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In-situ Ion-Exchange Preparation and Topological Transformation of Trimetal-

Organic Frameworks for Efficient Electrocatalytic Water Oxidation

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Experimental Section

Materials: Cobaltous nitrate hexahydrate (Co(NO₃)₂·6H₂O), iron nitrate (Fe(NO₃)₃·9H₂O), Nickel (II) nitrate hexahydrate (Ni(NO₃)₂·6H₂O), 2,6-Naphthalenedicarboxylic acid dipotassium (K-NDA), iridium (IV) oxide (IrO₂), KOH and ethanol were analytical grade and purchased from Adamas-beta. Nickel foam (NF) was obtained from Shengshijingxin New Material Corporation. All chemical reagents were used without any further treatment or purification.

Synthesis of NiCoFe-NDA nanosheets: In a typical fabrication procedure, a piece of the pre-treated nickel foam (thickness: 1.5 mm) was transferred into a vial containing 13 mg $Co(NO_3)_2 \cdot 4H_2O$, 22 mg $Fe(NO_3)_3 \cdot 9H_2O$ and 3 mL DI water, and maintained for 1 h at room temperature (the obtained product denoted as NiCoFe(OH)_x). Then, 30 mg of organic ligand (K-NDA) was added into the above solution and heated at 60 °C for 20 h. Finally, the products (NiCoFe-NDA/NF) were obtained by washing with ethanol and DI water sequentially and drying at 60 °C under vacuum. For comparison, the monometallic catalysts (Ni/Co/Fe-NDA/NF) and bimetallic NiFe/NiCo-NDA/NF were also synthesized by adjusting the species of added metal source. Noted that the Fe-NDA/NF was prepared under an inert atmosphere reaction system. To perform the different oxidation-reduction reaction times, a series of times including 0, 0.5 and 2 h were used.

Synthesis of NiCoFe-LDH nanosheets: 65 mg Co(NO₃)₂·4H₂O, 110 mg Fe(NO₃)₃·9H₂O, and 180mg urea were dissolved in 15 ml of water, stirred for 20 min and then 1×3cm NF was added and left for 1 h. Then the solution as well as NF was transferred to the Teflon-lined stainless-steel vessel (20ml) and hydrothermally heated at 120 °C for 12 h. The obtained products were washed using ethanol and water and then dried at 60 °C ambient.

Synthesis of IrO_2/NF electrode: IrO_2 (5.0 mg) were dispersed in the solution containing ethanol (980 µL), and 5 wt% Nafion solution (20 µL) with ultrasonic treatment for 1 h. Then, 20 µL of above suspension was dropped on the NF electrode with a size of 1.0 cm × 2.0 cm and then dried at room temperature.

Characterization:

The morphology and microstructure of the samples were obtained by using field-emission scanning electron microscopy (FESEM, Quanta FEG450) and transmission electron microscopy (TEM, FEI, TECNAI F30). Energy dispersive X-ray spectroscopy (EDS) attached to the FETEM was utilized to investigate compositions of the samples. The phase structure of the as-prepared samples was analyzed by X-ray diffraction (XRD, Bruker D8-Advance) at a scanning rate of 2° min⁻¹ in the 20 ranges from 5 to 50. X-ray photoelectron spectroscopy (XPS, ESCALAB250Xi, Thermo) was conducted to investigate the chemical composition of the samples. The Raman spectrum (Raman Station 400F system) of as-prepared samples was conducted at 532 nm laser. FT-IR spectra were recorded in the range of 4000-500 cm⁻¹ with a SPE CTRUM 100 spectrophotometer. The Ni, Co and Fe contents were analyzed by Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES, PerkinElmer Optima 2100DV).

XAS measurements

The X-ray absorption fine structure spectra Fe K-edge were collected at BL14W1 beamline of Shanghai Synchrotron Radiation Facility (SSRF) while Co and Ni K-edge were collected at 1W1B beamline of Beijing Synchrotron Radiation Facility (BSRF). The data were collected in fluorescence mode using a Lytle detector while the corresponding reference sample was collected in transmission mode. The sample were grinded and uniformly daubed on the special adhesive tape.

Electrochemical measurements

The electrochemical measurements were performed with a CHI 760E electrochemical workstation (CH Instruments, Inc., Shanghai) in a standard three-electrode electrochemical cell with modified NF electrode as working electrode directly, Ag/AgCl electrode as reference electrode and Pt plate as counter electrode, respectively. All potentials in this study were given versus reversible hydrogen electrode (RHE) according the following equations: $E_{RHE} = E_{Ag/AgCl} + 0.197 + 0.0591 \text{pH}$, where $E_{Ag/AgCl}$ is the experimentally measured potential against the Ag/AgCl reference electrode. 1.0 M KOH electrolyte was treated by oxygen (for OER) bubbles before the test. The linear sweep voltammetry (LSV) curves were collected at a scan rate of 2 mV s⁻ ¹. The Tafel slope was obtained by fitting the linear portion of the Tafel plots to the Tafel equation $[\eta =$ b*log(j)+a]. Electrochemical impedance spectroscopy (EIS) for OER were tested in a frequency range of 100 kHz-0.01 Hz at the potential 0.5 V vs. RHE. All polarization curves were corrected by the iR drop compensation. Cyclic voltammetry (CV) was tested at different scan rates to estimate the double-layer capacitances (C_{dl}) of the catalysts. Moreover, the electrochemical active surface area (ECSA) was estimated Cdl based on the following equation: ECSA = \overline{Cs} , where C_s value of 0.04 mF cm⁻² was thus adopted according to previously reported Co-based catalysts. The long-stability of catalysts was also tested by chronopotentiometry, which was carried out at constant overpotential of 100 mA cm⁻² for 50 hours.

In situ Raman characterization:

For the *In-situ* Raman spectra study, all the spectroscopy experiments were measured at a Confocal Renishaw Micro-Raman Spectroscopy System. Use the same electrode configuration as the previous electrochemical test except that the electrolyte is changed to 0.1 M KOH to reduce the impact of bubbles on spectra collection during the OER process. Each spectrum was accumulated twice at 532 nm with 5 % power of laser intensity

and 20 s exposure time. Open circuit voltage Raman spectra is the spectra collected by the sample directly immersed in 0.1 M KOH. *In Situ* Raman spectra were collected using chronoamperometry at 1.03-1.63V vs. RHE without *iR* drop compensation.

Alkaline anion exchange membrane water electrolysis (AEMWE) test

The AEMWE system consists of the anode, cathode, gas diffusion layer and anion exchange membrane (AEM, X37-50 Grade T, Dioxide Materials). The direct grown NiCoFe-NDA on Ni foam (thickness: 0.15 mm) used as anode electrode. The cathode electrode was prepared by the catalyst-coated substrate method. Pt/C (20 wt.%) was used as the electrode for the hydrogen evolution reaction (HER) and loaded 3 mg cm⁻² on Ni foam. The gas diffusion layer (GDL) was prepared by pressing Ni foam. The fastening pressure of the AEMWE cell is 20 N/m at each of the 8 bolts. The performance of an AEMWE with an active area of 2 cm \times 2 cm was evaluated through CV in the range of 1.2 to 2.4 V cell at a scan rate of 10 mV/s and stability test at different potential (1.8 and 2.0 V) in 0.1 M KOH at 50 °C.

DFT calculation

The density-functional theory (DFT) calculations were performed with the Vienna Ab-initio Simulation Package (VASP) codes 5.4.4¹⁻³. The projected augmented wave (PAW) pseudo-potentials, the revised Perdew-Burke-Ernzerhof (RPBE) exchange-correlation functional, and a plane wave cutoff of 500 eV have been used in the calculations⁴⁻⁶. A vacuum spacing of at least 14 Å are constructed and 4 * 2 * 1 Monkhorst-Pack k-point mesh was sampled for the calculations. Spin-polarized calculations were identified for all surfaces. The convergence of energy and force were set to be 10⁻⁵ eV and 0.03 eV/Å, respectively. Van der Waals correction was considered by introducing the DFT-D3 method⁷⁻⁸.

Figures and Tables



Figure S1. (a) Image of reactor in each step during the synthesis process; (b) Image of pristine NF, NiCoFe(OH)_x/NF and NiCoFe-NDA/NF.



Figure S2. SEM image of NiCoFe-NDA/NF with different oxidation-reduction time: (a) 0 h; (b) 0.5 h; (c)2h.(d) LSV, (e) corresponding Tafel plots and (f) Nyquist plots of NiCoFe-NDA-0h/0.5h/1h/2h.

Note: 2D nano-scaled morphology have the positive role for OER since high percentages of exposed active atoms guarantee high catalytic activity and rapid mass transport and charge transfer. Thus, we studied the effects of various experimental parameters on the unique morphology and found that the redox reaction at the initial stage of preparation is the key factor in the morphology of this unique 2D nanosheet. At an excessive oxidation-reduction time (2h), the obtained sample exhibits different sizes of thick sheets. At a suitable time (1h), the target products are obtained, this is, ultrathin NiCoFe-NDA nanosheets. At a halved hydrolysis time (0.5h), the resulting product presents small-sized nanoflakes with the orthogonal arrangement. Without additional reaction time (0h), the sample displays dense nanorods vertically growing on the NF surface. The corresponding LSV curves, Tafel, and EIS results are shown in Figure S2, 2D MOF catalyst displays the superior electrocatalytic activity, which can be attributed to the desirable merits of nanosheets structure such as high percentages of exposed active atoms as well as nanometer thickness.



Figure S3. XRD patterns of (a) NiCoFe(OH)_x/NF, (b) NiCoFe-LDH/NF.



Figure S4. SEM images (a-c) and EDX spectra (d-f) of Ni-NDA (a, d), Co-NDA (b, e), and Fe-NDA (c, f).



Figure S5. (a, b) SEM image of NiCoFe(OH)_x; (c, d) SEM and (e, f) TEM images of NiCoFe-NDA.



Figure S6. Raman spectra of NiCoFe-NDA and Ni/Co/Fe-NDA.



Figure S7. The Brunauer-Emmett-Teller (BET) surface area and pore volume of the Ni/Co/Fe-NDA and NiCoFe-NDA.



Figure S8. XPS spectra for (a) Survey, (b) C 1s, (c) O 1s for NiCoFe-NDA and Ni/Co/Fe-NDA.



Figure S9. Contact angle measurements of (a) NiCoFe-NDA, (b) Fe-NDA, (c) Co-NDA, and (d) Ni-NDA.



Figure S10. CV curves at different scan rates for (a) NiCoFe-NDA/NF, (b) Ni-NDA/NF, (c) Co-NDA/NF, (d) Fe-NDA/NF, (e) NiCoFe-LDH/NF, (f) IrO₂/NF.



Figure S11. (a) LSV curves, (b) Tafel slopes, (c) Nyquist plots of NiCoFe-NDA/NF, NiFe-NDA/NF, and NiCo-NDA/NF.



Figure S12. (a) LSV curves, (b) Tafel slopes, (c) Nyquist plots, (d) C_{dl} values and (e) ECSA-normalized LSV curves of NiCoFe-NDA/NF, NiCoFe-LDH/NF and IrO₂/NF.



Figure S13. Polarization curves of PEM cell using NiCoFe-NDA on Ni foam (Inset: the PEM cell photograph).



Figure S14. In situ Raman spectra: (a) Ni-NDA, (b) Co-NDA and (c) Fe-NDA.



Figure S15. (a-b) SEM images of the t-NiCoFe-NDA. (c) TEM images, (d) HRTEM images of the t-NiCoFe-

NDA. (e-g) The intensity distribution profile of the atoms for the Figure 5b inset.



Figure S16. EDX spectra of t- NiCoFe-NDA



Figure S17. High-resolution XPS spectra in (a) Survey, (b) Ni 2p, (c) Co 2p, and (d) Fe 2p of t-NiCoFe-NDA and t-NiCoFe-NDA. (e) The ratio of Ni²⁺/Ni³⁺ and Co²⁺/Co³⁺ in XPS of t-NiCoFe-NDA and t-NiCoFe-NDA.



Figure S18. Details of different coordination number during OER process and calculated volcano plot. (a) 4coordination, (b) 5-coordination, (c) 6-coordination. (d)The PDOS of t-NiCoFe-NDA, t-Ni-NDA, t-Co-NDA and t-Fe-NDA. The solid line in the figure represents t-NiCoFe-NDA, and the dashed lines in three colors represent t-Ni-NDA, t-Co-NDA and t-Fe-NDA respectively.



Figure S19. Differential charge density of MOF under different coordination number. (a) 4-coordination, (b) 5-coordination, (c) 6-coordination.



Figure S20. Primitive steps of the OER process on the surface of t-M-NDA. (*) adsorption step, (*OH and *O) dissociation steps, and (*OOH) desorption step during the OER process.



Figure S21. (a-c) Fourier-transformed magnitude in R space for NiCoFe-NDA. (d-f) *K*²-weighted spectra in K space for NiCoFe-NDA. (g-i) Fourier-transformed magnitude in R space for Ni-NDA Co-NDA and Fe-NDA. The best fit parameters are shown in Supplementary Tables S3. For all samples, the measured and calculated spectra closely matched.

	EDS	ICP-OES
Ni	2.13	9.8
Co	1.04	5.4
Fe	0.87	5.9
С	77.83	/
Ο	18.02	/

 Table S1. The percentage of elements for NiCoFe-NDA.

	EDS	ICP-OES
Ni	3.35	14.9
Co	3.41	15
Fe	3.51	11.5
С	51	/
0	37.18	/

 Table S2. The percentage of elements for t-NiCoFe-NDA.

Sample	Path	Ν	R(Å)	$\sigma^2 \times 10^{-3}$ (Å2)	$\Delta E_0 (eV)$	R-factor	
NiCoFe-NDA	Ni-O	6.3 +/- 0.7	2.069+/-0.026	1.7+/-0.8	1.49+/- 1.17	0.0276	
Ni site	Ni-C	1.8 +/- 0.2	2.941+/-0.038	4.1+/-2.1	4.37+/-2.16	- 0.0376	
NiCoFe-NDA	Co-O	5.7 +/- 0.9	2.068+/-0.027	3.3+/-0.8	2.73+/- 1.51	0.0496	
Co site	Co-C	2.3 +/- 0.3	2.973+/-0.039	3.9+/-1.7	3.71+/-2.03	- 0.0486	
NiCoFe-NDA	Fe-O	5.9 +/- 1.1	2.029+/-0.066	3.3+/-1.0	2.03+/- 1.77	0.0529	
Fe site	Fe-C	2.4 +/- 0.3	3.001+/-0.098	2.3+/-1.1	3.22+/-2.12	- 0.0538	
Ni-NDA Ni site	Ni-O	6.8	2.05+/-0.01	5.1+/-0.9	-1.8+/-2.0	0.0191	
Co-NDA Co site	Co-O	6.7+/-1.0	2.08+/-0.01	7.5+/-1.8	-0.8+/-1.8	0.0182	
Fe-NDA Fe site	Fe-O	4.7	1.96+/-0.01	8.7+/-1.1	-1.5+/-2.6	0.0162	

Table S3. The fitting results of Ni/Co/Fe K-edge EXAFS spectra for NiCoFe-NDA and reference sample.

N, coordination number; R, distance between absorber and backscatter atoms; ΔE_0 , inner potential correction to account for the difference in the inner potential between the sample and the reference compound. σ^2 , Debye– Waller factor; S₀² fitting from Fe sample defined as 0.82.

Table S4. Summary of the electrocatalytic OER activity of the previously reported TMs MOF and MOFs-

derived composites catalysts.

Cuthert	F1 to - 1 to		Tafel slope	Deferrere	
Catalyst	Electrolyte	Overpotential (mv)	(mV dec ⁻¹)	Kelerence	
		$215@10 \text{ mA cm}^{-2}$		This work	
NiCoFe NDA/NF	I M KOH	256@50 mA cm ⁻²	64.1		
(Ni ₂ Co ₁) _{0.925} Fe _{0.075} -					
MOF	1 M KOH	257@10mA cm ⁻²	41.3	9	
NiCoFeP/C	1M KOH	270@10mA cm ⁻²	65	10	
NiCo/Fe ₃ O ₄ /MOF-74	1 M KOH	238@10mA cm ⁻²	29	11	
NCF-MOF@Ni foam	1 M KOH	280@30mA cm ⁻²	49	12	
Ni-MOF@Fe-MOF	1 M KOH	265@10mA cm ⁻²	82	13	
FN-2	1 M KOH	$316@50 \text{ mA cm}^{-2}$	56.7	14	
NiFe-NFF	1 M KOH	253@100mA cm ⁻²	38.9	15	
NiFe-MOF-74	1 M KOH	223@10mA cm ⁻²	71.6	16	
FeNi ₃ N/NF	1 M KOH	202@10mA cm ⁻²	40	17	
MIL-53(FeNi)/NF,	1 M KOH	233@50mA cm ⁻²	31.3	18	
MIL-53(Co-Fe)/NF	1 M KOH	262@100mA cm ⁻²	69	19	
Fe(OH) ₃ @Co-MOF-		202@10m A am ⁻²	11	20	
74	I M KUH	292@10MA cm ⁻²	44	20	
CTGU-10c2	1 M KOH	240@10mA cm ⁻²	58	21	
NiCoP/NC PHCs	1 M KOH	297@10mA cm ⁻²	51	22	

 Table S5. Comparison of state-of-the-art AAEMWE performance reported in the literature. (Cell operation

 conditions: temperature= 50 °C, cell voltage =1.8V).

Anode/cathode	E _{onset} (V)	$j_{1.8V}$ (mA cm ⁻²)	Electrolyte	Ref.
Pt/C NiCoFe-NDA	1.34	325	0.1 M KOH	This work
Pt/C IrO ₂	1.52	200	1.0 M KOH	23
NiCo ₂ O ₄ -HSp NiCo ₂ O ₄ -HSp	1.39	150	1.0 M KOH	24
Pt/C Cu _{0.81} Co _{2.19} O ₄	1.375	341	0.1 M KOH	25
Pt/C PISPVA 46	1.44	320	0.5 M KOH	26
$Cu_{0.81}Co_{2.19}O_4 NS \parallel 3-Co_3S_4 NS$	1.55	115	1.0 M KOH	27
Pt-Ni Ni	1.56	162	1.0 M KOH	28
Pt/C LDPE	1.5	299	1.0 M KOH	29

coordination		$\Delta G1(* to$	∆G2(*OH	ΔG3(*O	$\Delta G4(*OOH$	
number	site	*OH)	to *O)	to *OOH)	to *)	η (ν)
4 —	Fe-NDA	0.834	1.232	1.965	0.889	0.735
	CoNDA	0.994	1.411	1.724	0.791	0.494
	Ni-NDA	1.176	1.848	1.237	0.659	0.618
	NiCoFe-NDA (Fe site)	0.780	1.102	1.787	1.251	0.557
	NiCoFe-NDA (Co site)	1.139	1.351	1.521	0.909	0.291
	NiCoFe-NDA (Ni site)	1.342	1.784	0.931	0.863	0.554
5	Fe-NDA	1.729	1.897	1.204	0.090	0.667
	CoNDA	1.709	1.604	1.396	0.211	0.479
	Ni-NDA	2.074	1.838	1.135	-0.127	2.074
	NiCoFe-NDA (Fe site)	1.813	1.884	1.191	0.032	0.654
	NiCoFe-NDA (Co site)	1.667	1.628	1.365	0.260	0.437
	NiCoFe-NDA (Ni site)	2.024	1.836	1.112	-0.052	0.794
6 —	Fe-NDA	2.236	1.642	1.193	-0.151	1.006
	CoNDA	2.276	1.828	1.161	-0.345	1.046
	Ni-NDA	2.425	1.660	1.129	-0.294	1.195
	NiCoFe-NDA (Fe site)	1.803	1.615	1.208	0.294	0.573
	NiCoFe-NDA (Co site)	2.145	1.695	1.219	-0.139	0.915
	NiCoFe-NDA (Ni site)	2.423	1.672	1.134	-0.309	1.193

Table S6. Details of different coordination number OER process

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