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Facile synthesis, structure and enhanced photocatalytic activity of novel BiOBr/Bi(C_2O_4)OH composite photocatalysts

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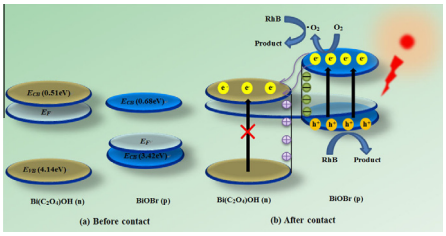
GRAPHICAL ABSTRACT



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ABSTRACT

Novel composite photocatalysts BiOBr/Bi(C_2O_4)OH were successfully fabricated via a chemical etching method. After flower-like Bi(C_2O_4)OH microstructure assembled by nanorods was etched by KBr under an appropriate acidic condition, BiOBr nano-rods could be in-situ generated in nanorods, forming a heterostructure. The heterostructures exhibited a commendable photocatalytic performance toward the degradation of rhodamine B under the visible light irradiation. The effective separation and transfer of the photogenerated electrons and holes were believed to be the main factor for the enhanced activity, which resulted from the intrinsic characteristic of p-n junction. The responsible mechanism was detailedly discussed, and the photogenerated holes and $\cdot O_2^-$ radicals were confirmed to be the main active species for the photodegradation of RhB.

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1. Introduction

With the population growth and intensified industrialization, water contamination is becoming an increasing issue all over the world. Many technologies such as biodegradation, physical adsorption and chemical oxidation, have been used to deal with the serious phenomenon. Unfortunately, these methods may not remove pollutants from aqueous solutions completely, on the contrary, sometimes they cause secondary pollution, which are even more dangerous to the environment. Recently, semiconductor photocatalysis has been known as a potential strategy to drastically transform the pollutants into harmless compounds [1,2]. A variety of photocatalysts such as TiO_2, SnO_2 and Ag_3PO_4, have been developed till now, and they are reported to exhibit considerable photocatalytic activity under UV or visible light irradiation [3–5]. In spite of this, exploring novel photocatalysts with strong oxidation and reduction ability is still intriguing ever increasing interest [6,7].

Recently, Bi-contained compounds receive wide attention as a kind of potential photocatalysts. In these compounds, the intrinsic polarizability induced by the $6s^2$ lone pair electrons of Bi^{3+} can be helpful to the separation of the photogenerated carriers, while the hybridization between O 2p and Bi 6s states can narrow the band gap well and extend the visible light response, both of which favor an enhancement in the photocatalytic performance. To date, many Bi-contained photocatalys such as $BiVO_4$, Bi_2WO_6, $BiPO_4$ and BiOX (X = Cl, Br, I) have been reported, and they all show attractive

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photocatalytic ability [8–11]. Nonetheless, it is worth noting that these reported Bi-contained photocatalysts are mainly composed of Bi ions and inorganic group. Recent studies demonstrate that the participation of organic groups may have a beneficial effect on the photocatalytic reaction. They can not only improve the adsorption ability of photocatalyst toward organic molecule but also facilitate the formation of active ·OH radicals [12,13]. Therefore, we imagine that Bi-contained compounds participated by the organic group may have a great potential in solar photocatalytic applications.

Bismuth oxalate, as a simple organic group-contained compound, has been known for years [14]. Due to its simple preparation and special morphology, it was often used as a precursor material to fabricate other Bi-contained photocatalysts [15,16]. Few investigations were carried out on its photocatalytic performance. Recently, Xiao et al. [17] reported a novel hydroxyl Bismuth oxalate($Bi(C_2O_4)OH$), which exhibited excellent activity toward the degradation of RhB. Unfortunately, it can only be activated under UV light irradiation due to wide band gap, just like TiO_2. To extend the light response range, coupling with narrow-band-gap semiconductor to construct composite photocatalysts is considered as an efficient method. Many photocatalytic composites such as TiO_2/BiOI [18], BiOBr/$BiPO_4$ [19] and C_3N_4/BiOCl [20] have been designed, and they are all resultful. Therefore, $Bi(C_2O_4)OH$-based composites can also be expected to exhibit an excellent photocatalytic activity under the visible light irradiation based on the same strategy.

Herein, we have in-situ constructed novel BiOBr/$Bi(C_2O_4)OH$ composite photocatalysts via a chemical etching method. The obtained samples exhibit interesting morphologies. Compared to pristine $Bi(C_2O_4)OH$ and BiOBr, composite photocatalysts present superior activity toward the degradation of RhB under visible light irradiation. A tentative mechanism for the enhanced photocatalytic performance is proposed.

2. Experimental

2.1. Sample preparation

All the chemicals were purchased from Sinopharm Chemical Reagent Co., Ltd (China) and used as received without further purification. $Bi(C_2O_4)OH$ precursor can be fabricated by the chemical precipitation method. First, 2 mmol of oxalic acid and 2 mmol of $Bi(NO_3)_3{\cdot}5H_2O$ were orderly added in 20 ml of deionized water with vigorously stirring at room temperature. Next, 20 ml ethylene glycol (EG) solution containing 2 mmol of hexadecyltrimethylammonium bromide (CTAB) was added into the above solution. Then, the pH value of the solution above was adjusted to 7. After stirring 16 h, the products were washed repeatedly with ethanol and deionized water for several times, and then dried at 80 °C.

BiOBr/$Bi(C_2O_4)OH$ composites were fabricated through a chemical etching method using HNO_3 and KBr as etching agents. Briefly speaking, 0.942 g of $Bi(C_2O_4)OH$ was added into 30 mL 0.1 M KBr solution. The obtained suspension were divided into three parts, whose pH values were adjusted to 4.0, 2.0 and 1.0 with 1.0 M HNO_3 solution, respectively. After etching for 30 min under the strong stirring condition, the products were washed and then dried at 80 °C, and they were denoted as S4.0, S2.0 and S1.0, respectively. As a comparison, pure BiOBr was prepared as $Bi(C_2O_4)OH$ except for the addition of oxalic acid.

2.2. Characterization

The phase structures of the photocatalysts were determined by X-ray diffractometer (XRD, Bruker D8 Advance) using Cu Kα irradiation (λ = 0.154178 nm), operating at 40 kV and 40 mA. Fourier transform infrared spectra (FTIR) were recorded on VERTEX 80v FTIR spectrometer (Bruker, Germany). The morphologies were investigated by field emission scanning electron microscopy (SEM, Quanta 250) and transmission electron microscopy (TEM, FEI, Tecnai G2 F20). The surface properties of the samples were examined by X-ray photoelectron spectroscopy (XPS: Thermo ESCALAB250, USA). The optical properties of the samples were analyzed by both UV–vis diffuse reflectance spectra (DRS, Varian Cary 300) and Photoluminescence spectra (PL, Varian Cary-Eclipse 500).

2.3. Photocatalytic activity measurement

The photocatalytic activities of as-prepared samples were investigated by the degradation of rhodamine-B (RhB) aqueous solution under visible light irradiation. A 150 W xenon lamp was utilized as a visible light source. Photocatalytic activity measurements were carried out as follows: 0.1 g of photocatalyst was added into 100 mL of 10 mg/L RhB solution. Prior to irradiations, the suspension was magnetically stirred for 1 h in the dark to ensure establishment of an adsorption/desorption equilibrium. At given irradiation time intervals, 3 mL of the suspensions were sampled and centrifuged. The concentration evolution of RhB was analyzed by UV–vis spectrophotometry at the characteristic wavelength of 554 nm.

2.4. Photocurrent measurement

Photoelectrochemical measurements were carried out on an electrochemical workstation (CHI660B, China) with a standard three-electrode configuration. Platinum electrode and saturated Ag/AgCl electrode were used as counter electrode and reference electrode, respectively. An 0.5 M Na_2SO_4 aqueous solution was used as the electrolyte. The working electrode was prepared by dispersing photocatalyst powder on 1.0 cm × 1.0 cm fluoride-tin oxide (FTO) glass.

3. Results and discussion

3.1. Characterization

The phase composition and structure of the resultant samples was investigated by XRD. As shown in Fig. 1, all diffraction peaks of $Bi(C_2O_4)OH$ match well with the orthorhombic $Bi(C_2O_4)OH$ (ICSD Card No. 41-9313), while those of BiOBr are in good agreement with the tetragonal phase BiOBr (JCPDS Card No. 09-0393). No

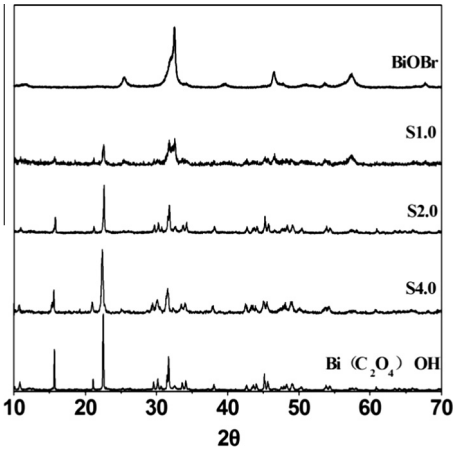

Fig. 1. XRD patterns of as-prepared samples.

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