

Supporting Information

A Highly Active Oxygen Evolution Electrocatalyst Derived From Co/Ni-Succinic Acid Framework Under Mild Conditions

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S1. Materials and General Methods

The organic ligand H₂L was used as received from commercial sources without further purification. Powder X-ray diffraction (PXRD) was carried out on a Bruker D8-Focus Bragg-Brentano X-ray Powder Diffractometer equipped with a Cu sealed tube ($\lambda = 1.54178 \text{ \AA}$) at 40 kV and 40 mA. Thermogravimetry analysis (TGA) was conducted on a TGA-50 thermogravimetric analyzer. The morphology was observed with a Sigma HD Thermal field emission scanning electron microscope (SEM). The elemental composition of the samples were characterized by energy-dispersive X-ray spectroscopy (EDS, Oxford instruments X-Max). The X-ray photoelectron spectroscopy (XPS) spectras were collected by Thermo ESCALAB 250XI spectrometer.

S2. Syntheses

Synthesis of Co-MOF.

First of all, in the glass vial of 10mL, 0.55g CoCl₂·6H₂O and 0.41g succinic acid ligand were added. Then, 5mL water containing 0.13g KOH was added. After ultrasonic dissolving, it was placed in a quiet place and volatilized without stirring at room temperature for about a week to form a red polyhedral crystal, which was named Co-MOF. Yield (based on ligand): 95%.

Synthesis of Co/Ni_x-MOF.

The synthesis process is similar to Co-MOF, except for the mixing different ratio of CoCl₂·6H₂O and NiCl₂·6H₂O. For example, Co_{0.9}Ni_{0.1}-MOF was synthesized via the utilization of 490mg CoCl₂·6H₂O and 30 mg NiCl₂·6H₂O; Co_{0.7}Ni_{0.3}-MOF was

constructed by utilizing 380mg $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ and 90 mg $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$. The similar XRD patterns illustrate the iso-structures of these materials. Yield (based on ligand): 91%.

Post-modification of different MOFs.

Because of the large crystal particles, which is not conducive to the post-modification process and the subsequent electrochemical test, the ball milling was carried out for 4 h. Then, the grinded samples was directly utilized for electrocatalytic measurements and post-modifications. 100 mg ball-milled $\text{Co}_{0.9}\text{Ni}_{0.1}$ -MOF samples were weighed in a vial, a certain amount of NaBH_4 was utilized at 75°C for 10 min with 0.1g NaBH_4 , 3h with 0.3g NaBH_4 , and 12h with 0.5g NaBH_4 . Yield (based on pristine MOF): 65%.

S3. Electrochemical measurements and products analysis.

Electrochemical testing was carried out in 1M KOH electrolyte using standard CHI760E electrochemical workstation with three electrodes. Glass carbon electrode (GCE) with diameter of 3mm was used as the working electrode, Pt network as the counter electrode, and Hg/HgO electrode as the reference electrode.

Preparation of catalyst dispersion solution. 5mg catalyst was dispersed in the mixed solution of 0.955mL ethanol and 0.005 ml5% Nafion, and ultrasonic treatment was conducted for 30min to form uniform dispersion solution. Then, The $10\mu\text{L}$ catalyst drops onto the polished GCE. To evaluate OER performance, a linear scan voltammetry curve (LSV) with a scan rate of 5mVs is obtained, from which the Tafel slope is obtained. Electrochemical impedance spectroscopy (EIS) in the range of

100000 ~0.1Hz was analyzed. When the current density was 10mA cm^{-2} , the stability of the catalyst was tested by chronopotentiometry method.

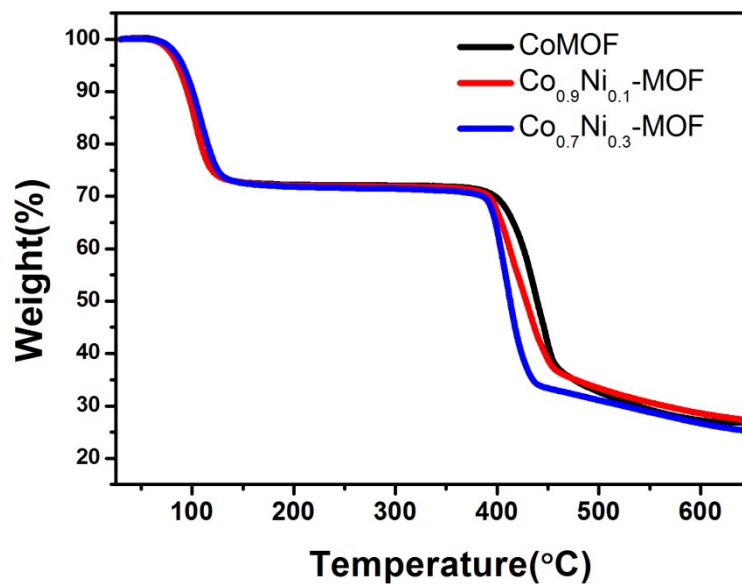


Figure S1. TGA curves for different samples

Table S1. ICP-OES values for different samples

Sample Name	Co (mg/mg Cat)	Ni (mg/mg Cat)	Co:Ni
Co-MOF	0.235	-	-
$\text{Co}_{0.9}\text{Ni}_{0.1}$ -MOF	0.264	0.0109	1:0.041
$\text{Co}_{0.7}\text{Ni}_{0.3}$ -MOF	0.232	0.03660	1:0.150

Table S2. The cost of the starting materials

materials	Price (dollar/1KG)
$\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	84.6
Succinic Acid	15.7
NaBH_4	342.6

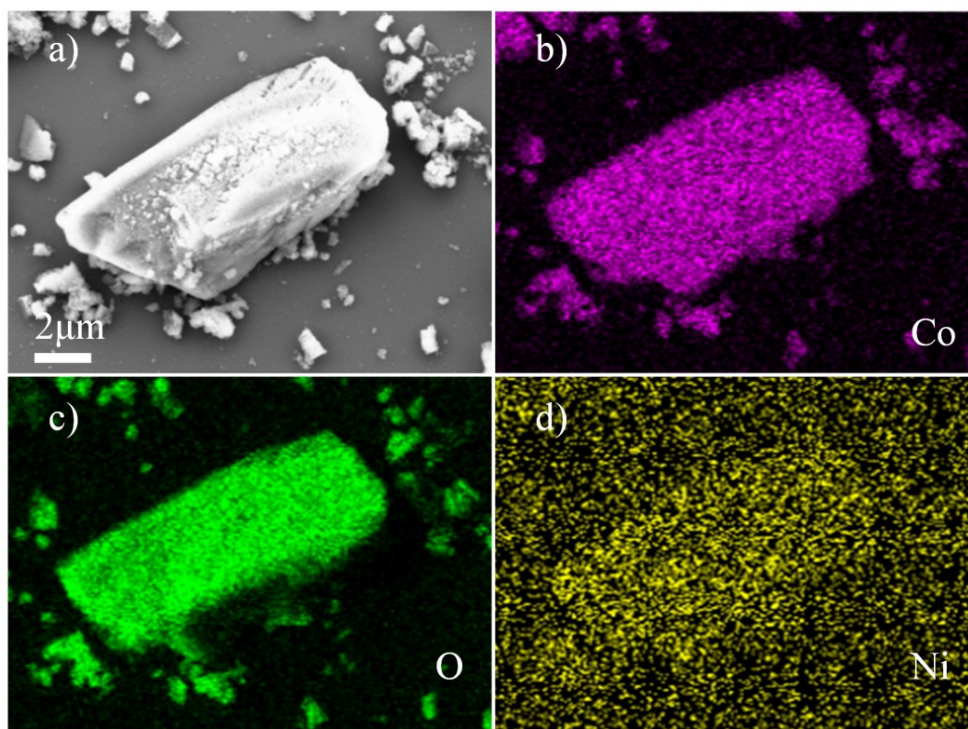


Figure S2. SEM and EDX images for $\text{Co}_{0.9}\text{Ni}_{0.1}\text{-MOF}$

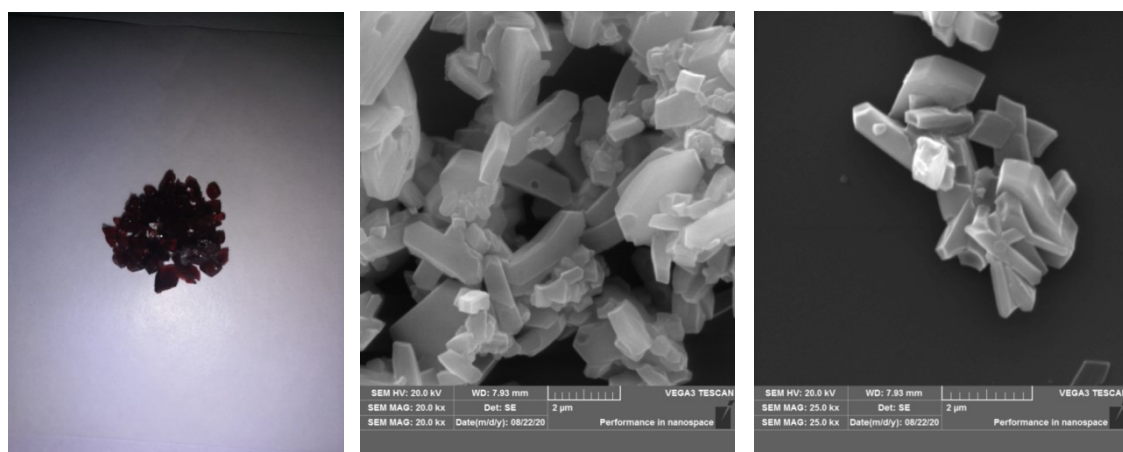


Figure S3. photo and SEM images for $\text{Co}_{0.9}\text{Ni}_{0.1}\text{-MOF}$

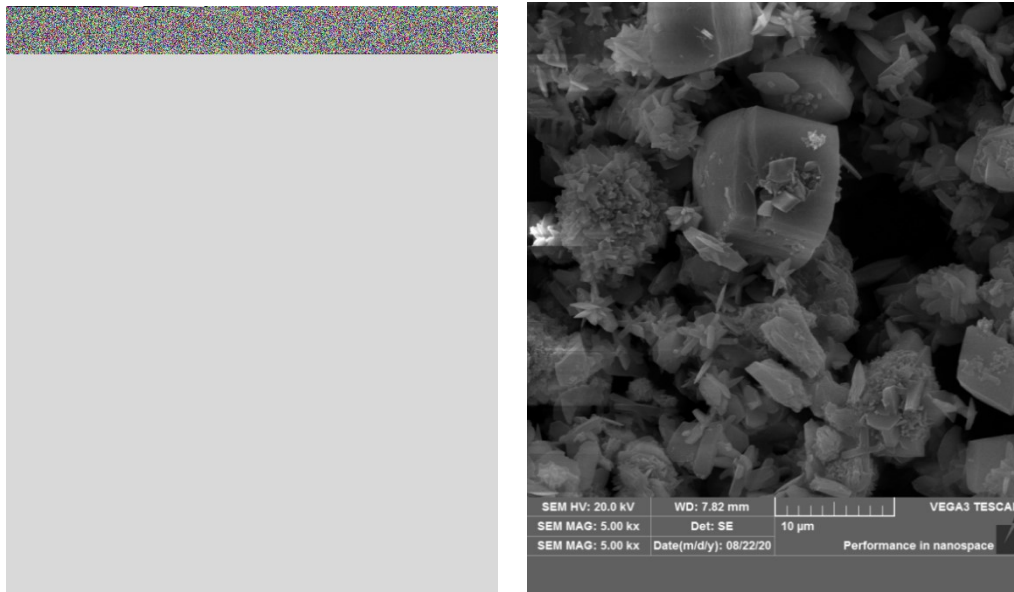


Figure S4. photo and SEM images for $\text{Co}_{0.7}\text{Ni}_{0.3}\text{-MOF}$

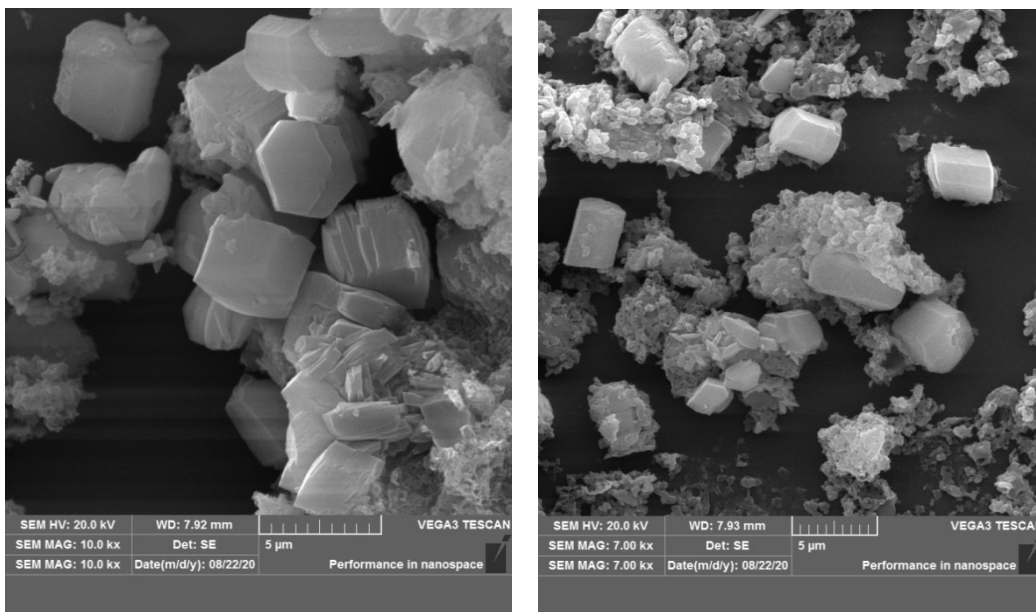


Figure S5. photo and SEM images for $\text{Co}_{0.9}\text{Ni}_{0.1}\text{-MOF-10min}$

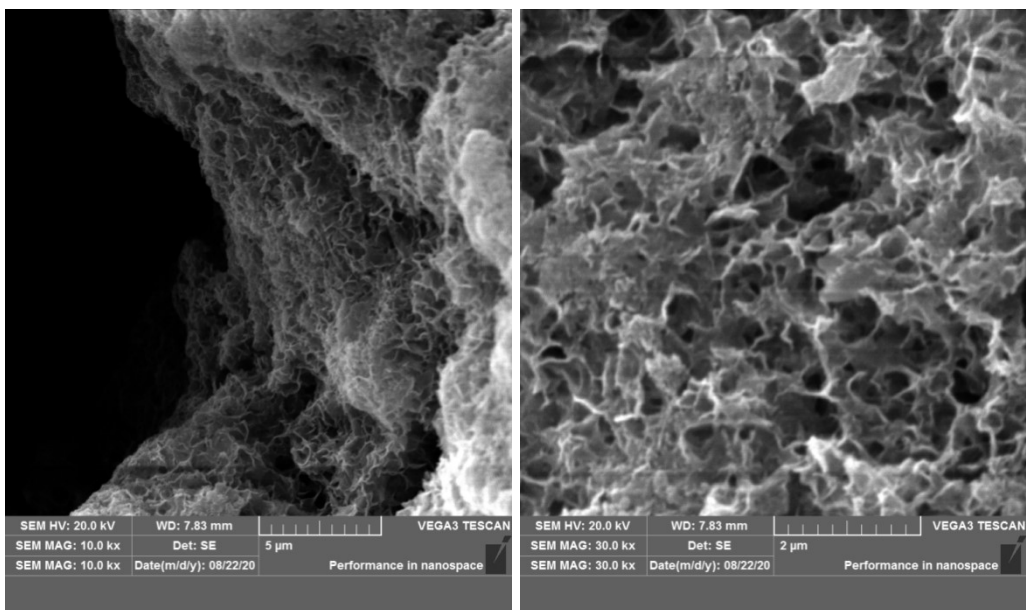


Figure S6. photo and SEM images for $\text{Co}_{0.9}\text{Ni}_{0.1}\text{-MOF-3h}$

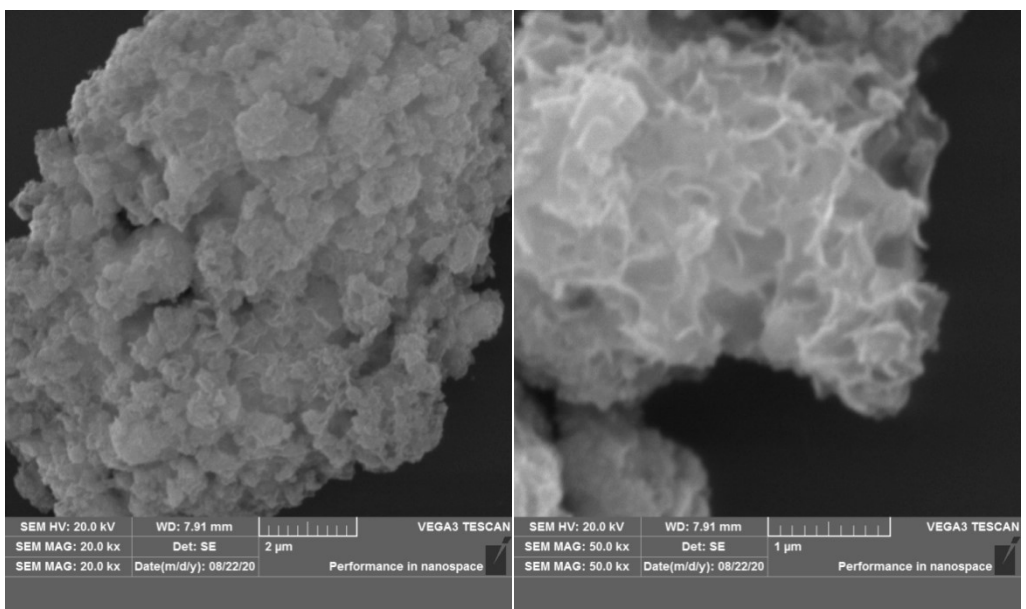
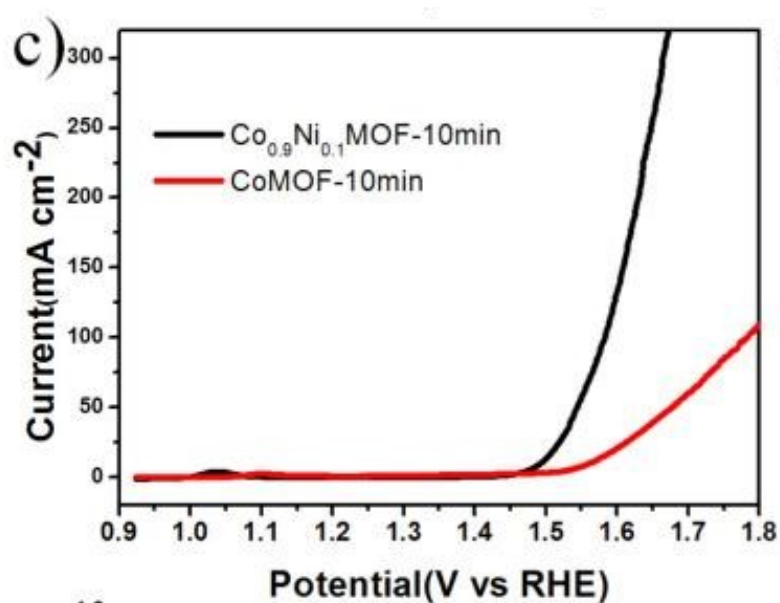
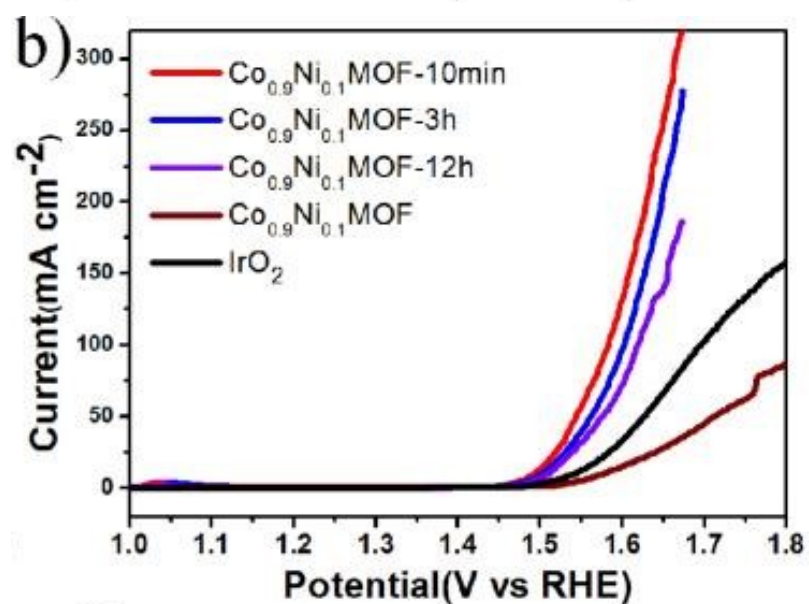
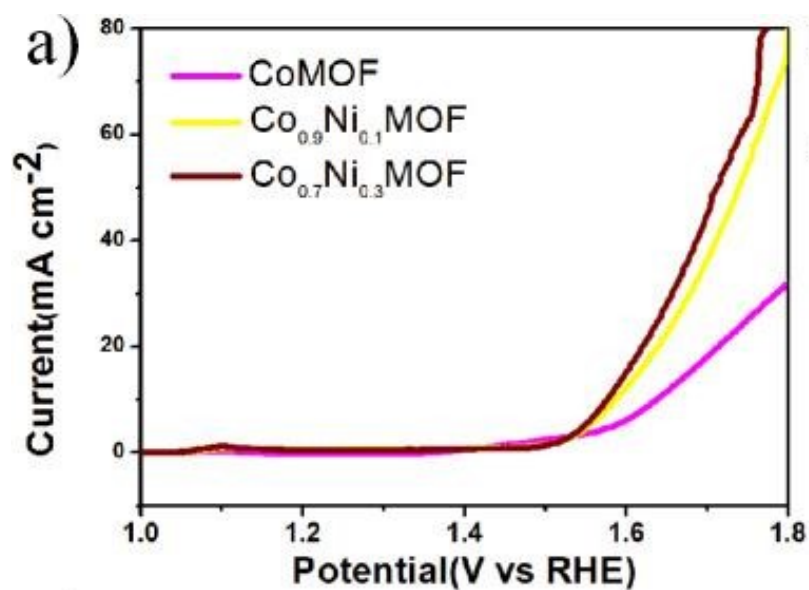


Figure S7. photo and SEM images for $\text{Co}_{0.9}\text{Ni}_{0.1}\text{-MOF-12h}$



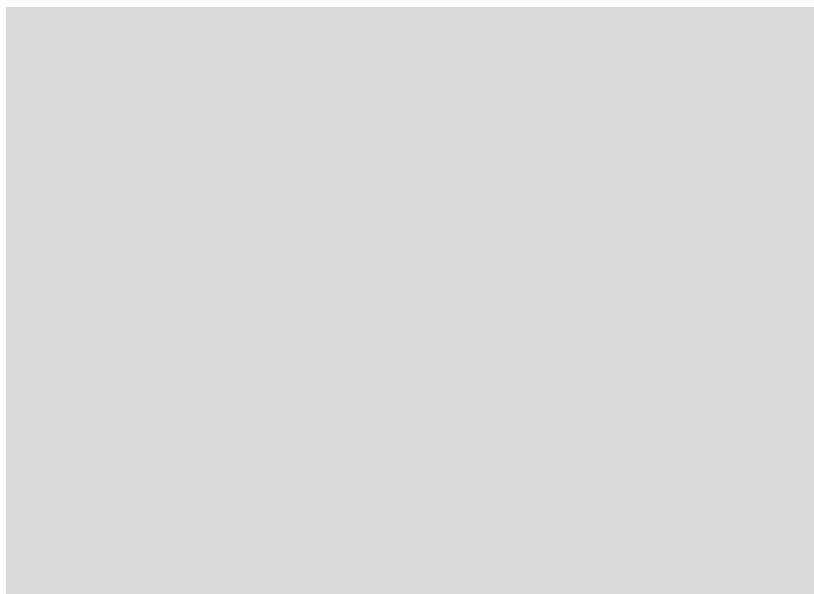


Figure S8. The enlarged OER results (a) and LSV plots (b, c) of different samples; EIS spectra (d) of different samples