

Enhanced electrocatalytic activity and stability of Ce doping NiFe layered double hydroxide for alkaline Seawater oxidation

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EXPERIMENTAL SECTION

Chemicals and Materials.

Commercial Ni foam (NF) was purchased from Changsha Liyuan New Materials Co. $\text{Fe}_2(\text{SO}_4)_3 \cdot 9\text{H}_2\text{O}$, $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, urea ($\geq 99.5\%$) and NH_4F ($\geq 99.5\%$) were purchased from Sinopharm Chemical Reagent Co., Ltd. All the reagents were of analytical grade and used as received without any further purification.

Synthesis of NiFeCe LDH on Ni foam substrate with various Ce doping level

NiFeCe LDH electrodes with various Ce level doping shared the same synthetic route, while the feeding amounts of $\text{Ce}(\text{NO}_3)_3$ are 0.05, 0.10, 0.15, and 0.20 mmol, respectively. The as-obtained electrode was denoted as NiFeCe_x LDH, and x represent represents the feeding molar amount of $\text{Ce}(\text{NO}_3)_3$.

Material Characterizations.

The morphological information was obtained by scanning electron microscopy (SEM, Hitachi SU8200) and transmission electron microscopy system (TEM, FEI Tecnai F30). X-ray diffraction (XRD) characterization was performed on a Rigaku D/MAX 2500V instrument (Rigaku Corporation, Japan) with a scanning speed of 4°min^{-1} in the 2θ range of $10\text{--}70^\circ$. The chemical valence states and compositions were analyzed by X-ray photoelectron spectroscopy (XPS) on an Thermo SCIENTIFIC Nexsa instrument, with the C 1s binding energy (284.8 eV) as the calibration reference.

The compositions of Ni, Fe, and Ce were analyzed by inductively coupled plasma optical emission spectroscopy (ICP-OES) on a Plasma 2000 full-spectrum ICP spectrometer.

Electrochemical Performance Tests

Electrochemical measurements were performed in a 1.0 M KOH solution or simulated seawater (1.0 M KOH + 0.5 M NaCl) solution using a DH7002 electrochemical workstation (Jiangsu Donghua Analytical Instrument Co., Ltd.) and a standard three-electrode system, while platinum sheet, Hg/HgO electrode, and the NF-supported nanosheet arrays served as counter electrode, reference electrode and working electrode, respectively. The potential was calibrated to the reversible hydrogen electrode (RHE) by using the following equation:

$$E_{\text{RHE}}(\text{V}) = (E_{\text{Hg/HgO}} + 0.0591) \times \text{pH} + 0.098 \quad (1)$$

Linear sweep voltammetry (LSV) polarization curves were collected at a scan rate of 5 mV s^{-1} with 90% iR compensation. The overpotential (η) was calculated as:

$$\eta = E_{\text{RHE}} - 1.23 \text{ V} \quad (\text{Eq.2})$$

The apparent Tafel slope was derived from the polarization curve by fitting experimental data to Equation (3):

$$\eta = a + b \log j \dots \dots \dots (\text{Eq. 3})$$

where a is the Tafel constant, b is the Tafel slope, and j is the steady-state current density.

The double-layer capacitance (C_{dl}) was determined through cyclic voltammetry (CV) curves measured at different scan rates in the non-faradaic region. The electrochemical active surface area (ECSA) was calculated using the formula $\text{ECSA} = C_{\text{dl}}/C_s$, where C_s is the specific capacitance of a smooth surface under alkaline conditions, typically taken as $40 \mu\text{F cm}^{-2}$.

Electrochemical impedance spectroscopy (EIS) experiments were performed over a frequency range of 0.01 Hz to 10^5 Hz. The long-term stability of the electrocatalyst was assessed using galvanostatic mode. The turnover frequency (TOF) was calculated according to $\text{TOF} = (J \times A)/(4 \times F \times n)$, where J (A cm^{-2}) is the current density at a given

overpotential, A (cm^2) is the surface area of the electrode material, 4 is the number of electrons transferred per mole of oxygen generated in the OER, F is the Faraday constant ($96485.3 \text{ C mol}^{-1}$), and n (mol) represents the molar number of Ni, Fe and Ce ions in NiFeCe LDH or NiFe LDH (without considering NF substrate), which was determined by ICP-OES. The stability of the catalyst was tested at a constant current density of 500 mA cm^{-2} .

Density Function Theory (DFT) calculation method

For all DFT calculations, the Vienna Ab initio Simulation Package (VASP) was employed, and the Perdew-Burke-Ernzerhof (PBE) functional and projector-augmented wave (PAW) method were applied to describe the exchange-correlation energy and electron-ion interactions. All slab models contain atomic layers and a vacuum layer of 15 \AA thickness to avoid artificial interaction between periodic images. To accurately describe the strong electronic correlations in the 3d orbitals of transition metals, the DFT+U method was employed. Structural optimizations, including both atomic positions and unit cell parameters, were performed using the Broyden–Fletcher–Goldfarb–Shanno (BFGS) algorithm as implemented in ASE. The UnitCellFilter was applied to allow for the simultaneous relaxation of atomic coordinates and cell dimensions. To improve the description of van der Waals interaction, DFT-D3 correction was employed. The energy cutoff was set to be 500 eV . The Brillouin zone was sampled by Gamma-centered k-point with $3 \times 3 \times 1$. The structures were fully relaxed until the maximum force on each atom was less than 0.03 eV/\AA , and the energy convergent standard was 10^{-5} eV .

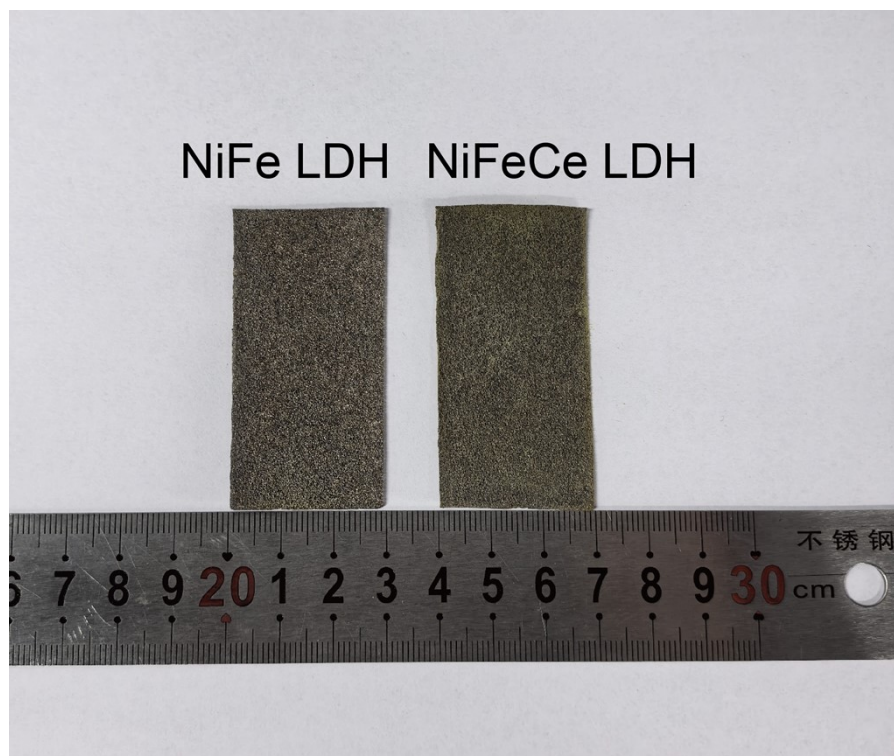


Figure S1. Photograph of NiFe LDH and NiFeCe LDH electrode with large size.

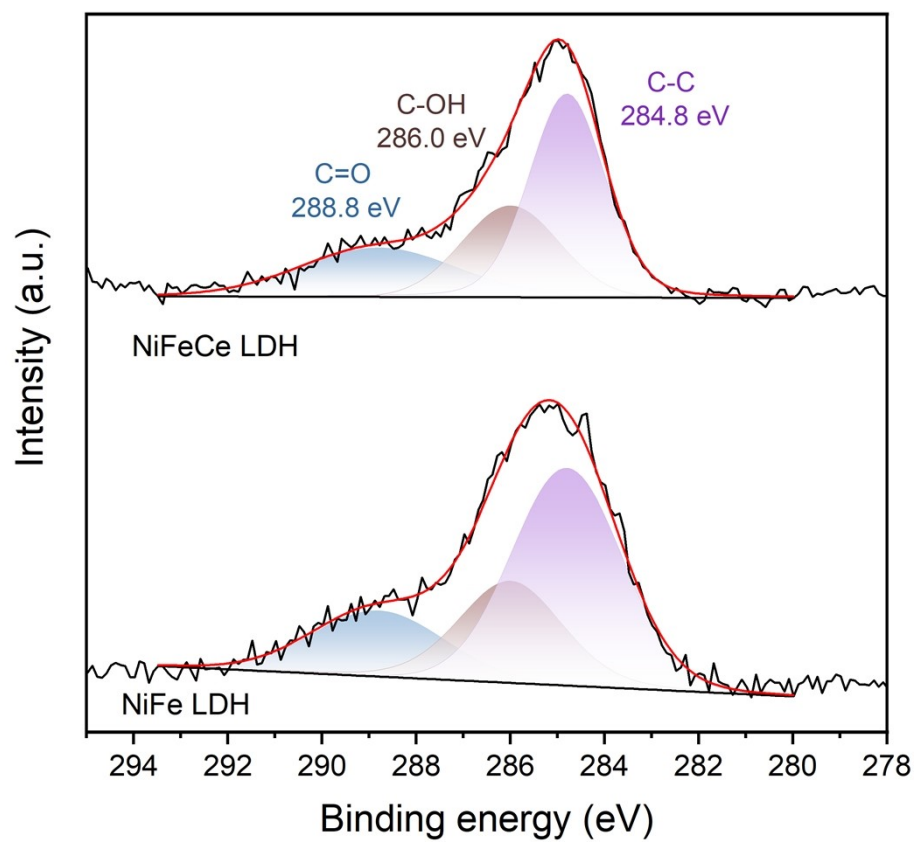


Figure S2. C 1s XPS spectra of NiFe LDH and NiFeCe LDH electrodes.

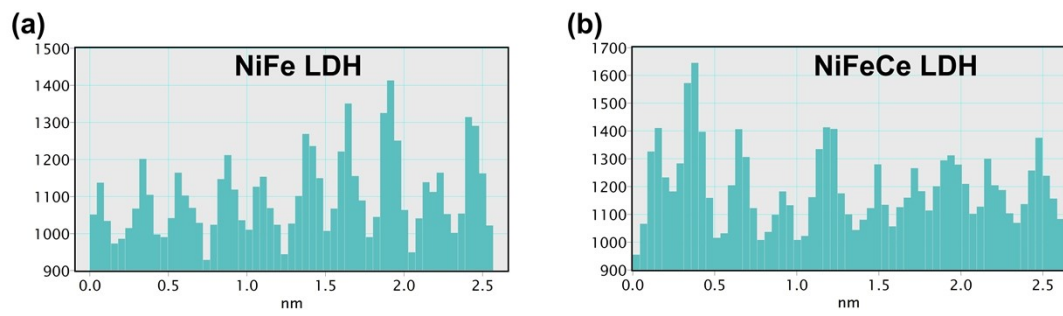


Figure S3. The line intensity profiles from HRTEM images. (a) NiFe LDH. (b) NiFeCe LDH.

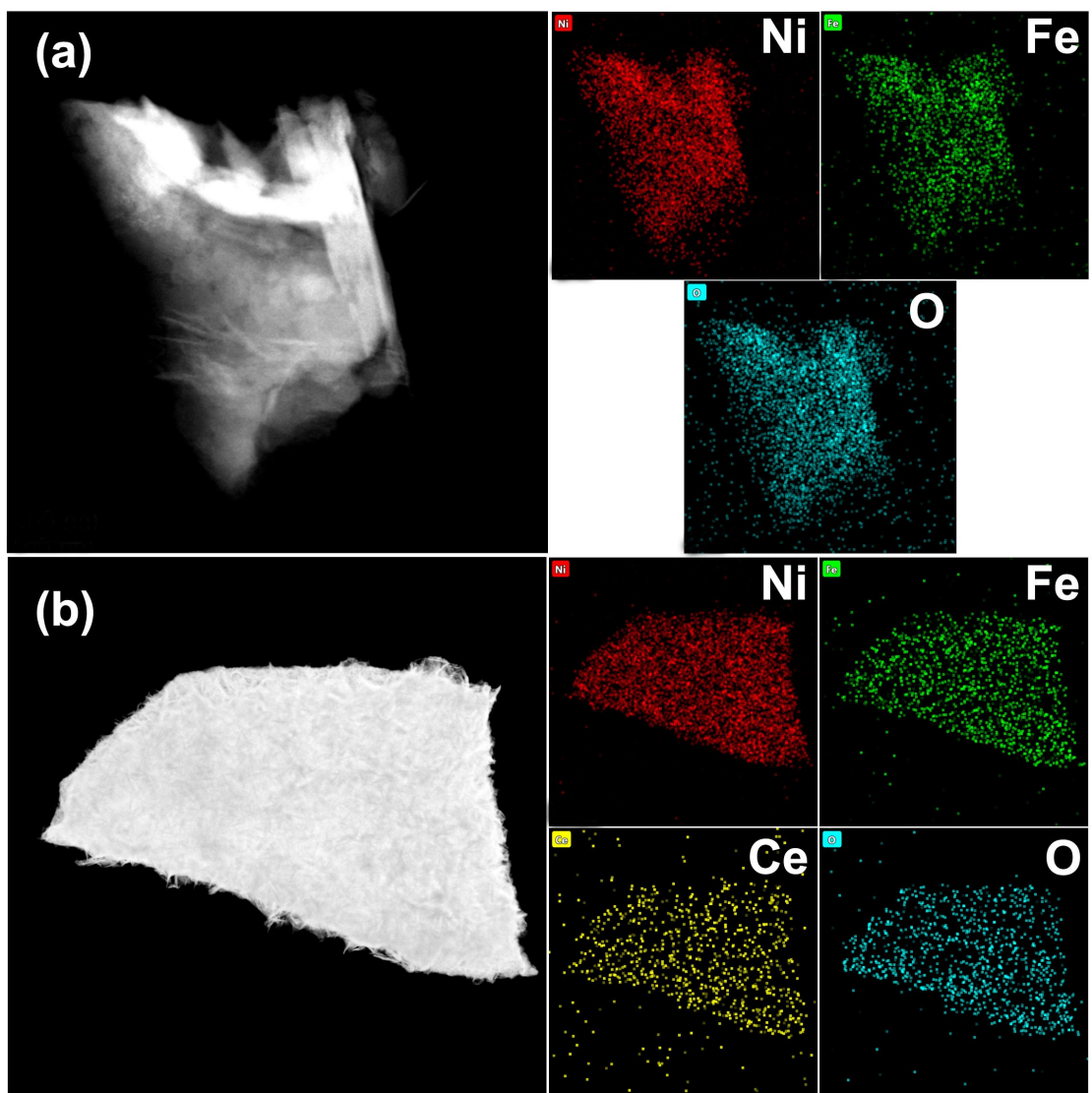


Figure S4. Elemental mapping of (a) NiFe LDH and (b) NiFeCe LDH nanosheets.

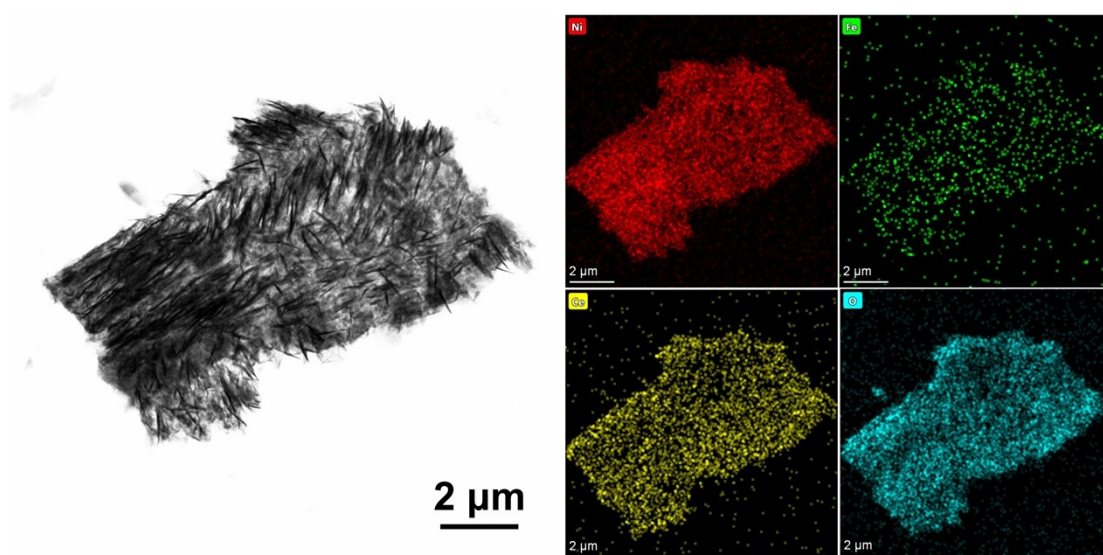


Figure S5. TEM and element mapping of NiFeCe LDH nanosheet.

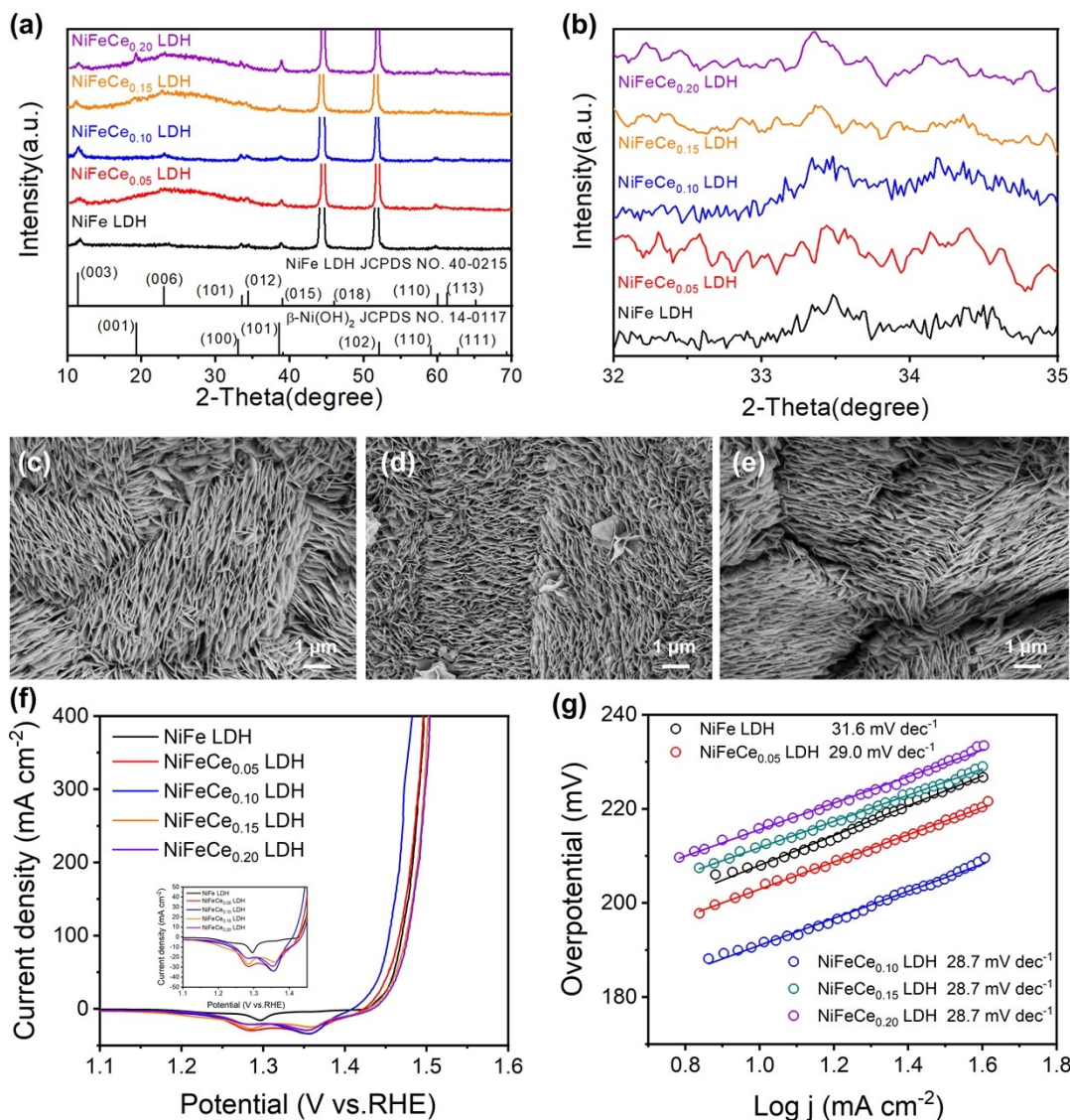


Figure S6. (a) XRD patterns and (b) the corresponding enlarged part of the NiFeCe LDH prepared with different Ce feeding amounts. SEM images of (c) NiFeCe_{0.05} LDH, (d) NiFeCe_{0.15} LDH and (e) NiFeCe_{0.20} LDH. (f) Linear sweep voltammetry (LSV) curves and (g) Tafel plots for the oxygen evolution reaction (OER) performance of NiFeCe LDH prepared with different Ce feeding amounts. The subscript number of Ce represents the feeding molar amount of Ce(NO₃)₃. NiFeCe_{0.10} LDH was selected as the representative research object in this work due to its optimal performance.

All NiFeCe layered double hydroxide (LDH) electrodes with varying Ce contents exhibit similar layered structures, as evidenced by the (003) diffraction peak appearing in the small-angle region (Figure S6a). However, at high Ce doping levels, the

formation of the β -Ni(OH)₂ phase becomes prominent, indicating that Ce doping disrupts the deposition of NiFe LDH. In the enlarged sections of the XRD patterns, the (101) and (012) peaks shift toward smaller angles with increasing Ce content, confirming the effective incorporation of Ce (Figure S6b). SEM images reveal quasi-parallel alignment of the as-obtained nanosheet arrays, suggesting similar growth behavior across the NiFeCe LDH nanosheets (Figure S6c-S6e). Thus, differences in their catalytic activity are primarily attributable to intrinsic activity.

The LSV curves of all NiFeCe LDH electrodes are shown in Figure S6f. NiFeCe_{0.10} LDH electrode achieves the lowest overpotential of 199 mV at a current density of 20 mA cm⁻². However, the difference in the overpotential among the electrodes are rather small, while the NiFeCe_{0.20} LDH electrode delivers the highest overpotential of 223 mV. The similar tafel slopes in Figure S6g also reveal the similar OER kinetics.

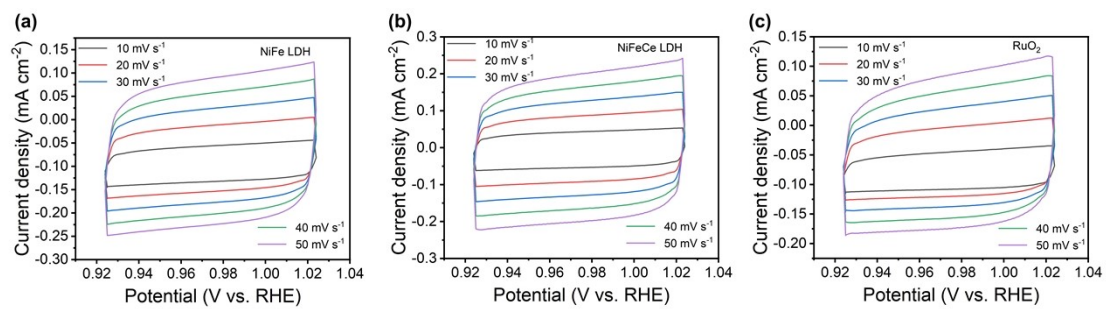


Figure S7. Cyclic voltammetry (CV) curves of the electrodes at non-faradic region. (a) NiFe LDH; (b) NiFeCe LDH; (c) RuO₂.

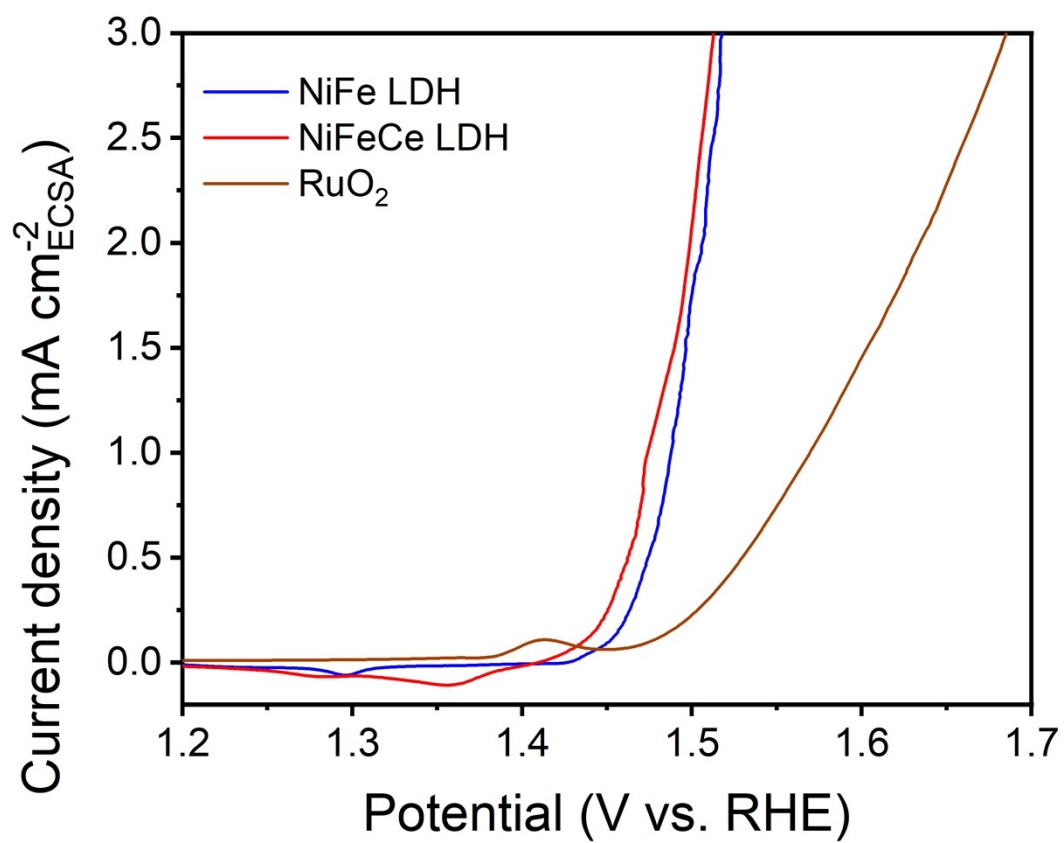


Figure S8. ECSA-normalized LSV curves of the as-obtained electrodes.

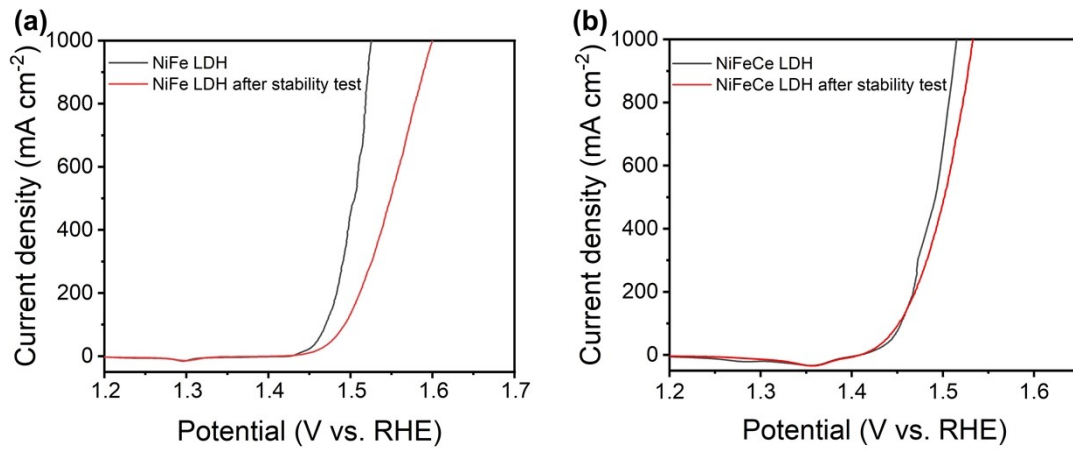


Figure S9. LSV curves of (a) NiFe LDH and (b) NiFeCe LDH electrodes before and after stability testing.

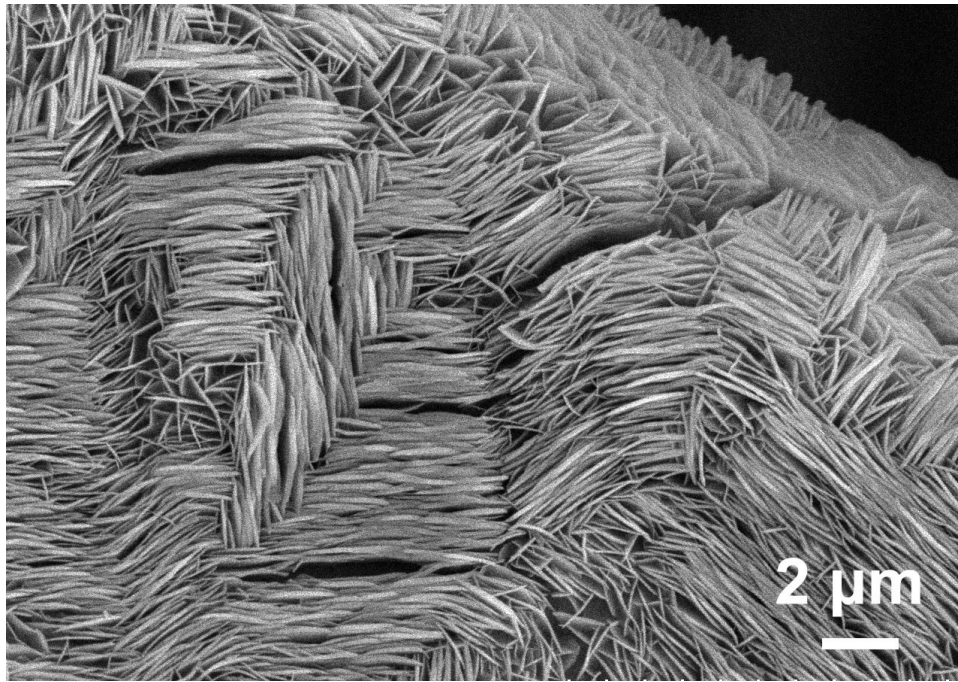


Figure S10. SEM image of NiFe LDH electrode after stability test.

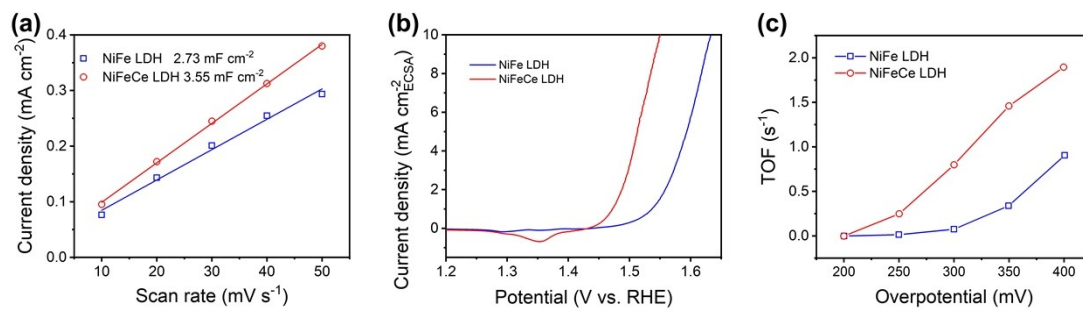


Figure S11. (a) Relationship between scan rate and Δj for verifying the C_{dl} values, (b) corresponding ECSA-normalized LSV curves, and (c) the TOF plots of NiFe LDH and NiFeCe LDH electrodes in simulated seawater electrolyte.

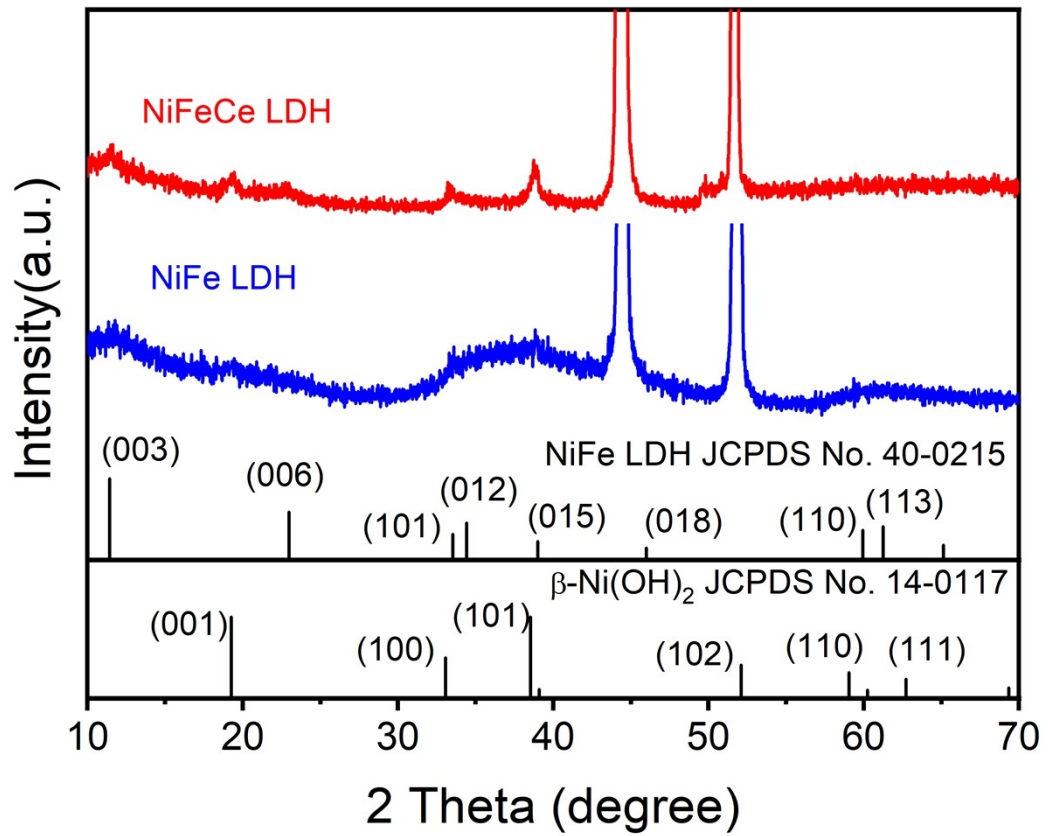


Figure S12. XRD pattern of the NiFe LDH and NiFeCe LDH electrodes after stability test in simulated seawater electrolyte.

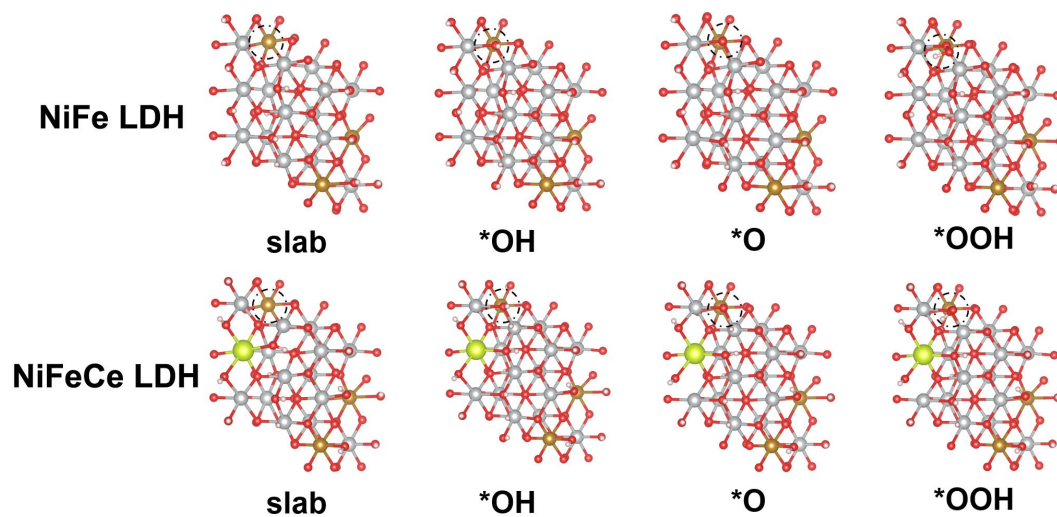


Figure S13 Illustration of the adsorption OER intermediates occurred on NiFe LDH and NiFeCe LDH.

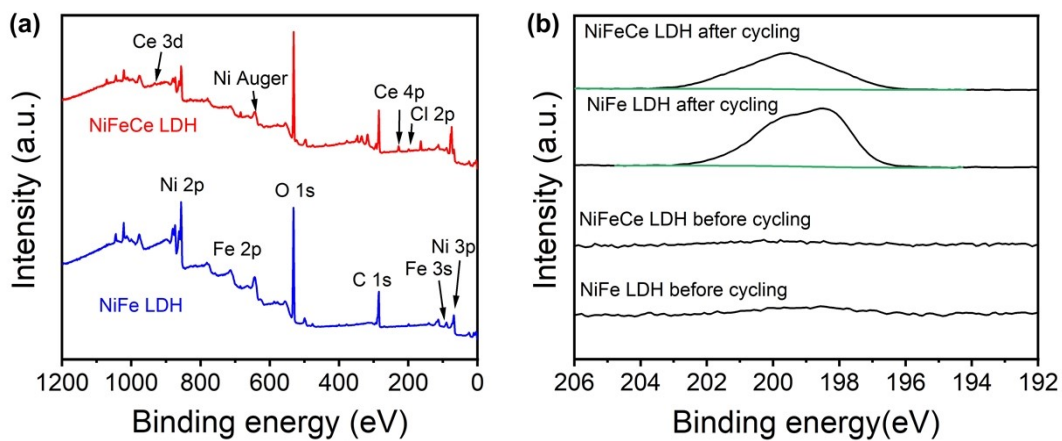


Figure S14. (a) XPS survey spectra of the NiFe LDH and NiFeCe LDH electrodes after stability test in simulated seawater electrolyte. (b) Cl 2p XPS spectra of the NiFe LDH and NiFeCe LDH electrodes before and after stability test in simulated seawater electrolyte.

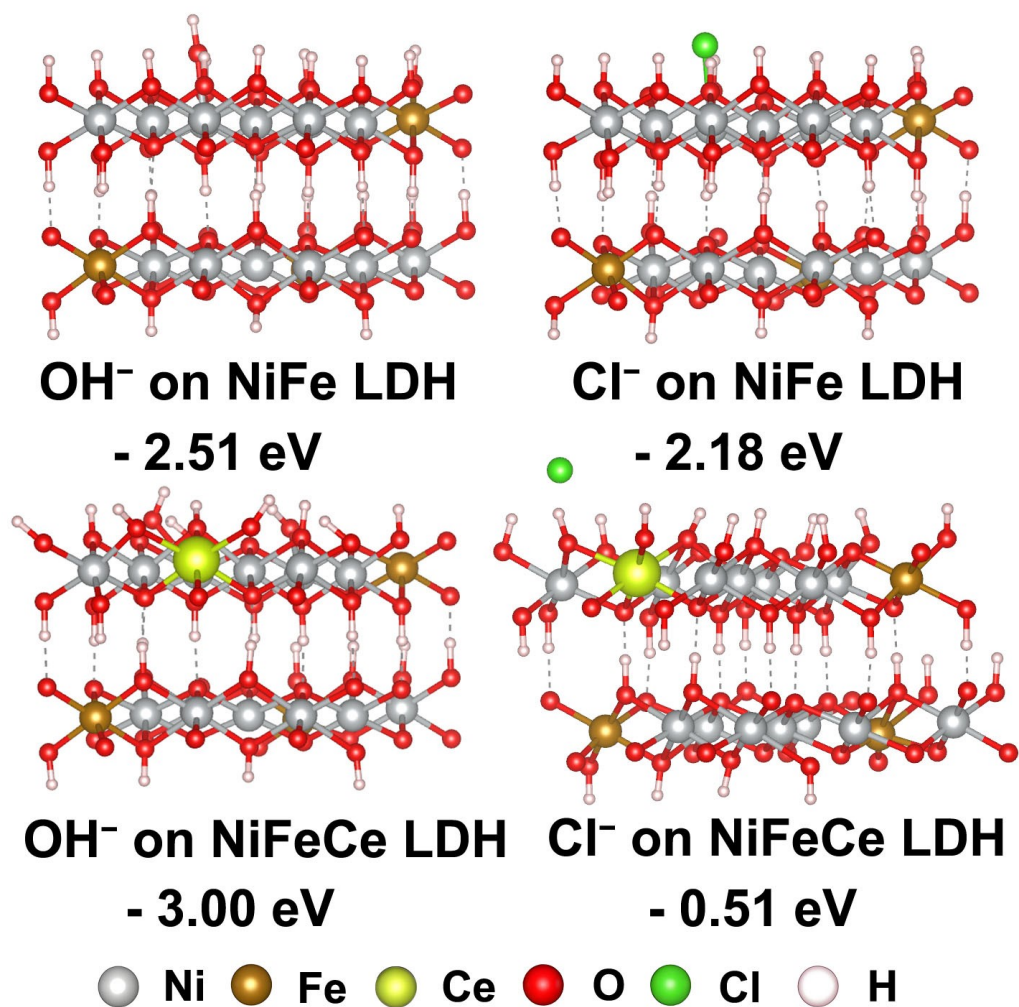


Figure S15. Scheme of adsorption models and the corresponding adsorption energies of OH⁻ and Cl⁻ anions on NiFe LDH and NiFeCe LDH.

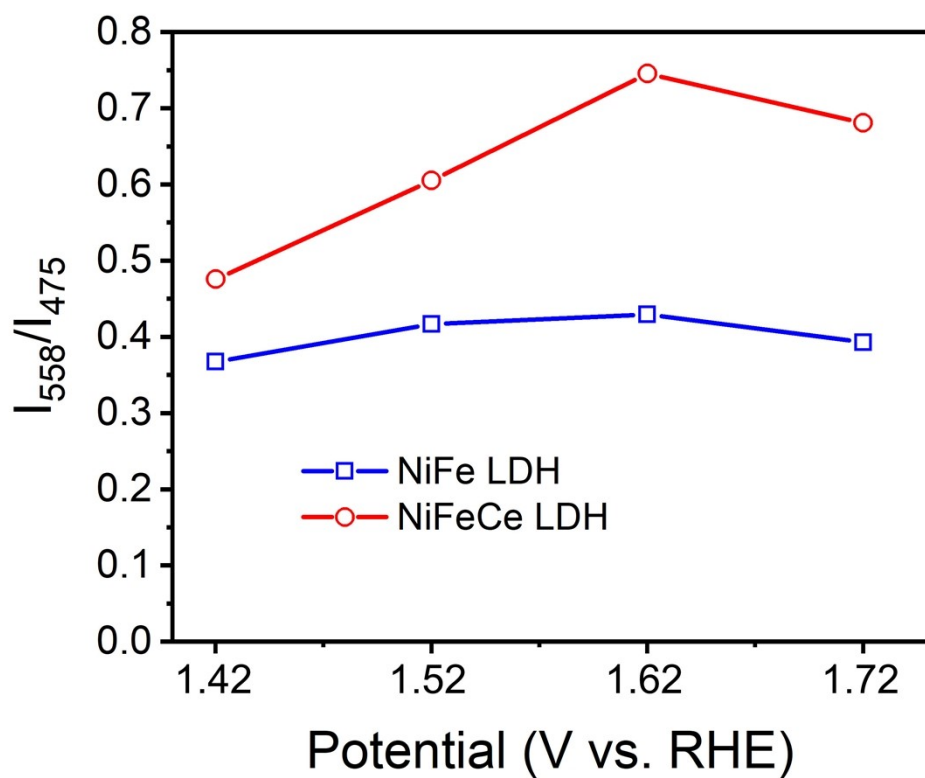


Figure S16. The I_{558}/I_{475} intensity ratio of NiOOH signal at the potential range of 1.42 to 1.72 V (vs. RHE).

Table S1. Content ratio of metal element in NiFeCe LDH and NiFe LDH based on

ICP-OES and XPS analysis.

Sample	Fe/Ni		Ce/Fe	
	ICP-OES	feed ratio	ICP-OES	feed ratio
NiFe-LDH	0.098	0.125	/	/
NiFeCe-LDH	0.102	0.125	0.021	2.5

Table S2. Comparison of OER performance for selected nickel-iron based electrocatalysts.

Electrocatalyst	η / mV				Tafel slope / (mV dec ⁻¹)	Durability time/h	Ref.
	10 mA cm ⁻²	100 mA cm ⁻²	500 mA cm ⁻²	1000 mA cm ⁻²			
NiFeCe LDH	191	224	262	285	28.7	1000	This work
IrNi@NiFe-MOFs	228	335	/	/	37.6	115	[1]
NiFe-S-0.6	213	252	294		32	260	[2]
Hf-NiFe-LDH/NF	230	258	/	/	30	90	[3]
Te-NiFeO _x H _y /NF	208	270	310		21.7	300	[4]
NiFe/MnNiCo-LDH	227	265	/	/	64.14	50	[5]
NiFe(S)/NM	/	310	370		41.4	500	[6]
E-NiFe	243	/	/		52	24	[7]
Ru/NiFe-LDH-40/NF	208	271	/	/	30	100	[8]
NiFe-LDH/MoS ₂ -0.25	/	284	336		37.34	24	[9]
Co-B/NiFe-LDH	244	/	/	/	56	50	[10]
NiFe@NiB	280	/	/	/	42.3	120	[11]
Sn-NiFeOOH@NF	223	270	/	/	41.9	200	[12]
NiFe-AL@Mo ₂ CT _x	230	300	/	/	53	50	[13]
Rh@NiFe-MIL-53	240	334	/	/	48.2	110	[14]
Ni _{0.85} Se@NiFe LDH	225	/	/	/	41.4	40	[15]
Ir _{SA} -NiFe LDH/NiMo	/	260	350		24	500	[16]

Table S3. Equivalent circuit fitting results of EIS.

Electrocatalyst	Impedance (Ω)		
	R_s	R_f	R_{ct}
NiFeCe LDH	1.265	0.041	0.546
NiFe LDH	1.378	0.265	3.385
RuO ₂	1.443	0.054	8.946
NiFeCe LDH in simulated seawater electrolyte	1.981	0.063	0.772
NiFe LDH in simulated seawater electrolyte	2.887	0.311	3.712

Table S4. Content ratios of Ni³⁺/Ni²⁺ and Fe³⁺/Fe²⁺ before and after stability test in 1 M KOH solution and in simulated seawater electrolyte.

Sample	Before stability test		After stability test in 1 M KOH solution for 1000 h		After stability test in simulated seawater electrolyte	
	Ni ³⁺ /Ni ²⁺	Fe ³⁺ /Fe ²⁺	Ni ³⁺ /Ni ²⁺	Fe ³⁺ /Fe ²⁺	Ni ³⁺ /Ni ²⁺	Fe ³⁺ /Fe ²⁺
	NiFe-LDH	0.477	0.913	0.568	2.248	0.655
NiFeCe-LDH	0.632	0.875	0.725	0.906	0.782	1.458

Table S5. Comparison of OER performance of the present electrocatalyst with the reported nickel-iron based electrocatalysts in 1 M KOH + 0.5 M NaCl electrolyte.

Electrocatalyst	η / mV			TOF / s ⁻¹	Tafel slope / (mV dec ⁻¹)	durability	Ref.
	100 mA cm ⁻²	500 mA cm ⁻²	1000 mA cm ⁻²				
NiFeCe LDH/NF	244	288	331	0.78@300 mV	21.6	600 h	This work
Sn-NiFeOOH@NF	340	/	/	/	63.3	200 h	[12]
NF/FeCoP/NiFe LDH	256	/	/	/	89.9	55 h	[17]
NiTe@NiFe-LDH/NF	277	359	/	/	59.79	50 h	[18]
TS-NiFe LDH/NF	/	333	398	2.89@ 410 mV	/	350 h	[19]
Li-NFL/CN	284	/	/	0.148@280 mV	/	25 h	[20]
NiFe LDH-CeW@NFF	/	330	386.7	0.54@300 mV	66	100 h	[21]
NiFe LDH-CNA3	264	/	/	0.195@270 mV	/	72 h	[22]
CoP@NiFe LDH	260	/	/	/	44.4	24 h	[23]
NiFe LDH@PCM/NF	266	314	344	0.75@270 mV	38.67	500 h	[24]
NiFe ₄ -VOx/NF	284	/	/	/	/	100 h	[25]
POM-CoFe LDH/NF	265	325	368	~ 1.2@300 mV	/	1300 h	[26]
NiFe LDH/NF (in PF ₆ ⁻ -containing seawater)	/	247	282	~ 0.17@at 270 mV)	28.7	5000 h	[27]
NiFePBA/Ni(OH) ₂ /NF	216	339	361	/	49.09	1000 h	[28]
Ce(OH) ₃ @NiFe LDH/NF	/	295	321	0.67@315 mV	23.5	1000 h	[29]

Table S6. Metal ion concentrations in the electrolyte after stability test.

Stability test	Electrode	Concentration of elements in the electrolyte		
		Ni (mg L ⁻¹)	Fe (mg L ⁻¹)	Ce (mg L ⁻¹)
In KOH solution for 1000 h	NiFe LDH	0.068	0.085	
	NiFeCe LDH	0.035	0.061	0.035
In simulated seawater electrolyte for 500 h	NiFe LDH	8.963	0.233	
	NiFeCe LDH	0.012	0.197	0.317

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