

## **Immobilization of BiOX (X=Cl, Br) on activated carbon fibers as recycled photocatalysts**

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### **EXPERIMENTAL SECTION**

#### **Materials**

Activated carbon fibers were provided by Toray Inc. Rhodamine B, Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O, 2-methoxyethanol, 1-hexadecyl-3-methylimidazolium chloride ([C<sub>16</sub>Mim]Cl), nitric acid, ethyl alcohol and 1-hexadecyl-3-methylimidazolium bromide ([C<sub>16</sub>Mim]Br) were purchased from the Sinopharm Chemical Reagent Corporation (Shanghai, China). All chemicals were of analytical grade and were used without further purification.

#### **Pretreat activated carbon fibers by nitric acid**

The activated carbon fibers were pretreated by dipping in concentrated nitric acid (65%) in three-necked flask and refluxed for 4-6 h in an oil bath at 388K. After it was cooled to room temperature, the activated carbon fibers were subsequently washed with deionized water, and then dried in a vacuum oven at 353 K.

#### **Hydrothermal synthesis of (BiOX/ACF) and (BiOX/ITO)**

Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O (2-4mmol) and 1-hexadecyl-3-methylimidazolium bromide ([C<sub>16</sub>Mim]Br) (3-6mmol) were separately dissolved in 30-40 mL of 2-methoxyethanol

and mixed together. The suspension was stirred and transferred into a 100 mL autoclave. The ACF pretreated by nitric acid or ITO glass was immersed in the solution. The autoclave was heated and maintained at 433K for 1 h and cooled down to room temperature. (BiOBr/ACF) was washed 7 times with deionized water and 3 time with ethyl alcohol, and (BiOBr/ITO) was washed in ethyl alcohol to remove any ionic residual, then dried in oven at 333K for 10 h. Likewise, (BiOCl/ACF) and (BiOCl/ITO) were prepared using [C<sub>16</sub>Mim]Cl instead, while keeping other experimental conditions unchanged.<sup>1</sup>

### **Characterization**

The zeta potential of ACF and BiOX were characterized by Zeta potential instrument. Powder X-ray diffraction (XRD) patterns were recorded on a Bruker AXS D8 advance powder diffractometer with Cu K $\alpha$ X-ray radiation. The scanning electron microscopy (SEM) images were obtained on a Hitachi S-4800 microscope with an accelerating voltage of 7.0 kV. The infrared (IR) spectra of ACF and BiOBr/ACF were recorded on an IR Prestige-21 Fourier transform spectrophotometer by dispersing samples in KBr pellets in 400-4000 cm<sup>-1</sup>.

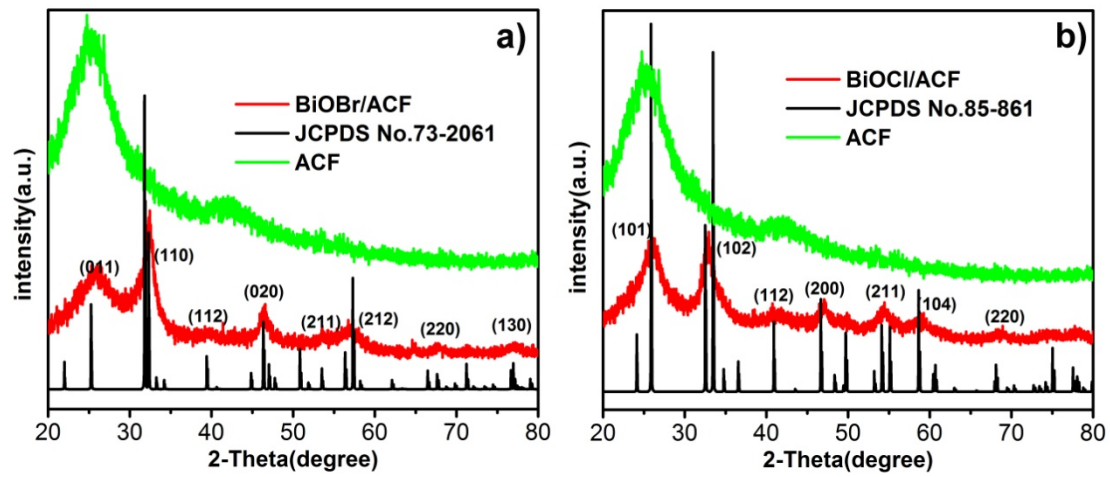
### **Photocatalytic Measurement**

Rhodamine B(RhB) is a common dye that has been widely used for investigating the photocatalytic efficiency of various types of photocatalysts. It is generally considered to be very stable to light and difficult to be decomposed, though RhB can absorb visible light at 554 nm. The photocatalytic performance of the prepared samples was conducted at ambient temperature. Experimental details have been shown as follows: The photocatalytic experiments were performed with the RhB

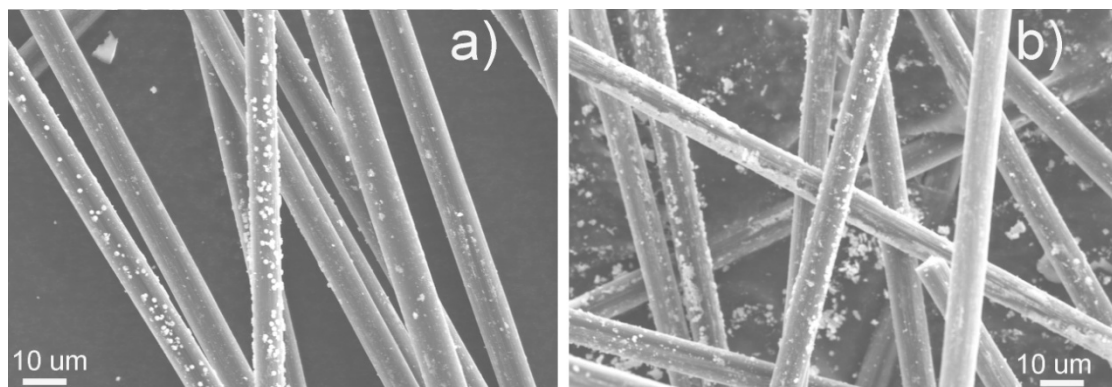
aqueous solution ( $20 \text{ mg L}^{-1}$ ). A 300 W Xe arc lamp (PLS-SXE300, Beijing Trusttech Co., Ltd.) was used as the light source. Prior to irradiation, all the samples were immersed in the RhB solution and kept in the dark for 12h to ensure the adsorption/desorption equilibrium. At the given time interval, the aqueous solution was taken for monitoring the absorbance of 554 nm as a function of irradiation time. As a comparison, the photocatalytic activity of the BiOX(X=Cl, Br)/ITO was also researched under the same conditions. The same amounts of BiOX have been used during the photocatalytic comparison. In addition, the colorless 2,4-Dichlorophenol (2,4-DCP,  $20 \text{ mg L}^{-1}$ ) was also used as the target organic substance to evaluate the photocatalytic performance of BiOX/ACF composite structures under same experimental conditions.

#### **Recycle experiment of the photocatalyst**

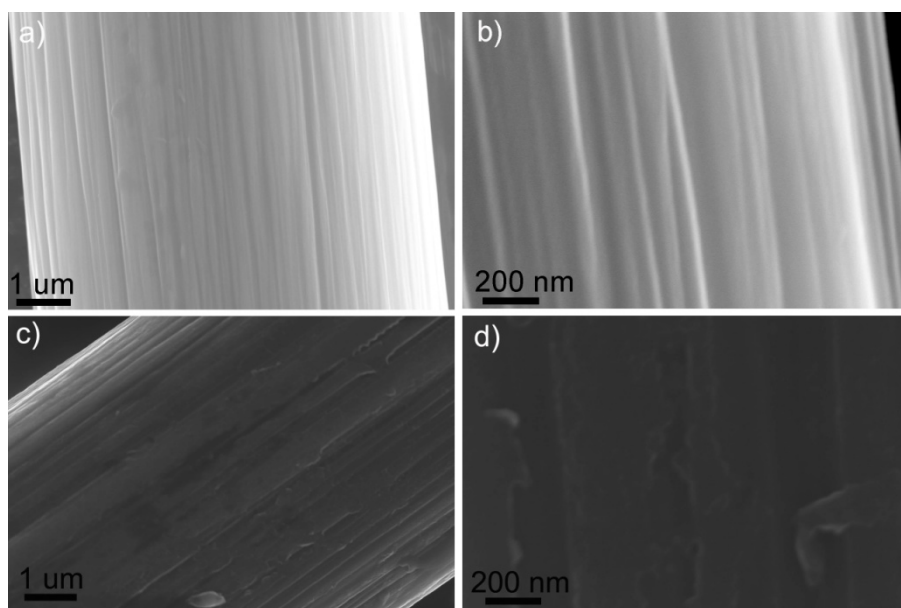
In order to research the recycling property of BiOX/ACF, recycle experiment for the photocatalytic degradation of RhB was designed under 300 W Xe arc lamp light. The BiOX/ACF photocatalyst was continuously used for five cycles to decompose RhB solution. After each cycle, aqueous solution after photocatalytic reactions was removed from reactive beaker, BiOX/ACF was washed thoroughly, and then fresh RhB solution was added into the reactive beaker and adsorbs 2 hours to carry out the next cycle.



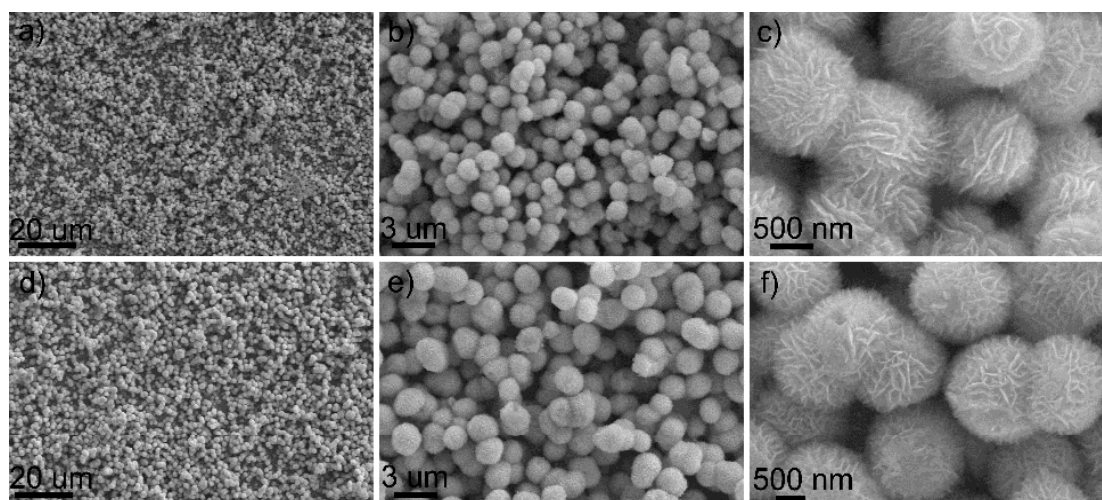
**Fig. S1.** XRD patterns of BiOBr(a) and BiOCl(b) grown on ACF pretreated by nitric acid



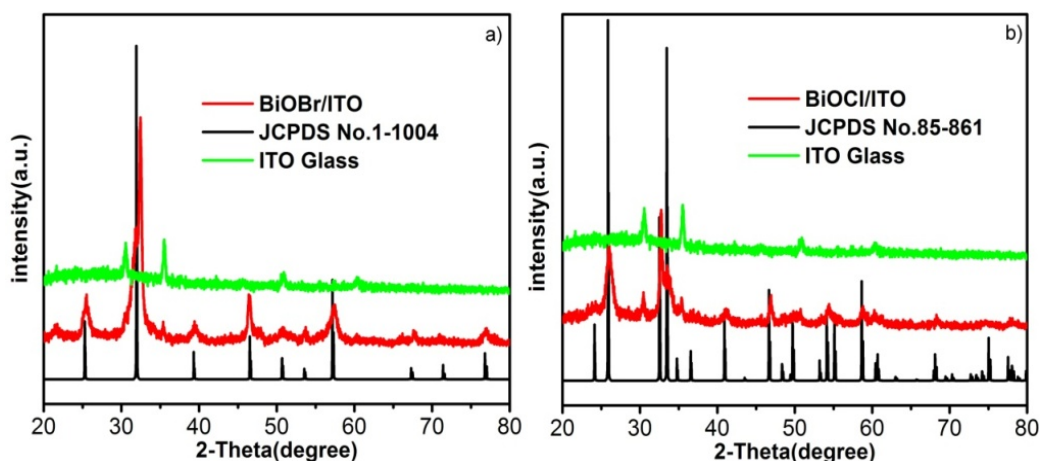
**Fig. S2.** SEM images of BiOBr(a) and BiOCl(b) grown on activated carbon fibers not pretreated by nitric acid



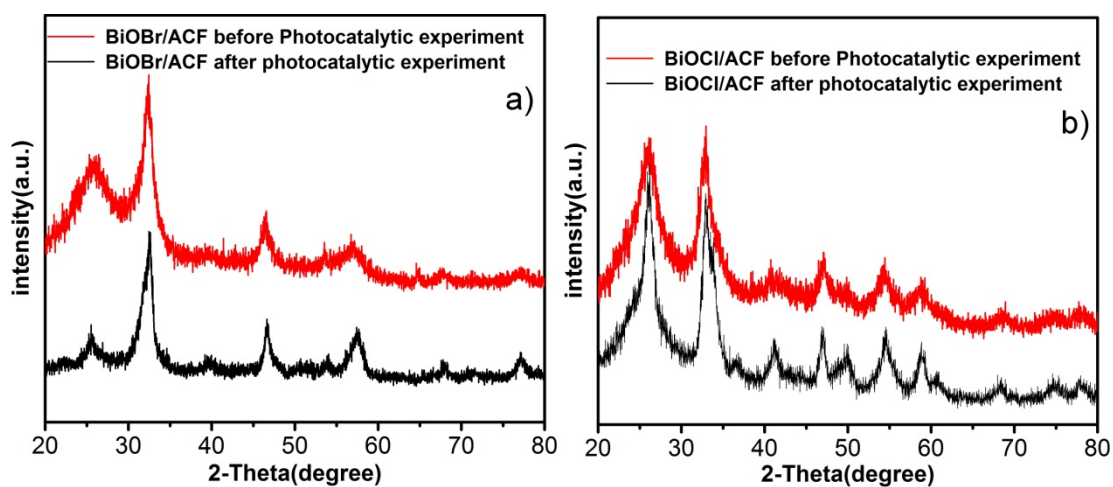
**Fig. S3.** (a,b) SEM image of ACF, (c,d) SEM image of ACF pretreated by nitric acid



**Fig. S4.** SEM images of BiOBr(a,b,c) and BiOCl(d,e,f) grown on ITO glass substrate

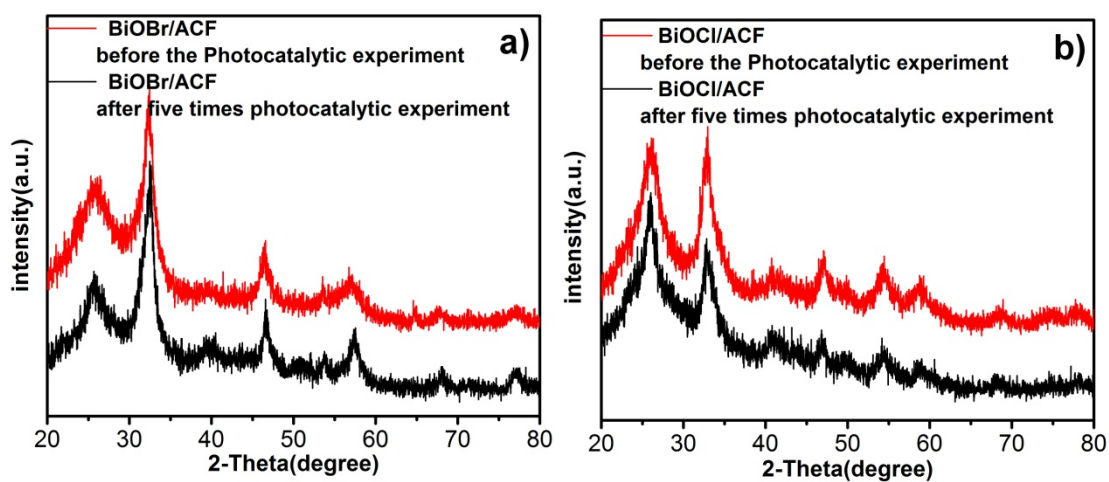


**Fig. S5.** XRD patterns of BiOBr(a) and BiOCl(b) grown on ITO glass substrate



**Fig. S6.** XRD patterns of BiOBr/ACF (a) and BiOCl/ACF (b) after photodegradation

2,4-DCP



**Fig. S7.** XRD patterns of BiOBr/ACF (a) and BiOCl/ACF (b) after five times

photocatalytic experiments

## REFERENCES

- [1] H. F. Cheng, B. B. Huang, Z. Y. Wang, X. Y. Qin, X. Y. Zhang, Y. Dai, *Chem. Eur. J.*, 2011, **17**, 8039.