



# Additive-free hydrothermal synthesis of novel bismuth vanadium oxide dendritic structures as highly efficient visible-light photocatalysts



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## ARTICLE INFO

Available online 14 November 2014

### Keywords:

Photocatalyst  
Bismuth vanadate  
Hydrothermal method  
Visible-light

## ABSTRACT

A novel bismuth vanadium oxide ( $\text{BiVO}_4$ ) dendritic structure was successfully synthesized via an additive-free hydrothermal route. The crystal structures, morphologies and photophysical properties of the as-obtained  $\text{BiVO}_4$  samples were characterized. The photocatalytic activities of the as-synthesized  $\text{BiVO}_4$  samples were evaluated by the degradation of rhodamine B (RhB) under visible-light irradiation.  $\text{BiVO}_4$  sample synthesized at 140 °C showed the highest photocatalytic degradation efficiency, up to 99.3%, due to its relatively high surface area and high crystallinity.

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## 1. Introduction

Since the discovery of photoelectrochemical water splitting used  $\text{TiO}_2$  by Fujishima and Honda in 1972 [1], great progress has been made in the research and application [2–5]. However, a major barrier to the practical application of  $\text{TiO}_2$  as photocatalyst is its band gap ( $E_g=3.0\text{--}3.2$  eV), limiting its photoresponse to visible-light which comprised of about 43% of the incoming solar energy [6]. Many researchers have devoted their efforts to find a suitable alternative for  $\text{TiO}_2$ . As one of the non-titania based visible-light-driven semiconductor photocatalysts, bismuth vanadate ( $\text{BiVO}_4$ ) has recently become a focus because of its unique properties such as narrow band gap, nontoxicity, higher stability and higher sunlight utilization [7].

$\text{BiVO}_4$  ( $E_g=2.4$  eV) has been widely investigated since the use of a  $\text{BiVO}_4$  photocatalyst for solar oxidation was first reported by Kudo et al. in 1998 [8]. It is well-known that  $\text{BiVO}_4$  mainly exists in three crystalline phases: tetragonal

zircon, monoclinic scheelite and tetragonal scheelite structure. Among the three crystal structures of  $\text{BiVO}_4$ , the monoclinic scheelite structure exhibited much higher photocatalytic performance than that of the other two crystalline forms under visible-light illumination [9]. Up until now, various methods for the preparation of  $\text{BiVO}_4$ , such as solid-state reaction [10], hydrothermal treatment [11,12], chemical bath deposition [13], organometallic decomposition [14] and sonochemical route [15], have been reported. A variety of morphologies, such as microsphere [16], hollow shell [17], hyperbranch [18], nanofibrous [19], nanosheet [11] and nanoellipsoid [20], have been achieved, most of which have been synthesized in the presence of organic additives or surfactants to obtain the desired morphologies and crystal structures. For example, Kudo et al. [19] synthesized one-dimensional (1D)  $\text{BiVO}_4$  nanofibrous via a hydrothermal process in the presence of cetyltrimethylammonium bromide (CTAB). Zhang et al. [11] reported the fabrication of two-dimensional (2D)  $\text{BiVO}_4$  nanosheet by a hydrothermal process using sodium dodecyl benzene sulfonate (SDBS) as a morphology-directing template. Dai et al. [21] fabricated monoclinic  $\text{BiVO}_4$  single-crystallites with polyhedral, rod-like, tubular, leaf-like and spherical morphologies using the

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triblock copolymer P123-assisted hydrothermal strategy. Xie et al. [22] reported a simple hydrothermal process to fabricate three-dimensional (3D)  $\text{BiVO}_4$  frameworks using a novel Gemini surfactant-monoalkylated disulfonated diphenyl oxide surfactant (C12-MADS) as structure-directing agent. However, this additive or surfactant method not only increased the production cost but also made it more difficult to scale up production. The additive-free synthesis of  $\text{BiVO}_4$  remained a challenge.

Herein, we reported a hydrothermal route without the use of any additive to synthesize  $\text{BiVO}_4$  photocatalyst. The properties of the as-synthesized  $\text{BiVO}_4$  samples were investigated by X-ray diffraction (XRD), field-emission scanning electron microscopy (FE-SEM) and UV-vis techniques. The photodegradation of rhodamine B (RhB) was employed to evaluate the photocatalytic activities of  $\text{BiVO}_4$  samples under visible-light irradiation. It was demonstrated that the as-prepared  $\text{BiVO}_4$  dendritic structures showed excellent photocatalytic performance. The  $\text{BiVO}_4$  sample obtained at 140 °C had the highest photocatalytic activity, and the decolorization rate of RhB reached to 99.3% under visible light irradiation in 210 min.

## 2. Experimental

### 2.1. Catalyst preparation

All chemicals were analytical-grade reagents and used directly without further purification.  $\text{BiVO}_4$  dendritic structures were prepared by an additive-free hydrothermal method modified from our previous literature [23]. In a typical synthesis, solution A was prepared by dissolving 0.2425 g of  $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$  (0.5 mmol) in 30 mL of deionized water at 50 °C. At the same time, solution B was prepared by dissolving 0.0585 g of  $\text{NH}_4\text{VO}_3$  (0.5 mmol) in 10 mL of deionized water at 50 °C. The two solutions were then mixed together. By stirring for 30 min, the mixture (pH  $\approx$  2) was transferred to a Teflon-lined stainless-steel autoclave of 50 mL capacity and maintained at a selected temperature (100, 140 and 180 °C) for 12 h in an electric oven. After the reaction, the autoclave was allowed to cool to room temperature. Finally, the products were centrifuged, washed with deionized water and absolute ethanol three times, and then dried at 70 °C for 10 h in air. For convenience, we denoted the samples prepared under various conditions as  $\text{BiVO}_4$  (X) (X = 100, 140 and 180 °C).

### 2.2. Characterization

The compositions and crystal structures of the products were analyzed by X-ray diffraction (XRD, D8 ADVANCE X-ray diffractometer, Cu K $\alpha$  radiation  $\lambda = 0.15418$  nm) with a scanning rate of  $10^\circ \text{ min}^{-1}$  in the  $2\theta$  from  $10^\circ$  to  $70^\circ$ . The surface morphologies and microstructures were analyzed by field-emission scanning electron microscopy (FE-SEM, JSM-7100F). The absorption spectra of samples were recorded on a UV-vis-NIR Spectrophotometer (UV-1901, Beijing Purkinje General Instrument Co. Ltd., China) with  $\text{BaSO}_4$  used as a reflectance standard in the UV-vis diffuse reflectance experiment. To determine the Brunauer-Emmett-Teller (BET) surface area of the samples, the  $\text{N}_2$  sorption measurements were

performed by using an Autosorb-iQ surface area analyzer (Quantachrome Instruments US).

### 2.3. Photocatalytic degradation of RhB

The photocatalytic reaction was carried out by degrading  $10 \text{ mg L}^{-1}$  RhB at room temperature under simulated solar light irradiation in an XPA-7 photocatalytic reactor (Nanjing Xujiang Machine-electronic Plant, China). Typically, 40 mg of  $\text{BiVO}_4$  was added to the reaction chamber containing 100 mL RhB solution ( $10 \text{ mg L}^{-1}$ ). Before irradiation, the system was stirred magnetically to achieve adsorption-desorption equilibrium in darkness for 30 min. A 500 W Xe lamp was used as a light source and an optical cut-off filter was employed to only permit the illumination of light with wavelength of  $> 400 \text{ nm}$ . A filter with a  $0.45 \mu\text{m}$  membrane was adopted to extract 3 mL of suspension at suitable time intervals and all the collected suspensions were analyzed by a UV-vis-NIR Spectrophotometer. The decolorization rate of RhB was calculated by the following equation:

$$D = \frac{C_0 - C_t}{C_0} \times 100\% = \frac{A_0 - A_t}{A_0} \times 100\% \quad (1)$$

where  $C_0$  and  $A_0$  were the concentration and absorbency of RhB solution before irradiation, respectively, and  $C_t$  and  $A_t$  were the concentration and absorbency of RhB in the solution after it was irradiated for  $t$  min, respectively.

## 3. Results and discussion

The crystal structure of the obtained  $\text{BiVO}_4$  products was confirmed using the X-ray diffraction (XRD) method. Fig. 1 shows XRD patterns of the  $\text{BiVO}_4$  samples synthesized at different hydrothermal temperatures for 12 h. After the hydrothermal reaction at 100 °C for 12 h (Fig. 1a), it is found that monoclinic  $\text{BiVO}_4$  (JCPDS no. 14-0688) forms along with a few tetragonal  $\text{BiVO}_4$  (JCPDS card no. 14-0133), indicating that  $\text{BiVO}_4$  (100) is the mixture of tetragonal and monoclinic  $\text{BiVO}_4$ . When the hydrothermal temperature is 140 °C, the diffraction peaks of the resulting powders can also be indexed to monoclinic  $\text{BiVO}_4$  and tetragonal  $\text{BiVO}_4$ . However, the intensity of diffraction peaks is tremendously increased with a rise in the hydrothermal temperature from

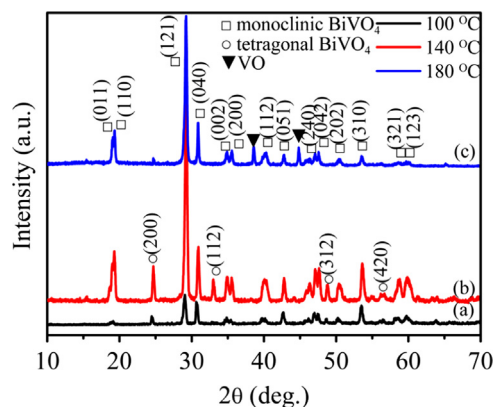


Fig. 1. XRD patterns of  $\text{BiVO}_4$  products derived from different hydrothermal temperatures for 12 h: (a) 100 °C, (b) 140 °C and (c) 180 °C.

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