



Synthesis and characterization of Ag/BiVO₄ composite photocatalyst

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ABSTRACT

Ag/BiVO₄ composite photocatalysts were hydrothermal synthesized and characterized by XRD, SEM, XPS and DRS techniques. Their photocatalytic activities were determined by oxidative decomposition of methyl orange in aqueous solution under visible light irradiation. It revealed that the doped Ag species greatly improved the visible light absorption abilities and morphologies of the composites, and thus lead to enhanced photocatalytic activities compared with that of the pure BiVO₄.

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1. Introduction

In the past few decades, various researches have been focused on exploiting novel and more efficient photocatalysts for degradation of organic contaminants and water splitting [1,2]. At the present time, developing visible-light-driven photocatalyst is the most urgent issue in this topic [3,4]. And among these works, monoclinic BiVO₄ with a band-gap of 2.4 eV has been reported to be an active photocatalyst under visible light irradiation and have nowadays attracted considerable attentions [5–8]. There are three crystalline phases reported for synthetic BiVO₄, the monoclinic sheelite-type, the tetragonal sheelite-type and the tetragonal zircon-type, and their photocatalytic properties are strongly related to the phase structure [9,10].

However, some of the researches indicated that the photo-activity of pure BiVO₄ was observed to be comparatively low due to its difficult in the separation of photogenerated electron–hole pairs. It was reported that modified BiVO₄ with metals or metal oxides doping made it difficult for the recombination of photogenerated hole–electron pair and thus solve this problem [11,12]. So far, variety of metal or metal oxides doped BiVO₄ systems, including Cu/BiVO₄ [12], Co/BiVO₄ [12,13], Pd/BiVO₄ [14,15], Fe/BiVO₄ [12], AgO/BiVO₄ [16–18], CuO/BiVO₄ [19], WO₃/BiVO₄ [20], V₂O₅/BiVO₄ [21], Bi₂O₃/BiVO₄ [22] and others, have been studied. Among these researches, most metals and metal oxides were doped or loaded by ion treatment or impregnation method after the synthesis of BiVO₄; and detailed investigation is

also needed to provide the clear evidence for explaining the enhanced activities in decomposition of organic pollutants and their mechanisms.

Herein, Ag/BiVO₄ series composites were hydrothermal synthesized, and their visible-light-response photocatalytic abilities were detected by the decolorization of methyl orange in aqueous solution. The possible working principle of Ag/BiVO₄ composite was also discussed.

2. Experimental

2.1. Synthesis of Ag doped bismuth vanadate

Bismuth nitrate pentahydrate (Bi(NO₃)₃·5H₂O) and ammonium metavanadate (NH₄VO₃) supplied from Beijing Chemical Company were used directly without any further purification. Other chemicals used were all analytical grade, and solutions were prepared using deionized water. In a typical preparation, 0.01 mol Bi(NO₃)₃·5H₂O and 0.01 mol NH₄VO₃ were dissolved in 20 mL of a 35% (w/w) HNO₃ and 20 mL of a 6 mol/L NaOH solution separately, and each stirred for 30 min at room temperature. After that, these two mixtures were mixed together in a 1:1 molar ratio and stirred for about 30 min to get a stable, salmon pink homogeneous solution. The different amounts of AgNO₃ (0.01, 0.05, 0.1, 0.2 g) were then added into these solutions individually with a continuing stirring for 30 min to get the precursors. Mixture of each precursor were then sealed in a 50 mL Teflon-lined stainless autoclave and heated to 180 °C for 6 h under autogenous pressure. Afterwards, the precipitate was filtered, washed with distilled water three times for each, and dried in vacuum at room temperature for 12 h.

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2.2. Apparatus and measurements

X-ray powder diffraction patterns (XRD, Puxi Co. Ltd., model XD-3) were recorded in the region of $2\theta = 10\text{--}55^\circ$ using Cu K α radiation ($\lambda = 0.15418\text{ nm}$) with a step scan of $2.0^\circ/\text{min}$ at room temperature using a counter diffractometer. The morphologies and microstructures of as-prepared samples were examined with scanning electron microscopy (SEM, FEI Co., model Quanta-600). Optical absorbance spectra of the samples were obtained on a doubled-beam UV–vis spectrophotometer (Puxi Co. Ltd., model TU-1901) equipped with an integrating sphere. UV–vis diffuse reflectance spectra (DRS) of samples were recorded by using BaSO $_4$ as a reference and were converted from reflection to absorbance by Kubelka–Munk method. X-ray photoelectron spectroscopy (XPS) analysis was performed on a VG MKII X-ray photoelectron spectrometer using the Mg K α radiation.

2.3. Photocatalytic activity test

Photocatalytic activities of the samples were determined by the decolorization of methyl orange (MO) under visible light irradiation. A 500 W Xe-illuminator was used as a light source and set about 10 cm apart from the reactor. The 420 nm cutoff filter was placed between the Xe-illuminator and the reactor to completely remove all incoming wavelengths shorter than 420 nm to provide visible light irradiation. Experiments were carried out at ambient temperature as follows: the same amount (0.2 g) of photocatalyst was added into 100 mL of a 10 mg/L MO solution. Before illumination, the solution was stirred for 30 min in darkness in order to reach the adsorption–desorption equilibrium for MO and dissolved oxygen. At different irradiation time intervals, about 5 mL suspensions were collected, and then centrifugalized to remove the photocatalyst particles. The concentrations of the remnant MO were monitored in the way of checking the absorbance of solutions at 464 nm during the photodegradation process.

3. Results and discussion

3.1. Powder formation

Fig. 1 shows XRD diffraction patterns of the pure BiVO $_4$ and Ag/BiVO $_4$ series photocatalysts. It is confirmed that all photocatalysts

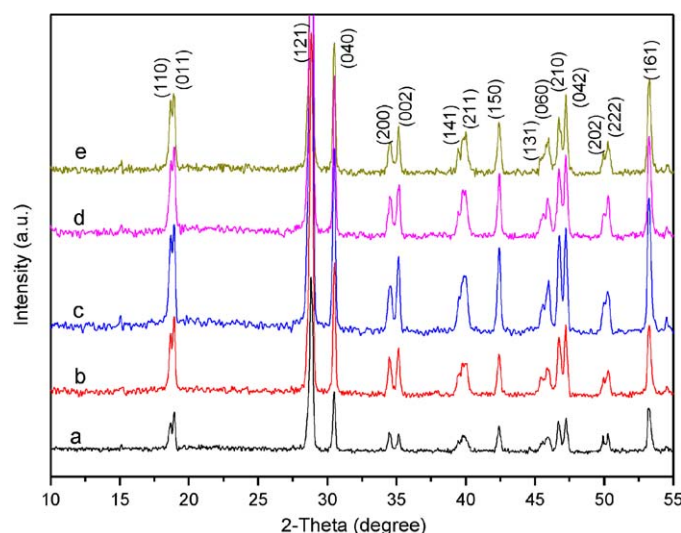


Fig. 1. XRD patterns of (a) pure BiVO $_4$; (b) 0.2 wt% Ag/BiVO $_4$; (c) 1.0 wt% Ag/BiVO $_4$; (d) 2.0 wt% Ag/BiVO $_4$; (e) 4.0 wt% Ag/BiVO $_4$.

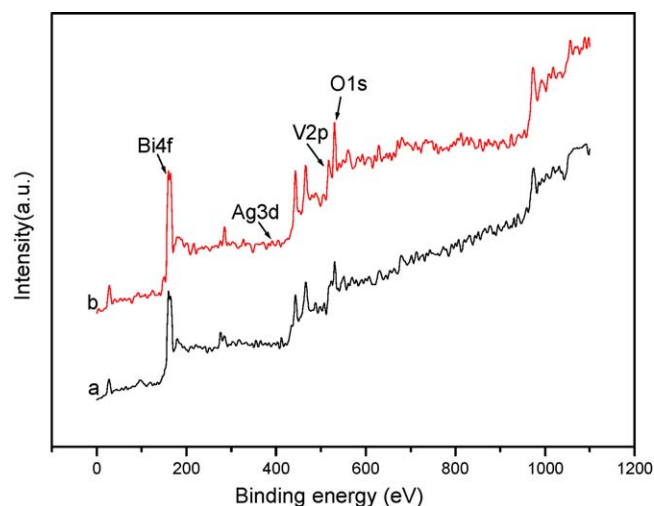


Fig. 2. XPS of (a) pure BiVO $_4$ crystallites and (b) 4.0 wt% Ag/BiVO $_4$ composite photocatalysts.

are the single-monoclinic-scheelite structure, and no peaks of any other phases or impurities can be detected in the composite samples. The diffraction peak of all samples is in conformity to the standard card of monoclinic BiVO $_4$ (JCPDS 14-0688) [5,7], indicating that all the prepared photocatalysts possess the same crystal structure. It is also found that the Ag/BiVO $_4$ composites have the higher diffraction intensities than the pure BiVO $_4$, which indicates that all composite samples display high crystallinity than the pure BiVO $_4$ sample. In addition, the obvious intensity reverses of the (2 0 0) and (0 0 2) peaks at 2θ equals to 34.5° and 35.2° were obtained, which can be attributed to the changes of crystalline orientation by the Ag doping.

The overall XPS spectra of pure BiVO $_4$ and Ag/BiVO $_4$ composite photocatalysts are shown in Fig. 2. It is indicated that the Ag 3d peak around 370 eV is detected for Ag/BiVO $_4$ composite sample. Fig. 3 shows high-resolution XPS spectra of the four primary elements of the sample. As shown in Fig. 3, the binding energies of Bi 4f $_{7/2}$, Bi 4f $_{5/2}$, V 2p $_{3/2}$, V 2p $_{1/2}$ and O 1s are 159, 164, 516, 523 and 528 eV, respectively. The Ag 3d region is displayed in Fig. 3d with the tiny peaks at 368 and 374 eV ascribed to the Ag 3d $_{5/2}$ and Ag 3d $_{3/2}$ respectively [23], and it has a 6 eV splitting of the 3d doublet. In addition, low content of Ag species may be merely placed on the surface of BiVO $_4$ crystal without doped into the crystal lattice according to the XPS and XRD results.

3.2. Morphologies and structures performance

SEM measurements were carried out to clarify the factor as shown in Fig. 4. For the pure monoclinic BiVO $_4$ in Fig. 4a and b, although the primary particles had small sizes, they sintered significantly to each other. In contrast, sintering was disappeared for the Ag/BiVO $_4$ composites, and dispersed particles with planes and edges appeared at the same time. In addition, the particle size appeared decrease with the increase of Ag content from SEM pictures, indicating an increase trend of adsorption ability of powders. In general, the higher the adsorption ability is, the higher the photocatalytic activity will be; and recombination between photogenerated electrons and holes can be suppressed in the edges and planes of photocatalysts [1,5]. So, the composite samples may have high photocatalytic activity than pure BiVO $_4$.

The UV–vis diffuse reflectance spectra of pure BiVO $_4$ and Ag/BiVO $_4$ series samples are depicted in Fig. 5. The absorption edge of pure BiVO $_4$ is about 550 nm, a slightly bigger value than the

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