Supporting Information

Ferrocene-based Porous Organic Polymers Derived High-performance Electrocatalysts for Oxygen Reduction

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Section 1. Materials and Methods

¹H NMR spectra of the monomers were recorded using an Avance Bruker DPX 400 (400 MHz) in solution. N₂ adsorption/desorption measurements were conducted at 77 K using Micromeritics ASAP 2020 surface area and porosimetry analyzer. All these samples were degassed for 12 h at 150°C under high vacuum before test. Thermal gravimetric analysis (TGA) datum were obtained using NETZSCH STA 449C analyzer at the temperature ranged from 30°C to 800°C with a heating rate of 10°C/min under the protection of N₂. FT-IR spectrum was recorded on KBr pellets in the range of 4000 to 400cm⁻¹ using Perkin-Elmer Spectroscopy. The element content was tested using ICP to and element analysis. Inductively Coupled Plasma OES spectrometer (ICP) was conducted on Ultima 2 produced in HORIBA Jobin Yvon. Elemental analyses (C, H, and N) were performed on an Elementar Vario MICRO elemental analyzer. Solid-state ¹H-¹³C CP/MAS NMR measurements were performed on a Bruker SB Avance III 500 MHz spectrometer. Powder X-ray diffraction (PXRD) patterns were collected on a Rigaku-DMAX 2500 diffractometer at a scanning rate of 5° min⁻¹ from 10° to 80°. Scanning electron microscopy (SEM) experiments were carried out on a JSM 6700 at 10.0 kV. Transmission electron microscopies (TEM) were performed on a FEI model Tecani 20 microscope and a JEOL model JSM-2100F. The TEM samples were prepared by dropcasting a sonicated methanol suspension of POPs onto a copper grid. The specific surface areas for N2 were calculated using the BET model over a relative pressure (P/P_0) range of 0.05-0.15. Total pore volumes were calculated from the uptake at a relative pressure of 0.995. Pore size distributions were calculated from the adsorption isotherms via the nonlocal density functional theory (NLDFT) method.

Section 2. Synthetic Procedures

DMSO, methanol, N,N-dimethylformamide (DMF) and tetrahydrofuran (THF) were used directly from commercial source without further purification. Dichloromethane (DCM) was used after distillation over calcium hydride under Argon atmosphere. Ferrocene, melamine and phosphorus oxychlorideare are purchased from the commercial supplier and used directly without further purification.

The detailed synthetic methods including the monomers and polymers are given as follows:

Preparation of melem^{S1}



Melamine (1, 25 g) was added into a crucible, and then transfered into a muffle furnace. The system was heated to 250 °C in a heating rate of 5 °C min⁻¹. Violent gas (ammonia) liberation began at 250 °C and continued for 9 h. Then, it was heated to 385 °C with a heating rate of 5 °C/min and continued for 15 h. Thereafter the reaction was allowed to cool to room temperature. Pure melem was obtained as a yellow-wish powder. Yield: 17.0 g, 96%; IR (KBr): 3487, 3425, 1620, 1485, 1310, 802 cm⁻¹.

Preparation of Ferrocenecarboxaldehyde (Fc-CHO)⁸²



To a 250 mL three-neck flask, a mixture of ferrocene (5.3 g, 28 mmol) and chloroform (60 mL) was placed. After degassing, the reaction system was kept at 5 to 10 °C under the protection of Ar. Afterward, DMF (15 mL) was added

into the reaction system dropwise, following by the injection of phosphorus oxychloride (10.5 mL) for 1.5 h. The resulting reaction mixture was refluxed for 12 h. After removal of solvent, the residue was poured into 100 mL ice water and filtered. The filtrate was neutralized to pH 8~9 using NaOH (10%, w/v) and then extracted with ether. The organic layer was washed with water and dried over anhydrous MgSO₄. After evaporation to dryness, the afforded red solid was further purified via column chromatography to give compound **3** (1.75 g) as a red solid in ca. 60% yield. ¹ H NMR (CDCl₃): δ 9.95 (s, 1H, - CHO), 4.80 (d, 2H, Cp-rings), 4.61 (d, 2H, Cp-rings), 4.28 (s, 5H, Cp-rings). FT-IR (KBr): v (cm⁻¹) 1679(C=O).

Synthesis of Fc-melamine-HCP S3,S4



Ferrocene-carboxaldehyde **3** (1.226 g, 6 mmol), melamine **1** (504 mg, 4 mmol) were added into a 100 mL two neck round bottom flask that equipped with a stirrer and a condenser. The system was degassed and backfill with argon for several times. DMSO (20 mL) was added and the reaction mixture was degassed again. Under the protection of argon, the reaction mixture was heated at 180 °C for 40 h. During this process, black gel was formed. Finally, the reaction mixture was cooled to room temperature. The solid was isolated by filtration and washed successively with methanol, DMF, and THF. The resultant solid was dried in a vacuum oven at 120 °C overnight to afford a dark blown powder (1.60 g, 99% yield). Element analysis and ICP analysis: Fe 6.91%, N 29.91%, C 37.06%, H 4.53%.

Synthesis of Fc-melem-HCP S3,S4



Ferrocene-carboxaldehyde (1.226 g, 6 mmol), melem (872 mg, 4 mmol) were added into a 100 mL reaction tube. After the injection of DMSO (20 mL), the reaction mixture was heated to 180 °C for 40 h. Black gel was formed during the polymerization process. Then the resulted black powder was filtrated and washed, dried in a vacuum oven at 120 °C overnight to afford a black powder (1.79 g) in a yield of 90%. Element analysis and ICP analysis: Fe 7.75%, N 30.00%, C 36.57%, H 3.25%.

Preparation of heteroatom-doped N-Fc-X catalysts



These series of catalyst denoted as N-Fc-700, N-Fc-800 and N-Fc-900 were prepared via the direct carbonization of as-synthesized Fc-melamine-POP at different temperatures (700 °C, 800 °C and 900 °C) with a heat rate of 5 °C/min under the protection of argon, and it kept at the target temperature for 2 h.

Electrochemical Measurements^{S5-S8}

All electrochemical measurements were performed in a standard threecompartment cell at the room temperature. The counter electrode was a platinum wire and the reference electrode was an Ag/AgCl electrode saturated with KCl solution. All potentials were referenced to the reversible hydrogen electrode (RHE) scale according to the Nernst equation ($E_{(RHE)} = E_{(Ag/AgCl)} +$ 0.059 × pH + 0.197 V, at 25 °C). The working electrodes, a rotating disc electrode (RDE) composed of a glassy carbon disc (5.0 mm diameter) and a rotating ring disc electrode (RRDE) composed by a glassy carbon disc (4 mm diameter) surrounded by an outer Pt-ring (5 mm inner-diameter and 7 mm outer-diameter), served as substrates for the loading of the catalyst ink. The catalyst ink and commercial Pt/C (20 wt%) was prepared by suspending 5 mg catalyst with 50 µL Naffion solution (5 wt%), 150 µL ethanol and 300 µl H₂O in an ultrasonic bath. A loading of 10 µL (6 µL for Pt/C) catalyst ink was pipetted onto the glassy carbon surface of the RDE/RRDE and leave to dry in air at room temperature.

The catalytic activities for the oxygen reduction reactions (ORR) were evaluated by CV, rotating ring-disk electrode (RRDE) and rotating disk electrode (RDE) with a three-electrode cell system on a CHI-760 electrochemical workstation (CH Instruments). In alkaline conditions, the RDE and RRDE tests were carried out in an O₂-saturated 0.1 M KOH solution. The scan rate was 50 mV s⁻¹ for CV measurements and 5 mV s⁻¹ for the RDE/RRDE tests from - 0.8 V to + 0.2 V at different rotation speeds from 400 to 2500 rpm. For comparison, commercial Pt/C (20 wt%) was also measured under the identical conditions. Koutecky-Levich plots were analyzed at various electrode potentials. To further investigate ORR activity at a RDE, the slopes of their best linear fit lines were used to calculate the electrons transformation number according to the basis of the K-L equations $(1)^{86}$ - $(3)^{87}$:

$$\frac{1}{J} = \frac{1}{J_L} + \frac{1}{J_K} = \frac{1}{Bw^{1/2}} + \frac{1}{J_K}$$
(1)

In this equation, j_L is the current that is measured; j_K is the kinetic-limiting current and ω is the rotating speeds of electrode.

$$B = 0.62 n F C_0 (D_0)^{2/3} V^{1/6}$$
⁽²⁾

In this equation, *n* is the whole number of transferred electrons during the ORR process; *F* is the Faradaic constant (96485 C/mol), C_0 is the oxygen concentration (solubility) in 0.1 M KOH (1.2×10^{-6} mol cm⁻³); D₀ is the oxygen diffusion coefficient in 0.1 M KOH (1.90×10^{-5} cm² s⁻¹) and *V* is the kinematic viscosity of the 0.1 M KOH (0.01 cm² s⁻¹).

For the measurements of RRDE, the electrodes were prepared in a same method as the same as the RDE. The disk electrode was scanned at a rate of 5 mV s⁻¹, with a constant ring potential at 1.5 V vs RHE. The yield of H_2O_2 and the number of electron transfer (n) toward the ORR process were determined by the followed equations^{S8}:

$$H_2 O_2 \% = 200 \times \frac{I_r / N}{I_d + I_r / N}$$

$$\times \frac{I_d}{I_d + \frac{I_r}{N}}$$

$$N = 4 \qquad (4)$$

In these equations, I_d stand for the disk current, and I_r represents the ring current, and N is current collection efficiency of the Pt ring which is 0.4424 from the reduction of K₃Fe[CN]₆.

The catalytic activities for the oxygen evolution reactions (OER) were evaluated by LSV, glassy carbon electrode (glassy carbon disc: 3 mm diameter) with a threeelectrode cell system on a CHI-760 electrochemical workstation (CH Instruments). In alkaline conditions, the LSV tests were carried out in an O_2 -saturated 1 M KOH solution with a scan rate of 5 mV s⁻¹ from 0 V to + 0.8 V. For comparison, commercial Pt/C (20 wt%) was also measured under the identical conditions.

Zinc-air battery measurement

To prepare Zn-air battery, the N-Fc-800 powder was ultrasonically dispersed in a 1 mL mixed solution (The Volume ratio of deionized-water, 0.05 wt% Nafion and ethanol was 6 : 1 : 3) for 30 min to form a concentration of 10 mg/mL catalyst ink. Then coated onto carbon paper and dried with a lamp. The loading density was about 1 mg cm⁻². A piece of Zinc plate was used as anode electrode and 6 M KOH was used as electrolyte. The Zn-air batteries were tested at room temperature. The polarization curves were obtained by LSV technique with CHI-760E electrochemical working station. The galvanostatic discharge and charge cycling (5 min discharge and 5 min charge with the current density of 10 mA cm⁻²) were performed in LAND testing system.

Section 3. ¹H-NMR Spectra



Figure S1 ¹H NMR spectrum of monomer ferrocene-carboxaldehyde (Fc-CHO).





Figure S2. a) FT-IR spectra of reaction monomers; b) FT-IR spectra Fc based porous polymers and Fc-CHO; c) ¹H-¹³C solid-state NMR spectra of Fc-melamine-POP and Fc-melem-POP.

Section 5. PXRD Profiles



Figure S3. PXRD profiles of Fc-melamine-POP, Fc-melem-POP and Fc-melamine-POP derived catalysts. a) PXRD of Fc-melamine-POP and Fc-melem-POP; b) PXRD of N-Fc-700 (red), N-Fc-800 (blue), N-Fc-900 (violet) and N-Fc-800 maintained under air for 5 months (black).

Section 6. SEM, TEM Images & EDX Analysis



Figure S4. SEM images of prepared catalysts. a-c) N-Fc-700; d-f) N-Fc-800; g-i) N-Fc-900.



Figure S5. SEM images and element-mapping of prepared catalysts a) SEM images of N-Fc-700; b-d) C, N and Fe mapping of N-Fc-700; e) SEM images of N-Fc-800; f-g) C, N and Fe mapping of N-Fc-800; i) SEM images of N-Fc-900; j-l) C, N and Fe mapping of N-Fc-900.



Figure S6. a-e) High-resolution TEM images of N-Fc-700; f) SAED patterns of the N-Fc-700.



Figure S7. a-e) High-resolution TEM images of N-Fc-800; f) SAED patterns of the N-Fc-800.

Figure S8. a-e) High-resolution TEM images of N-Fc-900; f) SAED patterns of the N-Fc-900.

Section 7. Thermogravimetric Analysis (TGA)

Figure S9. TGA curves of Fc-based POPs: a) TGA curves of Fc-melamine-POP; b) TGA curves of Fc-melem-POP.

To better understand the evolution mechanism from the amorphous porous polymers into the crystalline N-Fe/Fe₃C@C, and study the thermal stability, thermal gravity analysis (TGA) was carried out on the Fc-melamine to investigate the thermal properties (Figure S6). The main weight loss occurs over three different temperature ranges. In the initial range of 30-100°C, a drastic weight loss could be observed clearly which was ascribed to the existence of water absorbed in the N-rich porous networks. Another weight loss happened at the temperature from 150-250 °C which could be attributed to the repolymerization of the as-prepared polymers and the remaining part of DMSO. The last weight loss process occurred in the range from 400 to 700 °C corresponding with the decomposition of the N-rich polymer during which released the nitrogen containing gas for carbide reaction and growth of graphite layer. No significant weight loss was observed at temperatures above 700 °C.

Section 8. X-ray Photoelectron Spectra (XPS)

Figure S10. The XPS survey and high-resolution N 1s, C 1s, Fe 2p and Fe 2p XPS spectrum of N-Fc-700 a) XPS survey for N-Fc-700 b) C 1s N 1s spectrum of N-Fc-700; c) O 1s spectrum of N-Fc-700; d) N 1s spectrum of N-Fc-700; e) Fe 2p spectrum of N-Fc-700.

Figure S11. The XPS survey and high-resolution N 1s, C 1s, Fe 2p and Fe 2p XPS spectrum of N-Fc-800 a) XPS survey for N-Fc-800 b) C 1s and N 1s spectrum of N-Fc-800; c) O 1s spectrum of N-Fc-800; d) N 1s spectrum of N-Fc-700 e) Fe 2p spectrum of N-Fc-800.

Figure S12. The XPS survey and high-resolution N 1s, C 1s, Fe 2p and Fe 2p XPS spectra of N-Fc-900 a) XPS survey for N-Fc-900 b) C 1s, N 1s spectra of N-Fc-900; c) O 1s spectrum of N-Fc-900; d) N 1s spectrum of N-Fc-700 e) Fe 2p spectrum of N-Fc-900.

Section 9. BET and Poresize Distribution of N-Fc-700, N-Fc-800 and N-Fc-900

Figure S13. Nitrogen adsorption/desorption isotherm at 77K and the pore size distribution curve of prepared catalysts. a, d) Nitrogen adsorption/desorption isotherm and the pore size distribution curve of N-Fc-700; b, e) Nitrogen adsorption/desorption isotherm and the pore size distribution curve N-Fc-800; c, f) Nitrogen adsorption/desorption isotherm and the pore size distribution curve N-Fc-900.

4 6 Pore Width (nm) 4 6 Pore Width (nm)

4 Pore 6 dth (nm)

Section 10. Electrochemical Performance

Figure S14. Electrochemical performance of the prepared catalysts. a) CV curves of N-Fc-700 on glassy carbon electrodes in water solution of 0.1 M KOH saturated with O_2 with a sweep rate of 10 mVs⁻¹; b) LSV curves of N-Fc-700 in water solution of 0.1 M KOH saturated with O_2 at different rotation speeds; c) K-L plots for N-Fc-700 at various potential; d) CV curves of N-Fc-800 on glassy carbon electrodes in water solution of 0.1 M KOH saturated with O_2 with a sweep rate of 10 mVs⁻¹; e) LSV curves of N-Fc-800 in water solution of 0.1 M KOH saturated with O_2 with a sweep rate of 10 mVs⁻¹; e) LSV curves of N-Fc-800 in water solution of 0.1 M KOH saturated with O_2 at different rotation speeds; f) K-L plots for N-Fc-800 at various potential; g) CV curves of N-Fc-900 on glassy carbon electrodes in water solution of 0.1 M KOH saturated with O_2 with a sweep rate of 10 mVs⁻¹; h) LSV curves of N-Fc-900 in water solution of 0.1 M KOH saturated with O_2 with a sweep rate of 10 mVs⁻¹; h) LSV curves of N-Fc-900 in water solution of 0.1 M KOH saturated with O_2 at different rotation speeds; j) K-L plots for N-Fc-900 at various potential; j) CV curves of the N-doped Fe/C catalysts on glassy carbon electrodes in water solution of 0.1 M KOH saturated with O_2 with a sweep rate of 10 mVs⁻¹; k) RRDE voltammograms recorded with the prepared catalysts in O_2 -saturated 0.1 M KOH at 1600 rpm.

Figure S15. a) Polarization curves of N-Fc-800 for ORR measured by RDE in O_2 -saturated 0.1 M KOH at a rotating rate of 1600 rpm before (black line) and after (red line) the i-t tests; b) Polarization curves of N-Fc-800 for ORR measured by RDE in O_2 -saturated 0.1M KOH at a rotating rate of 1600 rpm before (black line) and after (red line) the addition of 3 M CH₃OH (black line).

Section 11. EDS of the Prepared Catalysts

Figure S16. EDS of N-Fc-700.

Figure S17. EDS of N-Fc-800.

Figure S18. EDS of N-Fc-900.

Fig. S19 OER performance of the N-Fc-X catalysts together with commercial Pt/C (20%) tested in O_2 -saturated 1.0 M KOH solution with a scan rate of 5 mV s⁻¹.

Section 12. Supporting Tables

Sample	S _{BET} (m ² /g)	V _{Total} (cm ³ /g)	Main pore size (nm)	
Fc-melamine	149	0.38	0.84/1.38/2.37	
Fc-melem	207	0.39	0.7/2.46/1.45	
N-Fc-700	419	0.42	1.409/0.70/2.45	
N-Fc-800	511	0.44	0.30/0.9/1.4/2.34	
N-Fc-900	433	0.43	0.28/0.7/1.3/2.2	

Table S1. Porosity Parameters of prepared porous polymers and the corresponding catalysts.

Table S2. The surface element contents of of different species including carbon, oxygen, nitrogen and iron in as-synthesized catalysts, calculated from the survey spectra data of Fig. S3.

Sample	Fe (%)	C (%)	Total N (%)	Pyridinic-N (% in total N)	Pyrrolic-N (% in total N)	Graphitic-N (% in total N)	Oxidized N (% in total N)
N-Fc-700	6.26	63.34	4.48	31.0	27.7	40.6	0.57
N-Fc-800	6.91	66.16	3.46	37.5	39.8	22.5	0.72
N-Fc-900	8.08	66.61	3.32	20.6	12.2	66.3	0.83

Table S3. Main parameters of the prepared catalysts combined with the commercial Pt/C catalysts

Sample	On-set Potential (Eon-set)	Half-wave potential (E _{1/2})	Current density (mA cm ⁻² ; at 0.5V)	Electron transfer number (n)
N-Fc-700	0.92 V	0.75 V	3.06	3.35
N-Fc-800	0.96V	0.82 V	5.27	3.94
N-Fc-900	0.94 V	0.80 V	4.62	3.60
Pt/C	0.99 V	0.84 V	4.76	3.99

Table S4. The comparison of the electrochemical performance toward ORR among different
POPs derived catalysts the date in the alkyl (The date given are in the basic conditions)

Sample	Monomers	Reactions	Catalysts	solvent	Onset potential (V vs RHE)	Half wave potential (V vs RHE)
N-Fc-800	Ferrocene melamine	Schiff base	Free	DMSO (analytic grade)	0.96	0.82
CoP-CMP- 800 ⁵⁹	Co-Porphyrin	Yamamoto polycondensation	Ni(COD) ₂ and 2,2'- bipyridyl	dry dioxane	0.84	0.78
Pt- CTF/CP ^{S10}	Pt and 2,6- dicyanopyridi ne	CN-tri- polymerization	molten ZnCl ₂	ZnCl ₂		
MWNT- CoP ^{S11}	MWNT and Alkynyl-Co- Porphyrin	Hay-coupling	TMEDA and copper(I) chloride	NMP free of oxygen		

C-COP-P- M(M=Fe,C o,Mn) ^{S7}	5,10,15,20- tetrakis(4'- bromo biphenyl) porphine-M	Yamamoto polycondensation	Ni(COD) ₂ and 2,2- bipyridyl	bis(1,5- cyclooctadie ne) dehydrate DMF	0.98	0.80
TTF-700 ⁵¹²	terephthalonit rile	CN-tri- polymerization (quartz ampoule)	molten ZnCl ₂	molten ZnCl ₂	0.82	0.73
TTF-F ^{S12}	Ttf-700, ammonium fluoride	CN-tri- polymerization(qu artz ampoule)	molten ZnCl ₂	molten ZnCl ₂	0.86	0.77
TTF-B ^{S12}	Ttf-700, boron oxide	CN-tri- polymerization(quartz ampoule)	molten ZnCl ₂	molten ZnCl ₂	0.83	0.76
PCN- FeCo/C ^{S13}	Co-Porphyrin, Fe-Porphyrin	Sonogashira reaction	Pd ₂ (dba) ₃ , AsPh ₃	Dry THF and Et ₃ N, oxygen-free	1.00	0.85
N-PAF- Carbon- 800 ⁸¹⁴	Tetrakis(4- bromophenyl) methane ,NH ₃	Yamamoto condensation	bis(1,5- cyclooctadie ne) nickel(0), 2,2- bipyridyl	dehydrated DMF	0.81	0.75
2DPPV- 800 ⁸¹⁵	p- phenylenediac etonitrile, 1,3,5-tris(4- formylphenyl) benzene	Knoevenagel polycondensation	cesium carbonate	1,2- dichloroben zene Pyrex tube	0.85	

Section 13. Supporting References

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