Supporting Information

Preferential Co substitution on Ni sites in Ni-Fe oxide arrays enabling large-current-density alkaline oxygen evolution

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Experimental Section

Chemicals

Ni(NO₃)₂•6H₂O (\geq 99.0%, AR), Co(NO₃)₂•6H₂O (\geq 99.0%, AR), FeSO₄•7H₂O (\geq 99.0%, AR), NaCl (\geq 99.0%, AR), RuO₂ (99.9%), H₂SO₄ and acetone were purchased from Sinopharm Chemical Reagent Co., Ltd. Nafion solution (5 wt %) was purchased from SigmaAldrich Chemical Reagent Co., Ltd. Ni foam was purchase from Shenzhen Meisen electromechanical equipment Co., Ltd. All of the reagents were used without further purification.

Materials Synthesis

Synthesis of Co-NiO/Fe₂O₃ Nanosheet Arrays

In this experiment, Ni foam with geometric area of $2\times 2 \text{ cm}^2$ was firstly washed by acetone, ethanol, and H₂SO₄ aqueous solution (2.0 M), respectively. For the synthesis of Co-NiO/Fe₂O₃ catalyst, the pre-cleaned Ni foam and the mixed solution (20 mL) containing Co(NO₃)₂•6H₂O (12.5 mM) and FeSO₄•7H₂O (12.5 mM) were successively transferred to a 50 mL Teflon-lined autoclave and kept at 80 °C for 12 hrs. Then, the sample was washed with deionized water and absolute ethanol for several times to get hydroxide nanosheet precursors after drying overnight at 60 °C. Subsequently, the Co-NiO/Fe₂O₃ was obtained by calcination the above precursor in air at 450 °C for 2 hrs. The mass loading of the catalysts supported on Ni foam substrate are around 2.15 mg cm⁻².

Synthesis of NiO/Fe₂O₃ Nanosheet Arrays

The NiO/Fe₂O₃ nanosheet arrays were prepared by the same procedure for the Co-NiO/Fe₂O₃ nanosheet arrays except that Ni(NO₃)₂•6H₂O (12.5 mM) to replace Co(NO₃)₂•6H₂O (12.5 mM).

Materials Characterizations

The composition and structure of the as-prepared samples were examined by X-ray diffraction (XRD) on a X-ray diffractometer (SmartLab, Rigaku, Japan) using a Cu K α radiation source (λ = 0.154178 nm). Field-emission scanning electron microscope (FESEM) (SU8020, Hitachi, Japan) and transmission electron microscope (TEM) (JEM-1400flash, JEOL, Japan) images of the

samples were obtained to characterize the morphology and microstructure. The thickness of the sample was measured by atomic force microscopy (AFM) (Dimension Icon, Bruker, Germany). Elemental mapping and select area electron diffraction (SAED) patterns of the samples were taken on a field-emission transmission electron microscope (FETEM) (JEM-2100F, JEOL, Japan). The valence state of the samples was recorded by X-ray photoelectron spectroscopy (XPS) (ESCALAB250Xi, Thermo, America) equipped with a hemispherical energy analyzer and a monochromatic Al Kα source.

Electrochemical Measurements

All electrochemical measurements were carried out on an electrochemical workstation (CHI 760E) via a conventional three-electrode system in 1.0 M O₂-saturated KOH media under ambient conditions. 1 M KOH + 0.5 M NaCl and 1 M KOH + seawater were used to evaluate the OER performance for actual industrial application and the natural seawater was collected from Bohai Sea, China. A Pt net (1×1.5 cm²), an Hg/HgO electrode, and the as-prepared catalysts were employed as the counter, reference electrode and working electrode, respectively. The linear sweep voltammetry (LSV) curves were collected at a scan rate of 5 mV s⁻¹ with 90% iR compensation. The C_{dl} values were evaluated based on cyclic voltammetry (CV) curves under non-Faradaic region. Electrochemical impedance spectroscopy (EIS) was investigated in an O₂-saturated electrolyte with amplitude of 5 mV and the frequency range from 10 kHz to 0.01 Hz. The multicurrent processes and chronopotentiometry curve were investigated without iR compensation.

For comparison, the RuO₂, NiO, Fe₂O₃ were evaluated. The working electrodes of these catalysts were prepared by dispersing 2 mg powders into a water–ethanol solution containing 10 μ L Nafion, respectively, and sonicating for 1 h to form a homogeneous catalyst inks. Then the catalyst ink was cast onto Ni foam and dried in air at room temperature.

Density Functional Theory Calculations

Quantum espresso (QE) based on the pseudopotential plane wave (PPW) method was used to complete density functional theory (DFT) calculations.^{1,2} The perdew-Bueke-Ernzerhof (PBE) functional was employed to describe exchange-correlation effects of electrons.³ Projected augmented wave (PAW) potentials were chosed to describe the ionic cores and take valence

electrons into account using a plane wave basis set with a kinetic energy cutoff of 500 eV.^{4,5} Slab models were built by slicing (111) and (110) planes of NiO and Fe₂O₃ respectively to model the surfaces of materials. An extra vacuum of 12 A were applied along z-direction to avoid interactions. In order to specify the most stable doping sites for Co, different possible sites were calculated in NiO and Fe₂O₃, where the most stable ones were used to carry out further simulation. Further, to explore the effects of NiO/Fe₂O₃, heterojunction models were built considering both un-doped and Co-doped structures. All of the structures were first optimized to reach their most stable configuration. All of the atom positions were allowed to relax during the geometry optimizations. The Brillouin-zone sampling was conducted using Monkhorst-Pack (MP) grids of special points with the separation of $0.04 \text{ Å}^{-1.6}$ The convergence criterion for the electronic self-consistent field (SCF) loop was set to $1 \times 10^{-5} \text{ eV}/\text{atom}$. The atomic structures, free energy along OER reaction path was calculated. The d-band center was calculated based on the following equation:

$$\epsilon = \frac{\int_{-\infty}^{E_{f}} Ef_{d}(E)dE}{\int_{-\infty}^{E_{f}} f_{d}(E)dE}$$

Where f_d represent the density of states (DOS) of the specified orbital.

Differential charge density was calculated by $D_{diff} = D_{tot} - D_{sub} - D_{abs}$, where D_{tot} , D_{sub} and D_{abs} were charge density of whole system, density of substrate and density of absorbed molecules respectively.



Fig. S1 (a) XRD pattern and XPS spectra of (b) Ni 2p, (c) Co 2p and (d) Fe 2p for the obtained precursor after hydrothermal reaction of Ni foam, $Co(NO_3)_2$ and $FeSO_4$.

Fig. S1a reveals the formation of $3Ni(OH)_2 \cdot 2H_2O$ and FeOOH precursors after hydrothermal reaction containing both Co²⁺ and Fe²⁺. The formation of FeOOH may be due to that the hydrolysis product Fe(OH)₂ could be easily oxidized to FeOOH by the dissolved oxygen. Furthermore, the Co signals could be detected in the XPS spectra although the corresponding diffraction peaks related to Co could not observed in the XRD patterns for the precursors. The high-resolution Co 2p and Ni 2p spectra of the precursors can be assigned to Ni²⁺, Ni³⁺ and Co²⁺, Co³⁺, respectively, and Fe 2p regions can be fitted into a pair of Fe³⁺ peaks and satellite peaks in the precursors (Fig. S1b-d).



Fig. S2 (a-b) FESEM images of pure Ni foam. (c-d) FESEM images, (e) SEM and (f) the corresponding EDS spectrum of the obtained Ni foam after hydrothermal reaction of Ni foam and FeSO₄ aqueous solution (without the addition of $Co(NO_3)_2$).

When only Ni foam and FeSO₄ are added into the hydrothermal system, as shown in the Fig. S2, the FESEM images exhibit that no nanosheets or other nanostructures are observed on the surface of the skeleton of smooth Ni foam, which is also confirmed by EDS results, suggesting that the co-deposition reaction cannot occur without Co^{2+} ions assistance.



Fig. S3 (a) XRD pattern, (b) survey XPS spectrum and high resolution (c) Fe 2p and (d) Ni 2p XPS spectra for the obtained Ni foam after hydrothermal reaction of Ni foam and FeSO₄ aqueous solution (without the addition of $Co(NO_3)_2$).

In the absence of Co^{2+} , only the diffraction peaks of Ni foam substrate could be observed after hydrothermal reaction (Fig. S3a), and no nanosheet arrays or other nanostructures could be observed on Ni foam (Fig. S2). Besides, the survey and Fe/Ni 2p XPS spectra confirm the existence of Ni and O elements while Fe signals could not be detected (Fig. S3b-d).



Fig. S4 The pH values of the solution for the synthesis of hydroxide precursors in hydrothermal reaction.



Fig. S5 XRD pattern of NiO/Co₃O₄.

 $Co(NO_3)_2$ •6H₂O and Ni foam were used to prepare Co-Ni based hydroxides under hydrothermal condition. Then, the hydroxide precursors were further treated in air at 450 °C for 2 hrs. Crystalline Co₃O₄ and NiO phases could be detected in the XRD pattern. In contrast, no crystalline Co₃O₄ could be found in the Co-NiO/Fe₂O₃. This suggests that substitutional incorporation of Co into the lattices of NiO or Fe₂O₃ may occur during the heat treatment of Fe-Co-Ni based hydroxide precursors.



Fig. S6 The top views of (a) pure NiO and (b) pure Fe_2O_3 .



Fig. S7 Rietveld refined XRD pattern of NiO/Fe₂O₃.

The XRD pattern of NiO/Fe₂O₃ is refined, which confirms that the as-prepared NiO/Fe₂O₃ also consists of NiO, Fe₂O₃, and Ni compared with the Co-NiO/Fe₂O₃ (Ni was peeled from Ni foam.)



Fig. S8 (a, b) FESEM images and (c) EDS spectrum of the obtained precursor after hydrothermal reaction of Ni foam, $Co(NO_3)_2$ and FeSO₄.

The Fe-Co-Ni based precursors exhibit typical vertically arrayed nanosheet morphology with Ni, Co, Fe and O elements.



Fig. S9 (a) FESEM image, (b) EDS spectrum, and (c) elemental mapping images of the NiO/Fe_2O_3 .

We used the similar method to prepare the Ni and Fe based precursors with the addition of Ni^{2+} instead of Co^{2+} in the hydrothermal reaction. The obtained precursors were then calcined at the same condition. The resulting NiO/Fe₂O₃ also maintains the morphology of nanosheets and shows uniform elemental distribution.



Fig. S10 (a) Polarization curves and (b) long-term stability tests at a constant current density of 500 mA cm⁻² for the Co-NiO/Fe₂O₃ electrode in different electrolytes.

Alkaline simulated seawater (1 M KOH + 0.5 M NaCl) and alkaline natural seawater (1 M KOH + seawater) electrolytes were used to further evaluate the OER performance of the Co-NiO/Fe₂O₃. The Co-NiO/Fe₂O₃ shows overpotentials of 265 and 300 mV at 100 mA cm⁻² in alkaline simulated seawater, and 313 and 373 mV at 500 mA cm⁻² in alkaline natural seawater. The Co-NiO/Fe₂O₃ also maintains stable potentials at 500 mA cm⁻² under continuous operation for 50 hrs both in either alkaline simulated seawater or alkaline natural seawater.



Fig. S11 CV curves of different catalysts under various scan rates (40 to 180 mV s⁻¹). (a) NiO, (b) Fe₂O₃, (c) NiO/Fe₂O₃, (d) Co-NiO/Fe₂O₃.

CV curves of different catalysts NiO, Fe₂O₃, NiO/Fe₂O₃ and Co-NiO/Fe₂O₃ have been added as shown in Fig. S11. We calculated the C_{dl} values of different electrocatalysts based on these CV curves and further estimated the electrochemical surface areas for different electrocatalysts. The C_{dl} values were calculated to be 0.69, 0.64, 1.86 and 3.16 mF cm⁻² for NiO, Fe₂O₃, NiO/Fe₂O₃, and Co-NiO/Fe₂O₃, respectively, suggesting that the Co-NiO/Fe₂O₃ can offer more abundant active sites as well as higher interfacial contact areas.



Fig. S12 Linear sweep voltammetry curves normalized by electrochemical double-layer capacitance.

To further evaluate the intrinsic catalytic activity of all the catalysts, the electrochemical double layer capacitance (C_{dl}) normalized linear scanning voltammetry (LSV) curves were collected. The Co-NiO/Fe₂O₃ requires only 204 mV overpotential to reach 10 A F⁻¹, which is significantly lower than those for the NiO, Fe₂O₃ and NiO/Fe₂O₃, demonstrating the high intrinsic OER activity of the Co-NiO/Fe₂O₃ with Co substitution.



Fig. S13 Overpotentials at current densities of 100, 200, and 500 mA cm⁻² for seven Co-NiO/Fe₂O₃ electrodes prepared in seven parallel experiments.

Seven Co-NiO/Fe₂O₃ electrodes exhibit nearly identical potentials at 100, 200 and 500 mA cm^{-2} in seven parallel experiments, demonstrating the superb reproducibility in this work.



Fig. S14 The calculated DOS for (a) NiO and (b) Co doped NiO.

Fig. S14 shows partial DOS plots before and after Co substitution. Asymmetric DOS is observed due to magnetic Ni atoms. Pure Ni has a wide band gap of the electrons (0.80 eV) and has a semiconducting band structure, showing a semiconductor nature. After Co substitution, a significantly narrower band gap of 0.42 eV is observed for Co doped NiO, revealing the highly improved electronic conductivity of the Co-NiO/Fe₂O₃.



Fig. S15 FESEM images and EDS spectra of the Co-NiO/Fe₂O₃ before (a, b) and after (c, d) chronopotentiometry at 500 mA cm⁻² for 300 hrs.

After long-term OER measurement, the morphology of Co-NiO/Fe₂O₃ remains the original nanosheet structure, revealing the high stability of the electrode. EDS spectrum of the Co-NiO/Fe₂O₃ after chronopotentiometry was further measured to investigate the reason for the slight stability degradation. As shown in Fig. S15d, EDS results confirm the presence of Ni, Co, and Fe after OER stability test. However, the atomic ratio of Co and Fe elements in the Co-NiO/Fe₂O₃ decreases to 2.2 and 8.4 at %, respectively. Co and Fe elements play an important role in the improvement of electrocatalytic performance of the Co-NiO/Fe₂O₃. The Co and Fe leaching during OER process may cause the slight stability degradation in a long-term stability test.



Fig. S16 XPS spectra of the Co-NiO/Fe₂O₃ before and after chronoamperometric operation. (a) survey, (b) Ni 2p, (c) Co 2p and (d) Fe 2p XPS spectra.

XPS spectra of the Co-NiO/Fe₂O₃ were collected to show the differences before and after stability test. Obviously, the results verify the presence of Ni, Co and Fe after OER stability test and show that the relative intensities of Ni³⁺ and Co³⁺ peaks are significantly increased, indicating that Ni and Co are oxidized to higher valence states which are considered as the real active sites in the previous reported results.



Fig. S17 Electrocatalytic activity for overall water splitting in 1.0 M KOH aqueous solution. (a) LSV polarization curves of the Co-NiO/Fe₂O₃ || commercial Pt/C cell and the commercial RuO₂ || Pt/C cell. (b) Long-term stability test of the Co-NiO/Fe₂O₃ || commercial Pt/C cell at constant current densities of 500 mA cm⁻².

The LSV polarization curve of the Co-NiO/Fe₂O₃ || Pt/C cell reveals that a low voltage of 1.64 V is required to reach 500 mA cm⁻², while the voltage of commercial RuO₂ || Pt/C cell is 1.65 V at a low current density of 10 mA cm⁻² (Fig. S17a), suggesting the superior activity of the Co-NiO/Fe₂O₃ in overall water splitting. Furthermore, the Co-NiO/Fe₂O₃ || Pt/C cell exhibits high stability for electrolysis of water at 500 mA cm⁻², and the voltage of the cell only increases from 1.64 to 1.72 V after OER test for 50 hrs (Fig. S17b).



Fig. S18 $\triangle G$ of four elementary reactions (M \rightarrow *OH \rightarrow *O \rightarrow *OOH \rightarrow O₂) for OER at Ni and Fe sites on surface model of NiO/Fe₂O₃ and Co-NiO/Fe₂O₃.

The elementary reaction with maximum change of ΔG is identified as rate-determining step (RDS) in the OER process. The second step from *OH to *O on the Ni and Fe sites in the NiO/Fe₂O₃ and Co-NiO/Fe₂O₃ has the lowest ΔG value and could be considered as RDS in our work. Co substitution in the Co-NiO/Fe₂O₃ reduces the energy barriers of RDS for both Ni (from 1.31 eV to 1.26 eV) and Fe (from 1.74 eV to 1.68 eV) sites, which contributes to the enhancement of OER activity.



Fig. S19 Projected density of states (PDOS) of Fe *d* orbitals for (a) NiO/Fe₂O₃ and (b) Co-NiO/Fe₂O₃ at *OH intermediates.

The *d*-band center of Ni is -5.5427 eV and that of Fe is -7.1802 eV with respect to the Fermi energy level in the Co-NiO/Fe₂O₃. For the NiO/Fe₂O₃, the *d*-band center of Ni is -5.8353 eV and that of Fe is -7.3221 eV relative to the Fermi energy level. The *d*-band centers of Ni and Fe in the Co-NiO/Fe₂O₃ are closer to the Fermi energy level, implying the better OER activity after Co substitution.

Phase No.	F	e ₂ O ₃	Ni	Co-NiO
Space group	F	R-3c	Fm-3m	R-3m
	a(Å)	5.0415(11)	3.52544	2.9556(20)
Lattice parameters	b(Å)	5.0415(11)	3.52544	2.9556(20)
	c(Å)	13.7574(62)	3.52544	7.2271(13)

Table S1. Lattice and structure parameters of the Co-NiO/Fe₂O₃ from XRD Rietveld refinement.

Phase No.	F	e ₂ O ₃	Ni	NiO
Space group	R	-3cH	Fm-3m	R-3mH
	a(Å)	5.0489(23)	3.5238	2.950(15)
Lattice parameters	b(Å)	5.0489(23)	3.5238	2.950(15)
	c(Å)	13.7568(77)	3.5238	7.240(75)

Table S2. Lattice and structure parameters of the NiO/Fe₂O₃ from XRD Rietveld refinement.

Table	S3. OI	ER	performance	es of	the (Co-NiC	P/Fe_2O_3	compared	with	other	advanced	transition
metal-b	ased e	lect	trocatalysts i	n 1.0	M K	OH alk	aline so	lution.				

Cotolyoto	Current density	Overpotential	Tafel slope	Stability	Poforonoo	
Catalysis	[mA cm ⁻²]	[mV]	[mV dec ⁻¹]	Stability	Kelelelice	
	100	220	33.0	300b@500 mA cm-2	This work	
	500 230			THIS WORK		
NiCo ₂ Fe O ₄	NiCo _{2-x} Fe _x O ₄ 10 274 42 100 ~320		42	25h@10 mA cm ⁻²	7	
					·	
CeO _{2-x} –FeNi	10	195	43	48h@10 mA cm ⁻²	8	
FeCoSeO _x	10	294	45.1	40h@10 mA cm ⁻²	9	
Cu@CeO2@NiFeCo-0.25	10	230	32.7	30h@10 mA cm ⁻²	10	
Fe-Co-O/Co@NC-mNS/NF	10	257	41.56	50h@10 mA cm ⁻²	11	
CoNiFeO _x -NC	100	286	64.05	40h@1.49V	12	
NiFe ₂ O _{4-x} /NMO-25	100	304	42.7	40 h@200 mA cm ⁻²	13	
CoNi/CoFe ₂ O ₄ /NF	100	290	45	48 h@100 mA cm⁻²	14	
Ir-NiO	10	215	38	10 h@10 mA cm⁻²	15	
Fe-NiO-Ni CHNAs	10	245	43.4	24 h@10 mA cm ⁻²	16	
Co ₃ O ₄ /CoO	10	302	68.6	4*10 ⁴ s@10 mA cm ⁻²	17	
CoVFeN@NF	10	212	34 8	50h@100 mA cm ⁻²	18	
	100	264	0.110		10	
(NilFeam) P	10	166	59.3	24h@10 mA cm-2	19	
(11), 01-x/2	100	~250	00.0	24h@100 mA cm ⁻²	10	
Cu@NiFe LDH	10	199	27.8	48h@10 mA cm ⁻²	20	
	100	281		48h@100 mA cm ⁻²		
Ni₅Co₃Mo-OH	100	304	56.4	100h@100 mA cm ⁻²	21	
Ni₃S₂/MnO₂	10	260	61	48 h@100 mA cm ⁻²	22	
0 2 2	100	348		U		
e-ICLDH@GDY/NF	100	249	43.6	80 h@100 mA cm ⁻²	23	
S-doped (Ni,Fe)OOH	100	281	48.9	100 h@100 mA cm ⁻²	24	
NiMoO _x /NiMoS	100	225	34	25 h@100 mA cm ⁻²	25	

Table S4. The EIS fit	tting data of different	catalysts at 1.47 V	(vs RHE).
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Sample	R _s [Ω]	R _{ct} [Ω]	CPE₁[F]
Co-NiO/Fe ₂ O ₃	1.028	1.015	0.5262
NiO	3.078	7.818	0.6797
Fe ₂ O ₃	1.618	5.834	0.7246
NiO/Fe ₂ O ₃	1.642	1.823	0.5580

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