Electronic Supplimentary Information

Calixarene intercalated NiCo Layered Double Hydroxide for enhanced oxygen evolution catalysis.

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The number of Figures: 7 (Figures: S1-S7)

The number of Tabels: 3 (Table S1-S3)

Conversion of Hg/HgO to RHE

Hg/HgO electrode calibration was carried in a three electrode system with Pt wires as working and counter electrode and Hg/HgO as reference electrode. Hydrogen saturated 0.1 M KOH was used as an electrolyte. Linear sweep voltammetry was performed at 1 mV/s scan rate and the potential at which current crosses zero was taken as thermodynamic potential (vs Hg/HgO) for the hydrogen electrode. The potential at which current crosses zero is -0.949V vs Hg/HgO So E (RHE) = E (Hg/HgO) + 0.949
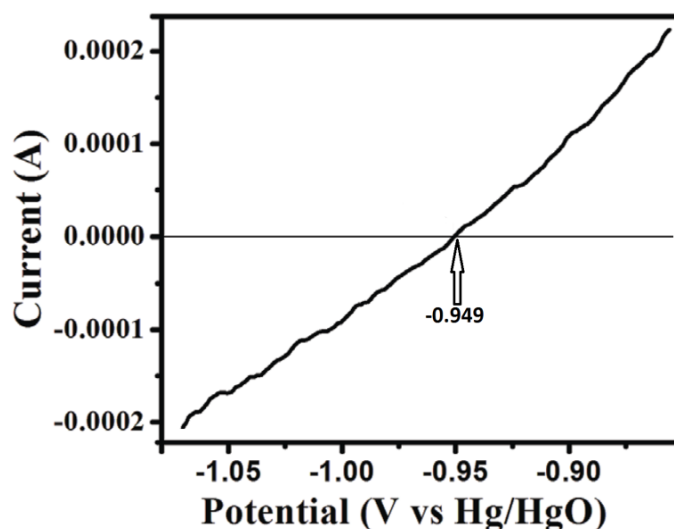

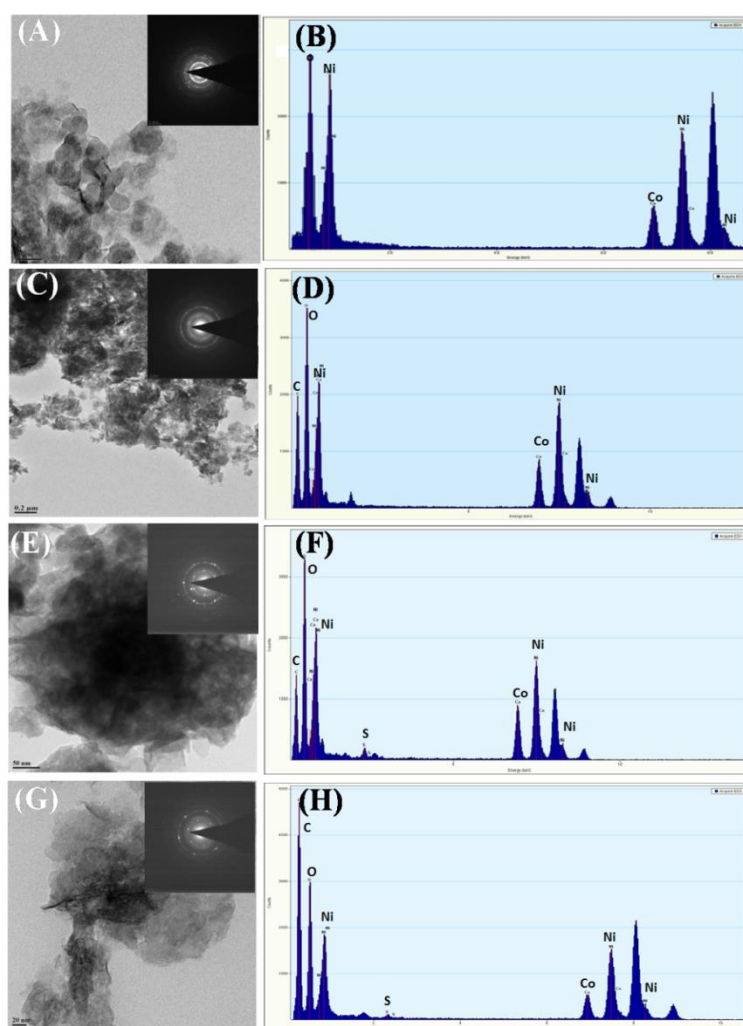**Figure S1.** Calibration curve for reference electrode

Figure S2. (A, C, E, and G) TEM micrograph and (B, D, F, and H) TEM-EDS of NiCo LDHs, CNO NiCo, CNO NiSO$_4^{2-}$Co and CNONiSC4Co nanocomposite material.
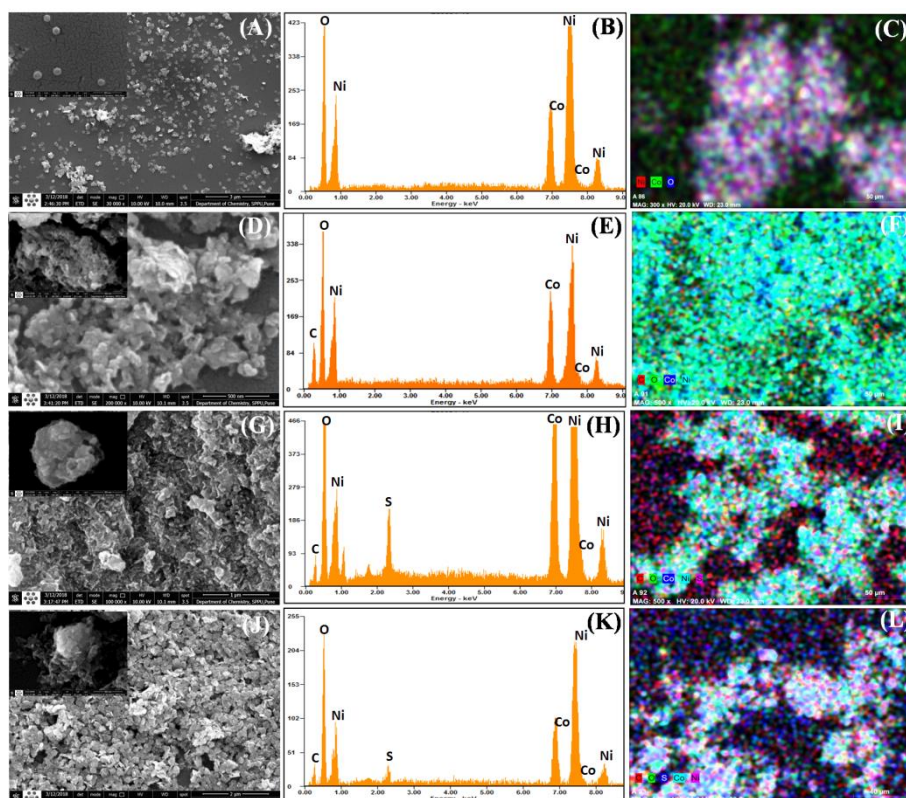
Figure S3. (A, D, G and J) SEM micrograph; (B, E, H and K) SEM-EDS; and (C, F, I and L) SEM-Elemental mapping of NiCo LDHs, CNO NiCo, CNO $NiSO_4^{2-}Co$ and CNONiSC4Co nanocomposite material.
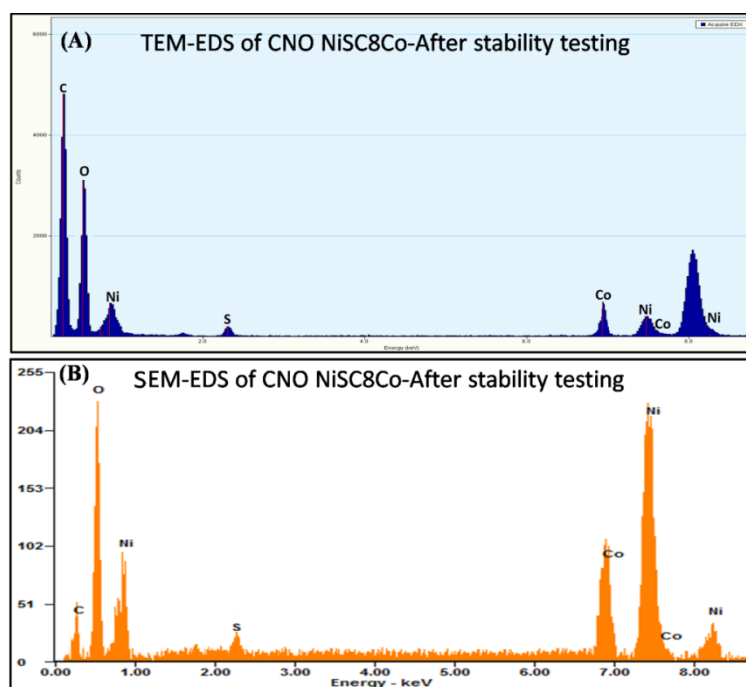

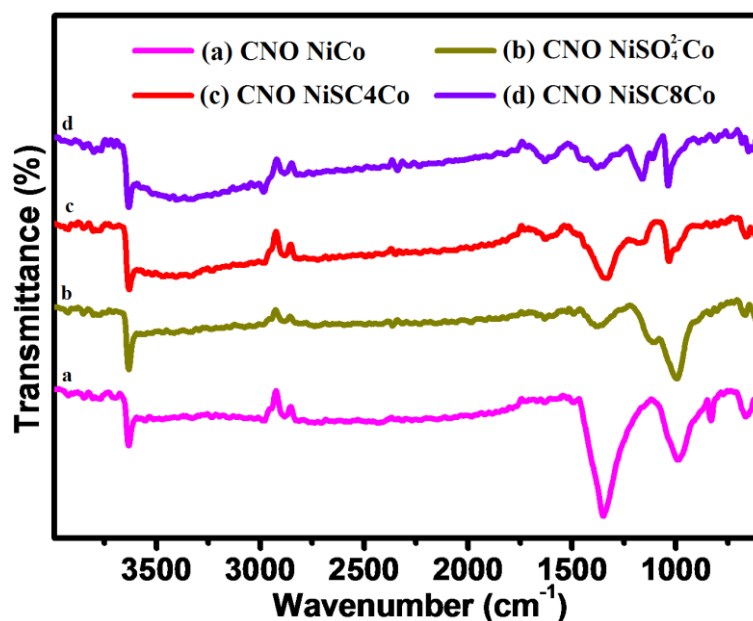**Figure S4.** (A) TEM-EDS and (B) SEM-EDS of CNO NiSC8Co nanocomposite taken after OER.

Figure S5. IR spectra of (a) CNO NiCo, (b) CNO NiSO$_4^{2-}$Co, (c)CNO NiSC4Co, and (d) CNO NiSC8Co electrocatalysts.

Table S1: Atomic percentage of elements obtained from TEM-EDS and SEM-EDS (From initial and after OER CNO NiSC8Co nanocomposite.

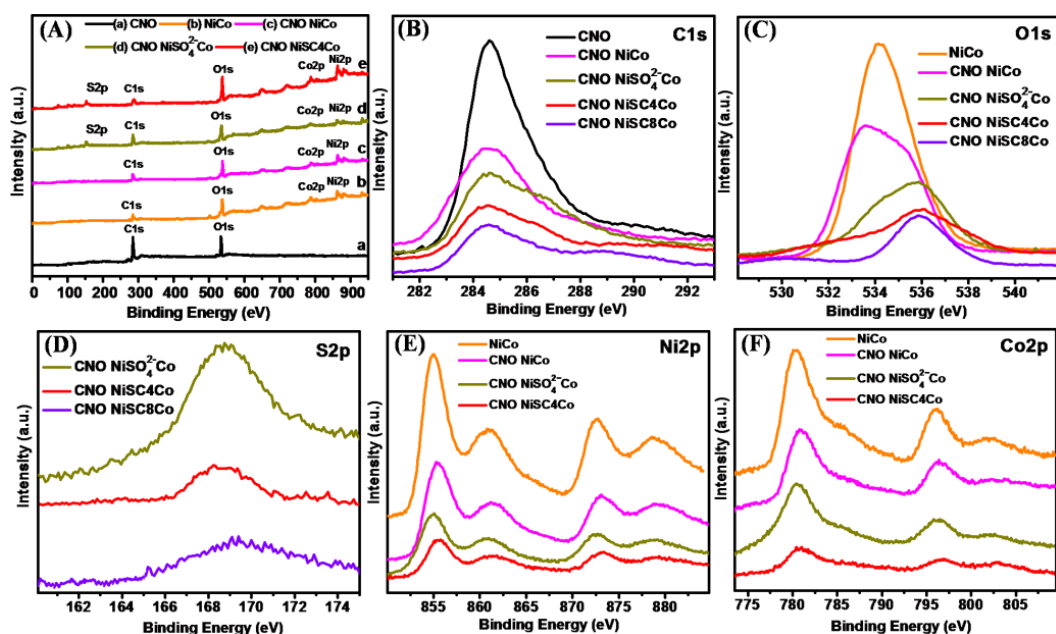| Sr. No.	TEM-EDS				Sr. No.	SEM-EDS			
		Element	Weight %	Atomic %			Element	Weight %	Atomic %
1	CNO NiSC8Co	C(K)	40.96	60.04	1	CNO NiSC8Co	C(K)	6.50	12.37
		O(K)	27.69	30.47			O(K)	49.21	70.31
		S(K)	0.37	0.20			S(K)	0.26	0.19
		Ni(K)	23.30	6.99			Ni(K)	32.18	12.53
		Co(K)	7.66	2.28			Co(K)	11.84	4.59
2	CNO NiSC8Co (Afterer 30 h anodisation)	C(K)	27.10	44.69	2	CNO NiSC8Co (Afterer 30 h anodisation)	C(K)	05.73	13.15
		O(K)	33.78	41.83			O(K)	40.74	65.06
		S(K)	1.02	0.63			S(K)	03.98	0.61
		Ni(K)	24.45	8.25			Ni(K)	33.02	14.09
		Co(K)	13.63	4.58			Co(K)	16.52	07.03

Figure S6. Comparative (A) XPS survey scan, (B) C1s, (C) O1s, (D) S2p, (E) Ni2p, and (F) Co2p spectra of CNO, NiCo LDHs and its respective nanocomposites.

Table S2: XPS analysis with species moieties of elements and its atomic percentage of initial and after OER, CNO NiSC8Co nanocomposites.

Sr. No.	Sample Name	Element present	Atomic %
1	CNONiSC8Co	C	23.86
		O	63.42
		S	0.19
		Ni	9.25
		Co	3.25
2	CNONiSC8Co (Afterer 30 h anodisation)	C	38.16
		O	51.48
		S	0.60
		Ni	6.61
		Co	3.13

Table S3 Comparison of electrocatalytic performance of the previously reported LDH based electrocatalysts for overall water splitting.

| Sr. No.	Catalyst	Over potential (mV)	Tafel slope (mV dec^{-1})	Electrolyte	Reference
1	Exfoliated Ni-Fe	300 mV	40 mV dec^{-1}	1 M KOH	*Nat. Commun.* **2014**, 5, 4477.
2	Ni-Co-OH	337 mV	-	1 M NaOH	Nano Lett. **2015**, 15, 2498.
3	Exfoliated Ni-Co	367 mV	40 mV dec^{-1}	1 M KOH	Nano Lett. **2015**, 15, 1421.
4	Ultrathin Co-Mn LDH	324 mV	43 mV dec^{-1}	1 M KOH	J. Am. Chem. Soc. **2014**, 136, 16481.
5	NiFe-Bi CC	363 mV	-	0.5 M K-Bi	ACS Appl. Mater. Int. **2017**, 9, 19502.
6	CoMn-LDH/MWCNT	300 mV	74 mV dec^{-1}	1 M KOH	ACS Appl. Mater. Int. **2016**, 8, 14527.
7	ultrathin Co-Fe LDH	300 mV	47 mV dec^{-1}	1 M KOH	ACS Appl. Mater. Int. **2016**, 8, 34474.
8	Fe-Ni nanoparticles	311 mV	-	1 M NaOH	ACS Catal. **2016**, 7, 365.
9	Co-Cr LDH (2:1)	340 mV	81 mV dec^{-1}	0.1 M NaOH	J. Mater. Chem. A **2016**, 4, 11292.
10	Ni-Fe(8:2)/3D	259 mV	39 mV dec^{-1}	1 M KOH	J. Mater. Chem. A **2015**, 3, 6921.
11	Ni-Fe LDH/N-Gr	337 mV	45 mV dec^{-1}	0.1 M KOH	Adv. Mater. **2015**, 27, 4516.
12	Ni0.6Co1.4P	300 mV	80 mV dec^{-1}	1 M KOH	Adv. Funct. Mater. **2018**, 1706008
13	3D NNCNTAs	460 mV	65 mV dec^{-1}	0.1 M KOH	Adv. Funct. Mater. **2014**, 24, 4698.
14	Co-Fe LDH (1:0.35)	350 mV	49 mV dec^{-1}	1 M NaOH	Chem Sus Chem **2017**, 10, 156.
15	Fe-Ni hydroxide	320 mV	57 mV dec^{-1}	1 M KOH	Chem Sus Chem **2016**, 9, 1835.
16	NiCo LDH	420 mV	113 mVdec^{-1}	0.1 M KOH	J. Power Sources **2015**, 278, 445.
17	ZnCo LDH/graphene	430 mV	73 mV dec^{-1}	0.1 M KOH	Dalton Trans. **2014**, 43, 15119.
18	Co-Fe LDH/rGO	340 mV	43 mV dec^{-1}	0.1 M KOH	Adv. Mater. Int. **2016**, 3, 1500782.
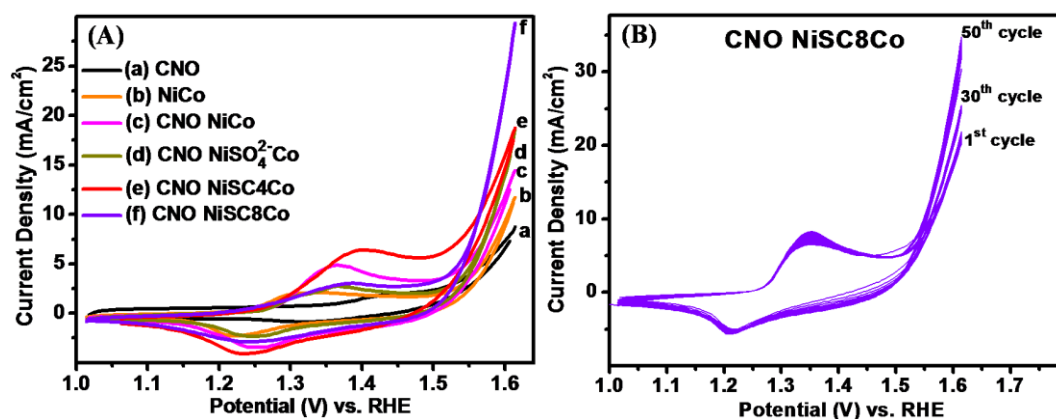
Figure S7. (A) CV of CNO, NiCo, CNO NiCo, CNO NiSO$_4^{2-}$Co, CNONiSC4Co, and CNO NiSC8Co electrocatalysts at 50 mv scan rate, (B) CV of CNO NiSC8Co electrocatalysts 1st cycle to 50th cycle.