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Supplementary Information

2 Experimental

2.1 Experimental materials

In this research, all solvents and reactants were of analytical grade and did not necessitate further purification during use. Ultrapure water was utilized for the preparation of all required solutions. Ferric chloride hexahydrate (FeCl₃·6H₂O; AR), and nafion(($C_7HF_{13}O_5S.C_2F_4$)_x) were procured from General-Reagent; 2,4-dinitrophenol (2,4-DNP; GR) was sourced from Xiya Reagent. Ethylene glycol (EG; AR), zinc chloride (ZnCl₂; AR), anhydrous ethanol (AR), *N*,*N*dimethylformamide (DMF; AR), H₂O₂ (AR), hydrochloric acid (HCl; AR), and sodium hydroxide (NaOH; AR) were purchased from Chengdu Kelong Chemicals Co., Ltd. Terephthalic acid (H₂BDC; RG), indium(III) chloride tetrahydrate (InCl₃·4H₂O; RG), thioacetamide (TAA; RG), 2,2,6,6-tetramethylpiperidine (TEMP; AR), and 5,5-dimethyl-1-pyrroline-N-oxide (DMPO; AR) were obtained from Adamas-beta. 2,2,6,6-Tetramethylpiperidine-1-oxyl radical (TEMPO; AR) and nitrobenzene (NB; AR) were sourced from Mackun. *P*-Nitrophenol (PNP; AR) and 3nitrophenol (3-NP, AR) were procured from Aladdin.

2.3 Characterization

The materials were characterized via X-ray powder diffraction (XRD; X'Pert PRO) with Cu K α radiation. Fourier transform infrared spectroscopy (FT-IR; ThermoFisher IS-50) was employed to analyze the structure of the materials. The fine morphology of the materials was investigated using a scanning electron microscope (SEM; ZEISS Sigma 360 and SU8600 Ultimax) and a transmission electron microscope (TEM; JEOL JEM-F200). X-ray photoelectron spectroscopy (XPS; Thermo Scientific ESCALAB 250Xi) was conducted to determine the binding energy of electrons in the materials, analyzing the elemental composition and chemical states, calibrated with C1s at 284.80 eV. Free radical testing was conducted using an electron paramagnetic resonance (EPR; Bruker EMX nano) spectrometer. The specific surface area and pore size distribution of the material were characterized using N₂ adsorption/desorption isotherm measurements at 77 K (BET; ASAP2460). Total orgaine carbon was evaluated using a TOC

analyzer (Japan Shimadu TOC-L CPH). The photocatalytic activity of the catalysts was characterized using an ultraviolet–visible (UV–vis DRS) diffuse reflectance spectrophotometer (Shimadzu solid spec-3700). Absorbance was tested with an UV-visible spectrophotometer (Cary 60, Agilent Technologies, Inc.) to assess residual PNP concentrations.

2.5 Electrochemical measurements

Electrochemical tests were conducted using a CHI660E electrochemical workstation with a three-electrode system. The 0.2 M Na₂SO₄ solution served as the electrolyte, with a Pt plate electrode acting as the counter electrode and an Ag/AgCl electrode serving as the reference electrode. The working electrode was conductive glass coated with the photocatalyst. The procedure for preparing the working electrode involved dissolving 10 mg of the photocatalyst in 40 μ L of ethanol and dispersing it uniformly via ultrasonication. Then, 20 μ L of Nafion solution was appended and the mixture was sonicated for another 30 min. Using a pipette, 30 μ L of this solution was evenly spread onto the conductive surface of the fluorine-doped tin oxide glass (with an effective area of 1.0 cm²) and dried at 60°C. The photocatalyst was then coated onto the working electrode under open-circuit potential and the electrochemical impedance spectroscopy (EIS) curve was recorded. The transient photocurrent (I-t) test was performed using a 300 W Xe lamp with a cutoff filter as the light source, with the control switch interval set to 20 s.

3 Results and discussion

3.4 XPS analysis

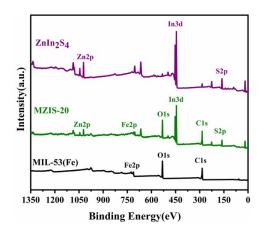


Fig. S1 Full XPS spectra of MIL-53, ZnIn₂S₄, and MZIS-20.

3.6 Photo degradation performance testing

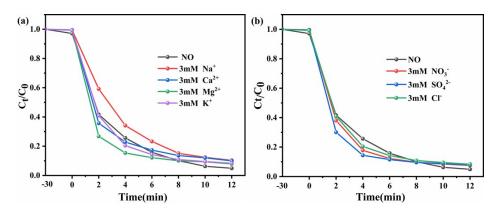


Fig. S2 Plots of the effects of (a) different cations and (b) anions on the degradation efficiency against PNP.