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Hydrothermal synthesis of phosphate-doped $BiVO_4$ with exposed (010) facets and enhanced sunlight-driven photocatalytic properties

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ABSTRACT

Phosphate-doped monoclinic $BiVO_4$ (mBiVO$_4$) single-crystalline sheets with exposed (010) facets were synthesized by a surfactant-free hydrothermal route for the first time. The results indicate that the appropriate addition of PO_4 oxoanion has significant influence on the crystal growth of mBiVO$_4$ along (010) facets, as well as morphology of the products. The sheet-like mBiVO$_4$ doped with 5% PO_4 oxoanion possesses the highest intensity ratio of $I_{(040)}/I_{(121)}$, and exhibits the best sunlight-driven photocatalytic performance for methylene blue degradation. The exposed (010) facets of the PO_4-doped mBiVO$_4$ which is in favor of increasing the separation of photogenerated electron-hole pairs plays a leading role on the enhanced photocatalytic activity of 5% PO_4-doped mBiVO$_4$.

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1. Introduction

Considering the serious shortage of energy, semiconductor photocatalysis technique provides a perfect way in the aspect of making use of the solar energy, and managing the environment pollution synthetically [1]. Monoclinic scheelite bismuth vanadate (mBiVO$_4$), as a non-titania based and ideal visible-light-driven photocatalyst, has attracted extensive interests because of its nontoxic, low cost and high photocatalytic activity [2,3]. However, the activity of pristine $BiVO_4$ is low due to its poor adsorptive performance and difficult migration of photo-generated electron-hole pairs [4–6]. Impurity doping (mental or non-mental) is an effect way to enhance the photocatalytic activity of $BiVO_4$, such as W [7], Mo [7,8], In [9], Eu [10], B [11], N [12], F [4], C [5], P [6], etc. Recently, selective synthesis of mBiVO$_4$ materials with exposed (010) facets has been confirmed to be effective in enhancing the photocatalytic activity [13–17]. Therefore, the combination of doping control and facet-dependent is expected to result in much better performance. For example, Thalluri et al. [7] used ammonium carbonate as directing agent during hydrothermal process to prepare W- and Mo-doped $BiVO_4$ with preferential growth along the (040) facets which demonstrated an enhancement of the water oxidation rate. Tan et al. [12] found that N-doped $BiVO_4$ single-crystalline nanoplates preferentially grow along the (040) facets exhibited higher photocatalytic activity. Phosphorus also has been used as dopant for mBiVO$_4$ to enhance photoelectrochemical water oxidation activity via the urea-precipitation method [6].

In this work, $BiVO_4$ was doped with different content of phosphate by a surfactant-free hydrothermal route for the first time. The PO_4 oxoanion doping indeed has significant influence on the crystal growth of mBiVO$_4$ along its (010) facets. Moreover, the relationship between exposed (010) facets and sunlight-driven photocatalytic properties for MB degradation was evaluated.

2. Experimental

2.1. Preparation of the phosphate-doped $BiVO_4$ sample

In a typical synthesis, 0.8 mmol $Bi(NO_3)_3 \cdot 5H_2O$ was dissolved in 32 mL nitric acid while a total 0.8 mmol of Na_3VO_4 and Na_3PO_4 with different $PO_4/(VO_4+PO_4)$ molar fraction (0%, 0.5%, 1%, 5% and 10%) was dissolved in 32 ml of distilled water. These two solutions were mixed to form a yellowish suspension. The pH value of the precursor was adjusted to 2 with NaOH. After stirring for 0.5 h, the precursor was poured into a 100 mL Teflon-lined stainless steel autoclave and heated at 180 °C for 48 h. The precipitate was then centrifuged, repeatedly washed and dried 12 h at 80 °C. The PO_4-doped $BiVO_4$ with different $PO_4/(VO_4+PO_4)$ molar fraction

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(0%, 0.5%, 1%, 5% and 10%) are denoted as BVP-0, BVP-0.5, BVP-1, BVP-5 and BVP-10, respectively.

2.2. Characterization

The crystalline phase was analyzed by X-ray diffraction (XRD, Rigaku D/max-3C) using a Cu k_α radiation generated at 45 kV and 35 mA. Surface morphology was observed by scanning electron microscopy (SEM, Hitachi s-570). The semi-quantitative chemical composition was measured by X-ray fluorescence spectrometer (XRF, Rigaku ZSX Primus II). The microstructure was performed on a transmission electron microscope (TEM, JEOL JEM-2100). Ultraviolet-visible absorption spectroscopy was recorded on a UV–vis spectrophotometer (Shimadzu, UV-3600).

2.3. Measurement of photocatalytic activity

The photocatalytic activity was evaluated by the degradation of MB solution under sunlight irradiation. A 300 W Xe lamp was used as the irradiation source. 0.05 g of photocatalyst was dispersed into 50 mL of 10 mg/L MB solution with constant magnetic stirring. Prior to irradiation, the solution was stirred for 0.5 h in dark to reach adsorption-desorption equilibrium. At irradiation time intervals of every 0.5 h, the sample was taken from the reaction suspension and centrifuged to remove the particles. The concentration of MB was measured according to the absorbance at $\lambda = 664$ nm with a Shimadzu 2700 spectrophotometer.

3. Results and discussion

Fig. 1A shows the XRD patterns of pristine $BiVO_4$ and PO_4-doped $BiVO_4$ samples, which can be indexed as pure *m*$BiVO_4$ (JCPDS card no. 14-0688) without any diffraction peaks of impurities, such as $BiPO_4$, Bi_2O_3 or V_2O_5 [6]. In addition, sharp diffraction peaks indicate good crystallinity, and before increasing the PO_4 oxoanion doping amount to 5%, the intensity of (121) and (040) peaks increases, and then declines. Fig. 1B displays the magnified views of (121) and (040) peaks which gradually shifts towards lower angles with increasing PO_4 oxoanion doping amount and the maximum shifting is 0.10° at 5% PO_4-doped sample and then drops back a little at 10% PO_4-doped sample. It is worthy to note that the relative intensity of $I_{(040)}/I_{(121)}$ increases significantly when increases PO_4 oxoanion doping level to 5%, and then drops obviously (see in Table 1), which suggests that appropriate PO_4-doped products have a preferred orientation along the (010) facets.

Table 1
Structural characterization and morphology for different $BiVO_4$ samples.

Samples	Crystal structure	XRD			Particle morphology
		Intensity(cps)		$I_{(040)}/I_{(121)}$	
		(121)	(040)		
BVP-0	Monoclinic	2638	1158	0.44	Stone-like
BVP-0.5	Monoclinic	3291	1931	0.59	Stone-like, strip-like
BVP-1	Monoclinic	3508	2091	0.60	Sheet-like
BVP-5	Monoclinic	4965	3770	0.76	Sheet-like
BVP-10	Monoclinic	3032	1139	0.38	Large bulk

The typical morphology of the as-prepared $BiVO_4$ samples was displayed in Fig. 2. The pristine $BiVO_4$ is composed of stone-like particles (Fig. 2a) with the size of about 2 μm which can be further demonstrated by TEM image (Fig. 2b). With increasing the PO_4 oxoanion doping level (Fig. S1a-b), the morphology of products turns into strip-like (BVP-0.5), and then sheet-like (BVP-1). For 5% PO_4-doped $BiVO_4$ (Fig. 2c and d), the particles mainly present irregular sheet-like morphology which relates to the preferential growth of (010) facets [17,18], but is thinner than that of BVP-1. However, continuous extending PO_4 oxoanion doping amount to 10% (Fig. S1c), the particles tend to form large bulk with nonuniform distribution. Fig. 2e gives the HRTEM image of BVP-5 recorded from Fig. 2d. The fringe spacing of 2.55 Å and 2.60 Å are close to (002) and (200) planes of *m*$BiVO_4$. The corresponding selected area electron diffraction (SAED) pattern (Fig. 2e insert) suggests that the plate is a single crystalline. Both HRTEM and SAED analyses reveal that the growth orientation of PO_4-doped *m*$BiVO_4$ single-crystalline is along its (010) facets, which is in accordance with the XRD analyses.

The real molar fractions of phosphorus to vanadium of the four PO_4-doped $BiVO_4$ samples were determined by XRF. The SQX results in Table S1 indicate that the actual P contents of the samples are much less than those introduced initially during the synthesis, which is similar to previous report [6]. It can be assumed that the PO_4 oxoanion not doped into the $BiVO_4$ has been dissolved in solution and removed during purification process.

The UV–vis absorption spectra of pristine $BiVO_4$ and PO_4-doped $BiVO_4$ samples are compared in Fig. 3A. All samples exhibit strong absorptions both in UV- and visible-light regions, while, the

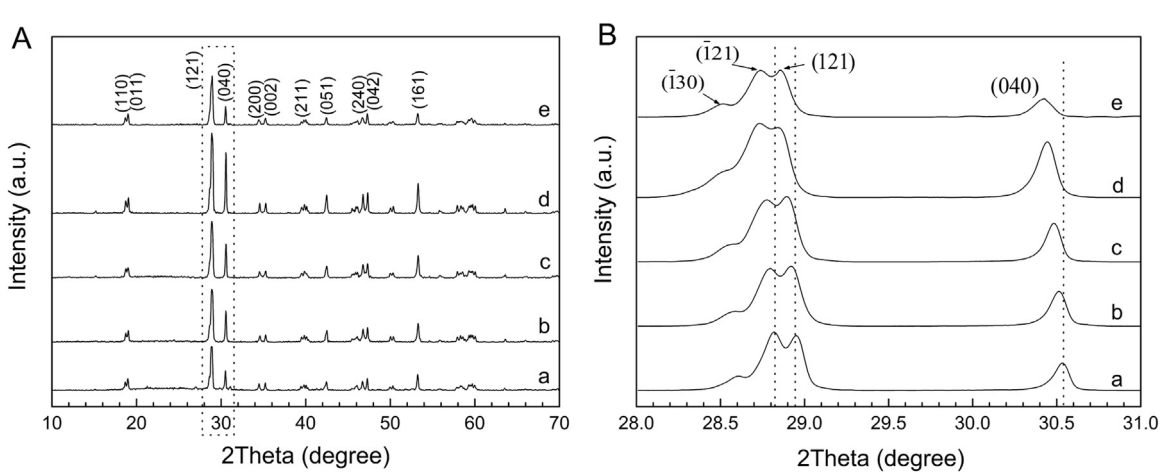

Fig. 1. (A) XRD patterns of pristine $BiVO_4$ and PO_4-doped $BiVO_4$; (B) magnified XRD patterns within 28–31°. (a) BVP-0, (b) BVP-0.5, (c) BVP-1, (d) BVP-5, and (e) BVP-10.

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