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A facile synthesis of a highly efficient β -Bi₂O₃/ Bi₂O₂CO₃ heterojunction with enhanced photocatalytic NO oxidation under visible light⁺

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The construction of a β -Bi₂O₃/Bi₂O₂CO₃ heterojunction is an effective approach to separate the photogenerated charge carriers and to improve the photocatalytic performance of the semiconductor photocatalysts. In this study, a composite nanosheet of β -Bi₂O₃/Bi₂O₂CO₃ was fabricated *via* an *in situ* annealing of Bi₂O₂CO₃ at 380 °C. The composite showed an excellent adsorption toward NO and an enhanced photocatalytic efficiency over NO oxidation from 5.7 v/v% (bare Bi₂O₂CO₃) to 32.5 v/v% (β -Bi₂O₃/Bi₂O₂CO₃). Furthermore, the presence of the β -Bi₂O₃/Bi₂O₂CO₃ heterojunction promoted the formation of additional $^{\circ}O_{2}^{-}$, and then improved the NO oxidation activity and safety of BOC in conjunction with $^{\circ}$ OH.

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1. Introduction

In the past decades, the quest for the efficient use of solar energy has been motivated by successfully employing the artificial anatase TiO₂ photonic crystals for the splitting of water.¹⁻³ To take full advantage of the solar energy, most efforts have been focused on the development of varied photocatalysts.4,5 Among the varied photocatalysts, bismuth photocatalysts (e.g., Bi₂O₃, BiO₂CO₃, BiVO₄, and BiOX, X = Cl, Br, and I) with their nontoxic properties and tunable electronic structures have been one of the most promising material series.⁶⁻¹⁴ Bi₂O₂CO₃, a Bi(III)-based semiconductor, has been widely explored during the past year^{15–17} due to the formation of hybridized valence band (VB) by the Bi 6s and 2p levels, which could produce increased mobility of photogenerated holes during photocatalysis. However, Bi₂O₂CO₃ has a wide band gap (ca. 3.4 eV), indicating that it can only be excited by UV light.8 To extend the response of Bi₂O₂CO₃ to the visible light region, the construction of a heterojunction of Bi₂O₂CO₃ with another narrow band gap semiconductor system has become an efficient option. It is worth noting that tetragonal-Bi(m) oxide (β -Bi₂O₃) with a narrow band gap of ca. 2.61 eV among the other Bi(III)-based semiconductors has been intensively studied for various applications.^{10,18} Although the valence band (VB) of β -Bi₂O₃ is sufficiently positive, it has been barely individually used in previous studies because of its less negative conduction band (CB), instability in the reaction

process and fast recombination of the photogenerated electrons and holes.^{7,19} Therefore, numerous related studies have been conducted to solve these problems, including the band structure modification, the crystal facet control and the heterojunction construction.^{20–24}

In particular, some studies demonstrated that Bi₂O₂CO₃ can be converted to a metastable β -Bi₂O₃ *via* an annealing process.^{25–27} The crystal structure of β -Bi₂O₃ is similar to that of Bi₂O₂CO₃, which is attributed to the same atomic arrangement in their Bi₂O₂²⁺ layers.²⁸ Therefore, the integration of these two phases is expected to form a p-n junction, in which the charge carrier separation is achieved through the migration of holes and electrons to the less positive VB and less negative CB of the component semiconductors, respectively. For example, a β-Bi₂O₃/Bi₂O₂CO₃ composite exhibited a much higher photodegradation efficiency toward MB than bare $Bi_2O_2CO_3$ and β - Bi_2O_3 .²⁹ Unfortunately, such type of charge carrier migration cannot include both the oxidizing power of holes and the reducing power of electrons simultaneously. In contrast, the proposed Z-scheme heterojunction could avoid this shortcoming, in which the migration of electrons is from the less negative CB of one semiconductor such as WO₃ to the less positive VB of the other semiconductor g-C₃N₄.³⁰

In this study, we reported the facile fabrication of a layered *Z*-scheme heterojunction of β -Bi₂O₃/Bi₂O₂CO₃ *via* a thermal treatment. The photocatalytic activity of the β -Bi₂O₃/Bi₂O₂CO₃ composite was evaluated by the photocatalytic oxidation of ppb-level NO under the irradiation of visible light. Moreover, the photogenerated superoxide radicals among the β -Bi₂O₃/Bi₂O₂CO₃ composite promotes fast migration owing to the *Z*-scheme mechanism, which is beneficial for obtaining enhanced photocatalytic activity.

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2. Experimental

2.1. Preparation of samples

Synthesis. All of the chemicals used in this study were of analytical grade and obtained from Shanghai Chemical. Bi₂O₂CO₃ (BOC) samples were synthesized by modifying a reported method.¹⁵ In a typical synthesis, 4.85 g of Bi(NO₃)₃·5H₂O was dissolved in 10 mL of 1 M HNO₃ aqueous solution and further stirred for 40 min at room temperature to form a clear solution (denoted as solution A). Moreover, 1.0 g of cetyltrimethylammonium bromide (CTAB) and 8.45 g of Na₂CO₃·10H₂O were added to 90 mL of distilled water (denoted as solution B). Then, solution A was dropped into solution B under constant stirring. After 10 min of stirring, the resulting white precipitate was filtered, washed with ultrapure water and ethanol for five times and dried at 60 °C for 12 h to obtain BOC samples with no further treatment. Finally, the BOC precursors were calcined in the temperature range from 350 °C to 420 °C for 4 h under atmospheric conditions to obtain different samples. The obtained samples were denoted as BOC-350, BOC-380, BOC-400 and BOC-420, corresponding to the temperatures 350 °C, 380 °C, 400 °C and 420 °C, respectively.

2.2 Characterization

Powder X-ray diffraction (PXRD) was conducted on a PANalytical X'pert diffractometer operated at 40 kV and 40 mA using a Cu Ka radiation. Diffuse reflectance spectra (DRS) were recorded on a Shimadzu UV-2600 spectrophotometer equipped with an integrating sphere using BaSO₄ as the reflectance standard sample. Scanning electron microscopy (SEM) was performed on a Hitachi S-4800 microscope. TEM images were obtained on an FEI Tecnai G2 20 microscope at 200 kV. X-ray photoelectron spectroscopy (XPS) measurements were conducted on a Thermo ESCALAB 250Xi with Al Ka emission at 1486.6 eV. All binding energies were referenced to the C 1s peak at 284.8 eV. A JE-SFA200 spectrometer was used to detect the in situ electron spin resonance (ESR) signals of the samples using DMPO as the capturing reagent. The photocatalytic measurements of NO oxidation were completed using a self-built photocatalytic evaluation device. The removal ratio (η) of NO was calculated as η (%) = $(1 - C/C_0) \times 100\%$, where C and C_0 are the concentrations of NO in the outlet stream and the feeding stream, respectively. The detailed measurements are shown in the ESI.†

3. Results and discussion

The PXRD spectra of the as-fabricated samples are shown in Fig. 1. In the PXRD spectra, the diffraction peaks of the as-prepared BOC can be assigned to the tetragonal $Bi_2O_2CO_3$ (JCPDS no. 41-1488). The signal of β -Bi₂O₃ appeared with the obtained BOC, which was annealed under atmospheric conditions at temperatures of 350, 380, 400 and 420 °C. When the annealing temperature reached about 380 °C, typical peaks appeared at around 27.9°, 31.8° and 32.7°, which corresponded well with the values from the β -Bi₂O₃ standard card (JCPDS no. 27-0050). With the further increase in temperature, the diffraction peaks of Bi₂O₂CO₃ in the samples gradually decreased, while the peaks of the β -Bi₂O₃ phase enhanced



Fig. 1 PXRD patterns of the as-prepared Bi₂O₂CO₃ and the samples after annealing at different temperatures.

simultaneously, which suggested the formation of the β -Bi₂O₃/ Bi₂O₂CO₃ composites. In line with the PXRD patterns, the UV-vis absorption edge of these samples also shifted from 356 to 507 nm with an increase in the temperature (Fig. 2). The band gap (E_g) was evaluated using the following equation $E_g = 1240/\lambda$. Therefore, the band gaps (E_g) of BOC, BOC-350, BOC-380, BOC-400 and BOC-420 were approximately determined as 3.25, 2.57, 2.52, 2.46 and 2.20 eV, respectively. The absorption band edge of BOC-380 was significantly smaller than that of BOC, which suggested that BOC-380 facilitated the visible light harvesting.

The photocatalytic activities of these samples were evaluated by the photocatalytic oxidation of ppb-level NO in air under visible light irradiation ($\lambda > 420$ nm). In Fig. S1 of the ESI,† it is observed that NO is very stable and cannot be photolyzed under visible light irradiation in the absence of photocatalysts. Before irradiation, all the catalysts were exposed to the starting NO gas until reaching an adsorption-desorption equilibrium under experimental conditions. The photooxidation of NO for BOC-380 could reach as high as 32.5% v/v in 7.5 min (Fig. 3a), while the removal ratio of NO over BOC was quite low (5.7% v/v). A conventional TiO₂ (P25) photocatalyst exhibited almost no photocatalytic NO oxidation activity under the same condition photocatalytic activity as shown in Fig. S2 (ESI[†]), which is due to a wide band gap. However, the photooxidative ability of NO on BOC-350, BOC-400 and BOC-420 was not in good agreement with BOC-380. Therefore, the optimum proportion of the catalyst concentration between Bi2O2CO3 and B-Bi2O3 may



Fig. 2 (a) UV-vis diffuse reflectance spectra and (b) plots of transformed Kubelka–Munk function *versus* the energy of absorbed light for asprepared samples under annealing at different temperatures.



Fig. 3 Photocatalytic oxidation of NO over the as-prepared samples of BOC, BOC-350, BOC-380, BOC-400 and BOC-420 under visible light irradiation ($\lambda > 420$ nm): (a) photocatalytic removal ratio of NO; (b) generation of NO₂.

effectively separate and transfer the photogenerated charge carriers for the production of ${}^{\bullet}O_2^{-}$ and ${}^{\bullet}OH$ reactive species.

The morphology and the microstructure of these samples were further investigated by the SEM and TEM observations. The SEM images of the samples prepared, BOC and BOC-380, are shown in Fig. 4a and b. The prepared BOC constitutes large amounts of loose microspheres. Evidently, the image of BOC-380 appeared to be irregular nanosheets due to the release of H₂O and CO₂ gas during decomposition. These results suggested that the Bi₂O₂CO₃ nanosheets gradually transformed to β-Bi₂O₃. The HRTEM image of BOC showed a clear lattice fringe with an interplanar lattice spacing of 0.274 nm and an angle of 90° (Fig. 4c), which corresponded with the (110) plane of the tetragonal Bi₂O₂CO₃. It clearly showed two different fringes with spacings of 0.274 and 0.323 nm (Fig. 4d), corresponding to the (110) lattice plane of Bi₂O₂CO₃ and (201) plane of the tetragonal β -Bi₂O₃. These results further confirmed that β -Bi₂O₃ nanosheets were formed *in situ* on Bi₂O₂CO₃.

The survey and the high-resolution XPS spectra of BOC and BOC-380 are shown in Fig. 5. It is observed that the peak of C 1s at 284.8 eV can be ascribed to the adventitious carbon species from the XPS measurements,³¹ which remained unchanged, whereas the peaks at 288.8 and 285.3 eV belonging to the carbonate in BOC shifted up to 288.9 and 287.3 eV in BOC-380 (Fig. 5a).

Consequently, the electronic densities of C in the CO_3^{2-} layers were also affected by the β -Bi₂O₃ formed. The peak at 530.9 eV can be ascribed to the oxygen of the C-O bond, and the dominant 529.6 eV peak of O 1s characterized by the Bi-O bonds in Bi2O2CO3 shifted to 529.9 eV, belonging to β -Bi₂O₃ (Fig. 5b).³² The other peaks are from adsorbed H₂O on the surface.³³ The two strong peaks at 158.9 and 164.2 eV are assigned to Bi $4f_{7/2}$ and Bi $4f_{5/2}$, which correspond to Bi₃⁺ (Fig. 5c).¹¹ These values are not exactly the same as those obtained from pure β -Bi₂O₃ or Bi₂O₂CO₃ after the annealing of BiO₂CO₃ at 380 °C, which indicates that an interfacial structure is formed and the local environment and electron density of the elements changed to some extent. These results were well agreed with the XRD analysis. The E_{VB} of Bi₂O₂C₃ and BOC-380 are estimated to be 1.77 and 2.13 eV, respectively, which is observed from the VB-XPS spectra (Fig. 5d). In addition, the E_{CB} of pure Bi2O2CO3 could be constructed using the following empirical formulae: $E_{CB} = E_{VB} - E_{g}$. According to the above formulae, the E_{VB} and E_{CB} values of pure Bi₂O₂CO₃ were calculated to be 1.77 and -1.48 eV, respectively. Zhou also reported that the $E_{\rm VB}$ value of pure Bi₂O₂CO₃ was 1.76 eV, which was obtained from the ultraviolet photoelectron spectrum.³⁴ The E_{VB} by XPS measurement and E_{CB} values of pure β-Bi2O3 were calculated to be 2.59 and 0.31 eV, respectively.10

The spin-resonance technique (ESR) was further employed to detect the active species generated during the photocatalytic NO oxidation of BOC and BOC-380 (Fig. 6a and b). Some negligible signals corresponding to DMPO-•OH of BOC and BOC-380 under the same conditions of light irradiation can be detected in Fig. 6a. As shown in Fig. 6b, the characteristic peaks of DMPO-•O₂⁻ for BOC are very weak; however, 6 strong signals in BOC-380 are detected after 10 min under visible light condition. These results confirmed that •O₂⁻ is the dominant active species in the photocatalytic degradation process. The activity of the photocatalyst is related to the absorption of light, the absorption intensity, and the separation of photogenerated electrons and holes. Photoluminescence (PL) is a typical technique for studying the photon-generated carrier properties of



Fig. 4 SEM (a and b) and HRTEM (c and d) images of BOC and BOC-380, respectively.



Fig. 5 XPS spectra of BOC and BOC-380: (a) C 1s, (b) O 1s, (c) Bi 4f, and (d) valence band.



Fig. 6 ESR spectra of (a) hydroxyl radicals (*OH) and (b) superoxide radicals (*O $_2^-$), and PL spectra of BOC and BOC-380 (c).

the semiconductor materials. Studies have shown that the fluorescence intensity is related to the recombination rate of electrons (e⁻) and holes (h⁺).^{13,14} For this reason, the PL spectra of BOC, BOC-380 and BOC-420 are measured under the excitation of a 367 nm laser (Fig. 6c). In general, the charge carrier migration followed the p-n heterojunction pathway, that is, electrons from the CB of BOC migrated to the CB of β -Bi₂O₃ and h^+ from the VB of β -Bi₂O₃ to the VB of Bi₂O₂CO₃, resulting in the PL intensity of BOC lower than that of BOC-380 and BOC-420. Nevertheless, the peak intensity of BOC-380 was obviously stronger than that of BOC and BOC-420, indicating stronger charge carrier recombination in BOC-380. According to the similar results from previous studies,^{35,36} the Z-scheme pathway was the only explicable reason for the increase in the PL intensity. This process could consume h^+ and e^- from the VB of $Bi_2O_2CO_3$ and the CB of β - Bi_2O_3 , while preventing the recombination of e⁻ and h⁺ from the CB of Bi₂O₂CO₃ and the VB of β -Bi₂O₃.

Based on the discussion of the abovementioned experimental results, we presented the photocatalytic mechanism of the NO removal by a composite catalyst sample under visible light. It is known that β -Bi₂O₃ is a p-type semiconductor, while Bi₂O₂CO₃ is an n-type semiconductor. Therefore, after their contact with each other, the p-n junction (β -Bi₂O₃/Bi₂O₂CO₃) would be formed when their Fermi levels (EF) reached the equilibrium state again, and the newly formed energy band structures would be observed in the interactive structures. From the above energy band calculations, the possible photocatalytic oxidation mechanism of NO on BOC-380 was presented (Fig. 7). In theory, the photogenerated e^- on the CB of $Bi_2O_2CO_3$ migrated to the CB of β - Bi_2O_3 and the photogenerated h^+ on the VB of Bi₂O₂CO₃ migrated to the VB of β -Bi₂O₃, which led to more accumulated electrons hardly migrating from O_2 to O_2^- (-0.33 eV). However, the BOC-380 was observed to be beneficial to produce more ${}^{\bullet}O_2^{-}$ according to the results of ESR. Therefore, the built-in electric field created during the formation of p-n (β-Bi₂O₃/Bi₂O₂CO₃) heterojunctions



Fig. 7 Possible photocatalytic oxidation mechanism of NO on BOC-380. (a) Before connection. (b) After connection.

can effectively separate the electron-hole pairs, which is called the Z-scheme heterojunction. The separated e⁻ on the CB of $Bi_2O_2CO_3$ and h^+ on the VB of β - Bi_2O_3 then freely migrated toward the surface to react with the adsorbed reactants, leading to an enhanced photocatalytic activity in the composites. In order to understand the photocatalytic process, in situ FTIR was subsequently carried out to intuitively reveal the conversion pathways for the photocatalytic NO oxidation (Fig. S3, ESI[†]). Two intensive broad bands at 1247 and 1189 cm^{-1} could be evidently observed in the NO adsorption process on β-Bi₂O₃/Bi₂O₂CO₃ (Fig. S3a, ESI⁺), which could be assigned to NO^{+.³⁷} The weak band at 1094 cm⁻¹ was observed for NO₃^{-.38} After light on, the NO₂⁻ bands at 1290 and 1056 cm⁻¹ gradually strengthened, which had formed by the oxidation of NO with ${}^{\bullet}O_2^{-}$ (reaction (3)). The bands at 817, 1020, 1056 and 1093 cm^{-1} assigned to NO₃⁻ was transformed from NO₂⁻ species (Fig. 3Sb, ESI⁺).³⁷⁻³⁹ This could be attributed to the •OH⁻ and NO₂⁻ participating in the photocatalytic oxidation (reaction (5)). During the photocatalytic reaction, new peak bands at 873 cm⁻¹ assigned to the adsorption of NO also appeared.40 From the comparative investigations above, the photocatalytic removal of NO over BOC-380 may involve the following reactions:

$$h\nu + \text{BOC-380} \rightarrow e^- + h^+$$
 (1)

$$e^- + O_2 \rightarrow \bullet O_2 \tag{2}$$

$$^{\bullet}O_2^{-} + NO \rightarrow 2NO_2 \tag{3}$$

$$h^+ + H_2O \rightarrow {}^{\bullet}OH + H$$
 (4)

$$OH + NO_2 \rightarrow NO_3^- + H^+$$
(5)

4. Conclusions

A β -Bi₂O₃/Bi₂O₂CO₃ composite with a *Z*-scheme heterojunction was successfully fabricated by the heat treatment of a Bi₂O₂CO₃ precursor at 380 °C. The photocatalytic activities were evaluated and compared with the photocatalytic oxidation of NO under visible-light irradiation. The β -Bi₂O₃/Bi₂O₂CO₃ composite exhibited much higher photocatalytic activity than pure-Bi₂O₂CO₃ and other temperature-Bi₂O₂CO₃. Furthermore, the *Z*-scheme heterojunction formation of β -Bi₂O₃/Bi₂O₂CO₃ could facilitate the separation of the photogenerated charge carriers, which improved the photocatalytic NO removal efficiency eventually.

Conflicts of interest

The authors report no conflicts of interest.

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