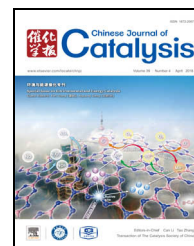


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In-situ transformation of Bi_2WO_6 to highly photoreactive $\text{Bi}_2\text{WO}_6@ \text{Bi}_2\text{S}_3$ nanoplate via ion exchange

Tingting Huang ^a, Yuhan Li ^{a,b}, Xiaofeng Wu ^a, Kangle Lv ^{a,*}, Qin Li ^a, Mei Li ^{a,#}, Dongyun Du ^a, Hengpeng Ye ^a

^a Key Laboratory of Catalysis and Materials Science of the State Ethnic Affairs Commission & Ministry of Education, Hubei Province, College of Resources and Environmental Science, South-Central University for Nationalities, Wuhan 430074, Hubei, China

^b Department of Science and Environmental Studies, The Education University of Hong Kong, Tai Po, N.T., Hong Kong, China

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ABSTRACT

As a two dimensional (2D) visible-light-responsive semiconductor photocatalyst, the photoreactivity of Bi_2WO_6 is not high enough for practical application owing to its limited response to visible light and rapid recombination of photogenerated electron-hole pairs. In this paper, 2D core-shell structured $\text{Bi}_2\text{WO}_6@ \text{Bi}_2\text{S}_3$ nanoplates were prepared by calcination of a mixture of Bi_2WO_6 (1.3 g) and a certain amount of $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ (0–3.0 g) at 350 °C for 2 h. The reactivity of the resulting photocatalyst materials was evaluated by photocatalytic degradation of Brilliant Red X-3B (X3B), an anionic dye, under visible light irradiation ($\lambda > 420$ nm). As the amount of $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ was increased from 0 to 1.5 g, the degradation rate constant of X3B sharply increased from 0.40×10^{-3} to $6.6 \times 10^{-3} \text{ min}^{-1}$. The enhanced photocatalytic activity of $\text{Bi}_2\text{WO}_6@ \text{Bi}_2\text{S}_3$ was attributed to the photosensitization of Bi_2S_3 , which greatly extended the light-responsive range from the visible to the NIR, and the formation of a heterojunction, which retarded the recombination rate of photogenerated electron-hole pairs. However, further increases in the amount of $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ (from 1.5 to 3.0 g) resulted in a decrease of the photocatalytic activity of the $\text{Bi}_2\text{WO}_6@ \text{Bi}_2\text{S}_3$ nanoplates owing to the formation of a photo-inactive NaBiS_2 layer covering the Bi_2WO_6 surface.

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1. Introduction

Recently, photocatalytic degradation of harmful pollutants has become an increasingly important technology for water and air purification [1]. TiO_2 is considered to be an excellent photocatalyst because of its biological and chemical inertness, strong photo-oxidization power, cost effectiveness, and long-term stability to photo-induced and chemical corrosion [2]; however, TiO_2 has a broad bandgap (3.2 eV for anatase

TiO_2) and absorbs only a small fraction of solar energy [3,4]. Considering that UV light constitutes only 3%–5% of the solar spectrum, more efficient photocatalysts, which can be activated both by UV and visible light, are highly desired [5–7].

As one of the simplest Aurivillius oxides with a layered 2D structure, Bi_2WO_6 nanoplates are notable for their visible-light-response and photocatalytic performance [8–12]. The perovskite-like structure is defined by WO_6 units that form a layer perpendicular to the [100] direction and sandwich

* Corresponding author. Tel: +86-27-67841369; Fax: +86-27-67843918; E-mail: lvkangle@mail.scuec.edu.cn

Corresponding author. Tel: +86-27-67842752; Fax: +86-27-67842752; E-mail: limei@mail.scuec.edu.cn

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(Bi_2O_2) $^{2+}$ units [13,14]. These nanoplate materials are usually characterized by high crystallinity and well-defined chemical compositions as well as extremely high anisotropy, with ultrathin plates [8]. However, the photocatalytic activity of Bi_2WO_6 nanoplates is not yet high enough for practical applications because of the rapid recombination of photogenerated electrons and holes, and the limited visible-light-response range [15,16]. Unmodified Bi_2WO_6 nanoplates can only adsorb UV or visible light of wavelengths shorter than 450 nm [17,18].

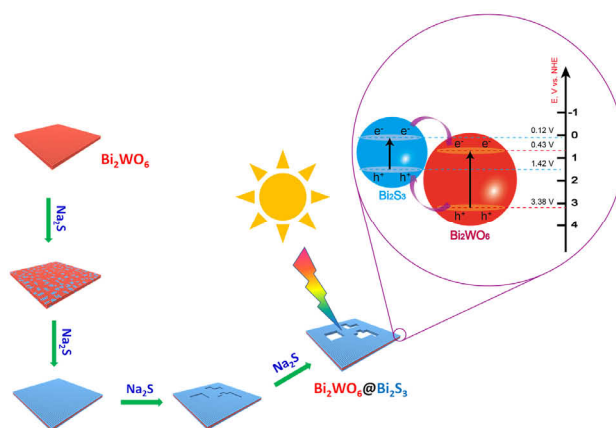
To improve the photocatalytic activity of Bi_2WO_6 nanoplates and/or extend the range of light response, many strategies have been studied, including semiconductor coupling [19], metal/non-metal doping and noble metal deposition [20]. For example, Ge *et al.* [21] reported a red shift of the light absorption and strong absorbance of visible light region after coupling Bi_2WO_6 with g- C_3N_4 . The enhancement in the photocatalytic performance of the g- C_3N_4 @ Bi_2WO_6 composite materials was attributed to the combination of polymeric g- C_3N_4 and Bi_2WO_6 , which improved separation of photogenerated electron-hole pairs [21]. Similarly, our group [3] reported the coupling of Bi_2WO_6 with TiO_2 using a layer-by-layer deposition and calcination strategy. We found that a hybrid coated with four layers of TiO_2 (containing 20 wt% TiO_2) showed the highest photocatalytic activity, which was 10.45 and 3.20 times as high as that of pure Bi_2WO_6 and TiO_2 , respectively. Zhu *et al.* [22] reported that, doping Bi_2WO_6 by F resulted in a two-fold increase in the photocatalytic degradation activity of MB under visible-light irradiation ($\lambda > 420$ nm). The enhanced photocatalytic activity was attributed to increased mobility of photo-excited charge carriers and a positive shift of the valence band position. Yu *et al.* [1] reported the fabrication of high visible photoreactive plasmonic Ag and graphene co-modified Bi_2WO_6 nanosheets, combining the surface plasmon resonance (SPR) effect of Ag and the electronic properties of graphene at the surface of Bi_2WO_6 .

Bismuth sulfide (Bi_2S_3) has been reported to be a semiconductor with a narrow bandgap of approximately 1.3 eV [12,23–25], which has been used as a sensitizer because of its ability to absorb visible and even near infrared light [26–28]. Here, we propose a structure of Bi_2WO_6 nanoplates with a surface layer of Bi_2S_3 to improve the photoreactivity of the material. Considering that strong interactions between two semiconductors is of great importance for efficient separation of carriers [29], the Bi_2S_3 was designed to grow on the surface of the Bi_2WO_6 nanoplate *via* an in-situ ion-exchange strategy, to form core-shell structured Bi_2WO_6 @ Bi_2S_3 nanoplates (Scheme 1). The effects of the amount of Bi_2S_3 loading on the structure and photocatalytic activity of the Bi_2WO_6 @ Bi_2S_3 nanoplates were systematically studied.

2. Experimental

2.1. Preparation of Bi_2WO_6 @ Bi_2S_3 nanoplates

All reagents were of analytical purity and were used as received from the Shanghai Chem. Co., China. The Bi_2WO_6 @ Bi_2S_3 nanoplate hybridized photocatalyst was synthesized by an ion



Scheme 1. Proposed formation mechanism of Bi_2WO_6 @ Bi_2S_3 nanoplate *via* ion exchange and the photoreactivity enhancement mechanism.

exchange strategy using Bi_2WO_6 nanoplates as a precursor, which were synthesized by a simple hydrothermal process according to the literature [8]. To synthesize the Bi_2WO_6 @ Bi_2S_3 nanoplates, 1.3 g of square Bi_2WO_6 precursor was added to a beaker containing 40 mL of $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ solution under magnetic stirring. This mixture was then evaporated to dryness in an oven at 60 °C for 4 h. The resulting powder was further calcined at 350 °C for 2 h in muffle furnace at a heating rate of 5 °C min^{-1} . Afterwards, the obtained samples were rinsed with anhydrous ethanol followed by drying in an oven at 80 °C for 2 h. The prepared hybridized photocatalysts are denoted as S_x , where x indicates the amount of $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ used (0–3.0 g, Table 1). Thus, the S0 sample represents the 350 °C-calcined Bi_2WO_6 precursor without treatment by the Na_2S solution.

2.2. Characterization

X-ray diffraction (XRD) patterns were obtained on a D8-advance X-ray diffractometer (German Bruker) using $\text{Cu } K\alpha$ radiation at a scan rate of 0.02° s^{-1} . The accelerated voltage and applied current were 15 kV and 20 mA, respectively. The morphology of the catalysts was characterized with a field emission scanning electron microscope (FESEM, S-4800, Hitachi, Japan) operating at an accelerating voltage of 10 kV and a transmission electron microscopy (TEM: Tecnai G² 20, USA) with an accelerating voltage of 200 kV. The optical absorption properties of the samples were investigated through diffuse reflectance spectrum (DRS) with a UV-Vis spectrophotometer (Lambda, Bio 35, PE Co., USA). Powder photoluminescence (PL) spectrum were measured at room temperature on a Fluorescence Spectrophotometer (F-7000, Hitachi, Japan). The excita-

Table 1
Physical property of the photocatalyst.

Sample	A_{BET} ($\text{m}^2 \text{g}^{-1}$)	PV ($\text{cm}^3 \text{g}^{-1}$)	APS (nm)
S0	18.8	0.08	18.7
S0.75	30.3	0.12	15.7
S1.0	29.5	0.12	14.6
S1.5	42.6	0.18	15.6
S2.0	39.7	0.23	23.1
S3.0	17.8	0.16	35.2

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