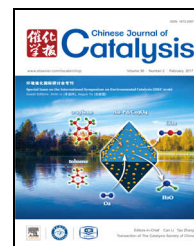


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# Surface plasmon resonance-induced visible-light photocatalytic performance of silver/silver molybdate composites

Xianglong Yang, Ying Wang, Xiao Xu, Yang Qu, Xing Ding<sup>#</sup>, Hao Chen<sup>\*</sup>

College of Science, Huazhong Agricultural University, Wuhan 430070, Hubei, China

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## ABSTRACT

Novel silver/silver molybdate ( $\text{Ag}/\text{Ag}_2\text{MoO}_4$ ) composites with surface plasmon resonance (SPR)-enhanced photocatalytic performance were successfully fabricated via a facile one-pot hydrothermal route with the presence of sodium dodecyl sulfate (SDS) in this study. The as prepared silver/silver molybdate ( $\text{Ag}/\text{Ag}_2\text{MoO}_4$ ) composites were systematically characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and ultraviolet-visible diffuse reflectance absorption spectroscopy (DRS) in order to investigate their crystal structure, morphology and optical property as well. The photocatalytic activities of the composites were subsequently evaluated by their ability to degrade rhodamine B (RhB) under visible-light irradiation. Various of controlled experiments were then carefully operated to gain a deep insight into the assembling of  $\text{Ag}/\text{Ag}_2\text{MoO}_4$  composites. It was found that preparation conditions such as pH, reaction time, and the amount of surfactant played important roles in the formation of composites with octahedral microstructures. And the composite obtained at 160 °C using 0.5 g of sodium dodecyl sulfate exhibited the highest photocatalytic performance under visible-light irradiation. Capture experiments were also conducted to clarify the function of different active species generated on the surface of  $\text{Ag}/\text{Ag}_2\text{MoO}_4$  during the photocatalytic process, in which both holes and  $\bullet\text{OH}$  radicals were found to play crucial role in photocatalytic removal of RhB under visible light irradiation. A possible photocatalytic mechanism of  $\text{Ag}/\text{Ag}_2\text{MoO}_4$  was finally proposed on the basis of all the results to explain the higher photocatalytic activity of the octahedral  $\text{Ag}/\text{Ag}_2\text{MoO}_4$  composites. It was inferred that the photoinduced "hot" electrons can quickly transfer from the Ag NPs to the conduction band of  $\text{Ag}_2\text{MoO}_4$  and react with oxygen and  $\text{H}_2\text{O}$  to generate a large quantity of active radicals such as  $\bullet\text{OH}$  and  $\bullet\text{O}_2^-$  because of the SPR effects. Besides, this SPR effects of Ag nanoparticles deposited on the surface of  $\text{Ag}_2\text{MoO}_4$  can not only dramatically amplify its light absorption, especially in the visible region, but also promote the separation of photoexcited electron-hole pairs and effectively decrease electron-hole recombination.

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## 1. Introduction

In the past decades, semiconductor photocatalysis has at-

tracted worldwide interest because of its potential applications in the fields of environment and energy [1–6].  $\text{TiO}_2$ , as the most popular photocatalyst has been extensively studied because of

<sup>\*</sup> Corresponding author. Tel/Fax: +86-27-87288246; E-mail: [hchenhao@mail.hzau.edu.cn](mailto:hchenhao@mail.hzau.edu.cn)

<sup>#</sup> Corresponding author. Tel/Fax: +86-27-87288246; E-mail: [dingx@mail.hzau.edu.cn](mailto:dingx@mail.hzau.edu.cn)

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its abundance, stability and nontoxicity [7,8]. However,  $\text{TiO}_2$  is only responsive to ultraviolet (UV) light ( $\lambda < 400$  nm), which is only about 4% of the spectrum of sunlight, leading to low photoconversion efficiency [9]. Therefore, many researchers have attempted to improve the photocatalytic efficiency of  $\text{TiO}_2$  under visible light through modifications such as doping with transition metals and nonmetal atoms, as well as developing other visible light-responsive semiconductor photocatalysts [10–12]. Various visible-light photocatalysts including doped classical photocatalysts, dye-sensitized photocatalysts, heterojunction photocatalysts and other composite semiconductor photocatalysts have been explored through different channels [13–17].

Among the original visible-light photocatalysts, those with deposited noble metals have received much attention recently, because the localized surface plasmon resonance (SPR) effect of the noble metals can both dramatically amplify the visible-light absorption of the semiconductor and markedly improve the rate of electron-hole separation at the metal/semiconductor interface [18–20]. For instance, Huang et al. [21,22] reported a series of silver (Ag)-silver halide ( $\text{AgX}$ ) plasmonic photocatalysts synthesized through photoreduction of  $\text{AgX}$  that showed improved photocatalytic activity in the degradation of methyl orange (MO) compared with that of  $\text{AgX}$  alone. Ye et al. [23] also found that necklace-like  $\text{Ag}/\text{Ag}_3\text{PO}_4$  heterostructures fabricated through a hetero-growth process, where uniform, ordered  $\text{Ag}_3\text{PO}_4$  submicrocubes formed on the Ag nanowires, displayed high MO degradation efficiency. Recently, Shen and colleagues [24] demonstrated that  $\text{Ag}_2\text{Mo}_2\text{O}_7@/\text{AgBr}-\text{Ag}$  composites allowed effective photocatalytic removal of both methylene blue and rhodamine B (RhB).

Molybdates, which play important roles in various technological applications, are promising photocatalysts because of their unique structural, physical, and chemical properties [25,26]. As a vital member of the molybdate family, silver molybdate ( $\text{Ag}_2\text{MoO}_4$ ) has been widely used in photoluminescence and surface-enhanced Raman scattering (because of its suitable physical and chemical properties [27,28]). To date, multifarious nano- and microstructures of  $\text{Ag}_2\text{MoO}_4$  have been fabricated, including nanoparticles (NPs), wire-like nanostructures, and even flower-like microstructures [29,30]. However, the photocatalytic activity of  $\text{Ag}_2\text{MoO}_4$  has rarely been observed, except for that of a cube-like  $\text{Ag}/\text{Ag}_2\text{MoO}_4$  composite recently prepared via a harsh microwave-assisted hydrothermal process in the presence of polyvinylpyrrolidone [31]. The function of different active species and the corresponding photocatalytic mechanism of  $\text{Ag}/\text{Ag}_2\text{MoO}_4$  composites have never been deeply investigated. Therefore, it is of great significance to develop a new route to fabricate  $\text{Ag}/\text{Ag}_2\text{MoO}_4$  composites under milder reaction conditions and examine their photocatalytic mechanism.

In this study, novel octahedral  $\text{Ag}/\text{Ag}_2\text{MoO}_4$  composites with SPR-enhanced photocatalytic performance are fabricated by a facile one-pot hydrothermal route in the presence of sodium dodecyl sulfate (SDS). The crystal structure, morphology and optical properties of the as-prepared samples are systematically investigated by a series of characterization methods in-

cluding X-ray diffraction (XRD), scanning electron microscopy (SEM), and UV-visible diffuse reflectance absorption spectroscopy (DRS). The photocatalytic activities of the as-prepared composites are subsequently evaluated by the degradation of RhB under visible-light irradiation. Various control experiments are performed to gain a deep understanding of the assembly of  $\text{Ag}/\text{Ag}_2\text{MoO}_4$  composites. The roles of reaction conditions such as pH, reaction time, and the amount of surfactant added in the formation of octahedral microstructure are studied in detail. In addition, the photocatalytic mechanism of the  $\text{Ag}/\text{Ag}_2\text{MoO}_4$  composites is also systematically investigated. This study provides a new strategy to achieve enhanced photocatalytic performance for other types of photocatalysts besides  $\text{TiO}_2$ .

## 2. Experimental

### 2.1. Preparation

All reagents were analytical grade, and used as raw materials without any further purification. Deionized water was used throughout the syntheses. The  $\text{Ag}/\text{Ag}_2\text{MoO}_4$  composites were synthesized via a hydrothermal process. In a typical synthesis,  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$  (0.286 mmol) and SDS (0, 0.2, 0.5, or 0.6 g) were first dissolved in deionized water (20 mL). Then  $\text{AgNO}_3$  solution (0.133 mol  $\text{L}^{-1}$ , 15 mL) was added dropwise to the above solution under constant vigorous magnetic stirring for 30 min. The pH of the mixture was adjusted to the desired value (2, 3, 4, 5, 6, 7, or 8) by addition of  $\text{NH}_3\cdot\text{H}_2\text{O}$  or  $\text{HNO}_3$  solution. The resulting mixture was then transferred into a 50-mL stainless steel autoclave with a Teflon liner. After treating the mixture at 180 °C for a time of 2, 4, 8, 14, or 24 h, the resulting product was cooled to room temperature. The precipitate was collected by centrifugation, washed with deionized water and absolute ethanol several times, and then dried at 60 °C overnight.

### 2.2. Characterization

The purity and crystallinity of the as-prepared composites were characterized by XRD using a diffractometer (Bruker D8 Advanced, USA) with  $\text{Cu } K_\alpha$  over the  $2\theta$  range from 5° to 80°. The morphologies and microstructures of the samples were observed by SEM (JSM-6390LV, Japan). DRS data for the samples were acquired using a Cary 5000 UV-vis-NIR spectrophotometer equipped with an integrating sphere using  $\text{BaSO}_4$  as the reflectance standard.

### 2.3. Photocatalytic activity evaluation

RhB was chosen as the target organic compound in this study to estimate the photocatalytic activity of the photocatalysts. During a typical test of RhB degradation, photocatalyst (20 mg) was dispersed in RhB solution (50 mL) with an initial concentration of 20 mmol  $\text{L}^{-1}$  in a photoreactor via vigorous stirring. Before exposure to light, the suspension was stirred in the dark for 30 min to establish an adsorption-desorption equi-

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