# **Supporting information**

### Activity of pure and transition metal-modified CoOOH for the oxygen evolution reaction in an alkaline medium

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### **Experimental methods**

#### Synthesis of Co(OH)2 nanowire/SS mesh

Briefly, 5 mmol of  $Co(NO_3)_2 \cdot 6H_2O$  (Aldrich, >99.00%) and 2.5 mmol of  $NH_4NO_3$  (Aldrich, >99.0%) were added to 35 mL of de-ionized water and 5 mL of 30 wt.% ammonia (Aldrich). After stirring in air for 10 min, the solution was poured into a glass reactor bottle and pre-heated at 85 °C for 1 h. Meanwhile, a SS mesh (1 by 0.5 cm) was sequentially cleaned in acetone and de-ionized water and then treated in 3 M HCl for 15 min. The SS mesh was added to the pre-heated Co precursor solution and the entire contents were kept at 85 °C for 12 h. The resultant material consisted of  $Co(OH)_2$  nanowires on SS mesh. The samples were gently rinsed with de-ionized water followed by a 10 s sonication in de-ionized water to remove loosely bound particles. The sonication time was kept short to not completely de-root the nanowires.

#### Synthesis of CoOOH nanowire by chemical oxidation

Prior to chemical oxidation, a mixture consists of 25 mL of 6 M NaOH and 5 mL of 30 wt%  $H_2O_2$  was prepared and pre-heated to 45 °C. After this, the cleaned Co(OH)<sub>2</sub> nanowire sample is submerged into the hot mixture. Chemical oxidation is indicated by rigorous bubbling around the submerged sample. Chemical oxidation proceeded for 8 h to completely convert Co(OH)<sub>2</sub> into CoOOH.

#### Impregnation of Co(OH)2 nanowire/SS mesh

The as-prepared Co(OH)<sub>2</sub> sample was immersed in 0.5 mM Ni(NO<sub>3</sub>)<sub>2</sub>•6H<sub>2</sub>O (Aldrich, >99.99%) for 5 h to modify the sample with Ni ions. Immediately after impregnation, the sample was rinsed and placed into a tube furnace at 100 °C under nitrogen protection for 3 h. Following this, the Ni-impregnated sample was subjected to chemical oxidation by H<sub>2</sub>O<sub>2</sub> to form Ni-CoOOH. To obtain samples with higher levels of Ni modification, 0.75 and 1 mM nickel nitrate solutions were used during the impregnation process. To prepare Mn-modified samples, 0.5 and 0.75 mM Mn(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O solutions were used during the impregnation process.

#### Synthesis of NiO<sub>x</sub>H<sub>y</sub> nanoparticles and electrode preparation

NiO<sub>x</sub>H<sub>y</sub> nanoparticles were synthesized using a hydrothermal method that combined 0.5816 g of nickel nitrate, 1.2012 g of urea, and 0.1 g of sodium dodecyl sulphate in a solution of a 50/50 vol % mixture of de-ionized water and ethanol. The contents were heated in an autoclave for 15 h at 110 °C. The precipitate was collected by centrifugation and dried in an oven at 60 °C and eventually calcined in air at 250 °C for 2h. Then the NiO<sub>x</sub>H<sub>y</sub> nanoparticles were mixed with Vulcan XC-72 at 30 wt.% and dispersed at 1 mg/mL in a 0.05 wt.% Nafion solution. A small amount of this dispersion (10  $\mu$ L) was drop-cast onto a glassy carbon electrode and allowed to dry overnight. The NiO<sub>x</sub>H<sub>y</sub>/C-modified glassy carbon electrode was used as working electrode in a three electrode electrochemical cell. The uncompensated resistance of this catalyst film was evaluated by impedance spectroscopy to be 45.2  $\Omega$ .

#### Sample preparation for electrochemical testing

Once the samples are ready for electrochemical testing, a clean plastic knife was used to carefully scrape the nanowires off a small spot (approximately 2 by 2 mm) to reveal the stainless steel mesh. A tantalum wire (0.02" diameter, 3N5, ESPI metals) was spot-welded to the exposed area to make electrical contact. After the spot-weld was made, a small amount of epoxy was applied to the weld junction to cover the exposed area and a small portion of the tantalum wire. During electrochemical testing, electrical contact between the potentiostat lead and the sample was made using an alligator clip.

#### Electrochemical testing: CV, LSV and EIS

Electrochemical (EC) experiments were performed in a three-electrode configuration using a potentiostat (VersaStat 3, Princeton Applied Research). CoOOH, Ni-, or Mn-CoOOH samples were used as working electrodes, a Pt wire (PINE Research Instrument, AFCTR5) was used as a counter electrode and a double junction Ag/AgCl (PINE Research Instrument, RREF0024) was used as a reference electrode. Cyclic voltammetry and linear sweep voltammetry were performed from 0 to 1 V vs. Ag/AgCl at 50 and 1 mVs<sup>-1</sup>, respectively. Electrochemical impedance spectroscopy (EIS) was carried out by applying 20 mV (peak-to-peak) AC voltage to the working electrode under 0.6 V DC bias, and the frequency was scanned from 10 kHz to 10 mHz. During EC testing, care was taken to not expose the tantalum wire to the electrolyte through proper positioning of the CoOOH/SS mesh electrode. Approximately 200 mL of 0.1M KOH was used for all EC experiments. The 0.1M KOH was scavenged for Fe ions using Co(OH)<sub>2</sub> nanoparticles before using in EC experiments and the electrolyte was purged with N<sub>2</sub> during all EC experiments.

SEM images of Mn-CoOOH-B, Ni-CoOOH-B and Ni-CoOOH-C



**Fig. S1** From left to right, SEM images of Mn-CoOOH-B, Ni-CoOOH-B, and Ni-CoOOH-C nanowires grown on a SS mesh. Insets show higher magnification SEM images, with scale bars to represent 500 nm.

SEM image of CoOOH nanowires showing hexagonal termination



**Fig. S2** SEM image of CoOOH nanowires resolving hexagonal nanoplates, outlined in blue. Particles at the tip of the nanowires are indicated by red arrows.

#### Estimation of increase in the electrolyte-accessible area

Consider a 10 µm thin film grown conformal to a segment (30 µm) of wire in the SS mesh. The estimated diameter of the wire is 30 µm from Figure 1a. Therefore the surface area accessible to electrolyte for a conformal film is

 $SA_{film} = 50\mu m * \pi * 30\mu m = 4700 \ \mu m^2$ 

Consider the same segment of SS wire but the surface of the wire is populated by CoOOH nanowires having averaged diameter of 375 nm and length of 10µm. If only 70 % of the said SS wire segment is covered with CoOOH nanowires, the surface area of the nanowires can be calculated. First we calculate the surface area of SS wire segment.

 $SA_{SSwire} = 30\mu m * \pi * 30\mu m = 2800 \mu m^2$ 

The cross section area  $(A_{CS})$  of nanowire is,

 $A_{CS} = (0.375 \mu m/2)^2 * \pi = 0.11 \mu m^2$ 

Since only 70% of the wire segment is populated with CoOOH nanowires, the total number of nanowires  $(n_{NW})$  grown in the SS wire segment is,

 $n_{NW} = 2800 \mu m^2 * 0.7/0.11 \mu m^2 = 18000$ 

Based on the dimensions of each nanowire, the total accessible area (SA<sub>NW</sub>) to electrolyte is,

 $SA_{NW} = 18000 * 0.375 \mu m * \pi * 10 \mu m = 210000 \mu m^2$ 

Compared to the surface area of conformal thin film, the nanowire electrode will show approximately 45 times greater surface area.

HR-TEM images of pure and modified CoOOH



**Fig. S3** TEM images of a) CoOOH, b) Mn-CoOOH-A, c) Mn-CoOOH-B, d) Ni-CoOOH-A, e) Ni-CoOOH-B, and f) Ni-CoOOH-C. Insets clearly show the lattice spacing of the  $\{003\}$  planes of  $\beta$ -CoOOH, with scale bars to represent 2 nm.

#### Raman spectroscopy of pure and modified CoOOH

At higher Ni and Mn modifications, the intensity of the  $E_g$  peak at 501 cm<sup>-1</sup> decrease which can be attributed to the distortion of the rhombohedral structure due to Ni and Mn incorporations. Similar observation was reported by Inaba et al. where the Raman peak intensity of LiCoO<sub>2</sub>—a structurally similar material to CoOOH is decreased due to Ni doping.<sup>1</sup> Another explanation for the weaker Raman peak at 501 cm<sup>-1</sup> could be the change in the polarizability of the  $E_g$  vibrational mode with Ni and Mn substitution into Co sites. Raman polarizability tensor for Co decreases to a greater degree upon addition of Mn compared to Ni substitution into Co sites. This is consistent with the expected high spin electronic configuration for Mn<sup>3+</sup> (in contrast to low spin for Ni<sup>3+</sup> and Co<sup>3+</sup>) resulting in weaker longer M-O bonds with less charge transfer. Chen et al have reported the change in Raman band polarizability of MnO<sub>2</sub> due to due to the substitution of interlayer neutral water molecules by positively charged cations.<sup>2</sup>

[1] M. Inaba, Y. Todzuka, H. Yoshida, Y. Grincourt, A. Tasaka, Y. Tomida, Z. Ogumi, *Chem. Lett.*, 1995, 24, 889-890.

[2] D. Chen, D. Ding, X. Li, G.H. Waller, X. Xiong, M. A. El-Sayed, M. Liu, Chem. Mater., 2015, 27, 6608-6619.



Co2p XPS spectra of pure and modified CoOOH nanowire catalysts

**Fig. S4** Co 2p XPS spectra of pure and modified CoOOH. Satellite feature at 10 eV above the principle line (780 eV) is indicative of  $Co^{3+}$  ions in all the samples.



#### Co LMM XPS spectra of pure and modified CoOOH nanowire catalysts

**Fig. S5** a) Co LMM region of the XPS survey spectra of the pure and modified CoOOH. b) Difference spectra of the Ni- and Mn-modified sample. The vertical lines on the X-axis indicate the Fe  $2p_{3/2}$  and Fe  $2p_{1/2}$  peak positions.

It is known that the Fe 2p XPS peaks overlap with the Co LMM region when an Al K $\alpha$  X-ray source is used in XPS. This issue makes it difficult to quantify the amount of Fe in the sample containing both Co and Fe, especially when Fe concentration is low. To address this issue, we obtained and examined the XPS difference spectra by subtracting the Co LMM signal of pure CoOOH from that of the modified samples and the results are shown in Fig S5 b). The difference spectra from the modified samples overlap in the region where the Fe 2p signal is expected, and no obvious peak is observed. Based on this result, we conclude that if Fe is present in our samples, the amount of Fe in all samples is comparable.

We further calculated the area under the difference spectra from 700 to 730 eV to establish a Fe concentration relative to the CoOOH. Using a linear baseline, Fe concentrations between 0.8% and 1% were obtained, with Mn-modified samples having the highest Fe concentration relative to pure CoOOH. Thus, at most, there is a 1% greater Fe content between the modified and pure CoOOH, and we believe such small difference is not enough to enable the degree of the OER activity improvement observed by us. Additionally, we observed significantly greater current density between the Ni-CoOOH-A and Ni-CoOOH-B samples where the Fe concentrations relative to CoOOH only differ by 0.1%. Based on the work from Burke et al, the OER activity of Fe-doped CoOOH showed improvement at greater Fe concentration than the 1% difference we observed from the analysis carried above.<sup>3</sup> We believe the approach implemented here for comparing Fe concentration relative to the pure CoOOH as a reference when comparing the OER activity improvements. Based on the analysis above, we believe that all samples have similar amount of Fe and that the observed improvement in OER activity is primarily due to the incorporation of Ni.

[3] M. S. Burke, S. H. Zou, L. J. Enman, J. E. Kellon, C. A. Gabor, E. Pledger and S. W. Boettcher, J. Phys. Chem. Lett., 2015, 6, 3737-3742.

### CV of pure and modified CoOOH nanowire catalysts



**Fig. S6** CV of pure and modified CoOOH. CV measurements were acquired in  $N_2$  purged 0.1M KOH at scan speed of 50 mVs<sup>-1</sup>. Due to the large surface area of the electrode, significant contribution from the capacitive current is observed before the onset of OER. Due to this reason, we focused our analysis on the LSV results (collected at 1 mVs<sup>-1</sup>), which has little contribution from capacitive current.



Comparison of the OER activity of pure and modified CoOOH with NiO<sub>x</sub>H<sub>y</sub>

Fig. S7 Comparison of the OER performance of a  $NiO_xH_y$  catalyst with that of pure, Ni- and Mn-modified CoOOH catalysts. Insets show an SEM image and a Raman spectrum of  $NiO_xH_y$  particles.

To further confirm the positive effects of Ni incorporation on the OER activity of these Ni-CoOOH catalysts, we synthesized NiO<sub>x</sub>H<sub>y</sub> particles and compared their OER activity to that of our Ni-CoOOH samples. The NiO<sub>x</sub>H<sub>y</sub> particles ranged in size between 500 nm to 1  $\mu$ m, as shown in the SEM image in Fig. S7, with their surface decorated by smaller nanoflakes. In Raman spectroscopy we observed clear phonon peaks corresponding to  $\gamma$ -NiOOH,<sup>4</sup> however an  $\alpha$ -Ni(OH)<sub>2</sub> phase was observed in XRD results (not shown). Tests for OER activity entailed mixing the NiO<sub>x</sub>H<sub>y</sub> particles with a carbon support (Vulcan XC-72) at 30 wt.% loading. We did not feel that it was important to obtain phase pure  $\gamma$ -NiOOH, since the exact local structure of Ni in our Ni-CoOOH samples is unclear, and multiple studies have discussed the transformation of Ni(OH)2 to NiOOH during OER, which means  $\alpha$ -Ni(OH)<sub>2</sub> in the NiO<sub>x</sub>H<sub>y</sub> sample will be transformed into NiOOH during reaction.4 Comparing the OER performance, the NiOxHy catalyst exhibited lower activity than the 5.5% and 9.7% Ni-modified CoOOH catalyst but higher activity than pure CoOOH. This result demonstrates the Ni modification of CoOOH creates highly sites that are more active than pure NiO<sub>x</sub>H<sub>y</sub>.

[4] B.S. Yeo, A.T. Bell, J. Phys. Chem. C, 2012, 116, 8394-8400.

Stability testing of pure and modified CoOOH



Fig. S8 Chronopotentiometry of pure and modified CoOOH electrodes.

Catalyst stability was evaluated for 25 hours by chronopotentiometry in 0.1M KOH under constant  $N_2$  purging. The counter electrode was Pt, and the reference electrode was a double junction Ag/AgCl cell. The working electrode was prepared according to the procedure described in the Supporting Information Experimental section. For this stability testing, the potential required to obtain a 10 mAcm<sup>-2</sup> current density was monitored over time. An increase in this potential reflects degradation in catalyst performance. Fig. S8 illustrates these chronopotentiometry results for pure and modified CoOOH samples. The potential required to maintain a 10 mAcm<sup>-2</sup> current density increased by 2-3% for all tested catalysts, indicating good stability of all of these catalysts.



Raman, XRD and XPS after OER stability testing

**Fig. S9** a) XRD and b) Raman spectroscopy of pure and Ni- and Mn-CoOOH after stability testing. c) Ni  $2p_{3/2}$  and d) Mn  $2p_{3/2}$  XPS spectra of Ni- and Mn-CoOOH samples.

The structure and chemical composition of pure and Ni- and Mn-modified CoOOH catalysts were investigated after OER stability testing using XRD, Raman spectroscopy, and XPS. The XRD data in Fig. S9a shows a single peak at 20.2° 2 $\theta$ , which corresponds to the (003) diffraction peak of  $\beta$ -CoOOH. Comparing the XRD curves obtained for pure and Ni- and Mn-modified samples prior to (Fig. 4a) and after reaction; there are no changes in peak positions. We note that the (003) peak FWHM increased slightly from 1.1° to 1.3° after stability testing. This could indicate a decrease in the overall crystallinity of the samples after stability testing, however this change in FWHM is too small to lead to a clear conclusion. In addition, we avoided the XRD peaks from the SS mesh support by collecting the nanowires by scraping and sonication from the SS mesh support after stability testing. For each pure and Ni- and Mn-CoOOH sample, stability testing was performed on three 1.5 x 1.5 cm samples in order to collect enough nanowires for an accurate XRD evaluation. Raman spectroscopy was performed on one of the triplicate samples and the result is shown in Fig. S9b, which indicates excellent local crystallinity before and after stability testing consistent with the minimal degradation in the OER activity observed, as shown

in Fig. S8. XPS analysis of Ni- and Mn-modified CoOOH samples was also carried out and some results are provided in Fig. S9. Ni 2p and Mn 2p spectra were fitted using the same components as used Fig. 5. Little difference was observed in comparing spectra obtained before and after the stability test, which indicates a similar near-surface composition and local chemical environment for the tested catalysts. The Ni and Mn content was altered measurably, to 9.2 and 2.3 at.%, respectively after subjecting the samples to the stability test. This could be explained by the detachment of weakly attached particles of NiO<sub>x</sub> or MnO<sub>x</sub> and their removal from the surface of the catalysts during OER experiments.

### STEM of Ni-modified CoOOH prior to OER testing



Fig. S10 STEM dark field and EDX images of a CoOOH nanowire containing 9.7% Ni.

Scanning transmission electron microscopy (STEM) was performed to investigate the spatial distribution of Ni in the CoOOH nanowire sample with 9.7% Ni concentration. The STEM EDX images in Fig. S10 clearly demonstrate a uniform Ni distribution with a slight Ni enrichment near the surface. No evidence of aggregated  $NiO_x$  phase was observed at the magnification value shown.

### Calculation of (003) d-spacing from SAED patterns

**Table S1** (003) d-spacing calculation based on the SAED pattern from TEM images in Figure 4. The plane spacing is calculated based on a gold reference. For Au,  $d_{(111)}=2.35478$  Å. The diffraction patterns were acquired at the camera length of 470 mm.

Sample	(h k l)	D (pixel)	d (Angstrom)
СоООН	(0 0 3)	502	4.433
Ni-CoOOH-A	(0 0 3)	496	4.486
Ni-CoOOH-B	(0 0 3)	496	4.482
Ni-CoOOH-C	(0 0 3)	498	4.464
Mn-CoOOH-A	(0 0 3)	501	4.441
Mn-CoOOH-B	(0 0 3)	500	4.446

## Elemental composition of pure and modified CoOOH nanowires by XPS

Sample	Carbon (%)	Oxygen (%)	Cobalt (%)	Nickel (%)	Manganese (%)
СоООН	15.9	61.2	22.9	0	0
Ni-CoOOH-A	9.9	49.9	34.7	5.5	0
Ni-CoOOH-B	13.9	44.2	32.2	9.7	0
Ni-CoOOH-C	9.1	44.5	30.9	15.5	0
Mn-CoOOH-A	9.5	57.2	30.8	0	2.5
Mn-CoOOH-B	8.9	54.4	30.7	0	6.0

 Table S2 Elemental composition of pure and modified nanowires based on XPS analysis

## Elemental composition of pure and modified CoOOH nanowires by EDX

Table S3 Elemental composition of pure and modified nanowires based on EDX analysis in atomic percent

Sample	Carbon (%)	Oxygen (%)	Cobalt (%)	Nickel (%)	Manganese (%)
СоООН	17.5	59.4	23.1	0	0
Ni-CoOOH-A	19.2	51.4	26.2	3.2	0
Ni-CoOOH-B	14.8	54.2	23.9	7.1	0
Ni-CoOOH-C	14.1	51.5	21.7	12.7	0
Mn-CoOOH-A	14.7	57.3	26.1	0	1.9
Mn-CoOOH-B	15.8	54.1	25.5	0	4.6

## Fitting parameters used in the decomposition of O1s XPS spectra

Sample		OL	OHL	OH <sub>ads</sub>	Ocarbon
СоООН	Position (eV)	529.8	530.8	531.6	532.5
	FWHM (eV)	1.17	1.17	1.17	1.8
	Area (%)	38.9	37.0	15.9	8.2
Ni-CoOOH-A	Position (eV)	529.8	530.9	531.7	532.5
	FWHM (eV)	1.22	1.22	1.22	1.8
	Area (%)	37.8	34.2	20.8	7.2
Ni-CoOOH-B	Position (eV)	529.8	530.9	531.6	532.5
	FWHM (eV)	1.22	1.22	1.22	1.8
	Area (%)	35.7	34.3	21.4	8.6
Ni-CoOOH-C	Position (eV)	529.8	530.8	531.8	532.7
	FWHM (eV)	1.25	1.25	1.25	1.7
	Area (%)	21.6	41.9	25.9	10.6
Mn-CoOOH-A	Position (eV)	529.9	530.8	531.6	532.6
	FWHM (eV)	1.17	1.17	1.17	1.8
	Area (%)	40.4	34.1	17.0	8.5
Mn-CoOOH-B	Position (eV)	529.9	530.8	531.6	532.5
	FWHM (eV)	1.18	1.18	1.18	1.8
	Area (%)	44.7	33.2	13.5	8.6

**Table S4** Fitting parameters for the O1s XPS spectra of pure and modified CoOOH

## Parameters used for fitting the EIS results

	Q <sub>dl</sub>				$Q_{\Phi}$			
	$R_{\Omega}$	Yo	n	Rp	Yo	n	R <sub>s</sub>	
	$(\Omega \cdot cm^2)$	(S·sec^n/cm <sup>2</sup>		$(\Omega \cdot cm^2)$	(S·sec^n/cm		(Ω·cm <sup>2</sup>	
		)			<sup>2</sup> )		)	
СоООН	15.1	0.072	0.80	1.18	0.204	0.94	6.39	
Ni-CoOOH_A	15.3	0.062	0.74	0.73	0.228	0.88	3.35	
Ni-CoOOH_B	15.2	0.051	0.68	0.54	0.312	0.79	1.87	
Ni-CoOOH_C	15.5	0.069	0.70	0.91	0.199	0.92	4.10	
Mn-	15.1	0.060	0.80	1.08	0.170	0.94	7.16	
CoOOH_A								
Mn-	15.2	0.054	0.80	0.91	0.211	0.92	7.20	
CoOOH_B								
			_	-				
			Percent	error from	fitting			
СоООН	0.14	20	10	26	14	6.1	4.4	
Ni-CoOOH_A	0.13	22	11	19	11	4.0	3.1	
Ni-CoOOH_B	0.13	23	11	19	8.9	5.3	5.2	
Ni-CoOOH_C	0.11	15	7.9	18	8.9	6.1	4.8	
Mn-								
CoOOH_A	0.11	29	12	22	12	4.3	2.3	
Mn-								
CoOOH_B	0.11	22	13	23	12	4.9	2.2	
		Stand	dard de	viation (thr	ee samples)			
СоООН	0.17	0.01	0.04	0.11	0.09	0.05	0.60	
Ni-CoOOH_A	0.18	0.01	0.03	0.12	0.08	0.03	0.63	
Ni-CoOOH_B	0.15	0.01	0.07	0.15	0.08	0.07	0.71	
Ni-CoOOH_C	0.15	0.02	0.05	0.20	0.07	0.07	0.77	
Mn-								
CoOOH_A	0.17	0.02	0.06	0.11	0.06	0.05	0.61	
Mn-								
CoOOH B	0.15	0.02	0.06	0.14	0.06	0.09	0.62	