

Supporting Information

One-pot synthesis of monodisperse Zn coordination polymer micro/nanostructures and their transformation to mesoporous ZnO photocatalyst

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Experimental details

Synthesis of Zn-CPs. All chemicals were obtained commercially and used as received without further purification. In a typical procedure, 3,5-pyridinedicarboxylic acid (H_2pydc , 0.1 mmol) and NaOH (0.2 mmol) were dissolved in the mixed solvent of THF (7.5 mL) and deionized water (6.5 mL) and stirred for a few minutes to prepare the solution of Na_2pydc . Then, 1 mL aqueous solution of $\text{Zn}(\text{NO}_3)_2$ (0.1 M) was dropped into above Na_2pydc solution and a white precipitate was formed immediately. After 10 min of stirring, the precipitate was collected by centrifugation, washed with ethanol for three times, and dried at 60 °C for 6 h. The obtained sample was defined as Zn-CP-1.

The synthesis procedures of Zn-CP-2 and Zn-CP-3 are similar to that of Zn-CP-1 except that the Na_2pydc solution was prepared with the mixed solvent of DMSO/ H_2O (7.5 mL/6.5 mL) and 1,4-dioxane/ H_2O (15 mL/8 mL), respectively.

Synthesis of ZnO micro/nanostructures. ZnO samples were obtained by the calcination of Zn-based CPs in air at 500 °C for 1 h with a heating rate of 1 °C min^{-1} . The samples obtained from Zn-CP-1, Zn-CP-2 and Zn-CP-3 were named as ZnO-1, ZnO-2 and ZnO-3, respectively.

Characterization of Zn-CP and ZnO samples. Scanning electron microscope (SEM) images were taken with a JEOL JSM-7500F scanning electron microscope. Transmission electron microscopy (TEM) images were obtained with JEM-2010FEF transmission electron microscope operating at 200 kV. Powder X-ray diffraction (PXRD) was performed on a Rigaku D/max-2500 diffractometer with Cu K α radiation ($\lambda=0.15406$ nm) at 40 kV and 100 mA. Elemental analysis (C, H, and N) was carried out with a Perkin-Elmer 240C analyzer. Thermogravimetric analysis (TGA) was operated on a Rigaku standard TG-DTA analyzer from ambient temperature to 700 °C with a heating rate of 10 °C min^{-1} in the air, and an empty Al_2O_3 crucible was used as the reference. Fourier transform infrared spectroscopy (FT-IR) was measured using a MAGNA-560 Fourier transform infrared spectrometer with nujol mull method. N_2 adsorption-desorption isotherm measurement was operated on a V-Sorb2800P surface area and pore size analyzer.

Photocatalytic degradation experiments. Rhodamine B (RhB) was used as the substrate to characterize the photocatalytic activities of the as-prepared ZnO powders. 40 mg of ZnO sample was dispersed into 80 mL aqueous solution of RhB (5 mg L^{-1}). The suspension was sonicated for 20 min and stirred in dark for another 30 min to ensure the establishment of an adsorption-desorption equilibrium. Then, the suspension was exposed to the irradiation of a 500 W Xenon light source and sampled periodically. The degradation process of RhB was characterized with its absorption at 552 nm (measured by a UV-2450 spectrophotometer).

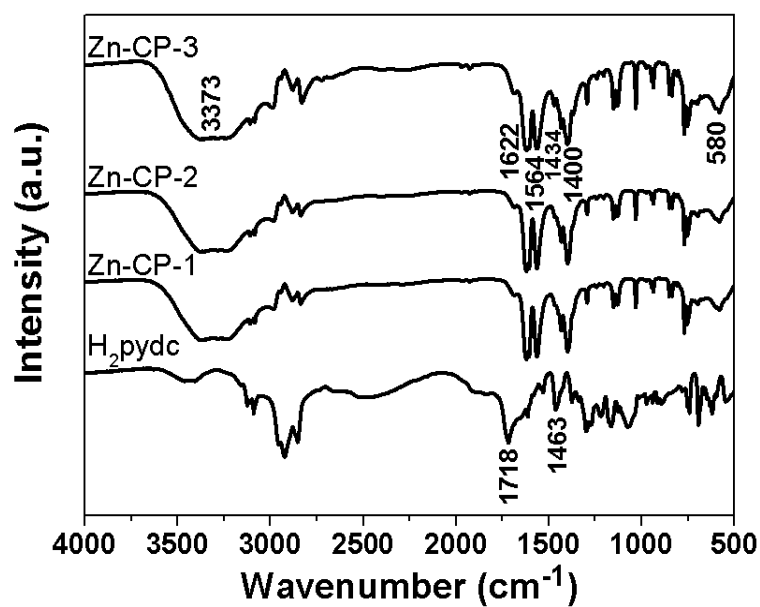


Fig. S1 FT-IR spectra of the as-prepared Zn-CPs and H_2pydc ligand.

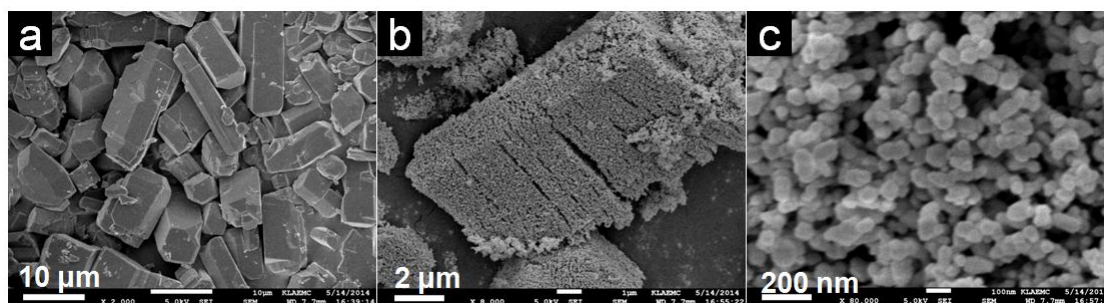


Fig. S2 SEM images of CP bulk crystals (a) and ZnO-bulk (b, c).

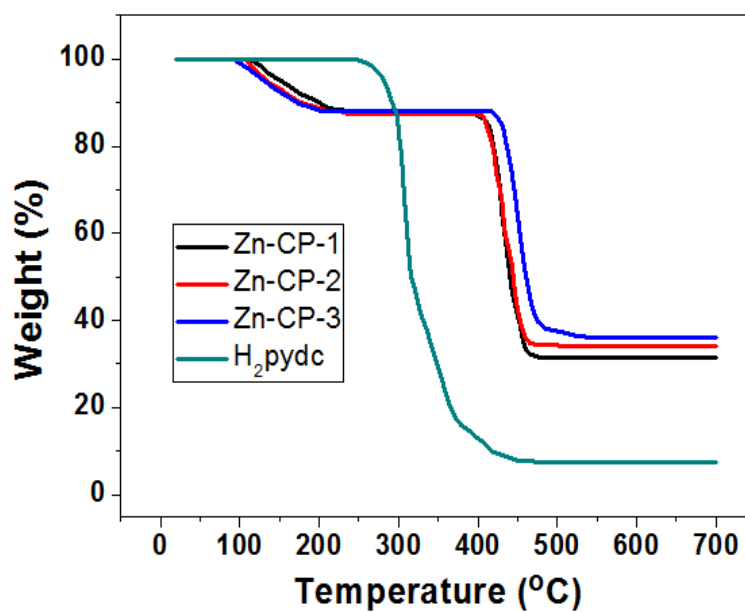


Fig. S3 TGA curves of the as-prepared Zn-CPs and H_2pydc ligand.

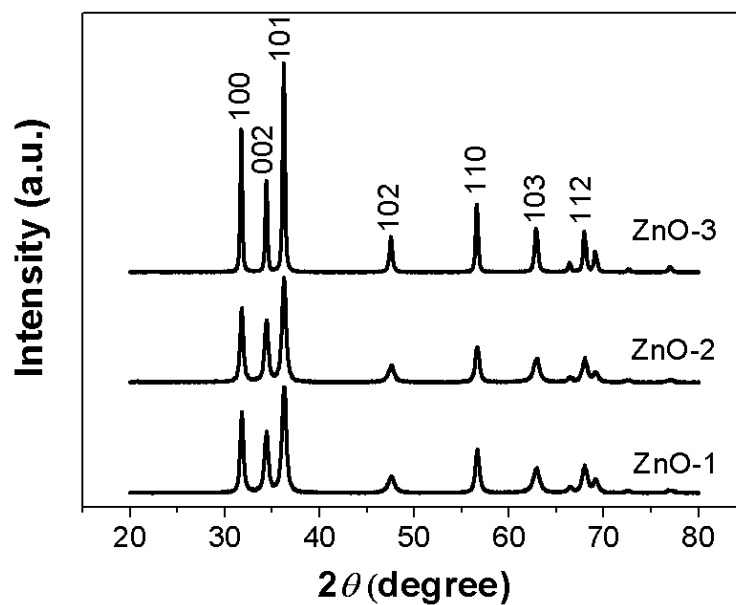


Fig. S4 PXRD patterns of the as-prepared ZnO micro/nanostructures.

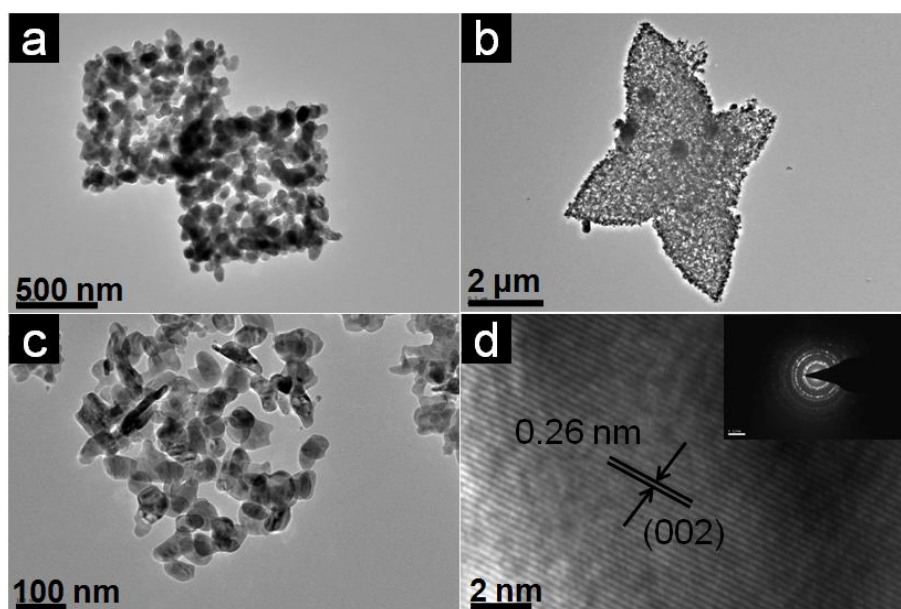


Fig. S5 TEM images of the as-prepared ZnO micro/nanostructures: (a) ZnO-1, (b) ZnO-2, (c) ZnO-3. (d) The typical HRTEM image and SAED pattern (inset) of ZnO-2, the adjacent lattice fringe distance of 0.26 nm corresponds to the (002) facet of wurtzite.

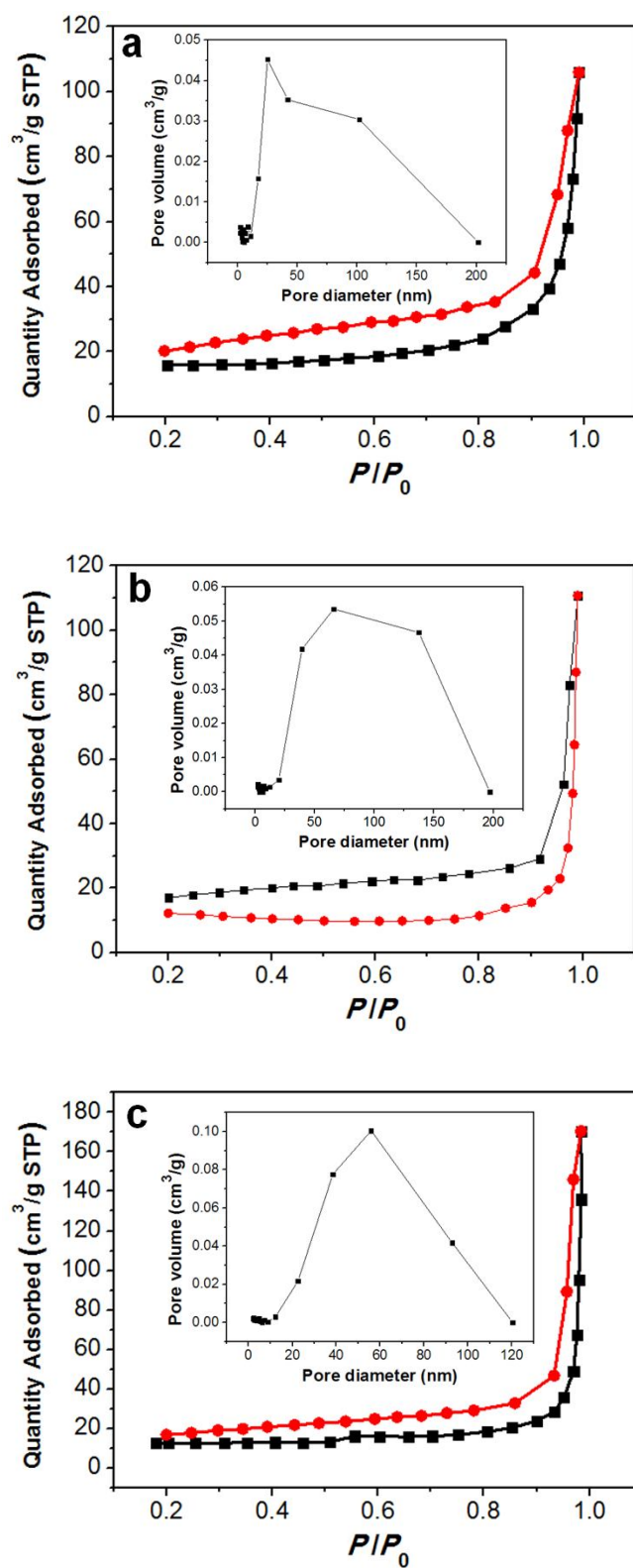


Fig. S6 N_2 adsorption-desorption isotherms of the as-prepared ZnO micro/nanostructures: (a) ZnO-1, (b) ZnO-2, (c) ZnO-3. Inset: the BJH pore-size distribution curves.

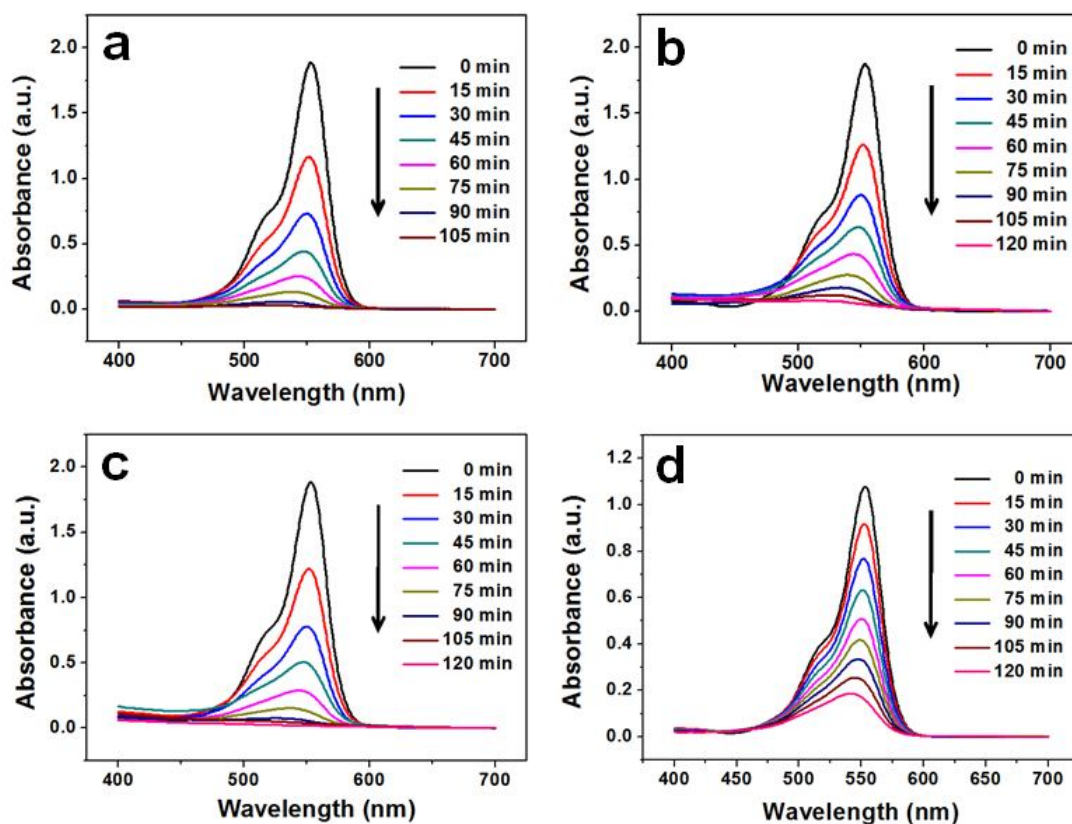


Fig. S7 UV-Vis absorbance spectra of the RhB solution during the degradation process with different ZnO samples: (a) ZnO-1, (b) ZnO-2, (c) ZnO-3, (4) ZnO-bulk.

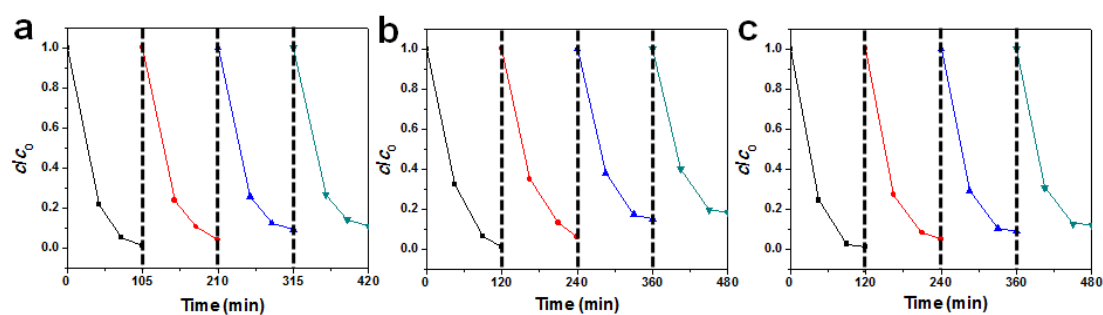


Fig. S8 Cyclic photodegradation of RhB with as-prepared ZnO samples as photocatalysts, 0.5 g L^{-1} ZnO, 5 mg L^{-1} RhB. (a) ZnO-1, (b) ZnO-2, (c) ZnO-3.

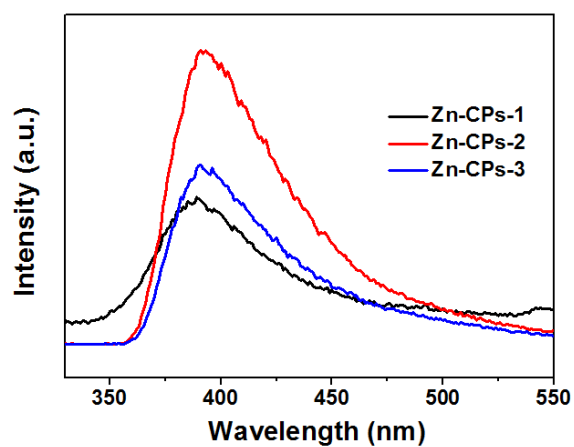


Fig. S9 Solid-state photoluminescent spectra of Zn-CP-1–3, $\lambda_{\text{ex}} = 300$ nm.

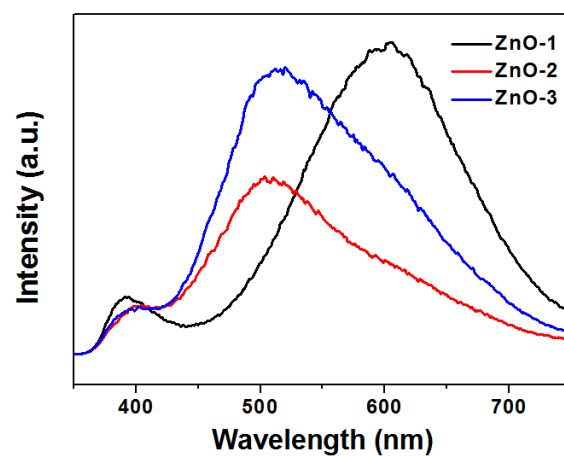


Fig. S10 Solid-state photoluminescent spectra of ZnO-1–3, $\lambda_{\text{ex}} = 320$ nm.

Table S1 EA results of the as-prepared Zn-CPs.

Zn-CPs	C%	H%	N%
Zn-CP-1	31.16	3.05	5.14
Zn-CP-2	31.54	3.13	5.29
Zn-CP-3	31.78	3.39	5.53
Zn(pydc)·2H ₂ O	31.31	3.38	5.22

Table S2 Weight loss percentages for the as-prepared Zn-CPs.

Zn-CPs	Weight (%)	
	25–250 °C	25–700 °C
Zn-CP-1	12	68.7
Zn-CP-2	12.5	65.8
Zn-CP-3	12.2	63.9
Zn(pydc)·2H ₂ O	13.42	69.69