

# Research Article

# Hydrothermal Synthesis of $Bi_2S_3$ Nanorods from a Single-Source Precursor and Their Promotional Effect on the Photocatalysis of $TiO_2$

## Juan Lu, Zuoshan Wang, Ying Zhang, and Xiufeng Zhou

College of Chemistry, Chemical Engineering and Materials Science, Soochow University, Soochow 215021, China

Correspondence should be addressed to Ying Zhang; yingzhang@suda.edu.cn

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As a direct bandgap semiconductor,  $Bi_2S_3$  has the potential ability to improve the photocatalytic activity of nano-TiO<sub>2</sub> due to its low energy gap (Eg = 1.3 eV). In this study, large-scale uniform  $Bi_2S_3$  nanorods were synthesized by a hydrothermal treatment, using  $Bi[S_2P(OC_4H_9)_2]_3$  as the single-source precursor. Characterization results show that as-prepared samples belong to an orthorhombic phase of  $Bi_2S_3$ , and the products mainly crystallize in the form of nanorods which measure ca. 200 nm in length and ca. 50 nm in diameter. The photo-catalytic experiments for the degradation of methyl orange under visible irradiation revealed that a small amount of as-prepared  $Bi_2S_3$  in our study would significantly improve the photo-catalytic activity of nano-TiO<sub>2</sub>, whether  $Bi_2S_3$  is introduced by a physical way or a chemical way. However, excess  $Bi_2S_3$  will lead to a decrease in the catalytic efficiency of TiO<sub>2</sub> when  $Bi_2S_3$  was introduced by a chemical way; it never happened when  $Bi_2S_3$  introduced by a physical way. Even so, among all as-prepared samples, the TiO<sub>2</sub>-based photo-catalyst with 3 wt.%  $Bi_2S_3$  introduced by a chemical way exhibits the best catalytic performance under visible irradiation.

### 1. Introduction

As a direct bandgap semiconductor,  $Bi_2S_3$  has the potential ability to improve the photocatalytic activity of nano-TiO<sub>2</sub> due to its low energy gap (Eg = 1.3 eV) [1–9]. As we all know, material's properties strongly depend on its structure which in turn has a close relationship with its preparation method. Therefore, in the case of the same TiO<sub>2</sub>, the photocatalytic activity of  $Bi_2S_3/TiO_2$  heterojunction will mainly rely on two aspects, including the different preparation methods for  $Bi_2S_3$ and the different ways to introduce  $Bi_2S_3$  into TiO<sub>2</sub>.

Currently, there are many classic preparation methods for  $Bi_2S_3$  nanoparticles or superstructures, such as hydrothermal method [10–12], solvothermal method [13], electrodeposition technique [14], chemical deposition [15, 16], spray pyrolysis deposition [17], microwave refluxing [18–21], and single-source precursor approach [22–28]. Among them, we select the single-source precursor approach to prepared  $Bi_2S_3$  in our study because this method is effective in synthesizing a large number of products with uniform size. Besides, in most of the previous reports,  $Bi_2S_3$  was introduced into  $TiO_2$  by a chemical way. There are few articles that simultaneously related to the introduction of  $Bi_2S_3$  into  $TiO_2$  by a physical way and a chemical way and discussed their different effects on photocatalytic efficiency. To the best of our knowledge, only Bessekhouad et al. [8] reported the relational research. Nevertheless, their study was a little rough for the proportion of  $Bi_2S_3$  in  $Bi_2S_3/TiO_2$  heterojunction increased from 10 wt.% to 50 wt.%, with an increase of 20 wt.% each time. Therefore, it is still interesting to study in detail the influence of different preparation methods for  $Bi_2S_3/TiO_2$  heterojunctions on their photocatalytic activity.

In this study,  $Bi[(S_2P(OC_4H_9)_2]_3$  was selected as the single precursor to prepare  $Bi_2S_3$  nanorods by a hydrothermal approach. As-prepared samples would significantly improve the photocatalytic activity of nano-TiO<sub>2</sub> for degradation of methyl orange (MO) under visible irradiation, whether  $Bi_2S_3$  is introduced by a physical way or a chemical way. Meanwhile, the influences of different proportions of  $Bi_2S_3$  on the catalytic efficiency of TiO<sub>2</sub> were discussed in detail.

#### 2. Experimental

All reactants and solvents are in analytical grade and are used without further purification.

2.1. Preparation of  $Bi[S_2P(OC_4H_9)_2]_3$ . At the beginning, 0.4 mol sec-butyl alcohol ( $C_4H_{10}O$ ) and 0.1 mol phosphorus pentasulfide ( $P_2S_5$ ) were mixed together and stirred at the room temperature for 3 h. 0.5 mol of NaOH was added into the system by every 20 min during this period. Then, the obtained product and bismuth nitrate ( $Bi(NO_3)_3 \cdot 5H_2O$ ) were dissolved in deionized water and DMF, respectively. Bismuth (III) dialkyldithiophosphate complex ( $Bi[S_2P(OC_4H_9)_2]_3$ ) was finally precipitated by mixing the two solutions with stirring.

2.2. Preparation of  $Bi_2S_3$  Nanorods. 1.5 g of clean and dry  $Bi[S_2P(OC_4H_9)_2]_3$  was dissolved in 16 mL of deionized water and was transferred into a teflon-liner autoclave of 20 mL capacity, maintained constantly at 180°C for 12 h. After the reaction, the mixture was cooled naturally to room temperature, and the precipitate was filtered, washed with water and absolute ethanol for several times, and dried in air for characterization.

2.3. Preparation of  $Bi_2S_3/TiO_2$  Heterojunctions.  $Bi_2S_3/TiO_2$  heterojunction was prepared by two different methods. One is designed as a physical way, in which the two constituents (as-prepared  $Bi_2S_3$  and commercial  $TiO_2$ -P25) were directly mixed together with different concentrations (1–20 wt. % for  $Bi_2S_3$ ) and ground for about 5 min, and the other is designed as a chemical way, carried out by adding commercial  $TiO_2$ -P25 into the reaction system during the preparation of  $Bi_2S_3$ .

2.4. Photocatalytic Experiments. The promotion effect of asprepared samples on the photocatalytic efficiency of TiO<sub>2</sub> was evaluated by measuring the degradation of MO under visible irradiation (500 W Xe lamp), using as-prepared Bi<sub>2</sub>S<sub>3</sub>/TiO<sub>2</sub> heterojunction as the catalyst. A cut filter (ZJB 420) was inserted between the Xenon lamp and reactor to eliminate ultraviolet light, when the experiments were performed under visible light irradiation. In a typical experiment, 0.2 g of Bi<sub>2</sub>S<sub>3</sub>/TiO<sub>2</sub> heterojunction was dispersed in 400 mL of methyl orange (MO,  $20 \text{ mg} \cdot \text{L}^{-1}$ ) solution, with a 300-W high pressure Hg lamp providing irradiation with a wavelength centered at 365 nm and an air flow at the rate of  $10 \text{ mL} \cdot \text{min}^{-1}$ . The actual effect of photocatalytic activity by chemical reaction was studied by maintaining the solution in dark for 1h before irradiation to reach the balance between adsorption and desorption. At given irradiation time intervals, the samples (5 mL) were taken out every 5 min and analyzed by UV-Vis spectrophotometer. The measure of the maximum absorbance was taken at 465 nm. The percentage of degradation is calculated via the formula  $(1 - A_i/A_0)$ , where  $A_0$  is the absorbance of original MO solution after being maintained in dark for 1 h before irradiation, and  $A_i$  is the absorbance of MO solution measured every 5 min during the process of photodegradation.

2.5. Characterization. The X-ray diffraction patterns (XRD) were recorded on a Bruker D8 Advanced X-ray diffractometer using Cu K $\alpha$  radiation ( $\lambda = 0.1542$  nm) with the range of the diffraction angle of  $2\theta = 15 \sim 75^{\circ}$ . Energy dispersion X-ray spectra (EDS) were performed with a GENESIS 2000 X-ray energy spectrometer (EDAX). Transmission electron microscopic (TEM) and scanning electron microscopic (SEM) images were carried out on a JEM-2100 microscope (JEOL) and JSM-6380LV scanning electron microscope, respectively. Ruili 1100 spectrophotometer was used to record the UV-Visible absorption spectra of the as-prepared samples.

#### 3. Results and Discussion

3.1. XRD and EDS Analyses of  $Bi_2S_3$ . Figure 1(a) shows the XRD patterns of the samples obtained at different temperatures. The main diffraction peaks are labeled, and all the reflections can be indexed to an orthorhombic phase of  $Bi_2S_3$ (JCPDS Files, No.17-320). No impurities such as  $Bi_2O_3$ , Bi and S are detected. Obviously, the shapes of the diffraction peaks indicate that the product should be well crystallized at 180°C. Therefore, the purity and composition of the sample obtained at 180°C are reflected by EDS analysis. The detected peaks in the EDS spectrum, shown in Figure 1(b), are assigned to Bi, S, C, and O, implying that there are no obvious impurities except trace amount of  $CO_2$  and  $O_2$  absorbed on the surface of the sample. Quantification of the EDS peaks gives the atomic ratio of Bi : S as 56.48 : 34.76 which is nearly consistent with the given formula of  $Bi_2S_3$ .

3.2. SEM and TEM Images of  $Bi_2S_3$ . The morphology of asprepared sample obtained at 180°C is revealed by SEM and TEM images, and the results are shown in Figure 2. The product mainly consists of many short rods with an average length of ca. 400 nm and a diameter of ca. 50 nm. The typical HRTEM image (Figure 2(c)) shows that the crystal lattice fringes, with an average neighboring distance of 0.42 nm, correspond to the (2 2 0) crystal plane of orthorhombicstructured  $Bi_2S_3$ , indicating as-prepared  $Bi_2S_3$  nanorods grow along the [0 0 1] direction.

3.3. TEM Images of Pure TiO<sub>2</sub> and  $Bi_2S_3$  (3 wt.%)/TiO<sub>2</sub> Heterojunctions. Figure 3 shows the TEM images of the pure commercial TiO<sub>2</sub> (Figure 3(a)),  $Bi_2S_3$  (3 wt.%)/TiO<sub>2</sub> heterojunctions prepared, respectively, by a physical way (Figure 3(b)) and a chemical way (Figure 3(c)). It is obviously observed that the pure TiO<sub>2</sub> was assembled particles with smooth borderlines, and the heterojunction contained TiO<sub>2</sub> particles and as-prepared  $Bi_2S_3$  nanorods, whether  $Bi_2S_3$ introduced by a physical way or a chemical way. The only difference is that the chemically introduced  $Bi_2S_3$  nanorods seemed to be growing on the surface of the TiO<sub>2</sub> particles.

3.4. Photocatalytic Activity. The photocatalytic activity of the  $Bi_2S_3/TiO_2$  heterojunction under visible irradiation is showed in Figure 4(a) (prepared by a physical way) and Figure 4(b) (prepared by a chemical way). It can be observed from Figure 4(a) that when  $Bi_2S_3$  was introduced



FIGURE 1: (a) XRD patterns of as-prepared samples at different temperatures; (b) EDS spectrum of the sample obtained at 180°C.



FIGURE 2: (a) and (b) SEM and TEM images of as-prepared sample obtained at 180°C; (c) the HRTEM image taken from an individual  $Bi_2S_3$  nanorod and its FFTs (inset).



FIGURE 3: TEM images of pure  $TiO_2$  (a) and  $Bi_2S_3/TiO_2$  heterojunctions prepared by different methods: (b) a physical way; (c) a chemical way.



FIGURE 4: Degradation efficiency of MO for  $Bi_2S_3/TiO_2$  heterojunctions under visible irradiation: (a) prepared by a physical way; (b) prepared by a chemical way.

by a physical way, all samples exhibited a higher efficiency than that of the pure TiO<sub>2</sub>, and the catalytic efficiency of the heterojunction increased with increasing the proportion of  $Bi_2S_3$  from 1 wt.% to 20 wt.%. This indicates that as-prepared  $Bi_2S_3$  nanorods introduced by a physical way have an indubitable promotional effect on the photocatalytic activity of TiO<sub>2</sub>. When the  $Bi_2S_3$  was introduced by a chemical way, the  $Bi_2S_3/TiO_2$  heterojunction also showed a better efficiency than that of the pure TiO<sub>2</sub> as the proportion of  $Bi_2S_3$  was in the range of 1 wt.% to 10 wt.%. However, further increasing  $Bi_2S_3$  till 15 wt.% and 20 wt.% caused the catalytic efficiency to be decreased instead. Above all, the chemical introduced  $Bi_2S_3$  with a proportion of 3 wt.% most significantly improved the catalytic efficiency of TiO<sub>2</sub> under visible irradiation.

3.5. Mechanism.  $Bi_2S_3$  was able to efficiently improve the photocatalytic performance of  $TiO_2$  mainly because it has a narrower bandgap than that of  $TiO_2$  (1.3 eV and 3.2 eV, resp.). When the two semiconductors get in contact, the photo-generated electrons in conduction band (CB) of  $TiO_2$  will transfer to valence band (VB) of  $Bi_2S_3$  first, but not to recombine with the photo-generated holes in VB of  $TiO_2$ , as the energy level of VB of  $Bi_2S_3$  is located between those of the VB and CB of  $TiO_2$ , as shown in Figure 5. In addition, a small number of electrons would further transfer to the higher CB of  $Bi_2S_3$  after being excited by UV irradiation. Consequently, more and more positive holes would be left and take part in the reactions of oxidizing  $OH^-$  and  $H_2O$  into hydroxyl radical (\*OH) which is finally responsible for the degradation of pollutants.

However, excess  $Bi_2S_3$  will lead to a decrease in the catalytic efficiency of  $TiO_2$ , when  $Bi_2S_3$  was introduced by



FIGURE 5: Energetic diagrams of Bi<sub>2</sub>S<sub>3</sub>/TiO<sub>2</sub> heterojunction.

a chemical way, it never happened when  $Bi_2S_3$  was introduced by a physical way. The most likely explanation can be elaborated from the structure of the material as follows. Originally, adding more  $Bi_2S_3$  would bring an increase of the probability of interparticle collisions which is beneficial for the improvement of the degradation efficiency. After that, the two constituents— $Bi_2S_3$  and  $TiO_2$ —were separated (Figure 3(b)) in the heterojunction prepared by a physical way. That is why the catalytic efficiency of the physically prepared  $Bi_2S_3/TiO_2$  heterojunction increased with increasing the proportion of  $Bi_2S_3$ . But if the photocatalyst was prepared chemically, some active points on the surface of  $TiO_2$  might be covered by excess  $Bi_2S_3$  and lose their catalytic activity, since  $Bi_2S_3$  nanorods were growing on the surface of  $TiO_2$  particles (Figure 3(c)), and  $Bi_2S_3$  itself has few effects on the degradation of MO (Figure 4(a)). That is why the chemically prepared heterojunction with too much  $Bi_2S_3$  will show a decreasing efficiency. In fact, it can be observed from Figures 4(a) and 4(b) that in the case of a small amount of  $Bi_2S_3$  added, the chemically prepared photo-catalyst exhibits a higher efficiency than that prepared by a physical way, and vice versa.

So far, some researchers have studied the photocatalytic activity of  $Bi_2S_3/TiO_2$  heterojunction, but there are few reports related to its photocatalytic activity using MO solution as the target pollutant. Compared with the reported value [8], although the final efficiency is very close, less  $Bi_2S_3$  (3 wt.%) is needed than that (10 wt.%) in their work. It can be concluded that the as-prepared  $Bi_2S_3$  in our study has an enhanced effect on the promotion of the photocatalytic activity of  $TiO_2$  mainly due to its different preparation method.

#### 4. Conclusions

Large-scale uniform  $Bi_2S_3$  nanorods were synthesized by a hydrothermal treatment, using  $Bi[S_2P(C_4H_9O)_2]_3$  as a singlesource precursor. Results of photocatalytic experiments showed that a small amount of as-prepared  $Bi_2S_3$  would significantly improve the photocatalytic activity of nano-TiO<sub>2</sub> under visible irradiation, whether  $Bi_2S_3$  is introduced by a physical way or a chemical way. After that, excess  $Bi_2S_3$  will make the catalytic efficiency of TiO<sub>2</sub> decreased when  $Bi_2S_3$ was introduced by a chemical way, but it never happened when  $Bi_2S_3$  was introduced by a physical way, mainly due to the different structures of  $Bi_2S_3/TiO_2$  heterojunction resulted from different preparation methods. Even so, among all asprepared samples, the TiO<sub>2</sub>-based photo-catalyst prepared chemically with 3 wt.%  $Bi_2S_3$  exhibits the best catalyst efficiency under visible irradiation.

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