

Regular Article

Double regulation of bismuth and halogen source for the preparation of bismuth oxybromide nanosquares with enhanced photocatalytic activity



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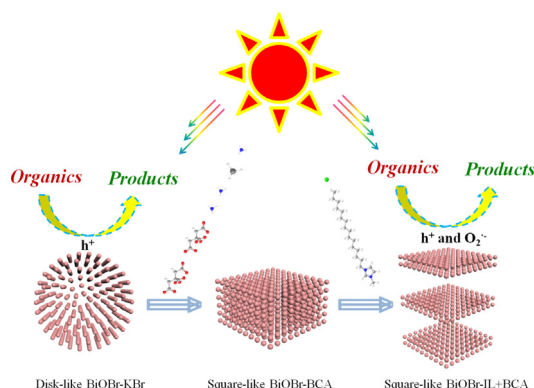
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GRAPHICAL ABSTRACT

Bismuth and halogen source bidirectional regulation has been employed to prepare the BiOBr nanosquares, which displayed improved photocatalytic activity.



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ABSTRACT

In this paper, bismuth oxybromide (BiOBr) nanosquares photocatalysts were synthesized via a facile hydrothermal method with the double regulation of the ionic liquid (IL) 1-hexadecyl-3-methylimidazolium bromide and ammonium bismuth citrate (BCA). To the best of our knowledge, this report is the first to describe the BiOBr material with simultaneous bismuth and halogen bidirectional source regulation. The structures, components, morphologies, optical properties and photocatalytic properties of the as-prepared samples were specifically explored. The photocatalytic ability was assessed using the degradation of rhodamine B under visible light irradiation. The BiOBr-IL + BCA exhibited improved photocatalytic activity compared with the BiOBr materials without double regulation. The primary active species were determined to be holes (h^+) and superoxide radicals (O_2^-) using electron spin resonance (ESR) analysis and free radical trapping experiments. This enhanced activity was attributed to its larger specific surface, the superior electron transfer ability, and the increased negative conduction band position, which favors the photogenerated electrons to trap the molecular oxygen to produce O_2^- . The production of more O_2^- can benefit the removal of pollutants.

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1. Introduction

In recent years, the unique properties of semiconductor photocatalysts have led to semiconductor photocatalysis for pollutant degradation, hydrogen production from water, and CO₂ reduction into hydrocarbon fuels becoming increasingly popular [1–4]. Titanium dioxide (TiO₂) has been considered as one of the most promising photocatalysts for the degradation of different pollutants. However, the high recombination rate of electron-holes pairs and the wide band gap (3.2 eV) result in the low quantum yield of light and poor efficiency in utilizing solar light, limiting its applications [5]. To overcome these drawbacks, several researchers have devoted to developing novel visible-light-response semiconductor materials, such as g-C₃N₄, Bi₂WO₆, and Bi₂MoO₆ [6–10].

Bismuth oxyhalides (BiOX, X = Cl, Br, and I) has the property of self-built, internal static electric fields due to the special layered structure, which features [Bi₂O₂] slabs interlaced with double halogen slabs, and has attracted more attention in the photocatalysis field [11–13]. Among the BiOX, bismuth oxybromide (BiOBr) has attracted significant attention owing to the proper energy band positions and the excellent photocatalytic properties under visible light irradiation. However, the high recombination rate of photo-generated electron-hole pairs limits the activity of BiOBr [14–17]. To improve the performance of BiOX in the photocatalysis field, several tactics have been proposed, such as coupling with other semiconductor materials to prepare hybrid materials, regulating the morphologies of the photocatalysts or controlling the surface structure with different facet exposure, which endow them with completely different properties [18–25]. However, the photocatalytic activities of these materials are still poor and there is great urgency to find appropriate strategies to further boost the photocatalytic behavior of BiOBr.

Ionic liquids (ILs) have been receiving increased attention due to its unique properties. The characteristics including being odorless, nonflammable, good solubility, and good thermal and chemical stability allow ILs to be used in large scale production and will not create environmental pollution. During the synthesis process of the photocatalysts, ILs may simultaneously act as reactants, templates and solvents [26–28]. In this study, ILs were used instead of inorganic salts to synthesize the different sample, the thickness and shape of the materials were changed and the photocatalytic activity has been improved.

In previous studies, Bi(NO₃)₃·5H₂O is typically used as the Bi source in the preparation of bismuth compounds. However, employing Bi(NO₃)₃·5H₂O may frequently generate the agglomeration during the synthesis process. Bismuth citrate ammonium (BCA) has attracted significant interests due to its unique special structure. The [(NH₃)₂C₆H₇O₇]³⁻ organic anion of the BCA can affect the growth of the crystal structure of the samples and can sustain the transparency of the reaction solution in the synthesis process [29–32]. In this study, compared with the Bi(NO₃)₃·5H₂O, BCA as the bismuth source can be used to synthesize the samples with a larger specific surface area and square-like nanostructure, which is important for the photocatalysis process.

In this study, BiOBr samples were prepared via a facile hydrothermal method with the ionic liquid 1-hexadecyl-3-methylimidazolium bromide ([C₁₆mim]Br) and BCA bidirectional regulation. The [C₁₆mim]Br acted not only as the Br source but also as the template at the system. The replacement of Bi(NO₃)₃·5H₂O by the BCA can affect the shape of the samples. The photocatalytic activity of the as-prepared samples was assessed with the degradation of rhodamine B (RhB) under visible light irradiation. The results indicate that the combination of [C₁₆mim]Br and BCA can regulate the sample morphology, increase the specific surface area,

improve the separation efficiency of the photogenerated electron-hole pairs, and ultimately result in an enhanced photocatalytic activity.

2. Experimental

2.1. Material

All chemicals were analytical grade and used as received without purification. The ionic liquid [C₁₆mim]Br (99%) was purchased from Shanghai Chengjie Chemical Co., Ltd.

2.2. The preparation of BiOBr samples

In a typical procedure, 1 mmol Bi(NO₃)₃·5H₂O and 2 mmol KBr were dissolved into 20 ml distilled water and the pH of the suspension was adjusted to 1 using nitric acid while continuously stirring. After the mixture stirred for 30 min, the precursor was transferred into a 25 ml Teflon-lined autoclave and subsequently heated at 140 °C for 24 h. The precipitate was separated by centrifuge and washed with distilled water and ethanol several times. Then, the product was dried at 50 °C for 12 h and was denoted as BiOBr-KBr. In keeping with the above process, using [C₁₆mim]Br instead of KBr, the product was denoted as BiOBr-IL. Using 1 mmol of BCA instead of Bi(NO₃)₃·5H₂O produced the product denoted as BiOBr-BCA. BCA and [C₁₆mim]Br were used instead of Bi(NO₃)₃·5H₂O and KBr, respectively, and the product was denoted as BiOBr-IL + BCA.

2.3. Photocatalyst characterization

The structure and crystalline phase of the as prepared samples were determined by X-ray diffractometry (XRD) using a Shimadzu XRD-6000 X-ray diffractometer with Cu K α radiation. X-ray photoelectron spectroscopy (XPS) using a VG MultiLab 2000 system with a monochromatic Mg-K α source at 20 kV was used to learn the chemical states and elements of the as prepared samples. The morphology of the samples was determined through scanning electron microscopy (SEM) (JEOL JSM-7001F) equipped with an energy dispersive X-ray (EDX) spectroscope and transmission electron microscopy (TEM) (JEOL JEM-2010). The absorption properties were explored via a UV-vis spectrometer (Shimadzu UV-2450 spectrophotometer) using the diffuse reflectance method with BaSO₄ powder as the substrate. After that, the band gaps of the materials were calculated through the Tauc equation. The surface areas of the samples were analyzed via the N₂ adsorption-desorption isotherms collected on a TriStar II 3020 surface area and porosity analyzer (Micromeritics Instrument Corporation, USA). The photoluminescence (PL) spectra of the as prepared samples were detected using a Varian Cary Eclipse spectrometer. The electron spin resonance (ESR) spectra were checked on a Bruker model ESR JES-FA200 spectrometer.

2.4. Photocurrent measurement

The photocurrent experiment was performed to explore the transfer efficiency of the photogenerated charge carriers, which were produced by the as prepared samples under visible light irradiation. The photocurrent experiment systems were equipped with an electrochemical working station (CHI660B, Chen Hua Instruments), a 500 W Xenon lamp and a standard three-electrode configuration. In the three-electrode system, a platinum wire was used as counter electrode, the saturated Ag/AgCl electrode was

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