## Supporting information

## Defective metal-organic framework derivative by roomtemperature exfoliation and reduction for highly efficient oxygen evolution reaction

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Fig. S1. The crystal structure and coordination of Co-MOF from the Cambridge Crystallographic Data Centre..



Fig. S2. The multi-layered structure of the Co-MOF (a-b) and CoFe-MOF (c-d).



**Fig. S3**. (a) LSV curves and (b) double-layer capacitance of CoFe-MOF and Fe@Co-MOF.



Fig. S4. XRD pattern of Fe@Co-MOF washed by DI-water and the standard card.



Fig. S5. The TG curve of r-CoFe.



Fig. S6. The photos of dialysate from mother liquid  $[FeSO_4 \text{ solution}, Co(NO_3)_2 \text{ solution}, Fe@Co-MOF suspension, CoFe-MOF suspension, Co-MOF suspension and DI water, left to right] with the addition of 1 M Na<sub>2</sub>S solution after a dialysis for 10 min (a) and 5 h (b). (molecular weight cutoff is 8000-14000)$ 



**Fig. S7**. XPS spectra of Co-MOF (a), Fe@Co-MOF (b) and r-CoFe (c), respectively. Inset: the corresponding magnification (i-iii).



Fig. S8. The XPS fitting of O 1s of r-CoFe.



Fig. S9. The UV-vis absorption spectra of Co-MOF, Fe@Co-MOF and r-CoFe.



**Fig. S10**. (a) LSV curves and (b) Tafel plots of r-CoFe, r-CoFe-OH, r-CoFe-MOF and r-Co-MOF, respectively.



Fig. S11. The summarized overpotentials of Co-MOF, r-Co-MOF, CoFe-MOF, r-CoFe-MOF, Fe@Co-MOF and r-CoFe, respectively.



Fig. S12. The CVs measured in a non-Faradaic region of Co-MOF (a), Fe@Co-MOF (b), r-CoFe-OH (c) and r-CoFe (d).



Fig. S13. (a) LSV curves and (b) Tafel plots of Co-MOF,CoFe-MOF and r-CoFe normalized by  $C_{dl}$ .

	*Position Co <sup>2+</sup> (eV)	**Position Co <sup>3+</sup> (eV)	***R(Co <sup>2+</sup> /Co <sup>3+</sup> )
Co-MOF	782.57	781.13	1.22
Fe@Co-MOF	782.76	781.29	1.36
r-CoFe	782.47	781.07	2.03

**Table S1**. The location of XPS fitting peaks and the ratio of peak area.

\*: the position of  $Co^{2+}$  XPS peak. \*\*: the position of  $Co^{3+}$  XPS peak. \*\*\*: the ratio of XPS peak area for  $Co^{2+}$  and  $Co^{3+}$ .

**Table S2**. Comparisons of OER activities of some representative Co/Fe-based MOF derivatives.

Electrocatalysts	$\eta_{10 \text{ mA cm}^{-2}}$ (mV)	Tafel slope (mV dec <sup>-1</sup> )	References
r-CoFe	253	39	This work
Co <sub>0.6</sub> Fe <sub>0.4</sub> P-1.125	298	48	<i>Chem. Sci.</i> , 2019, <b>10</b> , 464- 474.
CoFe-MOF-74-400°C-1h	300	-	J. Am. Chem. Soc. 2018, <b>140</b> , 15336-15341.
FeCo-Co <sub>4</sub> N/N-C	280	40	<i>Adv. Mater.</i> 2017, <b>29</b> , 1704991.
Co <sub>0.7</sub> Fe <sub>0.3</sub> P/C	270	27	Nanoscale, 2018, <b>10</b> , 19774- 19780.
Co-PBA-plasma-2h	274	53	<i>Adv. Energy Mater.</i> 2018, <b>8</b> , 1800085.
Co-Fe-P-1.7	244	55	ACS Appl. Mater. Inter. 2017, 9, 362-370.
NF@NC-CoFe <sub>2</sub> O <sub>4</sub> /C	240	45	<i>Adv. Mater.</i> 2017, <b>29</b> , 1604437.
Fe <sub>1</sub> Co <sub>3</sub> /Vo-800	250	53	J. Mater. Chem. A, 2019, 7, 3090-3100.
S-Co <sub>9-x</sub> Fe <sub>x</sub> S <sub>8</sub> @rGO-10	290	66	Small, 2018, 14, 1703748.

	Co-MOF	Fe@Co-MOF	r-CoFe-OH	r-CoFe
Co	15.3	11.9	40.0	30.8
Fe		5.7	18.2	20.4
В			6.1	3.0

**Table S3**. The ICP results of Co-MOF, Fe@Co-MOF, r-CoFe-OH and r-CoFe (unit:w%).