Synthesis of Bi$_6$O$_6$(OH)$_3$(NO$_3$)$_3$·1.5H$_2$O/ZnO composite material with excellent photocatalytic hydrogen production performance

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ABSTRACT
The heterojunction effect can effectively improve the separation efficiency of the photocatalyst’s photo-generated electron and hole pairs, thereby greatly improving the photocatalytic hydrogen production performance of the photocatalyst. In this paper, Bi$_6$O$_6$(OH)$_3$(NO$_3$)$_3$·1.5H$_2$O (BBN) and ZnO are used to construct and synthesize Bi$_6$O$_6$(OH)$_3$(NO$_3$)$_3$·1.5H$_2$O/ZnO (BBN/ZnO) heterojunction photocatalyst. Under UV-vis light irradiation, the BBN/ZnO composite could generate H$_2$ with a rate of 28.66 μmol·g$^{-1}$·h$^{-1}$, which is higher than pure BBN (0.92 μmol·g$^{-1}$·h$^{-1}$) and ZnO (6.54 μmol·h$^{-1}$·g$^{-1}$) at around 31.1 and 4.4 times, respectively. Moreover, the experimental results found that the composite still exhibits excellent photocatalytic activity and maintains a high and stable activity in the 12-hour experiment with 3 cycles. The possible mechanism to enhance the photocatalytic behavior is attributed to the expanded light absorption range, reduced surface migration resistance, and inhibited recombination of photo-generated electron and hole pairs.

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1. Introduction

With the continuous growth of global energy demand, the research on finding new energy sources has attracted more and more attention [1]. The exploration of clean energy, such as clean and environment-friendly hydrogen, is an important exploration direction of the energy revolution [2]. In 1972, Fujishima and Honda K reported for the first time the phenomenon of catalytic decomposition of water by TiO₂ to generate hydrogen, which paved the way for the research of using solar energy to produce hydrogen by photolysis water, revealed the possibility of hydrogen energy replacing fossil fuels, and it was extremely urgent to develop the photocatalysts to produce hydrogen [3,4]. However, as a single group of spectroscopic catalysts, there are some shortcomings that limit their development. For example, the rapid recombination rate of photoelectron-hole pairs limits their photocatalytic activity [5–7]. To overcome these shortcomings to accelerate the separation of photoelectron-hole pairs, a lot of work has been done to improve their photocatalytic activity. A variety of methods have been proposed to achieve this goal, such as morphology control, element doping, metal deposition, and construction of heterojunction [8–15]. The construction of heterojunction effectively reduces the recombination of photo-induced electrons and holes and improves the quantum efficiency, thereby significantly improving the photocatalytic activity of semiconductors [16–19].

ZnO is one of the most used photocatalysts for photocatalytic hydrogen production because of its abundant storage, chemical stability, high activity, low cost, and non-toxicity [20–24]. The band gap width of photocatalyst ZnO is 3.2 eV, and it only absorbs UV light with a wavelength below 387 nm, accounting for only 3–5% of the whole solar energy [25,26]. The wide band gap of ZnO causes it to be induced only under UV light, and the high recombination rate of photogenic electron-hole pair reduces their photocatalytic activity [27,28]. Similarly, as a semiconductor photocatalyst, basic bismuth nitrate (BBN) has been widely developed and studied to ascribe to its non-toxicity, corrosion resistance, suitable band gap, good photocatalytic degradation activity, and rich crystal chemical components [29–31]. However, the response range of basic bismuth nitrate to sunlight is mainly concentrated in the UV region, and the wide band gap greatly limits its photocatalytic activity [32–34]. By constructing a heterojunction of BBN and ZnO to promote photo-generated charge separation, and secondly, using the well-matched charged potential between BBN and ZnO, thereby significantly enhancing the photocatalytic efficiency of the composite material. At present, heterojunctions such as BBN/BiOCl, ZnO/CdS, ZnO/ZnS, and BiOI/ZnO were constructed to significantly enhance the photocatalytic activity [24,26,29,35].

In this study, BBN/ZnO heterojunction was obtained by calcination process. The experimental results show that the optical absorption range of the prepared composite is significantly expanded, the photocarriers separation ability and the surface electron transport efficiency are significantly improved. Therefore, the hydrogen production capacity of the composite was significantly higher than that of both pure BBN and ZnO. It provides an effective theoretical basis and practical solutions for improving the photocatalytic performance of composite materials prepared by constructing heterojunctions.
2. **Experiment**

2.1. **Materials**

Zinc acetate dihydrate (Zn(Ac)$_2$ · 2H$_2$O), trisodium citrate dihydrate (Na$_3$C$_6$H$_5$O$_7$ · 2H$_2$O), urea (CO(NH)$_2$)$_2$, HNO$_3$ (2 mol·L$^{-1}$), NaOH, Bi(NO$_3$)$_3$ · 5H$_2$O were purchased from Shanghai Macklin biochemical Co., Ltd, China. Triethanolamine (TEOA) was purchased from Xilong Scientific, China.

2.2. **Synthesis**

Solution 1 was obtained by dissolving 0.9 g Bi(NO$_3$)$_3$ · 5H$_2$O into 20 mL HNO$_3$ (2 mol·L$^{-1}$). 0.5 g NaOH was dissolved in 20 mL deionized water to obtain solution 2. Then, slowly add solution 1 to solution 2, stirring continuously for 20 minutes. The mixture was transferred to a Teflon-lined stainless-steel autoclave and heated at 190°C for 4 hours. After the precipitation was cooled to room temperature, it was washed twice with deionized water and ethanol and dried at 60°C for 2 hours to obtain BBN.

0.82 g Zn(Ac)$_2$ · 2H$_2$O, 0.9 g CO(NH)$_2$)$_2$ and 0.2 g Na$_3$C$_6$H$_5$O$_7$ · 2H$_2$O were dissolved in 80 mL distilled water, and the homogeneous solution was formed after acoustic wave treatment. The obtained superior products were placed in a 100-mL stainless steel autoclave lined with Teflon, sealed, stored at 120°C for 6 hours, centrifuged, and dried to obtain sediments. Finally, ZnO is obtained by calcination at 300°C for 2 hours.

We mixed BBN and ZnO in accordance with mass ratios of 1:1, 1:2, 1:3, and 1:6, added appropriate amount of ethanol to grind, and then calcined the grinded sample at 200°C for 3 hours to obtain BBN/ZnO-1-1, BBN/ZnO-1-2, BBN/ZnO-1-3, and BBN/ZnO-1-6.

2.3. **Characterization**

The X-ray Diffraction (XRD) patterns were examined by a BRUKER D8 ADVANCE. The morphology was studied using a scanning electron microscope (SEM, Hitachi Regulus8100). The light absorption capacity was measured by ultraviolet-visible diffuse reflectance spectroscopy (UV-vis DRS, Shimadzu UV 2600). The surface chemical compositions and state were recorded by X-ray photoelectron spectroscopy (XPS, *1/AXIS Ultra DLD). Photoelectrochemical measurements were performed using a CS310H electrochemical workstation (CorrTest Instrument, Wuhan, China) and 300 W xenon lamp (FX300).

2.4. **Photocatalytic reaction**

The hydrogen production rate was used in a full glass automatic on-line trace gas analysis system (Perfert Labsolar-6A) system equipped with gas chromatograph (FL 9700) to test the photocatalytic hydrogen generation performance of the samples. 50 mg of sample was dispersed in 100 mL of triethanolamine solution (10 vol%) at 5°C and a 300 W Xe lamp was served as the light source.
3. Results and discussion

3.1. Characterization

The crystal structures of the samples were determined by XRD patterns, which are showed in Figure 1. The diffraction pattern of BBN has obvious diffraction peaks at 10.14° and 31.13°, which match the (002) and (006) crystal planes (JCPDS card number 53–1038), respectively, and there are other diffraction peaks detected. The prepared ZnO has obvious diffraction peaks at 31.57°, 34.12°, 35.99°, 56.32°, 62.62°, and 67.83° which belong to the (100), (002), (101), (110), (103), and (112) diffraction planes (JCPDS card number 53–1038), respectively. In addition, the characteristic peaks of BBN and ZnO can be found in BBN/ZnO-1-2 composite, indicating the formation of BBN/ZnO composites [36].

The possible morphologies of pure BBN, ZnO and BBN/ZnO-1-2 composites were studied by SEM. As shown in Figure 2, pure BBN is in flake form. In addition, it can be seen from the figure that the morphology of the prepared ZnO sample is a layered microsphere structure composed of nanometer plates. The morphology of BBN/ZnO-1-2 composites is the same as that of ZnO layered microspheres, with BBN small flake particles distributed on the surface. The dense distribution of BBN on the periphery of the sphere verified the successful deposition of BBN on the surface of ZnO [26].

The microstructure of sample BBN/ZnO-1-2 was further investigated by TEM and HRTEM. The BBN was evenly dispersed on the surface of ZnO (Figure 3(a)). It can be clearly seen in Figure 3(b) that there are two different sets of lattice fringes. The interplanar spacing 0.246 and 0.284 nm correspond to the (101) plane of ZnO and (006) plane of BBN, respectively. Most importantly, the slightly deformed diffraction at the interface between BBN and ZnO was observed, revealing the formation of heterostructures. Figure 3(c-g) shows the EDS mapping image of sample BBN/ZnO-1-2, which proves the existence of Bi, N, Zn and O elements, and it also proves that BBN was dispersed on the surface of ZnO.

The valence and surface chemical composition of BBN/ZnO-1-2 sample was studied with XPS. Figure 3(a) shows the investigation spectra of BBN, ZnO and BBN/ZnO-1-2. Obviously, Zn, Bi, O, and N elements were also detected in BBN/ZnO-1-2 samples, and no other impurity elements were detected. Two peaks located at 1019.6 eV and 1042.6 eV could be assigned to Zn 2p$_{3/2}$ and Zn 2p$_{1/2}$ of Zn$^{2+}$ of ZnO, and the binding energy of the Zn 2p$_{3/2}$ and Zn 2p$_{1/2}$ in BBN/ZnO-1-2 increased by 0.3 eV compared with ZnO (Figure 3(b)) [37]. The Bi 4f spectra reveal two peaks at 158.7 and 164.0 eV (Figure 3(c)), which are assigned to the Bi 4f$_{7/2}$ and Bi 4f$_{5/2}$ in the Bi$^{3+}$ chemical state of BBN, and the binding energy of the Bi 4f$_{7/2}$ and Bi 4 f$_{5/2}$ in BBN/ZnO-1-2 increased to 159.3 eV and 164.5 eV compared with BBN [38]. The peak of the N 1s spectrum is 398.2 eV (Figure 3(d)), which can be attributed to the N element in BBN [9]. The enhancement of Bi 4 f and Zn 2p orbital energy can be attributed to the strong interaction between the BBN and ZnO interfaces, which also confirms the formation of BBN/ZnO heterojunction.

The light absorption capacity of pure BBN, ZnO, and BBN/ZnO-1-2 samples was recorded by UV-visible diffuse reflectance spectroscopy (DRS). UV-visible DRS spectrum of the samples prepared is shown in Figure 4(a). Obviously, the light absorption edge of BBN and ZnO is around 380 nm and 390 nm, respectively, while the light absorption edge of BBN/ZnO-1-2 increases to 430 nm. Based on the (ahv)$^{1/2}$ vs photon energy diagram in
Figure 4(b), the band gaps of BBN and ZnO are estimated to be 3.24 eV and 3.14 eV, respectively. The construction of BBN and ZnO heterojunction significantly enhances the absorption intensity of the sample, which was conductive to the formation of more photogenerated electron-hole pairs [39].

3.2. Photocatalytic performance

The photocatalytic activity of the sample was verified by decomposition of water under light to produce hydrogen. The hydrogen production performance of different samples can be clearly seen from Figure 5(a). Obviously, the photocatalytic performance of BBN and ZnO samples is lower, which is attributed to the faster recombination of photogenerated electrons and holes. As the content of ZnO in the composite material increases, the photocatalytic hydrogen production activity is significantly improved. When the mass ratio of BBN to ZnO is 1:2, hydrogen production activity is the highest. As the content of ZnO continues to increase, the photocatalytic activity decreases, but it is still higher than BBN and ZnO. The photocatalytic hydrogen production efficiencies of BBN and ZnO are 0.92 and 6.54, while the efficiencies of composite materials BBN/ZnO-1-1, BBN/ZnO-1-2, BBN/ZnO-1-3, and BBN/ZnO-1-6 are 22.94, 28.66, 17.22, and 16.12, respectively, and BBN/ZnO-1-2 approaches the highest photocatalytic hydrogen production efficiency, which is 31.1 and 4.4 times that of BBN and ZnO, respectively. The cycle H\textsubscript{2} production experiment was carried out for the best performance sample BBN/ZnO-1-2. It can...
be seen from Figure 5(b) that the BBN/ZnO-1-2 photocatalyst has always maintained a high and stable activity in the 12-hour cycle experiment with 3 cycles, which shows that the composite material has excellent activity and stability during the photocatalytic reaction. In addition, it can be seen from Figure 5(c) that the
photocatalytic hydrogen production efficiency of the BBN/ZnO-1-2 sample remained stable activity during the continuous 12-hour hydrogen production experiment.

### 3.3. Photoelectric performance

The photocurrent response measurements and electrochemical impedance spectroscopy (EIS) were performed to further investigate the charge separation and migration behavior of BBN and ZnO. The transient photocurrent–time curves of the samples were measured by several intermittent switching cycles. It can be seen from Figure 6(a) that the photocurrent increases rapidly under the activation of light. At the same time, after several intermittent exposures, the photocurrent remains stable and repeatable. Compared with pure BBN and ZnO, the BBN/ZnO-1-2 showed higher photocurrent response, indicating that the separation rate of photogenerated electrons and holes was significantly improved when BBN and ZnO formed heterojunction. The interface charge transport of BBN, ZnO and BBN/ZnO-1-2 was studied by using the electrochemical Nyquist diagram. As shown in Figure 6(b), the radius of each arc is related to the resistance of the electrode/electrolyte interface, the smaller the arc radius of the sample, the smaller the corresponding interface transmission resistance [40]. Compared with pure BBN and ZnO, the arc radius of BBN/ZnO-1-2 sample is the smallest, indicating that the formation of

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**Figure 4.** XPS spectrum of BBN, ZnO and BBN/ZnO-1-2: the survey spectrum (a), Zn 2p spectrum (b), Bi 4f spectrum (c), N 1s spectrum (d).
heterojunction enhances the interface charge transfer rate, and further improves the electron-hole separation efficiency. The photocurrent response of ZnO is stronger than that of BBN, and the surface impedance is larger than that of BBN. This indicates that as the content of ZnO increases, the photoresponse of the composite material increases, but the surface charge transfer efficiency is suppressed. Therefore, BBN/ZnO-1-2 exhibits the best photocatalytic hydrogen production activity.

3.4. Photocatalytic mechanism

The Mott-Schottky method was used to determine the semiconductor type and potential of BBN and ZnO. As shown in Figure 7(a-b), the slopes of the linear $1/C^2$ potential curves of BBN and ZnO are both positive, indicating that BBN and ZnO are n-type semiconductors. The flat band potential ($V_{fb}$) of a semiconductor could be evaluated by extrapolating the tangent of the curve to x-axis [7]. The resulted $V_{fb}$ of BBN and ZnO electrodes is $-1.64$ V vs. SCE (i.e. $-1.04$ V vs. NHE) and $-0.99$ V vs. SCE (i.e. $-0.39$ V vs. NHE). The conductor band ($E_{CB}$) is close to the $V_{fb}$ for n-type semiconductors [8]. Thus, the valence band edge potential ($E_{VB}$) of BBN and ZnO calculated by the equation ($E_{CB} = E_{VB} - E_{g}$) was 2.26 V and 2.81 V, respectively.

According to the above experimental results, the charge separation process of BBN/ZnO heterojunction is shown in Figure 7. Under UV-vis light irradiation, the electrons of BBN and ZnO are excited to the conduction band, while the holes remain in the valence band. The conduction band potential of BBN ($-1.04$ eV) is more negative than that of ZnO ($-0.39$ eV), so electrons are transferred from the conduction band of BBN to the conduction band of ZnO, and the $H^+$ adsorbed on the photocatalyst surface is reduced to $H_2$. In addition, the valence band potential of ZnO (2.81 eV) is higher than that of BBN (2.26 eV). The holes transfer from the valence band of ZnO to the valence band of BBN and react with TEOA to form TEOA$^+$. The construction of heterojunction effectively separates photogenerated electrons and holes, thereby obtaining higher photocatalytic hydrogen production activity.

Figure 5. UV-vis diffuse reflectance spectra (a) and plots of $(ahv)^{1/2}$ vs photon energy (b).
Figure 6. Time course of photocatalytic activities for various samples (a), the cycling stability for sample BBN/ZnO-1-2 (b), and a longer stability test of BBN/ZnO-1-2 sample.

Figure 7. (a) Transient photocurrent responses and (b) Nyquist plots of electrochemical impedance spectroscopy (EIS) plots of the as-prepared samples.
Conclusion

In summary, BBN/ZNO heterojunction is constructed and its photocatalytic hydrogen production performance is better than both pure BBN and ZNO. The photocatalytic

Figure 8. Mott-Schottky curves of BBN (a) and ZnO (b).

Figure 9. Schematic diagrams of formation of heterojunction and proposed charge separation process in the BBN/ZnO composite.

4. Conclusion

In summary, BBN/ZNO heterojunction is constructed and its photocatalytic hydrogen production performance is better than both pure BBN and ZNO. The photocatalytic
hydrogen production performance of BBN/ZnO composites was better than that of pure BBN and ZnO. The heterojunction between ZnO and BBN has highly efficient photocatalytic activity, which is mainly attributed to the enhanced light absorption capacity, reduced interface migration resistance, and electron-hole pair recombination rate.

**Disclosure of potential conflicts of interest**

No potential conflict of interest was reported by the author(s).

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