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Supplementary Information

Prussian blue analogue in-situ derived hierarchical flowerlike ZnS/FeS₂ Z-scheme heterojunction for boosted visiblelight responsive photocatalytic degradation of Rhodamine B with synergistic peroxydisulfate activation

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Characterization

The crystal structures of the photocatalysts were analyzed by a D8 Advance Xray diffractometer (Bruker AXS, Karlsruhe, Germany) with a scanning range of 5 to 70° and a scanning rate of 5° min⁻¹. The morphologies and elemental distributions of the samples were observed by a scanning electron microscope (Hitachi, S-4800, Japan), operating at 3.0 kV. The TEM images of the samples were obtained by a transmission electron microscope (Hitachi, JEM-2100 plus, Japan), operating at 200 kV. The surface elemental compositions, chemical states, and the position of the valence band of the samples were tested by X-ray photoelectron spectroscopy (Kratos, Axis supra, UK). The optical absorption properties of the samples were obtained using a UV-vis spectrophotometer (Shimadzu, UV-3600 plus, Japan) with BaSO₄ as a reference. The fluorescence properties of the samples were characterized by a fluorescence spectrometer (Edinburgh, FS5, UK) with an excitation wavelength of 335 nm. All electrochemical experiments were carried out on an electrochemical workstation (CH Instruments, CHI 660D, China) with Na₂SO₄ as the electrolyte. The as-prepared catalysts were used as working electrodes, and the counter and reference electrodes were Pt and Ag/AgCl electrodes respectively. The active species were detected by electron spin resonance spectrometry (Bruker, EMXplus-10/12, Germany).

Preparation of ZnS: 3 mmol ZnCl₂ was sonicated and dispersed in 50 mL of deionized water as A. 3 mmol thioacetamide was sonicated and dissolved in 30 mL of deionized water as B. B was slowly added dropwise to A and stirred for 30 min, after which the mixture was transferred to a 100 mL Teflon-lined stainless-steel autoclave and heated at 200 °C for 12 h. After the reaction, the sample was collected by centrifugation and washed 3 times with ethanol and deionized water, and then dried under vacuum at 60 °C for 12 h to obtain the ZnS sample.

Preparation of FeS₂: 3 mmol K₄[Fe(CN)₆] \cdot 3H₂O and 0.6 g PVP were dissolved in 50 mL of deionized water as C, 3 mmol thioacetamide was sonicated in 30 mL of deionized water as D. D was slowly added dropwise to C and stirred for 30 min, after which the mixture was transferred to a 100 mL Teflon-lined stainless-steel autoclave and heated at 200 °C for 12 h. After the reaction, the sample was collected by centrifugation and washed three times with ethanol and deionized water, and then dried under vacuum at 60 °C for 12 h to obtain FeS₂.



Fig. S1. The XRD patterns of (a) ZnS and (b) FeS₂.



Fig. S2. The SEM images of (a-c) ZnS and (d-f) FeS_2 .



Fig. S3. The EDX spectra of ZnS/FeS_2 .



Fig. S4. TOC removal rates of ZnS/FeS₂, ZnS/FeS₂+PDS, ZnS+PDS and FeS₂+PDS.



Fig. S5. The SEM images of (a-b) of ZnS/FeS_2 after 5 cycles.



Fig. S6. VB-XPS spectra of (a) ZnS and (b) FeS₂.



Fig. S7. Degradation efficiency of RhB in a light-free environment by ZnS/FeS₂ and ZnS/FeS₂+PDS.