

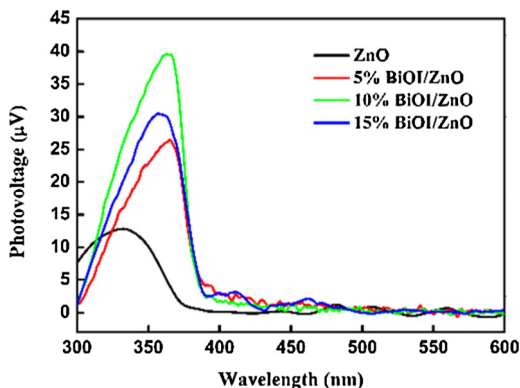


Regular Article

Enhanced photocatalytic degradation of phenol and photogenerated charges transfer property over BiOI-loaded ZnO composites

Jingjing Jiang^a, Hongtao Wang^a, Xiaodong Chen^a, Shuo Li^a, Tengfeng Xie^a, Dejun Wang^{a,b}, Yanhong Lin^{a,*}^a College of Chemistry, Jilin University, Changchun 130012, People's Republic of China^b Department of Chemistry, Tsinghua University, Beijing 100084, People's Republic of China

GRAPHICAL ABSTRACT



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ABSTRACT

In this paper, a series of BiOI/ZnO photocatalysts containing various BiOI contents were prepared by a facile two-step synthetic method. The structure and crystal phase, morphology, surface element analysis, optical property of as-prepared samples are measured by X-ray diffraction (XRD), Scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS) and UV-Vis diffuse reflectance spectrometry (DRS). BiOI/ZnO photocatalytic activities of the prepared photocatalysts were evaluated by photocatalytic degradation of phenol under simulated light irradiation. The phenol degradation rate reached 99.9% within 2 h under simulated solar light irradiation. The probable photocatalytic mechanism of composites photocatalysts is discussed by active species trapping experiments, the surface photovoltage (SPV), the transient photovoltage (TPV) and photoluminescence (PL) measurements. The results manifest that the superior photocatalytic activity of BiOI/ZnO composites is derived from the strong internal electric field between BiOI and ZnO, which is beneficial for the effective separation and transfer of photogenerated charges in ZnO. Moreover, the loading of BiOI on the surface of ZnO inhibited the recombination of photogenerated charge carriers in ZnO, resulting in excellent photocatalytic activity. On the contrary, the effect of an extension of the light absorption range induced by the introduction of BiOI on the phenol degradation activity is not significant.

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* Corresponding author.

E-mail address: linyh@jlu.edu.cn (Y. Lin).

1. Introduction

Phenol and phenolic compounds are among the most potential pollutants discharged from various industries, such as paper, mills, herbicide and fungicide production plants [1,2]. Phenol is toxic and people who are hypersensitive to it could experience death or serious side effects at very low concentration. Therefore, to protect the environment and public health, removal of phenol and phenolic compounds in wastewater effectively is a burning issue worldwide [3]. Various techniques were developed to treat the phenol containing wastewater including catalytic oxidation [4–6], microbial degradation [7], solvent extraction [8], membrane filtration [9], advanced oxidation processes [10], activated carbon adsorption [11], electrochemical [12], and others [13]. Among these methods, semiconductor photocatalysis, as an effective technology to address the global energy and environmental issues, has been widely applied in the removal of phenolic pollutants in wastewater [14]. Zinc oxide (ZnO) is widely recognized as a promising material for photocatalytic processes owing to rich morphologies, high electron mobility [15–17], environmentally friendly feature, excellent carrier mobility with a large exciton binding energy of 60 meV [18] and relatively low cost [19]. Nevertheless, the comparatively wide band gap (~ 3.2 eV) as well as high recombination ratio of electron-hole pairs greatly impede its practical application. Consequently, to make full use of solar energy, developing highly active photocatalysts under sunlight irradiation has become an imperative research topic [20,21].

Recently, BiOI (bismuth oxyiodide), as a protuberant photocatalyst, has drawn extensive interests of researchers owing to its potential applications in the photocatalytic degradation of organic pollutants [18]. BiOI, with a relatively narrow band gap (1.8 eV), has the strong absorption in the visible light range which can be served as an ideal sensitizer to sensitize the wide band-gap semiconductors, such as TiO_2 [22], ZnO [23], GO [24], BiPO_4 [25], $\text{Bi}_2\text{-MoO}_6$ [26]. Furthermore, BiOI displays outstanding photocatalytic activity for degrading organic pollutants owing to the high dipole moment (value above 2.00 D) and unique layered structures [27]. Huang et al reported that the $\text{BiVO}_4/\text{BiOI}$ composite photocatalysts fabricated by a facile in situ deposition method exhibited superior photocatalytic performance in degradation of phenol. The photocatalytic performance could achieve 64% after 16 h visible-light irradiation [28]. Liu et al. successfully prepared a $\text{BiPO}_4/\text{BiOI}$ heterojunction photocatalyst with high photocatalytic removal efficiency for phenol. After 3 h of visible light irradiation, the phenol could be removed 45.8% [29]. In addition, the photocatalytic activity of BiOI/ZnO heterojunctions has been reported. However, to the best of our knowledge, there was no report on the degrading uncolored phenol performance in the BiOI/ZnO . Furthermore, most of the photocatalytic studies focus on the net photoconversion efficiency, the investigations on the behavior of charges separation and transfer at the surface or interface and the origin of the enhanced photocatalytic activity of BiOI/ZnO are still lacking.

Thus, in this paper, a class of novel p–n heterostructures comprising p-type BiOI and n-type ZnO was successfully constructed by loading amounts of BiOI nanoflakes onto the surface of ZnO nanorod. The obtained BiOI/ZnO composites show remarkably enhanced photocatalytic performances for the degradation of phenol in aqueous solution under simulated solar light irradiation. After 2 h of solar-light irradiation, the degradation efficiency of phenol is about 99.9% for the BiOI/ZnO composite materials. Furthermore, the probable mechanism of phenol degradation in the presence of BiOI/ZnO composites and the process of charge separation and transfer at the surface or interface were also discussed in detail.

2. Experimental

2.1. Materials

All chemicals were analytical grade reagents, and used as received without further purification. zinc acetate dehydrate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$, Xilong Chemical Co., Ltd, $\geq 99.0\%$), sodiumhydroxide (NaOH, Beijing Chemical Works, $\geq 96.0\%$), bismuth nitrate pentahydrate ($\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$, $\geq 99.0\%$; Xilong Chemical Co., Ltd), ethylene glycol ($(\text{HOCH}_2)_2$, EG), potassium iodide (KI), and DI water.

2.2. Preparation of pure ZnO and different mole ratio BiOI/ZnO composites

The nanorod ZnO was prepared by a typical hydrothermal method [30]. In a typical procedure, 0.878 g zinc acetate dehydrate were dissolved in 60 mL deionized water, and then 2.4 g sodium hydroxide were added into above solution with constant stirring. After 5 min, the resulting solution was moved to a 100 mL Teflon-lined stainless steel autoclave, sealed and maintained at 160°C for 10 h. When the reactions were completed, the autoclave was cooled down to room temperature naturally. The white products were centrifuged, and washed several times with distilled water to remove residual salts. Finally, the products were dried at ambient air at 70°C and the ZnO samples were obtained.

A series of BiOI/ZnO composites were synthesized by a simple procedure. 0.081 g as-prepared ZnO nanorods above were ultrasonically suspended in 30 mL deionized water. Meanwhile, x mole bismuth nitrate pentahydrate (x = 5%, 10%, 15%) were added into 30 mL glycol under ultrasound, which will pour into the ZnO suspension. Finally, corresponding amount potassium iodides were added dropwise to the above ZnO suspension system and the mixed liquor was stirred for 5 h and centrifuged, dried. A series of BiOI/ZnO samples with various BiOI contents were obtained.

2.3. Characterization and photocatalytic performance

The phase and purity of the products were examined by XRD with a Rigaku D/Max-2550 diffractometer using $\text{Cu K}\alpha$ radiation at 50 kV and 200 mA in the 2θ range between 20 and 70° at a scanning rate of 10°min^{-1} . The surface morphologies and dimensions of pure ZnO and BiOI/ZnO photocatalysts were obtained by using SEM (shimadzu, ss-550) and HRTEM was carried out on a TECNAIG2 transmission electron microscope (FEI company) working at 200 kV. XPS measurements were performed on a Thermo VG Scientific ESCALAB 250 spectrometer using monochromatized $\text{Al K}\alpha$ excitation. The UV–Vis DRS was recorded with a UV–Vis–NIR spectrophotometer (Shimadzu UV-3600) equipped with an integrating sphere using BaSO_4 as a substrate to obtain absorption over the 300–800 nm. Room-temperature photoluminescence (PL) spectra with an excitation wavelength of 325 nm were measured on a FLUOROMAX-4. The SPV and TPV measurement were performed as described in our previous report [31]. The SPV measurement was conducted by the instrument that is composed of a computer, a sample cell, a lock-in amplifier (Sr830-D SP) with a light chopper (SR540). Monochromatic light was provided by passing light from a 500 W xenon lamp (CHFXQ500 W, Global Xenon Lamp Power) through a grating monochromator (Omni-5007, No.09010, Zolix). The TPV equipment was measured on a self-assembly instrument [32,33]. The samples were excited with a laser radiation pulse (wavelength of 355 nm and pulse width of 5 ns, wavelength of 532 nm and pulse width of 5 ns) from a third-harmonic Nd: YAG laser (Polaris II, New Wave Research, Inc). The 500 MHz digital

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