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One-pot solvothermal synthesis of Ag nanoparticles decorated BiOCOOH microflowers with enhanced visible light activity

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ABSTRACT

In this work, plasmonic Ag nanoparticles (NPs) decorated BiOCOOH microflowers as one kind of efficient visible-light-driven photocatalysts were prepared *via* a one-pot solvothermal method and systematically characterized. Compared with pristine BiOCOOH, Ag/BiOCOOH heterojunctions showed greatly enhanced visible light photocatalytic activities towards RhB degradation. Especially, the 5 wt% Ag/BiOCOOH heterojunction displayed the maximum activity, and the degradation rate is 10.9 times higher than that obtained by using pristine BiOCOOH. The exceptional photocatalytic activity of Ag/BiOCOOH is attributed to the surface plasmon resonance (SPR) effect induced by Ag NPs and formation of the heterojunction between Ag and BiOCOOH. Holes and superoxide radicals mainly dominate the RhB degradation. More importantly, Ag/BiOCOOH is highly stable in photocatalysis.

1. Introduction

Semiconductor photocatalysts, as the key of photocatalysis technique, have drawn stupendous attention [1]. Among them, Bi-based materials have been widely developed as efficient photocatalysts in virtue of their unique layer configuration [2–6]. BiOCOOH, as a new Bi-based compound, has emerged as a promising photocatalyst for pollutant removal [7,8]. However, its wide band gap and low quantum efficiency undermine its practical application.

Constructing heterojunctions can effectively overcome these drawbacks [9,10]. However, there is little work that reports on the exploration of BiOCOOH-based heterojunctions, except for BiOI/BiOCOOH [11], Ag_2O/BiOCOOH [12] and G/BiOCOOH [13]. These heterojunctions exhibited superior activity for pollutant removal. Thus, the further development of BiOCOOH-based heterojunctions with higher performance is highly desirable and anticipated.

The combination of semiconductors with metallic Ag to form plasmonic photocatalysts is an effective strategy towards ameliorating the photocatalytic activity. Several Ag-based heterojunctions, such as Ag/$Bi_2O_2CO_3$ [14], Ag/TiO_2 [15], and Ag/$SnWO_4$ [16] have been reported to present exceptional photocatalytic activity for pollutant degradation. However, to our knowledge, there is no report on the development of Ag/BiOCOOH heterojuctions.

In this work, Ag nanoparticles (NPs) uniformly anchoring on BiOCOOH microflowers were prepared by a one-pot solvothermal method. The photocatalytic properties of Ag/BiOCOOH were assessed by the decomposition of RhB dye under visible light. All heterojunctions depicted better photocatalytic activity and stability for RhB degradation. The mechanism for the improved activity of Ag/BiOCOOH was studied.

2. Experimental section

2.1. Preparation of Ag/BiOCOOH

2 mmol Bi(NO_3)·5H_2O was ultrasonically dissolved in the mixture (25 mL glycerol + 5 mL H_2O + 10 mL DMF). Then, a certain amount of $AgNO_3$ was dissolved in the solution under vigorously stirring. The resulting solution was transferred to a 100 mL autoclave and reacted at 160 °C for 15 h. After reaction, the system was cooled down naturally. Finally, the obtained samples with 1 wt%, 3 wt%, 5 wt% and 10 wt% Ag (named as Ag/BiOCOOH-1%, Ag/BiOCOOH-3%, Ag/BiOCOOH-5% and Ag/BiOCOOH-10%) were washed with water-ethanol for several times, and then dried at 70 °C overnight. For comparison, pristine BiOCOOH was prepared under same condition without adding $AgNO_3$.

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2.2. Characterization

The catalysts were studied by a X-ray diffractometer (XRD; Bruker), a scanning electron microscopy (SEM; Hitachis-4800), a transmission electron microscopy (TEM; JEM-2100F), energy dispersive X-ray spectrometer (EDX), and optical diffuse reflectance spectra were acquired on a spectrophotometer (Shimadzu, UV-2600).

2.3. Photocatalytic tests

The photocatalytic activity of all samples were evaluated by degrading RhB (50 mL, 5 mg L^{-1}) under visible light (300 W xenon lamp, $\lambda > 400$ nm). Specifically, 20 mg of the sample was ultrasonically dispersed in RhB aqueous solution for 2 min. Prior to illumination, the suspension was continuously stirred in the dark for 1 h. After that, the suspension was irradiated by visible light. 4 mL of the solution was taken from the reactor at certain intervals. The RhB concentrations were determined by measuring the characteristic absorption peak at 554 nm on a UV-2600 spectrophotometer.

Trapping experiments were carried out by adding different sacrificial agents into the RhB solution (50 mL, 5 mg L^{-1}), including 6 mM $AgNO_3$, 1 mM ammonium oxalate (AO), 1 mM *p*-benzoquinone (BQ) or 1 mM iso-propanol (IPA).

3. Results and discussion

3.1. Characterization of photocatalysts

The morphology of BiOCOOH and Ag/BiOCOOH (Ag/BiOCOOH-5%) was examined by SEM (Fig. 1a-d). Pure BiOCOOH exhibits a flower-like nanostructure (diameters: 2–4 μm) assembled by nanosheets with smooth surface (Fig. 1a, b). As for Ag/BiOCOOH-5%, it maintains the flower-like morphology, and these microflowers are constructed by BiOCOOH nanosheets and Ag NPs (Fig. 1c, d). Apparently, Ag NPs (size:~21 nm) are uniformly distributed on the surface of BiOCOOH microflowers (Fig. 1d). TEM images (Fig. 1e, f) clearly show that Ag/BOCH-5% is composed of BiOCOOH microflower (diameter:~3.5 μm) and Ag NPs (size:~21 nm). Ag NPs are well embedded in the BiOCOOH substrate, forming tight contact between BiOCOOH and Ag NPs.

Fig. 2a shows the XRD patterns of all samples. The XRD pattern of pristine BiOCOOH can be indexed to tetragonal BiOCOOH (JCPDS card No. 35-0939). The XRD patterns of Ag/BiOCOOH heterojunctions can be assigned to the combination of Ag and BiOCOOH phases. With the amount of Ag increasing, the intensity of Ag peaks at $2\theta = 38°$ and 44° was gradually strengthened, whereas those of BiOCOOH were weakened.

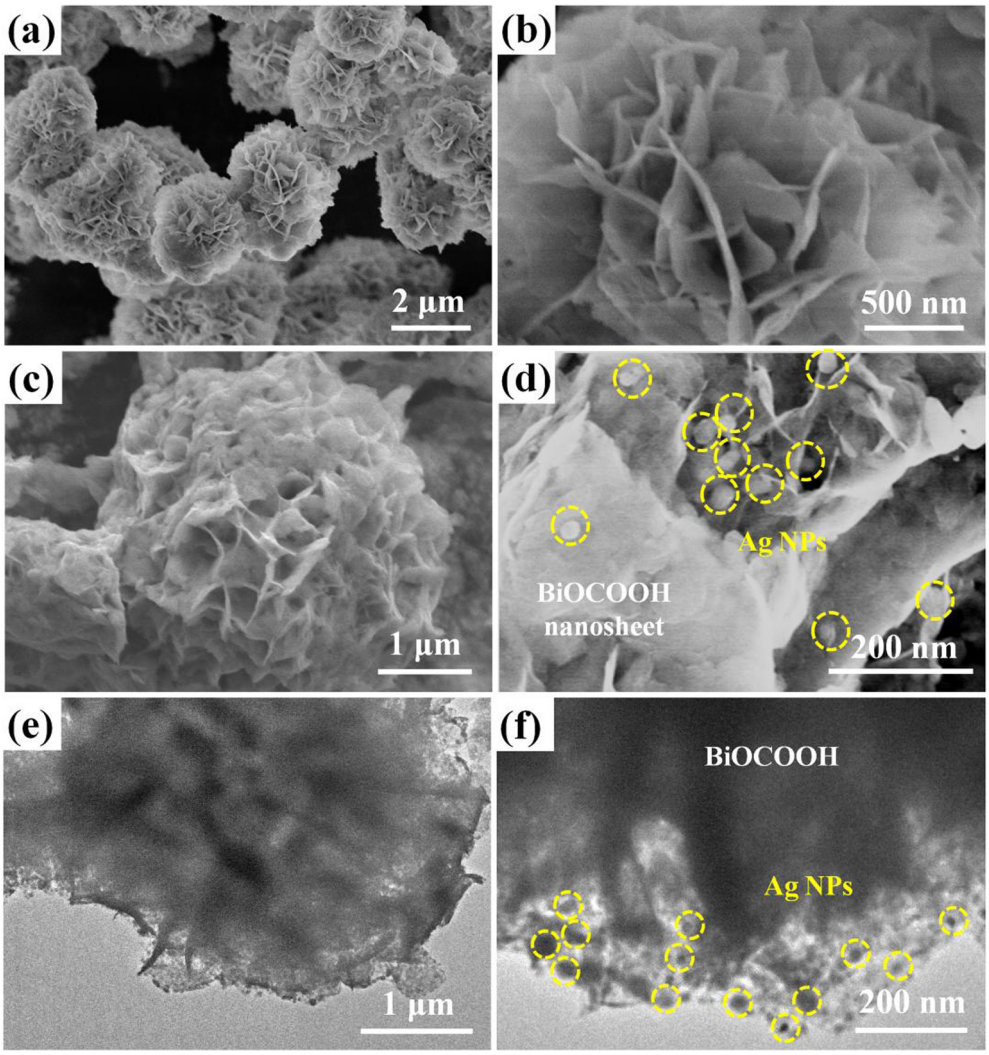

Fig. 1. SEM images of BiOCOOH (a, b) and Ag/BiOCOOH-5% (c, d); TEM images of Ag/BiOCOOH-5% (e, f).