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Photocatalytic Property of Hierarchical Structure Based on Fe-doped BiOBr Hollow Microspheres

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1. Experimental

0.6 g of $Bi(NO_3)_3 \cdot 5H_2O$ and appropriate amounts of $FeCl_3$ was dissolved in 60 mL of 2-methoxyethanol to from a clean solution, then 1.2 g of CTAB were added slowly to the solution by magnetic stirring. The resultant solution was transferred into a Teflon-lined stainless steel autoclave, followed by a solvothermal treatment at 160 °C for 0-24 h. After cooled to room temperature, the precipitates were washed with deionized water and ethanol six times, dried in a vacuum oven at 60 °C for 12 h.

2. Characterization

Structures of the as-prepared samples were analyzed with a SIEMENS Diffraktometer D5000 X-ray diffractometer using Cu K α radiation source at 35 kV, with a scan rate of 0.02° s⁻¹ in the 2 θ range of 10-80°. The morphologies were investigated by ULTRA-55 field-emission scanning electron microscopy (FE-SEM) equipped with an energy dispersive X-ray spectrum (EDS, Inca Energy-200) at an accelerating voltage of 10 kV and JSM-2100 transmission electron microscopy (TEM). The Brunauer-Emmet-Teller (BET) specific surface area of the samples were determined by a high speed automated area and pore size analyzer (F-Sorb3400, China). UV/Vis diffuse reflectance spectra were recorded with a UV-vis spectrometer (U -3010, Hitachi).

3. Measurement of photocatalytic activity

The photocatalytic activity of the as-prepared samples was investigated by the photo-degradation of RhB. The photo-degradation experiments were carried out under visible light irradiation whose source was an 11 W daylight lamp (25 Hz) equipped with UV cutoff filter to provide visible light ($\lambda \ge 400$ nm). 50 mg of the as-prepared catalysts was suspended in 50 mL RhB aqueous solution ($C_0 = 10 \text{ mg} \cdot \text{L}^{-1}$) with constant stirring. Prior to irradiation, the suspensions were stirred in the dark for 1 h to ensure the adsorption-desorption equilibrium. The temperature of suspensions was maintained below 283 K by a flow of cooling water during the reaction. The change of RhB concentrations (C) in accordance with the irradiation time was measured by JASCO V-570 UV/Vis/NIR spectrophotometer (Japan).

4. Electrochemical Measurement

Cyclic voltammetry test of the as-prepared samples was measured on a CHI 660D electrochemical workstation under ambient conditions. A three-electrode cell with 1 M KCl electrolyte was used, in which Pt foil was used as the counter electrode, glass carbon electrode as

the working electrode and an Hg/Hg₂Cl₂/KCl electrode as the reference electrode. And cyclic voltammetry was performed between -1.0 V and 0.5 V with scan rates of 20 mV s⁻¹, 50 mV s⁻¹, 100 mV s⁻¹. The photocurrent transient response toward glucose and Nyquist impedance plots were measured in 0.1 M Na₂SO₄ solution under an 11 W daylight lamp (25 Hz) equipped with UV cutoff filter to provide visible light ($\lambda \ge 400$ nm) irradiation.

5. HR-TEM image



Figure S1 HR-TEM image of Fe-doped BiOBr samples.





Figure S2 (A, B) FE-SEM images and (a, b) the corresponding EDS spectra of the different sections on Fe-doped BiOBr microsphere, (A, a) and (B, b).

7. The summary table of each element

| Element | Weight% |
|---------|---------|
| СК | 7.75 |
| ОК | 8.14 |
| Si K | 5.27 |
| Fe K | 4.16 |
| Br L | 15.21 |
| Ві М | 59.47 |
| Totals | 100.00 |

8. Plot of Kubelka-Munk function versus energy



Figure S3 The plot Kubelka-Munk function versus energy of BiOBr and Fe-doped BiOBr samples.

9. N₂ adsorption-desorption



Figure S4 Nitrogen desorption isotherms and corresponding pore-size distribution of the Fe-doped BiOBr and BiOBr samples.

10. XRD patterns



Figure S5 XRD patterns of the as-prepared Fe-doped BiOBr samples under different stages.

11. FE-SEM images and EDS spectra



Figure S6 FE-SEM images and EDS spectra of the Fe-doped BiOBr samples with different Fe doping content, (A, a) 3 % and (B, b) 15 %.

12. Cyclic voltammetry curves



Figure S7 CV curves of the Fe-doped BiOBr samples in KCl solution at different scan rates from 20 to 100 mV s^{-1} .

13. The plot of photocurrent transient response and Nyquist impedance



Figure S8 The photocurrent transient response and Nyquist impedance plots of BiOBr and Fe-doped BiOBr samples in $0.1 \text{ M} \text{ Na}_2\text{SO}_4$ solution under visible light irradiation.

14. Cycling degradation test



Figure S9 The rate of the cycling degradation on RhB of Fe-doped BiOBr samples.