



# Enhanced photocatalytic activity and synthesis of ZnO nanorods/MoS<sub>2</sub> composites

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## ABSTRACT

A stable and recyclable organic degradation catalyst based on MoS<sub>2</sub> functionalized ZnO nanorods was introduced. ZnO nanorods were synthesized on the glass substrates (2 cm\*2 cm) by sol-gel method and hydrothermal method and functionalized with MoS<sub>2</sub> via an argon flow annealing method. The structure and morphology of the as-prepared samples were characterized by XRD, SEM and TEM. Results showed that a small amount of MoS<sub>2</sub> was successfully wrapped on the surfaces of ZnO nanorods. XPS analyses showed the existence of Zn-S between ZnO and MoS<sub>2</sub>, indicating that the MoS<sub>2</sub> was combined with ZnO through chemical bonds and formed the ZnO/MoS<sub>2</sub> heterostructure. PL results revealed that ZnO/MoS<sub>2</sub> had lower fluorescence spectra indicating an electron transport channel between ZnO and MoS<sub>2</sub> which separated electrons and holes. Photocatalytic experiment showed that ZnO/MoS<sub>2</sub> composites showed a better photodegradation performance of Rhodamine B (RhB) after functionalized with MoS<sub>2</sub> under the UV light irradiation which could be attributed to the separation and transfer of photogenerated electrons and holes between ZnO and MoS<sub>2</sub>. Meanwhile, the high active adsorption sites on the edges of MoS<sub>2</sub> also accelerated the degradation process. Furthermore, the scavengers were used to investigate the major active species and results indicated that h<sup>+</sup> was the major reactive species for the degradation.

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## 1. Introduction

Water pollution has become the most serious problem due to the increasing number of industrialization around the world. The organic dye and other chemical solutions discharged into the river without any purification have caused immense damage to ecosystem, especially to human [1,2]. Thus, it's necessary to take some actions to degrade the organic dye and toxic substance. Photocatalysis is one of the effective methods to remove the organic dye from waste water [3–5]. Semiconductors (such as Ag<sub>3</sub>PO<sub>4</sub> [6], ZnO [7], TiO<sub>2</sub> [8], CdS [9], MoS<sub>2</sub> [10], SnO<sub>2</sub> [11]) have been studied a lot due to their good photoelectric properties. Among these semiconductors, zinc oxide (ZnO) has attracted much attention because of its low-cost and non-toxic [12]. Under UV irradiation, the electrons and holes generated by ZnO can efficiently degrade organic dye in water. However, there are also some shortcomings when using ZnO as photocatalyst, such as low separation rate of electron-hole pairs and poor photostability. Constructing hybrids and hetero-structures with noble metals, narrow-band semiconductors and other

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two-dimensional materials have been found to be efficient strategies to enhance the mobility of electrons and holes and suppress the recombination of electrons and holes [7,13,14]. Meanwhile, the photocatalytic performance of the catalyst can be changed by controlling the morphology of the catalyst. One-dimensional nanomaterials have been studied widely due to their large surface area and excellent photocatalytic effect [15–17].

As a typical 2D layered material, MoS<sub>2</sub>, composed of two layers of sulfur atoms sandwiching a layer of Mo atoms, has attracted a lot of attention due to its advantages in hierarchy morphology, electronic structure [18], active sites [19], and strong surface adsorption ability [20]. Generally, the MoS<sub>2</sub> sheets are combine together by van der Waals interactions to form MoS<sub>2</sub> bulk, which can be exfoliated into single- and few-layer nanosheets under ultrasonic effect in water or ethanol solutions [21]. Actually, MoS<sub>2</sub>-based composites have been widely researched and a lot of efficient photocatalysts have been found, such as TiO<sub>2</sub>/MoS<sub>2</sub> [21], Ag<sub>3</sub>PO<sub>4</sub>/MoS<sub>2</sub> [22] and CdS/MoS<sub>2</sub> [23]. Tian et al. also reported the ZnO and MoS<sub>2</sub> nanoflowers, which showed a good performance in photodegradation of methyl blue [7]. However, most MoS<sub>2</sub> based composite photocatalysts are powders which are difficult to recycle from aqueous solution and might lead to secondary pollution. Using glasses as the substrates to synthesis nanomaterial is a normal way to prepare recyclable photocatalyst which is environmentally friendly catalyst. Xu et al. reported FTO-based ZnO-Au@CdS catalyst, which was readily recycled and stable [24]. Although lots of researches about ZnO/MoS<sub>2</sub> catalysts reported the enhanced photocatalytic ability, their stability and recyclability had not got enough attention. Moreover, what a kind of role the MoS<sub>2</sub> sheets played in the ZnO/MoS<sub>2</sub> composites remains still unclear. The remarkable adsorption capacity of MoS<sub>2</sub> to organic dye was not separately discussed in many works which might influence the evaluation of photodegradation.

In this work, ZnO/MoS<sub>2</sub> composites based on glass substrate which can be easily recycled from solution have been synthesized by annealing under argon flow with ZnO nanorods and MoS<sub>2</sub> made by hydrothermal method. The adsorption capacity of MoS<sub>2</sub> nanoflowers was first tested by evaluating the concentration of RhB solution. Excluding the adsorption capacity of MoS<sub>2</sub>, the photocatalytic activities of the samples were tested by degrading RhB in water solution under UV light irradiation. By separating the adsorption and degradation process, we investigated the main effects of MoS<sub>2</sub> in the ZnO/MoS<sub>2</sub> composite. Moreover, the scavengers were used to investigate the major active species during the photocatalytic reaction for further understanding the photocatalytic mechanism.

## 2. Experimental

**Synthesis of MoS<sub>2</sub>:** 0.54 g NaMoO<sub>4</sub> · 2H<sub>2</sub>O and 0.36 g C<sub>2</sub>H<sub>5</sub>NS were dissolved in 40 ml deionized (DI) water. After stirring for about 30 min, the solution was poured into a 100 ml Teflon-lined stainless steel autoclave and then heated at 200 °C for 24 h. The obtained solid was washed with DI water and ethanol for several times, and dried at 70 °C for 12 h.

**Synthesis of ZnO nanorods:** The synthesis of ZnO nanorods could be divided into two steps: the syntheses of Al-doped ZnO film on glass substrates by sol-gel method and ZnO nanorods on the Al-doped ZnO film by hydrothermal method. As reported in our previous works [25,26], Zinc acetate sol was spin-coated on the glass substrates and dried in drying oven at 100 °C for 10 min followed by preheated in air at 500 °C for another 15 min. This process was repeated six times and then the obtained samples were annealed in air at 550 °C for 2 h. The synthesized Al-doped ZnO films were dipped into a 100 ml Teflon-lined stainless steel autoclave which contained 50 ml aqueous growth solution with 1 mmol Zinc nitrate hexahydrate [Zn(NO<sub>3</sub>)<sub>2</sub> · 6H<sub>2</sub>O] and 1 mmol hexamethylenetetramine (HMTA, C<sub>6</sub>H<sub>12</sub>N<sub>4</sub>). Then the stainless steel autoclave was heated in an electric oven at 90 °C for 3 h before the samples were washed by deionized (DI) water several times and dried at 90 °C for 6 h.

**Synthesis of ZnO/MoS<sub>2</sub> compounds:** 1 mg MoS<sub>2</sub> was dispersed in 100 ml ethanol and kept in ultrasonic for 2 h. Then one drop of the MoS<sub>2</sub> solution was dripped on the as-prepared ZnO nanorods followed by dried at 90 °C for 10 min before they were transferred in tube furnace and heated under the argon flow at 550 °C for 2 h. The ZnO nanorods without MoS<sub>2</sub> were annealed at the same condition.

**Photocatalytic:** The photocatalytic performance of the samples was carried out at room temperature by degrading Rhodamine B (RhB) under a mercury lamp (500w) irradiation. Typically, a piece of sample was suspended in the middle of test tube which contained 50 ml (4 mg/L) RhB solutions. The samples were kept in dark under stirring for 30 min to reach an adsorption-desorption equilibrium before the light was turned on. After irradiation for a certain interval, 3 ml reaction solution was taken out and centrifuged to remove the catalysts. Finally, the concentration of RhB in solution was analyzed by an UV-vis spectrophotometer. The efficiency of these catalysts were quantitatively evaluated by a pseudo-first order reaction equation:  $\ln(C/C_0) = -kt$ , where  $C_0$  was the initial concentration of RhB before irradiation, and  $C$  was the corresponding concentration in real time.

**Sample characterization:** X-ray diffraction (XRD, PANalytical XNalytical) with Cu-K $\alpha$  radiation ( $\lambda = 0.154$  nm) was used to characterize the different samples. The morphology and structure of the samples were examined by field emission scanning electron microscope (SEM, JSM-7000F) and transmission electron microscope (TEM, Tecnai F30G2). X-ray photoelectron spectroscopy (XPS, Kratos AXIS Ultra DLD) was used to analyze surface chemical states of these composites. Photoluminescence (PL) properties were measured by Gangdong F-320 photoluminescence spectrophotometer at an excitation wavelength of 325 nm. All these surveys were operated at room temperature.

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