Supporting Information

Electrochemical behavior of rapid-synthesized amorphous aerogel 3d-VIIIB-metal aerogels as bifunctional electrocatalysts for water splitting

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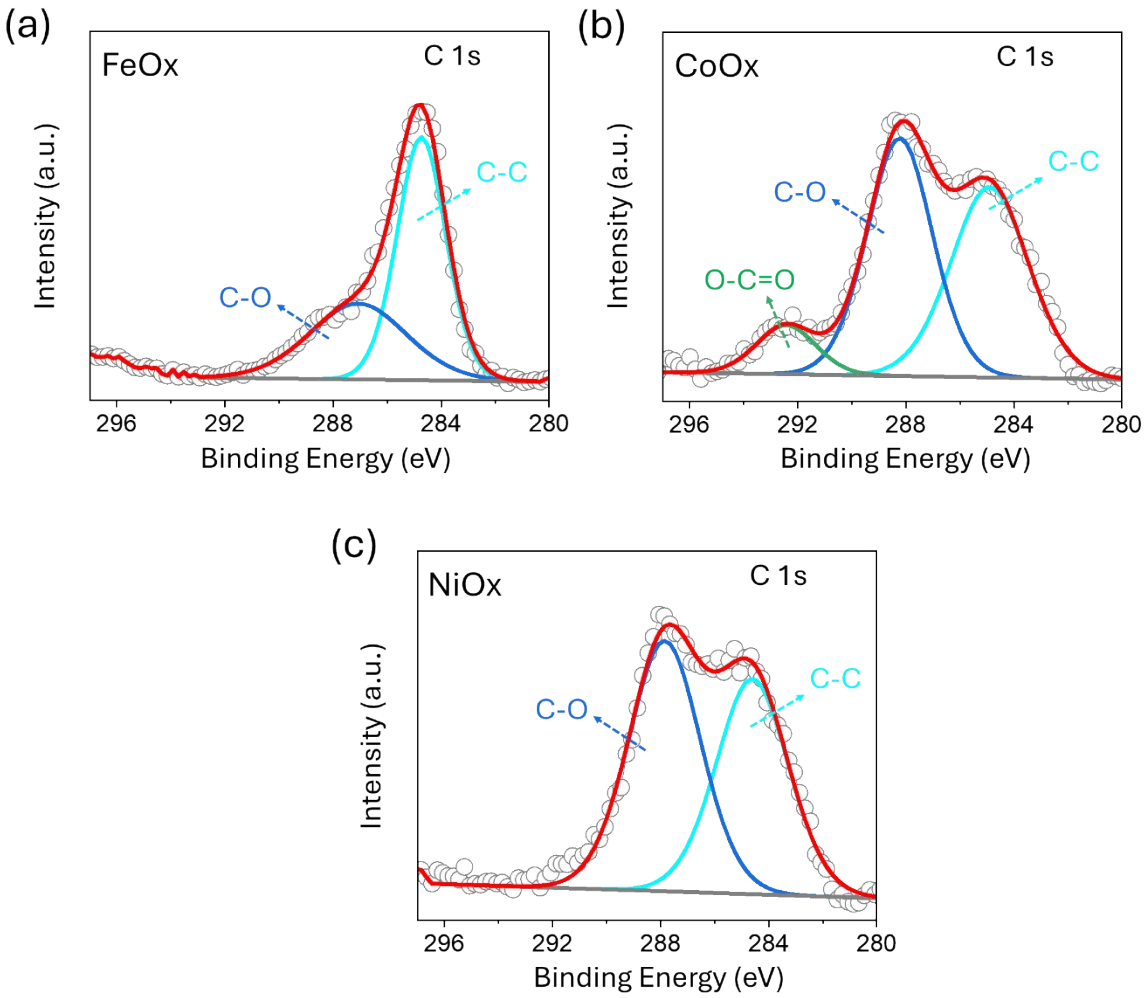

Figure S1. XPS C1s spectra of as-prepared catalysts.

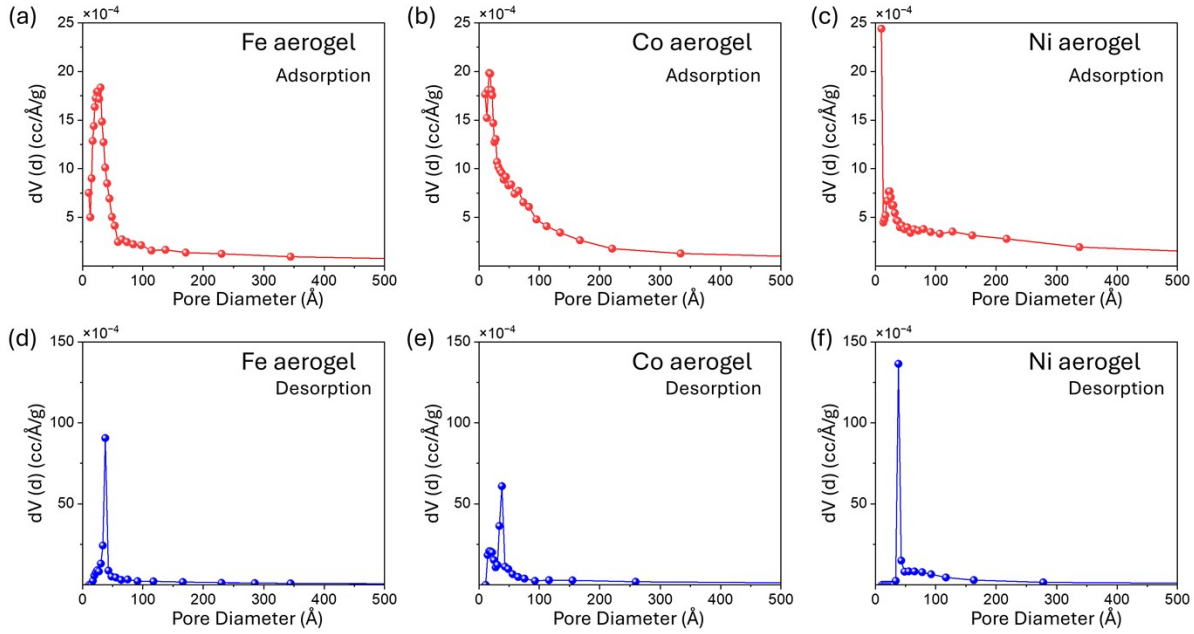

Figure S2. Adsorption-desorption pore-size distribution curves of as-prepared catalysts.

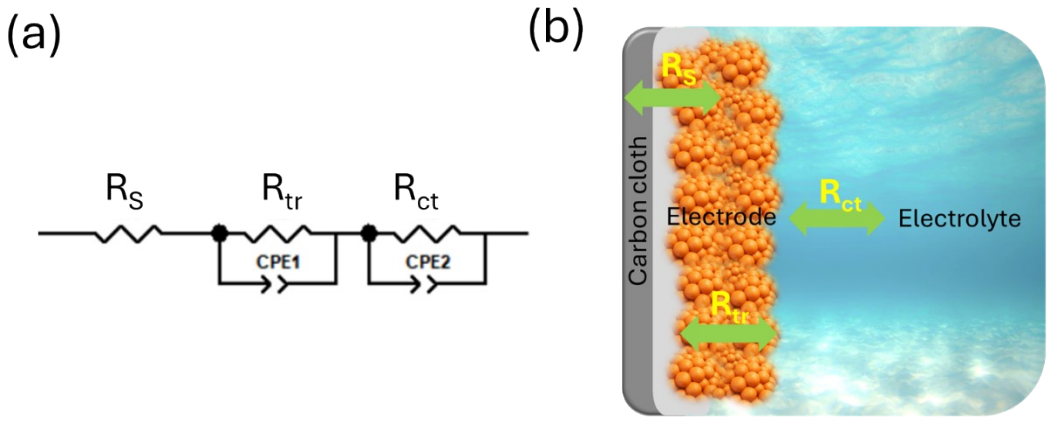

Figure S3. Schematic illustration of the fitted values of R_S, R_{tr}, and R_{ct} corresponding to series resistance, catalyst/catalyst interfacial resistance, and charge transfer resistance at the electrode/electrolyte interface, respectively.

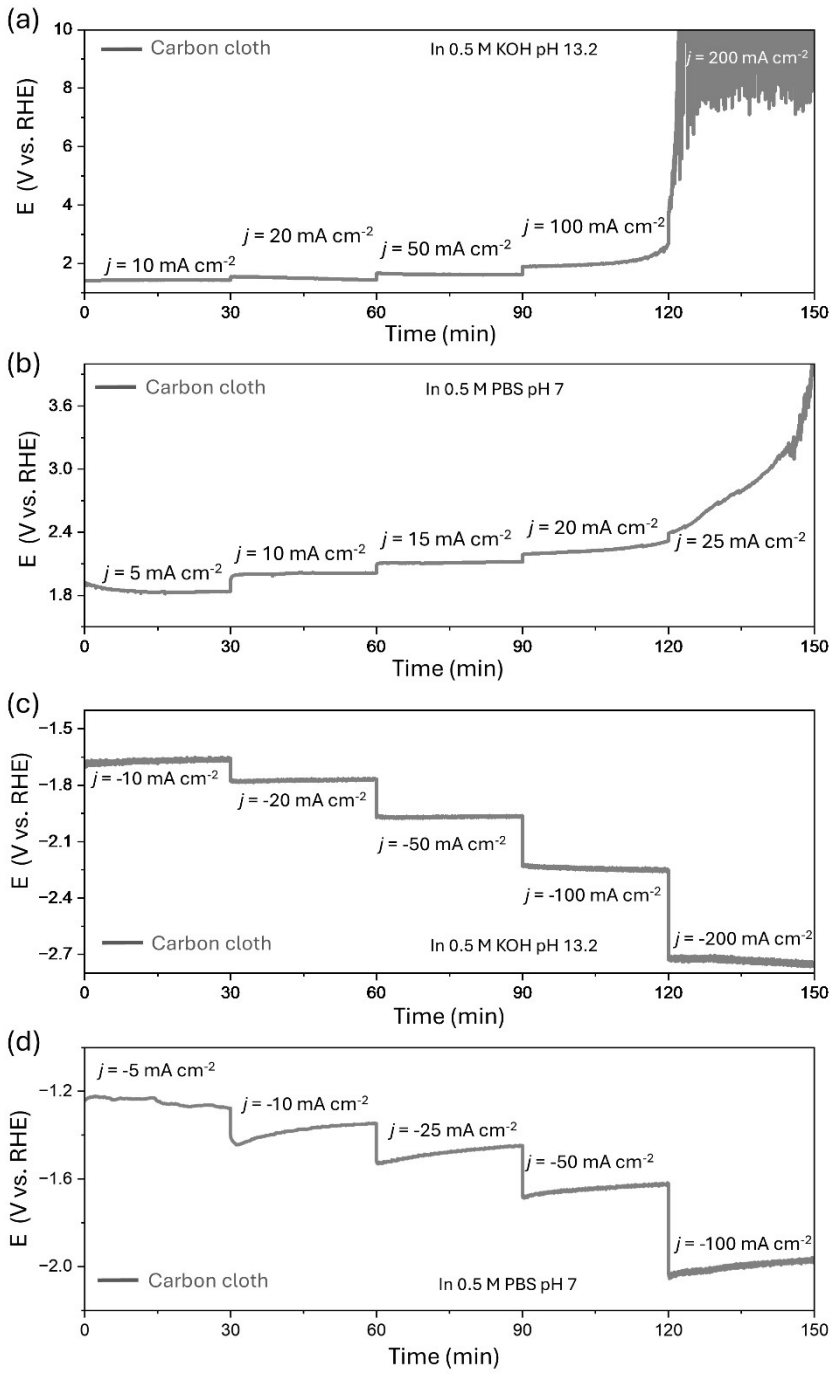

Figure S4. Short-term chronopotentiograms collected at different current densities of carbon cloth in (a) OER + 0.5 M KOH pH = 13.2, (b) OER + 0.5 M PBS pH = 7, (c) HER + 0.5 M KOH pH = 13.2, and (d) HER + 0.5 M PBS pH = 7.

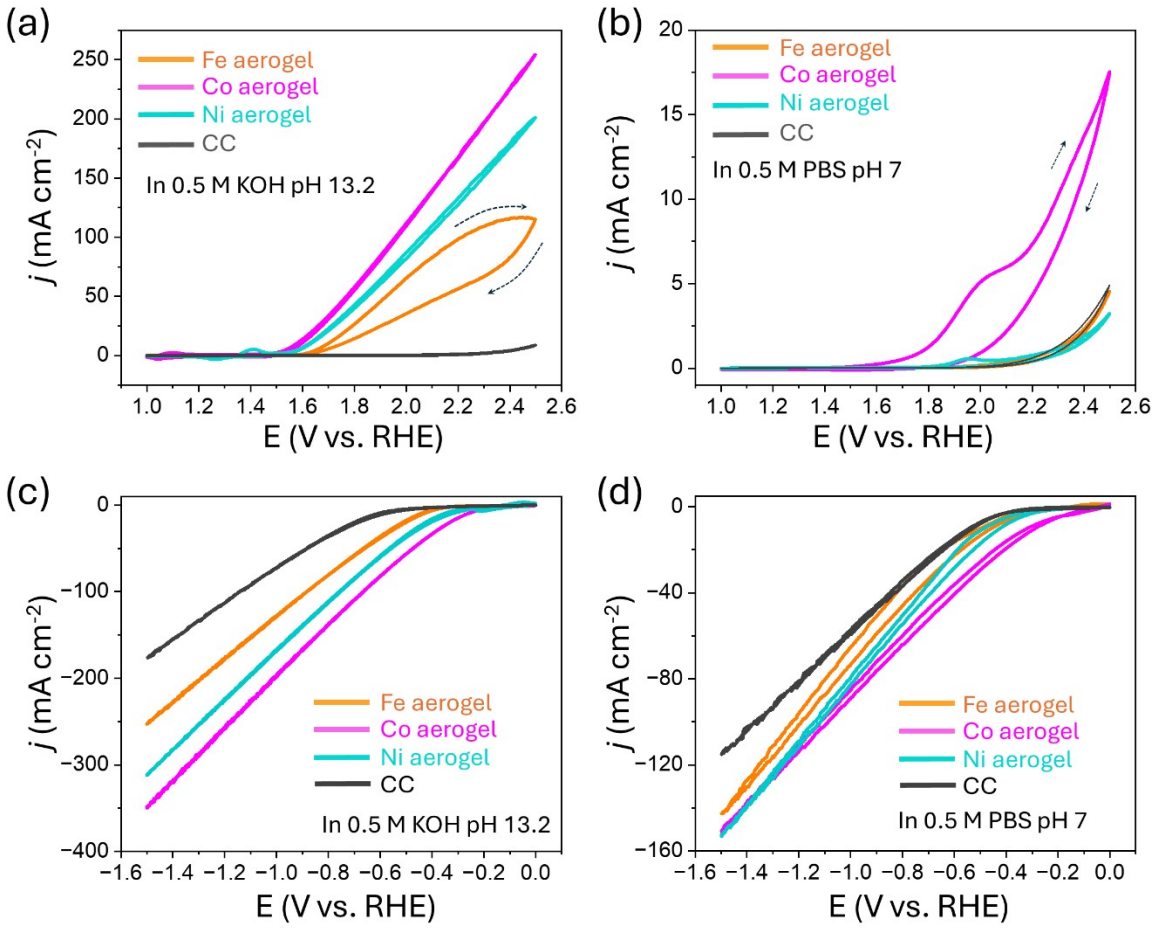

Figure S5. Cyclic voltammograms (v = 5 mV s^{-1}; 3rd scan) without iR_S correction applied of as-prepared catalysts of OER (a) in 0.5 M KOH (pH = 13.2) and (b) in 0.5 M PBS (pH = 7) and of HER (c) in 0.5 M KOH (pH = 13.2) and (d) in 0.5 M PBS (pH = 7).

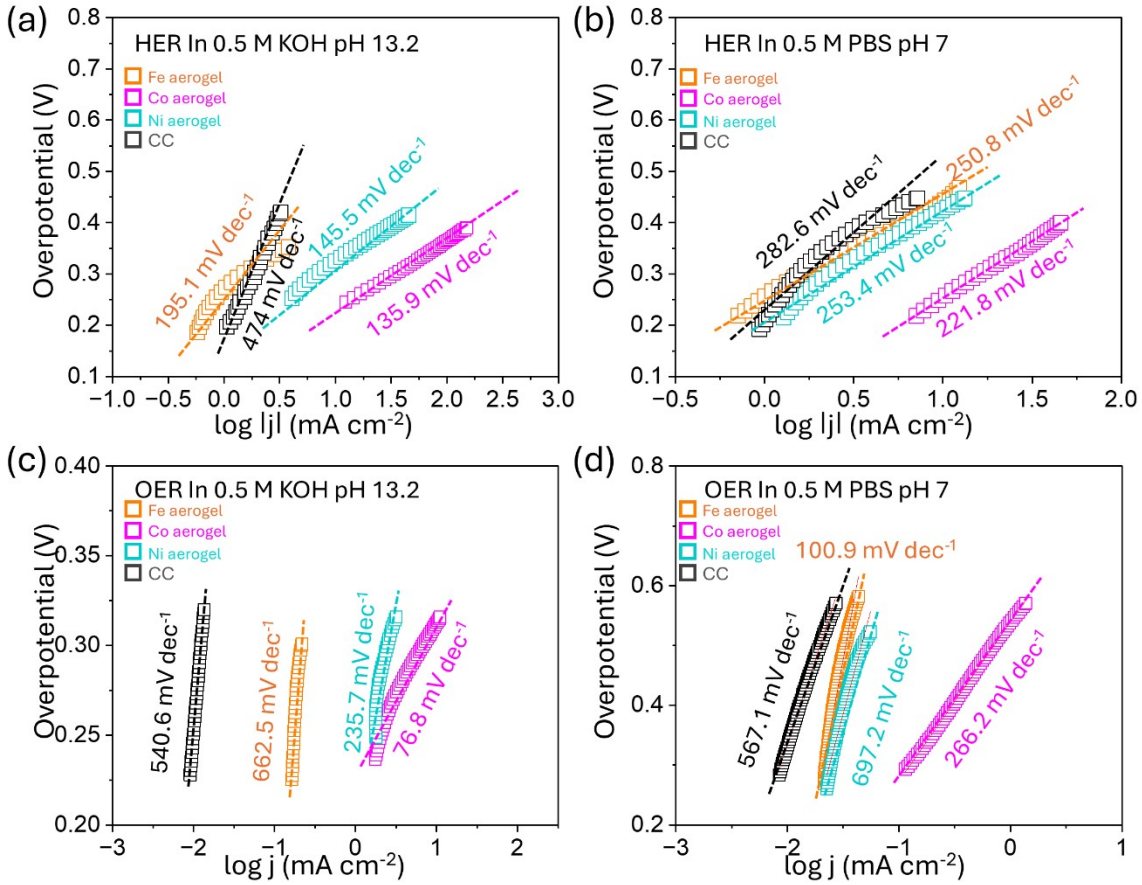

Figure S6. Tafel slopes of as-prepared catalysts of HER (a) in 0.5 M KOH (pH = 13.2) and (b) in 0.5 M PBS (pH = 7) and of OER (c) in 0.5 M KOH (pH = 13.2) and (d) in 0.5 M PBS (pH = 7).

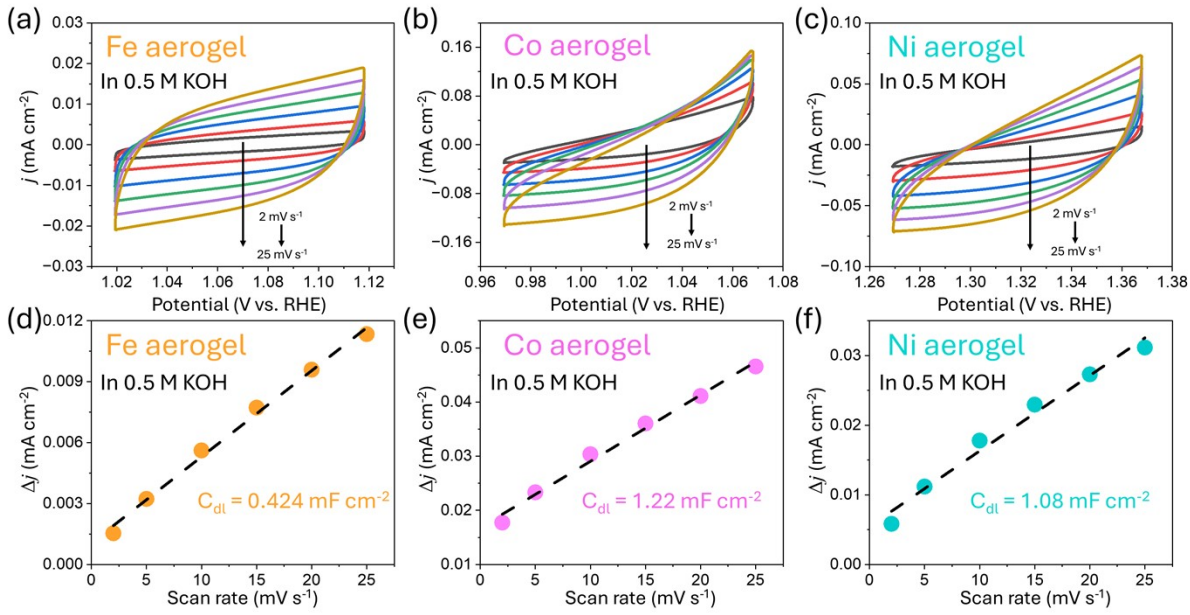

Figure S7. CV curves of as-prepared catalysts recorded at different scan rates (2; 5; 10; 15; 20; and 25 mV s^{-1}) in the non-faradaic potential range for (a) Fe aerogel; (b) Co aerogel; and (c) Ni aerogel. Corresponding double-layer capacitance for (d) Fe aerogel; (e) Co aerogel; and (f) Ni aerogel. Note: Electrolyte is 0.5 M KOH (pH = 13.2). Potentials are not corrected for ohmic losses. Currents are normalized to the geometric surface area of the electrodes (1 cm^2).

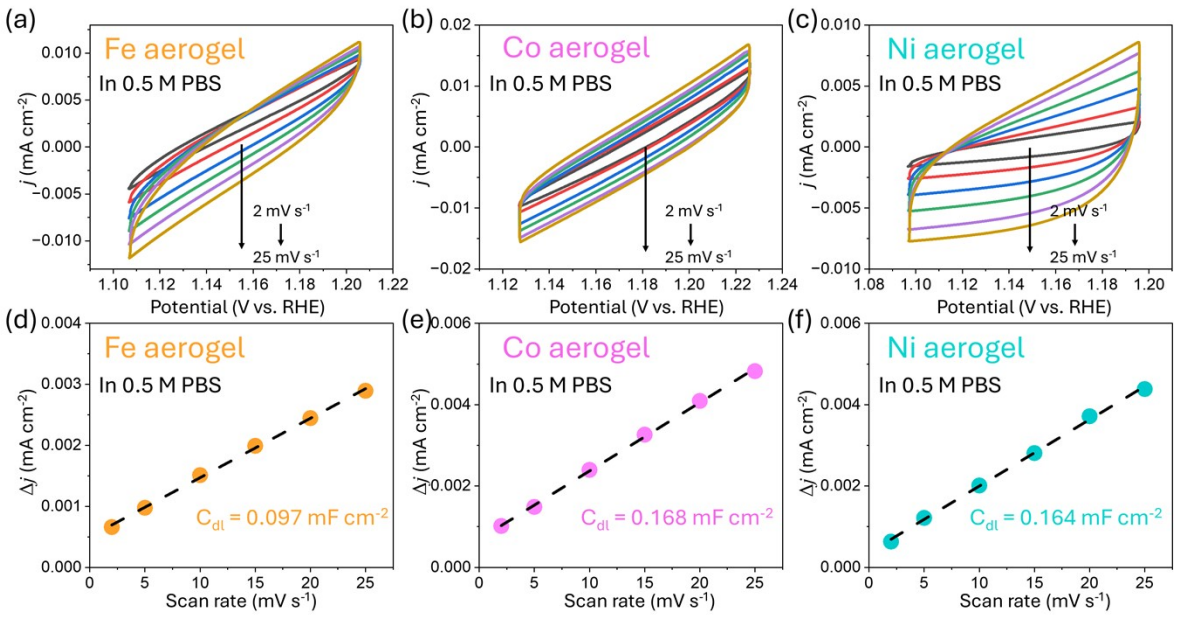

Figure S8. CV curves of as-prepared catalysts recorded at different scan rates (2; 5; 10; 15; 20; and 25 mV s^{-1}) in the non-faradaic potential range for (a) Fe aerogel; (b) Co aerogel; and (c) Ni aerogel. Corresponding double-layer capacitance for (d) Fe aerogel; (e) Co aerogel; and (f) Ni aerogel. Note: Electrolyte is 0.5 M PBS (pH = 7). Potentials are not corrected for ohmic losses. Currents are normalized to the geometric surface area of the electrodes (1 cm^2).

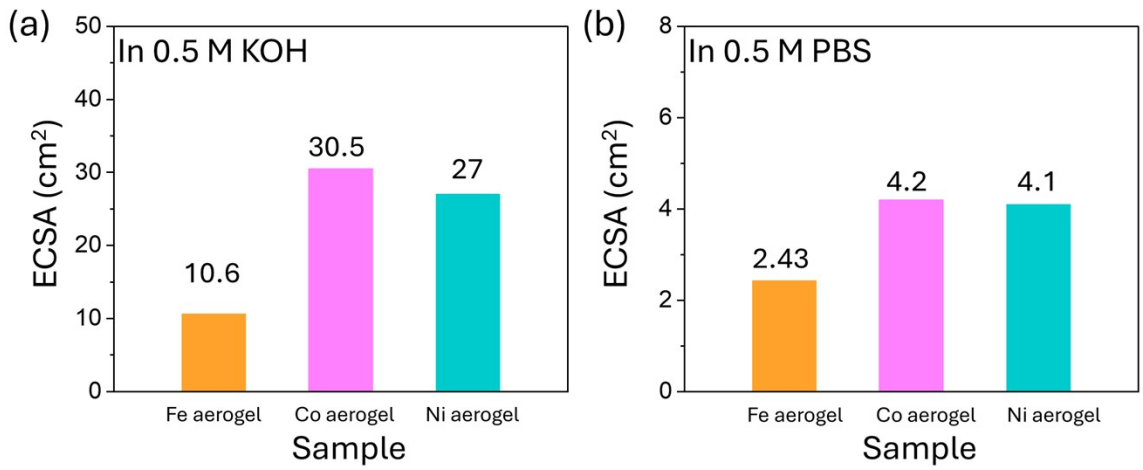

Figure S9. Electrochemical active surface area of as prepared catalysts in (a) 0.5 M KOH and (b) 0.5 M PBS.

Table S1. BET adsorption-desorption parameters of as-prepared catalysts.

Sample	**Adsorption**			**Desorption**		
	Surface area (m^2/g)	Pore volume (cc/g)	Pore diameter (Å)	Surface area (m^2/g)	Pore volume (cc/g)	Pore diameter (Å)
Fe aerogel	94.128	0.128	30.001	102.341	0.131	37.730
Co aerogel	134.461	0.176	16.945	141.002	0.178	38.065
Ni aerogel	103.382	0.184	9.993	113.964	0.197	38.193

Table S2. The fitted parameters of Nyquist plot of the as-prepared catalysts in OER with alkaline electrolyte.

OER	In 0.5 M KOH (pH = 13.2) @1.52 V vs. RHE			
	Fe aerogel	Co aerogel	Ni aerogel	Carbon cloth
R_S	3.4	3.45	3.53	4.9
R_{tr}	201.8	2	9	
CPE1-T	0.0015	0.00058	0.0019	
CPE1-P	0.94	0.7	0.65	
R_{ct}	7855	48	189	
CPE2-T	0.001644	0.000067	0.00084	
CPE2-P	0.9	0.961	0.934	

Table S3. The fitted parameters of Nyquist plot of the as-prepared catalysts in OER with neutral electrolyte.

OER	In 0.5 M PBS (pH = 7) @1.31 V vs. RHE			
	Fe aerogel	Co aerogel	Ni aerogel	Carbon cloth
R_S	6.11	6.53	7.03	6.95
R_{tr}	190	140	240	
CPE1-T	0.0002	0.00029	0.00091	
CPE1-P	0.89	0.5	0.72	
R_{ct}	8000	630	1700	
CPE2-T	0.00024549	0.0002549	0.00018	
CPE2-P	0.88515	0.86	0.83	

Table S4. The fitted parameters of Nyquist plot of the as-prepared catalysts in HER with alkaline electrolyte.

HER	In 0.5 M KOH (pH = 13.2) @-0.3 V vs. RHE			
	Fe aerogel	Co aerogel	Ni aerogel	Carbon cloth
R_S	3.38	3.02	2.9	4.24
R_{tr}	0.25	0.75	0.85	
CPE1-T	0.007	0.005	0.00085	
CPE1-P	0.67	0.55	0.78	
R_{ct}	28.55	2.89	23.15	
CPE2-T	0.018	0.00037	0.014	
CPE2-P	0.8524	0.96524	0.914	

Table S5. The fitted parameters of Nyquist plot of the as-prepared catalysts in HER with neutral electrolyte.

HER	In 0.5 M PBS (pH = 7) @-0.5 V vs. RHE			
	Fe aerogel	Co aerogel	Ni aerogel	Carbon cloth
R_S	5.78	6.23	5.94	7.3
R_{tr}	0.7	0.5	0.55	
CPE1-T	0.01	0.001	0.0005	
CPE1-P	0.8	0.75	0.827	
R_{ct}	7.85	3.9	3.97	
CPE2-T	0.01	0.014034	0.00025	
CPE2-P	0.8694	0.552	0.92721	

Table S6. Performance comparison of different reported Co-based catalysts for OER in alkaline electrolyte.

No.	Catalyst	Electrolyte	Tafel slope (mV dec^{-1})	OER Overpotential at 100 mA cm^{-2} (mV)	References
	Co aerogel	0.5 M KOH	76.8	390	This work
	$FePCoMo_2S_4$	1.0 M KOH	96	390	Zhou et al. 2026[1]
	VCoSe	1.0 M KOH	51.6	420	Sahu et al. 2025[2]
	Co-h-MoO_3	1.0 M KOH	102.02	359	Wang et at. 2025[3]
	Co-a-MoO_3	1.0 M KOH	112.38	463	Wang et at. 2025[3]
	Co/GC	1.0 M KOH	96	460	Vijayarangan et al. 2025[4]
	$CuS/Co_3O_4/C_3N_4$	1.0 M KOH	79	410	Ahmed et al.[5]
	CuS/Co_3O_4	1.0 M KOH	101	530	Ahmed et al.[5]
	$FeNiCo_2S_4$	1.0 M KOH	85	330	Singla et al. 2025[6]
	$NiCo_2S_4$	1.0 M KOH	155	250	Singla et al. 2025[6]
	$CuO/ZnCo_2O_4$	1.0 M KOH	70	510	Bhoj et al. 2025[7]

$ZnCo_2O_4$	1.0 M KOH	85	630	Bhoj et al. 2025[7]
$CuCo_2S_4/CoVOF_2$	1.0 M KOH	189.66	340	Zhang et al. 2025[8]
$CuCo_2S_4/CoF_2$	1.0 M KOH	73.69	350	Zhang et al. 2025[8]
$CuCo_2S_4/CoVOF_1$	1.0 M KOH	84.88	340	Zhang et al. 2025[8]
$CuCo_2S_4$	1.0 M KOH	256.92	385	Zhang et al. 2025[8]

Table S7. Performance comparison of different reported Co-based catalysts for HER in alkaline electrolyte.

No.	Catalyst	Electrolyte	Tafel slope (mV dec^{-1})	HER Overpotential at 100 mA cm^{-2} (mV)	References
	Co aerogel	0.5 M KOH	135.9	364	This work
	$FePCoMo_2S_4$	1.0 M KOH	125	350	Zhou et al. 2026[1]
	$ErPCoMo_2S_4$	1.0 M KOH	118	310	Zhou et al. 2026[1]
	VCoSe	1.0 M KOH	94.49	480	Sahu et al. 2025[2]
	Co-h-MoO_3	1.0 M KOH	116.22	130	Wang et at. 2025[3]
	Co-a-MoO_3	1.0 M KOH	124.62	235	Wang et at. 2025[3]
	CoB	1.0 M KOH	101	270	Sirichandana et al. 2025[9]
	$CuS/Co_3O_4/C_3N_4$	1.0 M KOH	89	490	Ahmed et al.[5]
	CuS/Co_3O_4	1.0 M KOH	118	700	Ahmed et al.[5]
	$FeNiCo_2S_4$	1.0 M KOH	122	300	Singla et al. 2025[6]
	$NiCo_2S_4$	1.0 M KOH	148	340	Singla et al. 2025[6]
	$CuCo_2S_4/CoVOF_2$	1.0 M KOH	189.66	320	Zhang et al. 2025[8]
	$CuCo_2S_4/CoF_2$	1.0 M KOH	176.92	295	Zhang et al. 2025[8]

$CuCo_2S_4/CoVOF_1$	1.0 M KOH	176.77	270	Zhang et al. 2025[8]
$CuCo_2S_4$	1.0 M KOH	248.39	250	Zhang et al. 2025[8]
Co_3O_4@NF	1.0 M KOH	163.9	540	Yang et al. 2025[10]

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