## **Electronic Supplementary Information**

# Facile synthesis of polyacrylonitrile-based N/S-codoped porous carbon

### as an efficient oxygen reduction electrocatalyst for znic-air battery

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### **Reagents and Materials:**

Acrylonitrile (AN) was analytical grade and commercially available from Sinopharm Chemical Reagent Co. Ltd (China) and distilled prior to use. 2-Mercaptoethanol (2-ME) purchased from Sigma Aldrich without any further purification. was Azobisisobutyronitrile (AIBN, Aladdin Reagents) was recrystallized in ethanol twice. Ferric chloride (FeCl<sub>3</sub>, ACS grade), zinc chloride (ZnCl<sub>3</sub>, ACS grade), HClO<sub>4</sub> (70%, ACS grade), KOH (ACS grade) and N,N-Dimethylformamide (DMF, ACS grade) were purchased from Aladdin. Commercial Pt/C catalyst (20 wt %, Pt) was obtained from Alfa Aesar. Nafion (5 wt %) was purchased from Sigma Aldrich. Ethanol and HCl (37%) were purchased from Sinopharm Chemical Reagent Co. Ltd (China). Ultra-pure water with a resistivity > 18 M $\Omega$  cm<sup>-1</sup> was used in all the experiments.

#### **First-principles calculations**

The calculation was performed on density functional theory (DFT) with Vienna ab initio package (VASP).<sup>1</sup> The exchange-correlation interactions was describe by general gradient approximation of Perdew-Burke-Ernzerhof (GGA-PBE).<sup>2</sup> The energy cutoff of plane wave functions were set to 500 eV. A 15 Å vacuum layers were used. The reciprocal space was sampled using a  $3 \times 3 \times 1$  point grid by Monkhorst-Pack K-points scheme. The DFT-D3 method was used to calculate van der Waals (vdW) interaction.<sup>3</sup> The structures were relaxed until the residual force on each atom is less than 0.01 eV Å<sup>-1</sup>. In the calculations, a  $10 \times 10 \times 1$  graphene supercell was used. The ORR is complete  $4e^{-}$  process, here the changes of free energy were calculated to reflect the ORR activity. For each one electron transfer step, the free energy change ( $\Delta G$ ) can be expressed as:

 $\Delta G = \Delta E + \Delta Z P E - T \Delta S + e U$ 

The  $\Delta E$ ,  $\Delta ZPE$ ,  $T\Delta S$ , e, and U represent the energy changes, zero-point energy correction, entropic energy, the elementary charge and the potential used during the ORR, respectively. The  $\Delta E$  and  $\Delta ZPE$  values are calculated by first principle calculations and the T $\Delta S$  can be obtained in standard thermodynamic data. The ORR process in this work include four steps:

- (1)  $O_2(g) + H_2O(l) + e^- + * \rightarrow OH^- + OOH^*$
- (2)  $OOH^* + e^- \rightarrow OH^- + O^*$
- (3)  $O^* + H_2O + e^- \rightarrow OH^- + OH^*$
- (4)  $OH^* + e^- \rightarrow OH^- + *$

Here the \* and OOH\* (O\* and OH\*) represent the adsorbed site of substrate and intermediate species OOH (O and OH) adsorbed on this site.



**Figure S1.** The <sup>1</sup>H NMR spectrum (a) and <sup>13</sup>C NMR spectrum (b) of LPAN with peak assignments as shown.

As is shown in Fig. S1, the structure of the obtained oligomer was confirmed by <sup>1</sup>H NMR. Most peaks are in agreement with the proton (<sup>1</sup>H) NMR structure of LPAN. The DP is calculated from 1 to 2 in view of the ratio of different peak areas.<sup>13</sup>C NMR was conducted on a 400 MHz spectrometer. C=N carbons (~120 ppm) and C-H carbons (20-60 ppm) peaks were clearly separated in spectrum.



Figure S2. EI-MS of LPAN with m/z with peak assignments.

The peak value of subtracting 1 from corresponding m/z is the molecular weight (MW) because of each molecule is protonized by positive-ion mode. MW<sub>LPAN</sub> can be evaluated based on the structure of LPAN by following equation:

 $MW_{LPAN} = MW_{ME} + MW_{AN} \times DP = 78 + 53 \times DP$ , which the value of  $MW_{ME}$  and  $MW_{AN}$  is 78 and 53. The numerical result is 131, 184 etc.

According to the maximum peak value of 184, the DP of most telomers is estimated to be 2, which is in line with the NMR spectra. Not all peaks match the value calculated by aforementioned equation, presumably due to the coupling/disproportionation of the initiating radicals. Essentially, the broad MW dispersity of the product stems from the radical polymerization using chain transfer agent, which differs from "living"/controlled free radical polymerization.<sup>4</sup>



Figure S3. Optical images of the synthesis process of doped porous carbon materials.



Figure S4. TGA (a) and DTG (b) of the AN and LPAN calcined in N<sub>2</sub> flow. Ramp rate, 10 °C/min; N<sub>2</sub> flow, 60 mL/min.

As is shown in Figure S3a, the monomer AN decomposed completely prior to reaching 85 °C. Correspondingly, the AN showed a sharp peak at 78 °C (Figure S4b), indicating a maximum weight loss rate. Notably, the LPAN shows two-part slow weight losses at 200 °C and 370 °C, owing reasonably to the thermal cross-linking and cyclization of polymer. It decomposed sluggishly upon increasing temperature above 400 °C revealing a good thermal stability for carbonization.<sup>5</sup>



**Figure S5.** (a) Nitrogen sorption isotherms for series of samples; (b) Pore size distributions and cumulative pore volumes for series of samples.



Figure S6. The SEM images of (a, b) CNS, (c, d) Fe-CNS and (e, f) Z-CNS



Figure S7. The SEM images of (a, b) FeZ-CNS-800 (c, d) FeZ-CNS-900 and (e, f)

FeZ-CNS-1000.



Figure S8. The HRTEM images (a, b, c and d) and the SAED (inset in d) of FeZ-

CNS-900.



Figure S9. The survey XPS spectrum of different samples.



Figure S10. N elemental analysis of the different samples based on the XPS results.



**Figure S11.** The high-resolution N 1s, S 2p and Fe 2p XPS spectra of FeZ-CNS-800 (a-c), FeZ-CNS-1000 (d-f) and Fe-CNS (g-i);The high-resolution N 1s, S 2p XPS spectra of Z-CNS (j, k) and CNS (l, m).



Figure S12. CV of different electrocatalysts in  $N_2$ - and  $O_2$ -saturated 0.1 M KOH solution at a scan rate of 50 mV s<sup>-1</sup>.



**Figure S13.** (a) RDE polarization curves of Pt/C and FeZ-CNS synthesized from different temperatures; (b) RDE polarization curves of FeZ-CNS at different loadings; (c) Tafel plots obtained from the RDE measurements in (a); (d) Tafel plots obtained from the RDE measurements in (a); Electrocatalytic ORR results in O<sub>2</sub>-saturated 0.1 M KOH at 5 mV s<sup>-1</sup> and 1600 rpm.



**Figure S14.** (a) RDE polarization curves of FeZ-CNS, FeZ-CN and a benchmark Pt/C at 5 mV s<sup>-1</sup> and 1600 rpm. (b)Comparison of E<sub>1/2</sub> and J<sub>k</sub> for Pt/C and FeZ-CNS synthesized from different temperatures.



**Figure S15.** RDE polarization curves of different electrocatalysts at different rotating speeds, the inset shows K–L plots at different potentials.



**Figure S16.** (a) RDE polarization curves of FeZ-CNS and Pt/C at 5 mV s<sup>-1</sup> and 1600 rpm; (b) CV of FeZ-CNS and Pt/C in N<sub>2</sub>- and O<sub>2</sub>-saturated 0.1 M HClO<sub>4</sub> solution at a scan rate of 50 mV s<sup>-1</sup>; (c) RDE polarization curves of FeZ-CNS at different rotating speeds, the inset shows K–L plots at different potentials; (d) Tafel plots obtained from the RDE measurements in (a); (e) RRDE curves for FeZ-CNS and Pt/C at 5 mV s<sup>-1</sup> and 1600 rpm; (f) Electron transfer number (n) (top) and H<sub>2</sub>O<sub>2</sub> yield (bottom) vs potential. Electrocatalytic ORR results in O<sub>2</sub>-saturated 0.1 M HClO<sub>4</sub> solution.



**Figure S17.** (a) Current–time (i–t) chronoamperometric response of FeZ-CNS and benchmark Pt/C at 0.70  $V_{RHE}$  in O<sub>2</sub>-saturated 0.1 M HClO<sub>4</sub> at a rotation rate of 1600 rpm; (b) Chronoamperometric response of FeZ-CNS and Pt/C upon addition of 1.0 M CH<sub>3</sub>OH at 0.70  $V_{RHE}$  in O<sub>2</sub>-saturated 0.1 M HClO<sub>4</sub> at a rotation rate of 1600 rpm; CVs of (c) FeZ-CNS and (d) Pt/C in O<sub>2</sub>-saturated 0.1 M HClO<sub>4</sub> without and with 1.0 M CH<sub>3</sub>OH at the scan rate of 50 mV s<sup>-1</sup>; RDE polarization curves of (e) FeZ-CNS and (f) Pt/C before and after 10000 potential cycles ranging from 0.6 to 1.0  $V_{RHE}$  with the scan rate at 100 mV s<sup>-1</sup>.



**Figure S18.** Specific capacities of the primary Zn-air battery with FeZ-CNS and Pt/C as the air catalyst, normalized to the weight of consumed Zn.

Samples	С	0	S	N	Fe
Samples	C	U	5	1	Γt
FeZ-CNS-800	85.61	5.20	2.84	5.25	1.05
FeZ-CNS-900	87.61	4.81	2.06	4.33	1.10
FeZ-CNS-1000	89.56	3.93	1.50	3.83	1.09
Z-CNS	90.29	5.25	1.47	2.94	/
Fe-CNS	87.98	5.73	1.51	3.23	1.49
CNS	86.79	6.32	2.27	4.56	/

Table S1. Elemental analysis and ICP-OES results of different samples (wt %).

Samples I <sub>D</sub> /I	Ir/Ic	Sbet (m <sup>2</sup> g <sup>-1</sup> )			Р	Average Pore		
	10/1G	Total	Micropore	Mesopore	Total	Micropore	Mesopore	Size (nm)
FeZ- CNS-800	0.98	2086.81	1823.79	263.02	1.29	0.67	0.62	2.21
FeZ- CNS-900	0.93	1730.75	1186.19	544.56	0.86	0.44	0.42	3.47
FeZ- CNS-1000	0.90	871.43	576.36	225.07	0.52	0.20	0.32	7.49
Z-CNS	/	1488.64	1118.11	370.53	0.64	0.37	0.27	4.32
Fe-CNS	/	689.17	548.54	140.63	0.44	0.15	0.12	5.65
CNS	/	146.39	54.21	92.18	0.24	0.02	0.22	40.99

**Table S2**.  $I_D/I_G$ , SSAs, total pore volumes and average particle sizes of different samples.

	XPS (wt %)								
Samples	С	0	S	Fe	Ν	Pyridinic -N	Pyrrolic -N	Graphitic -N	Oxidized -N
FeZ-CNS-800	87.98	5.60	1.38	0.39	4.65	1.55	0.56	2.13	0.41
FeZ-CNS-900	89.66	5.35	1.10	0.43	3.36	0.83	0.37	1.83	0.34
FeZ-CNS-1000	91.67	4.03	1.06	0.41	2.83	0.59	0.25	1.75	0.25
Z-CNS	89.35	6.52	1.33	/	2.80	0.71	0.16	1.72	0.21
Fe-CNS	93.30	4.17	0.76	0.61	1.16	0.31	0.04	0.60	0.21
CNS	85.94	8.36	1.86	/	3.84	1.15	0.17	2.40	0.12

 Table S3. Elemental compositions of different samples by XPS.

**Table S4**. ORR performance of samples and Pt/C electrocatalysts. Rotating disk electrode (RDE) results in terms of onset potential at 0.3 mA cm<sup>-2</sup>, half-wave potential, kinetic current density at 0.85 V and electron transfer number n under  $O_2$ -saturated 0.1 M KOH.

Catalyst	Loading (mg cm <sup>-2</sup> )	E <sub>onset</sub> (V <sub>RHE</sub> )	<i>E</i> <sub>1/2</sub> (V <sub>RHE</sub> )	J <sub>L</sub> (mA cm <sup>-2</sup> )	$J_k$ at 0.85 V (mA cm <sup>-2</sup> )	Tafel slopes (mV dec <sup>-1</sup> )	n
Pt/C	0.10	0.930	0.842	5.69	4.80	66	4.01
FeZ- CNS-900	0.50	0.963	0.881	5.63	24.10	62	4.00
FeZ- CNS-800	0.50	0.890	0.816	5.27	4.60	55	3.99
FeZ- CNS-1000	0.50	0.954	0.846	4.03	1.10	85	3.40
Fe-CNS	0.50	0.939	0.870	3.70	11.40	70	3.64
Z-CNS	0.50	0.871	0.785	4.96	0.60	64	3.95
CNS	0.50	0.764	0.640	2.43	0.10	103	2.28

Catalyst	Loading (mg cm <sup>-2)</sup>	E <sub>onset</sub> (V <sub>RHE</sub> )	<i>E</i> <sub>1/2</sub> (V <sub>RHE</sub> )	J <sub>k</sub> (mA cm <sup>-2</sup> )	Reference
Fe-N-CNFs	0.60	0.93	0.81	48.15 at -0.45 V vs. Ag/AgCl	Angew. Chem., Int. Ed. <b>2015</b> , 54, 8179.
Fe-N/C-800	0.079	0.98	~0.83	/	J. Am. Chem. Soc. <b>2015</b> , 137, 5555.
Co-N-C-0.8 NPHs	0.283	/	0.871	39.3 at 0.80 V	ACS Catalysis <b>2015</b> , <i>5</i> , 7068.
(Fe,Mn)-N-C- 3HT-2AL	0.80	0.98	0.900	36.8 at 0.80 V	<i>Nat. Commun.</i> <b>2015</b> , <i>6</i> , 8618.
FePhen@MO FArNH3	0.60	/	0.86	2.1 at 0.90 V	<i>Nat. Commun.</i> <b>2015</b> , <i>6</i> , 7343.
Fe-N-CC	0.10	0.94	0.83	18.3 at 0.58 V	ACS Nano <b>2016</b> , 10, 5922.
Fe-NMCSs	0.255	1.027	0.86	/	<i>Adv. Mater.</i> <b>2016</b> , <i>28</i> , 7948.
Co,N-CNF	0.12	0.883	0.81	/	<i>Adv. Mater.</i> <b>2016</b> , <i>28</i> , 1668
Co9S8@CNS9 00	0.40	0.915	0.80	27.3 at -0.45 V vs. Ag/AgCl	<i>Adv. Mater.</i> <b>2016</b> , <i>28</i> , 6391
FP-Fe-TA-N- 850	0.30	0.98	/	8.0 at 0.80 V	Angew. Chem., Int. Ed. <b>2016</b> , 55, 1355.
Co SAs/N- C(900)	0.408	0.982	0.881	21.2 at 0.80 V	Angew. Chem. Int. Ed. <b>2016</b> , 55, 10800.
Co <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub> C-N- HA/rGO	0.25	0.962	0.837	38.46 at 0.75 V	Energy Environ. Sci. <b>2016</b> , 9, 2563.
Fe@C-FeNCs- 2	0.70	/	0.899	41.6 at 0.80 V	J. Am. Chem. Soc. <b>2016</b> , 138, 3570.
CNT/PC	0.80	/	0.88	2.4 at 0.90 V	J. Am. Chem. Soc. <b>2016</b> , 138, 15046.
NCNTFs	0.20	0.97	0.87	/	<i>Nature Energy</i> <b>2016</b> , <i>1</i> , 15006.
Fe/N/C@BMZ IF	0.20	0.95	0.85	/	ACS Appl. Mater. Interfaces <b>2017</b> , 9,5213
COP- TPP(Fe)@MO	0.20	0.99	0.85	/	ACS Nano <b>2017</b> , 11, 8379.

**Table S5** Comparison of ORR performance of FeZ-CNS-900 product with various non-<br/>precious electrocatalysts in the literature under O2-saturated 0.1 M KOH.

F-900					
pCNT@Fe1.5 @GL	0.20	0.957	0.87	/	<i>Adv. Mater.</i> <b>2017</b> , <i>29</i> , 1606534.
CalCoZIFVX C72	0.40		0.84	13.8 at 0.80 V	<i>Adv. Mater.</i> <b>2017</b> , <i>29</i> , 1701354.
Fe/NMC-11	0.51	/	0.862	/	<i>Adv. Energy.</i> <i>Mater.</i> <b>2017</b> , 7, 1701154.
Fe-SNC-900	0.35	0.979	0.834	/	Adv. Energy. Mater. <b>2017</b> , 7, 1602002.
Core/shell NPME	0.60	/	0.87	/	<i>Adv. Funct. Mater.</i> <b>2017</b> , <i>27</i> , 1604356.
S,N-Fe/N/C- CNT	0.60	/	0.85	/	Angew. Chem., Int. Ed. <b>2017</b> , <i>56</i> , 610.
FeN2/NOMC	~0.51	1.04	0.863	45.2 at 0.79 V	Nano Energy <b>201</b> 7, 35, 9
Fe-N-CNBs- 600	0.429	1.03	0.875	~25 at -0.4 V vs. Ag/AgCl	J. Mater. Chem. A <b>2017</b> , 5, 11340.
FeBNC-800	0.60	0.968	0.838	/	ACS Energy Letters <b>2018</b> , <i>3</i> , 252.
Co-CNTFs	0.20	0.988	0.835	/	Energy Storage Materials, <b>2018</b> , 11, 112
Fe-N-C/rGO	~0.36	0.94	0.81	/	ACS Appl. Mater. Interfaces <b>2018</b> , 10, 2423.
Pt/C	0.10	0.930	0.842	19.6 at 0.80 V 4.80 at 0.85 V 0.85 at 0.90 V	This work
FeZ-CNS-900	0.50	0.963	0.881	58.70 at 0.80 V 24.10 at 0.85 V 2.89 at 0.90 V	This work

Catalyst	Loading (mg cm <sup>-2)</sup>	Electrolyte	E <sub>onset</sub> (V <sub>RHE</sub> )	<i>E</i> <sub>1/2</sub> (V <sub>RHE</sub> )	Reference
Fe-N-rGO	0.40	0.5 M H <sub>2</sub> SO <sub>4</sub>		0.63	<i>Chem. Mater.</i> <b>2011</b> , <i>23</i> , 3421.
Fe-N-C/CNTs	1.20	0.1 M HClO4	0.83	0.67	<i>Electrochim. Acta</i> <b>2013</b> , <i>107</i> , 126.
CoP-CMP800	0.60	0.5 M H <sub>2</sub> SO <sub>4</sub>	0.74	0.64	<i>Adv. Mater.</i> <b>2014</b> , <i>26</i> , 1450.
PpPD-Fe-C	0.90	0.5 M H <sub>2</sub> SO <sub>4</sub>	0.826	0.718	Angew. Chem., Int. Ed. <b>2014</b> , <i>53</i> , 2433.
Fe3C/C-700	0.60	0.1 M HClO4		0.71	Angew. Chem. Int. Ed. <b>2014</b> , 53, 3675.
ZIF-67-900-AL		0.1 M HClO4	0.85	0.71	J. Mater. Chem. A <b>2014</b> , 2, 14064.
CPM-99Co/C	0.6	0.1 M HClO4		0.55	J. Am. Chem. Soc. <b>2015</b> , 137, 2235.
Fe3C/CNT	1.20	0.1 M HClO <sub>4</sub>		~0.60	J. Am. Chem. Soc. <b>2015</b> , 137, 1436.
LDH@ZIF-67-800	0.2	0.1 M HClO4	0.875	0.675	<i>Adv. Mater.</i> <b>2016</b> , <i>28</i> , 2337.
NPC-1000	0.42-0.99	0.5 M H <sub>2</sub> SO <sub>4</sub>	0.818	0.698	<i>Adv. Funct. Mater.</i> <b>2017</b> , <i>14</i> , 1606190.
PPy/FeTCPP/Co	0.30	0.1 M HClO4		0.72	<i>Adv. Funct. Mater.</i> <b>2017</b> , <i>27</i> , 1606497.
1100-CNS	0.6	0.1 M HClO <sub>4</sub>	0.88	0.73	Energy Environ. Sci. <b>2017</b> , 10, 742.
Fe-SNC	0.35	0.1 M HClO4	0.560		<i>Adv. Energy. Mater.</i> <b>2017</b> , <i>7</i> , 1602002.
Fe-N-C-950	0.4	0.1 M HClO <sub>4</sub>	0.92	0.78	ACS Catalysis <b>2018</b> , <i>8</i> , 2824.
Co-N-C-10	0.40	0.1 M HClO4	0.92	0.79	Nano Energy <b>2018</b> , 46, 396.
Pt/C	0.10	0.1 M HClO4	0.861	0.780	This work
FeZ-CNS-900	0.50	0.1 M HClO4	0.820	0.720	This work

**Table S6.** Comparison of ORR performance of FeZ-CNS-900 product with variousnon-precious electrocatalysts in the literature under O2-saturated acid medium.

Catalyst	Loading (mg cm <sup>-2)</sup>	Open circuit potential (V)	Peak power density (mW cm <sup>-2</sup> )	Specific capacity (mAh g <sup>-1</sup> <sub>Zn</sub> ) at 10 mA cm <sup>-2</sup>	Reference
NiO/CoN PINWs	2.0	1.46	79.6	648	6
ZnCo2O4/N-CNT	2.0	1.47	82.3	428.47	7
NPMC-1000	0.5	1.48	55	735 at 5 mA cm <sup>-2</sup>	8
NCNF	2.0	1.48	185	626	9
CoO/N-CNT	1.0	1.40	256	576	10
Нрс-800	2.0	1.50		647	11
PBSCF-NF		1.25	127		12
Ni-MnO/rGO	10.0		123	758 at 5 mA cm <sup>-2</sup>	13
C0O <sub>0.87</sub> S <sub>0.13</sub> /GN	2.0	1.43		709	14
Co <sub>3</sub> FeS <sub>1.5</sub> (OH) <sub>6</sub>			113.1	898 at 20 mA cm <sup>-2</sup>	15
Co-N-CNTs	1	1.365	101		16
Cu@Fe-N-C	1	1.48	92		17
Pt/C	1.0	1.49	153	688 at 10 mA cm <sup>-2</sup>	This work
FeZ-CNS-900	2.0	1.49	168	778 at 5 mA cm <sup>-2</sup> 756 at 10 mA cm <sup>-2</sup>	This work

Table S7. Summary of the performance of Zn-Air batteries reported in recent literature.

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