



Facile synthesis of flower-like Pd/BiOCl/BiOI composites and photocatalytic properties

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ABSTRACT

In this study, through the BiOI and different quality of PdCl₂ reactions, preparation of Pd/BiOCl/BiOI photocatalyst with diffident content of Pd by ultraviolet light reduction method. The synthesis of BiOI from Bi(NO₃)₃·5H₂O and KI by solvent thermal method. The as-synthesized samples were characterized by X-ray diffraction, scanning electron microscopy, Transmission electron microscopy, ultraviolet-visible diffuse reflectance spectroscopy, photoluminescence and X-ray photoelectron spectroscopy. The Photodegradation performance of the photocatalysts were assessed by photodegradation of rhodamine B illuminating with a 350W Xe lamp. The results showed that 3%Pd/BiOCl/BiOI photocatalyst exhibited good photocatalytic activity.

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1. Introduction

Semiconductor applies in many ways, in which semiconductor as a photocatalyst to degrade organic pollutants has become a hot research in recent years. Many semiconductor materials can be used as photocatalysts, such as TiO₂ [1], metal sulfides [2–4] and BiOX [5]. BiOI is a new kind of photocatalytic material and has a layered structure. The band gap energy of BiOI is 1.8 eV, which has a good response under visible light [6,7]. As the band gap is too narrow, photogenerated electrons and holes easily recombine, reducing the photocatalytic activity [8]. Usually by forming a heterojunction with another semiconductor or depositing the noble metal to prevent recombination of photoinduced electrons and holes and improve the photocatalytic activity. The heterojunction including BiOI/Bi₅O₇I [9], BiOI/BiOCl [10], BiOI/BiPO₄ [11] and BiOI/AgI [12] have been reported, which have an obvious effect in the removing of organic pollution. Yu [13] et al. reported that BiOI loaded Pd has higher efficiency in the photodegradation of acid orange II. Ag deposition on BiOI can significantly improve the efficiency of dye degradation [14]. The efficiency of Ag/AgI/BiOI to separate photogenerated electrons and holes is higher than BiOI [15]. A number of other novel plasmonic photocatalysts with excellent photocatalytic properties also have been reported, such as Ag/AgCl/ZnO [16], Ag/ZnMgO [17] and ZnO/Ag/Ag₂WO₄ [18].

In recent years, adsorption and photodegradation are two more effective methods for the treatment of organic pollutants in water. In order to make these two methods have better application in the treatment of polluted water, we reported a novel composite of Pd/BiOCl/BiOI with both higher adsorption capacity and enhanced photocatalytic properties. The composite has a significant effect on the degradation of organic pollutants. As far as we know, there are few reports on this serious of composite [19,20]. The purpose of this work is to investigate the influence of Pd deposition on photocatalytic activity by degradation of rhodamine B (RhB). We also explored the effect of the Pd content on the removing of rhodamine B. The results and possible photodegradation mechanisms will be described in detail in the article.

2. Experimental section

2.1. Materials

2.1.1. Synthesis of BiOI sample

1 mmol of KI was added into 17 mL of distilled water at room temperature in a polytetrafluoroethylene liner with a volume of 40 mL. Absolute ethanol (17 mL) containing 0.4851 g of Bi(NO₃)₃·5H₂O was stirred for 30 min to obtain a suspension, which was dropped to the KI solution at a rate of 1.5 mL per minute while stirring. The final mixture was stirred for an additional 20 min and then sealed in an autoclave of stainless steel and allowed to react at 150 °C for 24 h. The autoclave was then allowed to drop the temperature to ambient condition. The resulting solid was rinsed

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by DW and absolute ethanol for five times, and dried in oven at 60 °C.

2.1.2. Synthesis of Pd/BiOCl/BiOI

The different volume of PdCl₂ solution (1 mmol/L) was added to the deionized water, the volume of the final solution was 20 mL. 0.05 g BiOI was put into the PdCl₂ solution, and then magnetic stirring in the dark for 12 h 3 mL methanol as a sacrificial agent was added into the above suspension. The result suspension was allowed to react for 4 h under irradiation of a 500 W high-pressure mercury lamp. The resulting precipitate rinsed by DW and absolute ethanol for five times, and dried in oven at 60 °C. Finally, the composites of Pd/BiOCl/BiOI with different theoretical mass percentage of Pd to initial BiOI (1%, 2%, 3% and 4%) were obtained.

2.2. Characterization

X-ray diffraction (XRD) patterns of the as-synthesized products were carried out using a PANalytical X'Pert PRO X-ray diffractometer with Cu K_α radiation ($\lambda = 1.5418 \text{ \AA}$). The morphologies of products were characterized by scanning electron microscopy (SEM, JEOL, JSM-6390LA) in conjunction with a system of energy dispersive spectrometer (EDS) analysis. Transmission electron microscopy (TEM), high-resolution transmission electron microscopy (HRTEM) images are obtained on a JEM-2100F (JEOL, Japan). Ultraviolet-visible (UV-vis) diffuse reflectance spectroscopy (DRS) was carried out using a UV-vis spectrophotometer (Thermo Evolution 220), with BaSO₄ as a baseline. The photoluminescence (PL) emission spectra of the samples were recorded by a spectrofluorometer (Spex 500 M, USA). The Surface components of the composite were measured using X-ray photoelectron spectra (XPS, Thermo 250×i).

2.3. Photocatalytic degradation experiment

The photodegradation abilities of the catalytic were estimated by degrading of RhB, illumination with a 350 W Xe lamp. Photodegradation experiments were completed in a XPA-system photochemical reactor (Xujiang Electromechanical Plant, Nanjing, P.R. China). The reaction vessels were cooled by the running water. In the dark, a reaction suspension composed of catalyst (0.01 g) and an aqueous solution of RhB (10 mL, 0.02 mmol/L–1) was magnetically stirred for 30 min. After that, the suspensions were illuminated with a 350 W Xe lamp under magnetic stirring. The reactor was sealed until one solution become colorless. Suspensions were centrifuged at 10000 revolutions per minute to separate the photocatalyst, and the supernatants were then analyzed using a UV-vis spectrophotometer (Shimadzu UV-2550).

3. Results and discussion

3.1. XRD analysis

Fig. 1 shows the XRD patterns of samples. The diffraction peaks of BiOI were consistent with those of tetragonal BiOI (JCPDS File No. 10-0445). There is no other peaks appeared, indicating that the obtained sample is pure. In addition, the intense peaks show the prepared sample is well crystallized. When PdCl₂ was added, a new peak appeared at $2\theta = 12.0^\circ$, which corresponds to the (001) plane in the tetragonal BiOCl (JCPDS File No. 06-0249). The intensity of this peak grows in stages with the Pd contents from 1% to 4%. However, the peak of Pd was not observed in the XRD pattern of Fig. 1, which may be due to the content of Pd below its detection limit.

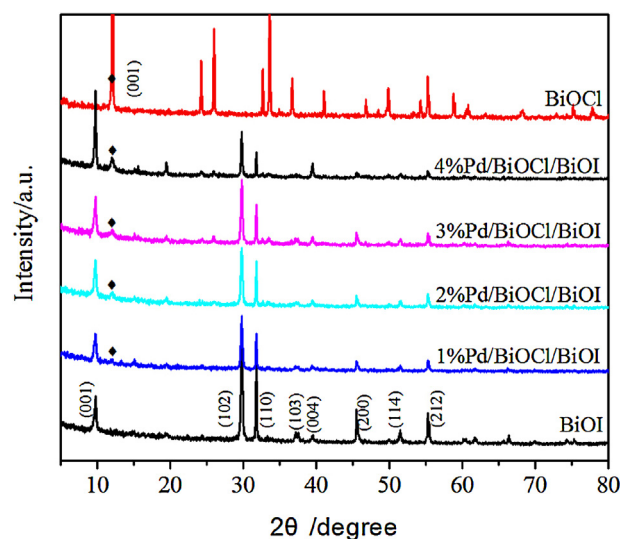


Fig. 1. XRD patterns of BiOI and Pd/BiOCl/BiOI.

3.2. Morphology

Fig. 2 shows the morphologies of as-synthesized sample. The morphology of BiOI (Fig. 2a) sample is a homogeneous, flower-like structure with a size of approximately 4–5 μm . The surface morphologies of Pd/BiOCl/BiOI composites (Fig. 2b–2f) with Pd content increased from 1% to 4% were similar, and no obvious change was possible due to the low amount of BiOCl and Pd.

More details structure of 3%Pd/BiOCl/BiOI was detected by TEM and HRTEM shows in Fig. 3. The high magnification SEM images of 3%Pd/BiOCl/BiOI shows in Fig. 3a. The surface of the sheet structure is not smooth and has small particles. Fig. 3b is the low TEM image of 3%Pd/BiOCl/BiOI with a number of irregular small particles on the surface that are clearly visible, which is consistent with SEM results. Fig. 3c is the HRTEM diagram, in which three different lattice spacing can be found. The lattice parameters of 0.737 nm is belong to the (001) plane of BiOCl, which is consistent with the XRD diffraction peak. The lattice spacing of 0.265 nm and 0.193 nm correspond to the (111) plane of BiOI and (200) plane of Pd, respectively, which illustrates the successful preparation of the Pd/BiOCl/BiOI. The EDS spectrum (Fig. 3d) shows that 3%Pd/BiOCl/BiOI composite included Bi, O, Cl, I and Pd elements, which also demonstrates the successful loading of Pd.

3.3. UV-vis DRS

Fig. 4 shows the UV-vis DRS spectra of the obtained samples. These catalysts can absorb the visible light effectively. The absorption wavelength edges of BiOI at around 700 nm, while after loading Pd, the absorption edge of Pd/BiOCl/BiOI moves in the long-wave direction. When the Pd content was increased, the visible light absorption ability of the catalyst gradually increased. The optical absorption of 1%, 2% Pd/BiOCl/BiOI catalysts showed red-shift compared with BiOI. The 3%, 4% Pd/BiOCl/BiOI composites exhibit intense absorption in entire wavelength range. According to the formula [21] $E_g = 1240/\lambda_g$ (eV), the forbidden width of BiOI can be estimated $E_g = 1.77$ eV. The voltages of valence band (VB) and conduction band (CB) of BiOI can be estimated by the following formula $E_{VB} = X - E^e + 0.5E_g$ and $E_{CB} = E_{VB} - E_g$ where X and E^e are 5.99 eV and 4.5 eV [22]. Thus, the voltages of the VB and CB are reckoned to be 2.38 eV and 0.61 eV.

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