

Electronic Supplementary Information (ESI) for Chemical Communications
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Supporting Information

Activating FeMoO₄ nanosheet arrays by partial nickel substitution for efficient electrocatalytic seawater oxidation

Jun-Ya Gao,^{a,#} Yin-Lei Ma,^{a,#} Guang-Sheng Qian,^a Meng-Ying Si,^a Ling-Li Han,^a and
Ji-Sen Li^{a,b,*}

^aSchool of Chemistry, Chemical Engineering and Materials, Jining University, Qufu
273155, P. R. China

^bKey Laboratory of Advanced Energy Materials Chemistry (Ministry of Education),
Nankai University, Tianjin 300071, P. R. China

[#]These authors contributed equally to this work.

*Corresponding authors. E-mail: senjili@sina.com (J.-S. Li)

EXPERIMENTAL SECTION

Synthesis of FNMO/NF. In typical procedure, a piece of NF (1 cm × 3 cm) was pre-treated sequentially with hydrochloric acid, acetone, and deionized water, respectively. Then, 50 mL deionized water consisting of 811 mg FeCl₃·6H₂O, 242 mg L-cysteine, and 618 mg Na₂MoO₄·2H₂O was prepared by ultrasonication and transferred into a Teflon reactor. Next, the treated NF was soaked into the mixture and heated at 170 °C for 12 h. The Fe_xNi_{1-x}MoO₄ (named FNMO/NF) was obtained by washing with ethanol and water for several times, and then dried at 60 °C in a vacuum oven.

Synthesis of FMO/NF and NMO/NF. The fabrication of FMO/NF and NMO/NF are similar to that of FNMO/NF apart from using FeCl₂·4H₂O and NiCl₂·6H₂O instead of FeCl₃·6H₂O, respectively.

Synthesis of Pt/C/NF and IrO₂/NF. 20 mg of commercial Pt/C was added into a mixture (1930 μL ethanol and 70 μL Nafion) and thus form a uniform ink by ultrasonication. Then, a certain amount of the ink was coated on a piece of NF (1 cm × 1 cm) with a loading mass of 1.4 mg cm⁻². IrO₂/NF was also prepared via the same way.

Instruments. Power X-ray diffraction (PXRD) patterns were achieved by D/max/2500PC. Scanning electron microscope (SEM, Regulus 8100), high-resolution transmission electron microscopy (HRTEM, JEM-F200), thermogravimetric analysis (TG, PE DSC8500) and X-ray photoelectron spectroscopy (XPS, PHI 5000 Versa) were performed to examine the morphology and component of the resultant materials.

Electrochemical Measurements. The Electrochemical measurements were performed on an electrochemical workstation (CHI 760E) with a three-electrode setup, wherein the self-supported catalyst was served as the working electrode, a graphite rod as the counter electrode, and the reversible hydrogen electrode as reference electrode, respectively. The linear sweep voltammetry (LSV) tests were carried out at a scan rate of 2 mV s⁻¹ in 1 M KOH, 1 M KOH + 0.5 M NaCl, and 1.0 M KOH + seawater electrolytes toward the OER, respectively. The double-layer capacitance (*C_{dl}*) was assessed according to cycle voltammetry (CV) curves with different scan rates from 20 to 100 mV s⁻¹.

S1. Figures in Supporting Information

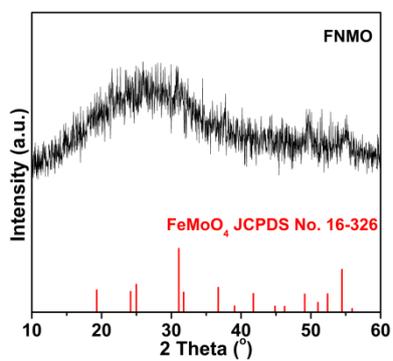


Fig. S1 PXRD pattern of FNMO scraped from NF.

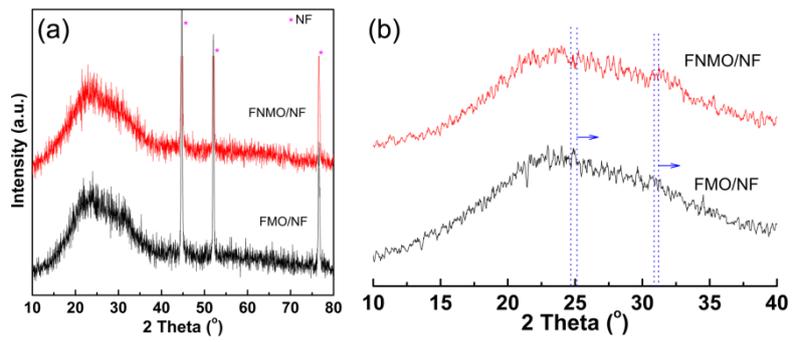


Fig. S2 (a) XRD patterns and (b) the magnified patterns of FNMO/NF and FMO/NF.

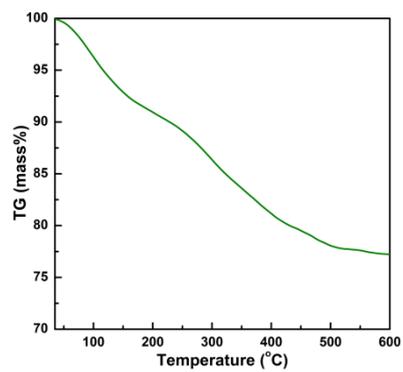


Fig. S3 TG profile of FNMO.

When FNMO is heated in the temperature range of 35-600 °C (Fig. S3, ESI†), the mass loss is ascribed to the removal of water (35-180 °C) and phase transformation of MoO_x and FeO_x oxides (180-600 °C).

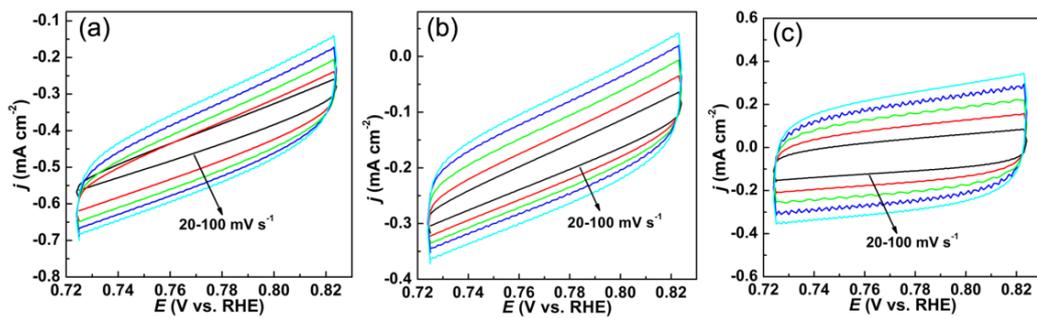


Fig. S4 (a-c) Cyclic voltammograms of NMO/NF, FMO/NF, and FNMO/NF, respectively.

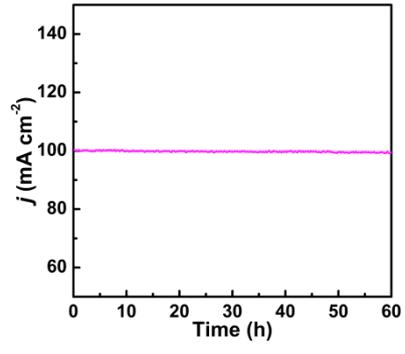


Fig. S5 Stability test of FNMO/NF in 1 M KOH.

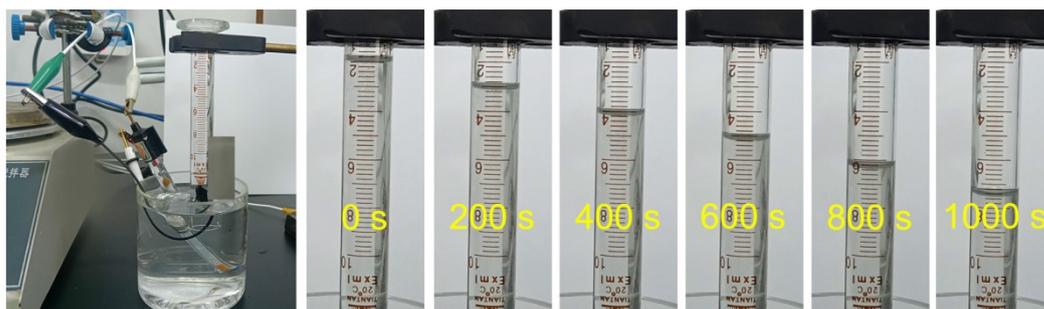


Fig. S6 Gas collection device toward the OER and digital photographs of O₂ produced at different time.

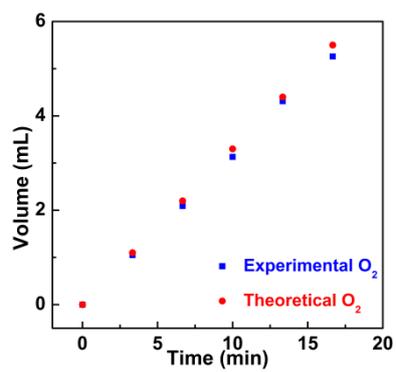


Fig. S7 Faraday efficiency of FNMO/NF toward the OER.

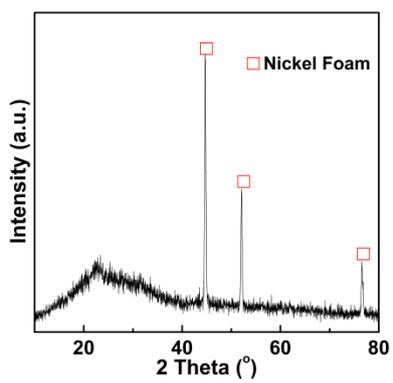


Fig. S8 XRD of FNMO/NF after stability testing.

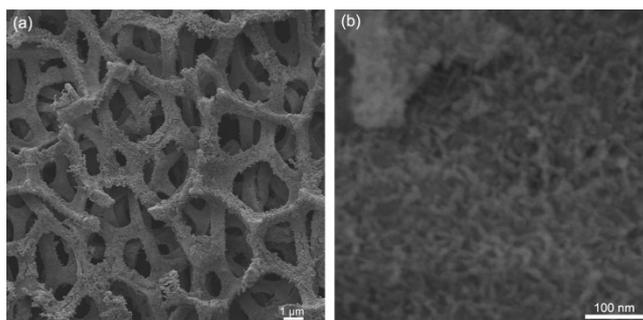


Fig. S9 SEM image of FNMO/NF after stability testing.

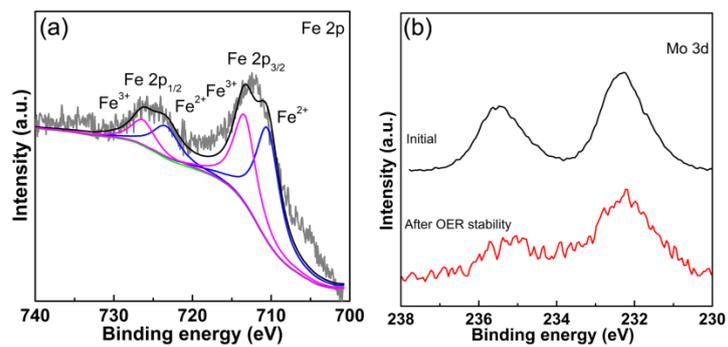


Fig. S10 High-resolution XPS spectrum of Fe 2p (a) and Mo 3d (b) for FNMO after stability testing.

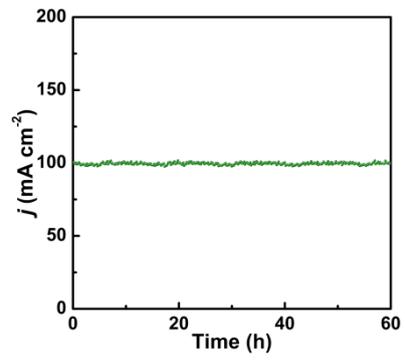


Fig. S11 Stability tests of FNMO/NF in 1 M KOH + seawater.

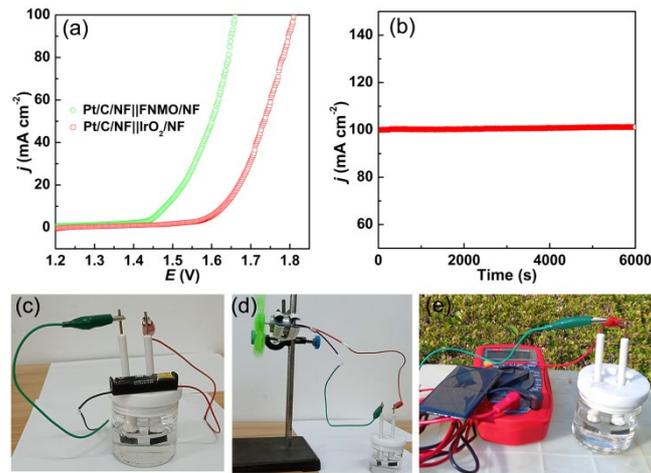


Fig. S12 (a) LSV curves of Pt/C/NF||FNMO/NF and Pt/C/NF||IrO₂/NF for overall seawater splitting and (b) long-term durability test at a voltage of 1.66 V in 1 M KOH + seawater. (c-e) 1.5 V battery, wind, and solar energy drive the overall seawater splitting.

S2. Tables in Supporting Information

Table S1. Comparison of catalytic OER performance of FNMO/NF and other reported catalysts in 1 M KOH solution.

Catalyst	Overpotential (mV) at 10 mA cm ⁻²	Overpotential (mV) at 100 mA cm ⁻²	Tafel (mV dec ⁻¹)	Reference
FNMO/NF	215	251	32.8	This work
NiFe-Ppy	229	268	38	Angew. Chem. Int. Ed. 2024, e202409628
Fe/P-NiMoO ₄	213	327	/	Appl. Catal. B Environ. Energy 2024, 347, 123805.
FeMoO ₄ /NF	/	263	39	Nano Res. 2024, 17, 2270-2275.
60Fe/NF	/	340	57	Adv. Energy Mater. 2023, 13, 2301921.
Ru-Ni(Fe)P ₂ /NF	/	251	91.6	Small 2023, 19, e2300030.
P-Mo-Co ₃ O ₄ @CC	276	315	53.9	Carbon Energy. 2022; 1, 14.
MoNiFe-27%	242	280	23	Nat. Commun. 2022, 13, 2191.
NiFe LD-PMo12	206	249	47.5	Adv. Mater. 2022, 34, 2110696.
CF-FeSO	192	230	40.1	Nat. Commun. 2022, 13, 605.
CoNiFeCu	291	345	43.9	Adv. Mater. 2022, 2109108.
CoOOH/CoS	240	360	86.4	Angew. Chem. Int. Ed. 2022, 61, e202117178.
Ni-Mo-B HF	293	290	79	Adv. Funct. Mater. 2021, 2107308.
FeP-CoP/NC	230	390	73	Nat. Commun. 2021, 12, 4143.
NiCoFe-NDA/NF	215	270	50.7	Energy Environ. Sci. 2021, 14, 6546.
NiFe LDH/NiS ₂	220	386	60.1	Adv. Energy Mater. 2021, 2102353.
CoCu-MOF NBs	260	320	63.5	Angew. Chem. Int. Edit. 2021, 60, 26397.
MOF-Fe/Co	220	300	52	Angew. Chem. Int. Edit. 2021, 60, 12097.
NiCo _{2x} Fe _x O ₄ NBs	274	308	42	Angew. Chem. Int. Edit. 2021, 60, 11841.
NiCe@NiFe	220	254	59.9	Appl. Catal. B Environ. 2020, 260, 118199.

Table S2. Comparison of catalytic OER performance of FNMO/NF and other reported catalysts in 1 M KOH + seawater.

Catalyst	Overpotential (mV) at 100 mA cm ⁻²	Reference
FNMO/NF	269	This work
FeMoO ₄ /NF	303	Nano Res. 2024, 17, 2270.
Fe-doped Ni&Ni _{0.2} Mo _{0.8} N	234	Energy Environ. Sci. 2022, 15, 3945.
NiCoHPi@Ni ₃ N/NF	396	ACS Appl. Mater. Interfaces 2022, 14, 22061.
Ni(OH) ₂ -TCNQ/GP	382	Nano Res. 2022, 15, 6084.
Mo-Co _x P	520	Mater. Today Nano 2022, 18, 100216.
NiMoO ₄ @NiFe-LDH	251	Sustain. Energ. Fuels. 2022, 6, 5521.
BZ-NiFe-LDH/CC	300	Nano Res. Energy 2022, 1, e9120028.
S-NiMoO ₄	315	J. Colloid Interf. Sci. 2022, 613, 349.
Fe-Co ₃ (PO ₄) ₂ ·4H ₂ O	287	Chem. Commun. 2022, 58, 6761.
CoPx@FeOOH	290	Appl. Catal. B Environ. 2021, 294, 120256.
NiMoN@NiFeN/NF	307	Nat. Commun. 2019, 10, 10.
NiCoS/NF	360	Appl. Catal. B Environ. 2021, 291, 120071.