



# Facile assembly of Bi<sub>2</sub>O<sub>3</sub>/Bi<sub>2</sub>S<sub>3</sub>/MoS<sub>2</sub> *n-p* heterojunction with layered *n*-Bi<sub>2</sub>O<sub>3</sub> and *p*-MoS<sub>2</sub> for enhanced photocatalytic water oxidation and pollutant degradation

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## ABSTRACT

As an important half reaction in solar-driven water splitting, it is still a challenging issue to develop low-cost and high efficient photocatalysts for water oxidation process. In this study, we reported a facile strategy for controllable synthesis of three-component Bi<sub>2</sub>O<sub>3</sub>/Bi<sub>2</sub>S<sub>3</sub>/MoS<sub>2</sub> *n-p* heterojunction based on the formation of the intermediate Bi<sub>2</sub>S<sub>3</sub> by coupling Bi<sub>2</sub>O<sub>3</sub> and MoS<sub>2</sub>. The Bi<sub>2</sub>S<sub>3</sub> was easily formed due to the strong interaction between Bi<sup>3+</sup> and S<sup>2-</sup> ions with the assistance of the hydrothermal treatment. As a result, the prepared Bi<sub>2</sub>O<sub>3</sub>/Bi<sub>2</sub>S<sub>3</sub>/MoS<sub>2</sub> nanocomposite exhibits enhanced ability of photocatalytic water oxidation (529.1 μmol h<sup>-1</sup> g<sup>-1</sup><sub>cat</sub>), which is 1.5 and 12.5 times higher than that of pure Bi<sub>2</sub>O<sub>3</sub> and MoS<sub>2</sub>, respectively, under simulated solar light irradiation. Furthermore, the photoelectrochemical results reveal that the charge transportation feature and the donor density were apparently enhanced after the introduction of highly conductive layered MoS<sub>2</sub>, which indicates the enhancement of the photo-response and the improvement of charge separation efficiency.

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## 1. Introduction

Energy crisis triggers the rapid development of promising and inexpensive methods for clean energy production, such as photocatalytic water splitting [1,2]. It is well-known that the photocatalytic water splitting process includes hydrogen evolution reaction and oxygen evolution reaction. In general, oxygen evolution reaction is multi-electrons transfer reaction, which is a sluggish process and involves complicated breaking of O–H bond and formation of O–O bond [3]. Currently, most of efficient water oxidation catalysts (WOCs) consist of noble metals Ru and Ir, however, the scarcity and high cost hinder their widespread application [4–6]. In order to reduce the cost, some transition metals are utilized to synthesize promising WOCs due to their less expense, earth-abundance, and low toxicity [7–9]. Bismuth trioxide (Bi<sub>2</sub>O<sub>3</sub>) stands out among many attractive candidate materials because of its low cost, favorable direct band gap of 2.5–2.8 eV, and good photostability in acidic conditions [10,11]. The valence band edge of Bi<sub>2</sub>O<sub>3</sub>

(+3.13 V vs NHE) is enough positive to provide sufficient potential for oxygen production. In addition, Bi<sub>2</sub>O<sub>3</sub> is one of semiconductors with multiple crystal structures, possessing either *n*-type or *p*-type properties [12] and allowing it to be easily used in sensor, catalysis, and photovoltaic field [13–15]. Nevertheless, as similar to many semiconductors, the poor harvesting of solar energy and charge carrier separation of pure Bi<sub>2</sub>O<sub>3</sub> leads to the low photocatalytic activity and thus cannot meet the demand of commercial applications.

On the other hand, in contrast to single component photocatalysts, heterojunction photocatalysts that combine at least two disparate functional materials in one system have received more attention in the past decades [16–18]. Among advanced heterojunction materials, *p-n* heterojunctions are extensively studied and reported, which simultaneously realize high utilization of solar energy and efficiency of photoinduced charge separation by means of formation of an inner electric field between the *n*-type and *p*-type semiconductors [19]. Therefore, to improve the photocatalytic performance of Bi<sub>2</sub>O<sub>3</sub>, it is necessary to couple Bi<sub>2</sub>O<sub>3</sub> with other semiconductors for constructing a heterojunction system. Among numerous semiconductors, molybdenum disulphide (MoS<sub>2</sub>), as a graphene-like transition metal dichalcogenide, may be a good candidate for tuning photo-response and improving charge carrier transportation properties [20–23]. In fact, MoS<sub>2</sub> is often used

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as an effective cocatalyst in the photocatalytic or electrocatalytic hydrogen evolution reaction due to its large surface area and high electrical conductivity [20,24–26]. These studies reveal that the incorporation of layered MoS<sub>2</sub> with a metal oxide can promote the photocatalytic activity.

Based on the above strategy, herein, by means of coupling Bi<sub>2</sub>O<sub>3</sub> microplates with layered MoS<sub>2</sub>, we successfully fabricated Bi<sub>2</sub>O<sub>3</sub>/Bi<sub>2</sub>S<sub>3</sub>/MoS<sub>2</sub> composites to form a three-phase *p-n* heterojunction for improving efficiency of solar energy utilization and photoinduced charge transportation. The three-component *n-p* heterojunction system displays enhanced photocatalytic performance for gaseous oxygen evolution under simulated solar light irradiation. To deeply understand the charge transportation, the photoelectrochemical properties of the heterojunction system were systematically investigated. The results demonstrate that the *n-p* heterojunction system based on *n*-Bi<sub>2</sub>O<sub>3</sub> and *p*-MoS<sub>2</sub> were successfully prepared for the first time. It is evidently observed that owing to the introduction of highly conductive layered MoS<sub>2</sub>, the formation of Bi<sub>2</sub>S<sub>3</sub> and *n-p* heterojunction is the key role in the enhancement of photocatalytic performance, which could provide deep insight into design and application of excellent photocatalysts in the future.

## 2. Experimental section

### 2.1. Materials

Bismuth nitrate pentahydrate (98.0%), sodium hydroxide (99.8%), sodium molybdate dehydrate (99.5%), thioacetamide (99.0%), 5,5-dimethyl-1-pyrroline (DMPO, >99.0%) were purchased from Sigma-Aldrich. All the chemicals were used as received without any further purification.

### 2.2. Synthesis of Bi<sub>2</sub>O<sub>3</sub> microplates

In a typical synthesis of Bi<sub>2</sub>O<sub>3</sub> microplates, 4.8 g of Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O was dissolved into 100 mL of deionized water, stirring for 30 min. Then the pH value of the solution was adjusted to 8.0 by using NaOH (10 M) and sonicated for 60 min. The mixed solution was stirred overnight and then precipitated. The precipitate was washed three times using absolute ethanol, purified water, and dried at 60 °C for 12 h. Finally, the light yellow precipitate was calcined at 400 °C for 2 h in air to obtain Bi<sub>2</sub>O<sub>3</sub> microplates.

### 2.3. Synthesis of layered MoS<sub>2</sub>

0.108 g of sodium molybdate dehydrate and 0.216 g of thioacetamide were dissolved in 36 mL of purified water, respectively. Then they were mixed, stirred for 30 min, and transferred to a 100 mL Teflon-lined autoclave. The mixture was placed at 200 °C for 24 h. A black precipitate was obtained by centrifuging, and washed using absolute ethanol and purified water. Then the precipitate was dispersed into deionized water and sonicated. The solution was transferred to a centrifugation tube and the centrifuge was set at 2000 rpm for removal of the MoS<sub>2</sub> aggregation at the bottom of the tube. Finally, the nanosized MoS<sub>2</sub> was collected by filtration using 0.22 μm filter paper, and then dried at 60 °C for 4 h.

### 2.4. Synthesis of Bi<sub>2</sub>S<sub>3</sub>

The pure Bi<sub>2</sub>S<sub>3</sub> was prepared by using excess sulfur source. In a typical procedure, 0.2 g of Bi<sub>2</sub>O<sub>3</sub> microplates and 0.4 g of thioacetamide were dissolved in 36 mL of purified water and stirred for 1 h. The solution was transferred to a 45 mL Teflon-lined autoclave and maintained at 200 °C for 24 h. The resulting sample was col-

lected by centrifugation, washed by deionized water, and dried in air at 60 °C for 12 h.

### 2.5. Synthesis of Bi<sub>2</sub>O<sub>3</sub>/Bi<sub>2</sub>S<sub>3</sub>/MoS<sub>2</sub> heterojunction

0.2 g of Bi<sub>2</sub>O<sub>3</sub> microplates and 0.02 g of MoS<sub>2</sub> were added into 36 mL of absolute ethanol and stirred for 30 min. And then the mixed solution was sonicated for 30 min and transferred into a 45 mL Teflon-lined autoclave which was maintained at 140 °C for 6 h. The resulting sample was collected by centrifugation, washed by deionized water, and dried in air at 60 °C for 12 h.

### 2.6. Synthesis of Bi<sub>2</sub>S<sub>3</sub>/MoS<sub>2</sub> heterojunction

0.2 g of Bi<sub>2</sub>O<sub>3</sub> microplates, 0.108 g of sodium molybdate dehydrate and 0.216 g of thioacetamide were dissolved in 36 mL of purified water and stirred for 1 h. The solution was transferred to a 45 mL Teflon-lined autoclave and maintained at 200 °C for 24 h. The resulting sample was collected by centrifugation, washed by deionized water, and dried in air at 60 °C for 12 h.

### 2.7. Characterizations

Powder X-ray diffraction (XRD) patterns of samples were determined on a Bruker D8 X-ray diffractometer (Bruker-AXS, Karlsruhe, Germany) with filtered Cu K $\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ) at accelerating voltage and current of 40 kV and 40 mA, respectively. The structure and morphology of samples were observed on a field emission scanning electron microscope (FESEM, ZEISS NEON 40EsB) and transmission electron microscope (JEOL 2100). Fourier transform infrared spectra (FTIR) were recorded on a Bruker instrument with an ATR correction mode. Raman analysis was performed on an ISA dispersive Raman spectroscopy using argon ion laser with a wavelength at 514 nm. To investigate the light absorption and emission behavior, UV–vis absorption spectra were determined in diffuse reflection mode using an integrating sphere (Cary 4000). X-ray photoelectron spectroscopy (XPS) was used to determine the chemical states of elements using a Thermo Escalab 250 with Al-K $\alpha$  X-ray. EPR measurements of the free radicals was recorded by using 5,5-dimethyl-1-pyrroline (DMPO, >99.0%) as a probe on a Bruker EMS-plus instrument, operated under the following conditions: center field, 3520 G; sweep width, 100 G; microwave frequency, 9.87 GHz; power setting, 18.75 mW; scan number, 3.

### 2.8. Photoelectrochemical measurement

Electrochemical tests were carried out using a Zahner Zennium electrochemical workstation operated in a standard three-electrode configuration with a FTO electrode deposited with the samples as a photoanode, a Pt wire as counter electrode, and Hg/Hg<sub>2</sub>Cl<sub>2</sub>/saturated KCl (SCE) as the reference electrode. In a typical experiment, 0.05 M Na<sub>2</sub>SO<sub>4</sub> (20 mL, pH = 6.8) purged with N<sub>2</sub> was used as the electrolyte. For the fabrication of the photoanode, 40 mg of the solid sample was homogeneously mixed with 2 mL of absolute ethanol and 40 μL of Nafion solution (5%) by using a vortex oscillator. The obtained sample was deposited onto the as-washed FTO glass with a controlled area of 1 cm<sup>2</sup> using dip-coating to form a film electrode, and then dried in air. A 300 W Xenon lamp (Philip) coupled with an AM 1.5 G filter was applied as the simulated sunlight source ( $I_0 = 100 \text{ mW cm}^{-2}$ ). The electrochemical impedance spectroscopy (EIS) was carried out in the frequency range of 10<sup>-1</sup>–10<sup>5</sup> Hz with an AC voltage amplitude of 10 mV at a DC bias of 0.298 V vs. SCE in a 0.05 M Na<sub>2</sub>SO<sub>4</sub> solution.

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