



Enhanced photocatalytic activity of Bi₂WO₆ loaded with Ag nanoparticles under visible light irradiation

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ARTICLE INFO

Article history:

Received 27 May 2009

Received in revised form 9 July 2009

Accepted 20 July 2009

Available online 28 July 2009

Keywords:

Silver

Bi₂WO₆

Photocatalytic activity

Bacterium

Inactivation

ABSTRACT

Ag-loaded Bi₂WO₆ nanoparticles were prepared via a facile alcohol-thermal process in ethylene alcohol system. The products were characterized by XRD, TEM, HRTEM and energy-dispersed X-ray (EDX) microanalysis. The results revealed that it was the metallic Ag deposited on Bi₂WO₆, which was further confirmed by the surface plasmon absorption band existed in the UV–vis absorption spectrum. Compared with pure Bi₂WO₆, Ag-loaded Bi₂WO₆ photocatalysts exhibited significantly enhanced photocatalytic activity in inactivating *E. coli*, a Gram-negative bacterium, and *S. epidermidis*, a Gram-positive bacterium under visible light irradiation ($\lambda > 420$ nm). The influence of the content of silver on the catalytic activity of Bi₂WO₆ photocatalyst has been investigated. The results concerning the effect of silver on the catalytic activity of Bi₂WO₆ photocatalyst revealed that the significant enhancement of photocatalytic activity can be attributed to the synergetic effects between noble metal and semiconductor component.

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1. Introduction

Since photoinduced decomposition of water on TiO₂ electrodes was discovered, semiconductor-based photocatalysis has attracted extensive interest [1]. Photocatalysis is one of the most promising technologies for water purification characterized by the generation of •OH radicals and other oxidative radicals. The general process for the photocatalytic destruction of organic substances can be divided into three steps: (1) the electrons in the valence band are excited to the conduction band leaving holes in the valence band simultaneously, when the energy of the incident light is equal or higher than the band-gap of the semiconductor; (2) the photogenerated electrons and holes transfer separately to the surface of the semiconductor to be captured by the oxygen and H₂O absorbed, forming the oxidative species •OH, O₂^{•-}, etc.; (3) the oxidative species attack the objects, resulting in the degradation of organic compounds and the inactivation of microorganisms. In the second step, the photogenerated electrons and holes may recombine before they arrived at the surface of the semiconductor, leading to the decrease of the photocatalytic activity. Therefore, the competition between the transfer and the recombination process of photogenerated electron–hole pairs is an important

factor, which limits the photocatalytic activity of photocatalysts. In recent years, many studies have been carried out to overcome the limitation, including the coupling two kinds of matching compounds [2–5], and the deposition of noble metals such as silver [6,7], gold [8,9], platinum [10,11], and palladium [12,13].

Recently, semiconductor–metal composites were designed to improve the catalytic ability inspired by the synergetic action between semiconductor and metal component. One kind of synergetic action brought by combining noble metal nanoparticles with semiconductor oxides works via facilitating the charge separation and improving the photocatalytic activity. The noble metal nanoparticles deposited on the semiconductor oxides could act as a sink for electrons and promote interfacial electron transfer process due to its high Schottky barriers at the metal–semiconductor interface [14,15]. There have been reports that noble metals such as Pt, Au or Ag deposited on semiconductor TiO₂ were achieved so as to enhance the photocatalytic activity [16–18]. Photoelectrochemical studies operated on the Au–TiO₂ composite system also exhibited enhanced photocurrent generation in virtue of the improved interfacial charge transfer [19]. Therefore, it is believed that the metal nanoparticle deposition facilitates separation of charge carriers and promotes interfacial electron transfer process. Surface plasmon resonances of noble metal particles, which can be induced by light irradiation, may also contribute to the enhanced photocatalytic activity. A plasmonic photocatalyst was proposed by Awazu et al. [20]. It was suggested that the photocatalytic behavior

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of TiO₂ was greatly boosted by the enhanced near-field amplitudes of localized surface plasmon, which arisen from an Ag core covered with a silica shell.

Bi₂WO₆ has been found to possess excellent intrinsic physical and chemical properties, such as ferroelectric piezoelectricity, catalytic behavior, and non-linear dielectric susceptibility [21]. Thus, many efforts have been devoted to study the properties of Bi₂WO₆, including the catalytic property [22–25]. Previously, our group has reported the preparation, photocatalytic activity, and stability of Bi₂WO₆ in the degradation of dyes [26–28]. The results exhibited high photocatalytic activity of Bi₂WO₆ in the degradation of dye. Inspired by the disinfection by TiO₂ photocatalysts, hereby, we expected that Bi₂WO₆ may also can inactivate microorganisms via photocatalytic process thus extend the researches on Bi₂WO₆ further. Considering the synergetic effect between the noble metal and semiconductor components, for the first time Ag nanoparticles were deposited on Bi₂WO₆ nanoparticles, to further improve the photocatalytic activity of photocatalyst Bi₂WO₆. And the results demonstrated that Ag-loaded Bi₂WO₆ exhibited significantly enhanced photocatalytic activity compared with pure Bi₂WO₆ in inactivation of both *E. coli*, a Gram-negative bacterium and *S. epidermidis*, a Gram-positive bacterium.

2. Experimental

2.1. Preparation of photocatalysts

All the reagents were of analytical grade and were used without any further purification. Ag-loaded Bi₂WO₆ was synthesized by a facile alcohol-thermal process in ethylene alcohol system. In a typical procedure, 0.97 g Bi(NO₃)₃·5H₂O and 0.33 g Na₂WO₄·2H₂O were dissolved in 20 mL ethylene alcohol, respectively. A transparent solution was obtained when the two solutions were mixed together under magnetic stirring at room temperature. Then, certain amount of AgNO₃ (0.1 or 1.0 mmol) was added to the above solution, and the solution was still transparent. The transparent solution was added into a 50 mL Teflon-lined autoclave up to 80% of the total volume. Then the autoclave was sealed in a stainless steel tank and heated at 160 °C for 24 h. Subsequently, the reactor was cooled to room temperature naturally. The resulting samples were collected and washed with de-ionized water and dried at 60 °C in air. Ag-loaded samples with different Ag/Bi₂WO₆ molar ratios were prepared and denoted as AB-0.1 and AB-1.0 according to the molar of reagent AgNO₃ used in the preparation. To find the appropriate content of Ag deposition so as to achieve the best photocatalytic activity, other loading contents of Ag–Bi₂WO₆ samples (AB-0.05, AB-0.5, and AB-0.75, denoted according to the mmolar used of reagent AgNO₃ in the preparation) were prepared and the photocatalytic bactericidal activities were evaluated by the inactivation of *E. coli* under visible light irradiation. Pure Bi₂WO₆ sample denoted as AB-0 was also synthesized under the same condition to compare their photocatalytic inactivation activities.

2.2. Characterization

The powder X-ray diffraction (XRD) patterns of the as-synthesized samples were recorded on a D/MAX 2250 V diffractometer (Rigaku, Japan) using monochromatized Cu K α ($\lambda = 0.15418$ nm) radiation under 40 kV and 100 mA and with the 2θ ranging from 10° to 70°. UV–vis diffuse reflectance spectra of the samples were obtained on an UV–vis spectrophotometer (Hitachi U-3010) using BaSO₄ as reference. Transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HRTEM) images were obtained using a JEOL JEM-

2100F field emission electron microscope at acceleration voltage of 200 kV.

2.3. Preparation of the bacterial culture

Two types of bacteria, *E. coli*, a Gram-negative bacterium, and *S. epidermidis*, a Gram-positive bacterium, were used as model bacteria in this study. They were incubated in Lysogeny Broth (LB) medium at 37 °C for 18 h. Cells were harvested from overnight culture by centrifugation at 4000 rpm for 5 min and then washed twice with 0.9% saline.

2.4. Bactericidal activity

The bactericidal activity of the samples was evaluated by the inactivation of *E. coli* and *S. epidermidis* under visible light irradiation ($\lambda > 420$ nm). All materials used in the experiments were autoclaved at 121 °C for 40 min before use to ensure sterility. The treated cells were suspended and diluted to a cell suspension of $\sim 2 \times 10^7$ cfu/mL with 0.9% saline to remove the influence of osmotic pressure on bacterial cells. The final photocatalyst concentration was adjusted to 0.5 mg/mL. A 500 W Xe lamp was used as the light source with a UV cutoff filter ($\lambda > 420$ nm) to provide visible light irradiation. The reaction mixture was stirred with a magnetic stirrer to prevent settling of the photocatalysts. The experiments were carried out at room temperature. Before and after the experiments, an aliquot of the reaction mixture was immediately diluted with 0.9% saline and plated on LB-agar plates. The colonies were counted after incubation at 37 °C for 24 h. All of the above experiments were repeated three times and the average values were given.

3. Results and discussion

3.1. XRD analysis

The powder X-ray diffraction (XRD) patterns of the as-synthesized Ag-loaded Bi₂WO₆ samples were recorded. As shown in Fig. 1, all the diffraction peaks can be categorized into two sets. The unmarked peaks can be indexed as an orthorhombic Bi₂WO₆ corresponding to JCPDS files no. 39-0256 ($a = 5.457$ Å, $b = 16.435$ Å, and $c = 5.438$ Å), while the marked agree well with face-centered cubic Ag corresponding to JCPDS files no. 04-0783. No other peaks from possible impurities are detected. Moreover, the changes of all diffractions and lattice parameters of Bi₂WO₆ in all Ag-loaded

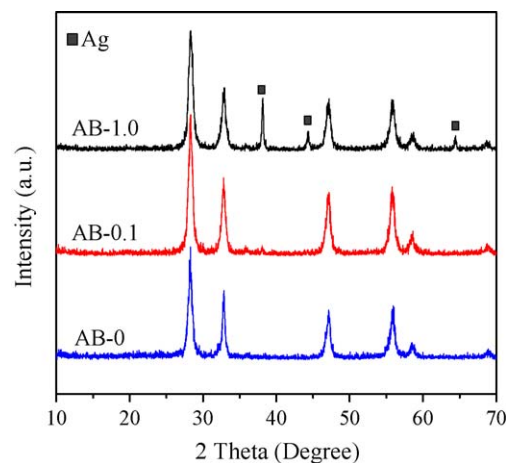


Fig. 1. XRD patterns of the Bi₂WO₆ and Ag-loaded Bi₂WO₆ samples with different silver contents.

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