Supporting Information

Urea-assisted Synthesis of Fe Nanoparticles Modified N-doped Three-Dimensional Porous Carbon Framework for Highly Efficient Oxygen Reduction Reaction

Hui Peng[a]*‡, Xuan Xie[a]‡, Kanjun Sun[b], Miaoran Zhang[a], Rui Zhao[a], Guofu Ma[a]*, Ziqiang Lei[a]

[a]Key Laboratory of Eco-Environment-Related Polymer Materials of Ministry of Education, Key Laboratory of Polymer Materials of Gansu Province, College of Chemistry and Chemical Engineering, Northwest Normal University, Lanzhou 730070, China.

[b]College of Chemistry and Environmental Science, Lanzhou City University, Lanzhou 730070, China.

‡These authors contributed equally to this work. They should thus be considered co-first authors.

*Corresponding authors. E-mail addresses: penghui@nwnu.edu.cn (H. Peng), magf@nwnu.edu.cn (G. Ma)

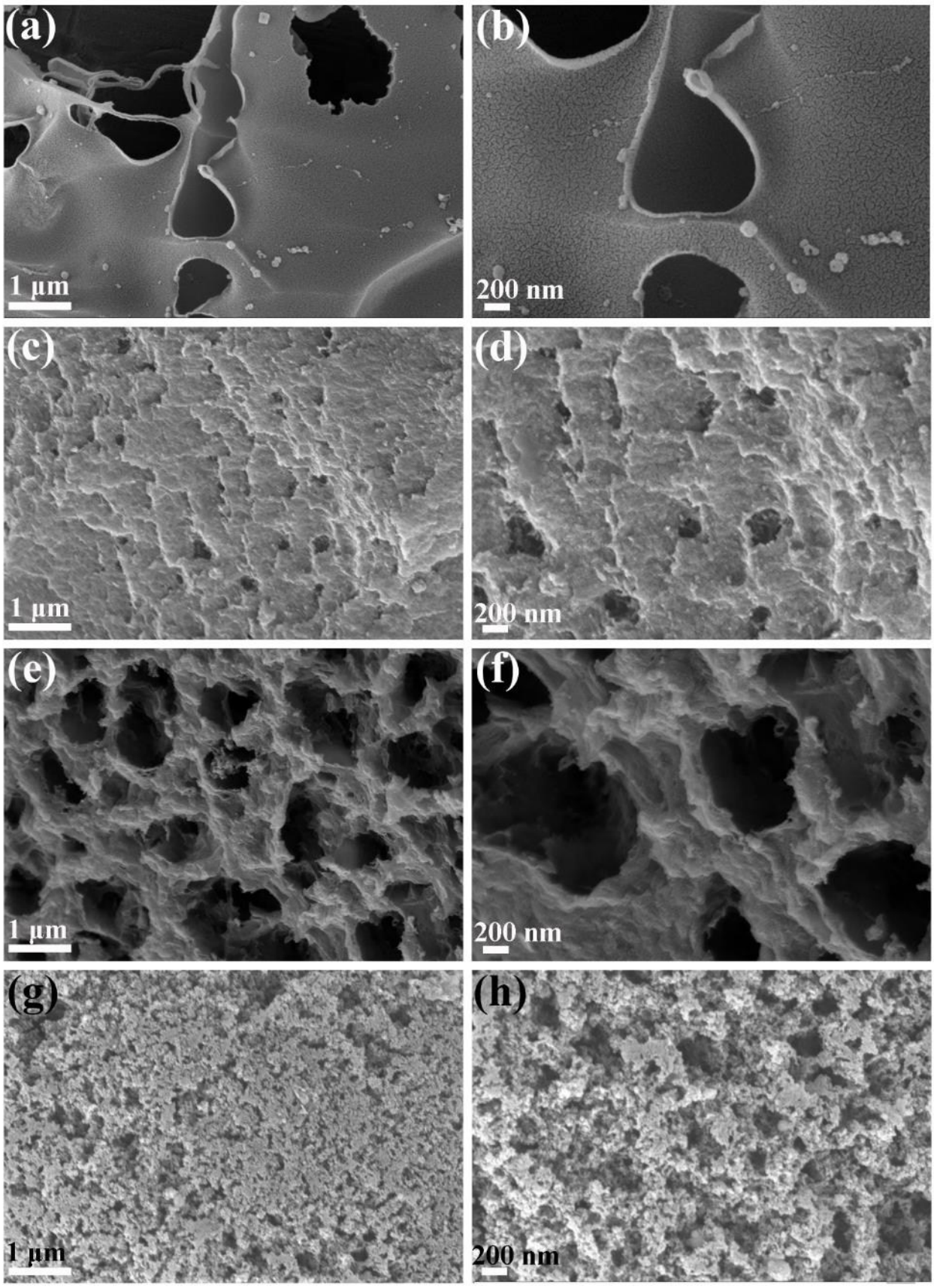

Figure S1. SEM images of (a, b) Fe/3DNC-0, (c, d) Fe/3DNC-1, (e, f) 3DNC-2 and (g, h) Fe/3DNC-3 at different magnification.

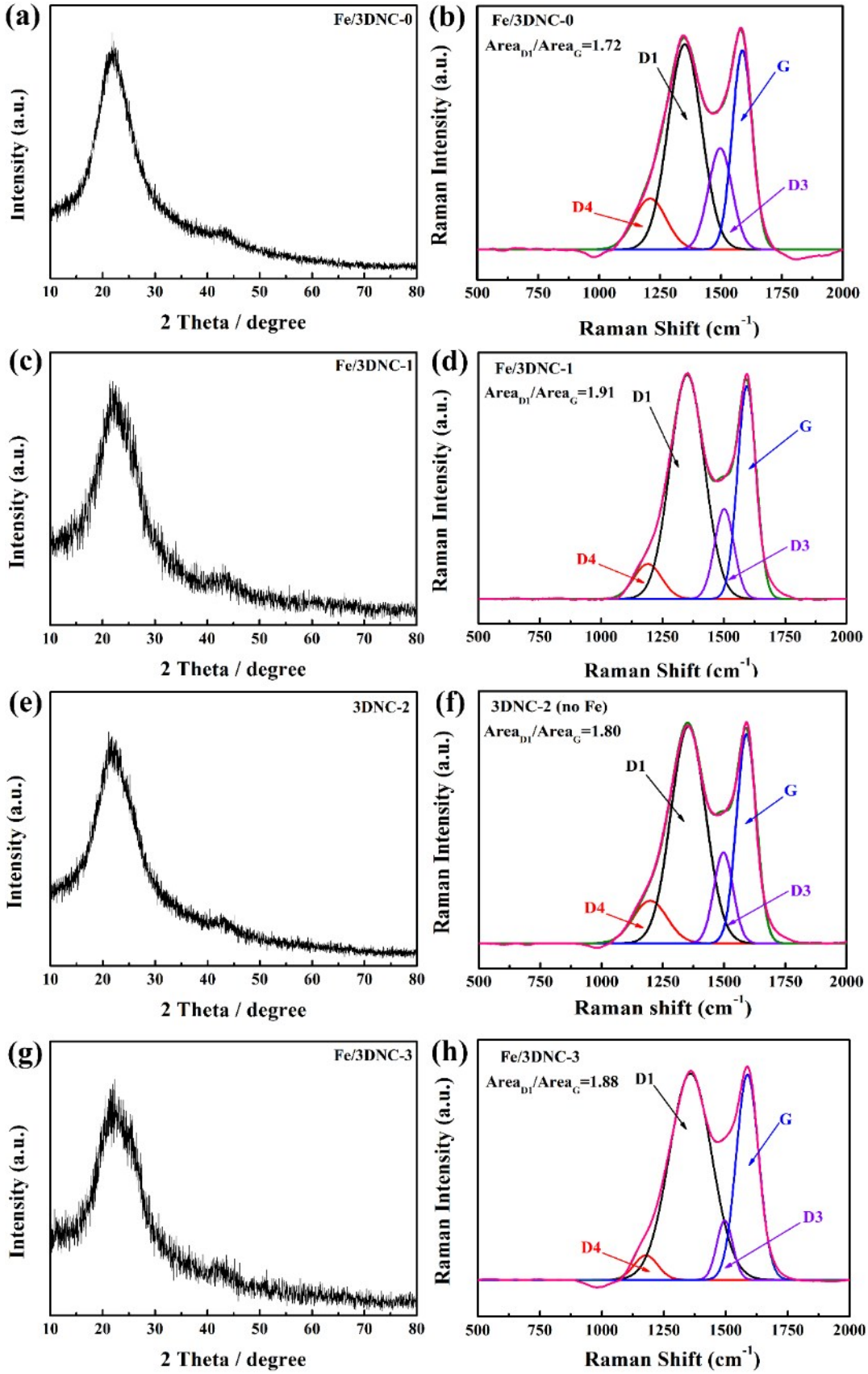

Figure S2. XRD patters and Raman spectra of (a, b) Fe/3DNC-0, (c, d) Fe/3DNC-1, (e, f) 3DNC-2 and (g, h) Fe/3DNC-3.

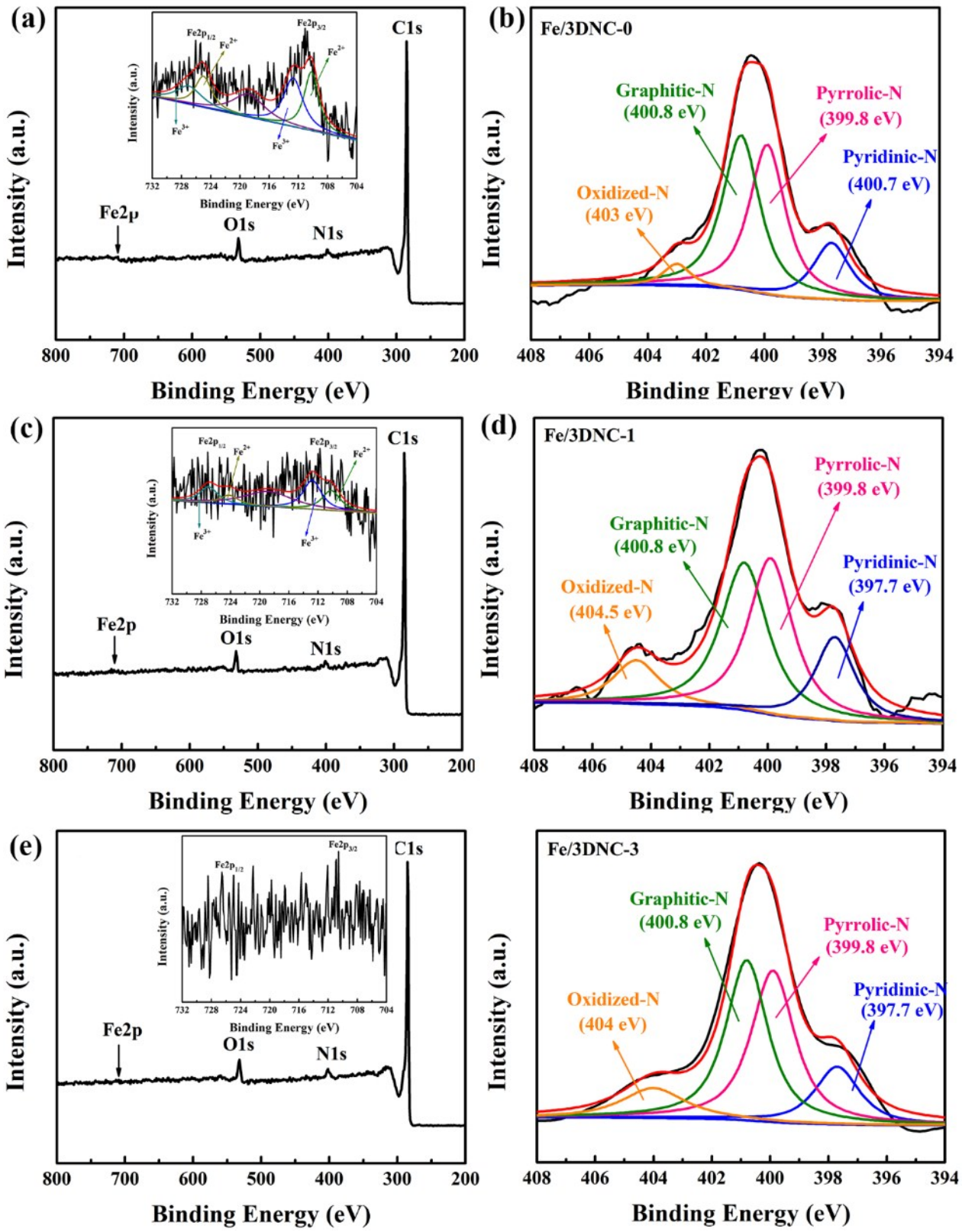

Figure S3. XPS survey spectrum of (a-b) Fe/3DNC-0, (c-d) Fe/3DNC-1, (e-f) Fe/3DNC-3 (The inset shows Fe 2p) and he high-resolution XPS spectra of N1s.

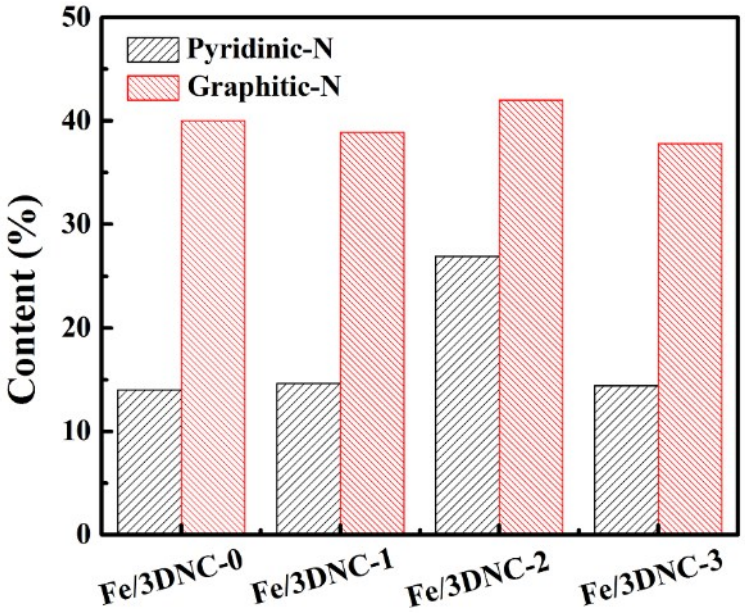

Figure S4. Histogram of pyridinic-N and graphitic-N contents in the Fe/3DNC catalysts.

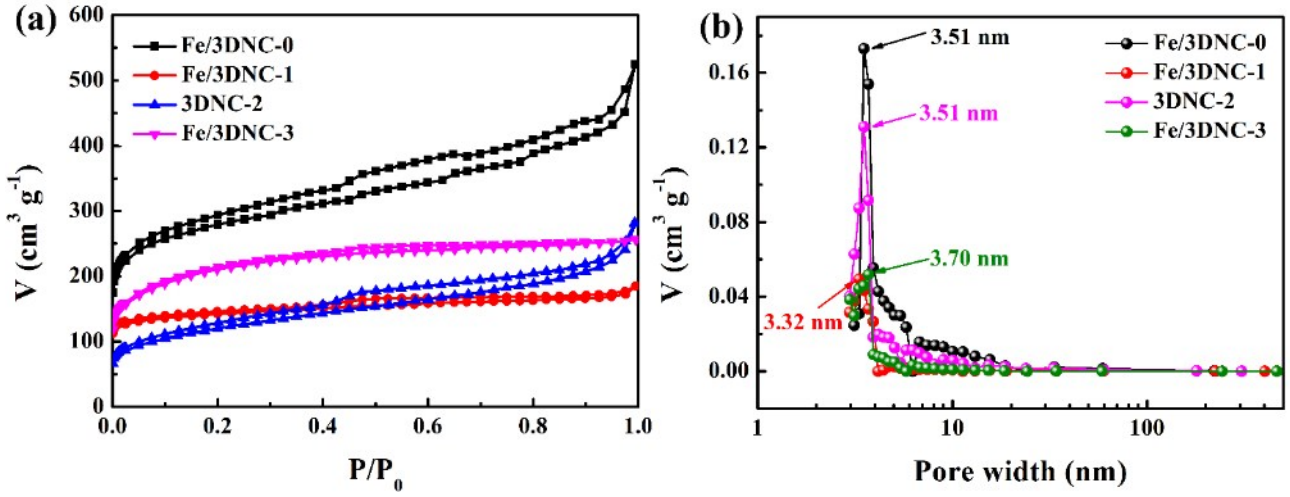

Figure S5. (a) N_2 absorption/desorption isotherms and (b) pore size distribution of Fe/3DNC-0, Fe/3DNC-1, 3DNC-2 and Fe/3DNC-3.

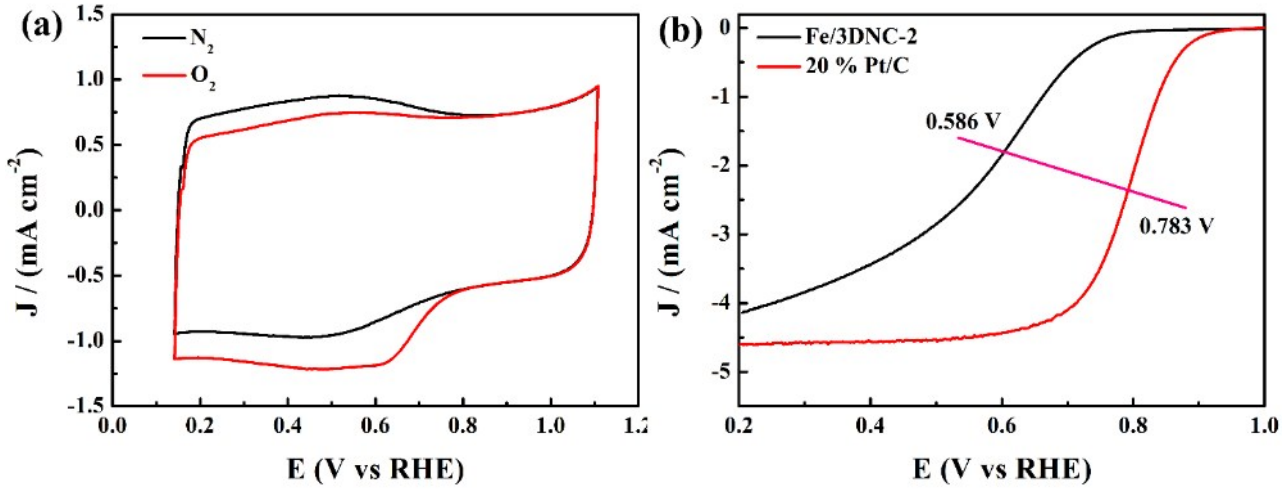

Figure S6. (a) CV curves of Fe.3DNC-2 catalyst in N_2 saturated (Black line) and O_2 saturated (Red line) 0.1 M $HClO_4$ electrolyte (scan rate: 50 mV s^{-1}), (b) LSV curves of the Fe/3DNC-2 and 20 % Pt/C catalysts (rotation speed: 1600 rpm; scan rate: 5mV s^{-1}),

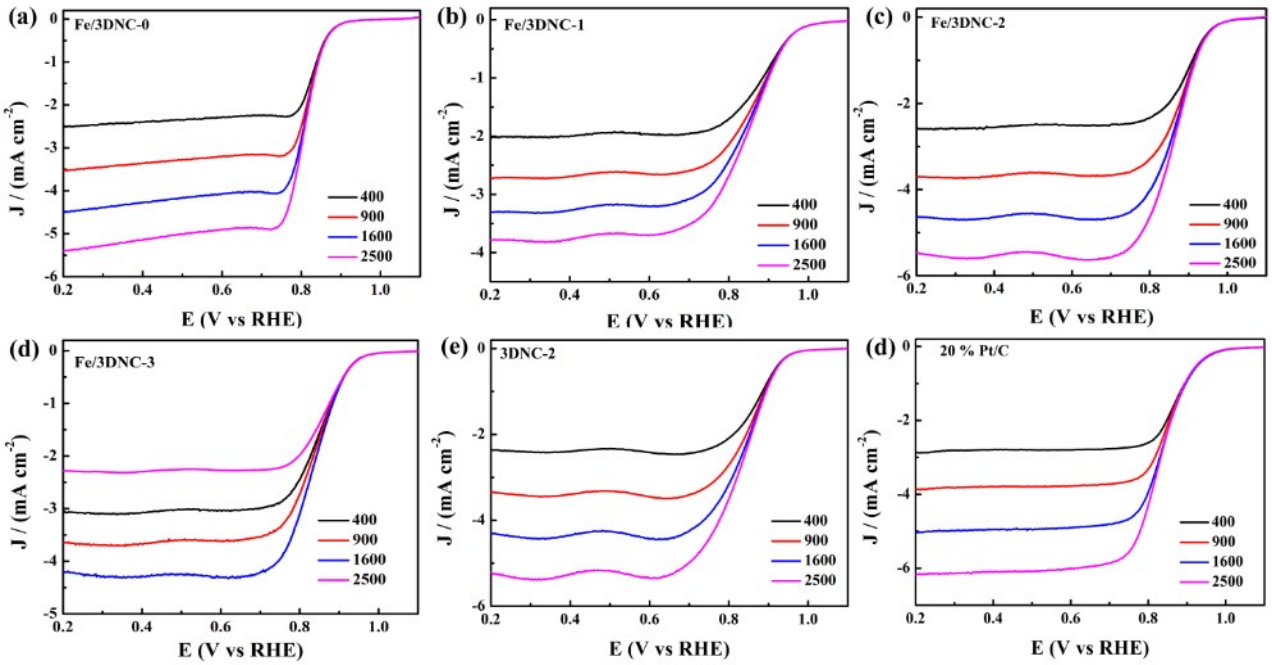

Figure S7. LSV curves at different rotating speeds for (a) Fe/3DNC-0, (b) Fe/3DNC-1, (c) Fe/3DNC-2, (d) Fe/3DNC-3, (e) 3DNC-2 and (f) Pt/C in O_2-saturated 0.1 M KOH electrolyte.

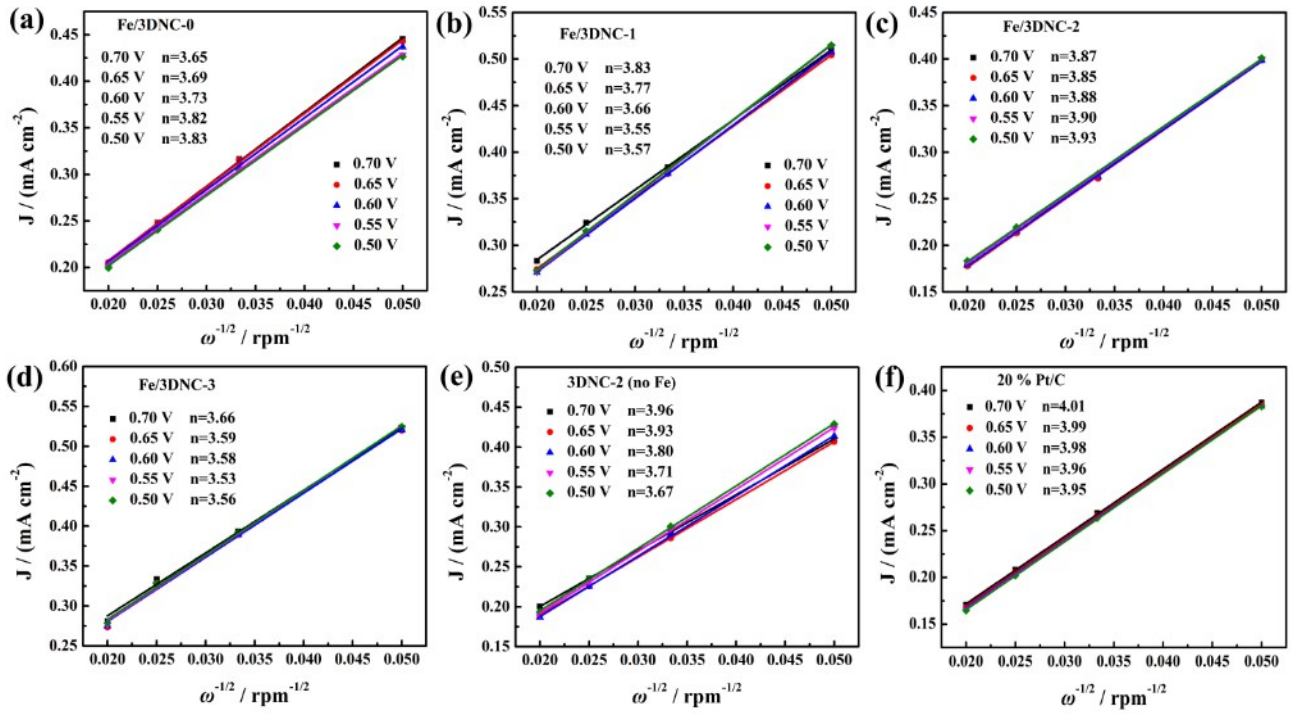

Figure S8. K-L plots and transferred electron numbers at various potential for (a) Fe/3DNC-0, (b) Fe/3DNC-1, (c) Fe/3DNC-2, (d) Fe/3DNC-3, (e) 3DNC-2 and (f) Pt/C in O_2-saturated 0.1 M KOH electrolyte.

Table S1. X-ray photoelectron spectroscopy (XPS) and Full spectrum direct reading inductively coupled plasma optical emission spectrometer (ICP-OES) results for element contents in Fe/3DNC-2 samples.

	C (wt %)	N (wt %)	O (wt %)	Fe (wt %)
XPS	94.09	2.50	3.32	0.09
ICP	_	_	_	1.18

Table S2. The element contents by XPS for Fe/3DNC-0, Fe/3DNC-1, Fe/3DNC-2 and Fe/3DNC-3 samples.

XPS	C (wt %)	N (wt %)	O (wt %)	Fe (wt %)
Fe/3DNC-0	93.72	2.41	3.46	0.41
Fe/3DNC-1	93.89	2.33	3.65	0.13
Fe/3DNC-2	94.09	2.50	3.32	0.09
Fe/3DNC-3	94.01	2.65	3.42	0

Table S3. BET surface area and pore structure characterization parameters of all catalysts.

catalysts	S_{BET} (m^2 g^{-1})	Average pore Diameter (nm)	Total pore Volume (cm^{-3} g^{-1})
Fe/3DNC-0	812	3.61	0.790
Fe/3DNC-1	408	3.31	0.282
Fe/3DNC-2	438	2.65	0.296
3DNC-2	430	2.62	0.439
Fe/3DNC-3	763	2.08	0.389

Table S4. Comparison of Fe/3DNC-2 with some M-N-C catalysts reported in literatures.

Catalysts	Loading Mass ($mg\ cm^{-2}$)	E_{onest} (V vs RHE)	$E_{1/2}$ (V vs RHE)	Reference
Fe/3DNC-2	0.20	0.995	0.874	This work
Zn/Fe_2-N-C	0.34	1.08	0.86	[1]
Fe(0)@FeNC	0.3	0.946	0.852	[2]
Fe-N-C/MXene	-	0.92	0.84	[3]
Fe-NAHPC-900	0.23	0.94	0.86	[4]
Cu-Fe-N-C	-	0.967	0.864	[5]
Fe_{DZ}-NC	-	0.98	0.863	[6]

References

[1] J. L. Xue, Y. S. Li and J. Hu, *J. Mater. Chem. A*, 2020.

[2] Z. T. Li, L. Q. Wei, W. J. Jiang, Z. P. Hu, H. Luo, W. N. Zhao, T. Xu, W. T. Wu, M. B. Wu and J. S. Hu, *Appl. Catal. B-Environ.*, 2019, 251, 240-246.

[3] L. L. Jiang, J, J. Duan, J. W. Zhu, S. Chen and M. Antonietti, *ACS Nano*, 2020.

[4] P. F. Tian, Y. H. Wang, W. Li, S. W. Song, S. Y. Zhou, H. W. Gao, H. Q. Xu, X. Q. Tian and J. B. Zang, *J. Cata*., 2020, 382, 109-120.

[5] J. S. Li, J. J. Chen, H. Wan, J. Xiao, Y. G. Tang, M. Liu and H. Y. Wang, *Appl. Catal. B-Environ*., 2019, 242, 209-217.

[6] M. L. Mao, J. Deng, T. T. Yan, J. J. Shen, J. P. Zhang, L. Y. Shi and D. S. Zhang, *ACS Sustainable Chem. Eng.*, 2019, 7, 19268-19276.