Novel ternary Ag/CeVO₄/g-C₃N₄ nanocomposite as a highly efficient visible-light-driven photocatalyst

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Abstract: The CeVO₄/graphitic C₃N₄ composites have exhibited much enhanced photocatalytic property for degrading methylene blue (MB) pollutant under visible light irradiation compared with single-phase g-C₃N₄ or CeVO₄. The composite S5 obtained from an optimized mass ratio (5%) of CeVO₄ to dicyanamide (DCDA) exhibits the highest photocatalytic activity. Here, ternary Ag/CeVO₄/g-C₃N₄ composites denoted as X%Ag/S5 were prepared by an ultrasonic precipitation method to improve the photocatalytic property of S5. The TEM images show that CeVO₄ and Ag nanoparticles are well distributed on the layered g-C₃N₄, which agree well with the XRD results. The UV spectra show that the 7%Ag/S5 sample has the widest absorption range and the enhanced absorption intensity under visible light irradiation. The corresponding band gap of 7%Ag/S5 (2.5 eV) is much lower than that of S5 (2.65 eV). The corresponding *k* value of 7%Ag/S5 is much higher than those of g-C₃N₄ and CeVO₄. The degradation experiments for MB solution suggest that the 7%Ag/S5 sample has the optimal photocatalytic performance, which can degrade MB solution completely within 120 min. The enhanced photocatalytic property of the composites is ascribed to not only the effect of heterojunction structure, but also the surface plasma resonance effect of Ag nanoparticles.

Keywords: g-C₃N₄; CeVO₄; Ag; nanocomposites; photocatalytic performance

1 Introduction

Since the 20th century, excessive use of fossil fuels and large-scale emissions of CO₂ had made the problems of environmental pollution and energy crisis become increasingly serious [1–3]. Semiconductor photocatalytic technology, as a green and efficient method, can be

applied for absorbing visible light directly at room temperature and degrading organic pollutants and dye waste without secondary pollution. In recent years, a number of photocatalysts have been synthesized and applied into practice, including TiO₂ [4], ZnO [5], Ag₂CO₃ [6], MoS₂ [7], and so on. However, the experiment results prove that single-component semiconductors exhibit poor photocatalytic performance under visible light irradiation because of wide band gap and high recombination efficiency of photo-induced charge carriers [8]. Therefore, in order to eliminate these

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disadvantages, many composites such as $\text{Ag}_3\text{PO}_4/\text{CuBi}_2\text{O}_4$ [9], $\text{MoS}_2/\text{grapheme}$ [10], $\text{Ag}/\text{AgCl}/\text{TiO}_2$ [11], and $\text{BiOBr}/\text{BiOI}/\text{Fe}_3\text{O}_4$ [12] were studied. All of them show much higher photocatalytic degradation efficiency compared with the single-component semiconductors. The main advantage of these composites is the formation of heterojunction structure between different semiconductors with matched band energy, which improves the separation efficiency of photo-generated electron–hole pairs during photocatalytic process.

Graphitic carbon nitride ($\text{g-C}_3\text{N}_4$), as a visible-light-driven and metal-free photocatalyst, has attracted much attention on account of a suitable band gap (2.7 eV) and its environmental friendliness [13–15]. Depended on the advantages of rich precursor source and low-cost raw materials, $\text{g-C}_3\text{N}_4$ has been widely used in many photocatalytic application fields, such as photocatalytic hydrogen evolution [16], pollutant degradation, and CO_2 reduction. However, pure $\text{g-C}_3\text{N}_4$ still has many drawbacks that restrict its practical applications, such as small surface area, fast recombination of electron–hole pairs, and low electrical conductivity [17,18]. Coupling with other semiconductors to form heterojunction structure and improve the visible light absorption performance is a good way to improve the photocatalytic performance of $\text{g-C}_3\text{N}_4$ [19]. There are various $\text{g-C}_3\text{N}_4$ -based photocatalysts including $\text{AgX}/\text{g-C}_3\text{N}_4$ [20], $\text{AgI}/\text{g-C}_3\text{N}_4$ [21], $\text{HSbO}_3/\text{g-C}_3\text{N}_4$ [22], $\text{BiPO}_4/\text{g-C}_3\text{N}_4$ [23], $\text{g-C}_3\text{N}_4/\text{Zn}_2\text{GeO}_4$ [24], $\text{g-C}_3\text{N}_4/\text{MoS}_2$ [25], $\text{g-C}_3\text{N}_4/\text{Fe}_x\text{P}$ [26], $\text{g-C}_3\text{N}_4/\text{RP}/\text{MoS}_2$ [27], $\text{g-C}_3\text{N}_4/\text{Fe}_3\text{O}_4/\text{Ag}_3\text{VO}_4$ [28], $\text{g-C}_3\text{N}_4/\text{AgBr}/\text{Fe}_3\text{O}_4$ [29], and $\text{g-C}_3\text{N}_4/\text{RGO}/\text{Bi}_2\text{WO}_6$ [30]. The formation of heterojunction between the semiconductors promotes the transmission of electrons obviously and consequently improves the separation efficiency of photo-induced electron–hole pairs. The other promising way is to introduce noble metal nanoparticles (Pt, Au, and Ag) into $\text{g-C}_3\text{N}_4$. Ag nanoparticles are often used to combine with other semiconductors, such as $\text{g-C}_3\text{N}_4/\text{Ag}/\text{MoS}_2$ [31], $\text{Ag}/\text{Fe}_3\text{O}_4/\text{g-C}_3\text{N}_4$ [32], $\text{g-C}_3\text{N}_4/\text{Ag}_2\text{WO}_4/\text{Ag}$ [33], $\text{Ag}@\text{AgBr}/\text{g-C}_3\text{N}_4$ [34], and $\text{g-C}_3\text{N}_4/\text{Ag}/\text{TiO}_2$ [35]. The important reasons are that Ag nanoparticles have unique electrical, optical, and magnetic features [36], and a relatively cheap price compared with platinum or gold. Furthermore, Ag nanoparticles can absorb much visible light because of the surface plasmon resonance (SPR) [37].

In previous report [1], we had prepared $\text{CeVO}_4/\text{g-C}_3\text{N}_4$ composites successfully. When the mass ratio of

CeVO_4 and DCDA as raw materials is 5%, the obtained composite denoted as S5 has the best photocatalytic performance [38]. Inspired by the effect of Ag nanoparticles, we synthesized a series of ternary $\text{Ag}/\text{CeVO}_4/\text{g-C}_3\text{N}_4$ composites with different mass ratios of Ag to S5 by an ultrasonic precipitation method. The photocatalytic performance of the ternary composites was investigated by degrading methylene blue (MB). As expected, all ternary samples exhibit much higher photocatalytic activity than S5. The reasons of the enhanced photocatalytic performance and the possible photocatalytic mechanism were also proposed.

2 Experiments

2.1 Preparation of $\text{Ag}/\text{CeVO}_4/\text{g-C}_3\text{N}_4$

$\text{CeVO}_4/\text{g-C}_3\text{N}_4$ composites were prepared by using a calcination method according to our previous report [1]. The ternary composites $\text{Ag}/\text{CeVO}_4/\text{g-C}_3\text{N}_4$ were prepared by an ultrasonic precipitation method using S5, silver nitrate, and sodium citrate as raw materials, and denoted as $X\%\text{Ag}/\text{S5}$ ($X\%$ represents different mass ratios of Ag to S5). The typical procedure is shown in Fig. 1. First, Ag nanoparticle solution was prepared according to the literature [39]: 90 mg of AgNO_3 was dissolved in 500 mL deionized water and heated to boiling temperature. Second, 10 mL 1% sodium citrate solution was added into beaker and kept boiling for 1 h. The Ag nanoparticle solution was obtained when it was cooled down to room temperature. Third, we mixed the same content of S5 with different contents of Ag nanoparticle solution and put a small quantity of CuSO_4 solution into the mixture with an additional 48 h of ultrasonic vibration. Then, the obtained solutions were dried in vacuum oven. Finally, we got ternary composites containing different Ag contents, which were denoted as 1%Ag/S5, 4%Ag/S5, 7%Ag/S5, and 10%Ag/S5. Herein, all ternary samples were compared with S5 at different aspects in order to prove the advantages of the ternary composites.

2.2 Characterizations

The phases of all samples were analyzed on a Rigaku D/max2500 X-ray diffractometer (XRD) with monochromatized $\text{Cu K}\alpha$ ($\lambda = 1.5406 \text{ \AA}$) radiation at a scanning speed of $8 (^\circ)/\text{min}$ in the range of 10° – 80° .

Fig. 1 Schematic diagram of preparation of Ag/CeVO₄/g-C₃N₄.

The SEM images were obtained on a field emission scanning electron microscope (SEM, JSM-7001F). The TEM pictures of the photocatalysts were taken by using a transmission electron microscope (TEM, JEOL, JEM-2100). UV–Vis diffuse reflection spectra (DRS) were recorded on an UV–Vis spectrophotometer (UV-2450, SHIMADZU) in the range of 300–800 nm using BaSO₄ powder as a reference. The photoluminescence (PL) spectra of the photocatalysts were obtained by using a Quanta Master TM40 fluorescence spectrophotometer at room temperature.

2.3 Photocatalytic experiment

The photocatalytic activity of the as-prepared ternary samples was evaluated by the degradation of MB equipped with a 350 W Xe lamp and a 420 nm cut off filter as light source. All photocatalytic experiments were taken under the same condition. Typically, 50 mg catalysts were dispersed in 50 mL of 10 mg/L MB solution. Prior to irradiation, the mixed solution was magnetically stirred in the darkness for 1 h to obtain adsorption equilibrium at room temperature. During the whole photocatalytic degradation process, about 4 mL samples were withdrawn at regular intervals, centrifuged, and examined at 663 nm on a UV–Vis spectrophotometer (UV-2450, SHIMADZU). The concentration of MB after adsorption equilibrium was denoted as the initial concentration (C_0). After all samples were evaluated, the optimal photocatalyst was chosen to continue the recycling experiments. When each run was finished, the residual samples were washed several times with deionized water, dried at 60 °C in an oven and prepared to be used for the next run.

3 Results and discussion

3.1 Characterizations

As shown in Fig. 2, all samples were characterized by XRD analysis (S5 represents a CeVO₄/g-C₃N₄ composite [1]). The XRD pattern of the ternary composite

Fig. 2 XRD patterns of S5 and Ag/CeVO₄/g-C₃N₄ composites with different contents of Ag.

1%Ag/S5 is similar with that of S5 due to the low content of Ag. When the content of Ag increases from 4% to 10%, the diffraction peaks appear at 38.1° and 44.2°, corresponding to the (111) and (200) planes of Ag crystal, respectively [32]. Their intensities become stronger with the increasing content of Ag. With further increasing the content of Ag, the peak intensities display different variations. It is clear to see that the characteristic diffraction peaks of g-C₃N₄, CeVO₄, and Ag from the ternary composites all exist, indicating that the ternary composites Ag/CeVO₄/g-C₃N₄ are successfully prepared.

Figure 3(a) displays the SEM image of 7%Ag/S5. Because of the small size of Ag nanoparticles, only g-C₃N₄ and CeVO₄ can be seen. g-C₃N₄ shows layered structure supporting large surface area for the ternary system and CeVO₄ particles with diameter of 50 nm are well distributed on the surface of g-C₃N₄. The TEM image of the ternary composites is shown in Fig. 3(b). It is obvious to see that there are three different components in 7%Ag/S5 composite. The results are in good agreement with above XRD analysis. CeVO₄ and Ag nanoparticles (about 10 nm) are deposited on the surface of g-C₃N₄ compactly, which can be in favor of the transmission of electrons.

Fig. 3 (a) SEM image and (b) TEM image of 7%Ag/S5.

The optical properties of the ternary composites were investigated by UV–Vis diffuse reflectance spectroscopy. As shown in Fig. 4(a), the sample S5 shows a relative low intensity of light absorption. After modifying Ag nanoparticles on the surface of $g\text{-C}_3\text{N}_4$, the intensities of all samples become stronger, which can be ascribed to the SPR absorption of plasmonic photocatalyst [34]. Particularly, the sample 7%Ag/S5 presents the highest absorption intensity, the largest absorption range of visible light irradiation, and the biggest absorption edge of 580 nm. The band gap of the composites was also studied according to Kubelka–Munk formula $F(R) = A(h\nu - E_g)^{n/2}$, where, $h\nu$ responses photon energy and n is a constant which depends on the nature of optical transition. The values of n are 1 and 4 for indirect semiconductor and direct semiconductor, respectively. In Fig. 4(b), the band gaps of S5, 1%Ag/S5, 4%Ag/S5, 7%Ag/S5, and 10%Ag/S5 are 2.65, 2.76, 2.8, 2.5, and 2.7 eV, respectively. The band gap of 7%Ag/S5 is lower than that of S5. The composites with lower band gap can absorb more visible light, and stimulate the generation of photo-induced electrons and holes. Therefore, it can be suggested that the sample 7%Ag/S5 would have the best photocatalytic performance.

3.2 Photocatalytic performance

The photocatalytic performance of the ternary composites was evaluated by the degradation of MB (10 mg/L) under visible light irradiation. Before the light irradiation, MB solution and photocatalyst were mixed together and stirred in the dark for 1 h to achieve adsorption–desorption equilibrium. Figure 5(a) illustrates the photocatalytic activities of as-prepared ternary photocatalysts for MB degradation under visible light irradiation. After 120 min, the removal efficiency of S5, 1%Ag/S5, 4%Ag/S5, 7%Ag/S5, 10%Ag/S5 are 72%, 73%, 90%, 99%, 94%, respectively. The results suggest that the ternary composites exhibit higher photocatalytic performance than S5. When the content of Ag increases from 1% to 7%, the photocatalytic activity is significantly improved. However, the overdose of Ag limits the improvement of photocatalytic activity, which indicates that the photocatalytic performance of the ternary composites is well influenced by Ag content. Much more or less Ag content will not promote the transmission of electrons. The optimal Ag content is 7%, so 7%Ag/S5 has the best photocatalytic property. Figure 5(b) shows the photocatalytic degradation kinetics constant (k) of MB over all samples, which represents the reaction rate of the MB solution degradation. It can be seen clearly that 7%Ag/S5 has the highest rate constant (0.0462 min^{-1}), which is much higher than that of S5 (0.0222 min^{-1}).

Practical applications require new photocatalysts with good stability and reusability. Therefore, we did recycling experiments of photocatalytic degradation on 7%Ag/S5 for 4 runs using MB solution (10 mg/L) as the target pollutant. The results are shown in Fig. 5(c). After the first test, the degradation efficiency of MB

Fig. 4 (a) UV–Vis absorption spectra of the ternary samples and (b) the band gap of all samples.

Fig. 5 (a) Photocatalytic activities of as-prepared ternary photocatalysts for MB degradation under visible light irradiation; (b) photocatalytic degradation kinetics of MB over all samples; (c) recycling experiments of photocatalytic degradation on 7%Ag/S5; (d) XRD patterns of 7%Ag/S5 before and after four-time recycling experiments.

decreases gradually which may be caused by the mass loss of the photocatalyst and the absorbed dye on the surface of the photocatalyst. The XRD patterns of 7%Ag/S5 before and after four-time recycling experiments are displayed in Fig. 5(d). It can be seen that the phases of the ternary composites do not be changed, which confirms that the as-prepared composite has good stability.

3.3 Photocatalytic mechanism

One of the most important effect factors of the photocatalytic performance is the separation efficiency of electron–hole pairs in the interface of photocatalyst. The photoluminescence (PL) spectrum is extensively used to investigate the migration, transfer, and recombination processes of the photo-generated electron–hole pairs [40]. As we all known, the higher PL emission intensity means the bigger recombination rate of the photo-induced charge carriers, leading to lower photocatalytic property. The PL spectra of g-C₃N₄, S5, and the ternary composites are shown in Fig. 6. The PL emission intensity of g-C₃N₄ is the highest, whereas the intensity of S5 is relatively low. It can be found that the intensity of ternary composites become much lower after the addition of Ag nanoparticles, showing that the

Fig. 6 PL spectra of g-C₃N₄, S5, and the ternary Ag/CeVO₄/g-C₃N₄ composites.

introduction of Ag nanoparticles into S5 inhibits the recombination of photo-induced charge carriers. The sample 7%Ag/S5 has the lowest emission intensity, suggesting that it has the highest separation efficiency. This result corresponds to the photocatalytic activity experiments.

The possible photocatalytic mechanism of the ternary composites is proposed as shown in Fig. 7. According to previous report [38], a heterojunction structure exists in the interface between g-C₃N₄ and CeVO₄, which effectively accelerates the separation of photo-induced electron–hole pairs and leads to an

Fig. 7 Schematic diagram of the charge carrier separation and transfer in ternary composites under visible light irradiation.

enhanced photocatalytic performance than pristine g-C₃N₄ and CeVO₄. After adding Ag nanoparticles, the photocatalytic property of ternary composites is improved greatly, which can be ascribed to the SPR effect of Ag nanoparticles, absorbing much visible light, causing local electromagnetic fields, and irradiating electron–hole pairs [33,41]. Plasmon activation of Ag nanoparticles transfers electrons to conduction band (CB) of g-C₃N₄ and CeVO₄. Also, a part of them react with O₂ existed on the surface of Ag particles to directly produce •O₂[−], and then •O₂[−] may react with H⁺ to further produce •OH. Both •O₂[−] and •OH are quite important active species which will play good roles for degrading pollutant.

4 Conclusions

In summary, a series of Ag/CeVO₄/g-C₃N₄ ternary composites have been prepared successfully by a simple ultrasonic precipitation method. CeVO₄ (about 50 nm) and Ag nanoparticles (about 10 nm) are distributed on the surface of g-C₃N₄. The ternary composites have higher absorption intensity and photocatalytic performance for degradation of MB under visible light irradiation than S5. In the photocatalytic process, the sample 7%Ag/S5 degrades MB solution completely within 120 min, and its kinetics constant is 0.0462 min^{−1}. The recycling experiments show that the ternary composites have good stability. The enhanced photocatalytic activity is attributed to the high visible light absorption intensity and the formation of heterojunction structure which promotes the transmission of photo-induced carriers.

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