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# **Supporting information**

# Ni or Co single atomic anchored conjugated microporous polymer for highperformance photocatalytic hydrogen evolution

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### **S1. Experimental section**

*S1.1. Chemicals.* 2,2'-Bipyridyl-5,5'-dialdehyde (BPD, 99.0%) was purchased from the Shanghai Kylpharm Co., Ltd. 1,3-Diacetylbenzene (m-DAB, 97.0%), cobaltocene (98.0%) and nickelocene (98.0%) were obtained from Macklin Co., Ltd. Ammonium acetate, triethanolamine (99.5%), pyridine (99.5%), sodium sulphate (99.0%), isopropyl alcohol (IPA, 99.7%), lanthanum oxide (La<sub>2</sub>O<sub>3</sub>, 99.99%) and trichloromethane (TEOA, 98.0%) were purchased from Sinopharm Chemical Reagent Co., Ltd. Chloroplatinic acid hexahydrate (37.5%), acetonitrile, silver nitrate (AgNO<sub>3</sub>, 99%) and Nafion solution (5.0%) were bought from Sigma-Aldrich Company. Tetrabutylammonium hexafluorophosphate (99.0%) was purchased from Adamas Company. All other chemicals were analytic grade and used without further purification. The solvent medium used for the reaction system was deionized water.

*S1.2. Synthesis of PCMP.* Pyridyl-functionalized conjugated microporous polymer (PCMP) was obtained by the Chichibabin reaction of BPD and *m*-DAB according to the modified method reported previously.<sup>1</sup> Firstly, 2 mmol (162.19 mg) of *m*-DAB, 1 mmol (212.2 mg) of BPD, and 30 mmol (2.312 g) of ammonium acetate were added into 50 mL of round bottom flask. Secondly, 35 mL of pyridine was slowly added into the mixture and stirred 24 h under 115°C in an oil bath. Then, the reaction mixture was filtered and as-obtained powder was washed using deionized water and trichloromethane at 60 °C and dried in vacuum oven at 80°C offering PCMP yellow powder with the yield of 80%.

*S1.3. Synthesis of Ni@PCMPs and Co@PCMPs.* A general gaseous diffusion strategy was used to dope PCMP via anchoring Ni and Co single atoms according to the method reported previously.<sup>2</sup> Typically, 40 mg of PCMP and a certain amount of nickelocene (0-50% in mass related to PCMP) were placed on the back and front sides of a corundum boat covered with a lid, respectively. Direct heating the boat in a tube furnace under an Ar flowing for 2 h offered a series of Ni single-atom anchored PCMP photocatalysts (xM@PCMP, where x represents the amount of nickelocene or cobaltene in mass related to 40 mg PCMP, M represents Ni) *i.e.* 10%Ni@PCMP, 20%Ni@PCMP, 30%Ni@PCMP, 40%Ni@PCMP, and 50%Ni@PCMP. The same procedure also provided a series of Co single-atom anchored PCMP photocatalysts i.e. 10%Co@PCMP, 20%Co@PCMP, 30%Co@PCMP, 40%Co@PCMP, and 50%Co@PCMP by replacing the nickelocene by cobaltocene. Furthermore, 30%Fe@PCMP was prepared using ferrocene under the same condition.

## S2. Characterization and measurements

*S2.1. Structure and morphology characterization.* The <sup>13</sup>C CP/MAS NMR spectra were taken at a Bruker Advance 400 (400 MHz) spectrometer. X-ray photoelectron spectroscopy (XPS) analyses were determined by a Thermo Escalab 250 Xi spectrometer and a Prodigy-ICP inductively coupled plasma mass spectrometer. The crystallinity of as prepared samples was characterized by an Arigaku D Max 2550 VB PC X-ray diffraction spectrometer (18 KW, Cu K<sub>a</sub> radiation with  $\lambda$ =1.548 Å). The FT-IR spectra were taken on a Thermo Scientific Nicolet iS5 spectrometer. Ultraviolet visible

diffuse reflectance spectra (UV-vis DRS) were obtained by a UV-3600 diffuse reflectance spectroscopy. Steady photoluminescence and time resolved fluorescence spectra were recorded on a QM/TM fluorometer with the excitation wavelength of 380 and 337 nm, respectively. The scanning electron microscope (SEM) images were obtained on a Hitachi SU8010 microscope. The transmission electron microscope (TEM) images were obtained on a Talos F200S microscope. Aberration-correction high-angle annular dark field scanning transmission electron microscopy (AC HAADF-STEM) images and the corresponding energy dispersive spectroscopy (EDS) were recorded on a FEI Tecnai Osiris and Titan3. The specific surface area, pore volume, and pore size distribution data were obtained on a Micro ASAP 2046 surface area and porosity analyzer.

*S2.2. Cyclic voltammetry measurements.* Cyclic voltammetry measurements were carried out in a three-electrode-cell system: glassy carbon electrode as the working electrode, Ag/AgCl electrode as the reference electrode, and platinum wire as the counter electrode. The samples were prepared by first mixing ground photocatalyst with 5% Nafion, the mixture was then dropped casting on top of a glassy carbon working electrode and let the solvent evaporate in a vacuum oven for 60 min. The measurements were carried out in a 0.1 M of tetrabutylammonium hexafluorophosphate solution as supporting electrolyte in acetonitrile with a scan rate of 50 mV s<sup>-1</sup> in the range of -1.5 to 0 V. For the conversion from Ag/AgCl redox couple to the normal hydrogen electrode (NHE), the equation  $E_{NHE} = E_{SCE} - 0.22$  V was applied.<sup>3</sup>

*S2.3. Photocurrent measurements.* The photocurrent measurements were performed on an Interface 1000E (GAMRY) electrochemical workstation, and the standard three-electrode system includes a platinum plate as the counter electrode, an Ag/AgCl electrode as the reference electrode, and a glassy carbon electrode as the working electrode. The working electrode was prepared as follows: 4 mg of each photocatalyst was thoroughly mixed with 1 mL of methanol containing 80  $\mu$ L of 5% Nafion, and the 2  $\mu$ L resulting suspension was carefully loaded on a glassy carbon electrode with an active area of 0.07 cm<sup>2</sup> and dried at 60 °C under vacuum for 60 min. Here, 0.5 M sodium sulphate aqueous solution was acted as the electrolyte. A constant potential of 1.5 V was applied with the 60 s light on-off after a certain time interval to record the photo and dark current under a 300W Xe lamp ( $\lambda$ >420 nm) irradiation.<sup>4</sup>

*S2.4 Electrochemical impedance measurements.* The electrochemical impedance measurements were performed on an Interface 1000E (GAMRY) electrochemical workstation, and the standard three-electrode system includes a platinum plate as the counter electrode, an Ag/AgCl electrode as the reference electrode, and a glassy carbon electrode as the working electrode. Typically, 5 mg catalysts, 100  $\mu$ L isopropyl alcohol (IPA), 100  $\mu$ L Nafion and 395  $\mu$ L deionized water were mixed by sonicating for at least 30 min to form a homogeneous ink, which were then dip-coated onto the glassy carbon electrode and natural drying in air. Before testing, pure nitrogen gas was used to purge the 0.1 M Na<sub>2</sub>SO<sub>4</sub> electrolyte for 30 min to keep the solution without air. The working electrodes were firstly activated for several times until the signals were stabilized and the electrochemical impedance measurements were performed at open-circuit voltage with AC amplitude of 5 mV in

the frequencies range of 0.01 Hz to 10<sup>5</sup> Hz.<sup>5</sup>

*S2.5. Photocatalytic hydrogen evolution measurements.* Typically, 10 mg photocatalysts were loaded with 3.0 wt% Pt dispersed (chloroplatinic acid hexahydrate as precursor) into a 50 mL TEOA solution (with volume concentration of 10%) via ultrasonic treatment for 30 min under sealed environment. After that, the mixture was transferred into the closed gas circulation system (Perfect Light Company Labsolar-6A). Prior to the photocatalytic test, the system was purged with Ar flow to remove air. A 300W Xe lamp (Perfect Light PLS-SXE 300) was used as the light source. A cutoff filter (Kenko L-42) was used to achieve a light irradiation ( $\lambda$ >420 nm). The amount of H<sub>2</sub> evolved was determined using gas chromatography (SHIMADZU GC-2014, thermal conductivity detector (TCD), Ar carrier, Agilent). Cycling photocatalytic stability tests were performed using the same way after the samples were collected and washed with deionized water.

*S2.6. Photocatalytic oxygen evolution measurements.* Typically, 10 mg photocatalyst suspended in 50 mL water with 0.01 M AgNO<sub>3</sub> as the sacrificial electron acceptors and La<sub>2</sub>O<sub>3</sub> (0.1 g) as a pH buffer agent was subjected to photoirradiation (Xe lamp, Perfect Light PLS-SXE 300, using  $\lambda$ >420 nm cutoff filters). The suspension was sonicated for 30 min for a better dispersion then purged with argon gas for at least 120 min to expel the dissolved oxygen. The amount of oxygen evolved was determined using gas chromatography (SHIMADZU GC-2014, thermal conductivity detector (TCD), Ar carrier, Agilent).

*S2.7. The apparent quantum efficiency (AQE) measurements.* AQE measurements for hydrogen evolution were performed under the illumination of a 300 W Xe lamp with 420 nm band pass filters ( $\lambda_0 \pm 20$  nm) for 5 hours. An optical diode power meter (FZ-A spectroradiometer) was used to measure its photon flux with an intensity of 3 mW cm<sup>-1</sup>. For these tests, 10 mg of photocatalyst was used. The apparent quantum efficiency was calculated using the following formula:

$$AQE = \frac{N_e}{N_p} \times 100\% = \frac{2 \times M \times N_A}{\frac{E_{total}}{E_{Photon}}} \times 100\% = \frac{2 \times M \times N_A}{\frac{S \times p \times t}{h \times \frac{c}{\lambda}}} \times 100\% = \frac{2 \times M \times N_A \times h \times c}{S \times P \times t \times \lambda} \times 100\%$$

Where,  $N_e$  is the amount of generated electrons,  $N_p$  is the incident photons, M is the amount of Hydrogen molecules (mol),  $N_A$  is Avogadro constant (6.022×10<sup>23</sup> mol<sup>-1</sup>), h is the Planck constant (6.626×10<sup>-34</sup> J·s), c is the speed of light (3×10<sup>8</sup> m s<sup>-1</sup>), S is the irradiation area (cm<sup>2</sup>), P is the intensity of irradiation light (W cm<sup>-2</sup>), t is the photoreaction time (s),  $\lambda$  is the wavelength of the monochromatic light (m).

### **S3.** Supplementary figures and tables



Fig. S1 A proposed mechanism for the synthesis of PCMP.



**Fig. S2** (a) FT-IR and (b) solid-state <sup>13</sup>C CP/MAS NMR spectra of PCMP. The FT-IR spectra of BPD and m-DAB monomers were provided for comparison.



Fig. S3 (a) N<sub>2</sub> adsorption/desorption isotherms and (b) corresponding DFT pore size distributions

of PCMP, 30%Ni@PCMP and 30%Co@PCMP.



Fig. S4 (a) Full survey, (b) C1s, (c) N1s, and (d) Ni2p core-level XPS spectra of Ni@PCMPs.



Fig. S5 (a) Full survey, (b) C1s, (c) N1s, and (d) Co2p core-level XPS spectra of Co@PCMPs.



Fig. S6 (a,c) FT-IR spectra and (b,d) powder XRD patterns of Ni@PCMPs and Co@PCMPs.



Fig. S7 (a) SEM and (b) TEM images of PCMP.



**Fig. S8** (a) SEM, (b) TEM, (c) medium and (d) low-resolution AC-HAADF-STEM, (e) element mapping, and (f) high-resolution AC-HAADF-STEM images of 30%Ni@PCMP.



**Fig. S9** (a,c) UV-vis di□use reflectance spectra and (b,d) Tauc plots of Ni@PCMPs and Co@PCMPs. Insets show the estimated band gap values determined by Tauc plots.



Fig. S10 CV curves of PCMP and Ni@PCMPs.



Fig. S11 CV curves of PCMP and Co@PCMPs.



**Fig. S12** (a,c) Time courses of hydrogen production and (b,d) Hydrogen evolution rate from water splitting by PCMP, Ni@PCMPs and Co@PCMPs.



**Fig. S13** (a) Time courses of hydrogen production and (b) Hydrogen evolution rate from water splitting by PCMP, 30%Ni@PCMPs and 30%Co@PCMPs without Pt.



**Fig S14** Hydrogen evolution rate from water splitting by PCMP, 30%Ni@PCMP and 30%Co@PCMP with or without Pt.



**Fig. S15** (a) Time courses of oxygen production and (b) oxygen evolution rate from water splitting by PCMP, 30%Ni@PCMPs and 30%Co@PCMPs.



Fig. S16 Photoluminescence emission spectra of (a) Ni@PCMPs and (b) Co@PCMPs.



Fig. S17 Nyquist plots of PCMP, 30%Ni@PCMP and 30%Co@PCMP.



**Fig. S18** SEM images of (a-c) PCMP, 30%Ni@PCMP and 30%Co@PCMP; (d-f) PCMP, 30%Ni@PCMP and 30%Co@PCMP SEM images of upon 25 hour cycling.



Fig. S19 (a) PCMP, (b) 30%Ni@PCMP and (c) 30%Co@PCMP XRD spectra of upon 25 hour cycling.



Fig. S20 (a-c) Full survey and (d-f) C1s core-level XPS spectra of PCMP, 30%Ni@PCMP and 30%Co@PCMP upon 25 hours cycling.



Fig. S21 N1s Core-level XPS spectra of PCMP upon 25 hours cycling.



Fig. S22 (a) SEM, (b) TEM and (c) element mapping of 30%Fe@PCMP.



**Fig. S23** (a) Time courses of hydrogen production and (b) hydrogen evolution rate from water splitting by PCMP, 30%Fe@PCMPs.



Fig. S24 Schematic diagram of xM@PCMP under light irradiation.

Sample	Advantages	Disadvantages	References
PCMPs	Metal-free synthetic approach	Low hydrogen evolution activity	S1
CTFs	High-throughput	Pd-mediated polycondensation	S6
PCPes	Controllable the optical band gap	Pd-catalyzed and low hydrogen evolution rate	S7
Py-X-CMPs	High hydrogen evolution activity	Pd-mediated polycondensation	S8
DPP-bdy-TPA	Wide photo action spectrum	Pd-mediated polycondensation	S9
DPCN-Cu-x	Metal-free catalyzed approach	Harsh reaction condition (anhydrous and oxygen-free environment)	S10
PFTBDD-IrTPy	Enhance hydrogen evolution activity	Pd-catalyzed and high temperature (400°C)	S11
PDBTSO@TiO <sub>2</sub>	High hydrogen evolution activity	Pd-catalyzed and harsh reaction condition (vigorous stirring in an argon atmosphere and 48h)	S12
P1	Dual-defect synergistically	Low hydrogen evolution activity and high temperature (550°C)	S13
Fe@g-C <sub>3</sub> N <sub>4</sub>	Enhanced solar-photon-driven activities	High temperature (550°C) and two-step process	S14
Pd/g-CN	Increase active sites for photocatalysis	Noble metal and long time (20 h)	S15
30%Co@PCMP	Metal-free synthesis conducted to prepare organic photocatalysts; mediate conditions are applied to prepare single atomic catalysts Good hydrogen evolution is obtained	Due to the sublimation temperature, the available metal precursors are relatively limited	This work

 Table S1 The comparisons of photocatalytic hydrogen production systems.

Ni (wt%)	Sample	Co (wt%)
2.2	10%Co@PCMP	0.06
2.95	20%Co@PCMP	0.11
2.97	30%Co@PCMP	0.14
3.79	40%Co@PCMP	0.16
4.26	50%Co@PCMP	0.32
	Ni (wt%) 2.2 2.95 2.97 3.79 4.26	Ni (wt%)         Sample           2.2         10%Co@PCMP           2.95         20%Co@PCMP           2.97         30%Co@PCMP           3.79         40%Co@PCMP           4.26         50%Co@PCMP

Table S2 The Ni and Co contents of Ni@PCMPs and Co@PCMPs determined by ICP-MS

Table S3 Band gaps ( $E_g$ ), band positions ( $E_{CB}$  and  $E_{VB}$ ) and photocatalytic hydrogen rates of Ni@PCMPs

Sample	E <sub>g</sub> (eV)	E <sub>CB</sub> (eV)	E <sub>VB</sub> (eV)	Hydrogen evolution rate (mmol g <sup>-1</sup> h <sup>-1</sup> )
РСМР	2.63	-0.93	1.7	0.83
10%Ni@PCMP	2.59	-0.93	1.66	0.86
20%Ni@PCMP	2.55	-0.94	1.61	0.94
30%Ni@PCMP	2.53	-0.95	1.58	0.97
40%Ni@PCMP	2.54	-0.91	1.63	0.91
50%Ni@PCMP	2.07	-0.59	1.48	0.36

Table S4 Band gaps ( $E_g$ ), band positions ( $E_{CB}$  and  $E_{VB}$ ), and hydrogen evolution rates of Co@PCMPs

Sample	Eg	E <sub>CB</sub>	E <sub>VB</sub>	Hydrogen evolution rate
	(eV)	(eV)	(eV)	$(mmol g^{-1} h^{-1})$
РСМР	2.63	-0.93	1.7	0.83
10%Co@PCMP	2.59	-0.92	1.67	1.07
20%Co@PCMP	2.57	-0.98	1.59	1.46
30%Co@PCMP	2.54	-1.0	1.54	1.72
40%Co@PCMP	2.56	-0.99	1.57	1.67
50%Co@PCMP	2.61	-0.97	1.64	1.54

Polymer	Optical gap (eV)	H <sub>2</sub> evolution rate $\lambda$ >420 nm	Reference
		$(mmol g^{-1} h^{-1})$	
РСМР	2.63	0.83	
30%Ni@PCMP	2.53	0.97	This work
30%Co@PCMP	2.54	1.72	
PCMP-1	2.83	0.1	S1
PCMP-2	2.96	0.02	
CTF-11	2.15	1.15	
CTF-12	2.41	1.51	
CTF-13	2.57	0.072	
CTF-14	1.55	0.016	<b>S</b> 6
CTF-15	2.58	2.94	
CTF-16	2.6	1.35	
CTF-17	2.42	0.53	
CTF-18	2.71	0.005	
DBTD-CMP1	2.53	4.6	S16
CTP1	2.98	0.15	
CTP2	2.66	0.5	S17
CTP3	2.36	0.05	
IEP-7	2.0	0.12	
IEP-8	1.5	0.26	S18
IEP-9	1.8	0.043	
IEP-10	1.8	0.16	
L-PyBT	2.23	1.67	S19
L-PyDFBT	2.28	0.42	
aza-CMP/RGO/C2N		0.45	S20
OB-POP-3	2.14	0.9	S21
CTF-N	2.17	0.54	S22
PCP1e	1.95	0.014	
PCP2e	2.45	0.15	S7 (Pd content 0.11-
PCP3e	2.19	0.079	0.67 wt%)
PCP4e	2.06	0.19	
P1-2	2.7	3.16	
P1-8	2.77	0.97	
P1-11	2.55	1.38	
P1-13	2.43	0.76	
P1-14	2.56	8.23	
P1-16	2.59	3.76	S23 (Pd content 0.81-
P1-20	2.25	1.49	1.6 wt%)

**Table S5** Optical properties and photocatalytic hydrogen evolution properties of the polymeric or

 metal-modified polymeric photocatalysts (Pt as the co-catalyst)

P1-34	2.61	1.53	
P1-39	2.56	4.24	
P1-51	2.5	1.47	
P1-60	2.65	7.65	
P1-71	2.33	1.54	
P1-94	2.71	1.83	
Cd-TBAPy	2.15	0.086	S24
Pt@MIL-125/Au	3.02	0.38	S25
Na <sub>0.02</sub> -C <sub>3</sub> N <sub>4</sub> /Pt@UiO-	2.88	0.47	S26
66			
Zn-ZnDETPP	1.5	0.27	
Co-CoDETPP	1.65	1.1	S27
Zn-CoDETPP	1.6	1.36	
Zn-CNN	2.74	1.5	S28
Fe@g-C <sub>3</sub> N <sub>4</sub> -0.5		1.93	
Fe@g-C <sub>3</sub> N <sub>4</sub> -1	2.61	3.39	S14
$Fe@g-C_3N_4-2$		1.45	

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