Supplementary Information

Cobalt Modification of Nickel-Iron Hydroxide Electrocatalysts: A Pathway to Enhanced Oxygen Evolution Reaction

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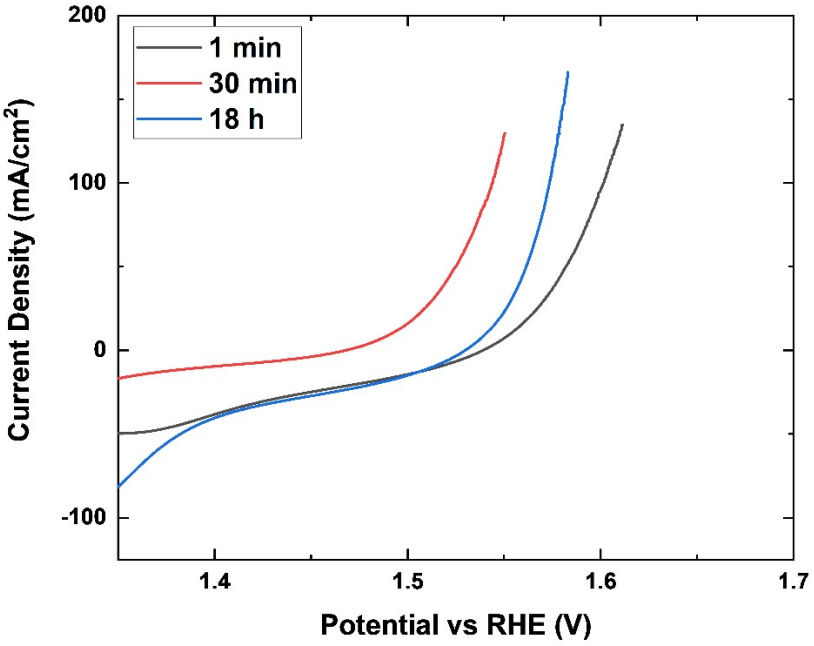

Figure S1: Reverse-scanned LSVs of NiFeCo hydroxide synthesized using 15 mM Co chloride concentration at different corrosion times.

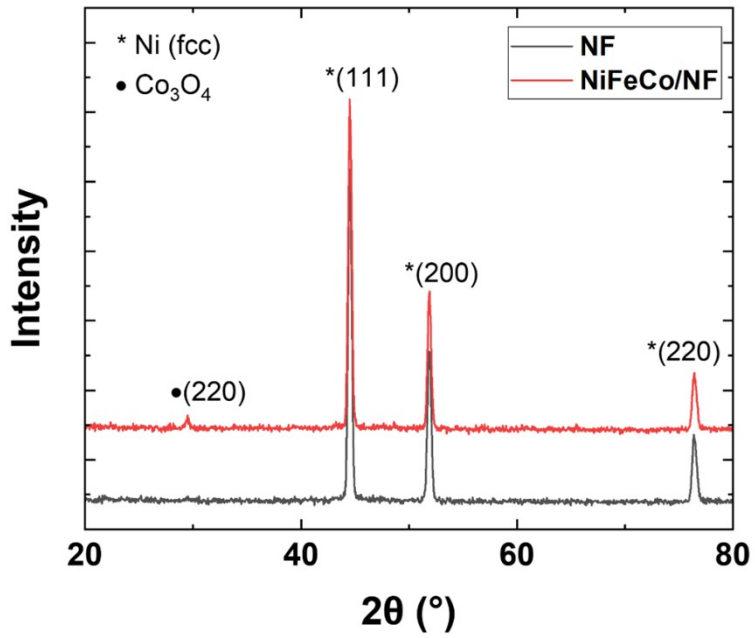

Figure S2: XRD plots of NiFeCo/NF and NF.

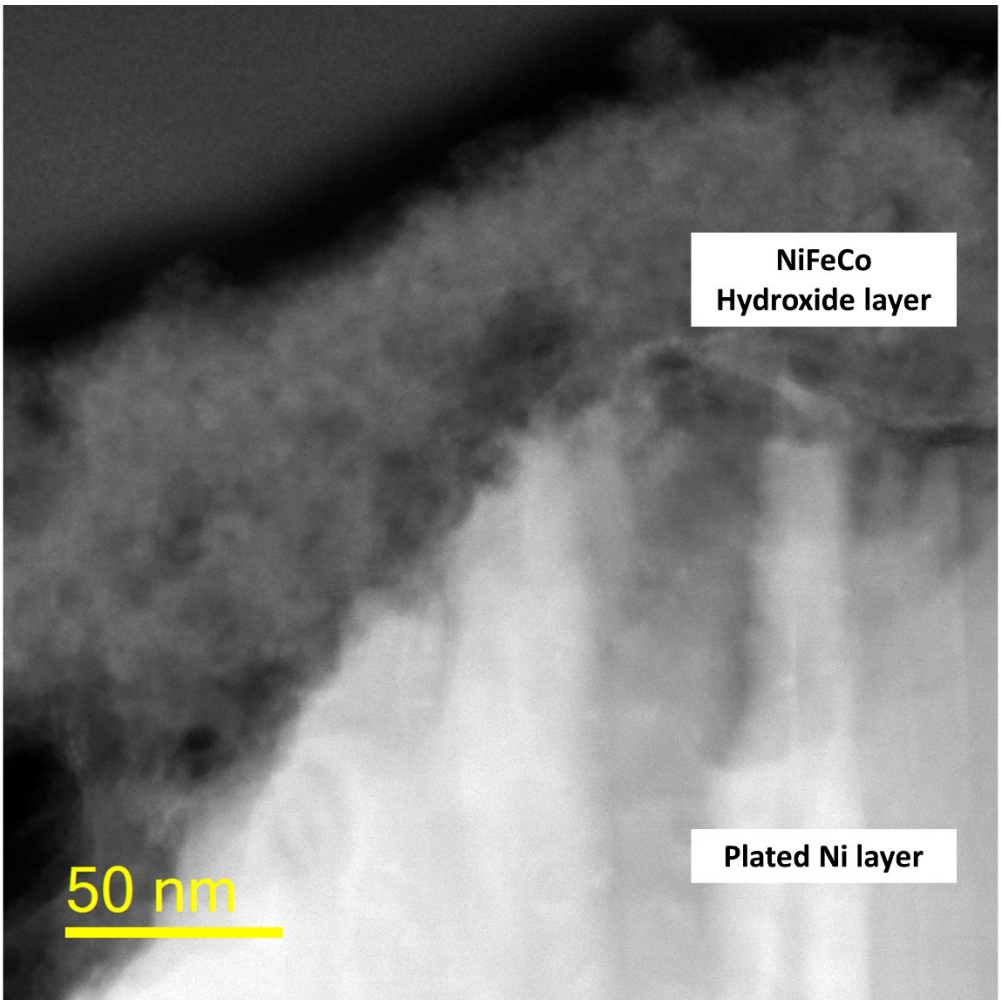

Figure S3: Grayscale of HAADF image of NiFeCo/Si with labelled regions.

Table S1: Peak assignments of NiFeCo/NF XPS Spectra from Figure 2(d-g).

Ni 2p			Fe 2p		
Peak	**Energy (eV)**	**Assignment**	**Peak**	**Energy (eV)**	**Assignment**
1	852.8	Ni^{0} $2p_{3/2}$	1	710.6	Fe^{3+} $2p_{3/2}$
2	855.6	Ni^{2+} $2p_{3/2}$	2	712.5	Fe^{3+} $2p_{3/2,}$ (multiplet)
3	857.2	Ni^{3+} $2p_{3/2}$			
4	861.1	Ni^{2+} $2p_{3/2}$, sat			
5	863.7	Ni^{3+} $2p_{3/2}$, sat			

Co 2p			O 1s		
Peak	**Energy (eV)**	**Assignment**	**Peak**	**Energy (eV)**	**Assignment**
1	781.0	Co^{2+} $2p_{3/2}$	1	529.9	M-O bonds
2	782.4	Co^{3+} $2p_{3/2}$	2	531.6	M-OH bonds
3	786.8	Co^{2+} $2p_{3/2}$, sat	3	532.9	Adsorbates
4	789.8	Co^{3+} $2p_{3/2}$			

Table S2: Comparison of NiFeCo/NF with other NiFeCo electrocatalysts and Ni-based electrocatalysts synthesized using solution corrosion method.

Sample	Synthesis Method	OER overpotential at	Ref

		10 mA/cm^2 (mV)	
NiFeCo/NF	**Solution corrosion**	**195**	**Own work**
NiFeCo/Iron foil	Hydrothermal	300	[1]
NiFeCo/NF	CV electrodeposition	207	[2]
NiFeCo/Ti felt	Co-precipitation	249	[3]
NiFeOOH/NiFeS$_x$/NiFe foam	Solution corrosion	227	[4]
NiFe LDH/NF	Solution corrosion	180	[5]
NiFe LDH/NF	Solution corrosion	269	[6]

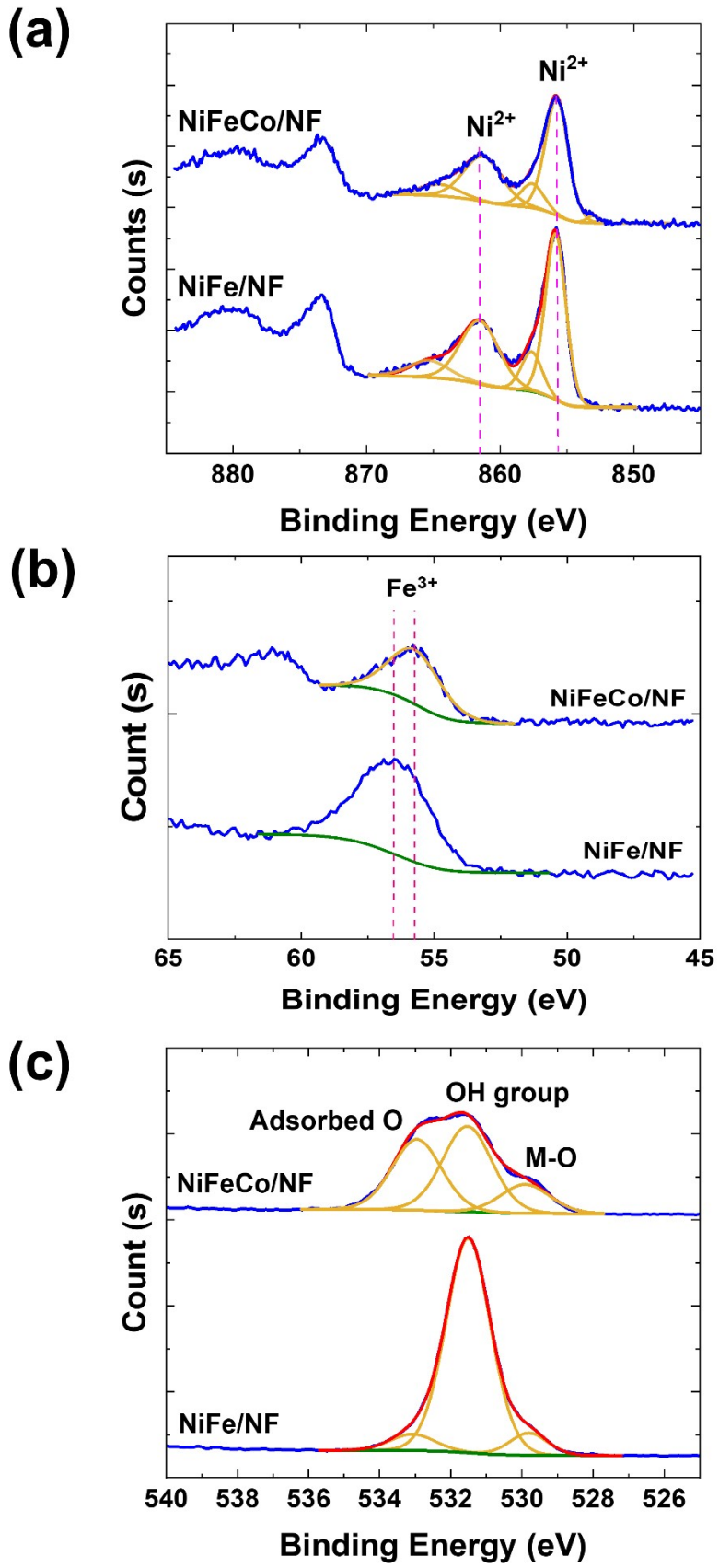

Figure S4: (a) Ni 2p, (b) Fe 3p, and (c) O 1s XPS spectra of NiFe/Si and NiFeCo/Si

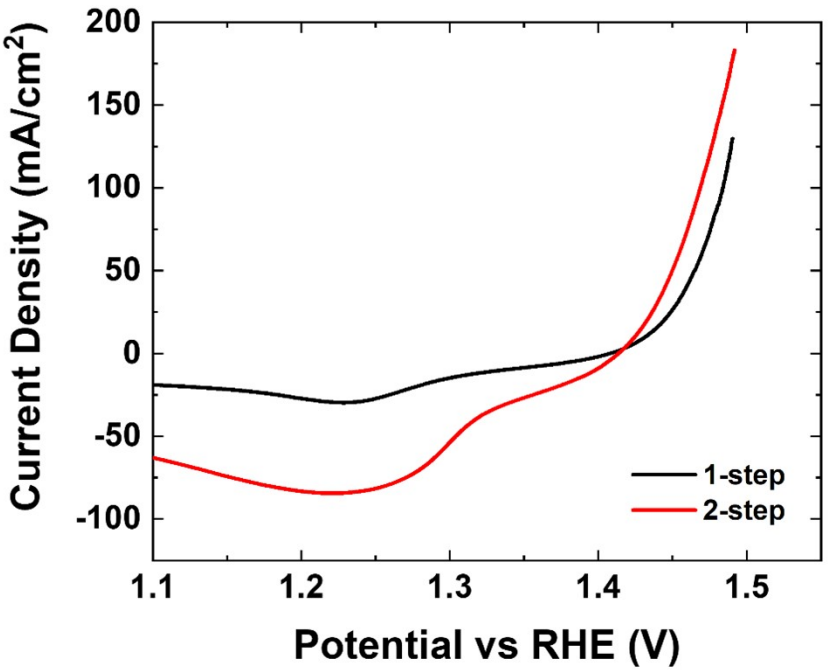

Figure S5: LSVs of NiFeCo/NF synthesized using 1 or 2-step processes.

Table S3: Randles cell equivalent circuit values of NiFe/Si and NiFeCo/Si

Sample	Rs	Rct	CPE (Y_0)	CPE (N)
NiFe/Si	0.965	0.590	0.028	0.651
NiFeCo/Si	0.812	0.269	0.092	0.789

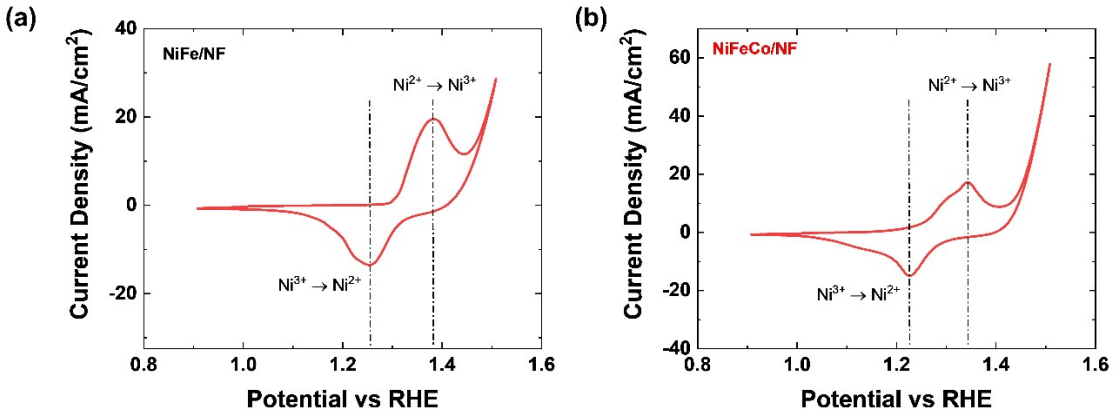

Figure S6: CV of (a) NiFe/NF and (b) NiFeCo/NF with labelled Ni^{2+}-Ni^{3+} redox couple.

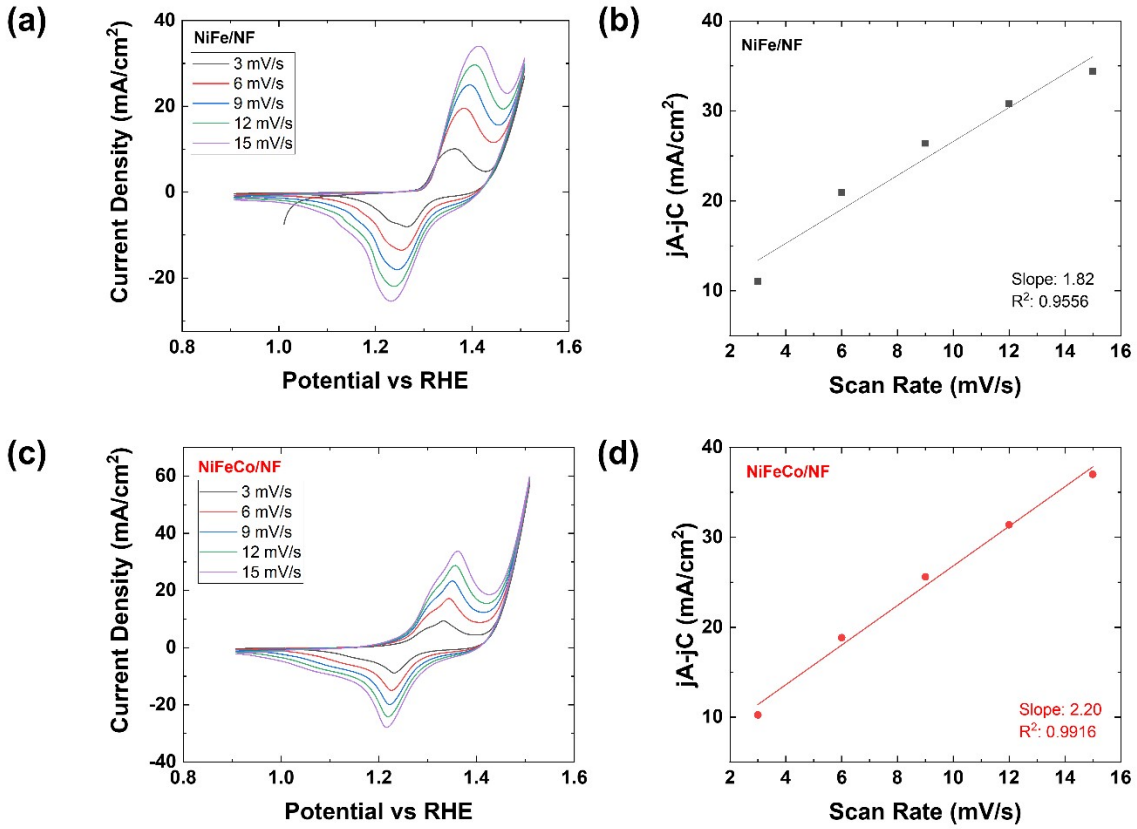

Figure S7: CV graphs at various scan rates (3 – 15 mV/s) and corresponding linear fitted plots of the maximum difference of anodic and cathodic sweep current densities vs scan rate for: (a-b) NiFe/NF and (c-d) NiFeCo/NF.

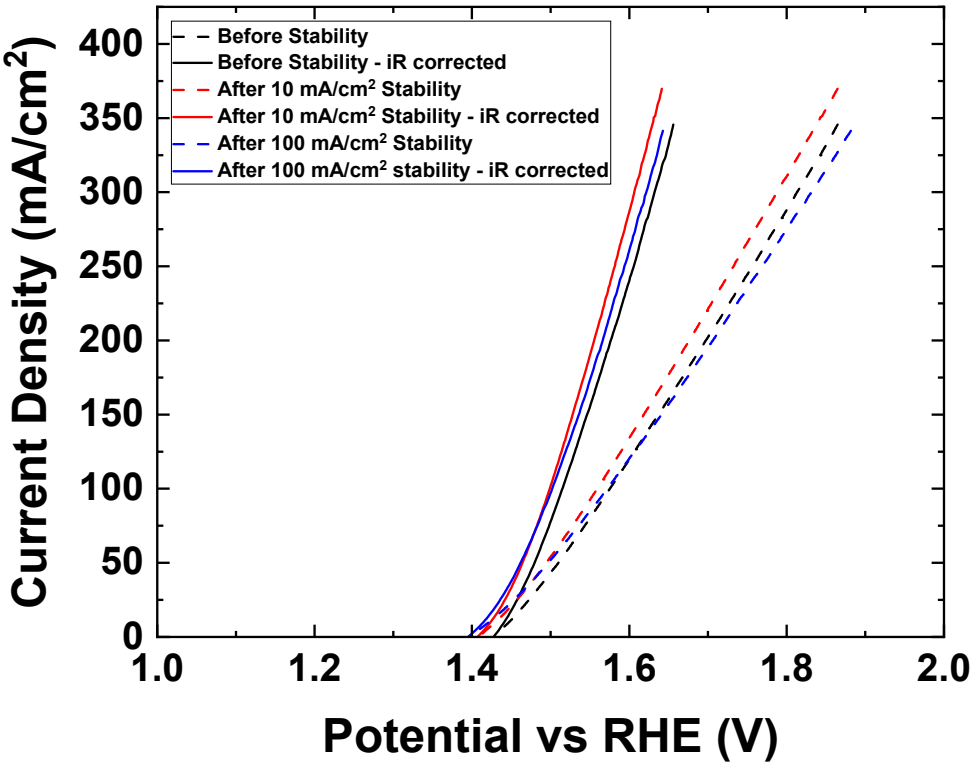

Figure S8: LSVs of NiFeCo/NF before and after chronopotentiometry (stability).

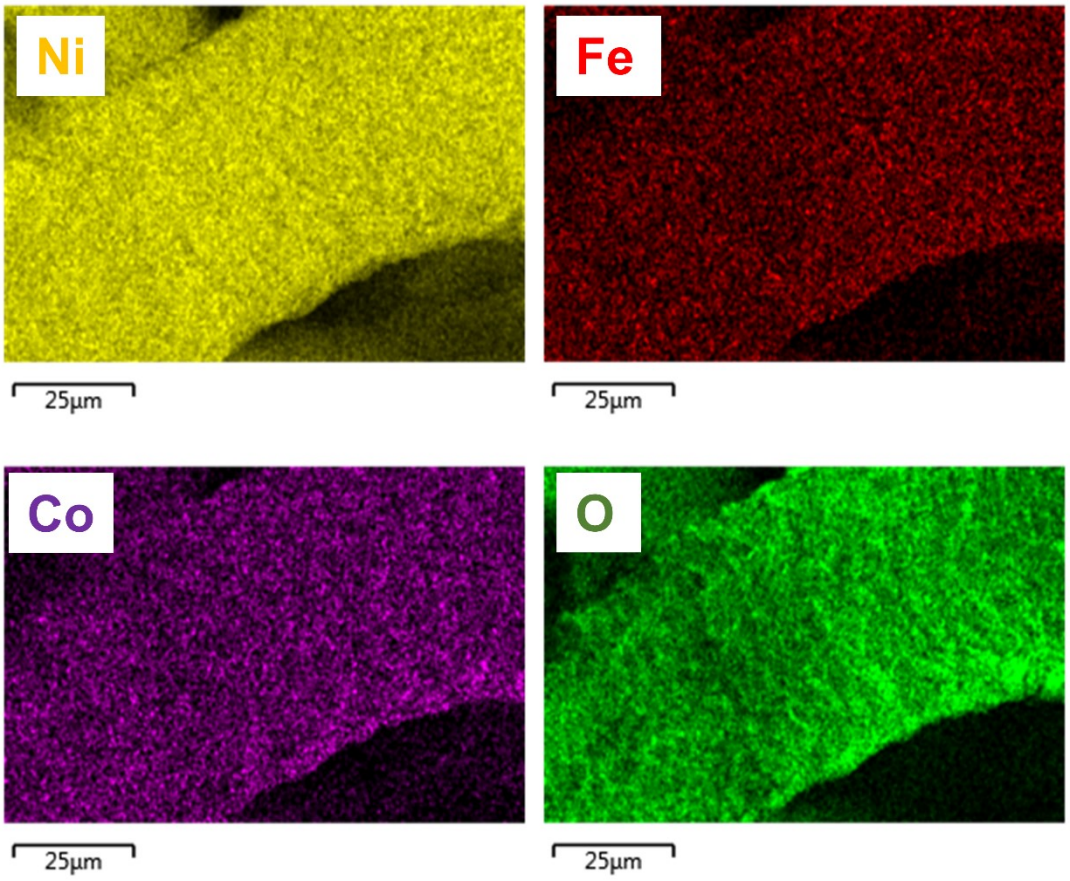

Figure S9: EDX of NiFeCo/NF surface after chronopotentiometric measurements.

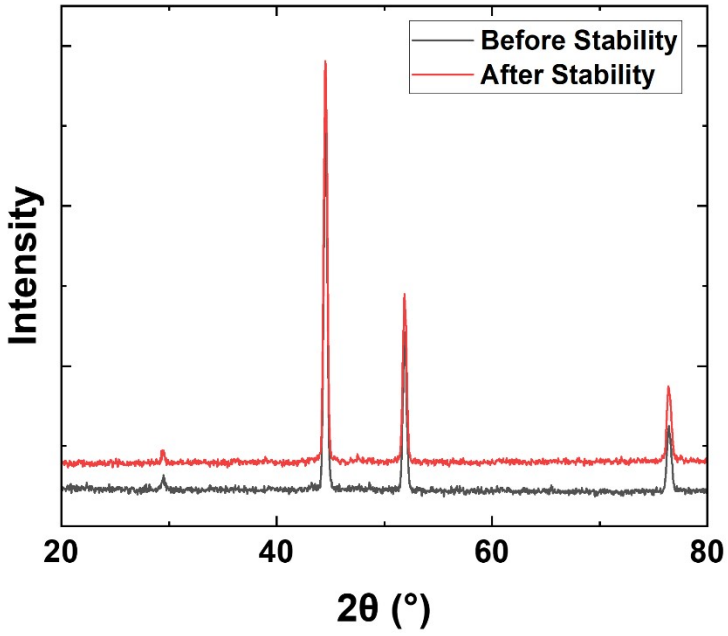

Figure S10: XRD plots of NiFeCo/NF before and after chronopotentiometry (stability) measurements.

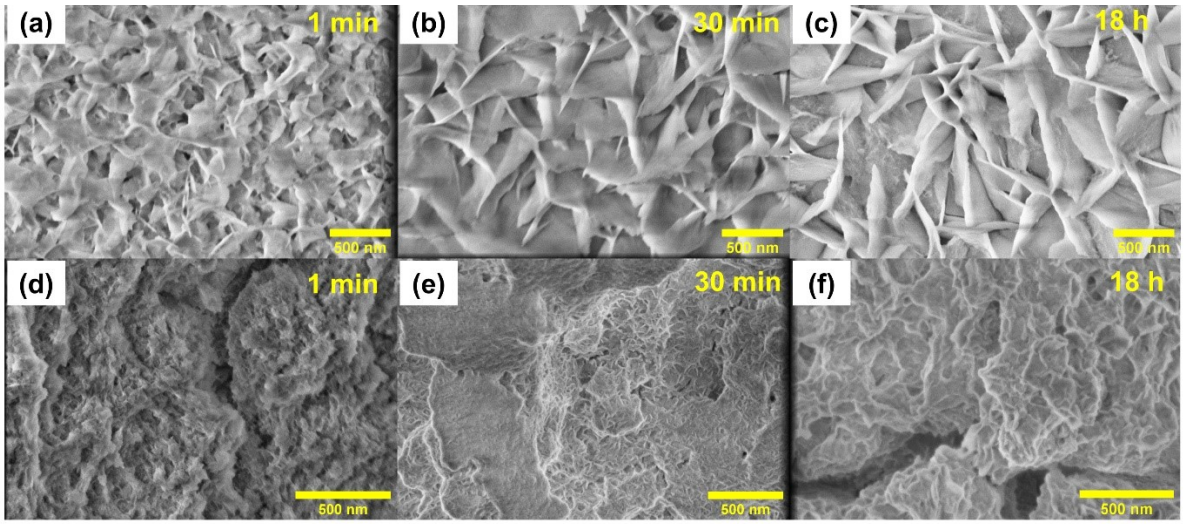

Figure S11: SEM images of NiFeCo synthesized using (a-c): Co chloride precursor and (d-f) Co nitrate precursor at different synthesis times.

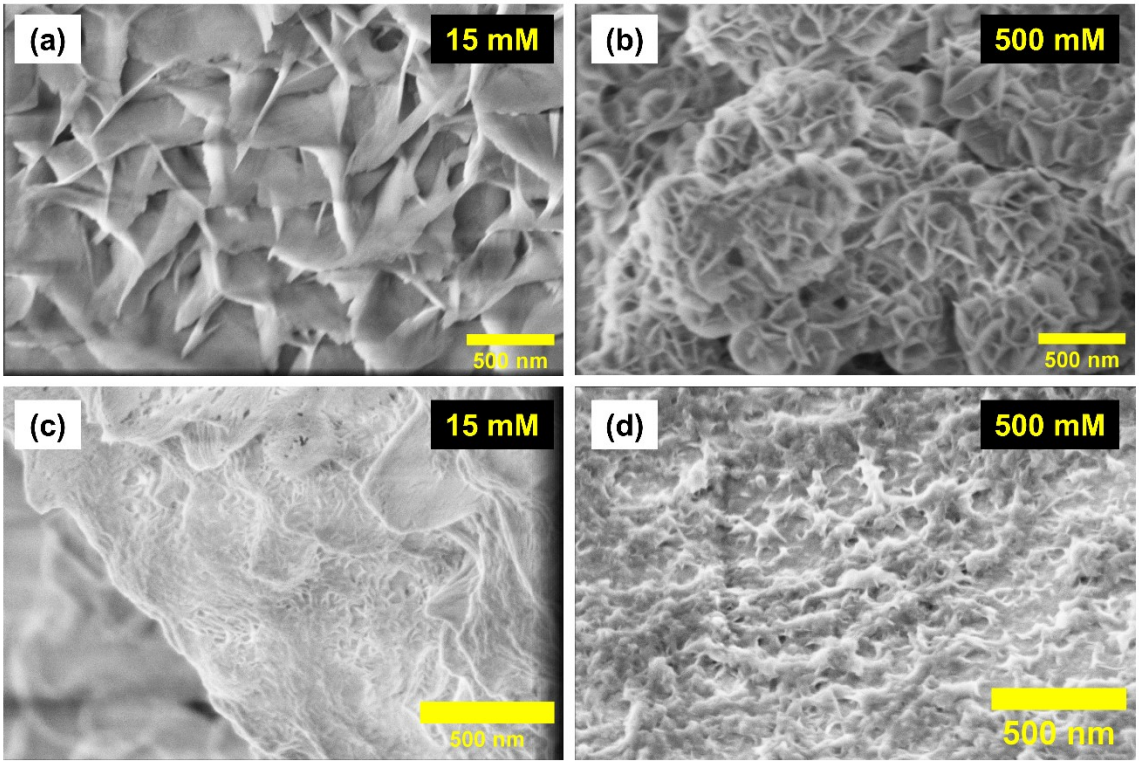

Figure S12: SEM images of NiFeCo/NF synthesized with (a-b) Co chloride precursor and (c-d) Co nitrate precursor; synthesized at 15 mM and 500 mM precursor concentrations, respectively.

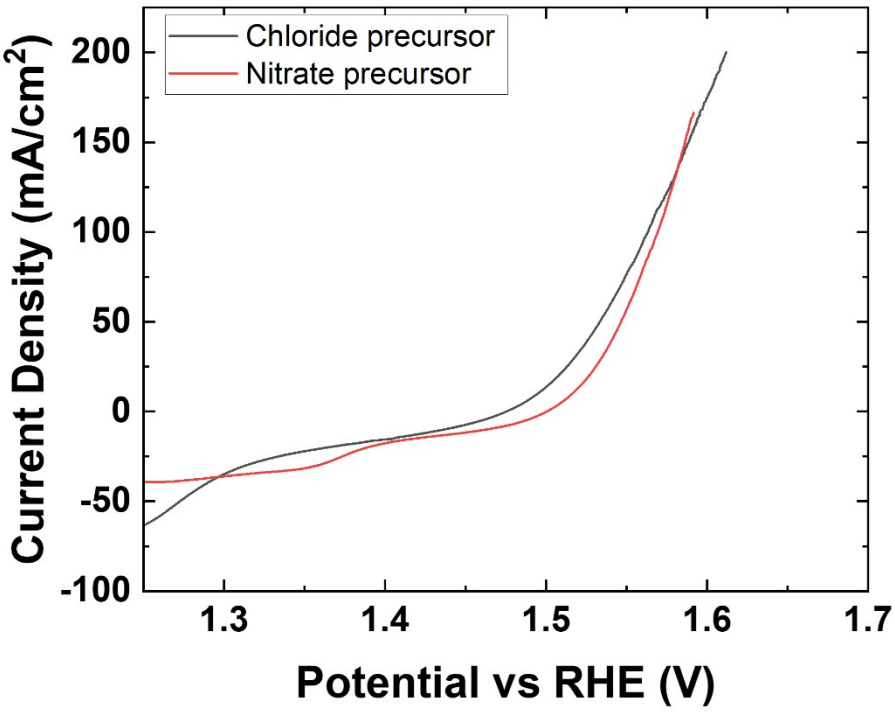

Figure S13: LSVs of NiFeCo/NF synthesized using different Co precursors at 15 mM concentration.

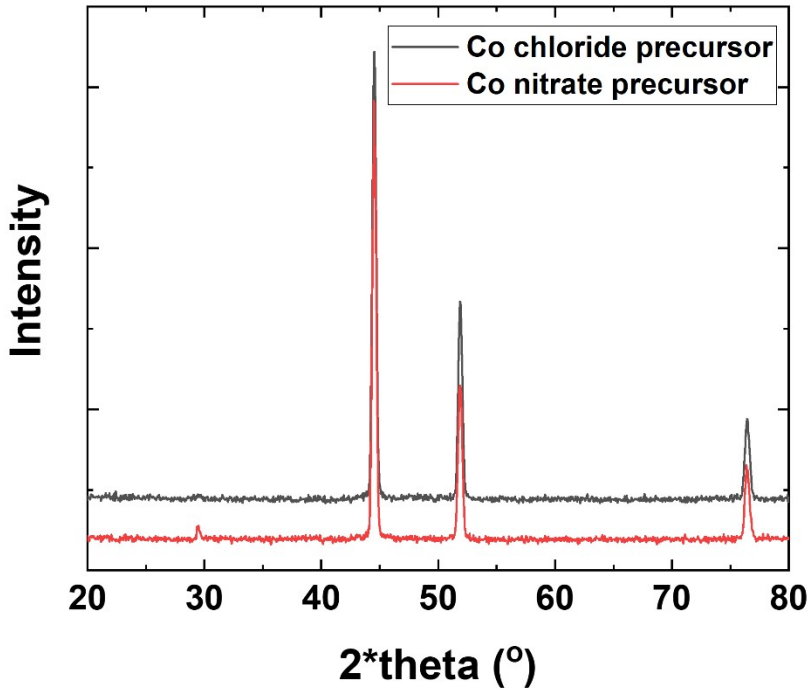

Figure S14: XRD plots of NiFeCo/NF synthesized using Co chloride and Co nitrate precursors at 15 mM precursor concentration.

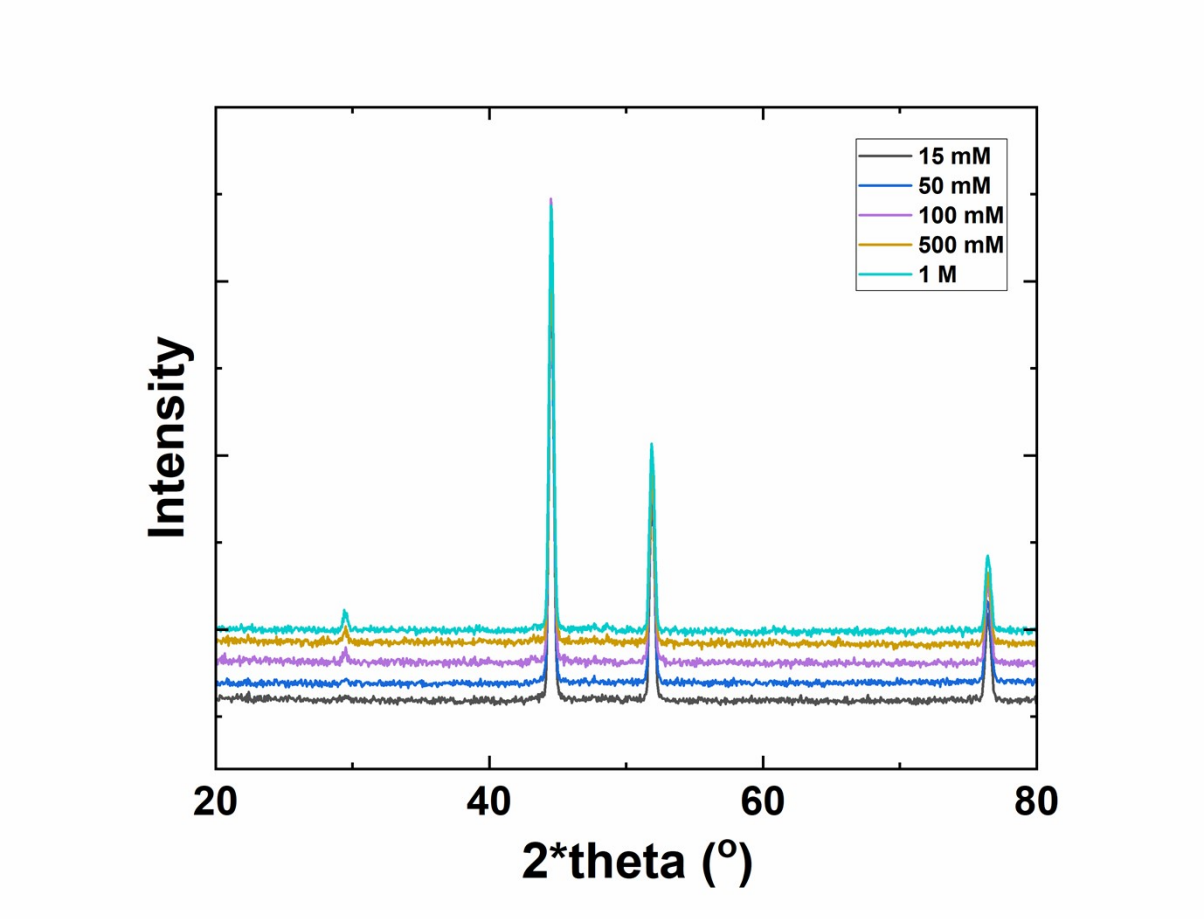

Figure S15: XRD plots of Co-chloride synthesized NiFeCo/NF at different precursor concentrations.

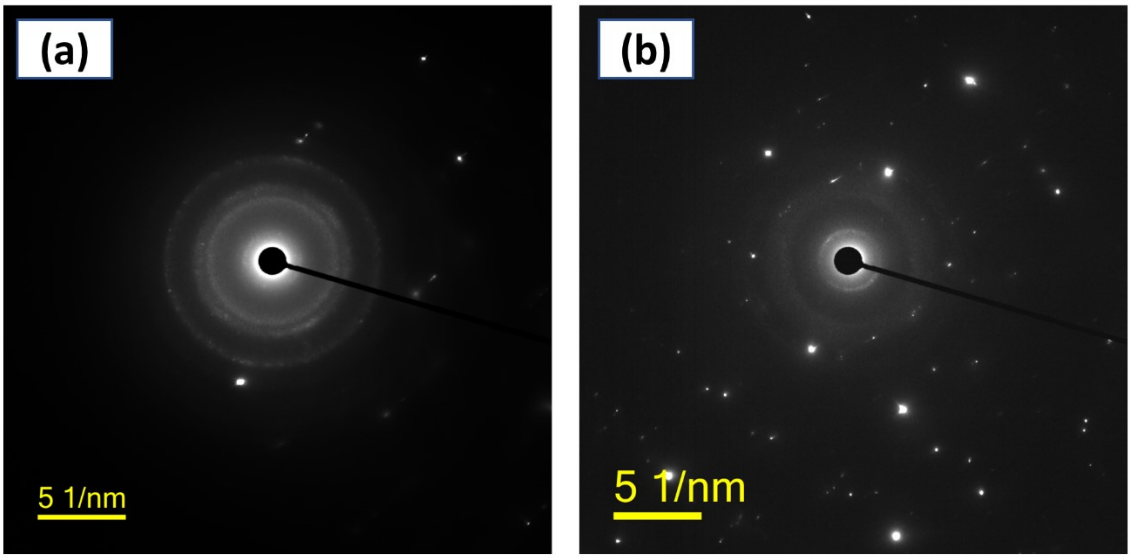

Figure S16: SAED patterns of Co –chloride synthesized NiFeCo/Si synthesized at (a) 15 mM and (b) 1 M precursor concentrations.

Table S4: pH values of Co chloride solution at different concentrations.

Co chloride concentration (mM)	Solution pH
15	7.0
50	7.0
100	6.7
500	6.2
1000	5.7

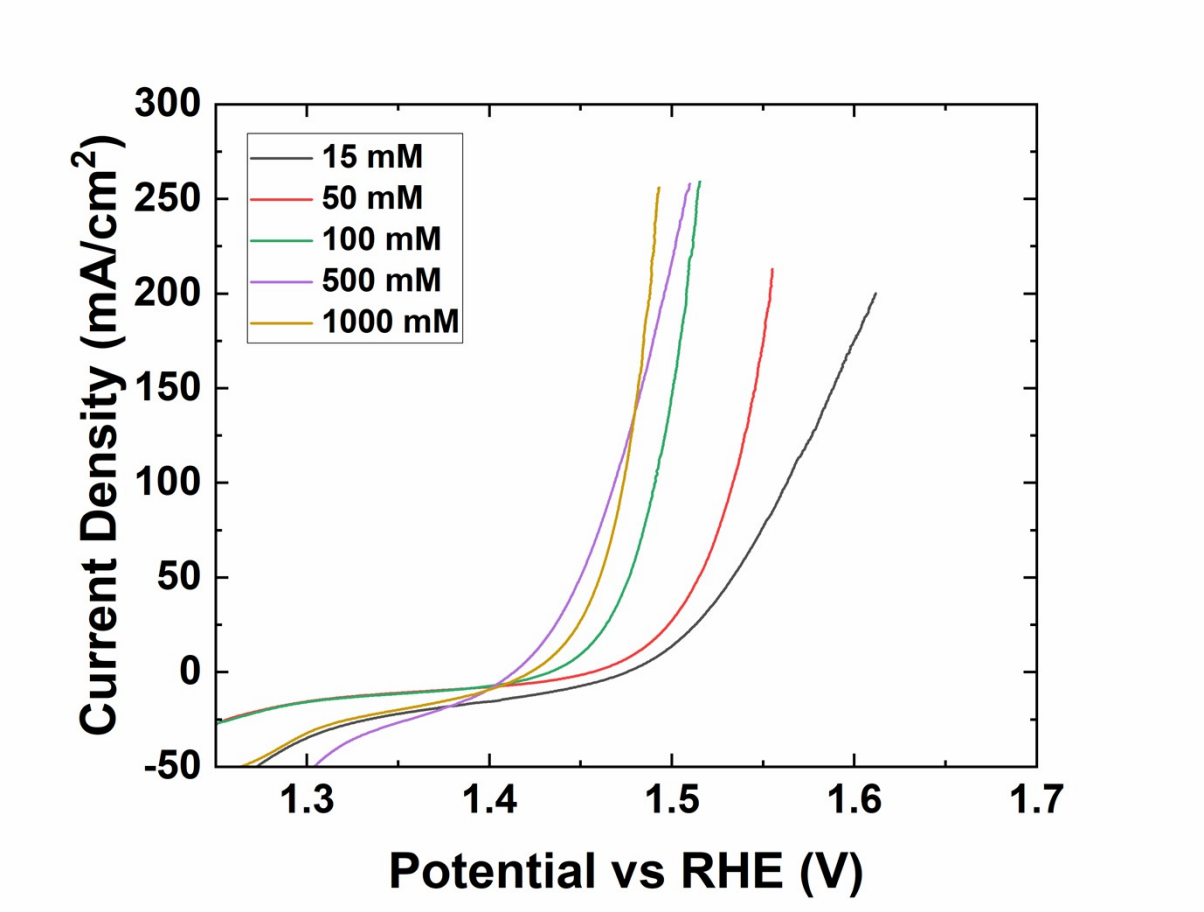

Figure S17: Reverse-scanned LSV of Co-chloride synthesized NiFeCo/NF at different Co chloride concentrations.

Table S5: Estimated R_{ct} values of Co-chloride synthesized NiFeCo/NF obtained from Nyquist plots in Figure 4(d).

Precursor Concentration (mM)	R_{ct} of NiFeCo/NF (Ω)
15	1.010
50	0.677
100	0.570
500	0.520
1000 (1 M)	0.676

References:

1. M. L. Lindstrom, R. Gakhar, K. Raja and D. Chidambaram, *Journal of The Electrochemical Society*, 2020, **167**, 046507.
2. H. Y. Jung, J. H. Park, J. C. Ro and S. J. Suh, *ACS Omega*, 2022, **7**, 45636-45641.
3. Y. S. Park, J.-Y. Jeong, M. J. Jang, C.-Y. Kwon, G. H. Kim, J. Jeong, J.-h. Lee, J. Lee and S. M. Choi, *Journal of Energy Chemistry*, 2022, **75**, 127-134.
4. M. Chen, W. Li, Y. Lu, P. Qi, H. Wu, G. Liu, Y. Zhao and Y. Tang, *Journal of Materials Chemistry A*, 2023, **11**, 4608-4618.
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