

## Supporting Information

### **Ball-milling preparation of ZnFe<sub>2</sub>O<sub>4</sub>/AgI nanocomposite with enhanced photocatalytic activity**

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## Section 1. Materials

Zinc sulfate heptahydrate ( $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ ) was obtained from Chengdu Kelong Chemical Reagent Company (Chengdu, China). Silver nitrate ( $\text{AgNO}_3$ ) was purchased from Chengdu Busan Chemical Reagent Co (Chengdu, China). Nitro blue tetrazolium (NBT), ferrous sulfate heptahydrate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ), potassium iodide (KI), oxalic acid ( $\text{H}_2\text{C}_2\text{O}_4$ ), ethylene glycol (EG), ethanol (EtOH), 1,4-DHP, AM, MO, indole, isopropanol (IPA), titanium dioxide ( $\text{TiO}_2$ , P25) and terephthalic acid (TA) were purchased from Titan Technology Exploration Platform. EDTA-2Na was obtained from Chongqing Maoye Chemical Reagent Co (Chongqing, China). All reagents were of analytical grade and used without further purification. All solutions were prepared using deionized water (DI), if not otherwise statement.

## Section 2. Synthesis of $\text{ZnFe}_2\text{O}_4$ and AgI

$\text{ZnFe}_2\text{O}_4$  was prepared following a previously reported method with minor modification<sup>1</sup>. Firstly, Solution A was prepared by dissolving 10 mmol of  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  and 20 mmol of  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  in 15 mL deionized water. Solution B was prepared by ultrasonically dispersing 30 mmol of  $\text{H}_2\text{C}_2\text{O}_4$  in 45 mL EG. The solution B was then poured into solution A and stirred for 10 minutes to form a yellow mixture. Subsequently, the yellow mixture was transferred into a 100 mL Teflon-lined stainless-steel autoclave and heated at 120 °C for 24 h in an electronic oven. Afterward, the solid-liquid mixture was centrifuged and washed with DI and EtOH until clarified. It was dried in a vacuum oven at 60 °C for 10 h, followed by calcination for 30 minutes at 350 °C (10 °C/min) in a muffle furnace to obtain the brown product of  $\text{ZnFe}_2\text{O}_4$ .

AgI was synthesized using a precipitation method<sup>2</sup>. In brief, 15 mL of KI solution (0.2 M) was added dropwise to 30 mL of  $\text{AgNO}_3$  (0.1 M) under magnetic stirring. After continuous stirring for 30 minutes, the precipitation was collected by centrifugation, washed with deionized water and ethanol, and then dried at 70 °C for 10 h.

## Section 3. Characterization

Many characterization measurements are performed to study the material's properties. XRD patterns were obtained on an X-ray diffractometer (Rigaku Dmax/Ultima IV). SEM images were recorded using a field emission scanning electron microscope (Hitachi S4800). TEM

and HRTEM were performed on an FEI Tecnai G2 F20 transmission electron microscope. XPS was collected by using an X-ray photoelectron spectroscopy (Thermo Fisher Nexsa). UV-Visible diffuse reflectance spectroscopy (UV-Vis DRS) was recorded on a Shimadzu UV-3600 spectrophotometer equipped with diffuse reflectance accessories using BaSO<sub>4</sub> as reference.

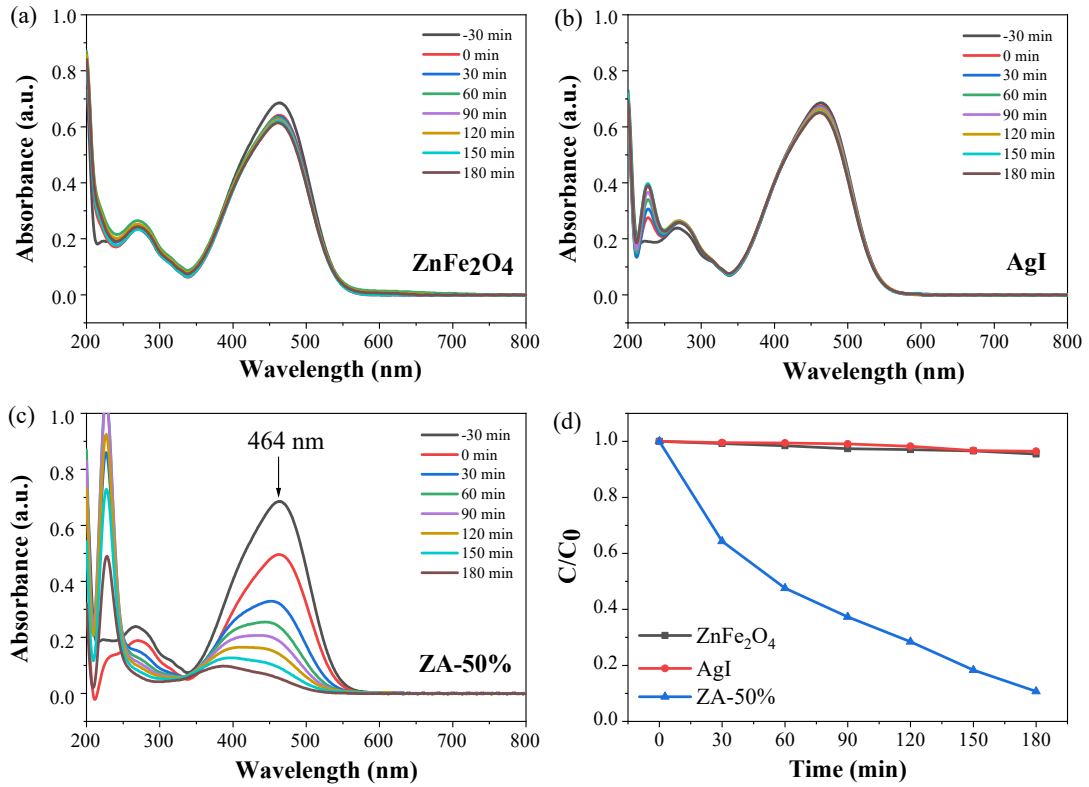


Fig. S1. UV-Vis spectral changes of the MO solution under visible light irradiation ( $\lambda \geq 400$  nm) in the presence of  $\text{ZnFe}_2\text{O}_4$  (a), AgI (b), ZA-50% (c). The kinetic diagram of  $\text{ZnFe}_2\text{O}_4$ , AgI and ZA-50% for MO degradation (d). The concentration of MO solution was 10 mg/L.

As shown in Fig. S1, both  $\text{ZnFe}_2\text{O}_4$  and AgI exhibited limited degradation capacity for MO (Fig. S1a-b). The characteristic peak of MO at 464 nm gradually weakened and shifted to a shorter wavelength with prolonged exposure to visible light in the presence of ZA-50% (Fig. S1c), indicating the degradation of MO<sup>3</sup>. And the degradation efficiency of MO reached 89.3% within 3 hours in ZA-50% (Fig. S1d).

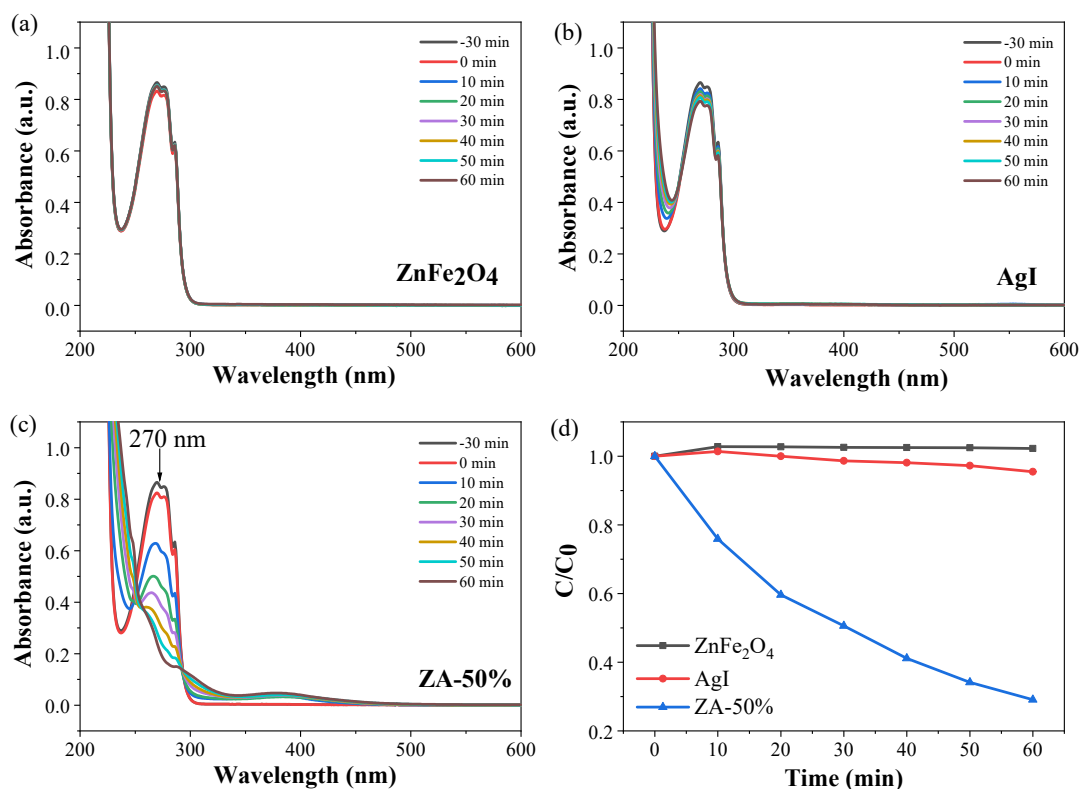


Fig. S2. UV-Vis spectral changes of the indole solution under visible light irradiation ( $\lambda \geq 400$  nm) in the presence of ZnFe<sub>2</sub>O<sub>4</sub> (a), AgI (b), ZA-50% (c). The kinetic diagram of ZnFe<sub>2</sub>O<sub>4</sub>, AgI and ZA-50% for indole degradation (d). The concentration of indole was 20 mg/L.

As shown in Fig. S2, according to the intensity change of the characteristic peak of indole at 270 nm<sup>4</sup>, it was found that the degradation of indole by ZnFe<sub>2</sub>O<sub>4</sub> was negligible (Fig. S2a), the degradation of indole by AgI was not obvious (Fig. S2b). However, the degradation of indole by ZA-50% was apparent (Fig. S2c), suggesting an enhanced photocatalytic activity of the ZnFe<sub>2</sub>O<sub>4</sub>/AgI composite. And the degradation efficiency of indole reached 70.9% within 1 hour in ZA-50% (Fig. S2d).

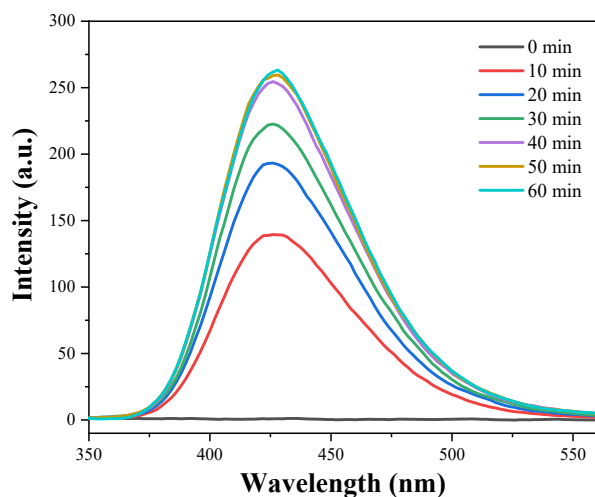


Fig. S3. The fluorescence spectra of terephthalic acid (TA) alkaline solution in the presence of  $\text{TiO}_2$  under UV-Visible light irradiation.

As illustrated in Fig. S3, the fluorescence absorption peak at 425 nm (excitation wavelength at 315 nm) in the TA alkaline solution increased with the irradiation time when  $\text{TiO}_2$  was present. This observation indicates the formation of  $\text{TA-OH}^5$  and the generation of  $\cdot\text{OH}$  radicals in the presence of  $\text{TiO}_2$  and under UV-Visible light irradiation.

## References

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