

SUPPORTING INFORMATION

Quasi-Double-Star Nickel and Iron Active Sites for High-Efficient Carbon Dioxide Electroreduction

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1 **Materials and Methods**

2 **Materials:**

3 If not specified, all chemical reagents were purchased from Sigma-Aldrich. Zinc
4 nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), 2-Aminoterephthalic acid, iron chloride
5 hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), cobaltous nitrate hexahydrate ($\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), nickel
6 chloride hexahydrate ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$), N,N-dimethylformamide (DMF), sodium
7 thiocyanate (NaSCN), ethanol and sodium bicarbonate (NaHCO_3) were all of
8 analytical grade and used as received without further purification. Meanwhile, all
9 solutions were prepared with Milli-Q water ($\text{DI-H}_2\text{O}$, Ricca Chemical, ASTM Type I).
10 The carbon paper was purchased from Alfa Aesar. The Nafion (N-117 membrane,
11 0.18 mm thick) was also purchased from Alfa Aesar and kept in 0.5 M NaOH
12 solution.

13 **Characterization:**

14 The X-ray diffraction patterns (XRD) were obtained through a Bruker D4 X-ray
15 powder diffractometer using $\text{Cu K}\alpha$ radiation ($1.54184 \text{ \AA}$). Field emission scanning
16 electron microscopy (FESEM) images were collected on a FEI Magellan 400 L
17 scanning electron microscope. The transmission electron microscopy (TEM) and high
18 angle annular dark field scanning TEM (HAADF STEM) images were obtained in a
19 Tecnai F20 field emission gun microscope with a 0.19 nm point-to-point resolution at
20 200 kV equipped with an embedded Quantum Gatan Image Filter for EELS analyses.
21 Images have been analyzed by means of Gatan Digital Micrograph software. Parts of
22 HAADF-STEM images and elemental mapping (EDX) were obtained in a spherical

1 aberration-corrected (AC) transmission electron microscope FEI Themis Z and
2 operated at 200 kV. X-ray photoelectron spectroscopy (XPS) was performed on a
3 Phoibos 150 analyser (SPECS GmbH, Berlin, Germany) in ultra-high vacuum
4 conditions (base pressure 4×10^{-10} mbar) with a monochromatic aluminum K α X-ray
5 source (1486.74 eV). Binding energies (BE) were determined using the C 1s peak at
6 284.5 eV as a charge reference. Inductively coupled plasma-mass spectrometry
7 (ICP-MS) measurements were carried out to determine the concentration of Fe.
8 Brunauer-Emmett-Teller (BET) surface areas were measured using nitrogen
9 adsorption at 77 K. Raman spectra were obtained using Senterra. Fourier transformed
10 infrared (FTIR) spectroscopy data were recorded on an Alpha Bruker spectrometer.
11 Thermogravimetric Analysis was measured by Pyris 1 TGA, Perkin Elmer.

XAFS Measurements:

The X-ray absorption fine structure spectra (Fe K-edge) were collected at 1W1B station in Beijing Synchrotron Radiation Facility (BSRF). The storage rings of BSRF were operated at 2.5 GeV with an average current of 250 mA. Using Si(111) double-crystal monochromator, the data collection were carried out in transmission/fluorescence mode using ionization chamber. All spectra were collected in ambient conditions.

XAFS Analysis and Results:

The acquired EXAFS data were processed according to the standard procedures using the ATHENA module implemented in the IFEFFIT software packages. The k^3 -weighted EXAFS spectra were obtained by subtracting the post-edge background from the overall absorption and then normalizing with respect to the edge-jump step. Subsequently, k^3 -weighted $\chi(k)$ data of Fe K-edge were Fourier transformed to real (R) space using a hanning windows ($dk=1.0 \text{ \AA}^{-1}$) to separate the EXAFS contributions from different coordination shells. To obtain the quantitative structural parameters around central atoms, least-squares curve parameter fitting was performed using the ARTEMIS module of IFEFFIT software packages.¹⁻³

1 **Ink Preparation:**

2 2 mg synthesized different samples and 50 μ l 5 wt% Nafion solutions were dissolved
3 in ethanol (1 ml) and ultrasonicated for 1 h to form evenly suspension for the further
4 electrochemical experiments. To prepare the working electrode, 500 μ L above
5 as-prepared inks were dropped onto the two sides of the carbon paper electrode with
6 $1 \times 1 \text{ cm}^2$ and then dried at room temperature for a few minutes, giving a catalyst
7 loading mass of $\sim 1 \text{ mg cm}^{-2}$.

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1 **Electrochemical Measurement:**

2 The electrocatalytic performance of different catalysts was measured at room
3 temperature by using a gas-tight H-cell with two-compartments separated by a cation
4 exchange membrane (Nafion N-117 membrane) with a continuously Ar or CO₂ gas
5 injection. Each compartment contained 70 ml electrolyte (0.5 M NaHCO₃ made from
6 de-ionized water). In a typical experiment, a standard three electrode setup in 0.5 M
7 NaHCO₃ solution was assembled: an Ag/AgCl electrode as a reference electrode, a Pt
8 plate as a counter electrode and a carbon paper coated with the different samples as a
9 working electrode (surface area = 1 cm²). The potentials were measured versus
10 Ag/AgCl and converted to the reversible hydrogen electrode (RHE) according to the
11 following equation: $E_{\text{RHE}} = E^0_{\text{Ag/AgCl}} + E_{\text{Ag/AgCl}} + 0.059 \times \text{pH}$, pH=7.⁴ All
12 electrochemical results were showed without iR-compensation by using a
13 computer-controlled BioLogic VMP3 electrochemical workstation. Meanwhile, the
14 linear sweep voltammetry (LSV) was performed at a scan rate of 20 mV s⁻¹ from 0 V
15 to -1.5 V vs. Ag/AgCl in Ar-saturated 0.5 M NaHCO₃ (pH=8.5) and CO₂-saturated
16 0.5 M NaHCO₃ (pH=7) as supporting electrolyte. The cyclic voltammetry (CV) was
17 performed at a scan rate of 20 mV s⁻¹. Moreover, electrochemical impedance
18 spectroscopy (EIS) of different samples was carried out in a frequency range from 100
19 kHz to 100 mHz.

20 Before the electrochemical CO₂ reduction experiments, an average rate of 20 ml
21 min⁻¹ Ar was injected into cathodic electrolyte in order to form an Ar-saturated
22 solution. During electrochemical CO₂ reduction experiments, the CO₂ gas was

1 delivered at an average rate of 20 ml min⁻¹ at room temperature and ambient pressure,
2 measured downstream by a volumetric digital flowmeter. The gas phase composition
3 was analyzed by gas chromatography (GC) during potentiostatic measurements every
4 20 min. The calibration of peak area vs. gas concentration was used for the molar
5 quantification of each gaseous effluent. The Faradaic efficiency was calculated by
6 determining the number of coulombs needed for each product and then dividing by
7 the total charge passed during the time of the GC sampling according to the flow rate.
8 Liquid products were analyzed afterwards by quantitative ¹H-NMR using water as the
9 deuterated solvent.

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Calculation Method:

Details concerning the Faradaic Efficiency (FE) calculations are shown below.⁴⁻⁷

The partial current density for a given gas product was calculated as below:

$$j_i = x_i \times V \times \frac{n_i F P_0}{RT} \times (\text{electrode area})^{-1}$$

Where x_i is the volume fraction of a certain product determined by online GC referenced to calibration curves from three standard gas samples, V is the flow rate, n_i is the number of electrons involved, $P_0 = 101.3$ kPa, F is the Faraday constant, and R is the gas constant. The corresponding FE at each potential is calculated by

$$FE = \frac{j_i}{j} \times 100\%$$

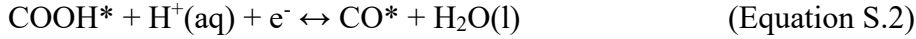
TOF for CO production was calculated as follow equation:

$$TOF = \frac{(I_{\text{product}}/nF)}{(m_{\text{cat}} \times \omega/M_{\text{metal}})} \times 3600$$

Where I_{product} is partial current for certain product, A , $n=2$, the number of electrons transferred for CO production, F is the Faraday constant, 96485 C mol⁻¹, m_{cat} is the mass of catalyst on the carbon paper, g. ω is the metal loading in the catalyst, and M_{metal} is atomic mass of Ni (58.69 g mol⁻¹) for Ni-N-C, atomic mass of Fe (55.85 g mol⁻¹) for Fe-N-C, and atomic mass of 56.99 g mol⁻¹ for Ni₇/Fe₃-N-C (based on the ratio of Ni and Fe).

DFT Calculations:

The spin-polarized DFT calculations with projector augmented wave (PAW) method⁸⁻¹¹ were performed using the Vienna ab initio Simulation Package (VASP) code.¹² The Bayesian error estimation functional with van der Waals correlation (BEEF-vdW) was employed to set the plane wave basis.¹³ The convergence criteria were 0.05 eV/Å in force and 1×10^{-4} eV in energy and the plane wave cutoff was 500 eV. The Monkhorst–Pack mesh k-point grids were $2 \times 2 \times 1$ for all models. All the vacuum thicknesses were higher than 15 Å. With the BEEF-vdW function, the energy of the gas phase molecules gave a systematic correction by +0.41 and +0.09 eV for gaseous CO₂ and H₂, respectively.¹⁴⁻¹⁶ For the electroreduction of CO₂ to CO, the following elementary steps were considered:



where (g), (aq) represent the gaseous phase and aqueous phase, respectively. The *, COOH* and CO* represent free site, adsorption state of COOH and CO, respectively.

The reaction free energies of each step was calculated by the following formula:

$$G = E_{\text{DFT}} + E_{\text{ZPE}} + \int C_p dT - TS + E_{\text{sol}} \quad (\text{Equation S.4})$$

the E_{DFT} is the DFT calculated energy, E_{ZPE} is the zero-point energy, C_p is the constant pressure heat capacity, T is temperature, S is the entropy. The E_{sol} is solvation correction, which is -0.1 eV for CO* and -0.25 eV COOH*.¹⁷ The temperature for the

1 reaction is considered as 298.15 K here.

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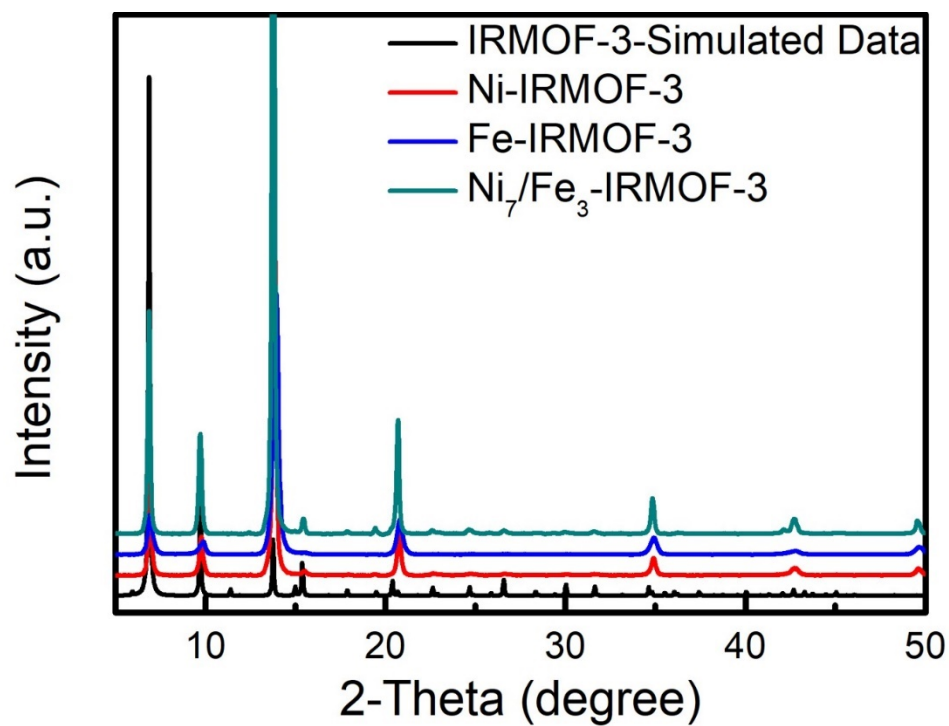


Fig. S1. XRD patterns of Ni-IRMOF-3, Fe-IRMOF-3 and Ni₇/Fe₃-IRMOF-3.

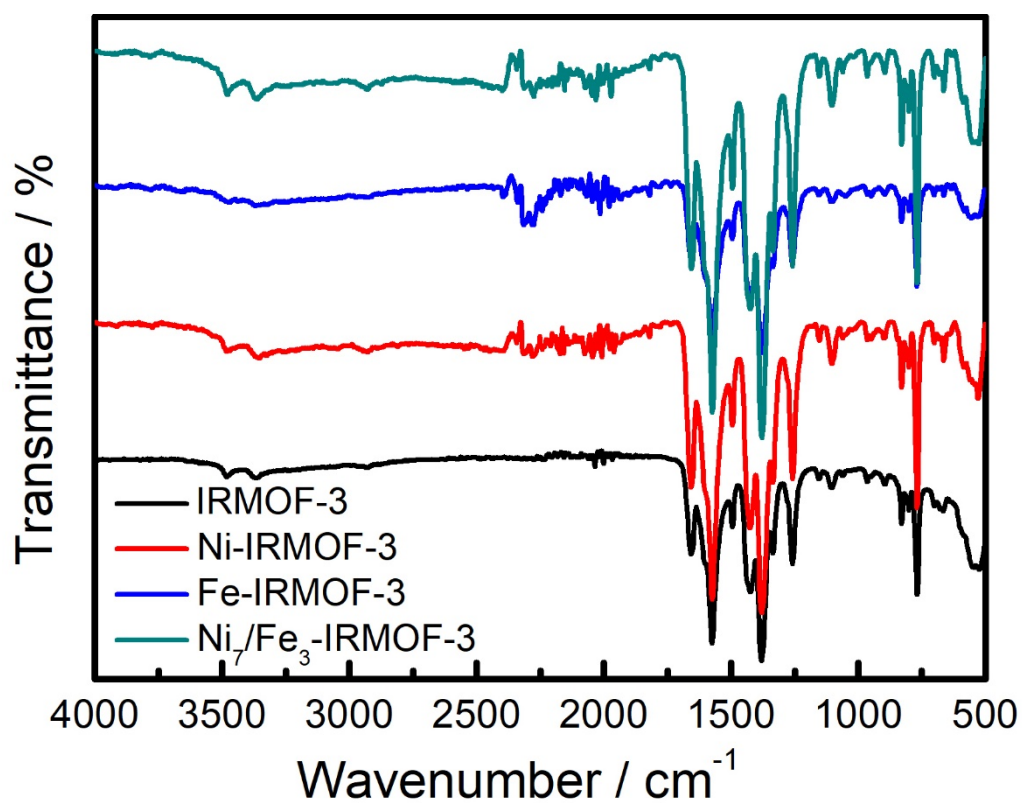


Fig. S2. FTIR spectrum of IRMOF-3, Ni-IRMOF-3, Fe-IRMOF-3 and Ni₇/Fe₃-IRMOF-3.

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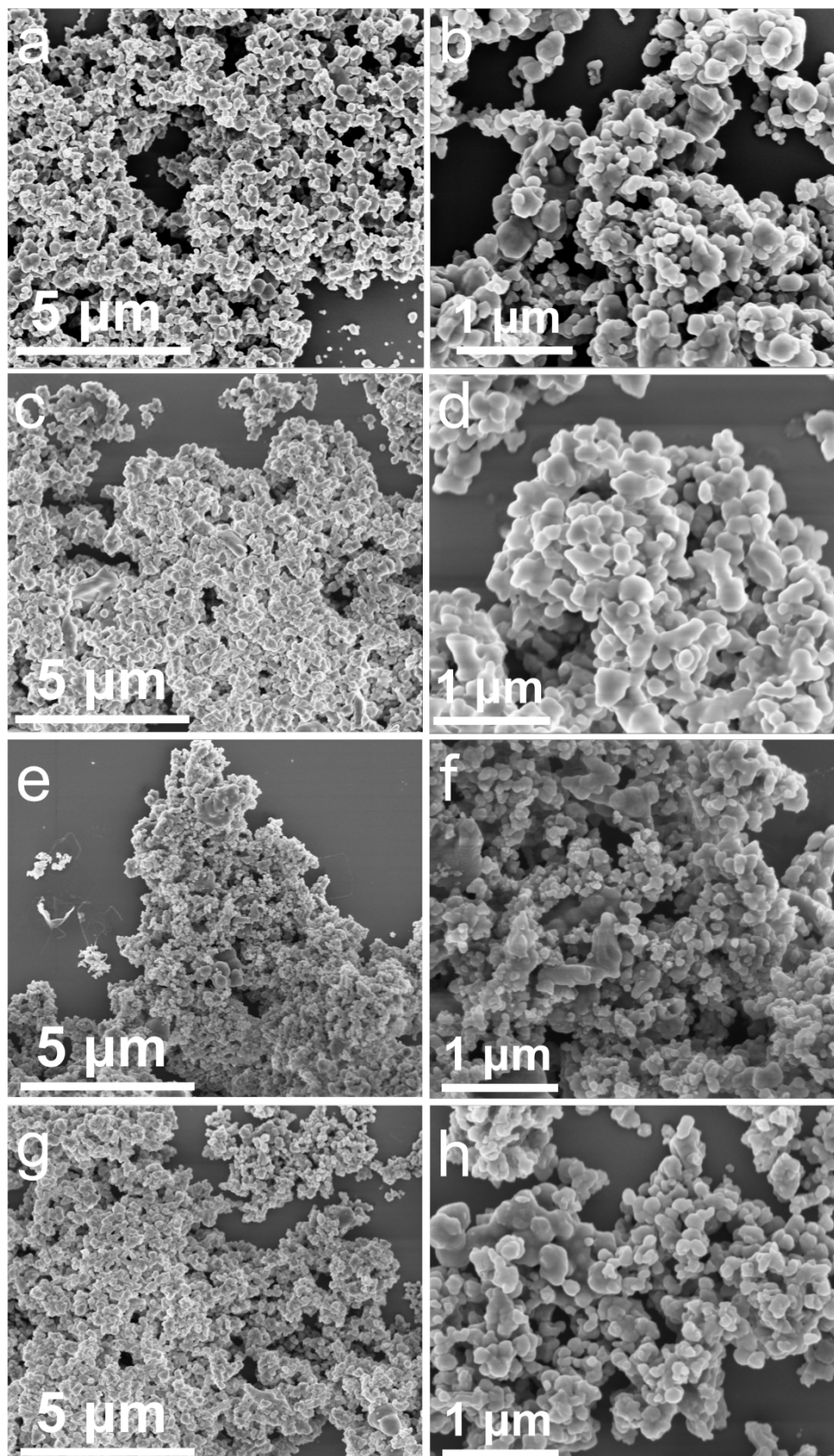


Fig. S3. SEM images of (a and b) IRMOF-3, (c and d) Ni-IRMOF-3, (e and f) Fe-IRMOF-3, (g and h) Ni₇/Fe₃-IRMOF-3.

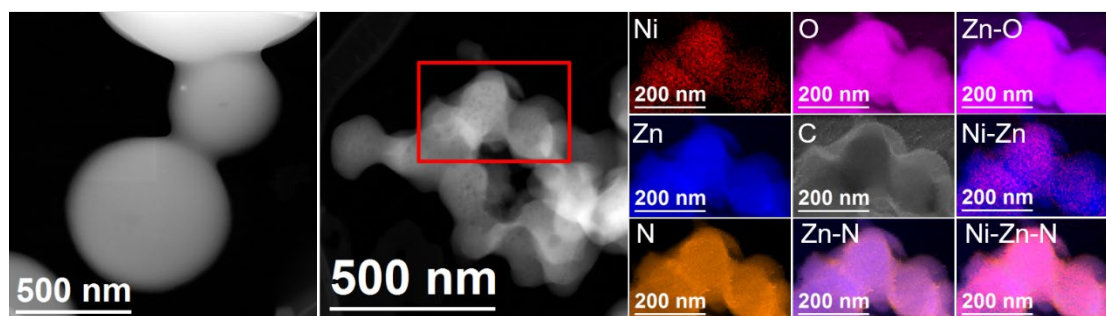


Fig. S4. HAADF STEM image of Ni-IRMOF-3 and representative EELS chemical composition maps obtained from the red squared area of the STEM micrograph. Individual Ni $L_{2,3}$ -edges at 855 eV (red), Zn $L_{2,3}$ -edges at 1020 eV (blue), N K-edges at 401 eV (orange), O K-edges at 532 eV (pink) and C K-edges at 285 eV (grey) as well as composites of Zn-N, Zn-O, Ni-Zn and Ni-Zn-N.

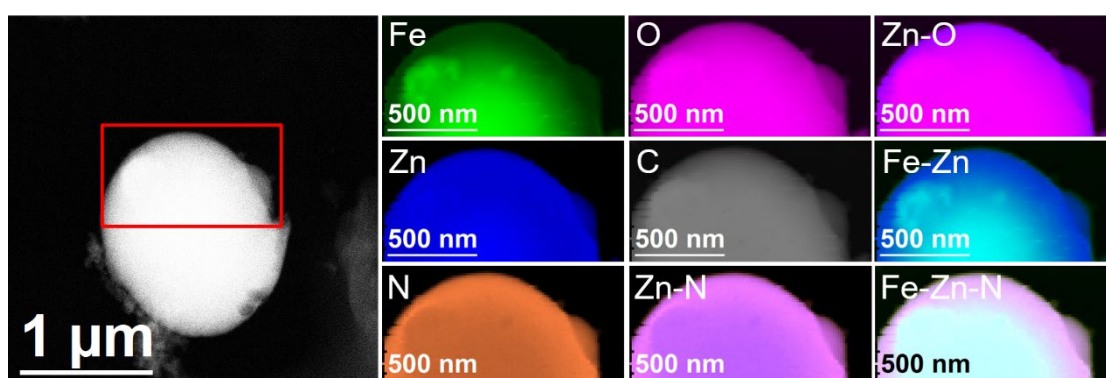


Fig. S5. HAADF STEM image of Fe-IRMOF-3 and representative EELS chemical composition maps obtained from the red squared area of the STEM micrograph. Individual Fe $L_{2,3}$ -edges at 708 eV (green), Zn $L_{2,3}$ -edges at 1020 eV (blue), N K-edges at 401 eV (orange), O K-edges at 532 eV (pink) and C K-edges at 285 eV (grey) as well as composites of Zn-N, Zn-O, Fe-Zn and Fe-Zn-N.

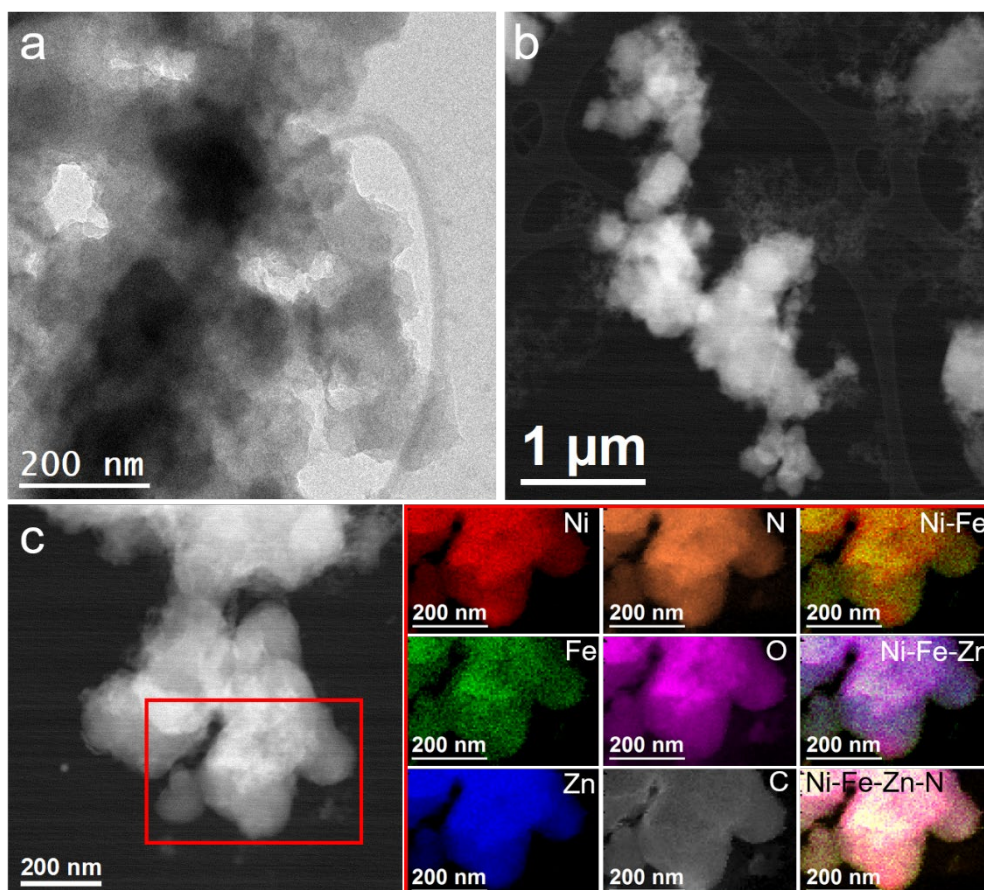


Fig. S6. (a) BF TEM, (b, c) HAADF STEM image of $\text{Ni}_7/\text{Fe}_3\text{-IRMOF-3}$ and representative EELS chemical composition maps obtained from the red squared area of the STEM micrograph. Individual Ni $L_{2,3}$ -edges at 855 eV (red), Fe $L_{2,3}$ -edges at 708 eV (green), Zn $L_{2,3}$ -edges at 1020 eV (blue), N K-edges at 401 eV (orange), O K-edges at 532 eV (pink) and C K-edges at 285 eV (grey) as well as composites of Ni-Fe, Ni-Fe-Zn and Ni-Fe-Zn-N.

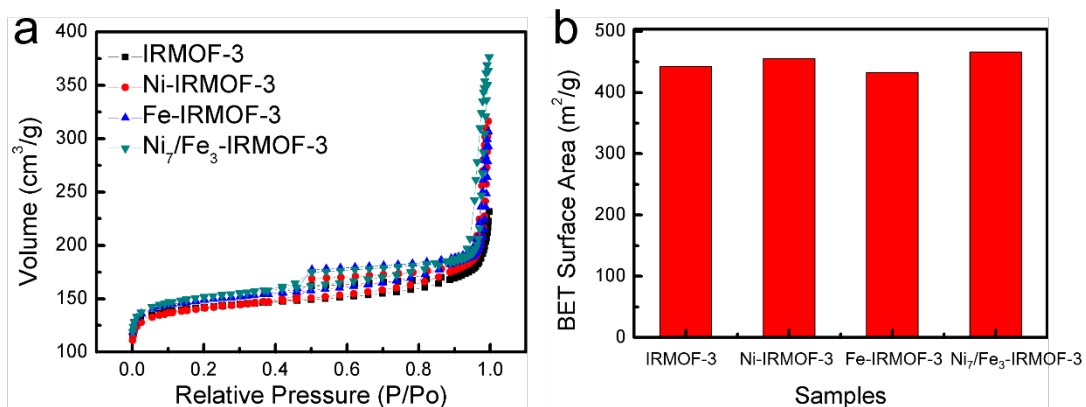


Fig. S7. (a) N₂ adsorption and desorption isotherm and (b) BET surface areas for IRMOF-3, Ni-IRMOF-3, Fe-IRMOF-3 and Ni₇/Fe₃-IRMOF-3.

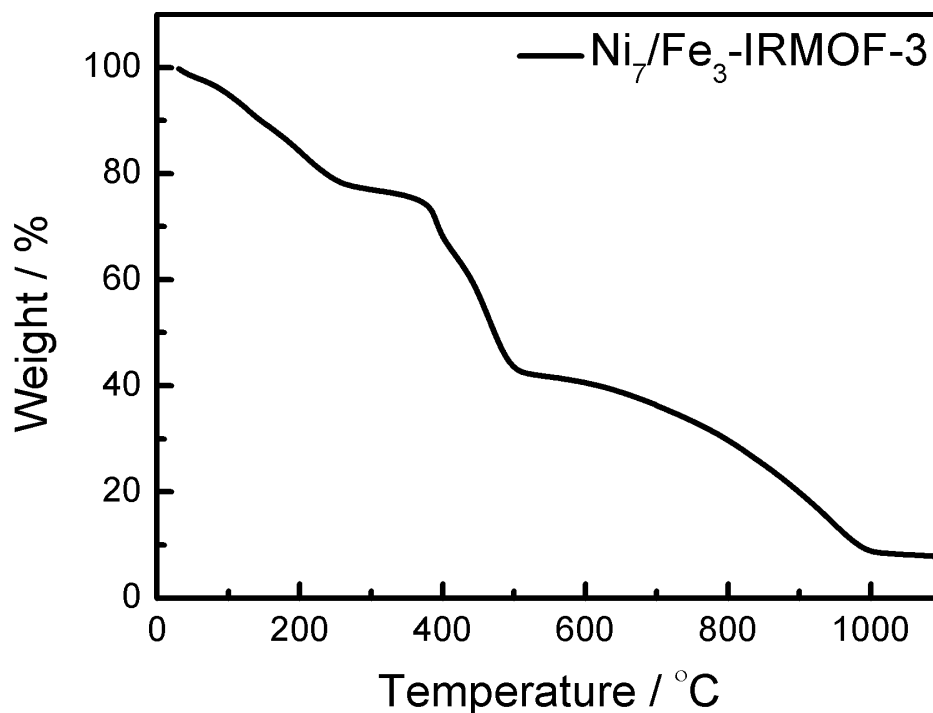


Fig. S8. TGA patterns of $\text{Ni}_7/\text{Fe}_3\text{-IRMOF-3}$.

Table S1. Ni and Fe loading ratios of different samples.

Samples	Feeding mass (Ni)	Final product ratio (Ni)	Feeding mass (Fe)	Final product ratio (Fe)
Ni-N-C	2.41×10^{-3} mmol	0.38 %	/	/
Fe-N-C	/	/	2.59×10^{-3} mmol	0.7 %
$\text{Ni}_7/\text{Fe}_3\text{-N-C}$	1.69×10^{-3} mmol	0.30 %	7.77×10^{-4} mmol	0.45 %

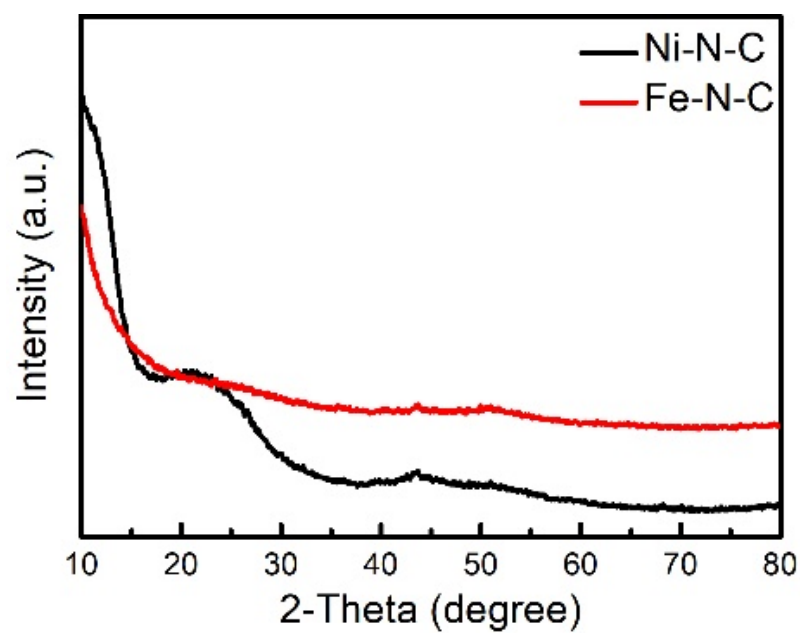


Fig. S9. XRD patterns of Ni-N-C and Fe-N-C.

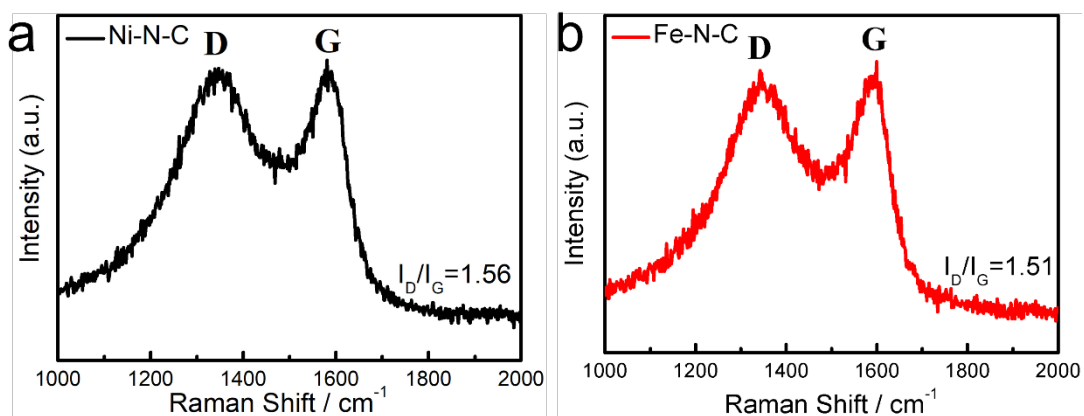


Fig. S10. Raman spectra of (a) Ni-N-C and (b) Fe-N-C.

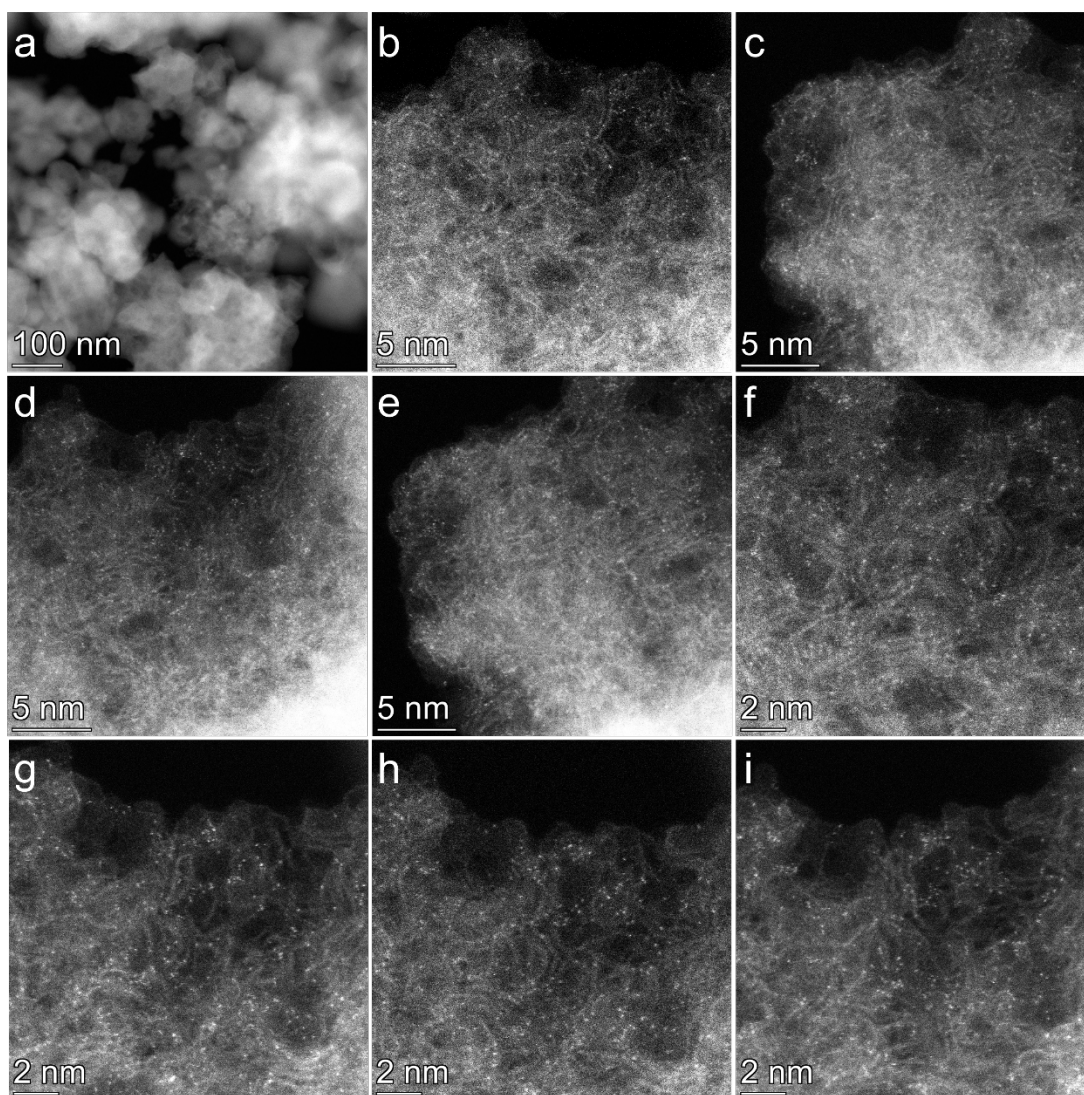


Fig. S11. (a) Low magnification and (b-i) high magnification aberration-corrected HAADF STEM images of $\text{Ni}_7/\text{Fe}_3\text{-N-C}$ sample.

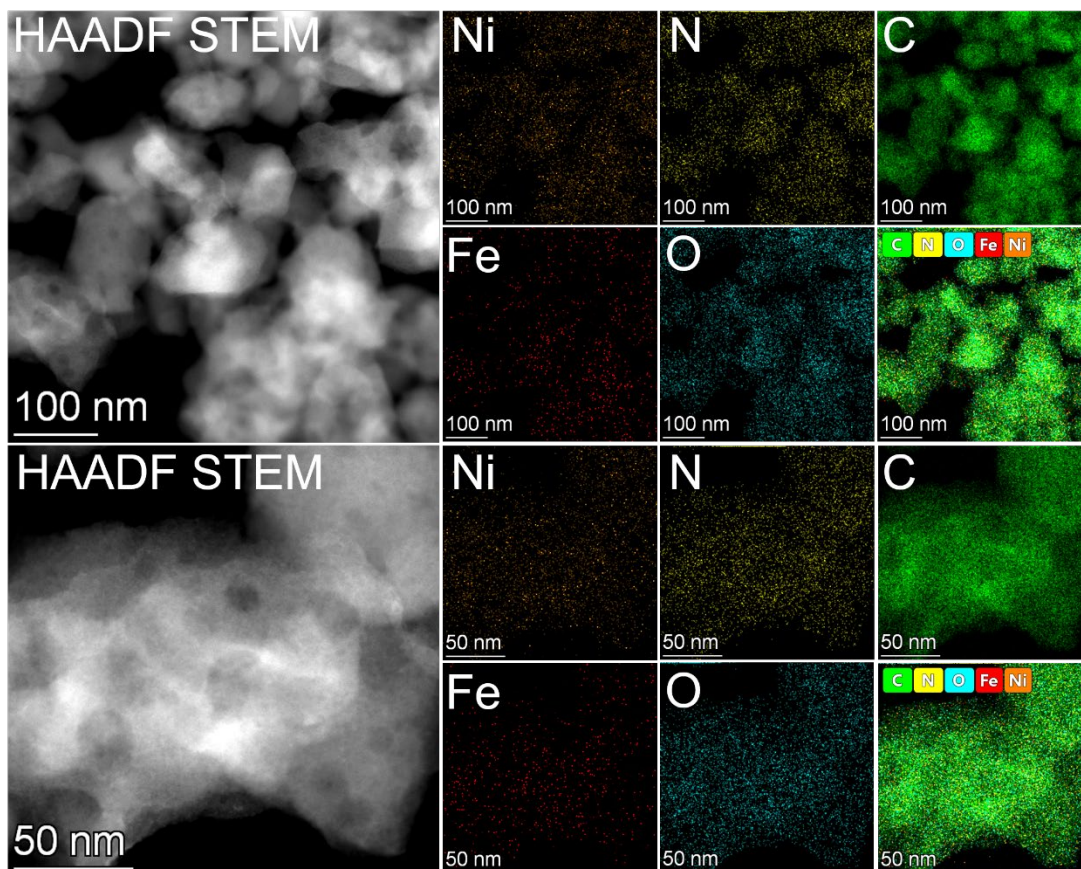


Fig. S12. HAADF STEM image of Ni₇/Fe₃-N-C and representative EDX chemical composition maps.

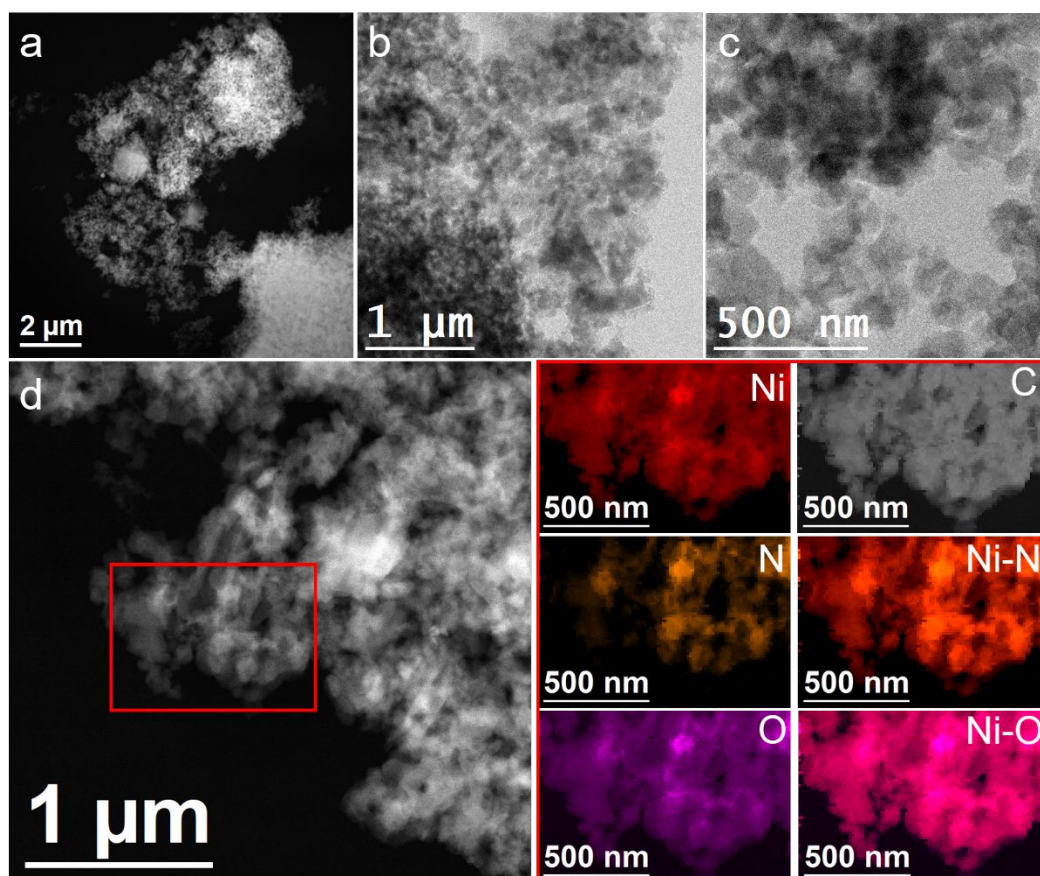


Fig. S13. (a) HAADF STEM, (b, c) BF TEM images of Ni-N-C, (d) HAADF STEM image and representative EELS chemical composition maps obtained from the red squared area of the STEM micrograph. Individual Ni L_{2,3}-edges at 855 eV (red), N K-edges at 401 eV (orange), O K-edges at 532 eV (pink) and C K-edges at 285 eV (grey) as well as composites of Ni-N and Ni-O.

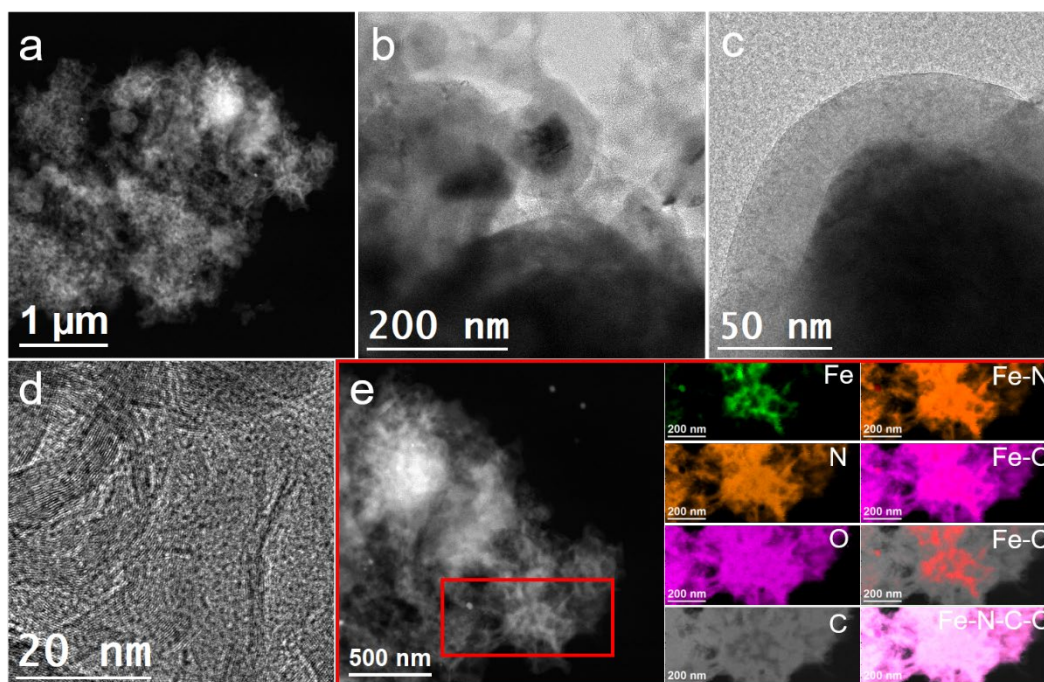


Fig. S14. (a) HAADF STEM, (b, c) BF TEM, (d) HRTEM images of Fe-N-C as well as (e) HAADF STEM image and representative EELS chemical composition maps obtained from the red squared area of the STEM micrograph. Individual Fe $L_{2,3}$ -edges at 708 eV (green), N K-edges at 401 eV (orange), O K-edges at 532 eV (pink) and C K-edges at 285 eV (grey) as well as composites of Fe-N, Fe-O, Fe-C and Fe-N-C-O.

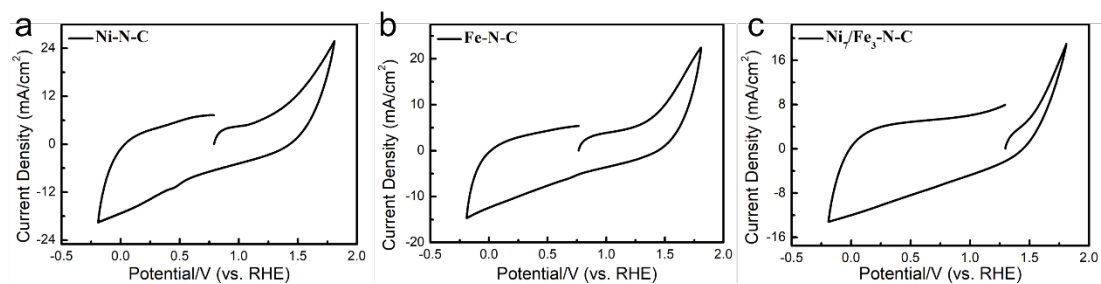


Fig. S15. CV curves of (a) Ni-N-C, (b) Fe-N-C and (c) Ni₇/Fe₃-N-C in 0.5 M NaHCO₃ electrolyte.

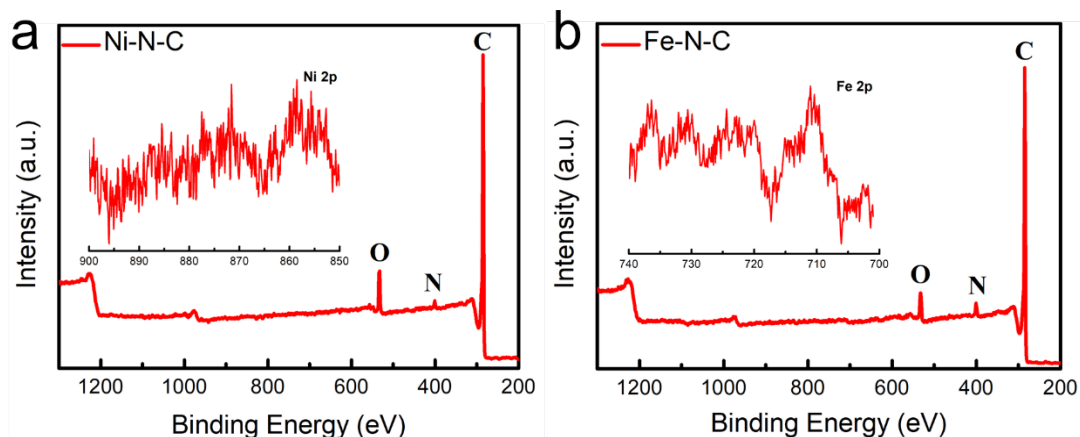


Fig. S16. XPS survey spectra of (a) Ni-N-C (inset) Ni 2p and (b) Fe-N-C (inset) Fe 2p.

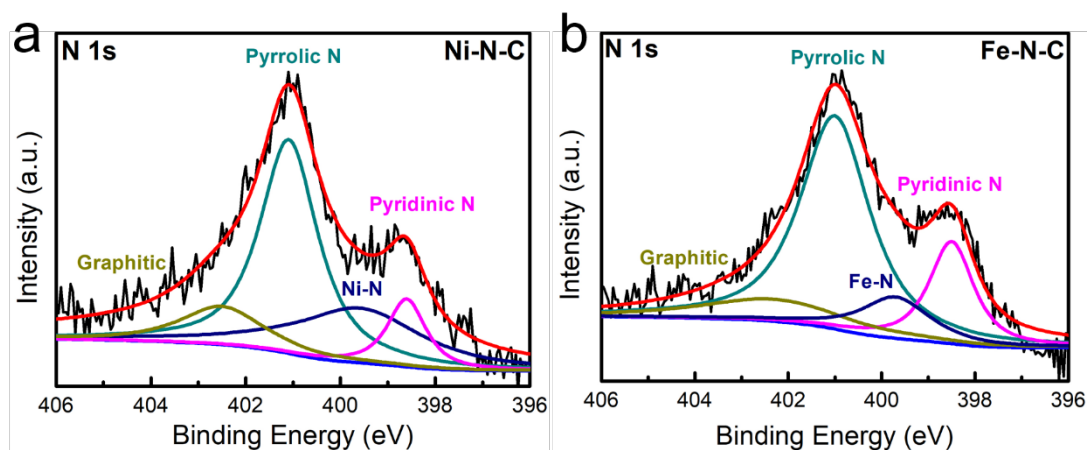


Fig. S17. High-resolution XPS N 1s of (a) Ni-N-C and (b) Fe-N-C.

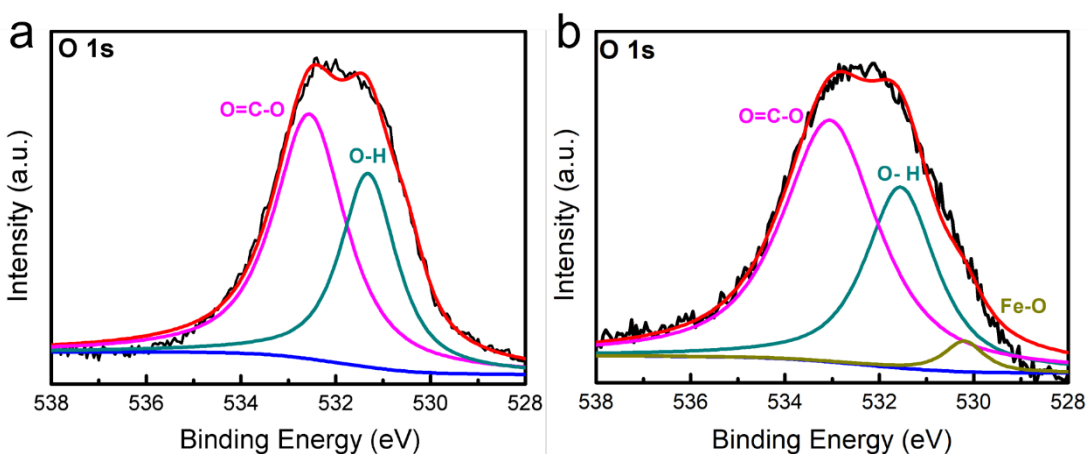


Fig. S18. High-resolution XPS O 1s of (a) Ni-N-C and (b) Fe-N-C.

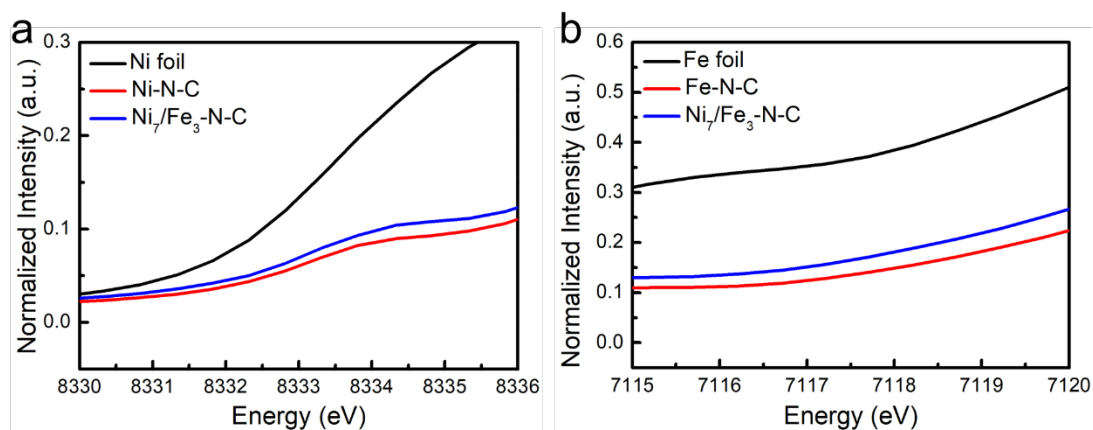


Fig. S19. The enlarged pre-edge region of (a) Ni K-edge XANES spectra and (b) Fe K-edge XANES spectra.

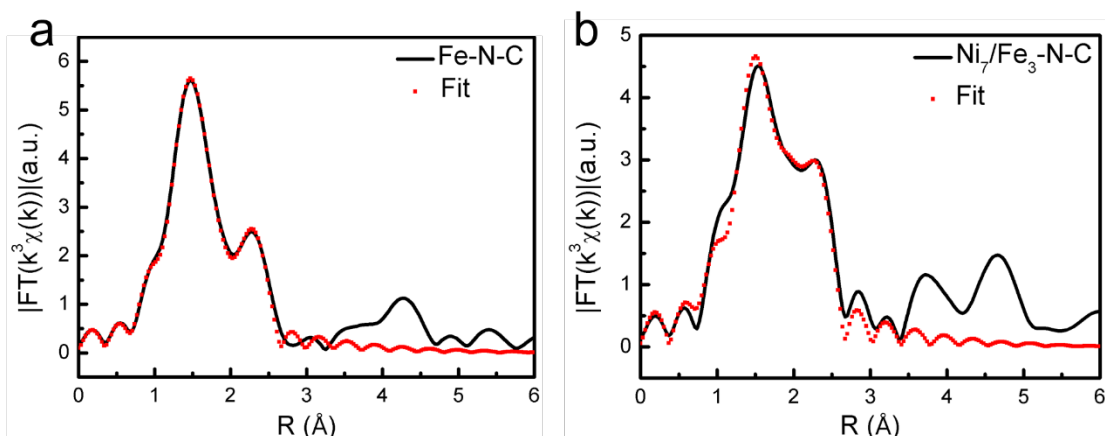


Figure S20. The corresponding Fe K-edge EXAFS fitting parameters for (a) Fe-N-C and (b) Ni₇/Fe₃-N-C samples.

Table S2. EXAFS fitting parameters at the Ni K-edge for various samples

Sample	Shell	N ^a	R (Å) ^b	σ ² (Å ² ·10 ⁻³) ^c	ΔE ₀ (eV) _d	R factor (%)
Ni ₇ /Fe ₃ -N-C	Ni-N	4.5	1.84	9.2	-5.1	0.3
Ni-N-C	Ni-N	3.9	1.87	6.5	-5.9	0.7
	Ni-C	3.2	2.12	6.3	-1.6	

^aN: coordination numbers; ^bR: bond distance; ^cσ²: Debye-Waller factors; ^dΔE₀: the inner potential correction. *R* factor: goodness of fit. *S*₀₂ were set as 0.85/0.80 for Ni-N/Ni-C, which was obtained from the experimental EXAFS fit of reference FePc by fixing CN as the known crystallographic value and was fixed to all the samples.

Table S3. EXAFS fitting parameters at the Fe K-edge for various samples

Sample	Shell	N ^a	R (Å) ^b	σ ² (Å ² ·10 ⁻³) ^c	ΔE ₀ (eV) _d	R factor (%)
Ni ₇ /Fe ₃ -N-C	Fe-N(O)	4.8	1.98	8.9	-2.4	1.1
Fe-N-C	Fe-N	6.1	1.99	10.1	-3.3	0.3
	Fe-Fe	1.0	2.52	7.2	-1.5	

^aN: coordination numbers; ^bR: bond distance; ^cσ²: Debye-Waller factors; ^dΔE₀: the inner potential correction. *R* factor: goodness of fit. *S*₀₂ were set as 0.85/0.90 for Fe-N/Fe-Fe, which was obtained from the experimental EXAFS fit of reference FePc/Fe foil by fixing CN as the known crystallographic value and was fixed to all the samples.

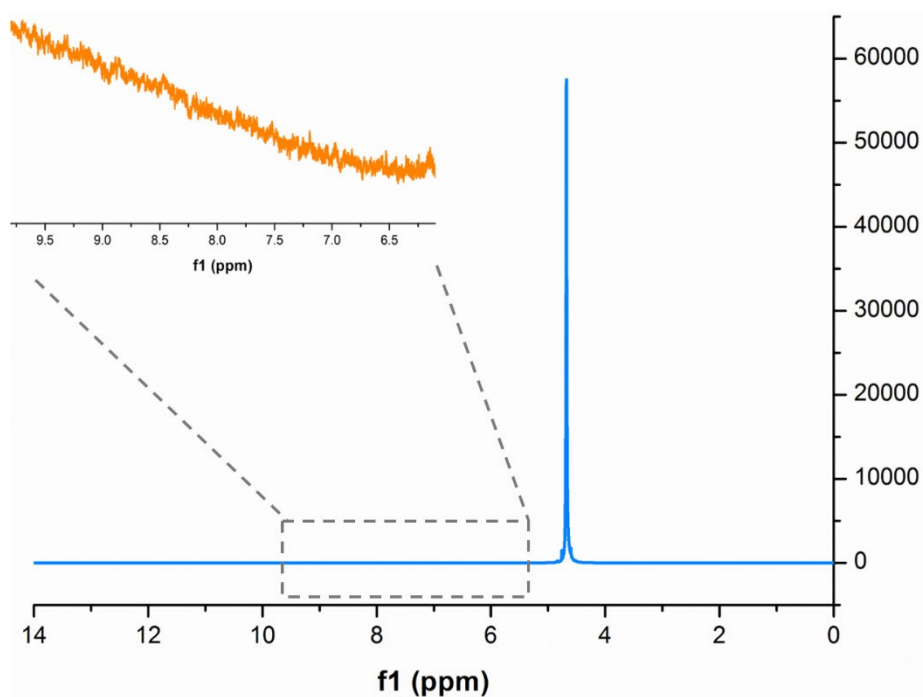


Fig. S21. The representative ^1H -NMR spectra of the electrolyte after electrolysis of -0.50 V for $\text{Ni}_7/\text{Fe}_3\text{-N-C}$ in CO_2 -saturated 0.5 M NaHCO_3 electrolyte for 30 h.

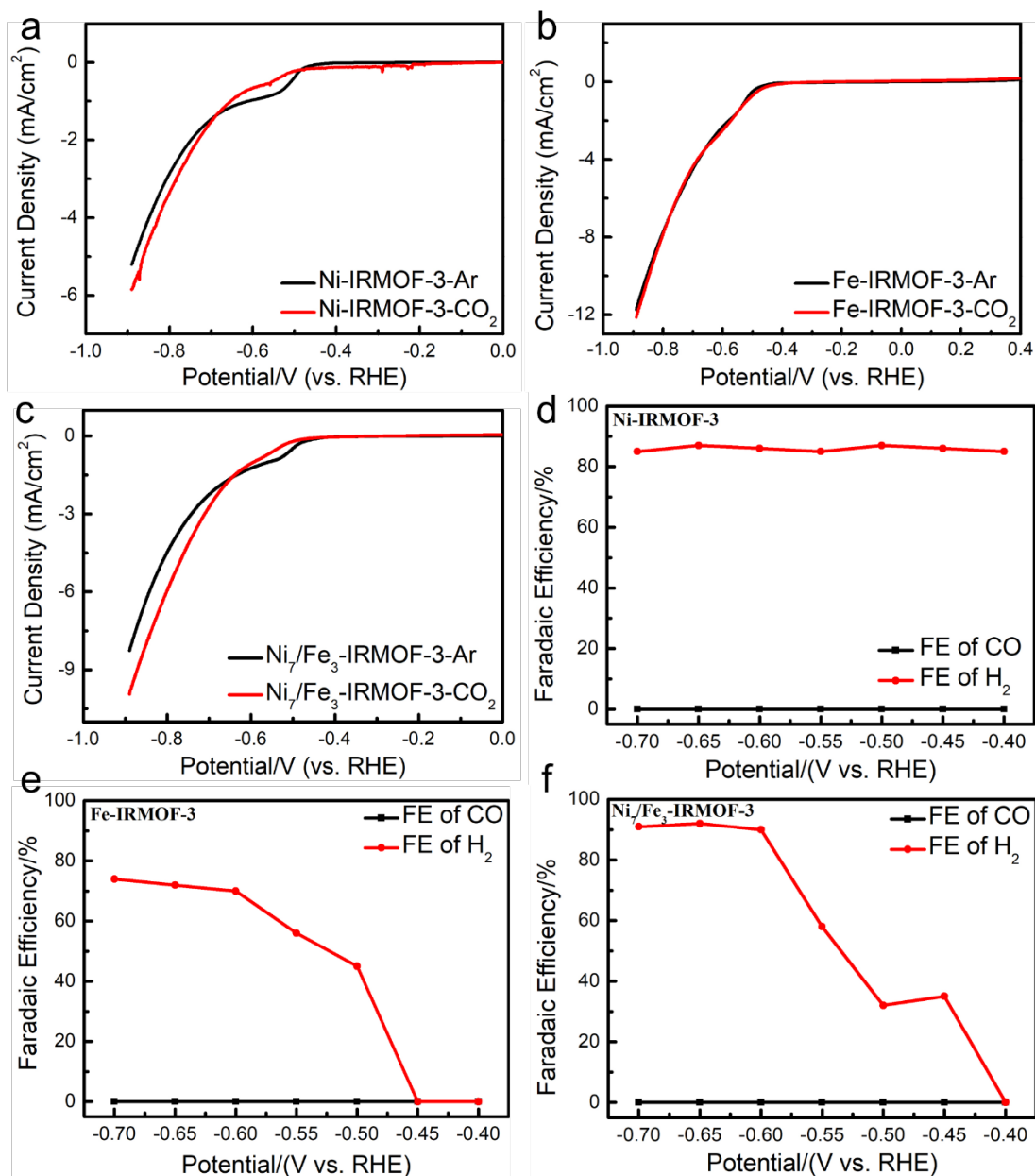


Fig. S22. LSV curves vs. RHE of (a) Ni-IRMOF-3, (b) Fe-IRMOF-3, and (c) Ni₇/Fe₃-IRMOF-3 obtained in Ar- or CO₂-saturated 0.5 M NaHCO₃ solution. FE of CO at various potentials and FE of H₂ at various potentials on (d) Ni-IRMOF-3, (e) Fe-IRMOF-3, (f) Ni₇/Fe₃-IRMOF-3.

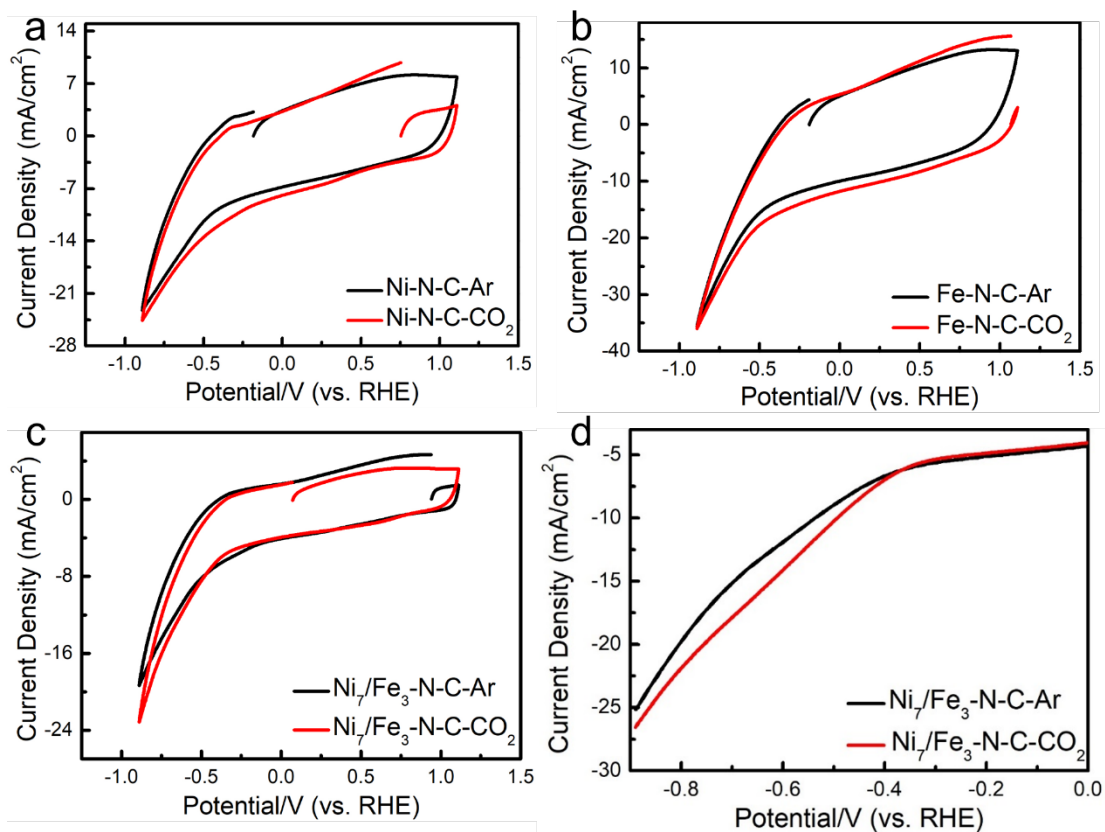


Fig. S23. CV curves vs. RHE of (a) Ni-N-C, (b) Fe-N-C, (c) Ni₇/Fe₃-N-C and (d) LSV curves vs. RHE of Ni₇/Fe₃-N-C obtained in Ar- or CO₂-saturated 0.5 M NaHCO₃ solution.

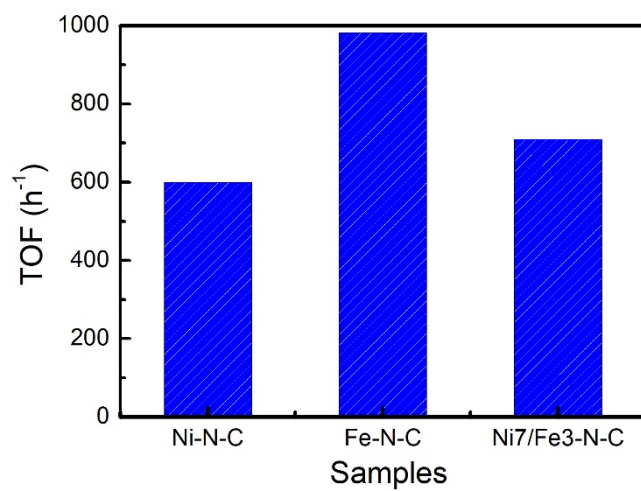
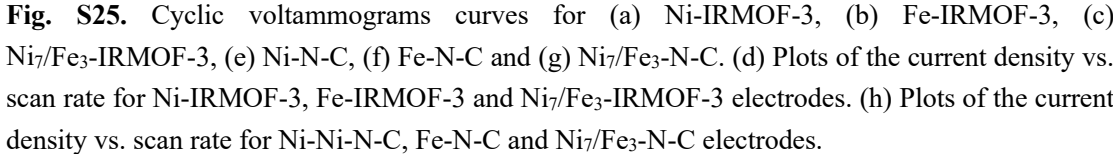


Fig. S24. TOF value of different samples.



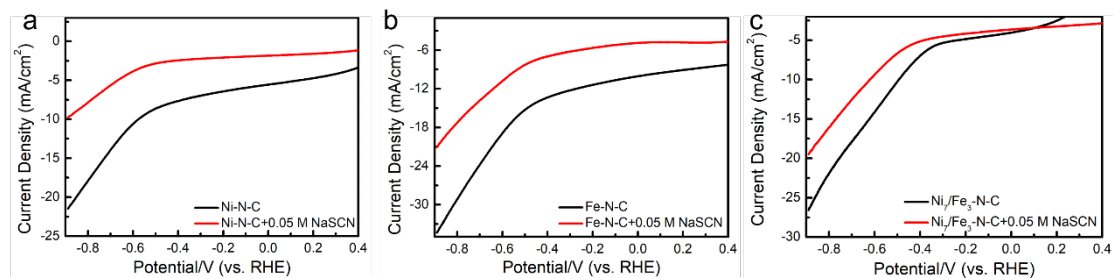


Fig. S26. Linear sweep voltammetry curves of (a) Ni-N-C, (b) Fe-N-C and (c) Ni₇/Fe₃-N-C with and without 0.05 M NaSCN.

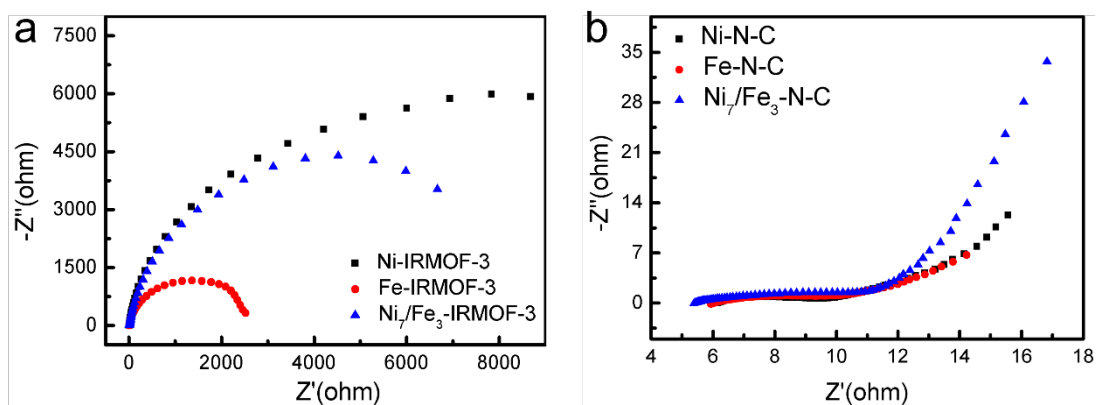


Fig. S27. Electrochemical impedance spectroscopy (EIS) of (a) Ni-IRMOF-3, Fe-IRMOF-3 and $\text{Ni}_7/\text{Fe}_3\text{-IRMOF-3}$, as well as (b) Ni-N-C, Fe-N-C and $\text{Ni}_7/\text{Fe}_3\text{-N-C}$.

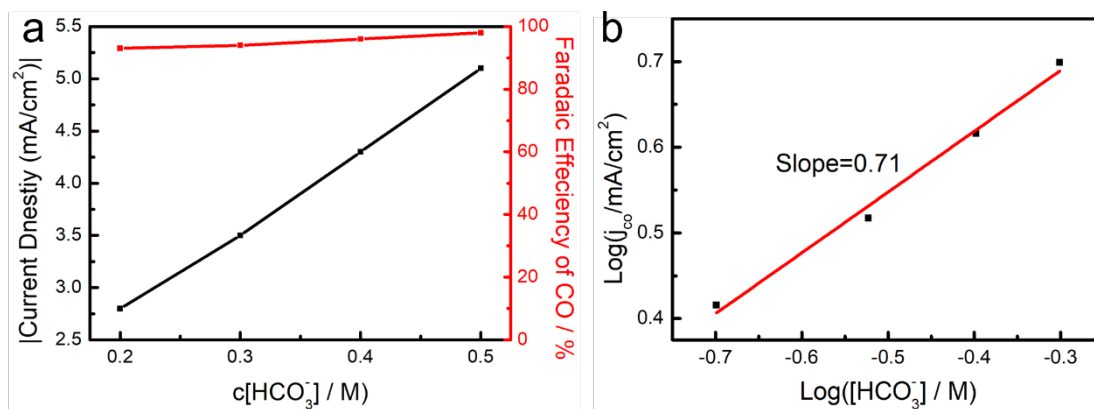


Fig. S28. (a) Current density and FE of Ni₇/Fe₃-N-C at different NaHCO₃ concentration at a constant potential (-0.50 V vs. RHE). (b) Partial CO current density of Ni₇/Fe₃-N-C vs. NaHCO₃ concentration at -0.50 V vs. RHE.

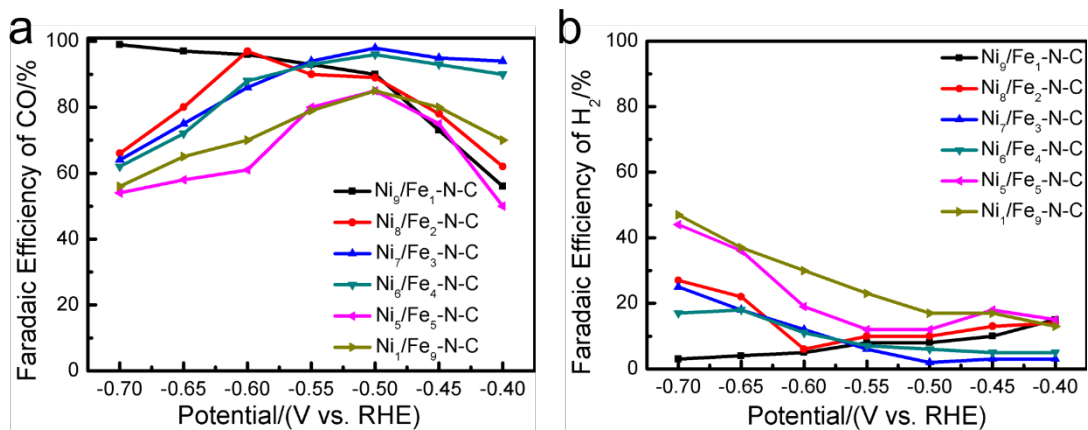


Fig. S29. (a) FE of CO and (b) FE of H₂ at various potentials on Ni_x/Fe_y-N-C.

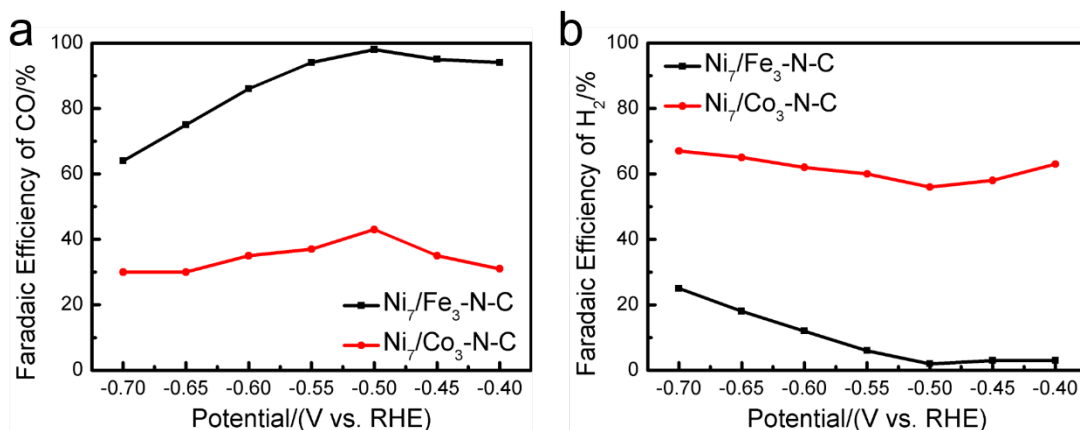


Fig. S30. (a) FE of CO and (b) FE of H₂ at various potentials on Ni₇/Fe₃-N-C and Ni₇/Co₃-N-C.

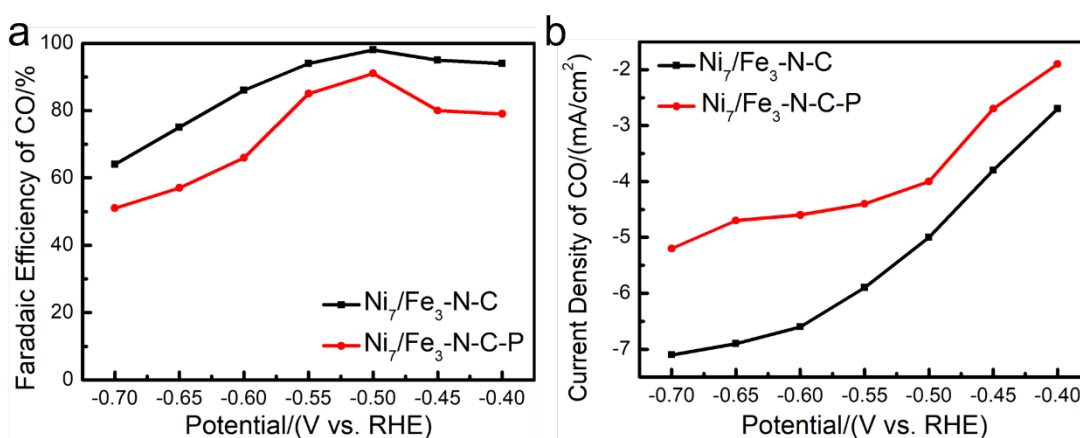


Fig. S31. (a) FE of CO at various potentials and (b) Current density for CO production on Ni₇/Fe₃-N-C and Ni₇/Fe₃-N-C-P.

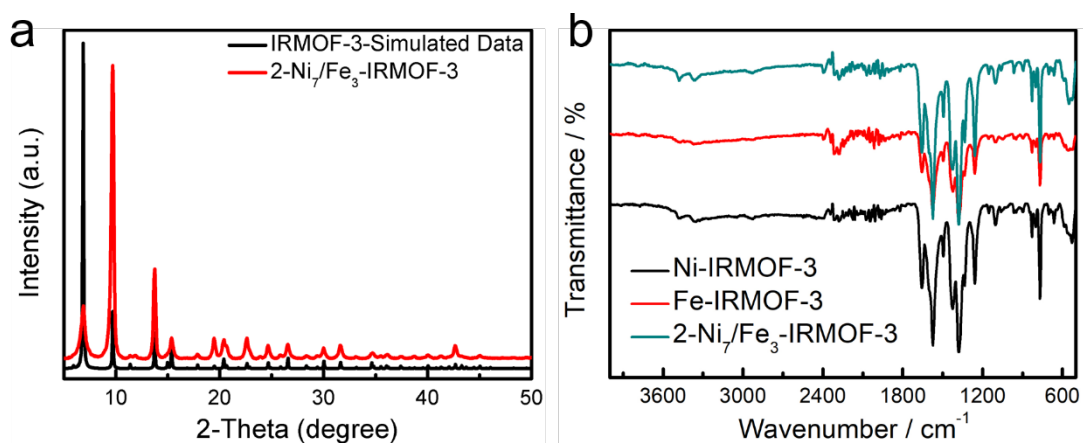


Fig. S32. (a) XRD patterns and (b) FTIR image of 2-Ni₇/Fe₃-IRMOF-3.

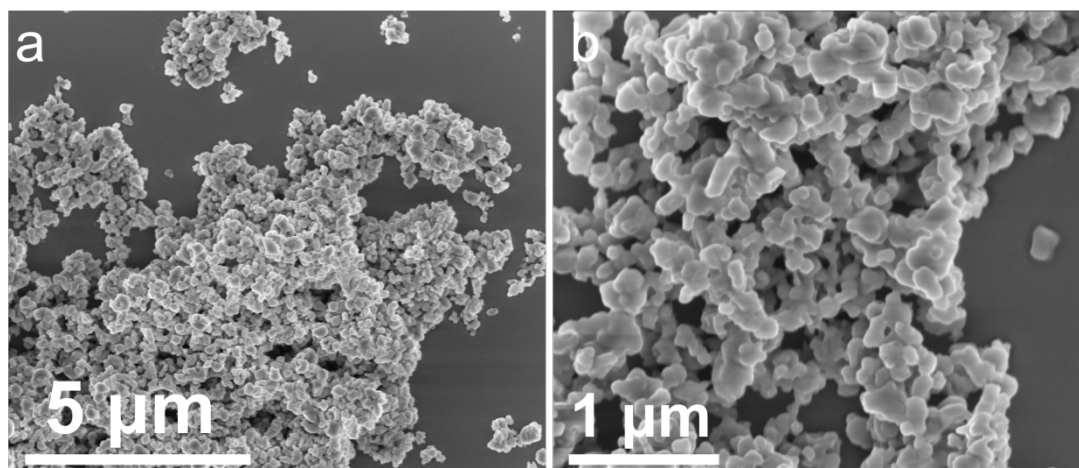


Fig. S33. SEM images of 2-Ni₇/Fe₃-IRMOF-3.

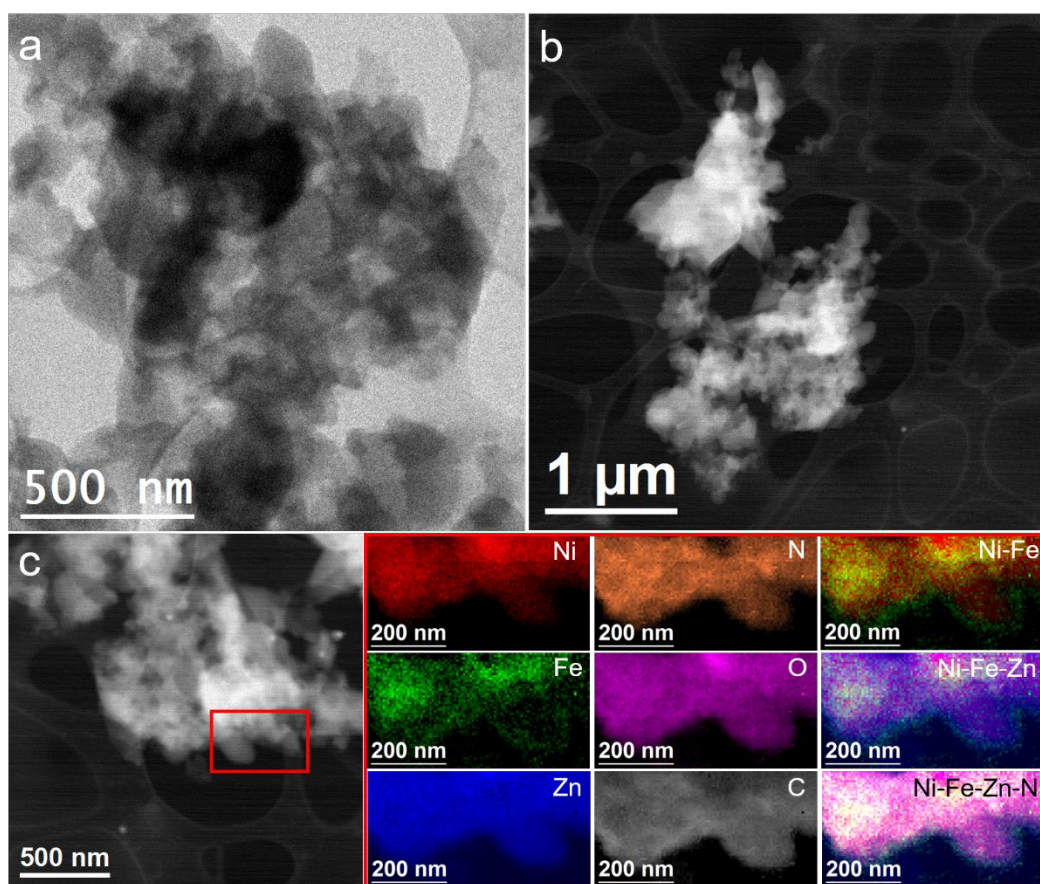


Fig. S34. (a) BF TEM, (b and c) HAADF STEM image of 2-Ni₇/Fe₃-IRMOF-3 and representative EELS chemical composition maps obtained from the red squared area of the STEM micrograph. Individual Ni L_{2,3}-edges at 855 eV (red), Fe L_{2,3}-edges at 708 eV (green), Zn L_{2,3}-edges at 1020 eV (blue), N K-edges at 401 eV (orange), O K-edges at 532 eV (pink) and C K-edges at 285 eV (grey) as well as composites of Ni-Fe, Ni-Fe-Zn and Ni-Fe-Zn-N.

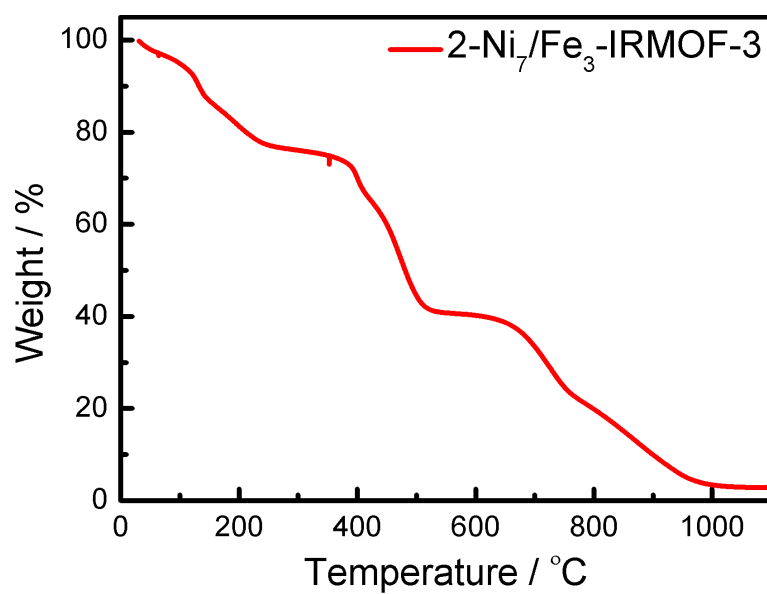


Fig. 35. TGA of 2-Ni₇/Fe₃-IRMOF-3.

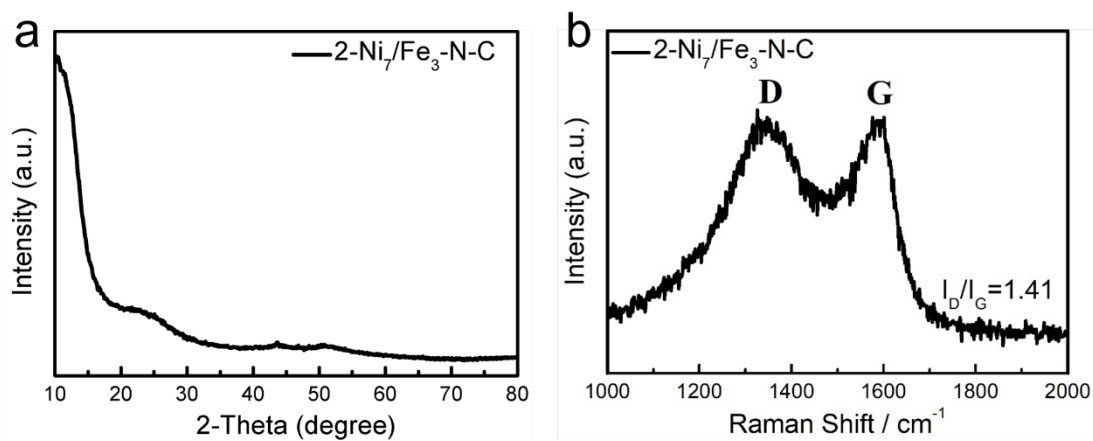


Fig. S36. (a) XRD patterns and (b) Raman spectra of 2-Ni₇/Fe₃-N-C.

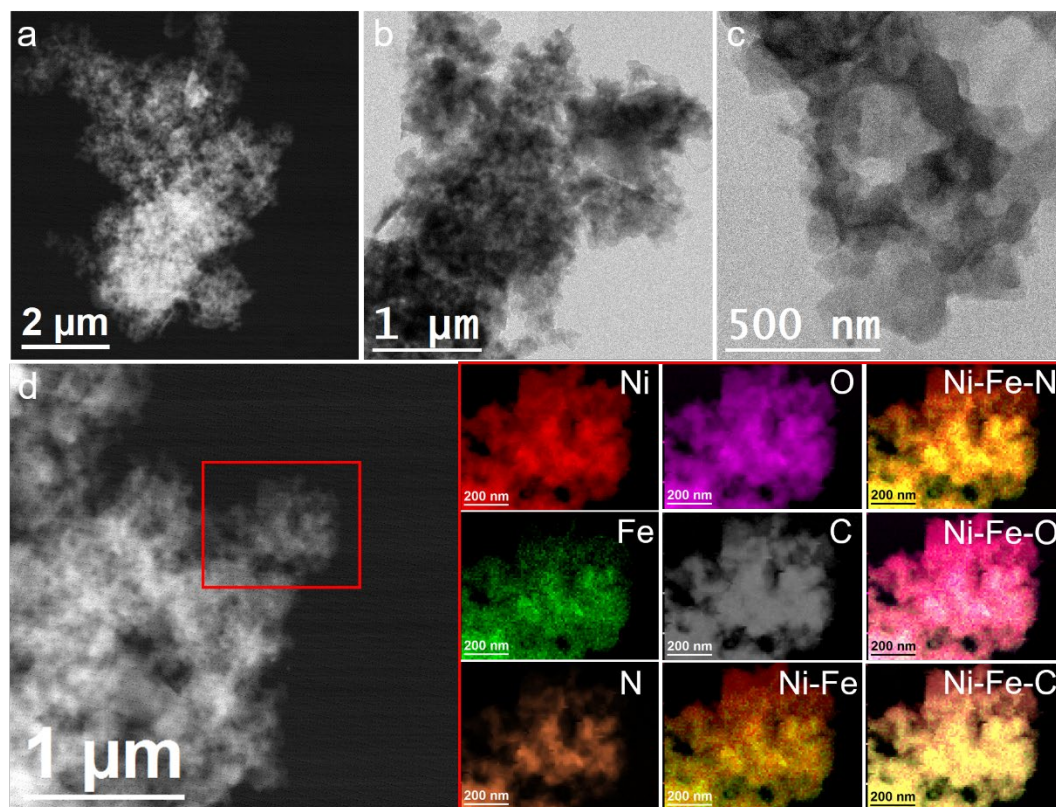


Fig. S37. (a) HAADF STEM, (b, c) BF TEM images of 2-Ni₇/Fe₃-N-C, (d) HAADF STEM image and representative EELS chemical composition maps obtained from the red squared area of the STEM micrograph. Individual Ni L_{2,3}-edges at 855 eV (red), Fe L_{2,3}-edges at 708 eV (green), N K-edges at 401 eV (orange), O K-edges at 532 eV (pink) and C K-edges at 285 eV (grey) as well as composites of Ni-Fe, Ni-Fe-N, Ni-Fe-O and Ni-Fe-C.

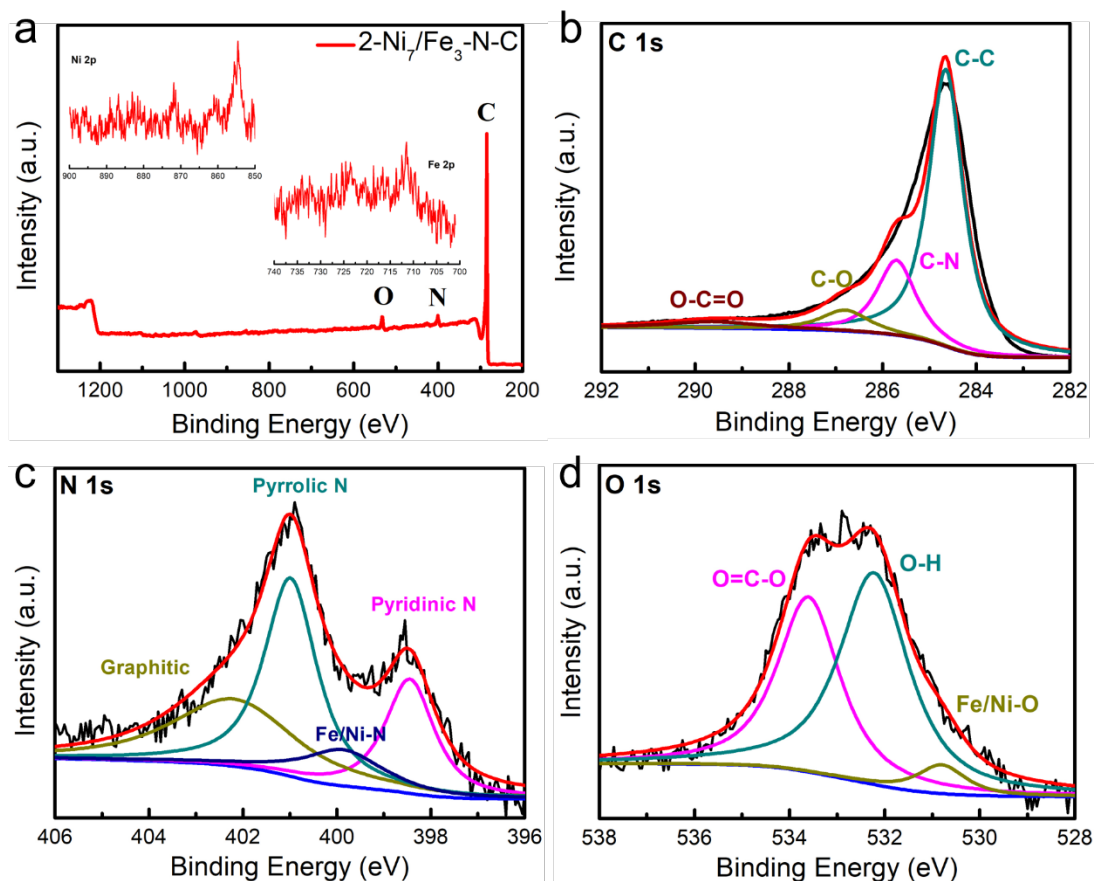


Fig. S38. XPS spectra for the (a) survey scan, (inset) Ni 2p and Fe 2p, (b) C 1s, (c) N 1s and (d) O 1s of 2-Ni₇/Fe₃-N-C.

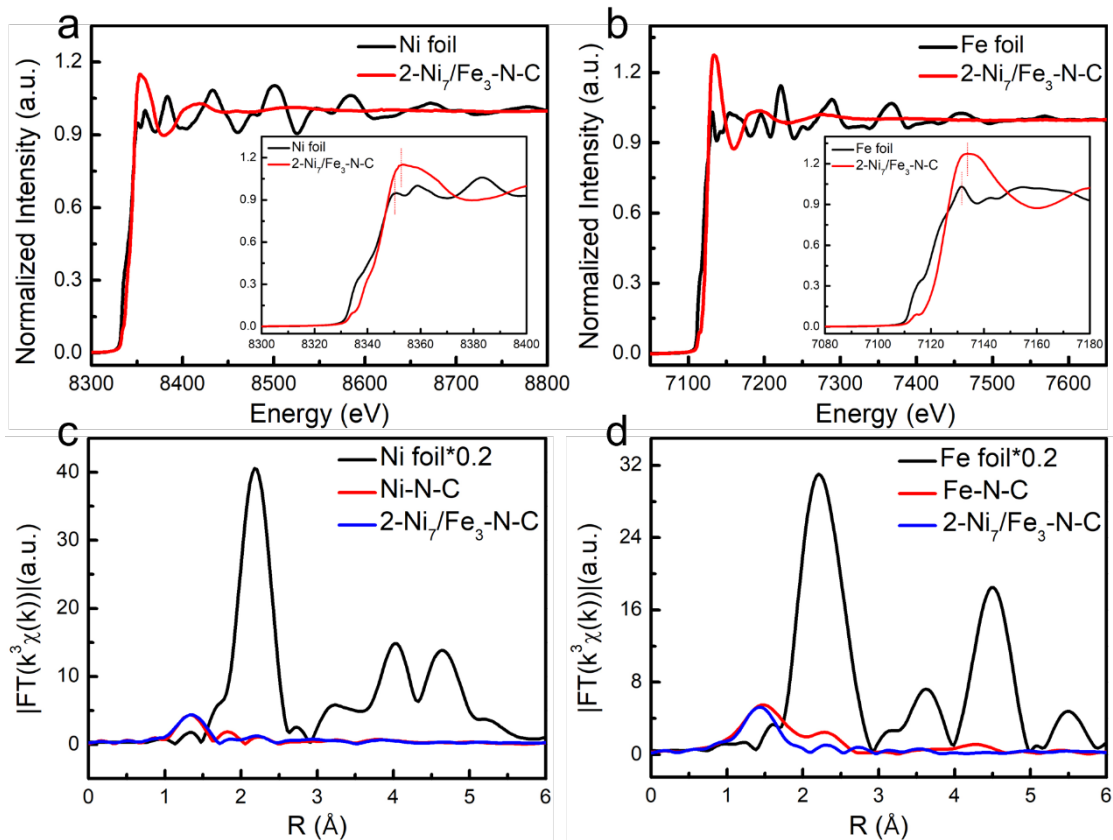


Fig. S39. (a) Ni K-edge XANES spectra of Ni foil and 2-Ni₇/Fe₃-N-C. (b) Fe K-edge XANES spectra of Fe foil and 2-Ni₇/Fe₃-N-C. (c and d) Fourier transformation of the EXAFS spectra at R space.

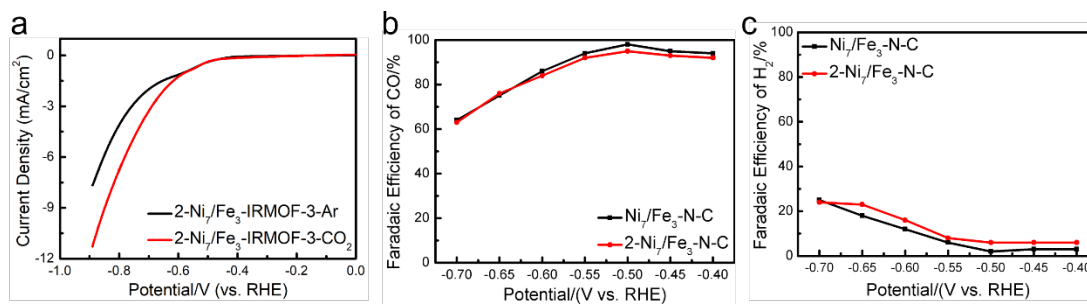


Fig. S40. (a) LSV of 2-Ni₇/Fe₃-N-C obtained in Ar- or CO₂-saturated 0.5 M NaHCO₃ solution. FE of (b) CO and (c) FE of H₂ at various potentials on Ni₇/Fe₃-N-C and 2-Ni₇/Fe₃-N-C.

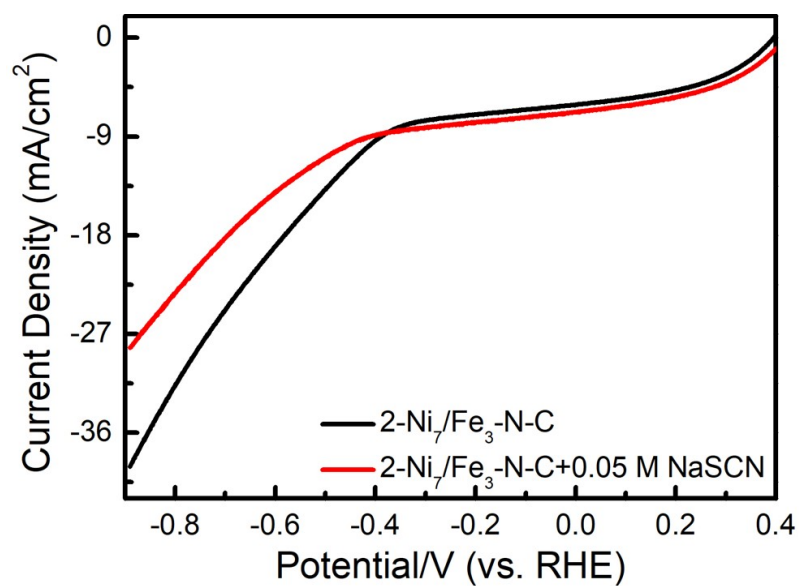


Fig. S41. Linear sweep voltammetry curves of 2-Ni₇/Fe₃-N-C with and without 0.05 M NaSCN.

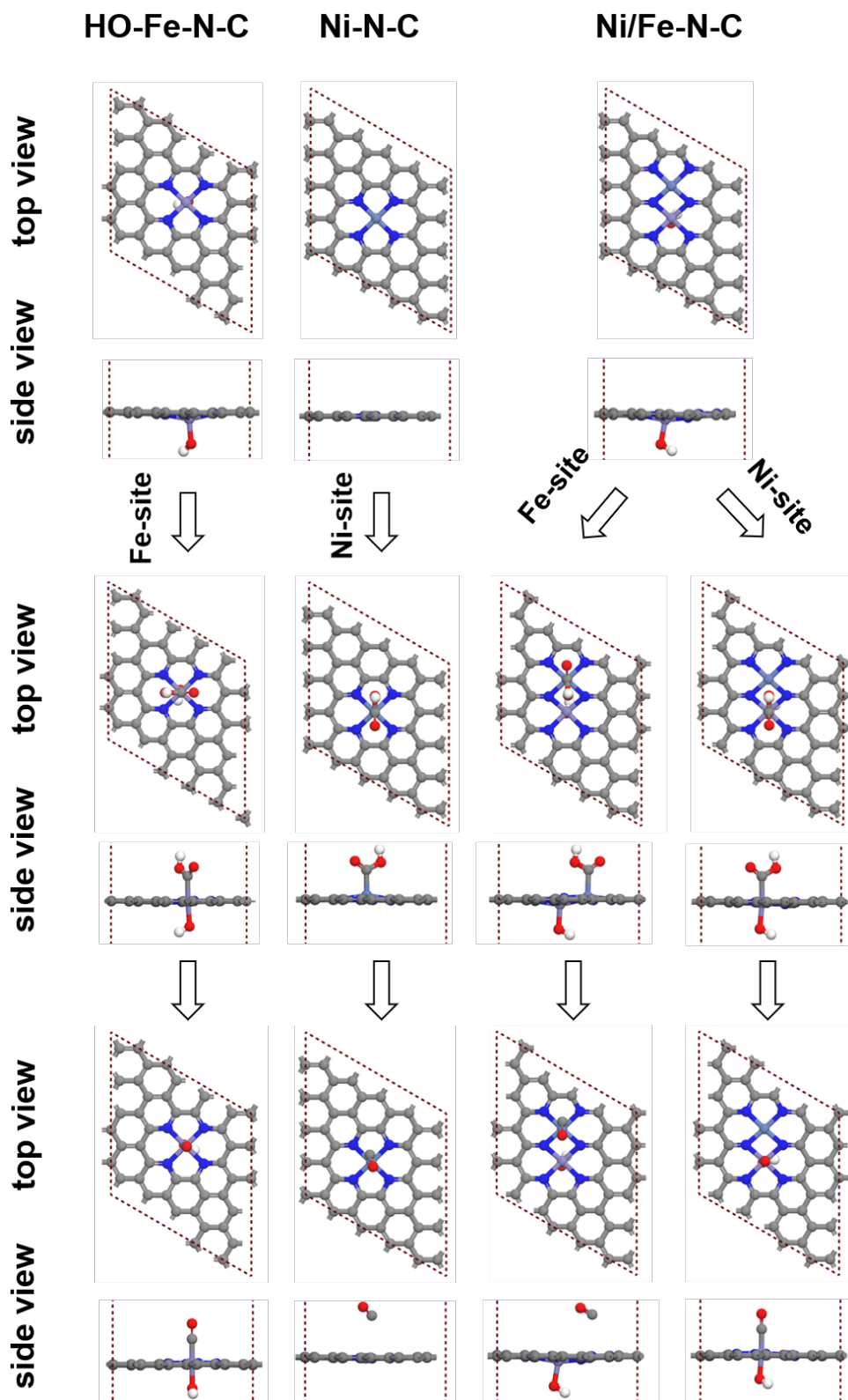


Fig. S42. The top view and side view of optimized adsorption configuration on simulated models (Ni, Fe, O, N and C atoms are represented in purple, cyan, red, blue and grey, respectively).

Table S4. Faradaic Efficiency (CO) of the reported single/double atom-based electrocatalysts for CO₂ electroreduction.

Catalyst	Product	FE(CO)	Potential	Reference
Fe-N-C	CO	93 %	−0.60 V vs. RHE	18
Fe-N-C	CO	81 %	−0.57 V vs. RHE	19
Fe-N-C	CO	93.5 %	−0.50 V vs. RHE	20
Fe-N-C	CO	93 %	−0.60 V vs. RHE	21
Fe-N-C	CO	80 %	−0.60 V vs. RHE	22
Fe-N-C	CO	92 %	−0.58 V vs. RHE	23
Fe-N-C	CO	64 %	−0.60 V vs. RHE	24
Fe-N-C	CO	95.47 %	−0.60 V vs. RHE	25
Fe-N ₅ -C	CO	97 %	−0.46 V vs. RHE	26
Fe-N-C	CO	95 %	−0.45 V vs. RHE	27
Ni-N-C	CO	99 %	−0.68 V vs. RHE	5
Ni-N-C	CO	71.9 %	−0.90 V vs. RHE	6
Ni-N-C	CO	95 %	−0.70 V vs. RHE	28
Ni-N-C	CO	99 %	−0.81 V vs. RHE	29
Ni-N-C	CO	96 %	−0.67 V vs. RHE	30
Ni-N-C	CO	96 %	−0.86 V vs. RHE	31
Ni-N-C	CO	95 %	−0.77 V vs. RHE	32
Ni-N-C	CO	91.2 %	−0.90 V vs. RHE	33
Ni-N-C	CO	97 %	−0.70 V vs. RHE	34
Ni-N-C	CO	95 %	−0.77 V vs. RHE	35
Ni-N-C	CO	99 %	−0.80 V vs. RHE	36
Ni-N-C	CO	94 %	−0.80 V vs. RHE	37
NiCo-N-C	CO	53 %	−0.60 V vs. RHE	38
Ni/Fe-N-C	CO	98 %	−0.70 V vs. RHE	39
Ni/Fe-N-C	CO	90 %	−0.65 V vs. RHE	40
Ni/Mn-C ₃ N ₄ -CNT	CO	90 %	−0.70 V vs. RHE	41
ZnCo-N-C	CO	93.2 %	−0.50 V vs. RHE	42
Ni-N-Co	CO	96.4 %	−0.48 V vs. RHE	43
Ni₇/Fe₃-N-C	CO	98 %	−0.50 V vs. RHE	This work

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