Band Alignment-Driven β-NiOOH Activation in NiFe LDH@CoO_x-

CNF Heterojunction for Efficient Alkaline Oxygen Evolution

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Experimental Section

Materials Characterization

The prepared materials were characterized using the following techniques: Field Emission Scanning Electron Microscopy (SEM, JSM-7500F), Transmission Electron Microscopy (TEM, JEM-2100F), X-ray Diffraction (XRD, equipped with Cu-K α radiation source, $\lambda = 1.5406$ Å), Raman Spectroscopy (Raman, Renishaw inVia, $\lambda = 532$ nm), X-ray Photoelectron Spectroscopy (XPS, Thermo Scientific ESCALAB 250 XI), Ultraviolet-Visible Spectroscopy (UV-vis, Shimadzu UV-3600i Plus), and Highangle Annular Dark Field Scanning Transmission Electron Microscopy (HADDF-STEM, Talos F200x).

OER performance tests

The OER performance of NiFe LDH@CoOx-CNF was tested in a three-electrode system with 1 M KOH as the electrolyte. A graphite rod served as the counter electrode, and a Hg/HgO electrode was used as the reference electrode. The working electrode was prepared by placing a 1×1 cm² NiFe LDH@CoO_x-CNF sample between two clean pieces of nickel foam and pressing them together using a hydraulic press at a pressure of 10 MPa. For other powdered samples such as NiFe LDH, RuO₂, and Pt/C, the working electrode was prepared as follows: 3 mg of the catalyst was ultrasonically dispersed for 30 minutes in a mixed solution of 165 µL deionized water, 55 µL ethanol, and 20 µL 5% Nafion. Then, 80 µL of this catalyst ink was drop-cast onto a 1×1 cm² piece of nickel foam and left to dry naturally in a vacuum drying oven. Before performing electrochemical tests, the catalyst was activated by conducting cyclic voltammetry scans (CV) in the potential range of 0-0.8 V (vs. Hg/HgO) at a scan rate of 100 mV s⁻¹ for 50 cycles. Linear sweep voltammetry (LSV) measurements were then conducted in the same potential range at a scan rate of 5 mV s⁻¹ without iR compensation. Electrochemical impedance spectroscopy (EIS) tests were carried out in the frequency range of 0.01 Hz to 100 kHz at a potential of 0.55 V (vs. Hg/HgO). Stability tests were performed using chronopotentiometry at a current density of 20 mA cm⁻². All potentials in this work were converted to the reversible hydrogen electrode (RHE) scale using the equation $E_{RHE} = E_{Hg/HgO} + 0.059 \times pH + 0.098$, with the pH of 1 M KOH assumed to be 13.8.

Electrochemical active surface area (ECSA) measurement: The electrochemical active surface area (ECSA) is calculated based on the electrochemical double-layer capacitance (C_{dl}) value. The C_{dl} value is obtained through CV tests conducted in the non-Faradaic potential region at scan rates of 20, 40, 60, 80, and 100 mV s⁻¹. In this study, the specific capacitance of nickel foam (NF) is taken as 1.75 mF cm⁻². The ECSA of the catalyst can be calculated using the following formula:

$$A_{ECSA} = \frac{Cdl \ of \ the \ catalyst \ (mF \ cm^{-2})}{1.75}$$

Turnover frequency (TOF) measurement: The turnover frequency (TOF) can be calculated using the following equation:

$$TOF = \frac{|j|A}{mFn}$$

where j is the current density of the catalyst at a given overpotential, A is the electrode area (1 cm² in this work), F is the Faraday constant (96475 C mol⁻¹), m is the number of electrons involved in the reaction (2 for HER and 4 for OER), and n is the number of active sites. The value of n can be determined through CV tests in phosphate buffer solution (PBS, pH = 7). During these tests, the potential range is from -0.2 to 0.6 V (vs. RHE), and the scan rate is 50 mV s⁻¹. The relevant formula is:

$$n = \frac{Q}{2F}$$

where Q is the charge obtained by integrating the CV curve, and F is the Faraday constant (96475 C mol⁻¹).

Mott-Schottky (MS) measurement: The Mott-Schottky (MS) tests were conducted in a three-electrode system using 0.2 M Na_2SO_4 as the electrolyte. An Ag/AgCl electrode and a platinum sheet electrode were used as the reference and counter electrodes, respectively, with a conductive glass (FTO) serving as the substrate for the working electrode. To prepare the working electrode, a catalyst mixture was applied onto the FTO substrate. Specifically, 2 mg of catalyst was dispersed in 100 μ L of isopropanol and 10 μ L of 5% Nafion solution, followed by ultrasonication to achieve a uniform dispersion. Then, 40 μ L of the mixture was coated onto a 1×1 cm² FTO and dried in a vacuum oven at 60°C. The MS measurements were performed using a CHI 660E electrochemical workstation. The positions of the conduction band and valence band were calculated using the RHE potential, according to the formula: $E_{RHE} = E_{Ag}/_{AgCl}$ + 0.197 + 0.059×pH (pH=7.5).

Density Functional Theory (DFT) calculations: In the DFT calculations of this work, the Vesta software was utilized for model construction, while the Vienna Abinitio Simulation Package (VASP) was employed for structural optimization and energy calculations. The Perdew–Burke–Ernzerhof (PBE) exchange-correlation functional was used within the generalized gradient approximation (GGA). The electronic properties were described using the projector augmented wave (PAW) method. The plane-wave cutoff energy was set at 520 eV. Convergence calculations were carried out with a convergence energy threshold of 10⁻⁵ eV. Optimization balanced the geometric shape and lattice constants, ensuring that the maximum stress on each atom was within 0.05 eV Å⁻¹. The magnetic properties of the model were described using spin polarization, and the charge density difference was set at 0.01 e Å⁻¹. In addition, we applied GGA+U in all the calculations, with U = 3.3 eV for Co, U = 6.2 eV for Ni, and U = 4.3 eV for Fe. Brillouin zone k-point sampling was conducted using the Monkhorst-Pack grid method, with a k-point mesh of 1×1×1 for all models. The DFT-D3 method was adopted to take van der Waals correction into consideration.

In this work, we neglected the small amount of CoO and constructed a structural model of NiFe LDH (001)/graphene (002)/Co (111). To avoid artificial interactions between periodic images, a 20 Å vacuum layer was added in the direction perpendicular to the layered structure. The adsorption energies (E_{ads}) were calculated as $E_{ads} = E_{ad/sub} - E_{ad} - E_{sub}$, where $E_{ad/sub}$, E_{ad} , and E_{sub} are the energies of the optimized adsorbate/substrate system, the adsorbate, and the substrate, respectively. Considering

the contributions from zero-point energies and entropies, the Gibbs free energies of adsorption are defined as:

$$G_{ads} = E_{ads} + \Delta ZPE - T\Delta S \tag{1}$$

The zero-point energies and entropies of the adsorbates were computed from vibrational frequency calculations, in which only the adsorbate vibrational modes were computed explicitly, while the catalyst was fixed. The zero-point energies and entropies of gas phase molecules were obtained from the NIST database. The oxygen evolution reaction is assumed to follow the conventional four-step electro-proton coupled mechanism:

$$*+ OH^{-} \to * OH + e^{-} \tag{2}$$

$$* OH + OH^{-} \rightarrow * O + H_2O + e^{-}$$
(3)

$$* 0 + 0H^{-} \rightarrow * 00H + e^{-} \tag{4}$$

$$* 00H + 0H^{-} \rightarrow *+ 0_{2} + e^{-} \tag{5}$$

The analysis of the crystal orbital Hamilton population (COHP) was done by VASP with the computer program LOBSTER.



Fig. S1. (a, b) TEM images of CNF.



Fig. S2. (a, b) TEM images, and (c) the diameter distribution diagram of CoO_x nanoparticles in CoO_x -CNF-1. (d, e) TEM images, and (f) the diameter distribution diagram of CoO_x nanoparticles in CoO_x -CNF-2. (g, h) TEM images, and (i) the diameter distribution diagram of CoO_x nanoparticles in CoO_x -CNF-4.



Fig. S3. SEM images of (a) NiFe LDH@CNF, (b) NiFe LDH@CoO_x-CNF-1, (c) NiFe LDH@CoO_x-CNF, and (d) NiFe LDH@CoO_x-CNF-4.



Fig. S4. The TEM image of NiFe LDH.



Fig. S5. (a) XRD patterns and (b) Raman spectra of CNF, CoO_x -CNF-1, CoO_x -CNF, and CoO_x -CNF-4.

As shown in **Fig. S5(a)**, with the increase of Co content from 0 to 0.4 g, the intensity of the Co and CoO diffraction peaks in the resulting CoO_x-CNF increases, indicating the formation of more CoO_x, which is consistent with the TEM results in **Fig. S2**. In the Raman spectra, the intensity ratio of the D band (located at 1347 cm⁻¹) to the G band (1589 cm⁻¹) is used to assess the degree of graphitization of the carbon material [1]. It can be seen that compared to CNF (with an I_D/I_G ratio of 1.06), CoO_x-CNF (with an I_D/I_G ratio of 0.98) contains more graphitic carbon, indicating that Co doping enhances the graphitic content in the carbon material, which helps improve its electrical conductivity. However, the I_D/I_G ratio of CoO_x-CNF-4 increases, which may be attributed to the excessive Co species causing damage to the carbon lattice. Notably, the new peaks at 473 cm⁻¹, 521 cm⁻¹, and 681 cm⁻¹ in CoO_x-CNF correspond to the E_g, F_{2g}, and A_{1g} modes of Co–O vibration peaks, respectively [2, 3].



Fig. S6. High-resolution XPS spectra of CoO_x-CNF: (a) C 1s, (b) N 1s, (c) O 1s, and (d) Co 2p.

XPS spectra were used to investigate the elemental states in the CoO_x -CNF substrate. As shown in **Fig. S6(a)**, the high-resolution spectrum of C 1s can be deconvoluted into three characteristic peaks. The binding energies at 284.0, 285.2, and 288.7 eV correspond to C=C, C-N/C-O, and C=O, respectively [4]. The N 1s spectrum can be deconvoluted into peaks at binding energies of 397.8, 398.5, 400.2, 402.5, and 405.5 eV, which are assigned to pyridinic N, Co-N_x, pyrrolic N, graphitic N, and oxidized N, respectively [3]. Additionally, the O 1s spectrum is deconvoluted into peaks corresponding to Co-O (529.4 eV), C=O (531.0 eV), and C-OH (532.5 eV) [5]. **In Fig. S6(d)**, the peaks at 778.4 and 793.7 eV are attributed to the $2p_{1/2}$ and $2p_{3/2}$ states of metallic Co, while the peaks at 779.8 and 795.5 eV correspond to the $2p_{1/2}$ and $2p_{3/2}$ states of Co²⁺ [6]. These results further confirm the presence of Co species in the carbon nanofibers in the forms of CoO and metallic Co.



Fig. S7. XRD patterns of NiFe LDH@CNF, NiFe LDH@CoO_x-CNF-1, NiFe LDH@CoO_x-CNF, and NiFe LDH@CoO_x-CNF-4.

Fig. S7 shows the phase composition of NiFe LDH grown on different substrates. The characteristic peaks of NiFe LDH are clearly observed in all samples, confirming the successful loading of NiFe LDH onto the carbon substrates. As the amount of Co doping increases, the intensity of the diffraction peak at 44.3° is enhanced.



Fig. S8. Raman spectra of the main samples.



Fig. S9. Bader charge plots of NiFe LDH@CoOx-CNF and NiFe LDH@CNF.



Fig. S10. (a) Tauc plot of NiFe LDH and (b) CoO_x -CNF. (c) XPS valence band spectra of NiFe LDH and (d) CoO_x -CNF.



Fig. S11. (a) Mott-Schottky plot, (b) XPS valence band spectra, and (c) Tauc plot of CNF. (d) Energy level diagram of NiFe LDH and CNF.

From the slope of the M-S curve, it can be seen that CNF is a p-type semiconductor. The flat band potential, valence band spectrum, and Tauc plot are used to determine the band information of CNF. The relevant data are shown in **Fig. S11(d)**.



Fig. S12. (a) LSV curves, (b) CV curves at the 50th cycle, (c) overpotential values, and (d) Tafel plot of NiFe LDH@CNF, NiFe LDH@CoO_x-CNF-1, NiFe LDH@CoO_x-CNF, and NiFe LDH@CoO_x-CNF-4.

Fig. S12 illustrates the effect of different Co introduction amounts on the OER performance of NiFe LDH@CoO_x-CNF. As shown in **Fig. S12(a)**, at a current density of 10 mA cm⁻², the overpotentials for NiFe LDH@CNF, NiFe LDH@CoO_x-CNF-1, NiFe LDH@CoO_x-CNF, and NiFe LDH@CoO_x-CNF-4 are 254, 232, 225, and 245 mV, respectively. As the amount of Co source increases, the reduction peak in the LSV curves gradually shifts to lower potentials, attributed to the strengthening interaction between NiFe LDH and CoO_x-CNF. **Fig. S12(b)** presents the CV curves of the catalysts after activation. Notably, as the Co amount increases from 0 to 0.2 g, the area of the CV curves increases, indicating enhanced redox activity [7]. However, when the Co amount reaches 0.4 g, the integral area of the CV curve for NiFe LDH@CoO_x-CNF-4 decreases, indicating a deterioration in performance. Therefore, NiFe LDH@CoO_x-CNF exhibits the lowest OER reaction overpotential and the fastest reaction kinetics

(Fig. S12c, d).



Fig. S13. CV curves of (a) NiFe LDH@CoO_x-CNF, (b) CoO_x-CNF, (c) NiFe LDH, and (d) RuO₂ at different scan rates in the non-Faradaic region (1.01-1.02 V).



Fig. S14. CV curves of (a) CNF and (b) nickel foam at different scan rates in the non-Faradaic region (1.01-1.02 V). (c) C_{dl} values of CNF and nickel foam.



Fig. S15 (a) ECSA values of the main catalysts and (b) LSV curves normalized by ECSA.



Fig. S16 Operando EIS plots and the corresponding fitting curves of (a) NiFe LDH@CoOx-CNF, (b) CoO_x-CNF, and (c) NiFe LDH.



Fig. S17. The TEM image of NiFe LDH@CoO_x-CNF after electrochemical tests.



Fig. S18. High-resolution Fe 2p XPS spectra of NiFe LDH@CoO_x-CNF before and after the OER tests.



Fig. S19. High-resolution Fe 2p XPS spectra of NiFe LDH@CoO_x-CNF before and after the OER tests.



Fig. S20. Optimized structural models of (a) NiFe LDH@CoOx-CNF, NiFeOOH@CoOx-CNF, (c) NiFe LDH@Co-CNF, (d) NiFeOOH@CNF, (e) NiFe LDH, and (f) NiFeOOH.

Element	С	0	Со	Ni	Fe
Atomic fraction (%)	62.3	23.4	0.8	10.0	3.5
Mass fraction (%)	38.3	19.1	2.4	30.2	10.0

Table S1. Element content in NiFe LDH@CoO_x-CNF

 Table S2. Comparison of OER activity of NiCo LDH@CoOx-CNF with other

 recently reported analogous catalysts in 1.0 M KOH.

Materials	Substrate	$\eta_{10}(mV)$	Tafel (mV dec ⁻¹)	Reference
NiFeP _x	Ni foam	234	41	[8]
Co-CAT/NiFeLDH/CNFs	Carbon nanofiber	330	85	[9]
Co-C@NiFe LDH	Glassy carbon	249	58	[10]
FeNi ₃ @GCDs	Glassy carbon	238	49	[11]
NiFe-[Cr(C ₂ O ₄) ₃] ₃ -LDHs	Glassy carbon	236	60	[12]
CoP/FeOOH/CNF	Glassy carbon	250	57	[13]
CoO/NiFe LDH	Carbon cloth	290	107	[14]
NiFe LDH@Fe-N-CNFs	Glassy carbon	360		[15]
MoS ₂ /NiFe LDH	Carbon cloth	257	95	[16]
Cr-NiFe ₂ O ₄ @NC	Ni foam	241	65	[17]
H-CoS _x @NiFe LDH	Ni foam	250	49	[18]
NiFe ₂ O ₄ @rGo	Ni foam	272	64	[19]
NiFe LDH@Cu _x O	FTO	270	67	[20]
ZnCo ₂ O ₄ @NiFe LDH	Ni foam	249	97	[21]
HPGC@NiFe	Glassy carbon	265	56	[22]
Ni _{0.83} Fe _{0.17} (OH) ₂	Glassy carbon	245	61	[23]
NiFeCo-LDH/CF	Glassy carbon	249	42	[24]
Ru/NiFe LDH-F/NF	Ni foam	230	50	[25]
Ni-FeLDH@MnCO ₃	Ni foam	275	45	[26]
NiFe LDH@CoO _x -CNF	Ni foam	225	80	This work

Table S3. The total charge transfer resistance (R) value of NiCo LDH@CoO_x-CNF,

Applied potential	$R(\Omega)$ of NiFe LDH@	$\mathbf{P}(\mathbf{O}) \circ \mathbf{f} \mathbf{C} \circ \mathbf{O} = \mathbf{C} \mathbf{N} \mathbf{E}$	$R(\Omega)$ of
(V)	CoO _x -CNF	$R(\Omega 2)$ of COO_x -CNF	NiFe LDH
1.112	2.82×10 ¹¹	1.00×10 ¹¹	2.66×10 ⁴
1.162	4.35×10 ¹⁰	4.05×10 ¹⁰	1.14×10 ⁴
1.212	1.61×10 ³	4.60×10 ¹⁰	5.95×10 ³
1.262	878	4827	4.40×10 ³
1.312	499	918	3.02×10 ³
1.337	366	479	1.47×10 ³
1.362	233	514	838
1.387	134	494	826
1.412	41.6	548	485
1.437	7.66	244	59.4
1.462	3.36	85.2	21.5
1.487	3.17	23.3	11.8
1.512	2.72	6.78	7.37

CoO_x-CNF, and NiFe LDH under different applied potentials.

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