# **Electronic Supporting Information(ESI)**

# Side-Chain-Tuned $\pi$ -Extended Porous Polymers for Visible Light-Activated Hydrogen Evolution

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#### **1. Experimental Section**

#### **General methods**

Liquid nuclear magnetic resonance (NMR) spectra were measured on Mercury plus 400 (400 MHz for proton, 100 MHz for carbon) spectrometer with tetramethylsilane as the internal reference using CDCl<sub>3</sub> as solvent in all cases. Solidstate <sup>13</sup>C NMR experiments were performed on a Bruker AVANCE 800 spectrometer operating at 201.2 MHz for <sup>13</sup>C using a doubleresonance 3.2 mm MAS NMR probe and a sample spinning rate of 18 kHz. Ramped <sup>1</sup>H-<sup>13</sup>C cross polarization (CP) and SPINAL proton decoupling during signal acquisition was applied. The chemical shifts were referenced with adamantane. Fourier transform infrared (FT-IR) spectra were recorded with a Spectrum 100 spectrometer (Perkin Elmer, Spectrum 100). UV-Vis DRS absorption spectra were recorded at room temperature on a HITACHI U-4100 Spectrophotometer. Fluorescence spectroscopy (PL) spectra were obtained with a FluoroMax-4 spectrophotometer. Cyclic voltammetry (CV) was performed on a Chenhua 650D electrochemical analyzer in anhydrous CH<sub>3</sub>CN containing recrystallized tetra-n-butyl-ammonium hexafluorophosphate (TBAPF6, 0.1M) as supporting electrolyte at 298 K. A conventional three electrode cell was used with a glassy carbon working electrode (surface area of 0.3 mm<sup>2</sup>) and a platinum wire as the counter electrode. The glassy carbon working electrode was routinely polished with a polishing alumina suspension and rinsed with acetone before use. The measured potentials were recorded with respect to Ag/AgCl reference electrode. The samples were prepared by first mixing 2 mg polymer with 2 mL isopropanol and 100  $\mu$ L 5 wt% Nafion, the mixture was ultrasonicated for 1h, then dropped cast on top of a glassy carbon working electrode and let the solvent evaporate in an oven at 80 °C for 30 min. All figures determined from cyclic voltammetry were checked three times to get average value. All electrochemical measurements were carried out under an atmospheric pressure of nitrogen. Thermal gravimetric analysis (TGA) were performed on a US TGA/Pyris 1 TGA thermogravimetric analyzer to investigate the thermal stability of all samples from ambient temperature to 900 °C at the rate of 20 °C/min in nitrogen atmosphere. X-ray powder diffraction patterns (PXRD) were recorded in transmission geometry using a RigakuD/Max 2500 X-ray diffractometer with Cu-Ka irradiation ( $\lambda = 0.15406$  nm) at 40 kV, 50 mA over the 2 $\theta$  range from 5 to 80° with a scanning speed of 6° min<sup>-1</sup>. The gas sorption isotherms were measured via an Autosorb-iQA3200-4 sorption analyzer (Quantatech Co., USA) based on N<sub>2</sub> adsorption/desorption. Samples were degassed at 150 °C for 12 h under vacuum before

analysis. Elemental analysis was carried out using elemental analyzer on a Vario-EL Cube. Scanning electron microscope (SEM) measurements were performed on a FEI Sirion-200 field emission scanning electron microscope. Transmission electron microscope (TEM) characterizations were conducted using a JEM-2100 (JEOL Ltd., Japan) with an accelerating voltage of 200 kV. Electron paramagnetic resonance (EPR) characterizations were performed on an EMX-8 (Bruker BioSpin Corp., Germany) with a 1-meter distance between the samples and the light source, irradiation time were 3 min.

#### Synthesis Procedure of the Monomers and Polymers:



Scheme S1 Synthetic route for the monomers.

**Dangerous Caution:** NaCN is highly toxic to human body, the users should use it in the cupboard with masks, gloves and goggles.

**2,4,6-Tris(4-formylphenyl)-1,3,5-triazine (TFPT):**<sup>1</sup> In a 250 mL flask, 2,4,6-Tris(4bromophenyl)-1,3,5-triazine (1.48 g, 2.71 mmol) was dissolved in dry THF (150 mL) under N<sub>2</sub> atmosphere. To the stirred solution, n-BuLi was added dropwise (2.5 M in nhexane, 11 mL, 27.5 mmol) at -78 °C for a 2-h reaction. The obtained green solution was treated with anhydrous *N*, *N*-dimethylformamide (DMF) (4.19 mL, 54.2 mmol) at -78 °C. The temperature of the mixture was allowed to resume to 25 °C, and stirred overnight. The milky opaque mixture was acidified with aqueous 3 M HCl (46 mL), petroleum ether (PE: 50 mL) was added to the mixture. After filtered, the white solid was collected as colorless micro0crystals in a yield: 74%. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 10.19 (s, 3H), 8.95 (d, 6H), 8.11 (d, 6H).

**1,4-Bis(chloromethyl)-2,5-dimethoxybenzene:**<sup>2</sup> In a 200-mL Schlenk flask, 1,4dimethoxy-benzene (6.90 g, 0.05 mol), paraformaldehyde (4.50 g, 0.15mol), concentrated HCl (20 mL) and anhydrous ZnCl<sub>2</sub> (0.90 g, 6.60 mmol) were added to 1,4-dioxane (50 mL). The system was heated to 65 °C for a 6-h reaction. After cooling down to the room temperature, the white solution was poured to ice water and the white precipitates were collected and washed with ethanol, dried, to get white solid product in a yield: 81.7%. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 3.86 (s, 6H), 4.64 (s, 4H), 6.92 (s, 2H).

**2,5-Bis(cyanomethyl)-1,4-dimethoxybenzene:**<sup>3</sup> 1,4-Bis(chloromethyl)-2,5dimethoxybenzene (11.70 g, 0.05 mol) and NaCN (4.90 g, 0.10 mol) were added to a 200 mL Schlenk flask and purged with nitrogen gas. Anhydrous DMSO (100 mL) was added to the flask under nitrogen atmosphere and the system was heated to 40 °C, and reacted for three days. The solution was cooled and poured into ice water and then filtered. The solid was washed with water, ethanol and dried, and purified on a silica gel column using PE: DCM=1:1 to afford a white solid in a yield: 79.6%. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 3.88 (s, 6H), 3.73 (s, 4H), 6.96 (s, 2H). <sup>13</sup>C NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 150.65, 119.06, 117.77, 111.86, 56.15, 18.65.

**1,4-bis(chloromethyl)-2,5-dimethyl benzene:**<sup>4</sup> *p*-xylene (11.10 mL; 90.00 mmol) and concentrated HCl (60 mL) were taken in to a 200 ml three necked round bottom flask and it was equipped with a reflux condenser. Then paraformaldehyde (10.80 g, 360.0 mmol), zinc chloride (4.00 g, 30.0 mmol) were added in to the flask and the system was heated to 100 °C under continuously stirring for 36 h. The resulting white precipitate was collected, washed with ethanol and dried over night to afford a white solid in a yield of 55.8%. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 2.38 (s, 6H), 4.56 (s, 4H), 7.15 (s, 2H).

**2,5-Dimethyl-1,4-bis(cyanomethyl)benzene:**<sup>3</sup> 1,4-bis(chloromethyl)-2,5-dimethyl benzene (10.05 g, 0.05 mol) and NaCN (5.39g , 0.11 mol) were added to a 200 mL Schlenk flask and purged with nitrogen gas. Anhydrous DMSO (100 mL) was added to the flask under nitrogen atmosphere and the system was heated to 40 °C for three days. The solution was cooled and poured into ice water and then filtered. The solid was washed with water, ethanol and dried. The solid was purified on a silica gel column using PE: DCM=1:1as eluent, to afford a white solid in a yield of 82.6%.<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 2.32 (s, 6H), 3.63 (s, 4H), 7.21 (s, 2H). <sup>13</sup>C NMR (400 MHz,

CDCl<sub>3</sub>):  $\delta$  (ppm): 134.60, 131.03, 128.75, 117.54, 21.65, 18.95.

#### Procedure approach to TFPT-OCH<sub>3</sub> upon Knoevenagel condensation reaction.

To a 25 mL long-necked Pyrex tube, were added 2,4,6-tris(4-formylphenyl)-1,3,5triazine (TFPT) (50 mg, 0.127 mmol), 2,5-Bis(cyanomethyl)-1,4-dimethoxybenzene (41.2 mg, 0.191 mmol), DMAc/EtOH (v/v= 2.5 mL/2.5 mL) and  $Cs_2CO_3$  (0.5mL, 1 M). The mixture was degassed by freeze–pump–thaw cycles for three times, and sealed under vacuum, then heated to 120 °C, reacted for 3 days. Afterward, the mixture was cooled to room temperature and the precipitate was collected by filteration, washed with acetone, DCM, THF and dilute hydrochloric acid. The resulting solid was dried at 80 °C under vacuum for 12 hours to afford orange red powder in a yield of 92%.

### Procedure for TFPT-PDAN using Knoevenagel condensation reaction.

In a 25 mL long-necked Pyrex tube, 2,4,6-tris(4-formylphenyl)-1,3,5-triazine (TFPT) (50.00 mg, 0.13 mmol), 1,4-phenylenediacetonitrile (29.80 mg, 0.19 mmol), DMAc/EtOH (v/v= 2.5 mL/2.5 mL) and Cs<sub>2</sub>CO<sub>3</sub>(0.5mL, 1 M) were added. The mixture was degassed by freeze–pump–thaw cycles three times and sealed under vacuum, then heated to 120 °C for a 3-day reaction. Afterward, the mixture was cooled to room temperature and the precipitate was filtered off, washed with anhydrous acetone, DCM, THF and dilute hydrochloric acid. The product was dried at 80 °C under vacuum for 12 hours to afford green powder in a yield: 82%.

#### **Procedure for TFPT-CH<sub>3</sub> using Knoevenagel condensation reaction.**

In a 25 mL long-necked Pyrex tube, 2,4,6-tris(4-formylphenyl)-1,3,5-triazine (TFPT) (0.050.g, 0.13 mmol), 2,5-Dimethyl-1,4-bis(cyanomethyl)benzene (0.35.g, 0.19 mmol), DMAc/EtOH (v/v= 2.5 mL/2.5 mL) and Cs<sub>2</sub>CO<sub>3</sub>(0.5mL, 1 M) were added. The mixture was degassed by freeze–pump–thaw cycles three times and sealed under vacuum, then heated to 120 °C, reacted for 3 days. After the reaction, the mixture was cooled to room temperature and the precipitate was filtered off, washed with anhydrous acetone, DCM, THF and dilute hydrochloric acid. After that, the product was dried at 80 °C under vacuum for 12 hours to afford dark yellow powder in a yield of 74%.

#### Hydrogen evolution test of the polymers.

A flask was charged with the polymer powder (50 mg), deionized water (80 mL), triethanolamine (TEOA) (20 mL), and sealed with a septum. The resulting mixture was ultrasonicated 30 min to obtain a well-dispersed photocatalyst suspension. Then the resulting suspension was transfer into a Pyrex top-irradiation reaction vessel connected to a closed gas system. 3 wt. % noble metals (Pt) as cocatalyst were loaded on the surface of the polymer catalyst by in situ photo-deposition method using H<sub>2</sub>PtCl<sub>6</sub>. The reaction mixture was evacuated 30min to insure complete removal of air prior to irradiation in a 90° angle with a 300 W Xe light-source. The temperature of the reaction solution was maintained at 5°C by the flow of cooling water during the reaction. The evolved gases were analyzed by gas chromatograph equipped with a 5 Å molecular sieve column at 40 °C with an argon flow of 25.0 mL min<sup>-1</sup>. Hydrogen was detected with a thermal conductivity detector (TCD) referencing against standard gas with a known concentration of hydrogen. Hydrogen dissolved in the reaction mixture was not measured and the pressure increase generated by the evolved hydrogen was neglected in the calculations. The rates were determined from a linear regression fit and the error is given as the standard deviation of the amount of hydrogen evolved.

#### The AQY measurement and Wavelength Experiment.

The apparent quantum yield (AQY) for H<sub>2</sub> evolution was measured using monochromatic LED lamps with band pass filter of  $405 \pm 4.6$  nm,  $420 \pm 4.4$  nm,  $450 \pm 4.0$  nm,  $490 \pm 4.1$  nm,  $520 \pm 4.4$  nm,  $578 \pm 4.1$  nm (errors of wavelength were depended on Guass Simulation of full width at half maximum). The irradiation area was controlled as  $3\times3$  cm<sup>2</sup>. Depending on the amount of hydrogen produced by the photocatalytic reaction in an average of one hour, and the AQY was calculated as follow:

$$\eta_{AQY} = \frac{N_{e}}{N_{p}} \times 100\% = \frac{2 \times M \times N_{A}}{\frac{E_{total}}{E_{photon}}} \times 100\% = \frac{2M \times N_{A}}{\frac{S \times P \times t}{h \times \frac{C}{\lambda}}} \times 100\% = \frac{2 \times M \times N_{A} \times h \times C}{S \times P \times t \times \lambda} \times 100\%$$

Where, M is the amount of H<sub>2</sub> molecules (mol), NA is Avogadro constant ( $6.022 \times 10^{23}$ 

mol<sup>-1</sup>), h is the Planck constant ( $6.626 \times 10^{-34} \text{ J} \cdot \text{s}$ ), c is the speed of light ( $3 \times 108 \text{ m s}^{-1}$ ), 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).

	TFPT-CH <sub>3</sub>		TFPT-	PDAN	<b>ТГРТ-ОСН</b> <sub>3</sub>			
	Theory	Test	Theory	Test	Theory	Test		
	(wt %)	(wt %)	(wt %)	(wt %)	(wt %)	(wt %)		
С	81.80	80.82	81.61	80.14	77.76	76.25		
Н	4.58	6.24	4.11	4.89	4.35	5.89		
N	13.63	12.81	14.28	14.97	12.95	12.12		
0	0	0	0	0	4.93	5.74		

## 2. Elemental analysis (EA)

Table S1 Elemental analysis of the polymers.

#### 3. Fourier transform infrared spectroscopy (FT-IR)



**Figure S1.** Transmission FT-IR spectra of the polymers with corresponding monomers as KBr pellets.



**Figure S2.** Transmission FT-IR spectra of the polymers before and after the hydrogen evolution process.

#### 4. Powder X-Ray diffraction (PXRD)



Figure S3. Powder X-ray diffraction spectra from 5-80°.

5. Nitrogen sorption analysis



**Figure S4.** BET plot (P/P<sub>0</sub> = 0.06–0.2) from N<sub>2</sub> isotherms at 77 K of (a) TFPT-CH<sub>3</sub>, (b) TFPT-PDAN, (c) TFPT-OCH<sub>3</sub>.



**Figure S5.** Nitrogen sorption isotherms for as-synthesized and after-photocatalytic porous polymers; (a) TFPT-CH<sub>3</sub>, (b) TFPT-PDAN, (c) TFPT-OCH<sub>3</sub>.



**Figure S6.** Pore size distributions of the polymers using non-local DFT modelling; (a) TFPT-CH<sub>3</sub>, (b) TFPT-PDAN, (c) TFPT-OCH<sub>3</sub>.

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Sample	$\begin{array}{c} S_{BET}{}^{[a]} \\ (m^2g^{\text{-}1}) \end{array}$	$\begin{array}{l} S_{Lang}^{[b]} \\ (m^2g^{\text{-}1}) \end{array}$	D <sub>av</sub> [c] (nm)	$\begin{array}{l} V_{Total}^{[d]} \\ (cm^3g^{\text{-}1}) \end{array}$
TFPT-CH <sub>3</sub>	275.4	555.9	9.90	0.682
TFPT-PDAN	588.0	1028.8	10.32	1.52
TTPT-OCH <sub>3</sub>	570.5	1033.8	5.98	0.853

 Table S2 Nitrogen physisorption data of as-synthesized polymers.

[a] Surface areas calculated from N<sub>2</sub> adsorption isotherm using BET (S<sub>BET</sub>) and [b] Langmuir (S<sub>Lang</sub>, ) theory. [c] Average pore size based on the adsorption isotherm. [d] Total pore volume at  $P/P_0 = 0.99$ .

6. DRS UV-Vis absorption spectra and photoluminescence spectra



**Figure S7.** The UV/vis diffuse reflectance spectra (DRS) (solid line) and photoluminescence spectra (dashed line) of the polymers (a) TFPT-CH<sub>3</sub>; (b) TFPT-PDAN; (c) TFPT-OCH<sub>3</sub>.



**Figure S8.** UV/vis diffuse reflectance spectra (DRS) of the polymers as synthesized and after experiment. (a) TFPT-CH<sub>3</sub>; (b) TFPT-PDAN; (c) TFPT-OCH<sub>3</sub>.

# 7. Kubelka-Munk-transformed reflectance spectra



**Figure S9.** Band gaps of the polymers determined from the Kubelka-Munk-transformed reflectance spectra of (a) TFPT-CH<sub>3</sub>; (b) TFPT-PDAN; (c) TFPT-OCH<sub>3</sub>.

#### 8. XPS spectra of the valance band



**Figure S10.** XPS spectra of the valance band for (a) TFPT-CH<sub>3</sub>; (b) TFPT-PDAN; (c) TFPT-OCH<sub>3</sub>.

9. Scanning electron microscope (SEM) spectroscopy



Figure S11. SEM images of (a) TFPT-CH<sub>3</sub>; (b) TFPT-PDAN; (c) TFPT-OCH<sub>3</sub>.



**Figure S12.** SEM images of TFPT-OCH<sub>3</sub> as synthesized and after experiment. After 16 hours under  $\lambda > 420$  nm irradiation showed no significant change in SEM spectroscopy.

# 10. Transmission electron microscope (TEM) spectroscopy



**Figure S13.** TEM images of the polymers: (a) TFPT-CH<sub>3</sub>; (b) TFPT-PDAN; (c) TFPT-OCH<sub>3</sub>.



**Figure S14.** TEM images of TFPT-OCH<sub>3</sub> as synthesized and after experiment with the Pt cocatalyst nanoparticle.

## 11. Electron paramagnetic resonance (EPR)



**Figure S15.** EPR analysis of the polymers. (a) TFPPy-OCH<sub>3</sub>; (b) TFPPy-CH<sub>3</sub>; (c) TFPT-OCH<sub>3</sub>; (d) TFPT-CH<sub>3</sub>.

# 12. Time-resolved transient photoluminescence (PL) decay



**Figure S16.** Time-resolved transient photoluminescence (PL) decay of the polymers. (a) TFPT-CH<sub>3</sub>; (b) TFPT-PDAN; (c) TFPT-OCH<sub>3</sub>

#### 13. The water wetting properties



Figure S17. A water droplet on (a) TFPT-CH<sub>3</sub>: (WCA=71.57 $\pm$ 0.13°); (b) TFPT-PDAN: (WCA=63.44  $\pm$  0.24°); (c) TFPT-OCH<sub>3</sub>: (WCA=49.21  $\pm$  0.33°).



Figure S18. Digital photographs of the as-prepared polymers and their dispersion in water

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after ultrasonicated for 30 min.



**Figure S19.** Periodic on/off photocurrent response for the polymers under visible light irradiation: (a) TFPT-CH<sub>3</sub>; (b) TFPT-PDAN; (c) TFPT-OCH<sub>3</sub>.

**Table S3** The apparent quantum yield (AQY) of the TFPT-OCH<sub>3</sub> sample, using different wavelength monochromatic LED lamps

λ/nm	405	420	450	490	520	578
AQY/%	1.031	0.980	0.877	0.617	0.232	0.050

Reaction condition: photocatalyst (50 mg); triethanolamine (10 mL), water (90 mL);  $H_2PtCl_6$  was added into the reaction solution to photo-deposite 3wt.% Pt nanoparticle on the surface of TFPT-OCH<sub>3</sub> sample.

# 15. NMR spectra of key monomer





Figure S22. <sup>1</sup>H NMR spectra of 2,5-bis(cyanomethyl)-1,4-dimethoxybenzene.



**Figure S23.** <sup>13</sup>C NMR  $\Box$  spectra of 2,5-bis(cyanomethyl)-1,4-dimethoxybenzene.



Figure S24. <sup>1</sup>H NMR spectra of 2,5-dimethyl-1,4-bis(cyanomethyl)benzene.



**Figure S25.** <sup>13</sup>C NMR spectra of 2,5-dimethyl-1,4-bis(cyanomethyl)benzene.

# **16.** Photocatalytic H<sub>2</sub> evolution activities of as-prepared organic photocatalysts

Table S4 Photocatalytic performance of the previously reported conjugatedorganic porous polymers for comparison.

Entry	Samples	Cocatalyst	λ/	AQY /	Ref.
	1	5	nm	%	
1	SP-CMP	Pd residue	420	0.23	Chem. Commun. <b>2016</b> , 52,
					10008.
2	N <sub>3</sub> -COF	Pt	450	0.44	Nat. Commun. 2015, 6, 8508.
3	CP-CMP-	Pd residue	420	0.56	J. Am. Chem. Soc. 2015, 137,
	10				3265.
4	PyBT-2	Pt	420	0.90	App. Catal. B: Environ. 2018,
					239, 46.
5	COP-TP <sub>3:1</sub>	Pt	400	1.5	ACS App. Mater. Interfaces.
					<b>2018</b> , 10, 30698.
6	CTF-2	Pt	420	1.6	<i>Polymer</i> <b>2017</b> , 126, 283.
7	PCP <sub>4e</sub>	Pt	350	1.8	J. Am. Chem. Soc. <b>2016</b> , 138,
					7681
8	OB-POP-3	Pt	420	2.0	Adv. Funct. Mater. 2017, 27,

					1703146
9	P7	Pd residue	420	2.3	Angew. Chem. Int. Ed. <b>2016</b> , 55, 1792
10	TFPT- OCH <sub>3</sub>	Pt	405	1.03	This work

## 17. Reference

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