

Supporting information

A Highly Efficient FeP/CeO₂-NF Hybrid Electrode for the Oxygen Evolution

Reaction

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Experimental section:

Chemicals and materials

The materials in the experiment were used directly without any purification: Cerium (III) nitrate hexahydrate (Ce(NO₃)₃·6H₂O, Sinopharm Chemical Reagent CO., Ltd, 99.0%), Iron(III) nitrate nonahydrate (Fe(NO₃)₃·9H₂O, Sinopharm Chemical Reagent CO., Ltd, 98.5%), Ammonium oxalate ((NH₄)₂C₂O₄·H₂O, Tianjing Damao Chemical Reagent CO., Ltd, 99.8%), Sodium chloride (NaCl, Tianjing Damao Chemical Reagent CO., Ltd, 99.5%), Potassium hydroxide (KOH, Aladdin Chemical Reagent CO., Ltd, 95%), Ni foam (1 cm×2 cm, thickness 1.5 mm, bulk density 200 mg/cm²) was purchased from Suzhou jiashide metal foam Co., Ltd.

Fabrication of FeP/CeO₂-NF, FeP-NF and CeO₂-NF electrodes

Firstly, NF (1 cm×2 cm) was washed by dilute HCl, ethanol and deionized water each 30 minutes to get rid of the surface oxide and pollutants, and dried in the air for subsequent use. In a typical procedure, first, CeO₂ was electrodeposited on the NF in a solution containing 2 mM Ce(NO₃)₃, 10 mM NaCl at 70 °C under the current density of 0.25 mA/cm² for 10 min. Second, FeOOH particles were electrodeposited on the CeO₂-NF electrode in a solution containing 1 mM Fe(NO₃)₃, 2.5 mM (NH₄)₂C₂O₄, 2 mM NaCl at 50 °C

under the current density of 0.2 mA/cm² for 10 min. Then the electrodes were washed by deionized water, and dried in the air. After that, the FeOOH/CeO₂-NF electrodes and NaH₂PO₂ were put in a porcelain boat and calcinated for 2 h at 350°C in a flowing Ar atmosphere. Finally, the FeP/CeO₂-NF electrode was obtained. The FeP-NF electrode was prepared in the same way without CeO₂.

Material characterization

X-ray diffraction (XRD) measurements were collected by a D/max-2400 diffractometer. The measurement was performed from 10° to 80° and the scanning speed was 10 degrees per minute. The microstructure and morphology of synthesized materials were observed by Nova NanoSEM 450 equipment. The energy-dispersion X-ray (EDX) spectrum analysis and elemental distributions mapping were obtained by using the energy-dispersion X-ray fluorescence analyzer. Transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HRTEM) was received by using FEI TF30. The X-ray photoelectron spectroscopy (XPS) images were recorded by a Thermo ESCALAB XI+. And the binding energy was calibrated according to C 1s (284.8 eV).

Electrochemical characterization

All the electrochemical characterization was performed by a computer-controlled electrochemical workstation (CHI 660E Shanghai Chenhua Instrument Co.,Ltd) at room temperature. In a three-electrode system: A previously prepared electrode was used as the working electrode, a Pt net (1×1 cm) was used as the counter electrode and a HgO/Hg electrode was used as the reference electrode. The reference electrode was calibrated with Ru(bpy)₃ before using. 1M Potassium hydroxide (KOH) solution was used as the electrolyte solution. The potential obtained by the test was converted to reversible hydrogen electrode (RHE): $E_{\text{RHE}} = E_{\text{HgO/Hg}} + 0.0591 \text{ pH} + 0.098 \text{ V}$. The working electrode was activated by performing an electrochemical test of 40 CV scans, which voltage was between 1.1 V and 1.6 V and the scan rate was 20 mv/s. Linear sweep voltammetry (LSV) measurements were performed at a scanning speed at 5 mv/s in 1 M

KOH solution with 90% iR compensation. Long-term stability measurements were obtained by chronopotentiometry experiments at a constant current density of 10 mA/cm² without iR compensation. Tafel curves were derived from Linear sweep voltammetry (with 90% iR compensation) by plotting overpotential η against $\log(J)$. Electrochemical impedance spectroscopy experiments were tested in 1.0 M KOH solution at 1.5 V vs. RHE with the frequency ranged from 10⁻² to 10⁵ Hz. Double-layer capacitance (C_{dl}) was obtained by measuring CV curves at different scanning speeds (10-100 mV/s) in the non-faraday region (1.03-1.13 V vs. RHE). Electrochemical active surface area (ECSA) was determined based on the double-layer capacitance (C_{dl}) by measuring the CV curves of the electrode at different scan rates in a non-Faradic region (1.03~1.13 V vs. RHE). C_{dl} was determined as the linear slope by plotting anodic current density at 1.08 V against the scan rate (10-100 mV/s)

$$C_{dl} = i_c/v \quad ECSA = C_{dl}/C_s$$

i_c is the charging current, v is the scan rate, C_s is the specific capacitance (0.040 mF/cm²), S is the area of the electrode (1 cm²).

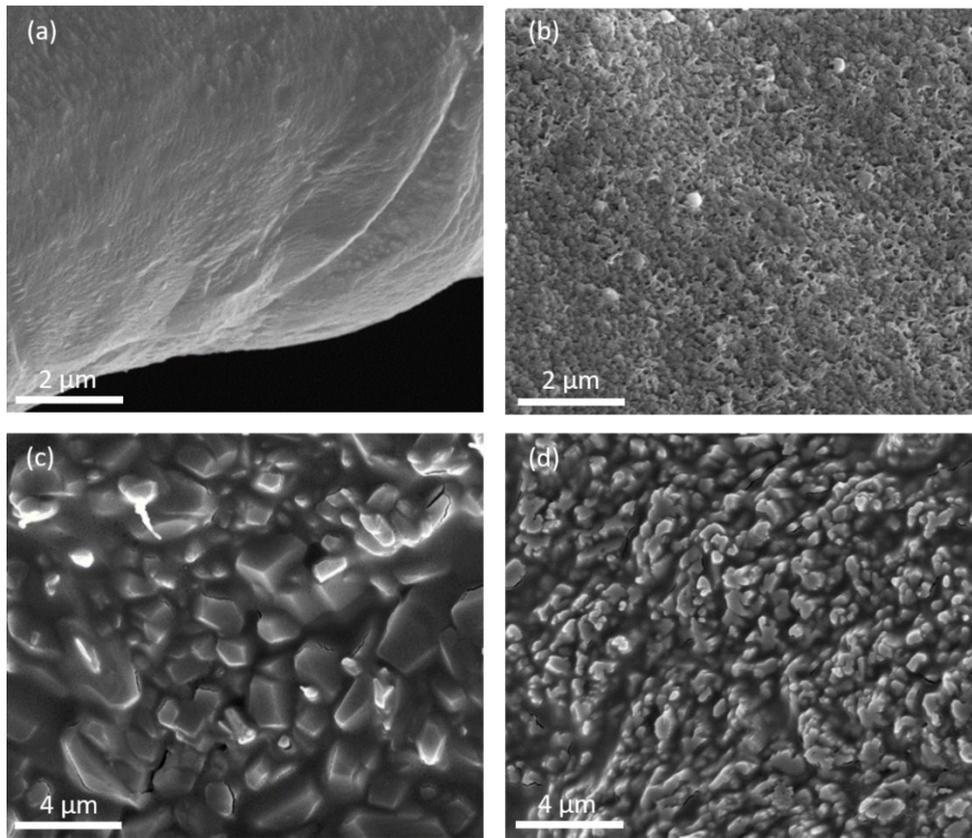


Figure S1. SEM image of the bare NF (a), CeO₂-NF (b), FeP-NF (c) and FeP/CeO₂-NF(d).

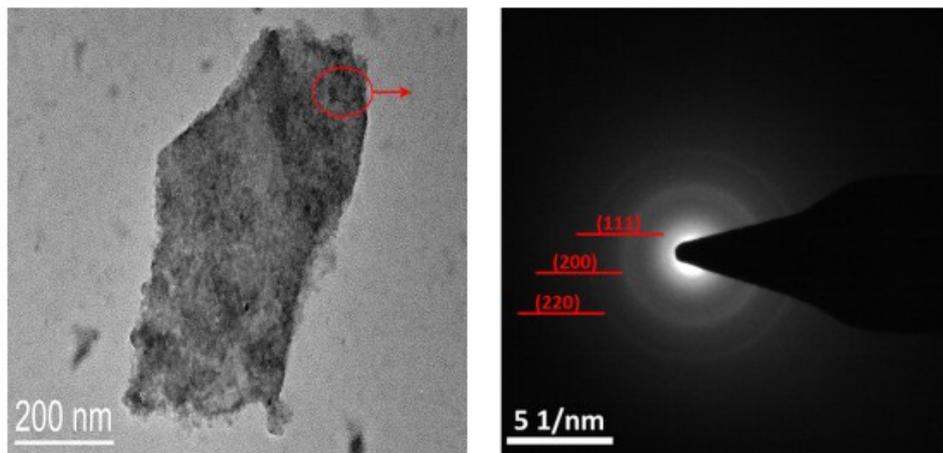


Figure S2. SAED pattern of the FeP/CeO₂-NF electrode.

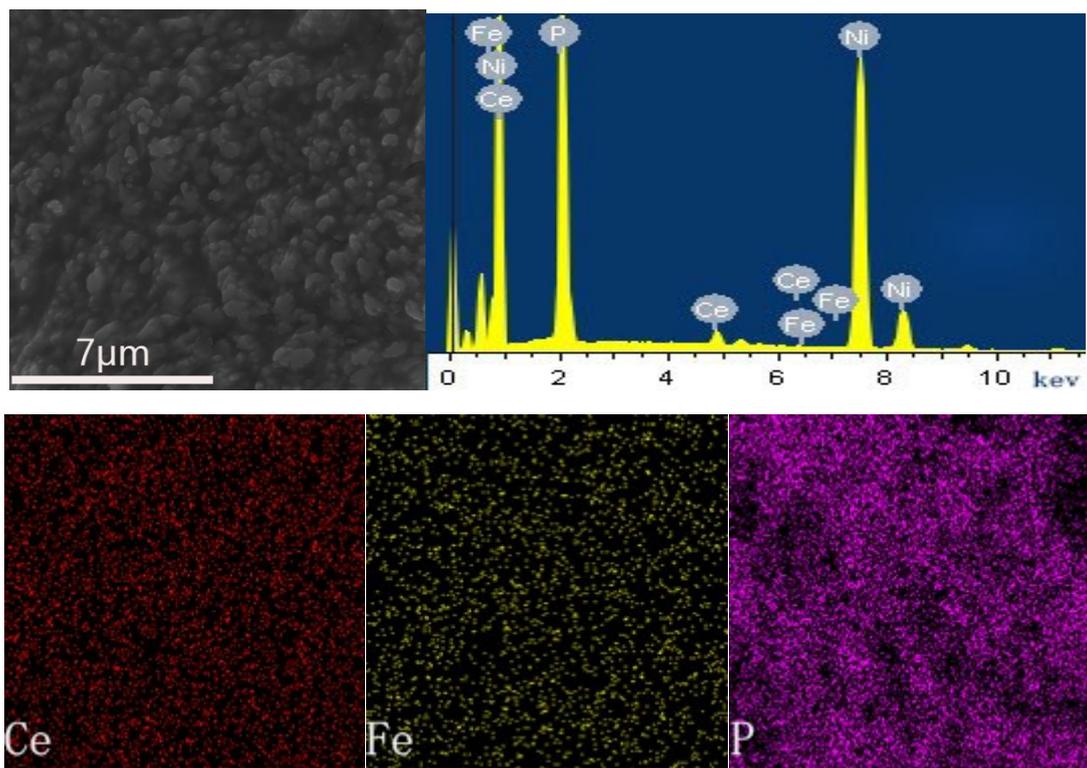


Figure S3. EDX spectra and EDX mapping of the FeP/CeO₂-NF electrode.

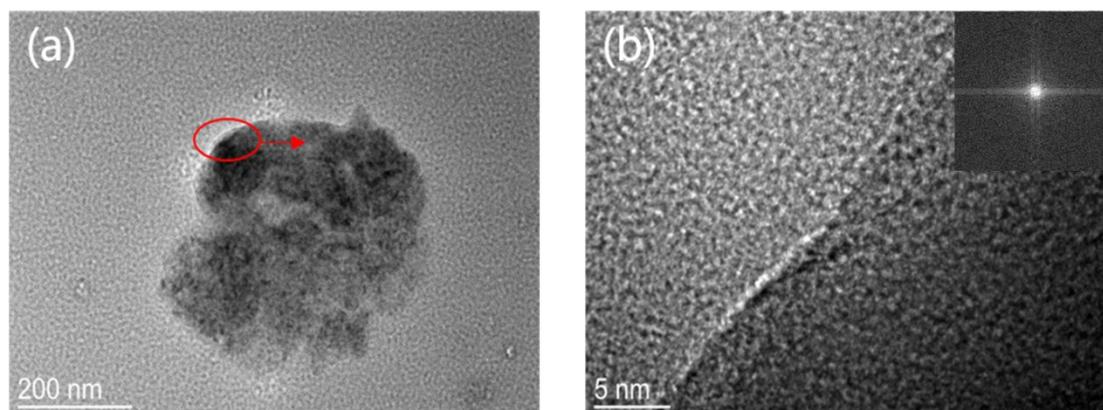


Figure S4. (a and b) TEM and HRTEM images of FeP-NF electrode. Inset in panel b shows the corresponding SAED pattern.

Table S1. Inductively coupled plasma (ICP) analysis of the deposition solution

Solution	Ce(mg/L)
Before deposition	-
After deposition	1.135

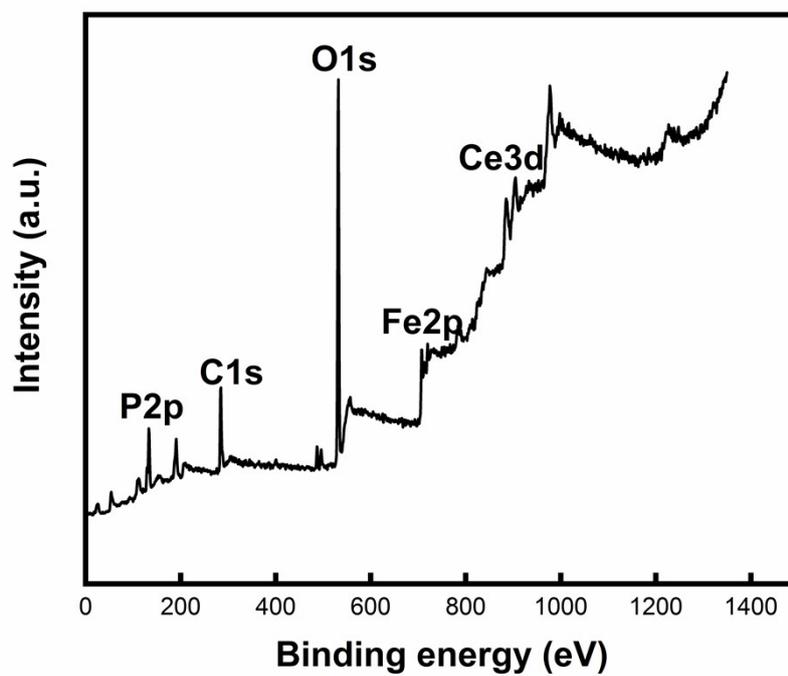


Figure S5. XPS survey spectrum of the FeP/CeO₂-NF electrode before OER.

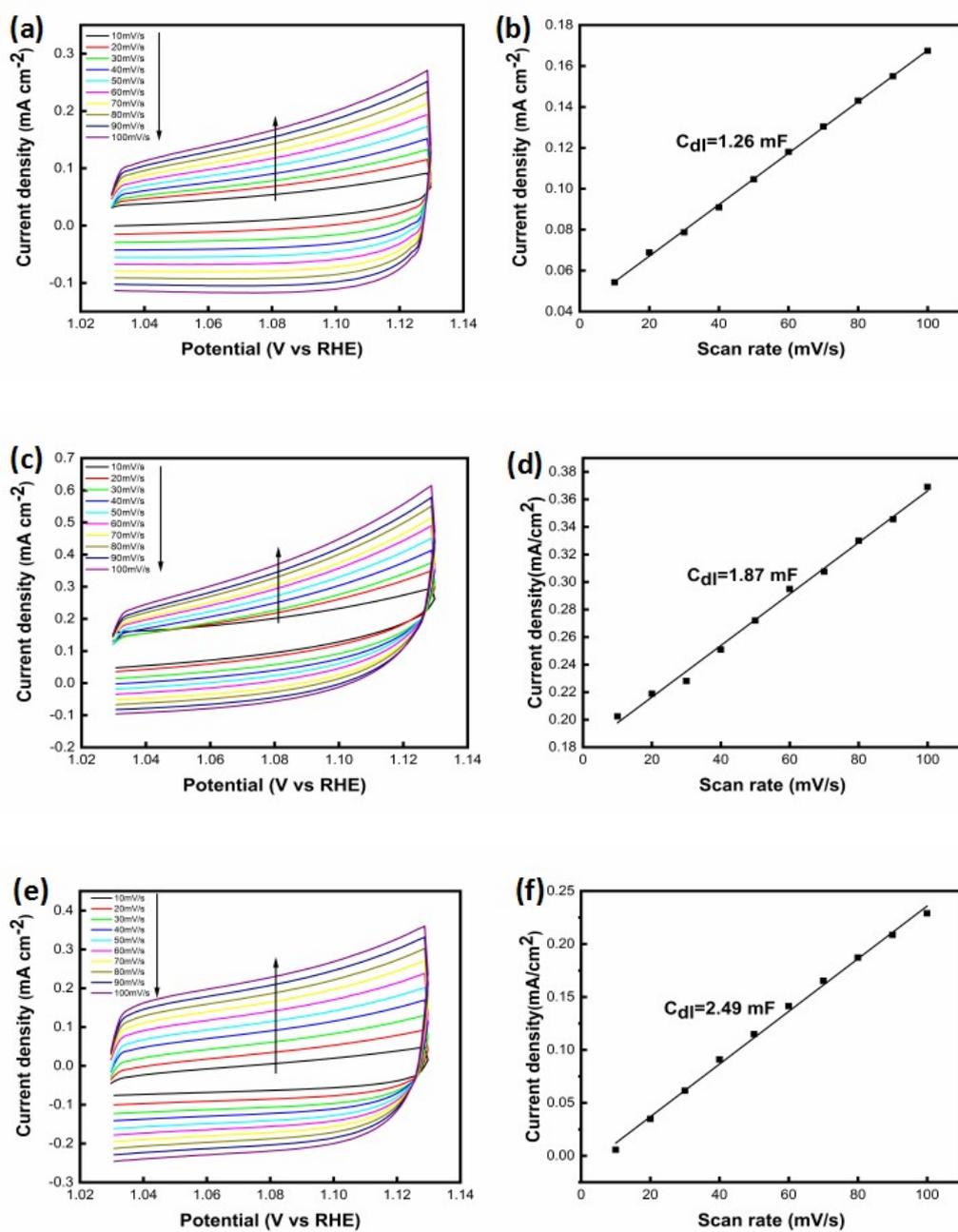


Figure S6. CV curves of CeO₂-NF (a) , FeP-NF (c) and FeP/CeO₂-NF(e) at various scan rates(10 mV/s,20 mV/s,30 mV/s,40 mV/s,50 mV/s,60 mV/s,70 mV/s,80 mV/s,90 mV/s and 100mV/s) in the potential of 1.03-1.13 V and the anodic charging current measured at 1.08 V plotted as a function of scan rate for CeO₂-NF (b) , FeP-NF (d) and FeP/CeO₂-NF(f).

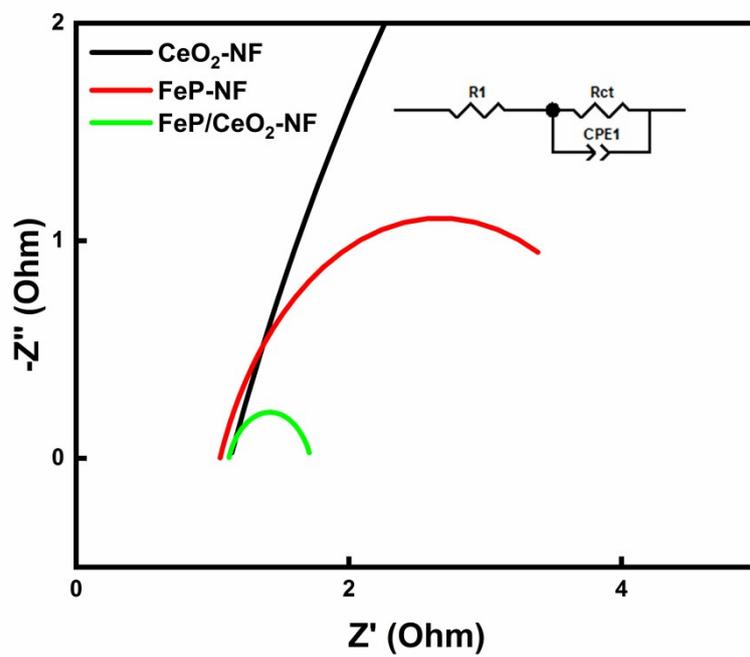


Figure S7. EIS spectra of FeP/CeO₂-NF, FeP-NF and CeO₂.

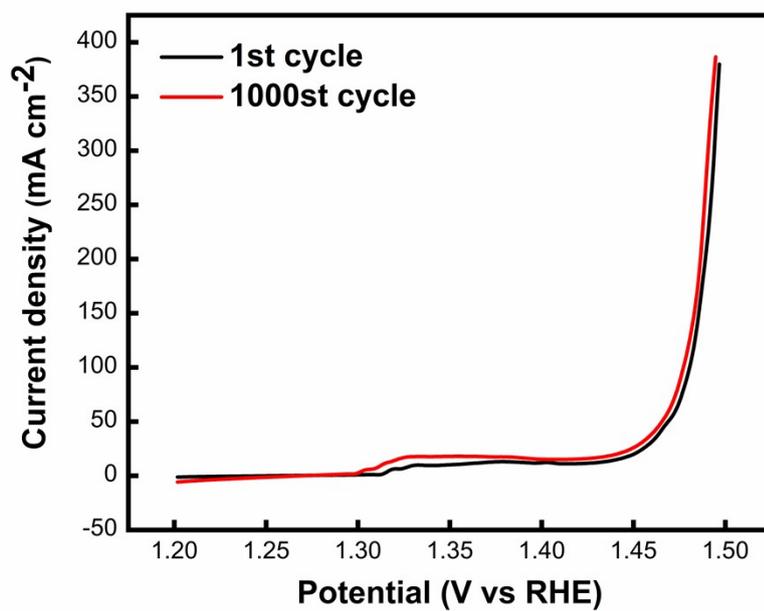


Figure S8. LSV curves before and after 1000 continuous CV cycles.

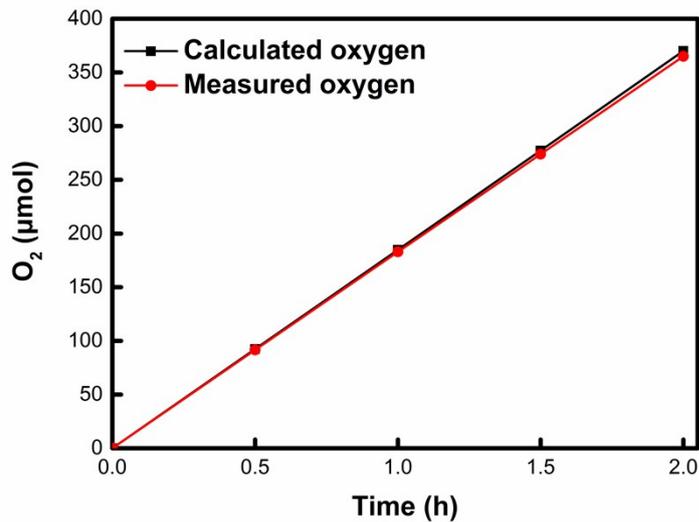


Figure S9. Determination of the Faradicefficiency of the FeP/CeO₂-NF on electrolysis of water at 20 mA/cm² in 1.0 M KOH solution.

Table 2. TOF calculated from current density at 20 mA/cm²

Catalyst	I (mA/cm ²)	Loading (mg)	TOF (s ⁻¹)
FeP/CeO ₂ -NF	20	0.28	0.01

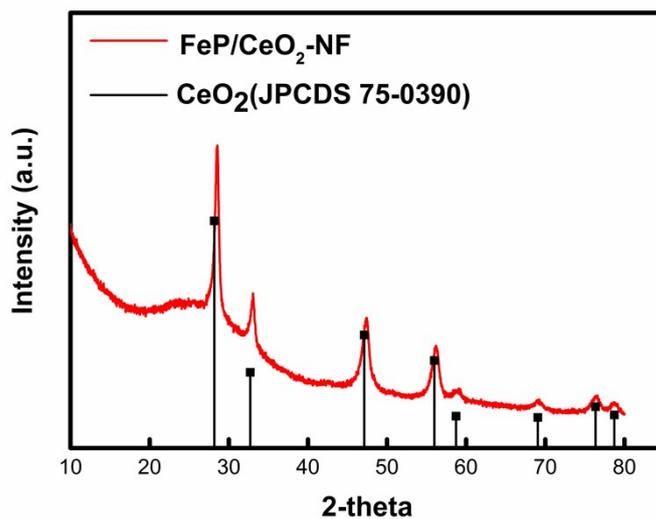


Figure S10. XRD pattern of FeP/CeO₂-NF electrode after OER.

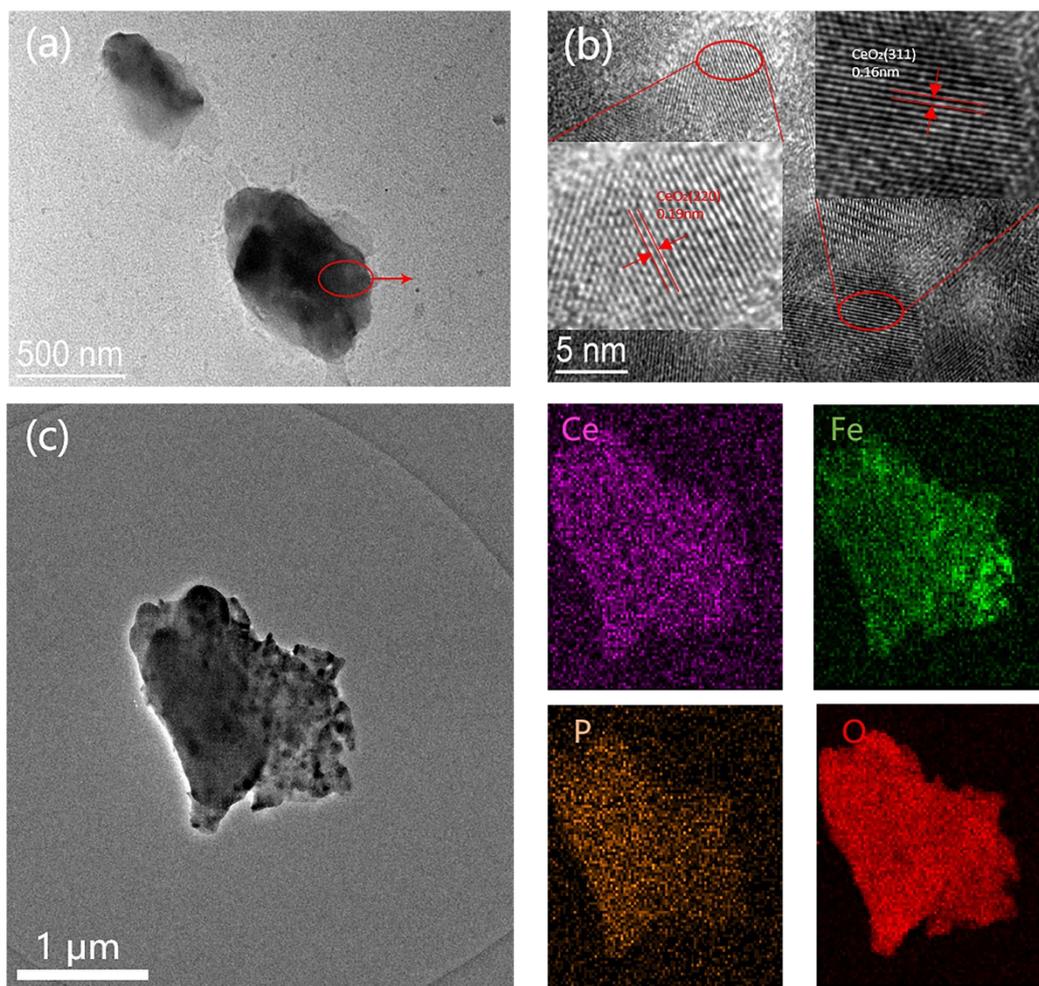


Figure S11. (a and b) The TEM and HRTEM images of FeP/CeO₂-NF electrode after OER; (c) HAADF-STEM image and STEM-EDS element mappings of FeP/CeO₂-NF electrode. Ce pink, Fe green, P yellow, O red.

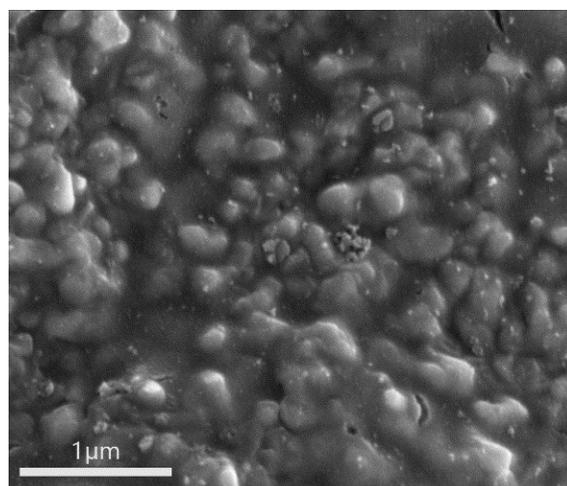


Figure S12. The SEM images of FeP/CeO₂-NF electrode after OER.

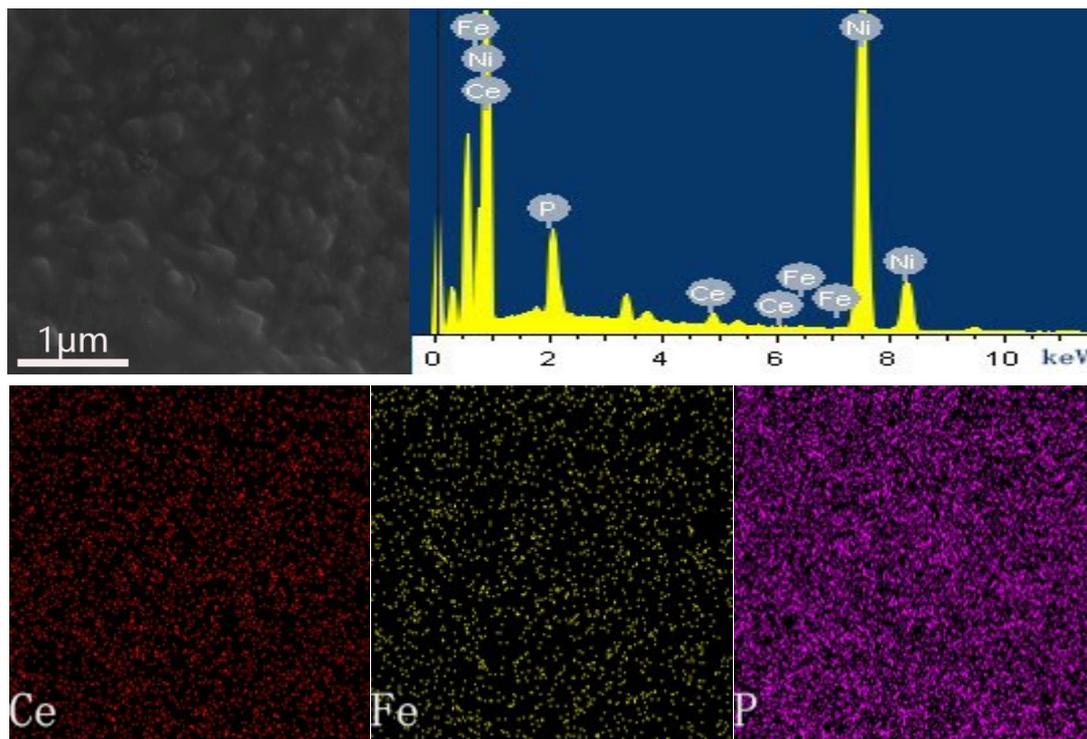


Figure S13. EDX spectra and EDX mapping of the FeP/CeO₂-NF electrode after OER

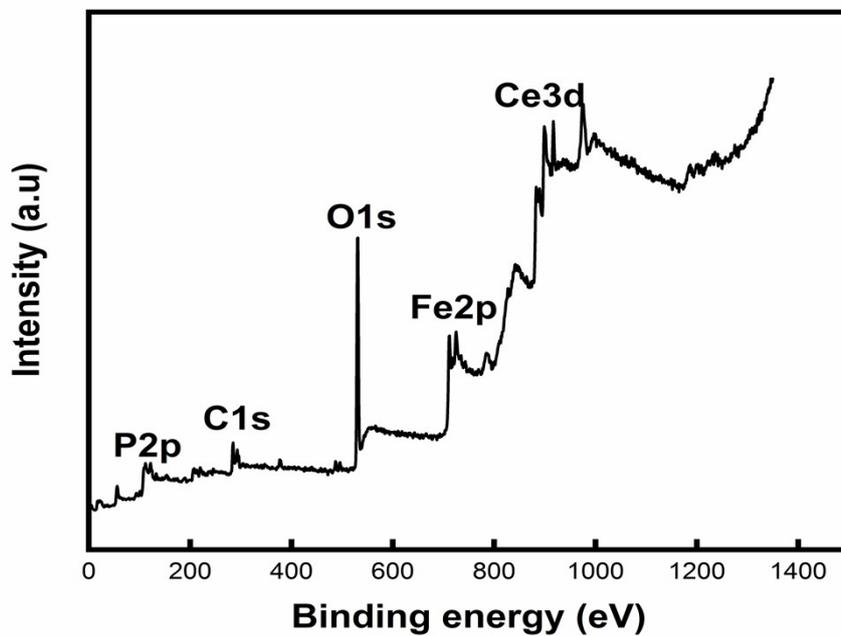


Figure S14. XPS survey spectrum of the FeP/CeO₂-NF electrode after OER.

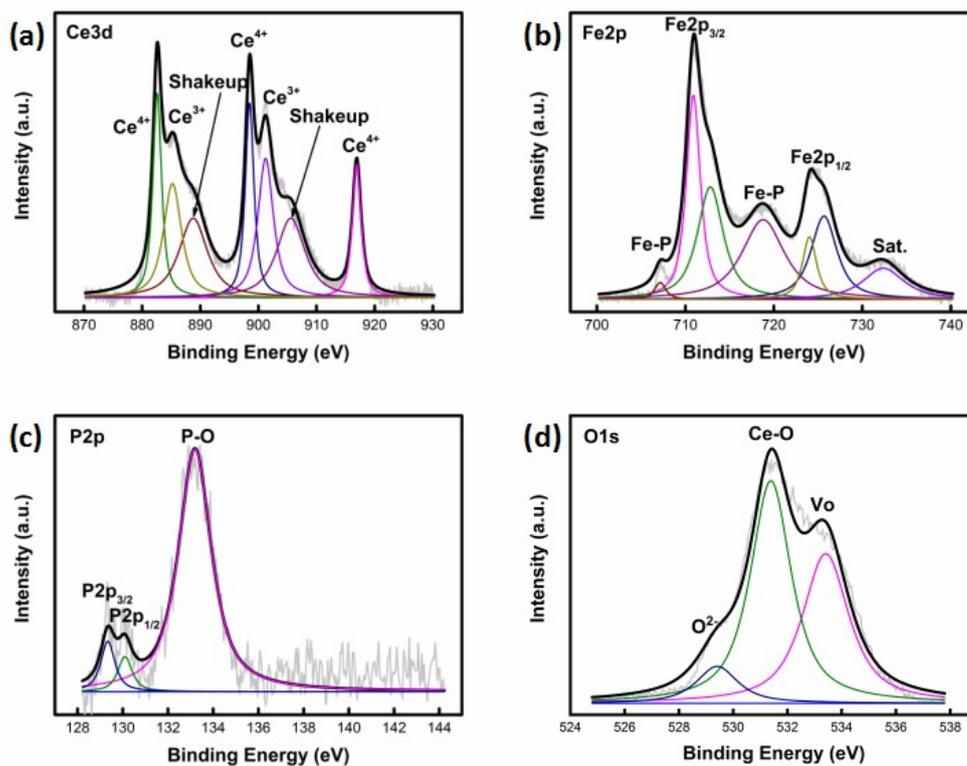


Figure S15. (a) XPS survey spectrum of the FeP/CeO₂-NF electrode and high-resolution XPS spectra of (b) Ce 3d, (c) Fe 2p, (d) P 2p and (e) O 1s after OER.

Table S3. Comparison of the OER performance of FeP/CeO₂-NF with the recently reported catalysts

Electrocatalyst	Electrolyte	I/mA/cm ²	Overpotential η/mV	Tafel slope mV/dec	Refs
FeP/CeO ₂ -NF	1M KOH	100	245	39.1	This work
NiFeP-NF	1M KOH	100	290	57	1
NiFe(3:1)-P	1M KOH	100	290	42.5	2
FeP-rGO	1M KOH	100	330	49.6	3
FeP NRs@CP	1M KOH	100	413.6	63.6	4
FeP/CoP-CC	1M KOH	100	317	67	5
FeP/NF	1M KOH	100	292	54	6
FeP/Ni ₂ P NSs	1M KOH	100	285	50.3	7
Ni ₄ Ce ₁ @CP	1M KOH	100	330	81.9	8
CeO ₂ /FeOOH-NF	1M NaOH	100	320	92.3	9
CeO ₂ /Ni(OH) ₂ /NOSCF	1M KOH	100	450	57	10

(Overpotentials were estimated from the LSV curves presented in the reference literature)

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