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In-situ generated 3D porous nanostructure onto 2D nanosheets to boost oxygen evolution reaction for water-splitting

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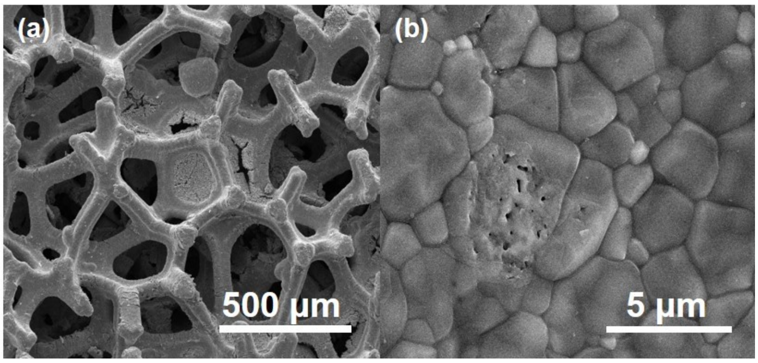

Figure S1. SEM image of iron foam with different magnification.

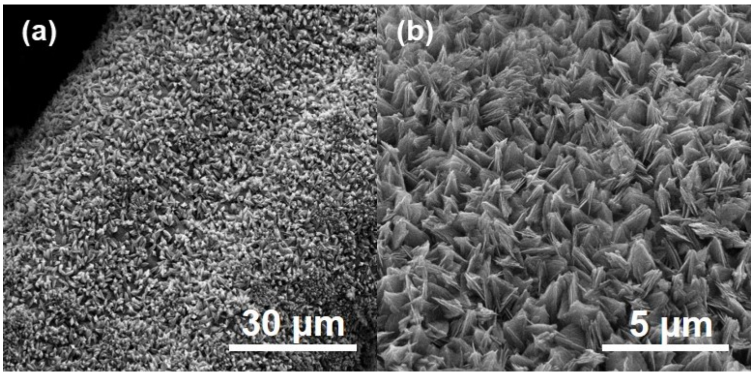

Figure S2 SEM image of FeCoNi-NS with different magnification.

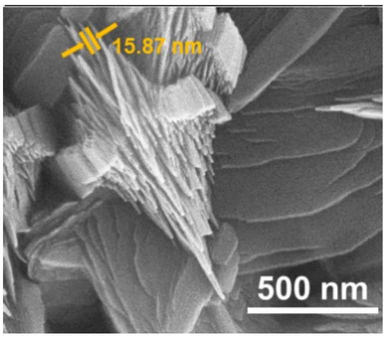

Figure S3. SEM image of FeCoNi-NS.

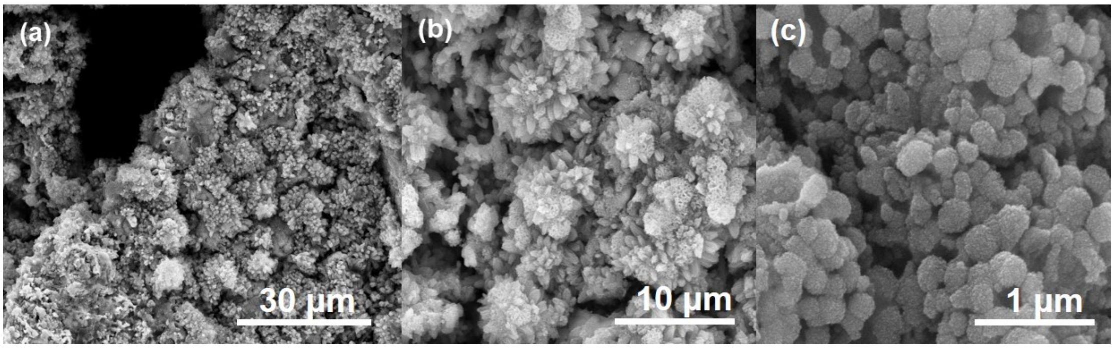

Figure S4. SEM image of FeCoNi-NS-ACVs with different magnification.

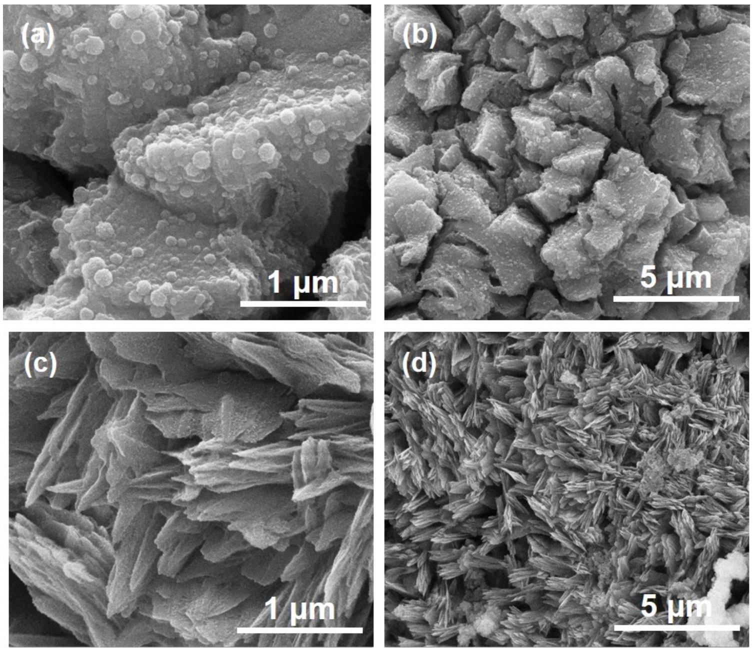

Figure S5 SEM images of FeNi-NS-ACVs (a, b) and FeCo-NS-ACVs (c, d).

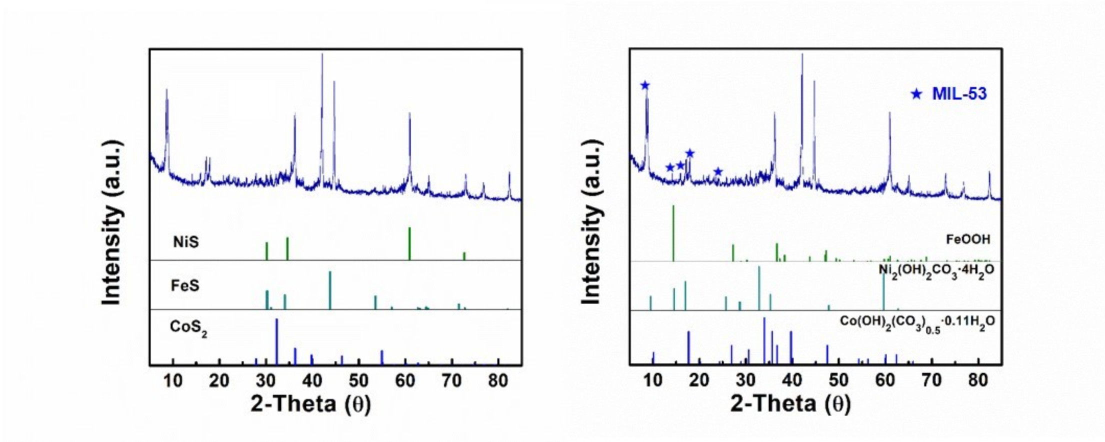

Figure S6. XRD partten of the designed FeCoNi-NS-ACVs.

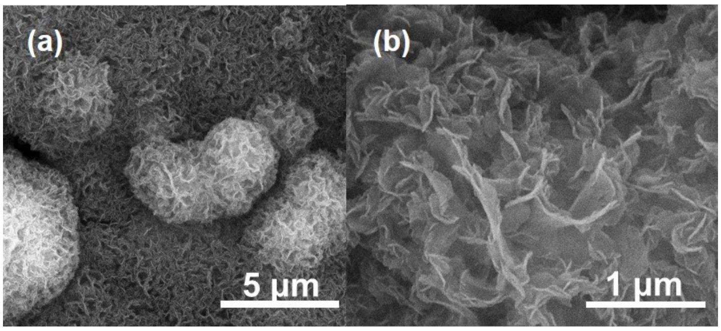

Figure S7. SEM image of FeCoNi-S with different magnification.

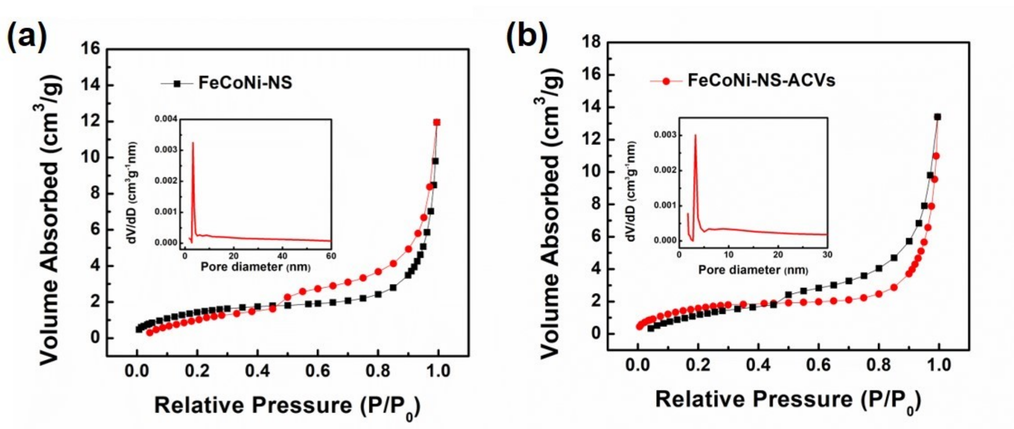

Figure S8 N_2 sorption isotherms and pore size distribution curves of FeCoNi-NS (a) and FeCoNi-NS-ACVs (b).

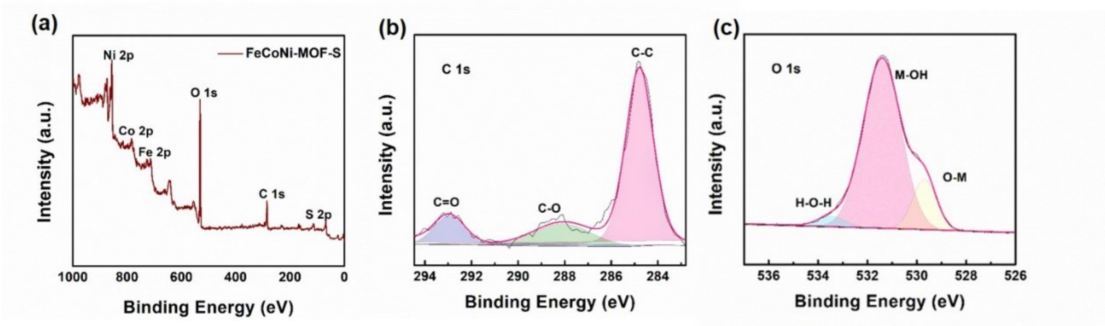

Figure S9. XPS survey spectrum (a) high-resolution of C 1s (b) and O 1s (c) of the designed FeCoNi-NS-ACVs.

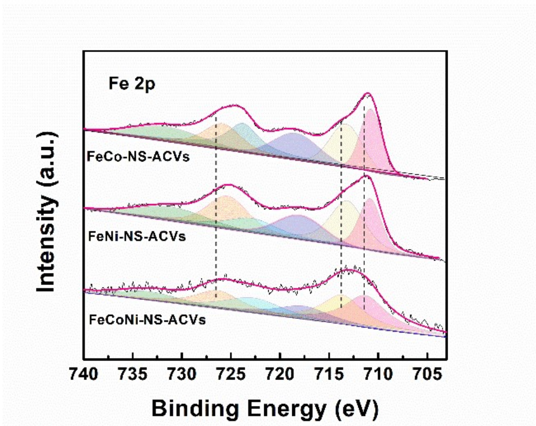

Figure S10. High-resolution Fe 2p XPS spectra of FeCo-NS-ACVs, FeNi-NS-ACVs and FeCoNi-NS-ACVs.

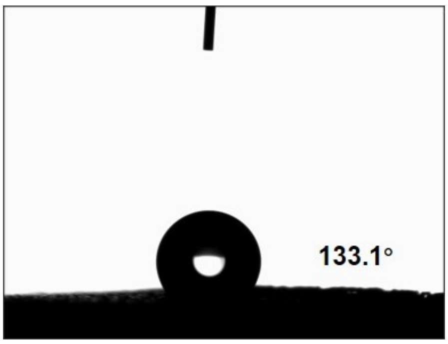

Figure S11. Contact angle measurement of IF.

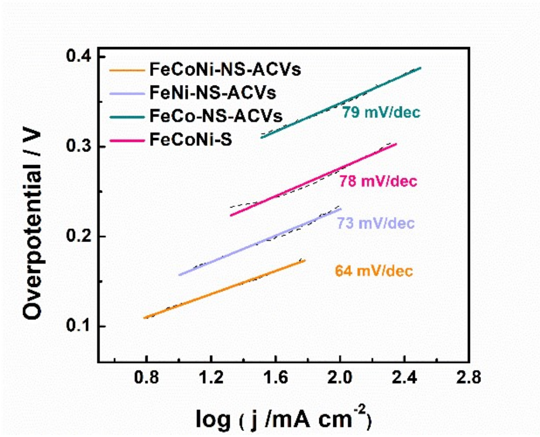

Figure S12. Tafel slopes of obtained catalysts in 1M KOH.

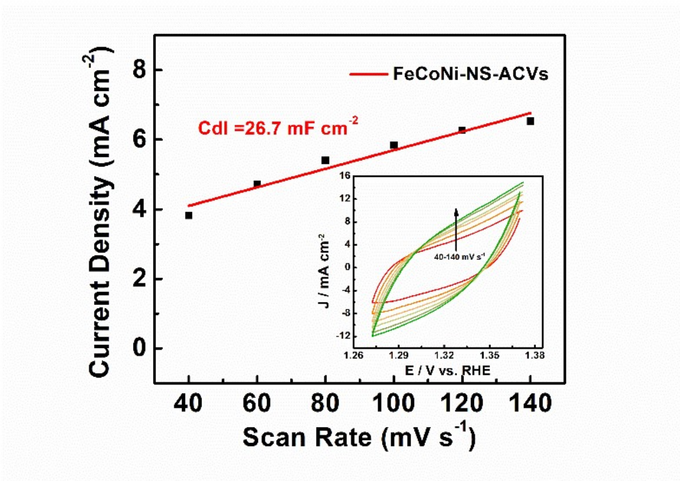

Figure S13 Linear fitting of scan rates with capacitive current densities at 0.25 V (vs SCE) under different scan rate (inset is CV curves of FeCoNi-NS-ACVs under different scan rates).

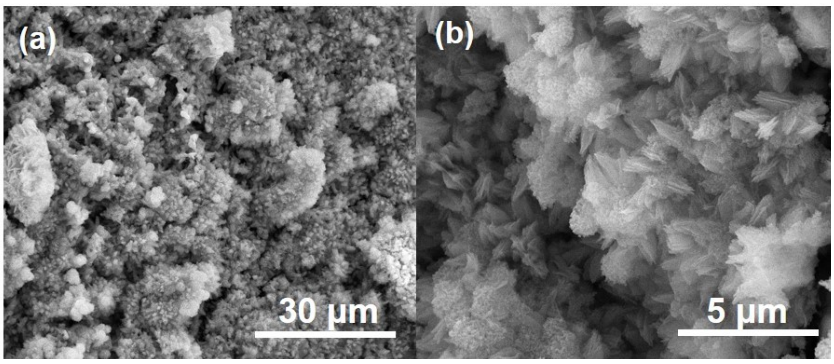

Figure S14 SEM images of FeCoNi-NS-ACVs after stability test of OER with different magnifications.

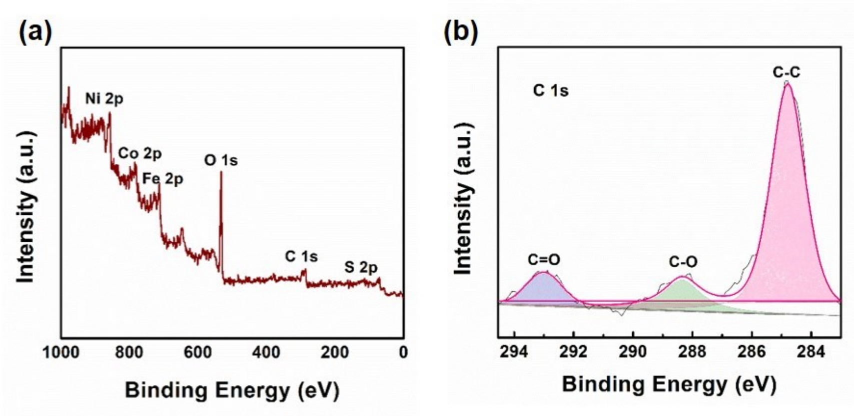

Figure S15 XPS survey spectrum (a) and high-resolution of C 1s (b) of the designed FeCoNi-NS-ACVs after long-time stability test.

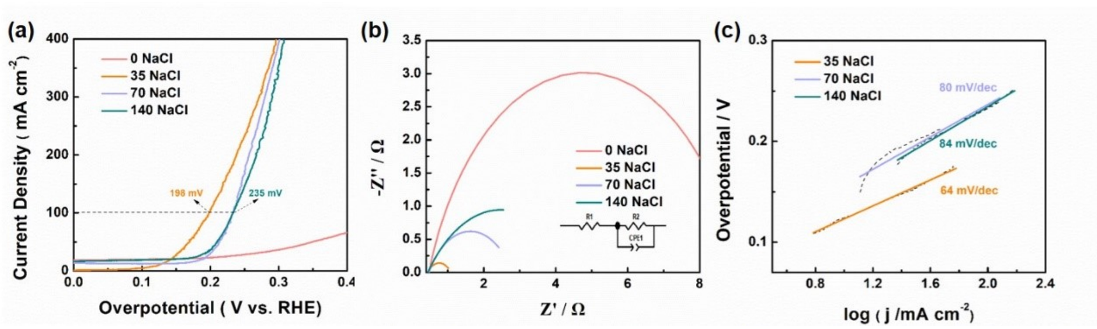

Figure S16 Electrochemical measurements: (a) OER polarization curves, (b) Nyquist plots, (c) Tafel slopes of obtained catalysts in 1M KOH.

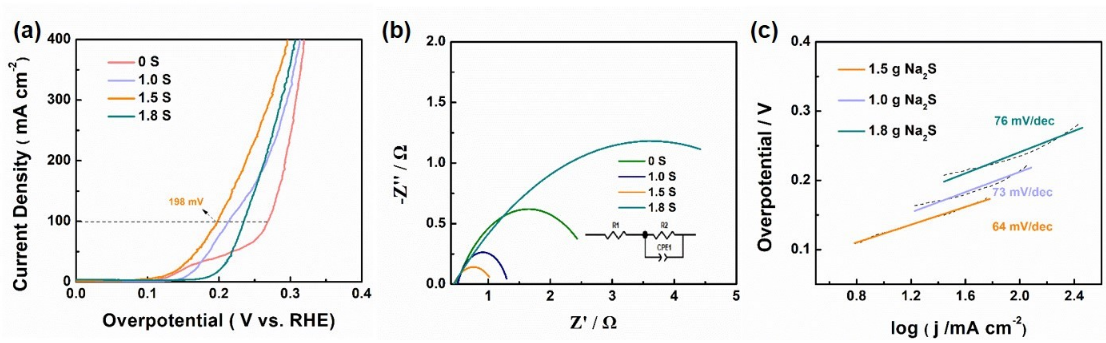

Figure S17 Electrochemical measurements: (a) OER polarization curves, (b) Nyquist plots, (c) Tafel slopes of obtained catalysts in 1M KOH.

Table S1 Element content of Fe, Co and Ni in 1M KOH containing Na_2S determined using ICP.

State of Na_2S solution	Element	the element content of the solution（mg/L)	Diluted multiples	Sample element content（mg/L)
initial	Fe	< 0.02	10	< 0.02
final	Fe	0.37	10	3.7
initial	Ni	< 0.02	10	< 0.02
final	Ni	0.15	10	1.46
initial	Co	< 0.02	10	< 0.02
final	Co	< 0.02	10	< 0.02
initial	S	8.35	1000	8354.02
final	S	8.28	1000	8279.81

Table S2 Comparison of the OER activity and Tafel slope between FeCoNi-NS-ACVs and other electrocatalysts.

Catalysts	Electrolyte	Overpotential (mV)	Tafel slope (mV dec^{-1})	Reference
FeCoNi-NS-ACVs	1 M KOH	125	64.0	This work
$NiCo_2S_4$/Fe-2	1 M KOH	200	71.0	*Nano Energy* 2020, 78, 105230
Ru-RuPx-CoxP	1 M KOH	291	85.4	*Nano Energy* 2018, 53, 270-276
NiCo-P/NF	1 M KOH	280	73.0	*Nano Lett.* 2016, 16, 7718−7725
Fe, Mn-Ni_3S_2/NF	1 M KOH	216	63.3	*J. Mater. Chem. A* 2017, 5, 14828–14837.

CoO/Co	1 M KOH	350	97.6	*ACS Energy Lett.* 2017, 2, 1208−1213
NiSe/NF	1 M KOH	270	64.0	*Angew. Chem. Int. Ed.* 2015, 54, 9351–9355.
S:Co_2P@Ni foam	1 M KOH	280	71.0	*Chem. Mater.* 2018, 30, 8861–8870.
NiCoP NR@NS	1 M KOH	268	75.0	*ACS Appl. Mat. Inter.* 2018, 10, 41237–41245.
Fe-Mn-O NSs/CC	1 M KOH	273	63.9	*Adv. Funct. Mater.* 2018, 28, 1802463.

Table S3 Comparison of electrocatalytic performances for OER.

Catalysts	**Electrolyte**	**Overpotential (mV) at 10 mA cm^{-2}**	**Reference**
FeCoNi-NS-ACVs	1 M KOH	125	This work
3D-V-Ni_3S_2-NiFe	1 M KOH	209	*J Mater Chem A 2019, 7, 18118-18125*
Ni_3S_2 nanorod@NiFe LDH nanofilms	1 M KOH	245	*J Mater Chem A 2018, 6, 10253-10263*
FeP/Ni_2P	1 M KOH	154	*Nat. Commun. 2018, 9, 2551*
MoS_2/Co_9S_8/Ni_3S_2/Ni	1 M KOH	166	*J. Am. Chem. Soc. 2019, 141, 10417*
MoS_2/Ni_3S_2/Ni	1 M KOH	218	*Angew.Chem. Int. Ed., 2016, 128, 6814*
NiFe LDH-NS@DG	1 M KOH	210	*Adv. Mater. 2017, 29, 1700017*
(Fe_xNi_{1-x})2P	1 M KOH	156	*Nano Energy 2017, 38, 553*
$NiMoO_x$/NiMoS	1 M KOH	186	*Nat. Commun. 2020, 11, 5462*
FeCoNi-HNTAs	1 M KOH	184	*Nat. Commun. 2018, 9, 2452*
V-CoP@a-CeO_2	1 M KOH	225	*Adv. Funct. Mater. 2020, 30, 1909618*

$Ni_3N/CMFs/Ni_3N$	1 M KOH	273	*J. Mater. Chem. A, 2017, 5, 9377*
NiCoP	1 M KOH	242	*ACS Catal.* 2017, *7*, 4131
Co_9S_8	1 M KOH	302	*Adv. Funct. Mater., 2017, 1606585*
Ni–Fe NP	1 M KOH	200	*Nat. Commun. 2019, 10*
$FeCo\text{-}NiSe_2$	1 M KOH	251	*Adv. Mater. 2018, 30, 1802121*
$FeNi_3N/Ni$	1 M KOH	202	*Chem. Mater., 2016, 28, 6934*
Ti_3C_2@mNiCoP	1 M KOH	237	*ACS Appl. Mater. Interf. 2020, 12, 18570-18577*
$N_2\text{-}CoS_2$-400	1 M KOH	240	*ACS Catal. 2017, 7, 4214*

Table S4 Comparison of the Rct (Ω) values of Fe-S, FeCoNi-S, FeNi-NS-ACVs, FeCo-NS-ACVs and FeCoNi- NS-ACVs in 1M KOH.

Catalysts	Rct / Ω
	1.0 M KOH
Fe-S	60.17
FeCoNi-S	1.29
FeNi- NS-ACVs	0.56
FeCo- NS-ACVs	15.7
FeCoNi- NS-ACVs	0.39

Table S5 Comparison of the electrocatalytic performances overall water splitting

Catalysts	**Electrolyte**	**Cell Voltage (V) at 10 mA**	**Reference**

		cm^{-2}	
FeCoNi-NS-ACVs	1 M KOH	1.37	This work
(NixFe1-x)2P@PC/P	1 M KOH	1.45	*Adv. Funct. Mater.* 2021, 2010912
Ni/γ-Fe_2O_3	1 M KOH	1.47	*Nat. Commun.* 2019, *10*, 5599
IrNi-$FeNi_3$/NF	1 M KOH	1.47	*Appl. Catal. B* 2021, 286, 119881
RuCu NSs/C	1 M KOH	1.49	*Angew. Chem. Int. Ed.* 2019, 58, 13983 – 13988
NiVIr LDH	1 M KOH	1.49	*ACS Energy Lett.* 2019, 4, 1823−1829
Fe_2Co-MOF/NF	1 M KOH	1.49	*J. Mater. Chem. A*, 2021,9, 11415-11426
FeNiS/Ni	1 M KOH	1.51	*Adv. Energy Mater.* 2020, 2001963
Ir@S–C/rGO	1 M KOH	1.51	*J. Mater. Chem. A,* 2021, 9, 4176–4183
Fe-CoP/$Ni(OH)_2$	1 M KOH	1.52	*Adv. Funct. Mater.* 2021, 2101578
CoNC@Co_2N/CPs	1 M KOH	1.52	*Adv. Energy Mater.* 2020, *10*, 2002214
NiFe-LDH@NiCu	1 M KOH	1.53	*Adv. Mater.* 2019, *31*, 1806769
FeCoNi/CC	1 M KOH	1.55	*Adv. Energy Mater.* 2019, *9*, 1901312
NiCo-P/NF	1 M KOH	1.58	*Nano Lett.* 2016, 16, 7718−7725
CoP-InNC	1 M KOH	1.58	Adv. Sci. 2020, *7*, 1903195
$Ni_{0.75}Fe_{0.25}$-N, P, S/C	1 M KOH	1.60	*J. Power Sources* 2018, 401, 312–321
Cu_3N	1 M KOH	1.60	*ACS Energy Lett.* 2019, *4*, 747
O-CoP	1 M KOH	1.60	*Adv. Funct. Mater.* 2020, *30*, 1905252
$NiCo_2O_4$	1M NaOH	1.65	*Angew. Chem. Int. Ed.* 2016, 55, 6290 –6294

NiCoFeB	1 M KOH	1.75	*Small* 2019, 15, 1804212