Electronic Supplementary Information

Experimental section

Materials: Ethanol (C₂H₅OH) was bought from Chengdu Changlian Chemical Reagent Co., Ltd. (Chengdu, China). Potassium hydroxide (KOH) and sodium hydroxide (NaOH) were provided by Chengdu Kelong Chemical Reagent Factory China). Hydrochloric acid (HCl), nickel nitrate hexahydrate (Chengdu, (Ni(NO₃)₂·6H₂O), iron nitrate nonahydrate (Fe(NO₃)₃·9H₂O), ammonium fluoride $(NH_4F),$ urea $(CO(NH_2)_2),$ N,N-Dimethylformamide (DMF) and p-benzenedicarboxylic acid (C₈H₄O₄) were purchased from Chengdu KeLong Chemical Co. Ltd. (Chengdu, China). RuCl₃·3H₂O, and a Nafion (5 wt%) solution were obtained from Sigma–Aldrich Chemical Reagent Co., Ltd. (Shanghai, China). Nickel foam (NF) was purchased from Shenzhen Green and Creative Environmental Science and Technology Co., Ltd. (Shenzhen, China), and was pretreated in HCl, deionized water and ethanol several times to ensure the surface impurities removed. All above reagents and chemicals were used as received without further purification and the water used throughout all experiments was purified through a Millipore system.

Preparation of NiFe–LDH/NF: NiFe–LDH/NF was prepared according to previous work with minor modifications.¹ Firstly, 4.05 mmol Ni(NO₃)₂·6H₂O, 0.45 mmol Fe(NO₃)₃·9H₂O, 20 mmol CO(NH₂)₂, and 8 mmol NH₄F were added in 80 mL ultrapure water. After vigorous magnetically stirred for 10 min, as–formed pellucid solution was transferred to a 100 mL Teflon–lined stainless–steel autoclave with a piece of cleaned NF (4 cm × 6 cm). Secondly, the autoclave was sealed and maintained at 120 °C for 6 h in an electric oven and then cooled down naturally to room temperature. Thirdly, the NF covered with yellow green products was taken out and then washed with ultrapure water and ethanol several times before dring at 70 °C for 3 h in an electric oven. For comparison, NiFe_x–LDH on NF with varied Ni/Fe atomic ratios of 1.00/0.00, 0.95/0.05, and 0.80/0.20 were also prepared by adjusting the Ni²⁺/Fe³⁺ salt amount, ensuring the total mole number of Ni²⁺ added with Fe³⁺

ions is 4.5 mmol without any other variations, and the corresponding samples are denoted as Ni(OH)₂/NF, NiFe_{0.05}–LDH/NF, and NiFe_{0.20}–LDH/NF, respectively.

Preparation of Fe_{0.1}–**Ni**–**MOF/NF:** 3.5 mmol C₈H₄O₄, 4.725 mmol Ni(NO₃)₂·6H₂O, and 0.525 mmol Fe(NO₃)₃·9H₂O were dissolved in 70 mL DMF under vigorous stirring to form a uniform solution. Then, 5 mL C₂H₅OH and 5 mL H₂O were added in dropwise to the solution under constant stirring for 30 min. Then, the pellucid solution was transferred to a 100 mL Teflon–lined stainless–steel autoclave with a piece of NiFe–LDH/NF (4 cm × 6 cm). The autoclave was sealed and maintained at 120 °C for 12 h in an electric oven. After cooled down naturally to room temperature, the sample was taken out and washed with distilled water and ethanol several times, followed by drying at 70 °C for 3 h in vacuum. Ni–MOF on NF (Ni–MOF/NF) and Fe doped Ni–MOF on NF with varied Ni/Fe atomic ratios of 0.95/0.05 (Fe_{0.05}–Ni–MOF/NF) and 0.80/0.20 (Fe_{0.2}–Ni–MOF/NF) were prepared in a similar way.

Preparation of RuO₂: RuO₂ was prepared according to previous report.² Specifically, 2.61 g of RuCl₃· $3H_2O$ was dissolved in 100 mL ultrapure water under vigorous magnetic stirring for 10 min at 100 °C. Then 30 mL NaOH solution (1.0 M) was added into the above solution. After stirred for 45 min at 100 °C, the mixed solution was centrifuged for 10 min and filtered to collect the precipitates, which was washed with distilled water several times and the dried at 80 °C overnight. Finally, the precipitates were annealed at 350 °C for 1 h in air atmosphere to obtain crystalline RuO₂. As–prepared RuO₂ powder (0.01 g) was dispersed into a solution of Nafion, ethanol and ultrapure water with a volume ratio of 10/250/250 *via* sonication, and then deposited onto NF with a mass loading of 3.2 mg cm⁻².

Characterizations: X–ray diffraction (XRD) measurements were operated on a LabX XRD–6100 X–ray diffractometer with Cu K α radiation (40 kV, 35 mA) at a wavelength 1.54 Å (SHIMADZU, Japan) from 5° to 80°. Fourier transform infrared (FT–IR) transmission spectrum was collected on a Nicolet–6700 spectrometer using the potassium bromide pellet method. Raman spectrum was recorded on a LABRAM HR micro–Raman spectroscope with an excitation wavelength of 532 nm. Scanning electron microscope (SEM) images were recorded on a XL30 ESEM FEG scanning

electron microscope with an accelerating voltage of 20 kV. Cross–section SEM analysis was conducted on a JSM–7500F scanning electron microscope with an accelerating voltage of 20 kV. Transmission electron microscopy (TEM) images were collected on a HITACHI H–8100 electron microscopy (Hitachi, Tokyo, Japan) with an accelerating voltage of 200 kV. X–ray photoelectron spectroscopy (XPS) measurements were made on an AXIS Ultra DLD X–ray photoelectron spectrometer using Mg as the excitation source.

Electrochemical measurements: Electrochemical measurements were performed on a RST electrochemical workstation (Zhengzhou Shiruisi Technology Co., Ltd.) respectively using a Fe_{0.1}–Ni–MOF/NF (0.5×0.5 cm) as the working electrode, a platinum plate (1.5×1.5 cm) as the counter electrode and an Hg/HgO as the reference electrode in a standard three–electrode system. All potentials reported in this work were calibrated to RHE unless especially stated according to the following equation: E (RHE) = E (Hg/HgO) + ($0.098 + 0.0592 \ pH$) V. All electrochemical measurements were carried out at room temperature (298.15 K) *via* the adjustment of air condition and heating support, ensuring the variation of diffusion coefficient within 1%.



Fig. S1. EDX spectrum for Fe_{0.1}–Ni–MOF/NF.



Fig. S2. XPS survey spectrum for Fe_{0.1}–Ni–MOF.



Fig. S3. (a) LSV curves of $Fe_{0.1}$ –Ni–MOF/NF samples prepared at 100, 120, and 140 °C for 12 h with a scan rate of 2 mV s⁻¹ for water oxidation. (b) Tafel plots for Fe_{0.1}–Ni–MOF/NF samples prepared at 100, 120, and 140 °C for 12 h.



Fig. S4. (a) LSV curves of $Fe_{0.1}$ –Ni–MOF/NF samples prepared for 8, 12, and 16 h at 120 °C with a scan rate of 2 mV s⁻¹ for water oxidation. (b) Tafel plots for $Fe_{0.1}$ –Ni–MOF/NF samples prepared for 8, 12, and 16 h at 120 °C.



Fig. S5. (a) LSV curves of $Fe_{0.05}$ –Ni–MOF/NF, $Fe_{0.1}$ –Ni–MOF/NF, and $Fe_{0.2}$ –Ni–MOF/NF with a scan rate of 2 mV s⁻¹ for water oxidation. (b) Tafel plots for $Fe_{0.05}$ –Ni–MOF/NF, $Fe_{0.1}$ –Ni–MOF/NF, and $Fe_{0.2}$ –Ni–MOF/NF.



Fig. S6. LSV curve of Fe_{0.1}–Ni–MOF/NF with a scan rate of 2 mV s⁻¹ for water oxidation in 30 wt% KOH.



Fig. S7. (a) Low– and (b) high– magnification SEM images for $Fe_{0.1}$ –Ni–MOF/NF after durability test.



Fig. S8. XRD pattern for Fe_{0.1}–Ni–MOF/NF after durability test.



Fig. S9. XPS survey spectrum for Fe_{0.1}–Ni–MOF after durability test.



Fig. S10. XPS spectra for Fe_{0.1}–Ni–MOF/NF in the (a) Fe 2p, (b) Ni 2p, (c) C 1s, and

(d) O 1s regions after durability test.



Fig. S11. CVs for (a) Ni–MOF/NF and (b) $Fe_{0.1}$ –Ni–MOF/NF in the faradic capacitance current range at scan rates from 5 to 25 mV s⁻¹ (inset: the corresponding plots of oxidation peak current densites versus the scan rates from CVs) in 1.0 M KOH. (b) Plots for TOFs for Ni–MOF/NF and Fe_{0.1}–Ni–MOF/NF versus the overpotential.

 $j (\text{mA cm}^{-2})$ Catalyst η (mV) Electrolyte Ref. 50 243 Fe0.1-Ni-MOF/NF 100 263 200 284 1.0 M KOH This work 298 100 Ni-MOF/NF 200347 Ni-MOF/NF 100 320 1.0 M KOH 3 NiPc-MOF modified FTO 10 ≈390 1.0 M KOH 4 Ni(OH)2-TCNQ/CF 50 322 5 1.0 M KOH benzoate-Ni(OH)₂/NF 60 242 1.0 M KOH 6 ultrathin NiFe-MOF film 150 ≈330 0.1 M KOH 7 NiFe-MOF-74 rhombic 100 8 ≈390 1.0 M KOH crystals/NF 2D Ni-MOF@Fe-MOF 10 1.0 M KOH 9 265 MIL-53(FeNi)/NF 50 233 1.0 M KOH 10 Fe-MOF/NF 100 270 1.0 M KOH 11 Fe(TCNQ)₂/CF 50 353 1.0 M KOH 12 Fe(TCNQ)₂/Fe 10 340 1.0 M KOH 13 10 NiCo-POM/Ni 360 1.0 M KOH 14 10 NiCo-UMOFNs 250 1.0 M KOH 15 CoCd-BNN 1 353 0.1 M KOH 16 UTSA-16 408 10 1.0 M KOH 17 UTSA-16-derived Co₃O₄ 465 50 311 Co-MOF/NF 1.0 M KOH 18 15 19 Co(TCNQ)₂/Co 310 1.0 M KOH 25 276 20 Co(OH)2-TCNQ/CF 1.0 M KOH 15 MOF derived Co₃O₄ NA/CF 308 1.0 M KOH 21 50 291 22 benzoate-Co(OH)₂/NF 1.0 M KOH CuO-TCNQ/CF 25 1.0 M KOH 23 317 Cu-N-C NA/CF 20 314 1.0 M KOH 24 NiO@Ni-Bi/CC 10 290 1.0 M KOH 25 20 270 NiSe/NF 1.0 M KOH 26 Fe0.1-NiS2 NA/Ti 100 231 1.0 M KOH 27 Fe11.8%-Ni3S2/NF 100 253 1.0 M KOH 28 Zn-Ni₃S₂/NF 100 330 1.0 M KOH 29 NiFe-LDH/CNT 10 247 1.0 M KOH 30 NiFe-LDH@NiFe-Bi/CC 50 294 1.0 M KOH 31 100 32 Fe-NiSe/FeNi foam 264 1.0 M KOH

Table S1. Comparison of water oxidation performance for Fe_{0.1}–Ni–MOF/NF with other earth–abundant WOCs working under alkaline conditions.

NiFe/NF	20	264	1.0 M KOH	33
De-LNiFeP/rGO	10	258	1.0 M KOH	34
NiCo ₂ S ₄ @NiFe LDH/NF	60	201	1.0 M KOH	35
Co _{0.13} Ni _{0.87} Se ₂ /Ti	100	320	1.0 M KOH	36
Ni _{0.75} V _{0.25} –LDH	57	350	1.0 M KOH	37
Fe-NiCr ₂ O ₄ /NF	20	228	1.0 M KOH	38
NiOOH–NiCr ₂ O ₄ /NF	20	271	1.0 M KOH	39
NiCo ₂ S ₄ NA/CC	100	340	1.0 M KOH	40
NiCoP NSAs/NF	50	308	1.0 M KOH	41
Ni _{2.3%} –CoS ₂ /CC	100	370	1.0 M KOH	42
Ni–Co–S/CF	100	363	1.0 M KOH	43
Co ₃ O ₄ –MTA	150	360	1.0 M KOH	44
CoTe ₂ nanofleeces	10	357	0.1 M KOH	45
MnCo ₂ S ₄ NA/TM	50	325	1.0 M KOH	46
Al–CoP/NF	50	280	1.0 M KOH	47
Hollow Fe _{0.5} V _{0.5} spheres	10	390	1.0 M KOH	48
$(Co_{0.54}Fe_{0.46})_2P$	10	280	1.0 M KOH	49
FeOOH/CeO ₂ HLNTs–NF)	31.3	250	1.0 M NaOH	50
Co–B@CoO	10	290	1.0 M KOH	51
Fe-CoP/Ti	10	230	1.0 M KOH	52
P–Co ₃ O ₄ /NF	20	260	1.0 M KOH	53
Co ₃ O ₄ /CoMoO ₄ –50	10	318	0.1 M KOH	54
FeB_2	10	296	1.0 M KOH	55
Mn@Co _x Mn _{3-x} O ₄	10	246	1.0 M KOH	56
NF@NC–CoFe ₂ O ₄ /C NRAs	10	240	1.0 M KOH	57
CuCo ₂ S ₄ /CF	100	295	1.0 M KOH	58
Co–Cu ₂ S/CF	20	298	1.0 M KOH	59
	50	357		
Cu ₃ P@NF	10	~320	1.0 M KOH	60

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