Electronic Supplementary Material (ESI) for Catalysis Science & Technology.

Supplementary Information

Facilitating active NiOOH formation via Mo doping towards high-efficiency oxygen evolution

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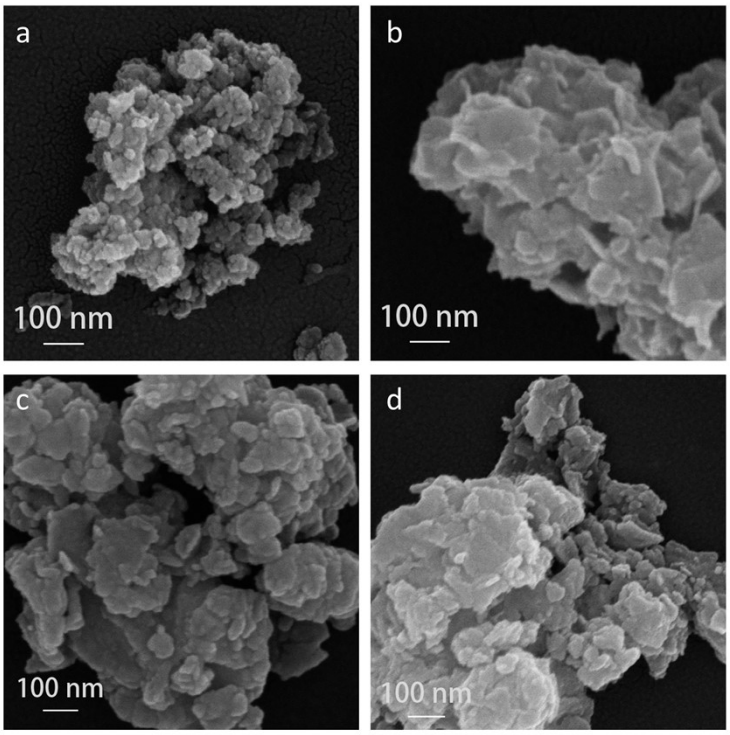

Fig. S1 SEM images of NiFe-LDH, NiFeMo-1, NiFeMo-2 and NiFeMo-3.

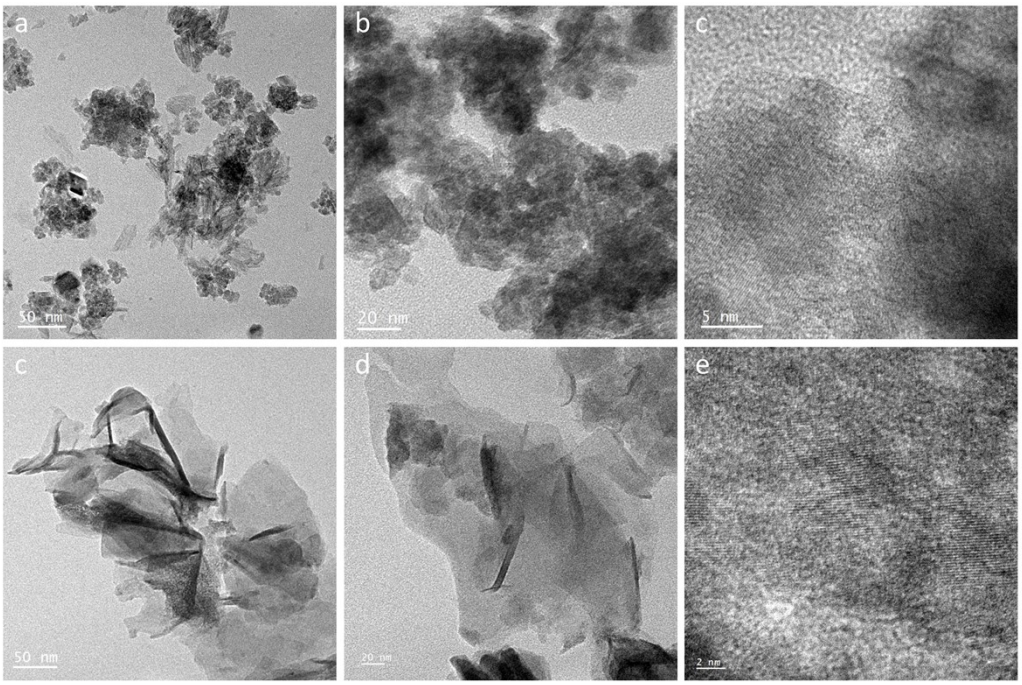

Fig. S2 TEM images of (a-c) NiFe-LDH and (c-e) NiFeMo-2.

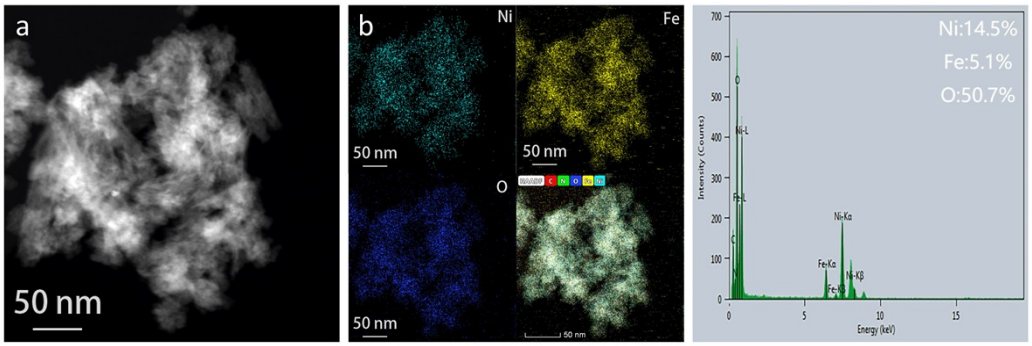

Figure S3. (a) HAADF-STEM image and (b, c) element mapping analysis for NiFe-LDH.

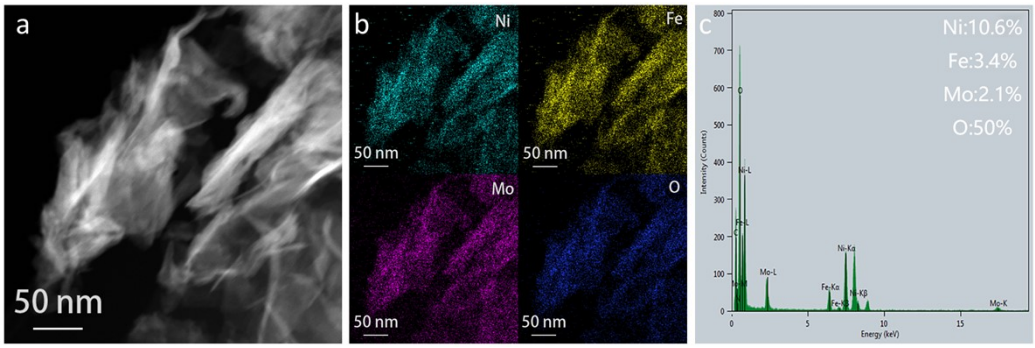

Fig. S4 (a) HAADF-STEM image and (b, c) element mapping analysis for NiFeMo-2

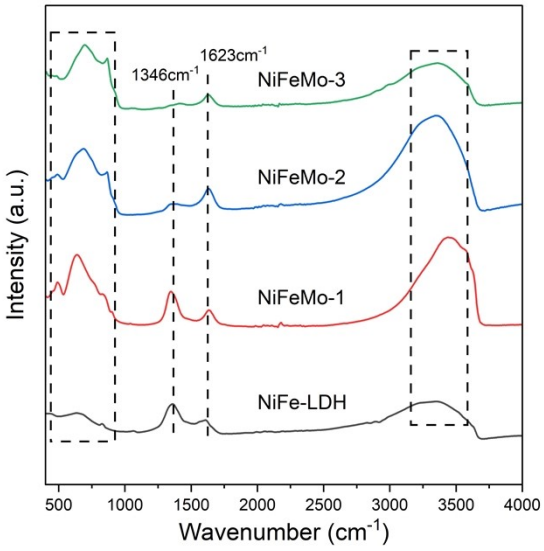

Fig. S5 FTIR spectra of NiFe-LDH, NiFeMo-1, NiFeMo-2 and NiFeMo-3.

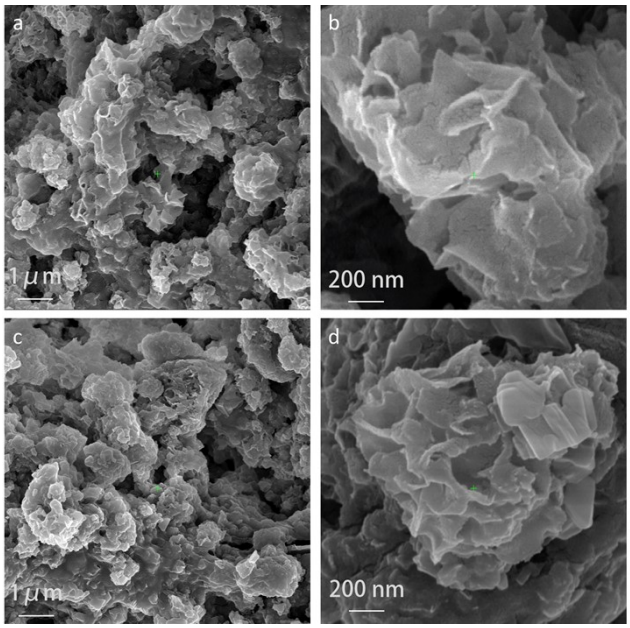

Fig. S6 SEM images of NiFeMo-2 catalyst (a, b) before stability test and (c, d) after stability test.

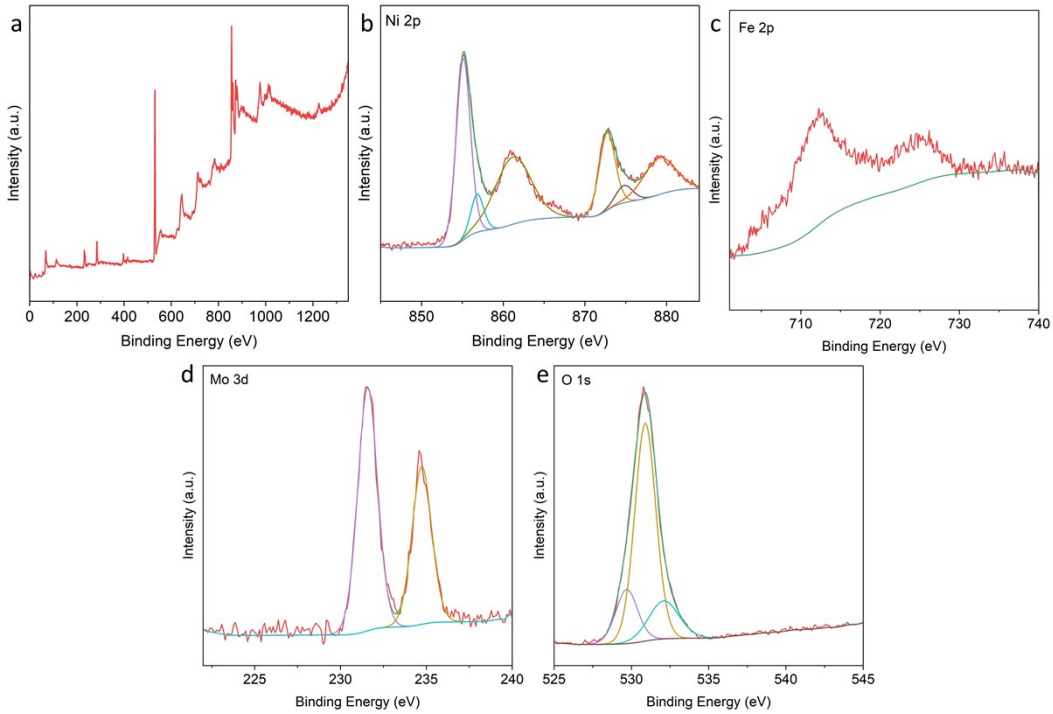

Fig. S7 (a) XPS survey and (b-e) High-resolution XPS spectra: (b) Ni 2p, (c) Fe 2p, (d) Mo 3d, and (e) O 1s of NiFeMo-1 catalyst.

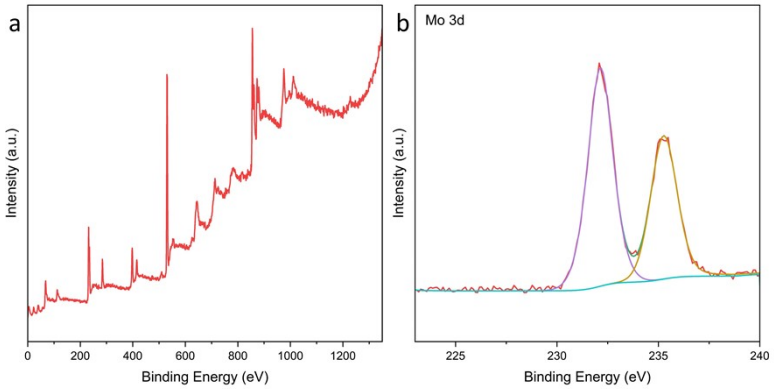

Fig S8 (a) XPS survey and (b) Mo 3d XPS spectra of of NiFeMo-2 catalyst.

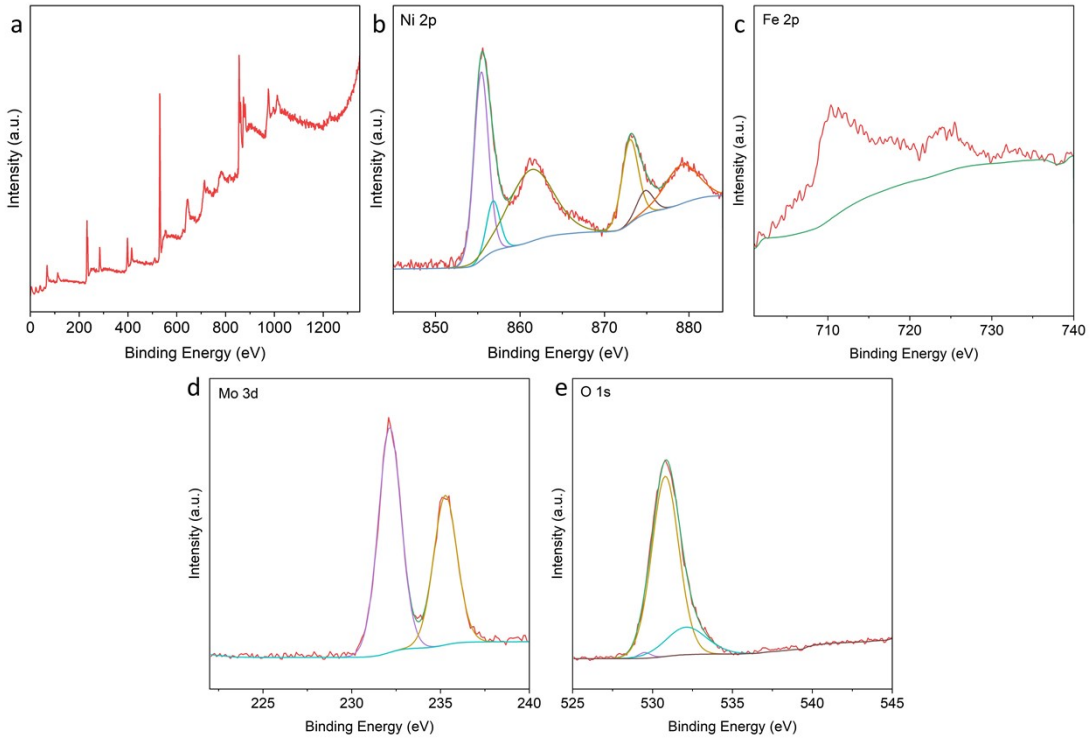

Fig. S9 (a) XPS survey and (b-e) High-resolution XPS spectra: (b) Ni 2p, (c) Fe 2p, (d) Mo 3d, and (e) O 1s of NiFeMo-3 catalyst.

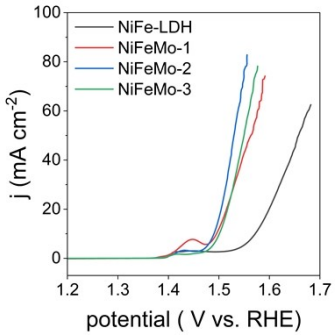

Fig. S10 positive scan LSV curves of NiFe-LDH, NiFeMo-1, NiFeMo-2 and NiFeMo-3

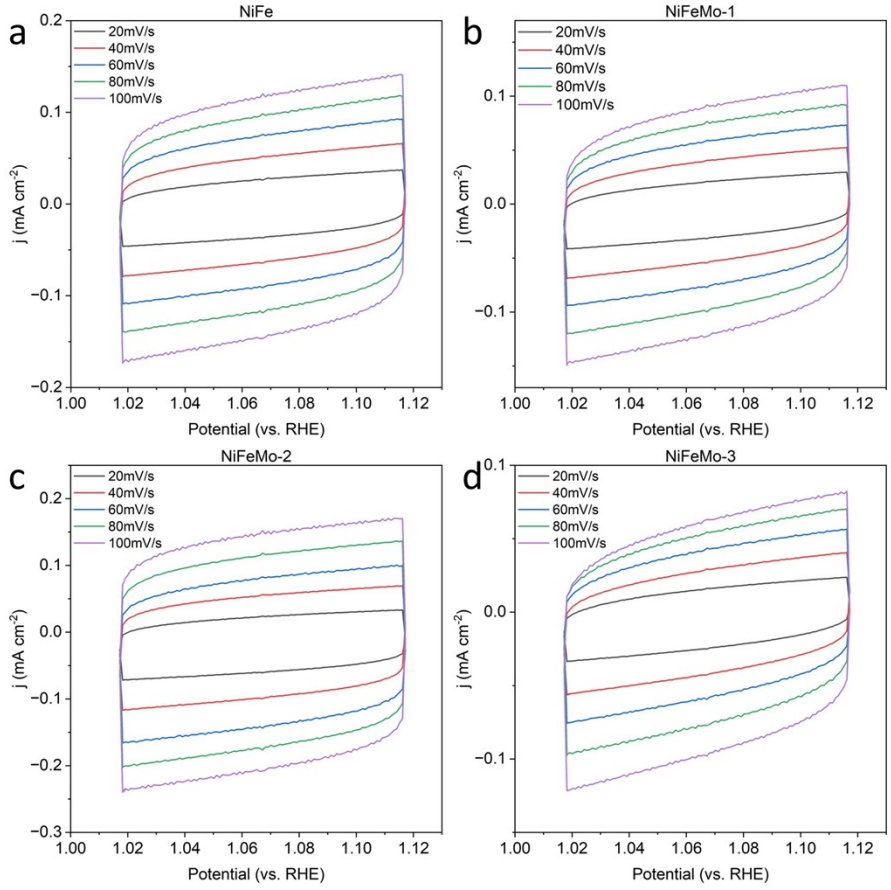

Fig. S11 ECSA curves of (a) NiFe-LDH, (b) NiFeMo-1, (c) NiFeMo-2 and (d) NiFeMo-3.

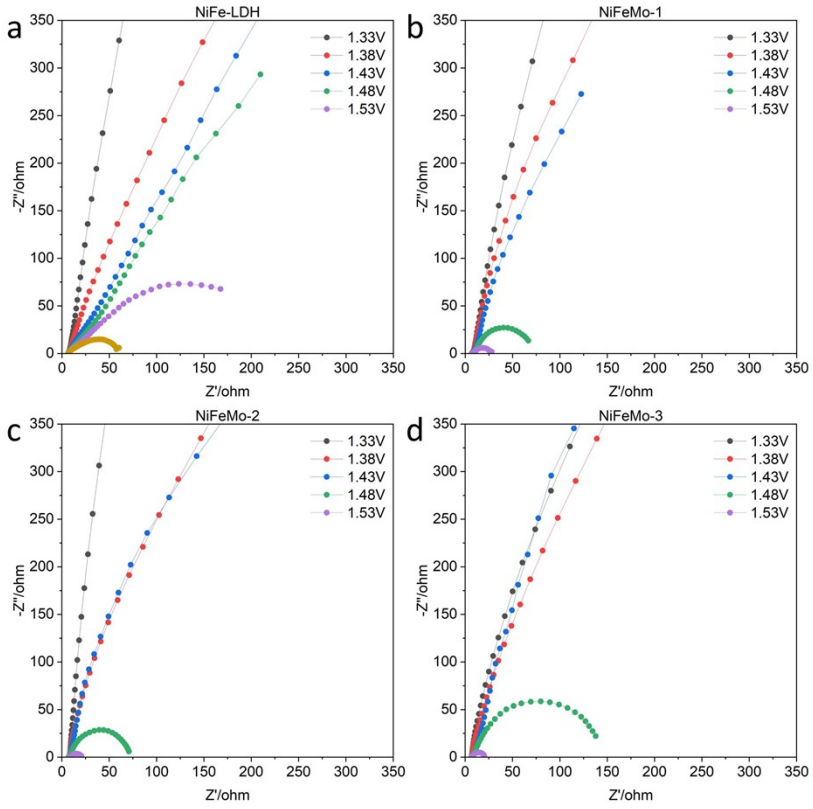

Fig. S12 (a-d) Nyquist plots of (a) NiFe-LDH, (b) NiFeMo-1, (c) NiFeMo-2 and (d) NiFeMo-3 at different potentials.

Table S1 Fitted equivalent circuit model parameters of the NiFe-LDH, NiFeMo-1, NiFeMo-2 and NiFeMo-3

Sample	R_s/Ω	R1(R_{ct})/Ω	CPE1/F	R2/Ω	CPE2/F
NiFe-LDH	6.561	243.8	4.007×10^{-3}	12.09	3.042×10^{-2}
NiFeMo-1	8.143	18.66	8.886×10^{-3}	1.628	3.572×10^{-4}
NiFeMo-2	6.881	9.07	5.927×10^{-3}	0.4694	7.199×10^{-5}
NiFeMo-3	6.497	11.6	2.894×10^{-3}	2.476	5.529×10^{-4}

Table S2 Comparison of the overpotentials (η_{10}) and Tafel slope of NiFeMo-2 with other transition metal-based OER catalysts

Catalysts	η_{10} (mV)	Tafel slope (mV dec^{-1})	Reference
NiFeMo-2	262	64.8	This work
S/N-CMF@$Fe_xCo_yNi_{1-x-y}$-MOF	296	53.5	Adv. Mater. 2023, 35, 2207888.
a-NiFeMo	280	49	Angew. Chem. Int. Ed. 2019, 58, 15772.
Ni-$FeNi_3$/$Ni_{0.5-b}Fe_{0.5-y}Mo_{1.5}O_x$	278	42	Small 2024, 20, 2303927.
Ni_xFe_{1-x}-THQ	272	47.9	Adv. Funct. Mater. 2024, 34, 2310902.
$FeCo_{0.6}Ni_{0.4}$-CAT	277	72	Sustainable Energy Fuels, 2020,4, 4589-4597
$FeNi_5$-MOF-450	307	56.2	Int. J. Miner., Metall. Mater., 2023 30, 1914–1921.
ANC-Fe_1Ni_2	266	39	J. Energy Chem. 2022, 71, 167-173.

Table S3 Comparison of the electrocatalytic stability of NiFeMo-2 with other transition metal-based OER catalysts

Catalysts	Stability	Current density (mA cm^{-2})	References
NiFeMo-2	65h	50	This work
NiFe-LDH/Sn NSs	60h	100	Appl. Catal., B. 2024, 124073.
Ni-Fe LDH nanosheets	100h	50	Chem Catal. 2022, 2(9), 2312-2327.
$NiFe(OH)_x$/(Ni, Fe)Se_2/CC	20h	10	Small 2021, 17, 2007334.
a-$NiFeO_xH_y$@NF	24h	50	Adv. Funct. Mater. 2021, 31, 2009032.
iron rusts/$Ni(OH)_2$	30h	10	Electrochim. Acta 2020, 353,

			136478.
$Ni_{0.3}Fe_{0.7}$-LDH@NF	84h	10	Appl. Catal., B. 2023, 323, 122091.

Supplementary Note 1

In situ DEMS with ^{18}O isotope labelling. First, the catalyst ink was prepared and dropped on a gold electrode. The ^{18}O-labelled catalyst was prepared by chronopotential test in KOH-$H_2{}^{18}O$ solution. Then the ^{18}O-labelled catalyst was rinsed with $H_2{}^{16}O$ to remove the $H_2{}^{18}O$ on the surface of electrode and drying, the DEMS experiments were performed. The in situ DEMS with isotope labelling was performed on electrochemical mass spectrometer purchased from Linglu Instruments (Shanghai) Co., Ltd. Typically, the test was carried out in a three-electrode cell with 1M KOH as electrolyte. The ^{18}O-labelled NiFe-LDH or NiFeMo-2 catalyst with ^{18}O-labeling, Ag/AgCl electrode prefilled with saturated KCl aqueous solution, and a Pt wire were used as working electrode, reference electrode, and counter electrode, respectively. LSV measurement was performed in KOH-$H_2{}^{16}O$ solution with a scan rate of 5 mV/s. In the meantime, gas products with different molecular weights were detected in real time by mass spectroscopy. The value of $^{34}O_2/^{32}O_2$ is defined as the ratio of $^{34}O_2$ signal to $^{32}O_2$ signal at the onset time of oxygen evolution reaction obtained by mathematical fitting calculation.