Supplementary Information (SI) for Materials Chemistry Frontiers.
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Supporting Information

Single-Crystalline NiO Octahedron with (111) facet as a Bifunctional Electrocatalyst for Overall Water Splitting

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Characterization Techniques

The $Ni(OH)_2$ and NiO materials were analyzed using various characterization techniques. The powder X-ray diffraction patterns of the samples were obtained using a Bruker D-8 Advance diffractometer, operating at 40 kV voltage and 40 mA current, and utilizing Cu Ka radiation with a wavelength of 0.15406 nm. The Powder X-ray diffraction patterns were analyzed using MATCH software, Version 3.x, by CRYSTAL IMPACT located at Kreuzherrenstr, 102, 53227 Bonn, Germany to determine the phase of the synthesized materials.

Morphology analysis was conducted using a JEOL JEM 6700 Field Emission Scanning Electron Microscope (FE SEM).

TEM images were captured using a JEOL 2010 TEM operating at 200 kV.

Raman spectra were recorded using a Bruker Senterra Raman microscope.

X-ray photoelectron spectroscopy (XPS) was performed on a Thermo Scientific (Model No ESCALAB 250Xi). X-ray Photoelectron Spectrometer operated at 15 kV and 20 mA with a monochromatic Al Kα X-ray source.

The electrochemical experiments were conducted using a computer-controlled electrochemical workstation (BioLogic 150e) within a standard three-electrode system.

Electrochemical Measurements

Electrode Preparation: The working electrode utilized in this experiment was the NiO octahedron materials on fluorine doped tin oxide (FTO), with an electrolyte solution of 1 M KOH. The working electrode was made according to usual technique. FTO glasses were cleaned with acetone and ethanol under sonication bath. To disperse 2 mg of powdered NiO, a combination of 40 μL dry ethanol and 20 μL 5% Nafion was used, followed by 30 minutes of sonication. A 5 μL suspension was gently drop cast onto an FTO−coated plate, resulting in a circular film. The film was then dried at ambient humidity to serve as a functioning electrode.

Electrochemical Characterization: The electrochemical experiments were carried out on a computer−controlled electrochemical workstation (BioLogic 150e) in a three−electrode system with a Pt wire and a saturated Ag/AgCl electrode (in 3 M NaCl) serving as the counter and reference electrodes, respectively. For the OER and HER experiment, the LSV studies were completed with a scan rate of 50 mV/s. The iR compensation LSV was done manually with eighty five percent of the Rs of impedance spectroscopy (EIS). LSV Stability test of NiO materials studies was carried out with a scan rate of 100 mV/s for 1000 cycles in the 1 M KOH solution for HER and OER. The cyclic voltammograms (CV) were measured in 1 M KOH solution with a scan rate 50 mV/s. The applied potentials vs. Ag/AgCl (NaCl Sat'd) were converted to RHE (Reversible Hydrogen Electrode) potentials using the following equations:

$E_{RHE} = E_{Ag/AgCl} + 0.0591pH + E^{\theta}_{Ag/AgCl}$ ($E^{\theta}_{Ag/AgCl} = 0.194\ V$ vs NHE at 25 °C)

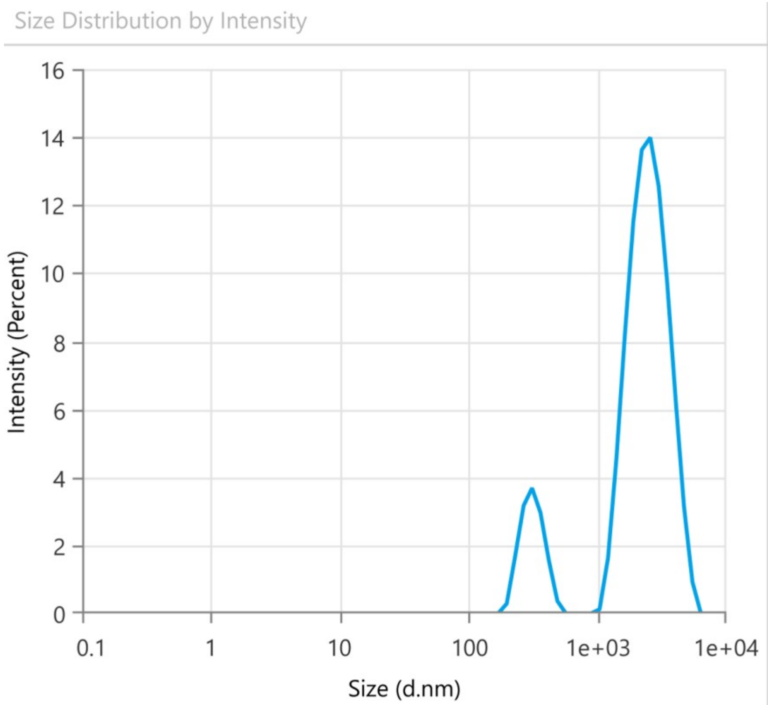

Fig. S1. DLS study of NO-8 sample showing bimodal distribution.

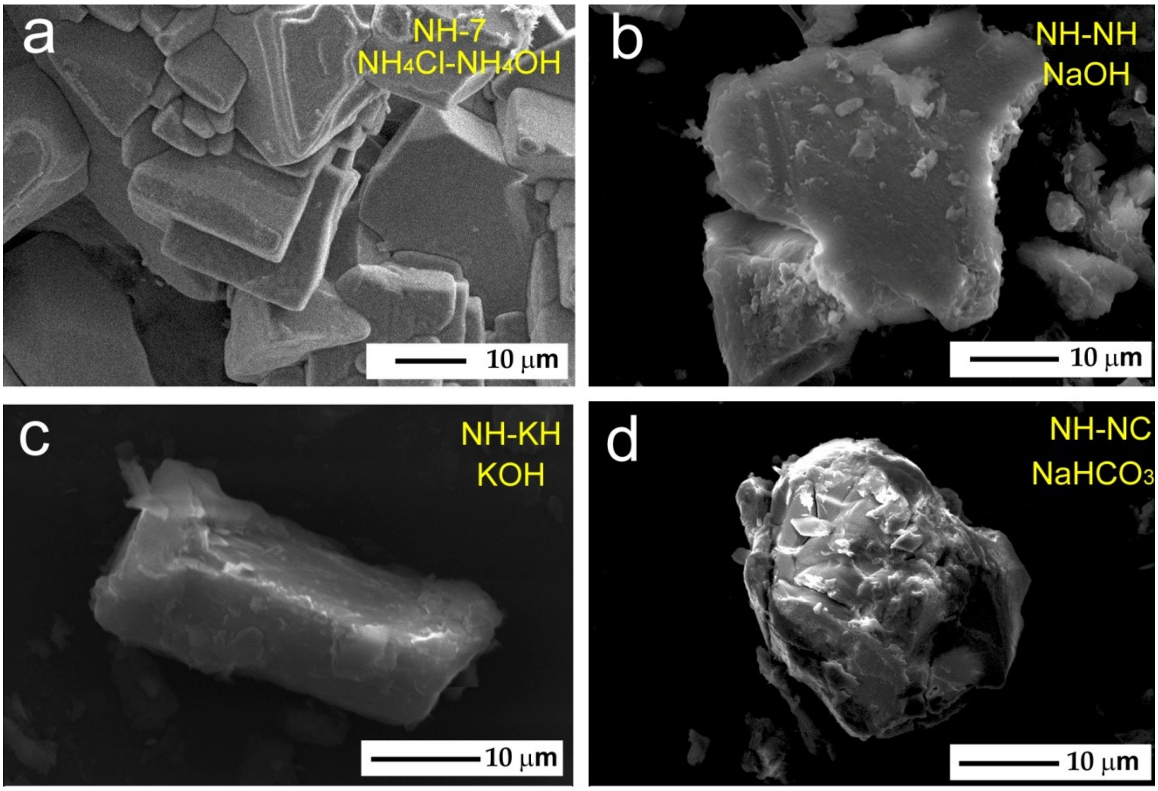

Fig. S2. FE SEM images of as-synthesized materials obtained in different base medium. a) NH-7 (NH_4Cl-NH_4OH), b) NH-NH (NaOH), c) NH-KH (KOH), and d) NH-NC ($NaHCO_3$).

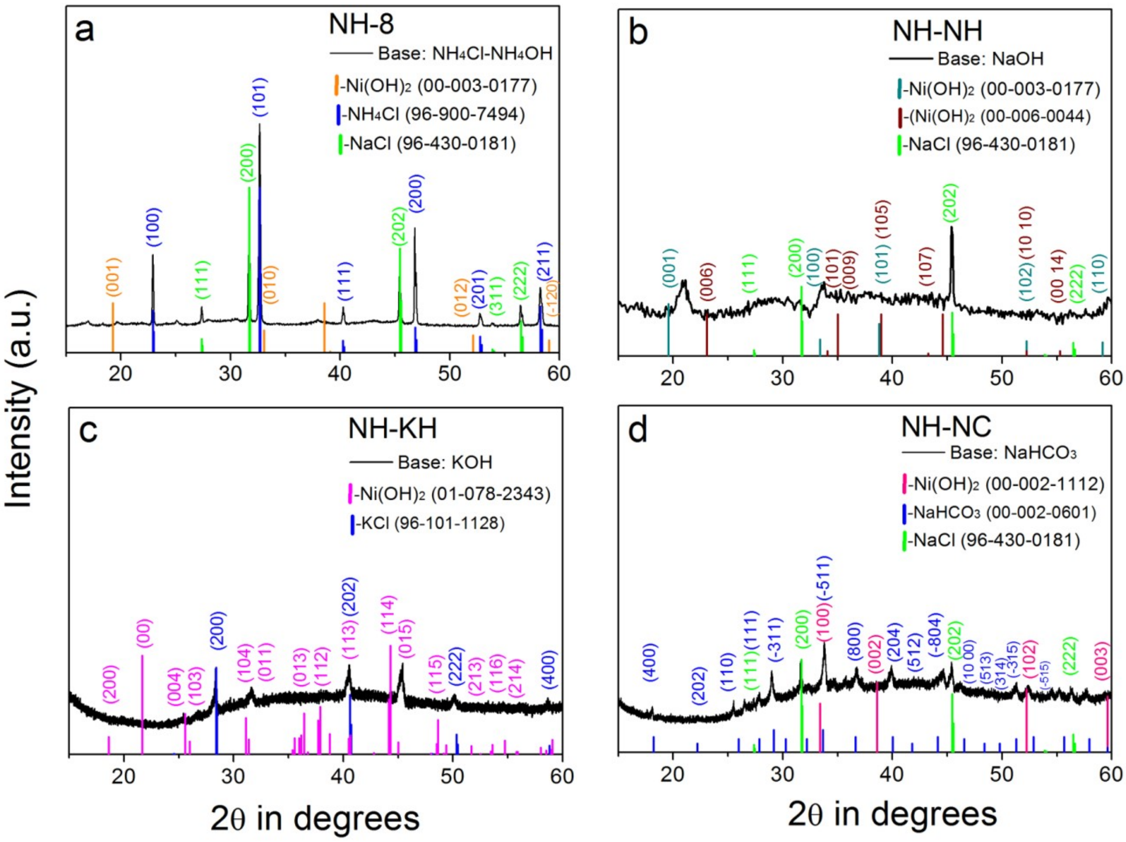

Fig. S3. Wide-angle powder XRD pattern of different as-synthesized materials at different base medium. a) NH-8 (NH_4Cl - NH_4OH), b) NH-NH (NaOH), c) NH-KH (KOH), and d) NH-NC ($NaHCO_3$).

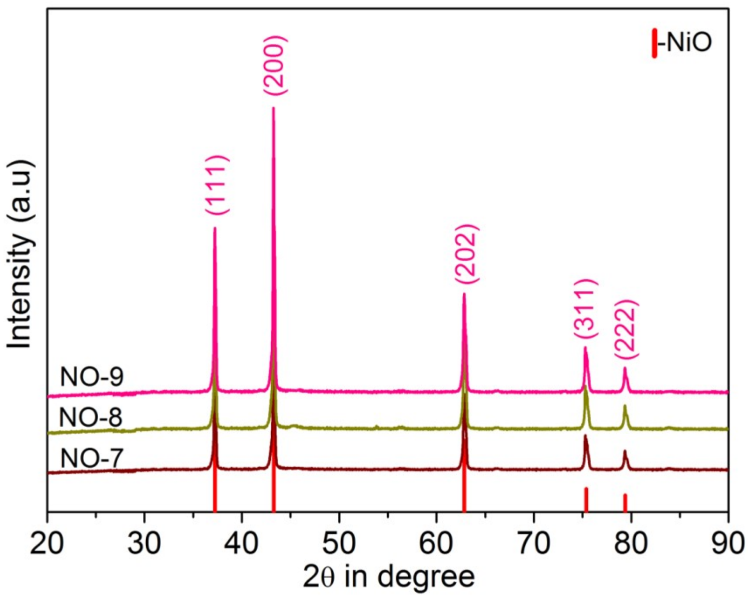

Fig. S4. Wide-angle powder XRD pattern of NO-7, NO-8, and NO-9.

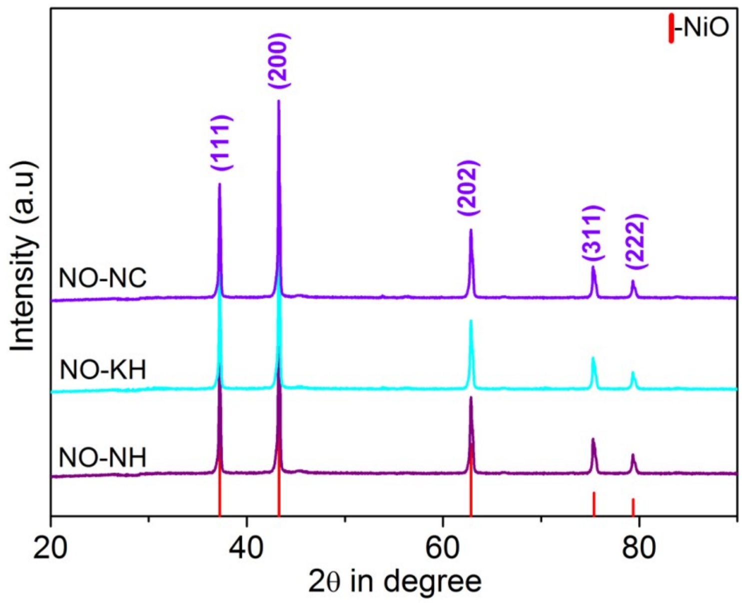

Fig. S5. Wide-angle powder XRD pattern of NO-NH, NO-KH, and NO-NC.

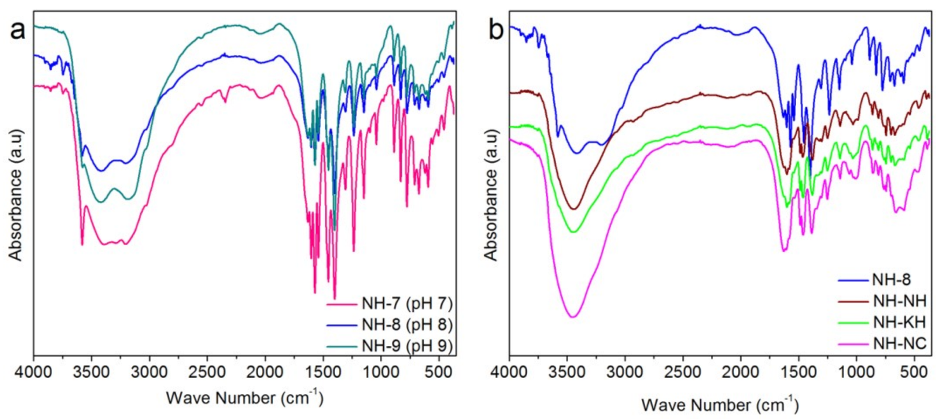

Fig. S6. FT IR images of material obtained in different condition. a) Different pH: NH-7 (pH 7), NH-8 (pH 8), NH-9 (pH 9), and b) different bases: NH-8 (NH_4Cl-NH_4OH), NH-NH (NaOH), NH-KH (KOH), NH-NC ($NaHCO_3$).

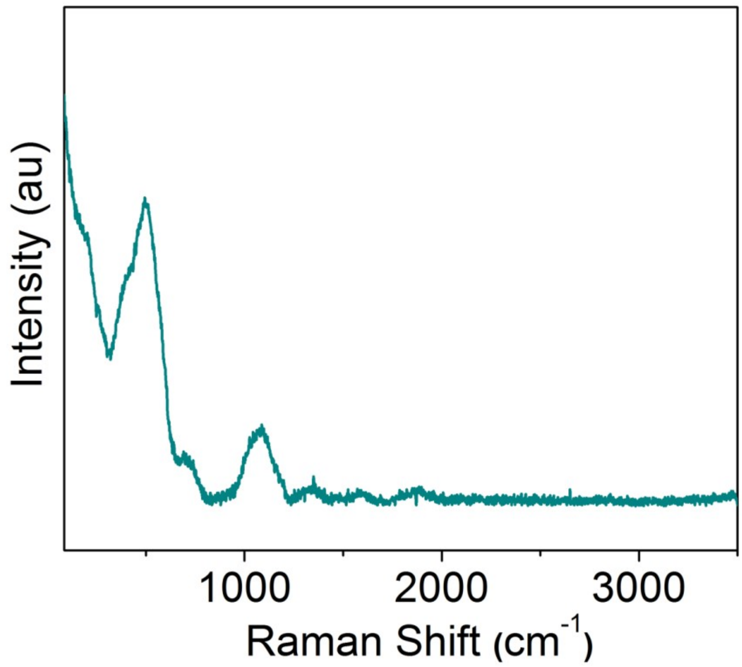

Fig. S7. Raman spectra of NiO octahedrons (NO-8 sample).

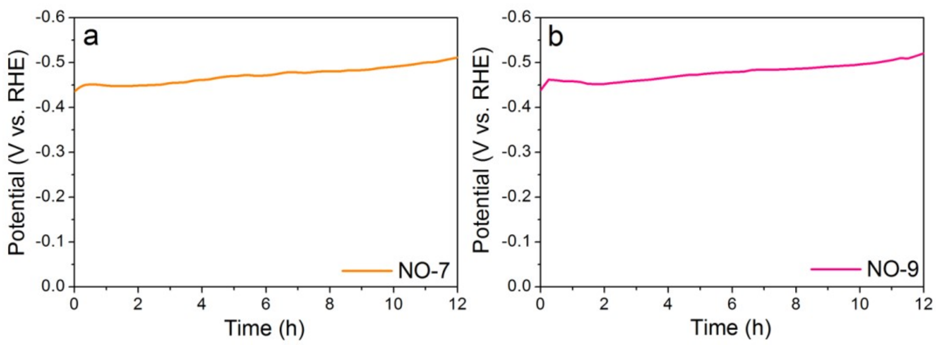

Fig. S8. Long-term chronopotentiometric stability test (HER) of NiO octahedrons applying a constant current -10 mA/cm^2. a) NO-7 and b) NO-9.

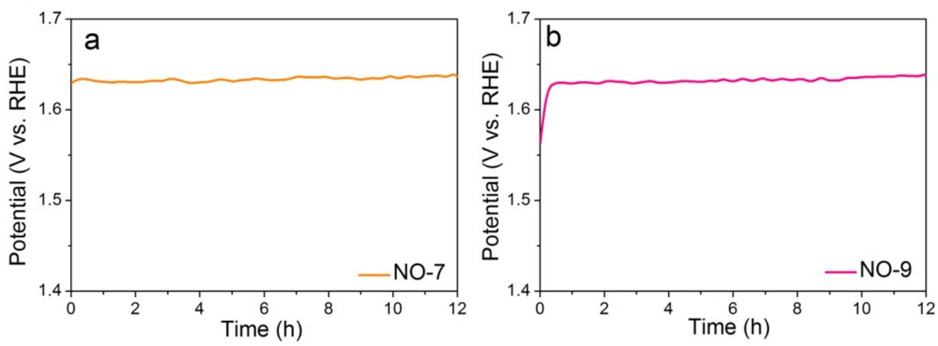

Fig. S9. Long-term chronopotentiometric stability test (OER) of NiO octahedrons applying a constant current 10 mA/cm2. a) NO-7 and b) NO-9.

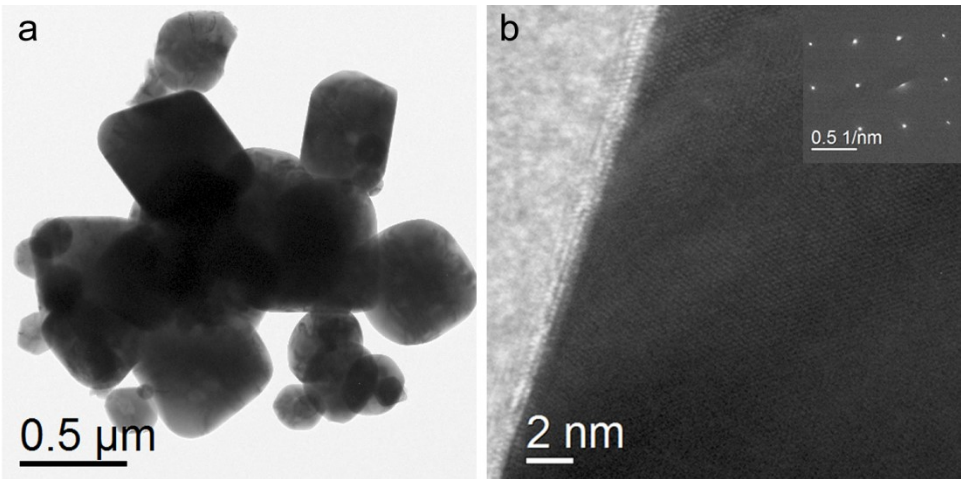

Fig. S10. TEM analysis of NO-8 after OER LSV measurement.

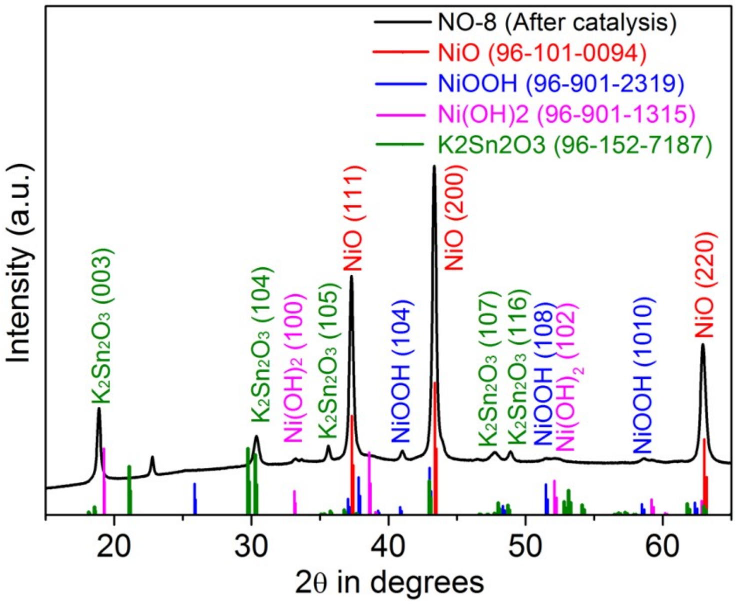

Fig. S11. Powder XRD analysis of NO-8 after OER LSV measurement.

Table S1. Comparison of HER catalytic performance with other catalysts on recently available literatures.

Sl No	Material	Medium	Overpotential (mV) @10 mA/cm^2	Tafel Slope (mV/dec)	Ref
1	Co_3O_4 nanocube {001} (NF)	1 (M) KOH	284	97	*ACS Appl. Mater. Interfaces* **2017**,*9*, 27736–27744
2	Co_3O_4 nanobelt {110}(NF)	1 (M) KOH	260	78	*ACS Appl. Mater. Interfaces* **2017**, *9*, 27736–27744
3	Co_3O_4 octahedron {111}(NF)	1 (M) KOH	195	50	*ACS Appl. Mater. Interfaces* **2017**, *9*, 27736–27744
4	Co_3O_4 nanosheet {112}(NF)	1 (M) KOH	232	59	*ACS Appl. Mater. Interfaces* **2017**, *9*, 27736–27744
5	$CoSn(OH)_6$ Nanocube {220} (GCE)	3 (M) KOH	262.7	309.5	*Electrochimica Acta,* **2024**, *507,* 145190
6	$CoSn(OH)_6$ Cube {400} (GCE)	3 (M) KOH	191.4	348.9	*Electrochimica Acta,* **2024**, *507,* 145190
7	$CoSn(OH)_6$ Octahedron {422} (GCE)	3 (M) KOH	168.8	268.9	*Electrochimica Acta,* **2024**, *507,* 145190
8	$CoSn(OH)_6$ Dodecahedron {420} (GCE)	3 (M) KOH	161.9	275.4	*Electrochimica Acta,* **2024**, *507,* 145190
9	$CuMn_2O_4${400}{200}(Cu-Foam)	1 (M) KOH	116	115	*J. Mater. Chem. A,* **2022**, *10,* 17710–17720
10	$NiSe_2$ Nanooctahedron {211} @NF	1 (M) KOH	345 @ 50 mV/dec	104	*ACS Appl. Energy Mater.* **2025**, *8 (4)*, 2088–2102
11	NiMoN Nanowire {100} (NF)	1 (M) KOH	290 @ 50 mV/dec	46	*ChemSusChem* **2018**, *11*, 3198 – 3207
13	NiO (Calcined at 300 °C) (GCE)	1 (M) KOH	424	105	*Int. J. Hydrogen Energy,* **2018**, *43,* 21665–21674
14	NiO (Calcined at 400 °C) (GCE)	1 (M) KOH	498	125	*Int. J. Hydrogen Energy,* **2018**, *43,* 21665–21674
15	NiO (Calcined at 500 °C) (GCE)	1 (M) KOH	553	145	*Int. J. Hydrogen Energy,* **2018**, *43,* 21665–21674
16	NiO Hollow Microsphere (GCE)	1 (M) KOH	372	165	*Electrochimica Acta,* **2020**, *361,* 137040
17	NiO_x-SC (FTO)	0.5 (M) KOH	350	89	*ACS Omega,***2020**, *5,* 10641−10650
18	Octahedron NiO {111} (FTO)	1 (M) KOH	369.92	239.8	***This Work***

TableS2. Comparison of OER catalytic performance with other catalysts on recently available literatures.

Sl No	Material	Medium	Overpotential (mV) @10 mA/cm^2	Tafel Slope (mV/dec)	Ref
1	Co_3O_4{111} truncatedOctahedron (Carbon Paper)	1 (M) KOH	423	78.8	*ACS Energy Lett.* **2024**, *9*, 2182−2192
2	Cubic Co_3O_4 {001} containing Ovs (Carbon Paper)	1 (M) KOH	375	58.2	*ACS Energy Lett.* **2024**, *9*, 2182−2192
3	Co_3O_4{001} Cubic (Carbon Paper)	1 (M) KOH	460	96.9	*ACS Energy Lett.* **2024**, *9*, 2182−2192
4	Co_3O_4 {111} Truncated Octahedron (Carbon Paper)	1 (M) KOH	460	107.3	*ACS Energy Lett.* **2024**, *9*, 2182−2192
5	Co_3O_4 nanocube {001} (NF)	1 (M) KOH	362	119	*ACS Appl. Mater. Interfaces* **2017**, *9*, 27736−27744
6	Co_3O_4 nanobelt {110} (NF)	1 (M) KOH	341	101	*ACS Appl. Mater. Interfaces* **2017**, *9*, 27736−27744
7	Co_3O_4 octahedron {111} (NF)	1 (M) KOH	320	49	*ACS Appl. Mater. Interfaces* **2017**, *9*, 27736−27744
8	Co_3O_4 nanosheet {112} (NF)	1 (M) KOH	312	73	*ACS Appl. Mater. Interfaces* **2017**, *9*, 27736−27744
9	$CoSn(OH)_6$ Nanocube {220} (GCE)	3 (M) KOH	235	108.2	*Electrochimica Acta,* **2024**, *507,* 145190
10	$CoSn(OH)_6$ Cube {400} (GCE)	3 (M) KOH	233.6	103.9	*Electrochimica Acta,* **2024**, *507,* 145190
11	$CoSn(OH)_6$ Octahedron {422} (GCE)	3 (M) KOH	232	89.7	*Electrochimica Acta,* **2024**, *507,* 145190
12	$CoSn(OH)_6$ Dodecahedron {420} (GCE)	3 (M) KOH	228	76.1	*Electrochimica Acta,* **2024**, *507,* 145190
13	$CuMn_2O_4${400}{200} (Cu-Foam)	1 (M) KOH	406	135	*J. Mater. Chem. A,* **2022**, *10,* 17710–17720
14	$NiSe_2$ Nanooctahedron {211} @NF	1 (M) KOH	119	165	*ACS Appl. Energy Mater.* **2025**, *8 (4),* 2088–2102
15	NiO {111} Microwave (GCE)	0.1 (M) KOH	414	50.8	*ACS Appl. Mater. Interfaces* **2024**, *16,* 62142−62154
16	NiO {111} Solvothermal (GCE)	0.1 (M) KOH	405	52.3	*ACS Appl. Mater. Interfaces* **2024**, *16,* 62142−62154
17	NiO (Calcined at 300°C) (GCE)	1 (M) KOH	370	156	*Int. J. Hydrogen Energy,* **2018**, *43,* 21665–21674
18	NiO (Calcined at	1 (M)	470	188	*Int. J. Hydrogen Energy,*

	400°C) (GCE)	KOH			**2018**, *43,* 21665–21674
19	NiO (Calcined at 500°C) (GCE)	1 (M) KOH	540	290	*Int. J. Hydrogen Energy,* **2018**, *43,* 21665–21674
20	NiO_x-SC (FTO)	0.5 (M) KOH	300	57	*ACS Omega,***2020**, *5,* 10641−10650
21	NiPc350 (FTO)	0.1 NaOH	610	147	*ChemistrySelect,***2018**, *3,* 11357−11366
24	NiCoO (FTO)	1 (M) KOH	460	191	*ACS Omega,***2023**, *8,* 9539−9546
26	$Ni_xCo_yMn_xO_4$ (Calcined at 500 °C) (FTO)	1 (M) KOH	436	78	*ACS Omega,***2024**, *9,* 43503−43512
27	$Ni_xCo_yMn_xO_4$ (Calcined at 700 °C) (FTO)	1 (M) KOH	465	84	*ACS Omega,***2024**, *9,* 43503−43512
28	NiO Octahedron{111}(FTO)	1 (M) KOH	389	65.67	***This Work***