4$ nanosheets, which corresponds to (002) diffraction plane (JCPDS No. 87-1526). Nevertheless, another typical peak located at about 13.2$^\circ$ (in-planar repeat period of aromatic) is not enough strong to be directly observed. The observation indicates that the interlayer structure of bulk $g$-$C_3N_4$ was destroyed during two-step calcination thermal exfoliation, and $g$-$C_3N_4$ nanosheets are successfully obtained. Additionally, the XRD pattern of Cu$_2$O shows five main peaks with $2\theta$ values of 29$^\circ$, 36$^\circ$, 42$^\circ$, 61$^\circ$ and 73$^\circ$, corresponding to the (110), (111), (200), (220) and (311) reflections, respectively (JPCDS No. 65-3288). The $g$-$C_3N_4$/Cu$_2$O composites only display the characteristic diffraction peaks of both $g$-$C_3N_4$ and Cu$_2$O without impurity phases, and the XRD peak intensities of Cu$_2$O become more evidently with the content increasing.

The morphology of as-prepared samples was observed by SEM and TEM, as shown in figure 2. The pristine $g$-$C_3N_4$ nanosheets exhibit irregular, laminar and curly morphology characteristic as shown in the SEM image (figure 2(a)) and TEM image (figure 2(d)). In contrast, the pure Cu$_2$O samples exhibit regular nanospheres structure with diameter of about 200 nm (figure 2(b)). After coupling $g$-$C_3N_4$ with Cu$_2$O, $g$-$C_3N_4$ nanosheets are randomly coated on Cu$_2$O nanospheres as shown in figures 2(c) and (d). Besides, there is an obvious and intimate contact interface formed between $g$-$C_3N_4$ and Cu$_2$O, which would facilitate the separation of the photo-generated carriers.

Besides, the XPS measurements were further carried out to determine the chemical composition and states of $g$-$C_3N_4$/Cu$_2$O-1:1. The survey scan spectrum is shown in figure 3(a), which indicates the simultaneous presence of C, N, O and Cu elements in the composite. Figure 3(b) shows the high-resolution XPS spectra of C 1 s, two characteristic peaks can be fitted locate at 284.4 and 288.0 eV, which can be regarded as sp$^2$ C–C bonds.
and sp² hybridized carbon in the g-C₃N₄, respectively [25]. As presented in figure 3(c) (N 1 s spectra), the corresponding binding energies are fitted locate at 398.3, 400.7 and 403.9 eV, which are caused by the sp² hybridized N involved in triazine rings (C−N=C), the tertiary nitrogen N−(C), and the amino groups (C−N−H), respectively [26]. Figure 3(d) shows the characteristic peaks of the O 1 s at 530.0 eV, which correspond to oxygen bonded to Cu [27]. With regard to Cu 2p, two characteristic peaks locating at 932.0 and 952.0 eV are

![Figure 2. SEM images of prepared photocatalysts: (a) g-C₃N₄ nanosheets, (b) Cu₂O, (c) g-C₃N₄/Cu₂O-1:1, (d) TEM images of g-C₃N₄/Cu₂O-1:1.](image)

![Figure 3. XPS spectra of g-C₃N₄/Cu₂O-1:1: (a) survey scan spectrum and high resolution spectra of (b) C 1 s, (c) N 1 s, (e) O 1 s and (e) Cu 2p.](image)
assigned to Cu 2p3/2 and Cu 2p1/2 of Cu+`, respectively [28, 29]. The XPS results strongly confirm the simultaneous existence of g-C3N4 and Cu2O in the composite.

3.2. Optical properties

Figure 4(a) shows the UV–DRS spectra of the as-prepared photocatalysts. As shown in figure 4(a), the pure g-C3N4 has a fundamental absorption edge at 460 nm, which can be attributed to the g-C3N4 band gap of 2.7 eV. The pure Cu2O exhibits an absorption edge at ca. 620 nm, corresponding to the Cu2O band gap of 2.0 eV. Compared with g-C3N4, the g-C3N4/Cu2O composites showed enhanced dramatically visible light absorption intensity with the introducing of Cu2O, owing to the stronger visible-light adsorption ability of Cu2O than that of g-C3N4. The enhanced visible-light utilization will much more efficiently create photogenerated charge carriers and undoubtedly improve the photocatalytic performance of g-C3N4. Furthermore, the bandgap energy of the samples can be calculated according to the tauc plot. As shown in figure 4(b), the Eg of pure g-C3N4, Cu2O and g-C3N4/Cu2O-1:1 were calculated as 2.86, 2.05 and 2.03 eV, respectively. The results suggest that g-C3N4/Cu2O-1:1 possesses an excellent visible-light harvesting behavior.

The PL experiment was performed to understand photogenerated charge carriers recombination and separation process in the corresponding photocatalyst. As can been seen from figure 4(c), pristine g-C3N4 exists in a strong emission in the visible light range. Compared with g-C3N4, the g-C3N4/Cu2O composites have a significantly lower fluorescent intensity, among of all composites, g-C3N4/Cu2O-1:1 presents a relatively lowest intensity. The remarkable reduction in the PL spectrum of g-C3N4/Cu2O-1:1 implies a fastest separation rate of photo-generated charge carriers.

3.3. Photoelectrochemical properties

The resistance of charge separation and transfer was firstly investigated by EIS measurements. Typically, a smaller diameter of the EIS Nyquist plot means the accelerated interfacial charge separation and transfer. EIS Nyquist plots of g-C3N4 and g-C3N4/Cu2O-1:1 electrodes are displayed in figure 5(a). The Nyquist plot diameter of g-C3N4/Cu2O-1:1 is smaller than that of g-C3N4. Furthermore, the charge separation and transfer efficiency was further analysed by photocurrent measurement. As shown in figure 5(b), g-C3N4/Cu2O-1:1 composite exhibits higher photocurrent density than g-C3N4. Hence, the above analysis demonstrates the
heterojunction between g-C3N4 and Cu2O with this unique structure can significantly enhance separation and transfer ability of photo-induced carriers.

3.4. Photocatalytic efficiency

MO photodegradation reaction of the as-prepared g-C3N4/Cu2O composites were measured under visible-light ($\lambda > 420$ nm) illumination in aqueous solution. As shown in figure 6(a), the concentration of MO does not show any decline in blank test indicating that the self-degradation of MO is negligible under visible-light irradiation [30]. While it can be observed that the concentration of MO diminished slightly in the presence of pristine g-C3N4. As expected, sharp decrease in MO absorbance was observed for all of the g-C3N4/Cu2O samples. Among of all composites, g-C3N4/Cu2O-1:1 displayed the highest photodegradation activity after irradiation for 160 min, the degree of MO degradation for g-C3N4/Cu2O-1:1 reached 97%.

For confirming practical application of g-C3N4/Cu2O-1:1, the reusability experiments need to be taken into consideration. As shown in figure 6(b), no apparent decrease of photocatalytic degradation is observed at the second time, while the MO photodegradation rate shows slightly decrease in the following recycling, which is probably connected with the reduced surface area for photocatalysis due to the absorption of MO on the surface of the photocatalyst [31]. The results indicate the relatively good reusability of the catalysts during entire photodegradation process, which is vital in the practical application of photocatalyst.

3.5. Photocatalytic performance enhancement mechanism

To clarify the photocatalytic performance enhancement mechanism, t-BuOH, BQ and TEOA were used as hydroxyl radical (·OH), superoxide radical (·O2−) and holes (h+) scavenger in MO degradation system of g-C3N4/Cu2O-1:1, respectively. As shown in figure 7(a), the MO photodegradation rate shows a slight decrease with the addition of t-BuOH, which indicates that ·OH just makes a minor contribution to the photocatalytic reaction. Nevertheless, obviously declined degradation rates are discovered in the presence of TEOA and BQ. Therefore, it is not difficult to deduce that h+ and ·O2− are the major active species affect the photocatalytic activity in the photocatalytic process. Thus, the possible chemical processes can be described by following equations:

$$\text{g} – \text{C}_3\text{N}_4/\text{Cu}_2\text{O} + \text{hv} \rightarrow \text{g} – \text{C}_3\text{N}_4(\text{e}^–, \text{h}^+) / \text{Cu}_2\text{O}(\text{e}^–, \text{h}^+)$$  
(1)

$$\text{g} – \text{C}_3\text{N}_4(\text{e}^–, \text{h}^+) / \text{Cu}_2\text{O}(\text{e}^–, \text{h}^+) \rightarrow \text{g} – \text{C}_3\text{N}_4(\text{e}^–) / \text{Cu}_2\text{O}(\text{h}^+)$$  
(2)

$$\text{Cu}_2\text{O}(\text{h}^+) + \text{H}_2\text{O} \rightarrow \text{Cu}_2\text{O} + \text{H}^+ + \cdot\text{OH}$$  
(3)

$$\text{Cu}_2\text{O}(\text{h}^+) + \text{OH}^– \rightarrow \text{Cu}_2\text{O} + \cdot\text{OH}$$  
(4)

$$\text{g} – \text{C}_3\text{N}_4(\text{e}^–) + \text{O}_2 \rightarrow \text{g} – \text{C}_3\text{N}_4 + \cdot\text{O}_2^–$$  
(5)

$$\text{MO} + \cdot\text{O}_2^– \rightarrow \text{Degraded productions(major)}$$  
(6)

$$\text{MO} + \cdot\text{OH} \rightarrow \text{Degraded productions(minor)}$$  
(7)

$$\text{MO} + \text{h}^+ \rightarrow \text{Degraded productions(major)}$$  
(8)

According to the above-mentioned results, the probable photocatalytic activity enhancement mechanism for the charge transfer of g-C3N4/Cu2O-1:1 is further proposed based on the energy band structure. As shown in figure 7(b), the band gaps of g-C3N4 and Cu2O are 2.86 and 2.05 eV, respectively, as calculated based on the...
aforementioned UV–vis DRS spectra, which means that both g-C3N4 and Cu2O can adsorb photons and generate e\(^{-}\)-h\(^{+}\) pairs under visible-light irradiation. Since both the conduction band (CB) and valence band (VB) of g-C3N4 are lower than those of Cu2O \([32, 33]\), the excited state electrons of Cu2O transfer directly to the CB of the g-C3N4. At the same time, the excited holes of g-C3N4 tend to migrate to the VB of Cu2O. Subsequently, electrons stored in CB of g-C3N4 could react with molecular oxygen dissolved in the solution to produce reactive \(\cdot\text{O}_2\)\(^{-}\), and then \(\cdot\text{O}_2\)\(^{-}\) and h\(^{+}\) are able to directly oxidize the organic pollutants due to their high oxidative capacity, therefore, the enhancement of photocatalytic activity was mainly owing to the effective carriers separation efficiency.

4. Conclusions

In summary, 2D/3D g-C3N4/Cu2O nanocomposites were successfully synthesized by a facile and in situ strategy. After introduction of Cu2O, g-C3N4/Cu2O composites exhibit enhanced visible light photocatalytic activities in MO degradation compared with pure g-C3N4. Furthermore, the g-C3N4/Cu2O-1:1 composite can maintain high photoactivity during repeated use. The enhanced visible light photocatalytic efficiency may be ascribed to strong visible-light absorption ability and effective separation and transfer of charge carriers. The results concluded here provide a new insight for further improving the practical application of photocatalytic materials.

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