

Supporting Information

In-situ synthesis of rosette-like Co-doped FeNiOOH/NF for seawater oxidation

Mingyuan Shi^{a,b}, Tianmi Tang^b, Liyuan Xiao^b, Jingyi Han^b, Xue Bai^b, Yuhang Sun^{a,b},

Yuanyuan Ma^{a,*}, and Jingqi Guan^{b,*}

^a College of Chemistry and Chemical Engineering, Qiqihar University, Heilongjiang Province 161006, China. *E-mail: mayuanyuan1219@126.com (Y.Y. Ma)

^b Institute of Physical Chemistry, College of Chemistry, Jilin University, 2519 Jiefang Road, Changchun 130021, P. R. China. *E-mail: guanjq@jlu.edu.cn (J.Q. Guan)

Synthesis of Co_x-FeNiOOH/NF

Nickel foam (2 cm × 2 cm) was treated in 3 M HCl, distilled water and absolute ethanol for 15 min, respectively. Then, Co(NO₃)₂·6H₂O (0, 0.1, 0.2, 0.4 and 0.6 mmol), 2 mmol Fe(NO₃)₃·9H₂O, 2 mmol Ni(NO₃)₂·6H₂O, 0.24 g NH₂CONH₂ and 0.075 g PVP were dissolved in 20 mL of H₂O via ultrasonic treatment. Afterwards, the mixture was transferred into a 25 mL Teflon-lined stainless steel autoclave, and a piece of NF was inserted, which was heated at 100 °C for 12 hours. After natural cooling, the in situ grown NF was washed several times with distilled water and ethanol and dried at 60 °C for 12 h. After drying, the synthesized sample was named Co_x-FeNiOOH/NF, where x is the molar content of Co.

Characterization

Powder X-ray diffraction (XRD) patterns were collected using a Shimadzu XRD-6000 with CuK α radiation (40 kV, 30 mA). SEM images were recorded on a HITACHI SU8020 field emission scanning electron microscope. Raman spectra were acquired using a Renishaw micro-Raman spectrometer with a 532 nm laser at 0.2 mW. The valence state was determined using XPS recorded on a Thermo ESCALAB 250Xi. The X-ray source selected was monochromatized Al K α source (15 kV, 10.8 mA). Region scans were collected using a 30 eV pass energy. Peak positions were calibrated relative to C 1s peak position at 284.6 eV.

Electrochemical measurements

All electrochemical measurements were performed on a CHI760E electrochemical working station at room temperature. The catalysts were measured in 1.0 M KOH aqueous solution using a typical three-electrode configuration. Linear sweep voltammetry (LSV) polarization curves were acquired at a scan rate of 5 mV \cdot s $^{-1}$. Electrochemical impedance spectroscopy (EIS) measurements were performed at open-circuit potential in the frequency range from 100 kHz to 0.1 Hz with an a.c. perturbation of 5 mV. All potentials measured were calibrated to RHE using the following equation: E (versus RHE) = E (versus SCE) + 0.241 V + 0.0591 pH.

The Faradaic efficiency was calculated using the equations:

$$FE = (V/V_m) e N_A Z/Q$$

where V is the volume of gas (L), V_m is the standard molar volume (22.4 L mol $^{-1}$), e is the electron charge (1.6×10^{-19}), N_A is the Avogadro number (6.02×10^{23}),

Z is the number of electrons needed to form O_2 molecular (for O_2 , $Z = 4$), and Q is the amount of power consumed during electrolysis (C).

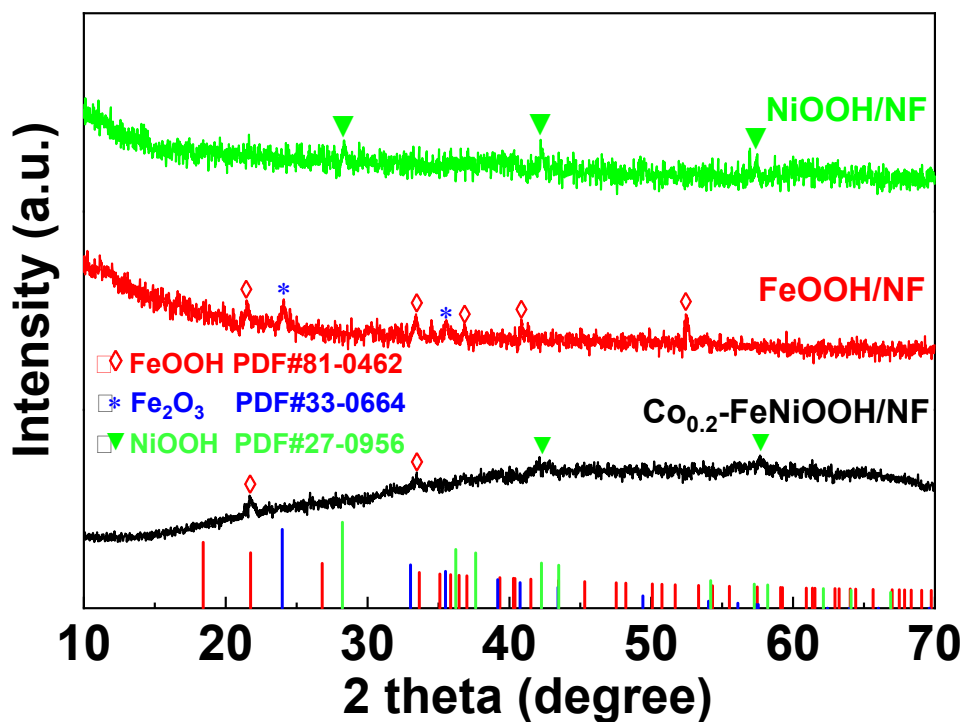


Fig. S1. XRD patterns of NiOOH/NF, FeOOH/NF and Co_{0.2}-FeNiOOH/NF.

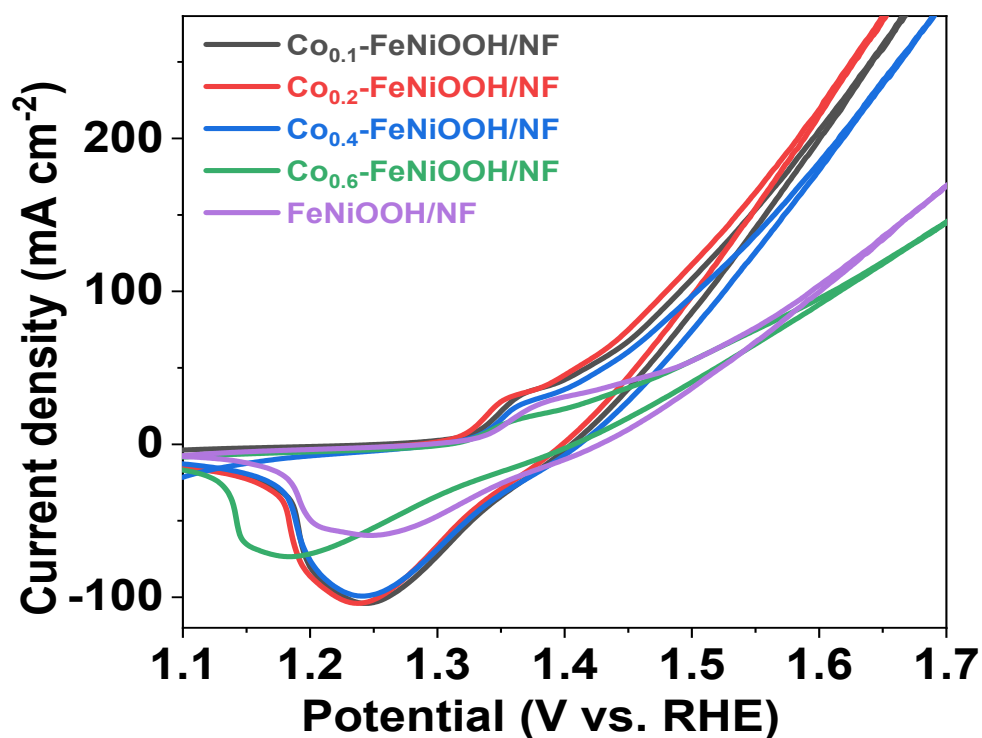


Fig. S2. CV curves of Co_{0.1}-FeNiOOH/NF, Co_{0.2}-FeNiOOH/NF, Co_{0.4}-FeNiOOH/NF, Co_{0.6}-FeNiOOH/NF and FeNiOOH/NF at a scan rate of 5 mV/s.

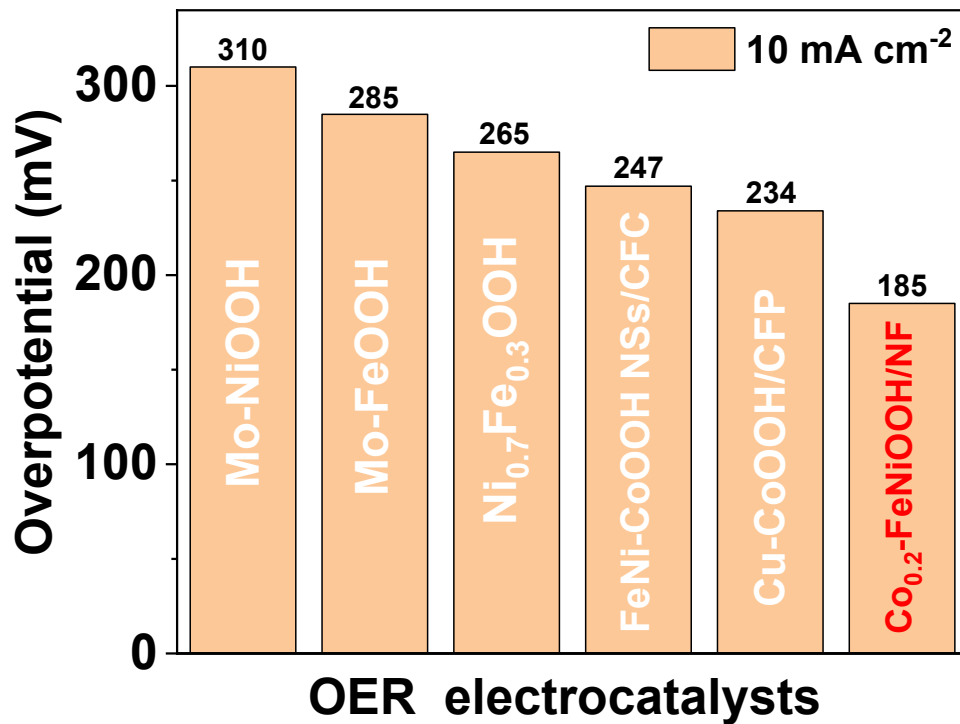


Fig. S3. Overpotential comparison at 10 mA cm⁻².

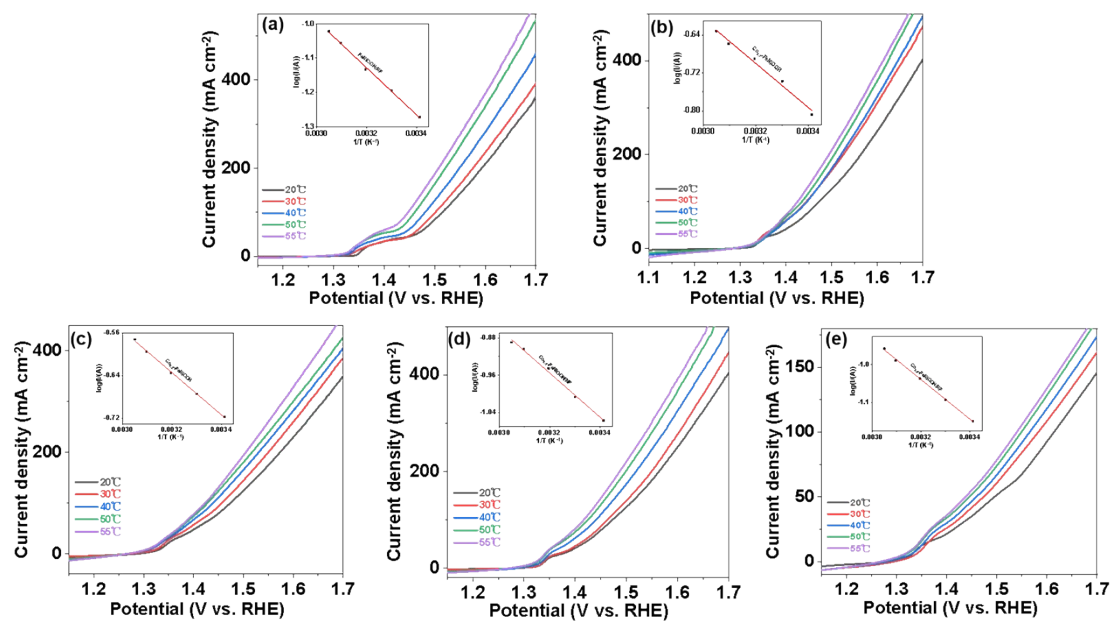


Fig. S4. OER polarization curves of FeNiOOH/NF, Co_{0.1}-FeNiOOH/NF, Co_{0.2}-FeNiOOH/NF, Co_{0.4}-FeNiOOH/NF and Co_{0.6}-FeNiOOH/NF in three-electrode configuration in 1 M KOH at 20 °C, 30 °C, 40 °C, 50 °C and 55 °C.

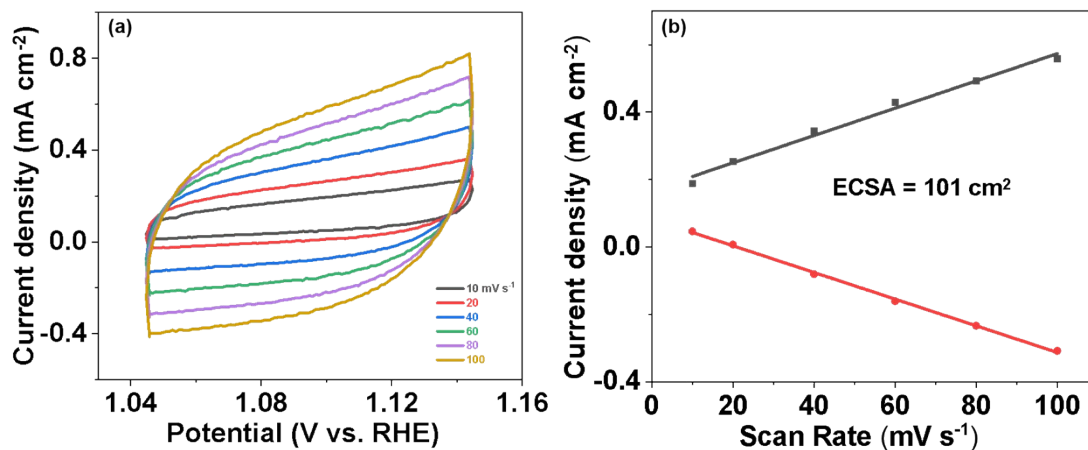


Fig. S5. (a) CVs of the $\text{Co}_{0.2}\text{-FeNiOOH/NF}$ measured in a non-Faradaic region at different scan rate. (b) The cathodic and anodic currents measured as a function of the scan rate.

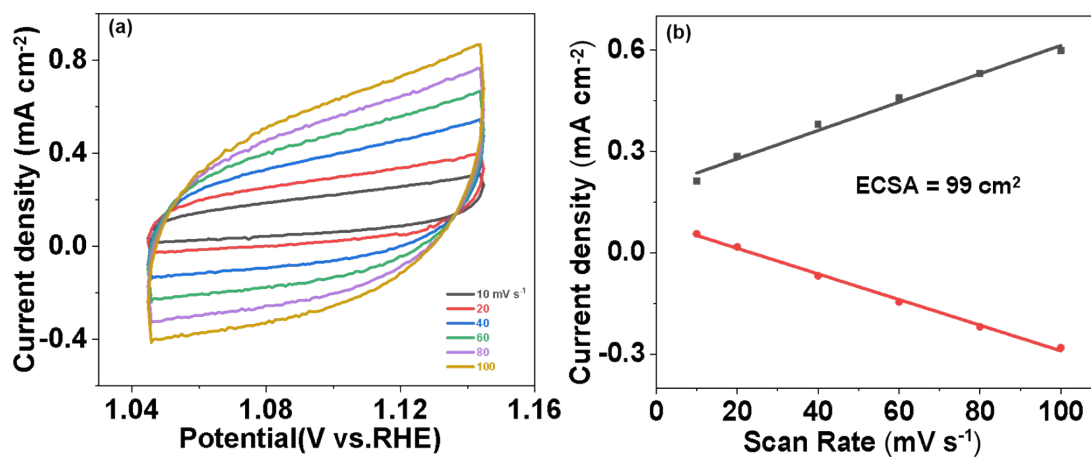


Fig. S6. (a) CVs of the $\text{Co}_{0.1}\text{-FeNiOOH/NF}$ measured in a non-Faradaic region at different scan rate. (b) The cathodic and anodic currents measured as a function of the scan rate.

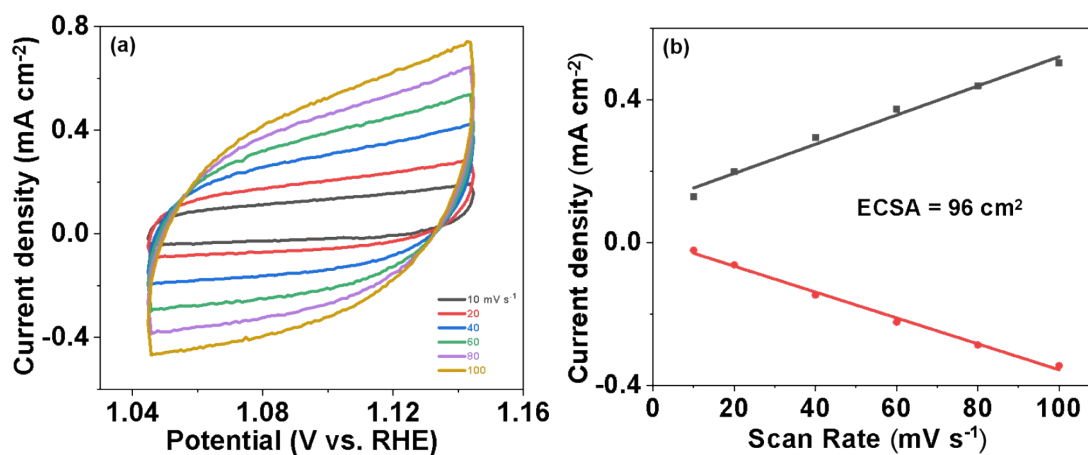


Fig. S7. (a) CVs of the Co_{0.4}-FeNiOOH/NF measured in a non-Faradaic region at different scan rate. (b) The cathodic and anodic currents measured as a function of the scan rate.

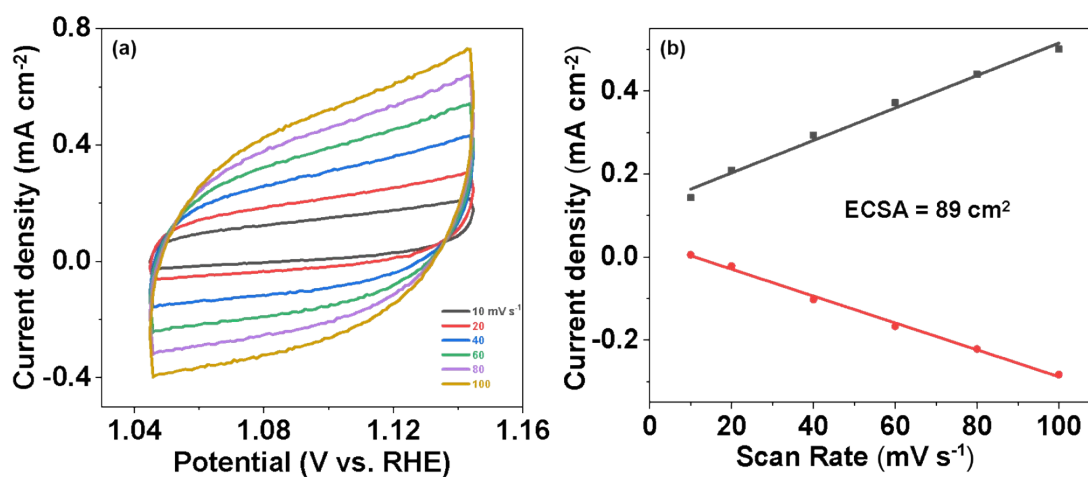


Fig. S8. (a) CVs of the Co_{0.6}-FeNiOOH/NF measured in a non-Faradaic region at different scan rate. (b) The cathodic and anodic currents measured as a function of the scan rate.

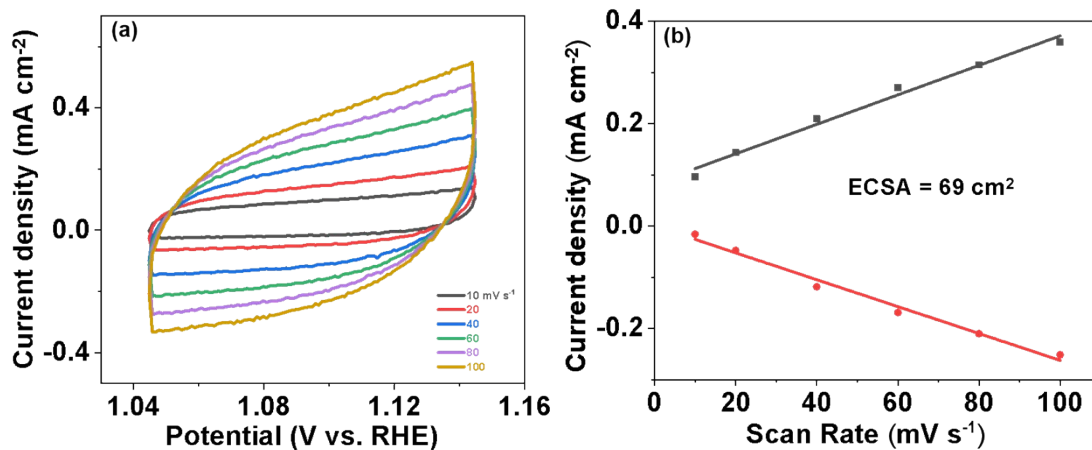


Fig. S9. (a) CVs of the FeNiOOH/NF measured in a non-Faradaic region at different scan rate. (b) The cathodic and anodic currents measured as a function of the scan rate.

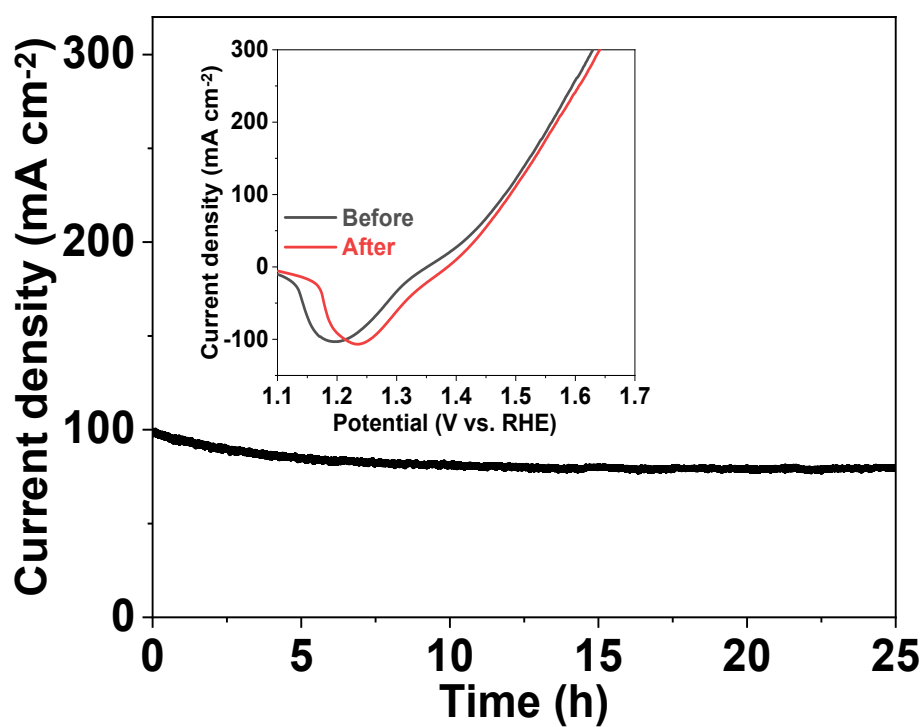


Fig. S10. Chronoamperometric curve of the Co_{0.2}-FeNiOOH/NF in 1 M KOH.

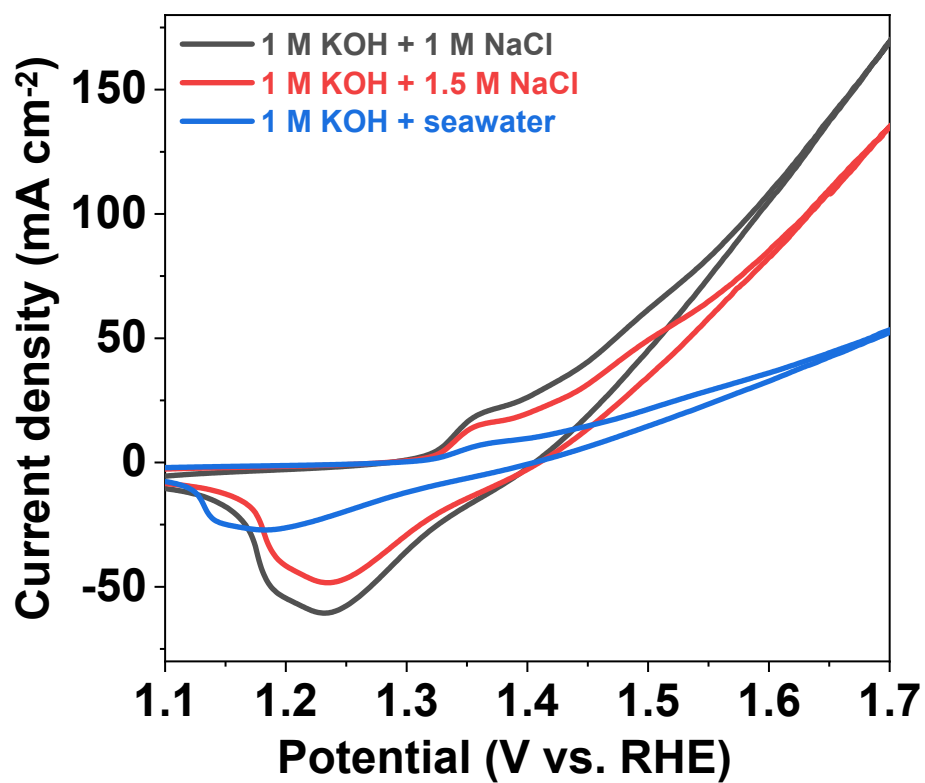


Fig. S11. CV curves of Co_{0.2}-FeNiOOH/NF in different electrolytes at 5 mV/s.

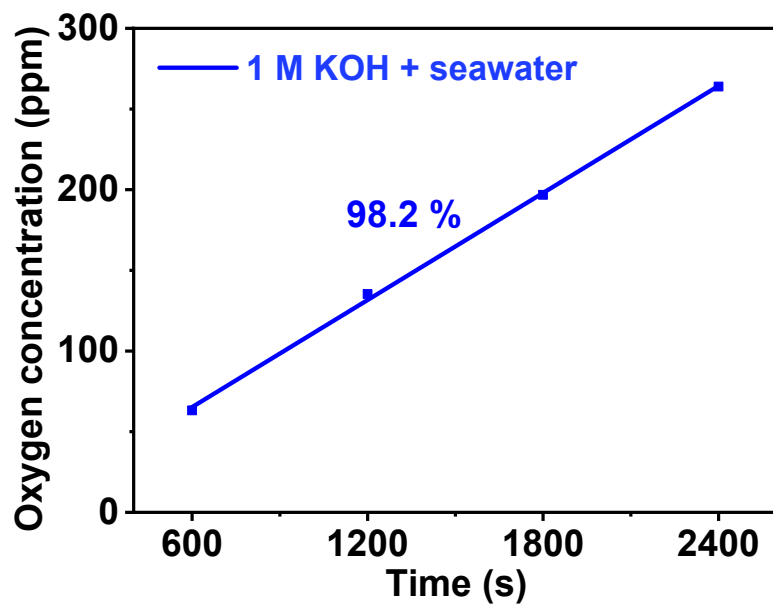


Fig. S12. Faraday efficiency of Co_{0.2}-FeNiOOH/NF in 1 M KOH + seawater at 10 mA cm⁻².

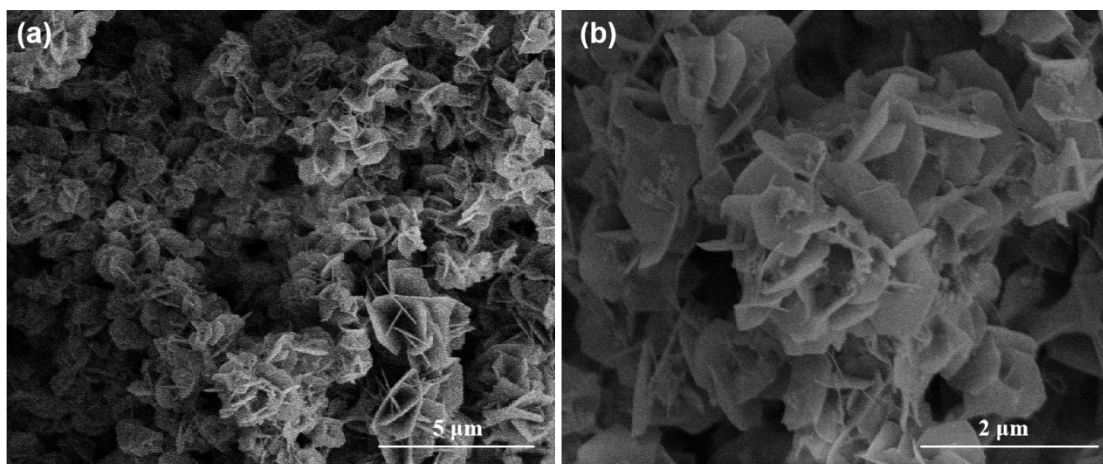


Fig. S13. (a, b) SEM images of $\text{Co}_{0.2}\text{-FeNiOOH/NF}$ after OER testing.

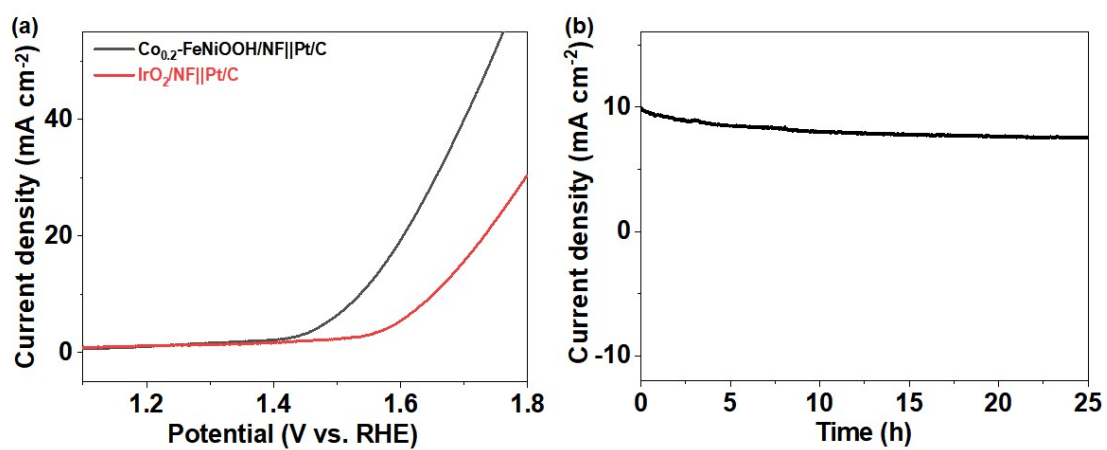


Fig. S14. (a) LSV curves of $\text{Co}_{0.2}\text{-FeNiOOH/NF||Pt/C}$ and $\text{IrO}_2\text{/NF||Pt/C}$ in overall water splitting. (b) Stability test.