Synthesis of a Novel 1D/2D Bi$_2$O$_2$CO$_3$–BiOI Heterostructure and Its Enhanced Photocatalytic Activity

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Abstract: A novel 1D/2D Bi$_2$O$_2$CO$_3$–BiOI heterojunction photocatalyst with high-quality interfaces was synthesized through a hydrothermal method by using Bi$_2$O$_2$CO$_3$ nanorods and KI as raw materials. Two-dimensional (2D) BiOI nanosheets uniformly and vertically grow on the 1D porous Bi$_2$O$_2$CO$_3$ rods. Bi$_2$O$_2$CO$_3$–BiOI heterojunctions exhibit better photocatalytic activity than pure Bi$_2$O$_2$CO$_3$ nanorods and BiOI nanosheets. Cr(VI) (30 mg/L), MO (20 mg/L) and BPA (20 mg/L) can be completely degraded in 8–15 min. The superior photocatalytic performance of 1D/2D Bi$_2$O$_2$CO$_3$–BiOI heterojunction is ascribed to the synergistic effects: (a) vertical 2D on 1D multidimensional structure; (b) the formation of the Bi$_2$O$_2$CO$_3$–BiOI p–n heterojunction; (c) high-quality interfaces between Bi$_2$O$_2$CO$_3$ and BiOI.

Keywords: 1D/2D nanostructure; photocatalysis; Bi$_2$O$_2$CO$_3$–BiOI heterogeneous

1. Introduction

Nowadays, one-dimensional (1D)/two-dimensional (2D) heterogeneous photocatalysts have been extensively studied for their unique dimensional advantages in environmental and sustainable energy applications [1–3]. A large number of studies have shown that 1D nanostructures can provide a short diffusion length perpendicular to its axis and a fast carrier transfer path along its axis [4–7]. Two-dimensional (2D) materials usually have a large surface area with good electrical conductivity and superior electron mobility [8–11]. So, compared with other heterostructures, 1D/2D heterostructures have unique advantages because of their intrinsic structural features [12–15]. Firstly, 1D/2D heterojunction has a larger surface/interface area, which can provide more photocatalytic active sites. Secondly, the photogenerated charge carriers on 2D nanosheets will transfer to 1D nanorods, which make electron–hole pairs separation more effectively and charge lifetimes longer [12,16–18]. At present, there are mainly two types of 1D/2D heterojunction based on the interfacial contact and morphology: the growth of 1D material on 2D nanomaterials and the growth of 2D nanosheets on 1D nanomaterials. Among them, the vertical epitaxial growth of 2D nanosheets on 1D nanomaterial can expose most of their overall surface [19,20]. Although 1D/2D heterojunction materials have many advantages, there are very few studies on bismuth-based 1D/2D heterojunction materials at present [21–25]. In recent years, our group has reported the synthesis of photocatalysts which 2D nanosheets vertically loaded on 1D bismuth-based materials [19,20,24,25]. These 1D/2D bismuth-based heterojunction photocatalysts exhibit superior photocatalytic activity in the removal of organic pollutants. However, the research in this field is still insufficient. So, it is necessary to extensively explore the synthesis of this kind of 1D/2D bismuth-based heterojunction photocatalysts with excellent photocatalytic performance.
As known, the unique layered structure of bismuth-based materials makes it has the characteristics of high connection quality between interfaces, internal electric field formation, asymmetric polarization effect [26–29]. Bi₂O₂CO₃ is a typical n-type semiconductor with wide band gap (3.55 eV) and only responds to ultraviolet light [30–32]. BiOI is a typical p-type semiconductor with narrow band gap (1.8–2.1 eV) [33–36]. BiOI is widely used to eliminate the pollutants because it can improve the absorption capacity of visible light [34,37,38]. BiOI and Bi₂O₂CO₃ have the similar layered structure which can couple into p–n typical heterostructure [39–48]. All of the above BiOI–Bi₂O₂CO₃ heterostructures display improved activities compared to pristine BiOI and Bi₂O₂CO₃. However, the reported BiOI–Bi₂O₂CO₃ photocatalysts show the 0D/2D, 0D/3D or 2D/2D coupling of BiOI with Bi₂O₂CO₃, and 1D/2D Bi₂O₂CO₃–BiOI heterojunction has not been reported yet.

In this article, a novel 1D/2D Bi₂O₂CO₃–BiOI heterojunction was synthesized through a hydrothermal method by using porous Bi₂O₂CO₃ nanorods as Bi source and KI as I source. BiOI nanosheets vertically grow on the Bi₂O₂CO₃ porous rods. This 1D/2D Bi₂O₂CO₃–BiOI heterostructure displays superior photocatalytic activity for degrading Cr (VI), methyl orange (MO) and bisphenol A (BPA) under solar light irradiation, and Cr (VI) (30 mg/L) could be completely reduced in 8 min. This excellent photocatalytic performance is due to the synergistic effect of some factors: (a) Unique 1D/2D nanostructure; (b) the formation of the p–n junction; (c) the high-quality interfaces between Bi₂O₂CO₃ and BiOI.

2. Results and Discussion

1D/2D Bi₂O₂CO₃–BiOI heterojunction was synthesized through hydrothermal method using porous Bi₂O₂CO₃ nanorods and KI as raw materials. The as-obtained Bi₂O₂CO₃–BiOI heterojunctions were labelled as S1, S2, S3 and S4 when the molar ratios of Bi₂O₂CO₃: KI are 10:1, 2:1, 1:2 and 1:20, respectively. The SEM images and XRD pattern of the obtained samples are shown in Figure 1. Bi₂O₂CO₃ is 1D rod-like structure with rough surfaces and porosity (Figure 1a). Few nanosheets grow vertically on the surface of Bi₂O₂CO₃ rods when the molar ratio of Bi₂O₂CO₃: KI is 10:1 (Figure 1b). With the molar ratio of Bi₂O₂CO₃: KI increase, more and more nanosheets are loaded on the surface of Bi₂O₂CO₃ rods (Figure 1b–d). When the molar ratio of Bi₂O₂CO₃: KI is 1:20, only out-of-order nanosheets can be obtained (Figure 1e). Figure 1f shows the XRD patterns of the obtained samples. It can be seen that the main diffraction peaks in S1–S3 samples are indexed to Bi₂O₂CO₃ (PDF#25-1464). In samples S2 and S3, a new diffraction peak (2θ = 31.65°) is found clearly, which is indexed to the tetragonal BiOI (PDF#10-0445). The diffraction peaks of Bi₂O₂CO₃ become weaker and weaker from S1 to S3. The results indicate Bi₂O₂CO₃ reacts with KI and form 1D/2D Bi₂O₂CO₃–BiOI heterostructure. In S4 sample, only BiOI peaks can be found, indicating all the Bi₂O₂CO₃ nanorods is consumed completely. The Bi₂O₂CO₃ nanorod skeleton is disappeared, which results in the collapse of 1D structure. So, BiOI nanosheets is formed when the molar ratio of Bi₂O₂CO₃: KI is 1:20.

The S2 sample was characterized by using TEM and HRTEM. As shown in Figure 2a, it can be seen that BiOI nanosheets grow vertically on the surface of Bi₂O₂CO₃ nanorods, which is consistent with the SEM images. In Figure 2b, the lattice spacing of 0.685 and 0.915 nm indexes to the (002) lattice plane of Bi₂O₂CO₃ and the (001) lattice plane of BiOI, respectively. BiOI nanosheets grow out from the Bi₂O₂CO₃ rod by oriented epitaxial nucleation and growth, which is beneficial for the formation of a high-quality interface [24].
Figure 1. FE-SEM images of (a) pure Bi₂O₂CO₃ nanorods, Bi₂O₂CO₃–BiOI heterojunctions of (b) S1, (c) S2, (d) S3, (e) pure BiOI. (f) XRD patterns of Bi₂O₂CO₃–BiOI heterostructure (S1–S4) and Bi₂O₂CO₃.

The full XPS spectra of S2, S4 and Bi₂O₂CO₃ nanorods is shown in Figure 3a. Bi, C, O and I elements are co-existence in S2 sample, indicating the formation of Bi₂O₂CO₃–BiOI. There is almost no C element in the S4 sample, confirming that S4 is pure BiOI. The high resolution XPS spectra of the Bi 4f, I3d and C1s are shown in Figure 3b–d. Two peaks centered at about 164.50 and 159.00 eV are attributed to Bi 4f⁷/₂ and Bi 4f⁵/₂, respectively, indicating that the Bi element in all samples is in the form of Bi³⁺ ion (Figure 3b). Compared with Bi₂O₂CO₃ nanorods and S4 (pure BiOI), the peak of Bi 4f moves to higher binding energies, which proves that high-quality interface forms between Bi₂O₂CO₃ and BiOI. The same phenomenon is also observed in the I 3d spectra (Figure 3c). The peak centered at 289.08 eV is indexed to C1s (Figure 3d). The intensity of C1s in S2 sample is obviously lower than in Bi₂O₂CO₃. This result confirms Bi₂O₂CO₃ is consumed to form BiOI during the reaction.
The FTIR spectra of Bi$_2$O$_2$CO$_3$, S2 and S4 samples were measured, as shown in Figure 4. The intensive peak which centered at 847.1 cm$^{-1}$ is attributed to the $\nu_2$ modes of the CO$_3^{2-}$ group. After Bi$_2$O$_2$CO$_3$ and BiOI are combined with each other, a new peak at 493.6 cm$^{-1}$ appears in S2 sample, further proving the formation of the strong interfacial junction between Bi$_2$O$_2$CO$_3$ and BiOI. Depending on the increase of the loaded content of BiOI, the main characteristic peak of the CO$_3^{2-}$ ($847.1$ cm$^{-1}$) almost disappears in the S4. This result is consistent with XRD and XPS results, verifying that the S4 sample is pure BiOI.

Figure 3. Survey (a) and high-resolution Bi 4f (b), I 3d(c), and C 1s (d) XPS spectra of Bi$_2$O$_2$CO$_3$, S2 and S4 samples.

Figure 4. FT-IR spectra of Bi$_2$O$_2$CO$_3$, S2 and S4 (pure BiOI) samples.
UV-vis diffuse reflectance spectroscopies (DRS) of Bi$_2$O$_2$CO$_3$, S2 and S4 were performed to study their optical absorption properties. The absorption band edge of Bi$_2$O$_2$CO$_3$ and BiOI are ~450 and 670 nm (Figure 5a), respectively, indicating the wider band gap of Bi$_2$O$_2$CO$_3$ than that of BiOI. The absorption band edge of the S2 heterostructure red-shifts compared with Bi$_2$O$_2$CO$_3$ due to the loaded-BiOI with narrower band gap (Figure 5b). The optical band gap of Bi$_2$O$_2$CO$_3$ and BiOI is obtained using the following equation:

$$a\hbar\nu = A(\hbar\nu - E_g)^{n/2}$$

where $a$ is the absorption coefficient, $h$ is Planck’s constant, $\nu$ is the light frequency, $A$ is the constant and $E_g$ is the bandgap energy [49]. In our study, both Bi$_2$O$_2$CO$_3$ and BiOI possess indirect band gaps, so $n = 4$ [25,50]. The band gap energies are estimated to 2.96 eV for pure Bi$_2$O$_2$CO$_3$, and 1.75 eV for pure BiOI (Figure 5b).

![Graphs showing UV-Vis diffuse reflectance spectra and plots of $(a\hbar\nu)^{2/n}$ vs. $\hbar\nu$](image)

**Figure 5.** (a) UV-Vis diffuse reflectance spectra of Bi$_2$O$_2$CO$_3$, S2 heterostructures and S4 (pure BiOI) and (b) the plots of $(a\hbar\nu)^{2/n}$ vs. $\hbar\nu$ ($n = 4$ for Bi$_2$O$_2$CO$_3$ and BiOI).

The photocatalytic activity of the Bi$_2$O$_2$CO$_3$–BiOI heterostructures are tested using the Cr (VI) (30 mg/L), MO (20 mg/L) and BPA (20 mg/L) as model pollutants under solar light irradiation. The degradation curves of the different photocatalysts and the UV-vis absorption spectra of Cr (VI), MO and BPA are shown in Figure 6. The results show that S2 owns the highest photocatalytic activity among all the samples, and Cr(VI) (30 mg/L) (pH = 7), MO (20 mg/L) and BPA (20 mg/L) can be completely photodegraded in 8, 15 and 15 min, respectively. Compared with the reported Bi$_2$O$_2$CO$_3$–BiOI heterostructures, S2 sample exhibits excellent photocatalytic activity [39,40]. From Figure S1, the reaction rate constant (k) of S2 (0.4661 min$^{-1}$) is much higher than that of Bi$_2$O$_2$CO$_3$, S1, S3 and S4 (0.0049, 0.0731, 0.1363 and 0.0594 min$^{-1}$) in degrading Cr (VI), exhibiting S2 sample superior photocatalyst. The reaction rate constant (k) of S2 is also higher than that of Bi$_2$O$_2$CO$_3$, S1, S3 and S4 in degrading MO (20 mg/L) and BPA (20 mg/L) (Figures S2 and S3).

The unique 2D vertical on 1D structure endows Bi$_2$O$_2$CO$_3$–BiOI photocatalyst with distinctive photocatalytic activity. Firstly, 2D BiOI nanosheets vertically grew on the 1D Bi$_2$O$_2$CO$_3$ nanorods which can provide almost exposure entire active sites; Secondly, 1D Bi$_2$O$_2$CO$_3$ structures provide quickly charge carriers transfer path along their axis; In addition, the high-quality interface between Bi$_2$O$_2$CO$_3$ and BiOI promotes the transfer rate of photo-generated charge carriers at junction interface, enhancing photocatalytic activity.
Free radical capture experiment is carried out to explore active species in photocatalytic process. tert-butanol (TBA), 1, 4-benzoquinone (BQ) and ammonium oxalate (AO) were used to trap hydroxyl radical (OH·), superoxide radical (O₂⁻) and hole (H⁺) for Cr(VI) (30 mg/L) degradation. As can be seen from Figure 7a, the addition of AO and BQ significantly inhibited the photocatalytic reduction of Cr(VI) (30 mg/L). However, with the addition of TBA, the photoreduction efficiency of Cr(VI) (30 mg/L) only partly changes. These results prove that the main active species are O₂⁻ and H⁺ during photocatalytic process.
In order to evaluate the reusability and photostability of Bi$_2$O$_2$CO$_3$–BiOI heterostructure, catalytic cycle experiment was done using S2 as photocatalyst to degrade the Cr(VI) (30 mg/L) (Figure 7b). We can see that the S2 sample had good reusability, and its photocatalytic efficiency almost remained stable after five cycles. In addition, the S2 sample after 5 cycles was characterized by using XRD and SEM, and the results are shown in Figure 7c,d, respectively. The results demonstrate S2 sample retains the original structure and morphology after five cycles, implying a good photostability of S2 sample under solar light irradiation.

The room temperature PL emission spectra of the pure Bi$_2$O$_2$CO$_3$, S2 and S4 (pure BiOI) are shown in Figure 8a. The PL emission intensity of S2 is the lowest one among the three samples, which implies 1D/2D heterostructure effectively suppresses the recombination of photogenerated e–h$^+$, and thus enhancing the photocatalytic performance [51].

The photocurrent of pure Bi$_2$O$_2$CO$_3$, S2 and S4 (pure BiOI) samples are shown in Figure 8b. The photocurrent density generated by the S2 sample is obviously higher than that of pure Bi$_2$O$_2$CO$_3$ and BiOI. Therefore, the PL and photocurrent measurements all demonstrate that the 1D/2D Bi$_2$O$_2$CO$_3$–BiOI heterostructure can significantly promote the separation and transfer of photogenerated electron–hole pairs.

In order to obtain the relative positions of the conduction band (CB) and valence band (VB) edges, VB-XPS of Bi$_2$O$_2$CO$_3$ and S4 (pure BiOI) were characterized (Figure 9). The E$_{VB}$ top of Bi$_2$O$_2$CO$_3$ and S4 locate at 1.78 and 1.15 eV, respectively. On the basis of the VB position and their band gaps, the CB edge potentials of Bi$_2$O$_2$CO$_3$ and BiOI are estimated to be $-1.18$ and $-0.6$ eV, respectively through the equation $E_{CB} = E_{VB}$. 

Figure 8. The room temperature PL spectra ($\lambda_{ex} = 320\, \text{nm}$) (a) and photocurrent spectra (b) of Bi$_2$O$_2$CO$_3$, S2 and S4 samples.

Figure 9. The VB-XPS spectra of Bi$_2$O$_2$CO$_3$ and S4 (pure BiOI).

Schematic diagram for energy band of Bi$_2$O$_2$CO$_3$–BiOI, the formation of the p–n junction and the possible charge separation is displayed in Figure 10. We know that p–n junctions can be formed between (Figure 10b), The internal electric field of Bi$_2$O$_2$CO$_3$–BiOI p–n heterojunction promotes the migration rate of photogenerated electrons and holes, which greatly improves the photocatalytic activity. The unique vertical 2D materials on 1D structure makes BiOI nanosheets expose almost entire surface, increasing the separation and transfer rate of photo-generated electron–holes pairs. Furthermore, high-quality interface between Bi$_2$O$_2$CO$_3$ and BiOI decreases the energy barrier for the photogenerated charge carriers transfer at the junction, enhancing the photocatalytic activity.
3. Experimental

3.1. Photocatalyst Preparation

Bismuth nitrate pentahydrate [(Bi(NO$_3$)$_3$·5H$_2$O), Potassium iodide (KI), sodium sulfate (Na$_2$SO$_4$), potassium dichromate (K$_2$Cr$_2$O$_7$), benzoquinone (BQ) and Butyl rhodamine B (RhB) were obtained from Sinopharm Chemical Reagent Co., Ltd. Methyl orange (MO) comes from Tianjin Guangfu Fine Chemical Research Institute. Phenol, Bisphenol A (BPA) were purchased from Shanghai Lingfeng Chemical Reagent Co., Ltd. Ammonium oxalate was obtained from Shanghai Sansihewei Chemical Co., Ltd. Tert-butyl alcohol was purchased from Sinopharm Shanghai Chemical Reagent Company. Ethylene glycol was purchased from Tianjin Fengchuan Chemical Reagent Technology Co., Ltd.

Bi$_2$O$_2$CO$_3$ nanorods: Bi$_2$O$_2$CO$_3$ nanorods were synthesized according to our early report [52].

Bi$_2$O$_2$CO$_3$–BiOI heterostructures: In a typical synthesis, the 0.2 mmol Bi$_2$O$_2$CO$_3$ nanorods and Bi(NO$_3$)$_3$·5H$_2$O were added into 15 mL ethylene glycol. Then, 15 mL KI solution was slowly added into above solution under stirring at 30 °C for 3 h. Deionized water and anhydrous ethanol were used to wash the obtained products and then dry them at 60 °C for 6 h. The as-make products were named as S1, S2, S3 and S4 when the molar ratios of Bi$_2$O$_2$CO$_3$ and BiOI are 10:1, 2:1, 1:2 and 1:20, respectively.

3.2. Photocatalytic Activity Measurements

The photocatalytic performance of Bi$_2$O$_2$CO$_3$–BiOI heterojunction was evaluated by degrading Cr (VI), methyl orange (MO) and bisphenol A (BPA) under solar light irradiation. The 300W Xe lamp (CER-HXF300F, Beijing China Education Au-Light Co. Ltd. Bei Jing, China) was used as light source. 30 mg photocatalyst was dispersed in 30 mL K$_2$Cr$_2$O$_7$ solution (30 mg/L), MO solution (20 mg/L) and BPA solution (20 mg/L), respectively. The suspension kept in dark place while it was stirred. Asan adsorption/desorption equilibrium was achieved, the suspension was illuminated under the solar light. Within a given time, 4 mL was collected and centrifugated. Finally, the supernatant is monitored by UV-vis spectrophotometer.

The trapping experiments of active species were done. During photocatalytic process, P-Benzoquinone (BQ) (0.001 mol/L), t-butanol (0.01 mol·L$^{-1}$) and ammonium oxalate (AO) (0.01 mol·L$^{-1}$) were added into reaction system, which can act as the scavengers to trap superoxide radicals (O$_2^-$), hydroxyl radicals (OH) and hole (h$^+$).

3.3. Electrochemical Impedance Spectroscopy (EIS) Measurements

Electrochemical impedance spectroscopy (EIS) measurements were tested at a frequency between 0.1 Hz and 100 kHz using the CHI760E instrument (Shanghai Chen Hua Company, Shanghai, China) Na$_2$SO$_4$ (0.2 M) was used as detecting electrolyte. The electrode system used a three-electrode system, which platinum wire was used as the counter
electrode and the saturated amphot-mercury electrode is used as a reference electrode. 

\[ \text{Bi}_2\text{O}_2\text{CO}_3, \text{ S2 sample and BiOI film electrodes served as the working electrodes, separately.} \]

3.4. Characterization

X-ray powder diffractometer (XRD) (Shimazu XRD-6000, Kyoto, Japan), scanning Angle of 10–80°. Field emission scanning electron microscope (SEM) (Hitachi S-4800, Tokyo, Japan, operating voltage: 5 Kv); Transmission electron microscope (Hitachi HT7700, Tokyo, Japan); Fluorescence spectrometer (FL, F-4500, Shimadzu, Kyoto, Japan); Infrared spectrometer (IR) (IR-21, Brock, Bochum, Germany); X-ray Photoelectron spectrometer (XPS) (Thermo EscalAB 250Xi, Waltham, MA, United States). A UV vis diffuse-reflectance spectroscopy (DRS) (UV-2450 spectrophotometer, Kyoto, Japan).

4. Conclusions

In conclusion, a novel 1D/2D Bi$_2$O$_2$CO$_3$–BiOI p–n heterojunction was synthesized by hydrothermal method. BiOI nanosheets were uniformly and vertically grown from the interior of Bi$_2$O$_2$CO$_3$ nanorods on the basis of a crystallography-oriented epitaxial mechanism, which provides a small barrier for the transport of photogenerated electron–hole pairs through the junctions because forming high-quality interfaces between Bi$_2$O$_2$CO$_3$ and BiOI. The Bi$_2$O$_2$CO$_3$–BiOI heterojunction photocatalyst exhibits a superior photocatalytic activity for the degradation of Cr (VI), MO and BPA. The outstanding photocatalytic performance is ascribed to the synergistic effects of unique 2D BiOI vertical on 1D Bi$_2$O$_2$CO$_3$ multidimensional structure, the formation of the p–n junction and the high-quality interfaces between Bi$_2$O$_2$CO$_3$ and BiOI. Trapping experiments show that h$^+$ and O$_2^-$ play key roles for photodegradation.

Supplementary Materials: The following are available online at https://www.mdpi.com/article/10.3390/catal11111284/s1, Figure S1: Kinetic linear simulation curves of Cr (VI) (30 mg/L) photodegradation over the synthesized samples under visible light irradiation; Figure S2: Kinetic linear simulation curves of MO (20 mg/L) photodegradation over the synthesized samples under visible light irradiation; Figure S3: Kinetic linear simulation curves of BPA (20 mg/L) photodegradation over the synthesized samples under visible light irradiation.

Author Contributions: Methodology, investigation, N.Z.; data curation, writing—original draft, H.Q.; writing—review and editing, Y.P.; editing and polishing, Y.L. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Acknowledgments: This work is supported by the Natural Science Foundation of Anhui Province (2008085MB33).

Conflicts of Interest: There are no conflict to declare.

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