Supporting Information

Ultrafine Ir Nanoparticles Decorated on FeP/FeOOH with Abundant Interface via Facile Corrosive Approach for Alkaline Water-splitting

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1. Experimental Section

1.1 Materials and synthesis

Materials: Fe Foam (FF) with the thickness of 2 mm was purchased from Kunshan Tengerhui Electronic Technology Co., Ltd. Sodium chloride (NaCl), Sodium hypophosphite monohydrate ($NaH_2PO_2 \cdot H_2O$), Potassium hydroxide (KOH) were purchased from Sinopharm Chemical Reagent Co., Ltd. Iridium chloride ($IrCl_3$) was purchased from Shanghai Macklin Biochemical Co., Ltd. Deionized water was utilized in all the experimental process. All the chemicals and reagents used in the experiment were directly used without further purification.

Synthesis of FF-NaCl-Ir Catalysts: The FF-NaCl-Ir catalysts were synthesized by a one-step oxygen corrosion method. Put a slice of FF (2 cm*2 cm), NaCl (0.0087 g), $IrCl_3$ (0.0448 g) into a beaker with 30 mL deionized (DI) water and stirred at room temperature (25 ^oC) for one hour. Then, the product was collected and washed by deionized water several times. Finally, the product was dried at 60 ^oC under vacuum for 12 h and denoted as FF-NaCl-Ir.

Synthesis of FF-NaCl-Ir-P Catalysts: Under the protection of nitrogen, put FF-NaCl-Ir catalysts and $NaH_2PO_2 \cdot H_2O$ in a tube furnace and then heated up to 360 ^oC in Ar for 2 h with a heating rate of 3 ^oC min to obtain FF-NaCl-Ir-P.

1.2 Material characterization

The morphologies of the catalyst is characterized by the scanning electron microscopy (SEM, S-4800, Hitachi) and transmission electron microscopy (TEM, JSM-2100). The composition and valence of elements are proved by X-ray photoelectron spectroscopy

(XPS, AXISULTRA DLD). The crystal structure of the material is proved by X-ray diffraction (XRD, X'Pert PRO). The contact angle is measured by Dataphysics-OCA40. The XRD test is a direct test of the synthesized catalyst block. The TEM test is to make the synthesized catalyst block into powder.

1.3 Electrochemical measurements

All the electrochemical tests were performed by using a CHI 760E electrochemical workstation with a three-electrode system at room temperature. A 1.0 M KOH solution was used as the electrolyte, FF products as the working electrode, a graphite rod as the counter electrode, and Ag/AgCl as the reference electrode. For comparison, all potentials were adjusted to the reversible hydrogen electrode (RHE).

$$E(RHE) = E(Ag/AgCl) + 0.059 * pH + 0.197.$$

The polarization curve of the electrode was obtained by linear sweep voltammetry (LSV) test with a scanning rate of 5 mV s^{-1}. During the electrochemical test, we immersed catalyst FF-NaCl-Ir-P with an area of 1cm*0.5cm into the electrolyte. In LSV test, the voltage setting intervals of HER, OER and water splitting are (-0.9V, -1.5V), (0V, 0.8V) and (0V, 3V) respectively. When testing commercial catalyst, the immersion area and voltage setting interval are the same as that of catalyst FF-NaCl-Ir-P. Long term stability measurements were performed at a specific potential. Overall water splitting measurements were performed using a CHI 760E potentiostat in a two-electrode system by applying FF-NaCl-Ir-P as the cathode and anode.

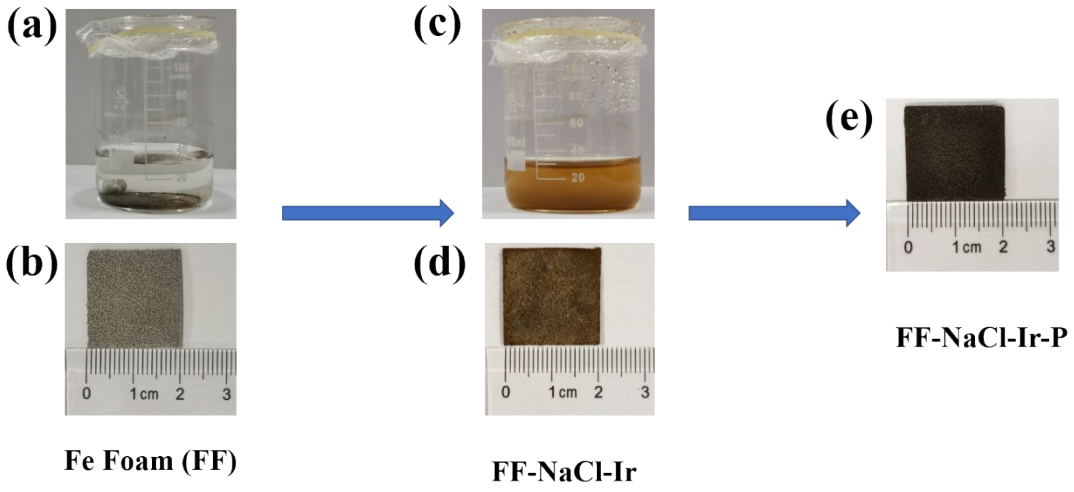

Figure S1. Pictures of the preparation process

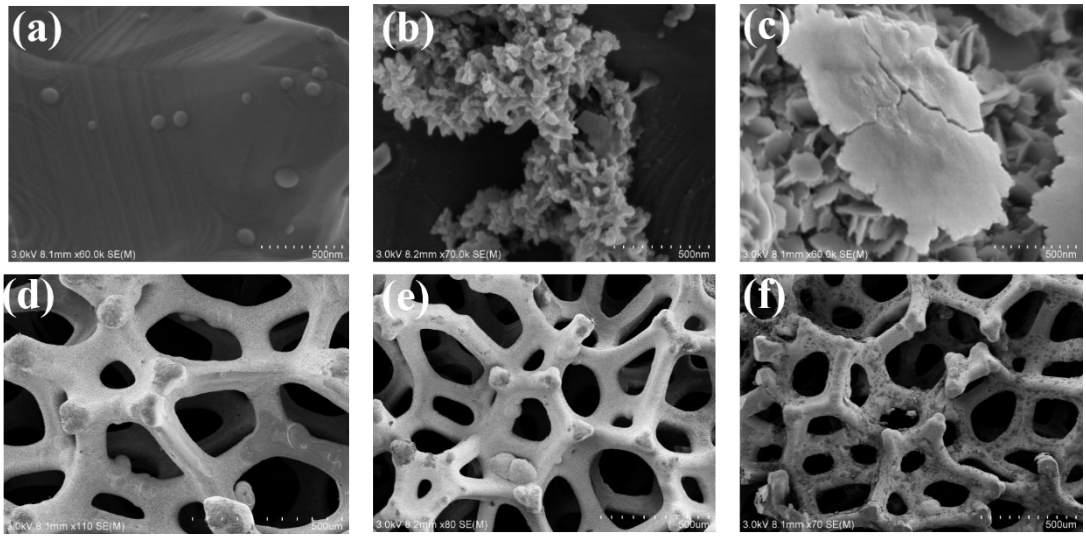

Figure S2. SEM images of Fe Foam (a, d), FF-NaCl (b, e) and FF-NaCl-Ir (c, f)

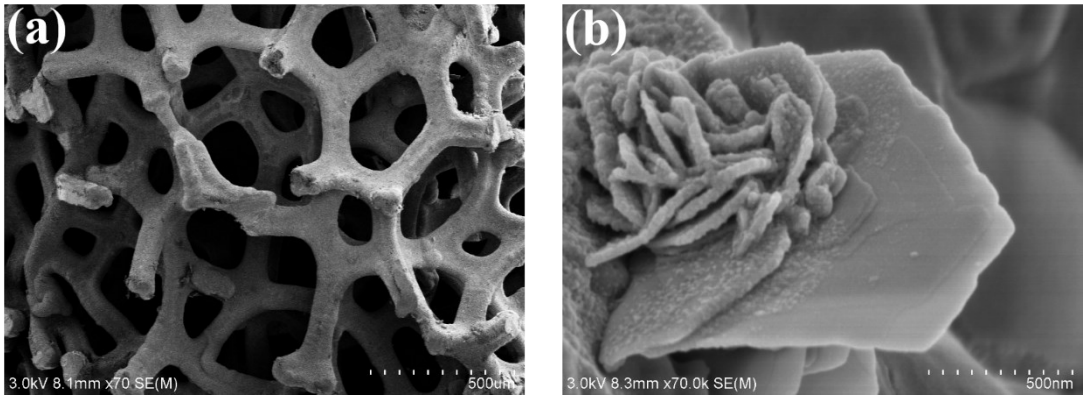

Figure S3. SEM images of FF-NaCl-Ir-P

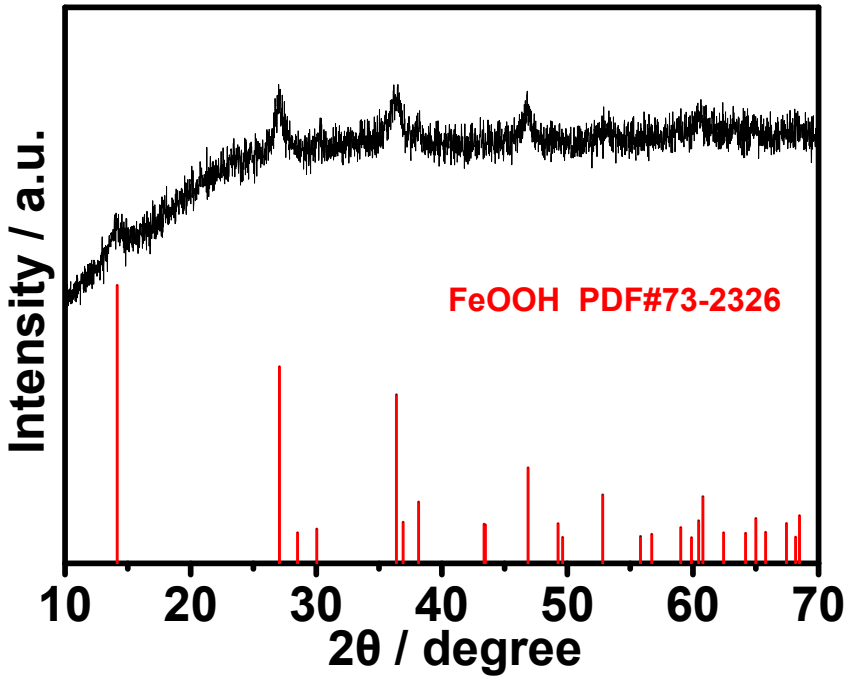

Figure S4. XRD pattern of FF-NaCl

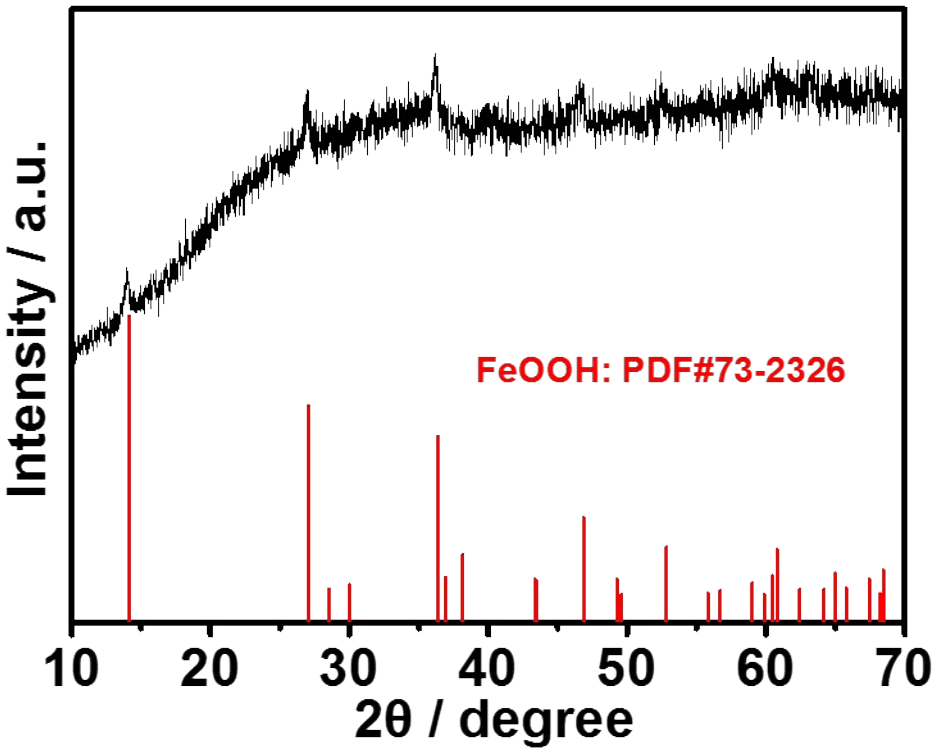

Figure S5. XRD pattern of FF-NaCl-Ir

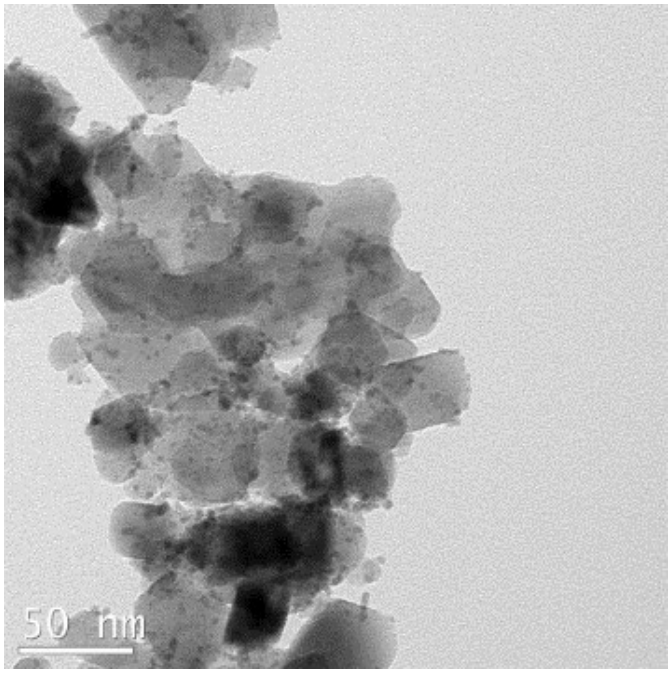

Figure S6. TEM image of FF-NaCl-Ir-P.

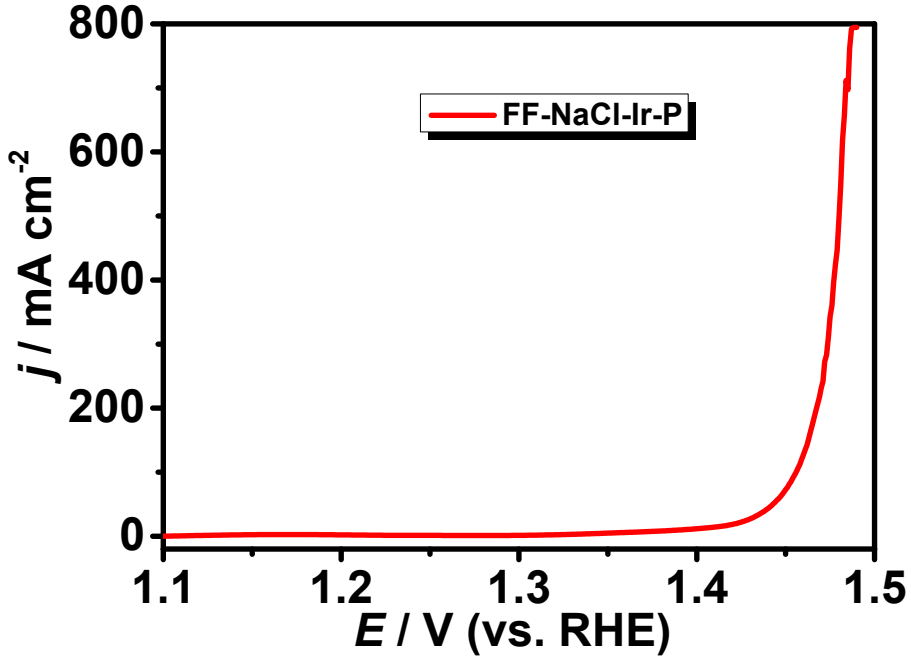

Figure S7. LSV of FF-NaCl-Ir-P

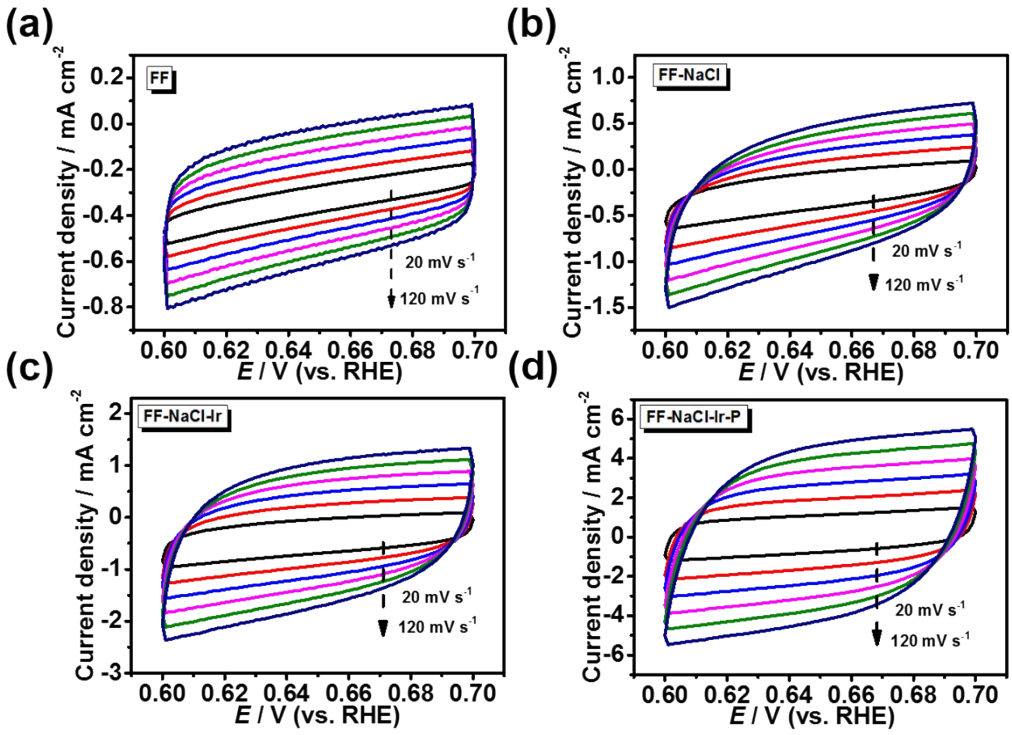

Figure S8. CV curves in the potential range from 0.6 V to 0.7 V at various scan rates in 1 M KOH solution: (a) FF, (b) FF-NaCl, (c) FF-NaCl-Ir and (d) FF-NaCl-Ir-P

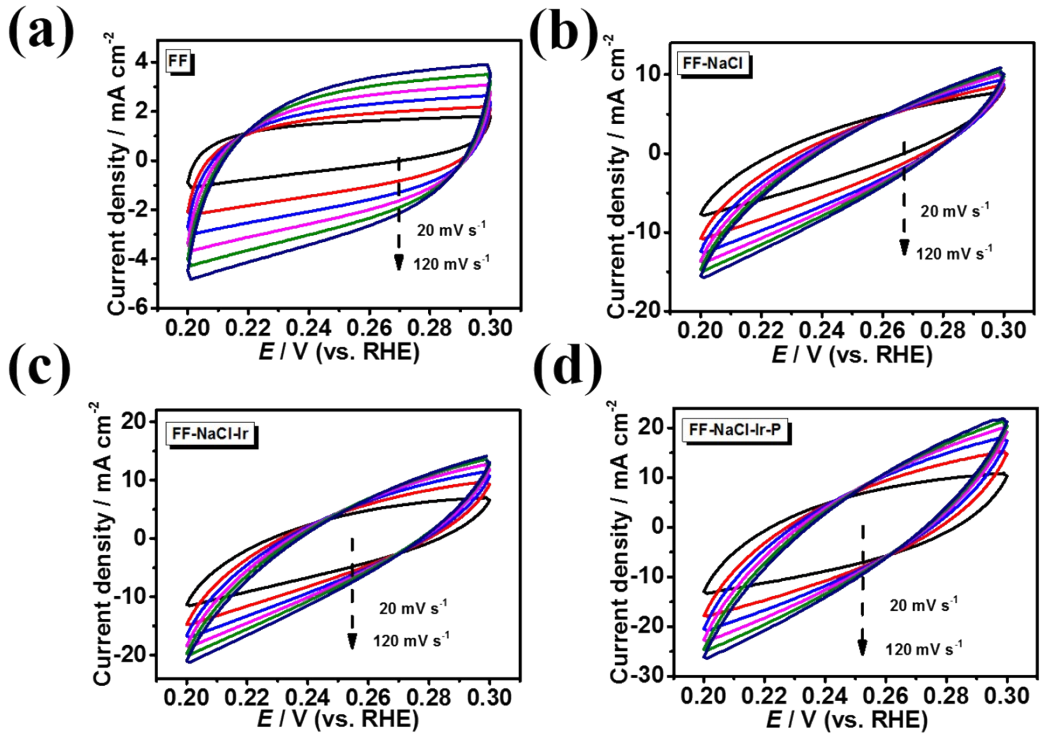

Figure S9. CV curves in the potential range from 0.2 V to 0.3 V at various scan rates in 1 M KOH solution: (a) FF, (b) FF-NaCl, (c) FF-NaCl-Ir and (d) FF-NaCl-Ir-P

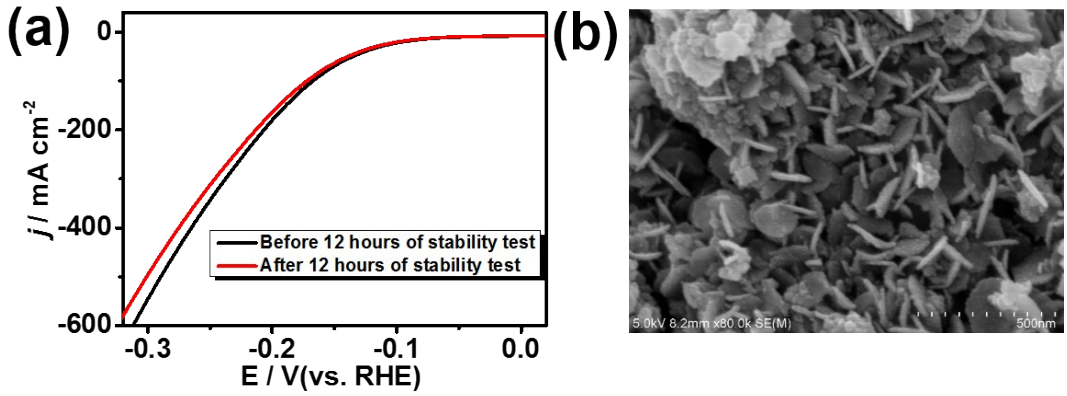

Figure S10. (a) LSVs of FF-NaCl-Ir-P before and after 12 hours chronopotentiometry measurement. (b) SEM image of FF-NaCl-Ir-P after 12 hours of HER.

Table S1. Comparative electrochemical OER performances of different electrocatalytic materials in alkaline medium

Catalysts	Electrolyte	Overpotential / mV (10 mA cm^{-2})	Ref.
FF-NaCl-Ir-P	**1.0 M KOH**	**169**	**This work**
NiCo-Ir-5	1.0 M KOH	240	1
CoIr-0.2	1.0 M KOH	235	2
Ir@Co nanosheets	1.0 M KOH	273	3
NiVIr LDH	1.0 M KOH	203	4
Ru/NiFe LDH-F/NF	1.0 M KOH	230	5
Ru_1Co_2NP	1.0 M KOH	240	6
Ir-NiCo-LDH	1.0 M KOH	192	7
$Fe_{1.0}CO_{1.1}Ni_{1.4}$-NC	1.0 M KOH	270	8
Fe_{MOFs}-SO_3	1.0 M KOH	218	9
Ru-NiFeP/NF	1.0 M KOH	179	10

Table S2. Comparative electrochemical HER performances of different electrocatalytic materials in alkaline medium

Catalysts	Electrolyte	Overpotential / mV (10 mA cm^{-2})	Ref.
FF-NaCl-Ir-P	**1.0 M KOH**	**69**	**This work**
Pt-αFe_2O_3/NF	1.0 M KOH	90	11
NiVIr LDH	1.0 M KOH	41	4
Ru/NiFe LDH-F/NF	1.0 M KOH	116	5
Ru_1Co_2NP	1.0 M KOH	188	6
Ru_2P	1.0 M KOH	57	12
Ru-WSe_2	1.0 M KOH	87	13
$Fe_{1.0}CO_{1.1}Ni_{1.4}$-NC	1.0 M KOH	175	8
Ru-NiFeP/NF	1.0 M KOH	56	10
NiFe-MOF-74	1.0 M KOH	195	14

Table S3. Comparative electrochemical overall water splitting performances of different electrocatalytic materials

Catalysts	Electrolyte	Overall Voltage (V) @ 10 mA cm^{-2}	Ref.
FF-NaCl-Ir-P	**1.0 M KOH**	**1.47**	**This work**
NiVIr LDH	**1.0 M KOH**	**1.49**	[4]
FeCoRuP	**1.0 M KOH**	**1.47**	[15]
Pt-αFe_2O_3/NF	**1.0 M KOH**	**1.51**	[11]
Ru/NiFe LDH-F/NF	**1.0 M KOH**	**1.53**	[5]
Ru_1Co_2NP	**1.0 M KOH**	**1.59**	[6]
np-IrO_2||IrAl	**0.5 M H_2SO_4**	**1.52**	[16]
$Fe_{1.0}CO_{1.1}Ni_{1.4}$-NC	**1.0 M KOH**	**1.52**	[8]
$IrCo_{0.14}$ NRs	**0.1 M $HClO_4$**	**1.53**	[17]
Ru-NiFeP/NF	**1.0 M KOH**	**1.47**	[10]
NiFe-MOF-74	**1.0 M KOH**	**1.58**	[14]

1. T. Huang, S. K. Moon and J.-M. Lee, *Electrochimica Acta*, 2019, **296**, 590-597.
2. Y. Zhang, C. Wu, H. Jiang, Y. Lin, H. Liu, Q. He, S. Chen, T. Duan and L. Song, *Advanced Materials*, 2018, **30**, 1707522.
3. D. D. Babu, Y. Huang, G. Anandhababu, X. Wang, R. Si, M. Wu, Q. Li, Y. Wang and J. Yao, *Journal of Materials Chemistry A*, 2019, **7**, 8376-8383.
4. S. Li, C. Xi, Y.-Z. Jin, D. Wu, J.-Q. Wang, T. Liu, H.-B. Wang, C.-K. Dong, H. Liu, S. A. Kulinich and X.-W. Du, *ACS Energy Letters*, 2019, **4**, 1823-1829.
5. Y. Wu, S. Ji, H. Wang, B. G. Pollet, X. Wang and R. Wang, *Applied Surface Science*, 2020, **528**, 146972.
6. Y. Bao, J. Dai, J. Zhao, Y. Wu, C. Li, L. Ji, X. Zhang and F. Yang, *ACS Applied Energy Materials*, 2020, **3**, 1869-1874.
7. R. Fan, Q. Mu, Z. Wei, Y. Peng and M. Shen, *Journal of Materials Chemistry A*, 2020, **8**, 9871-9881.
8. M. Khalid, A. M. B. Honorato, G. Tremiliosi Filho and H. Varela, *Journal of Materials Chemistry A*, 2020, **8**, 9021-9031.
9. K. Feng, D. Zhang, F. Liu, H. Li, J. Xu, Y. Xia, Y. Li, H. Lin, S. Wang, M. Shao, Z. Kang and J. Zhong, *Advanced Energy Materials*, 2020, **10**, 2000184.
10. Y. Lin, M. Zhang, L. Zhao, L. Wang, D. Cao and Y. Gong, *Applied Surface Science*, 2021, **536**, 147952.
11. B. Ye, L. Huang, Y. Hou, R. Jiang, L. Sun, Z. Yu, B. Zhang, Y. Huang and Y. Zhang, *Journal of Materials Chemistry A*, 2019, **7**, 11379-11386.
12. Z. Wu, D. Nie, M. Song, T. Jiao, G. Fu and X. Liu, *Nanoscale*, 2019, **11**, 7506-7512.
13. Y. Zhao, G. Mao, C. Huang, P. Cai, G. Cheng and W. Luo, *Inorganic Chemistry Frontiers*, 2019,

6, 1382-1387.

14. C. Chen, N. Suo, X. Han, X. He, Z. Dou, Z. Lin and L. Cui, *Journal of Alloys and Compounds*, 2021, **865**, 158795.
15. H. Liu, X. Li, L. Ge, C. Peng, L. Zhu, W. Zou, J. Chen, Q. Wu, Y. Zhang, H. Huang, J. Wang, Z. Cheng, Z. Fu and Y. Lu, *Catalysis Science & Technology*, 2020, **10**, 8314-8324.
16. Q. Li, J. Li, J. Xu, N. Zhang, Y. Li, L. Liu, D. Pan, Z. Wang and F. L. Deepak, *ACS Applied Energy Materials*, 2020, **3**, 3736-3744.
17. W. Gao, Q. Xu, Z. Wang, M. Wang, X. Ren, G. Yuan and Q. Wang, *Electrochimica Acta*, 2020, **337**, 135738.