

Supporting Information

Mesoporous K-doped NiCo₂O₄ derived from Prussian blue analog: High-yielding synthesis and assessment as oxygen evolution reaction catalyst

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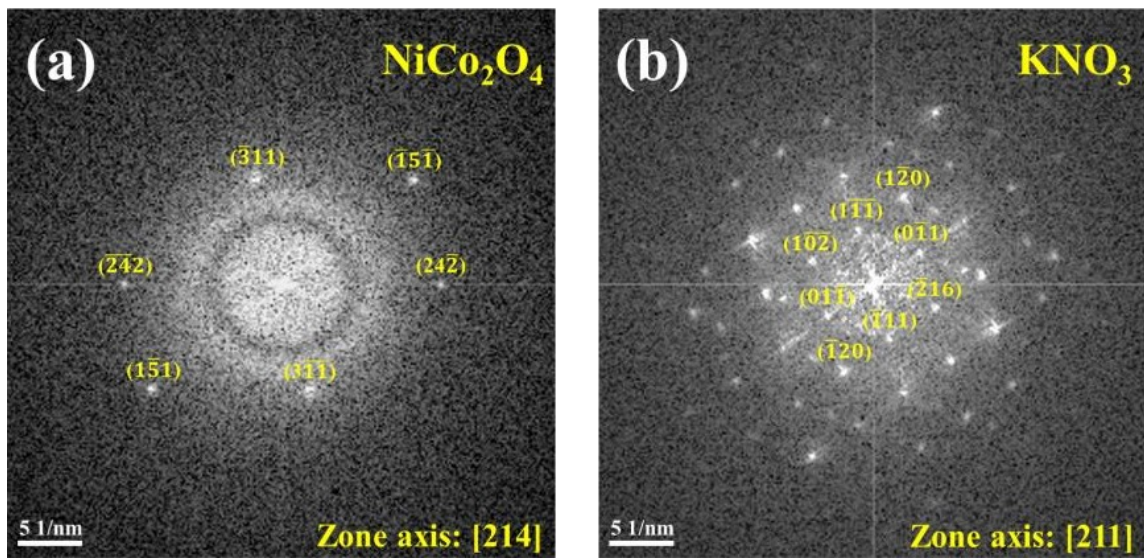


Figure S1. Fast Fourier transform patterns of (a) NiCo_2O_4 (white rectangle in Fig. 1b) and (b) KNO_3 (red rectangle in Fig. 1b).

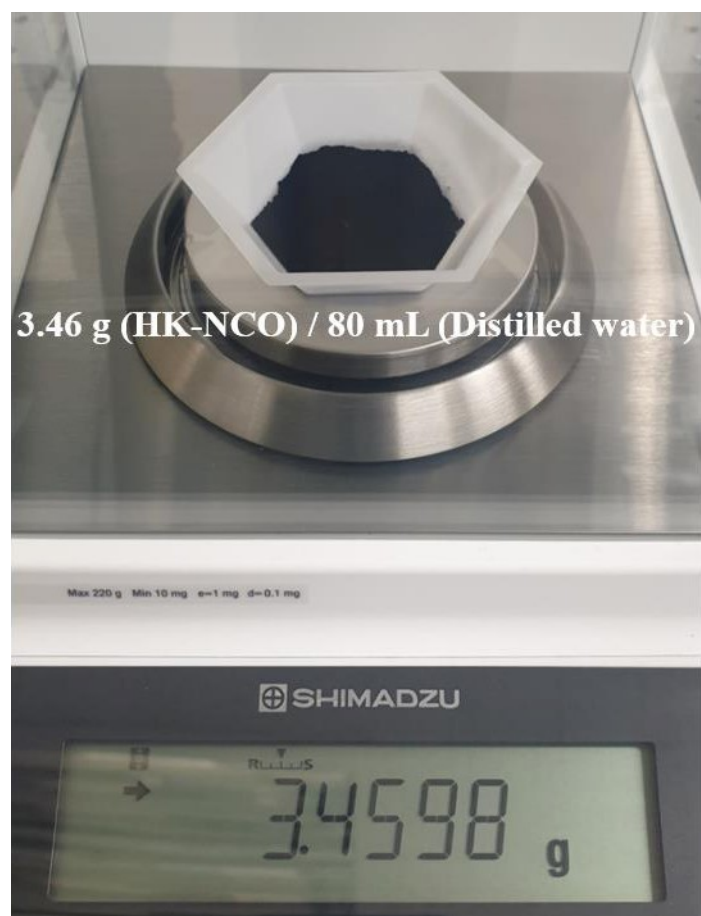


Figure S2. Weighing the HK-NCO catalyst powder produced by hydrothermal synthesis.

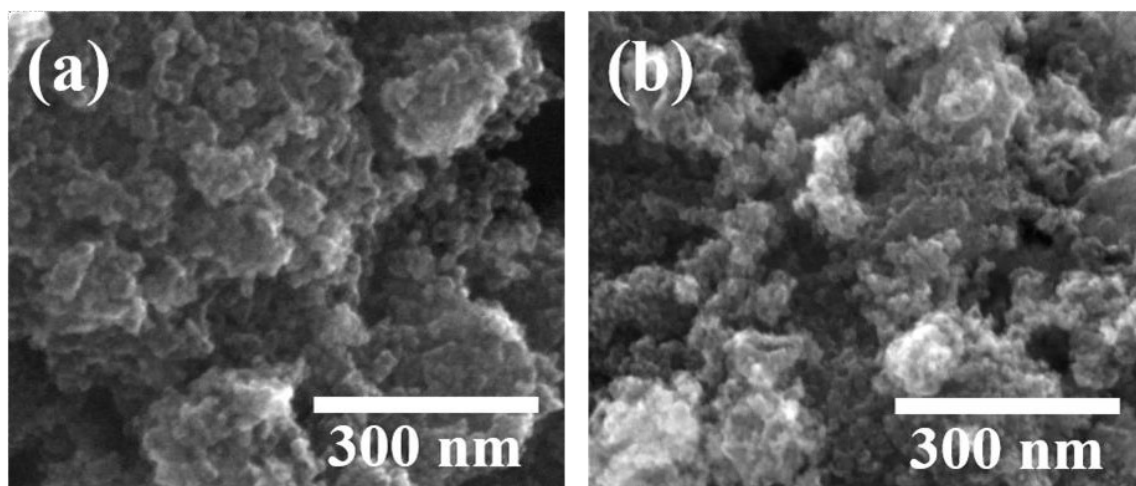


Figure S3. SEM images of catalysts prepared (a) hydrothermally (KNO_3 + high-concentration K-doped NiCo_2O_4) and (b) non-hydrothermally (KNO_3 + low-concentration K-doped NiCo_2O_4).

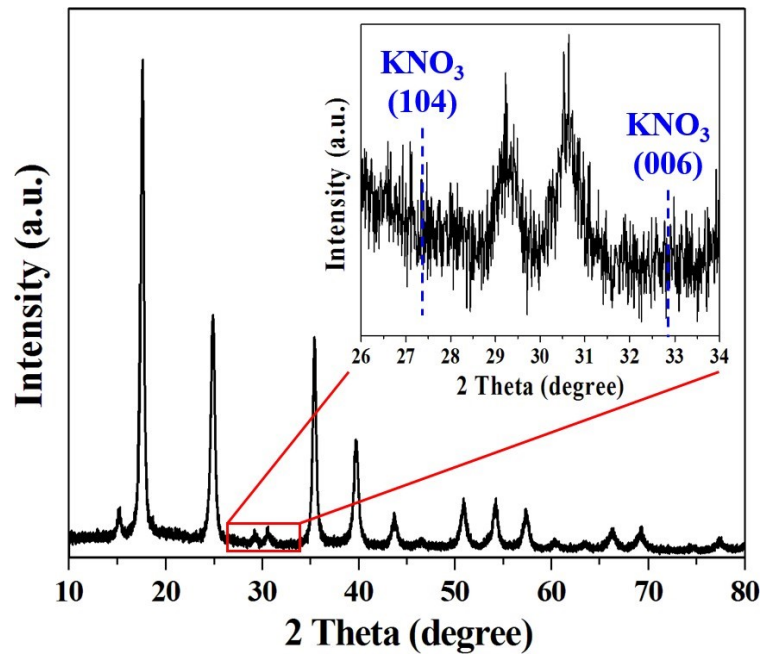
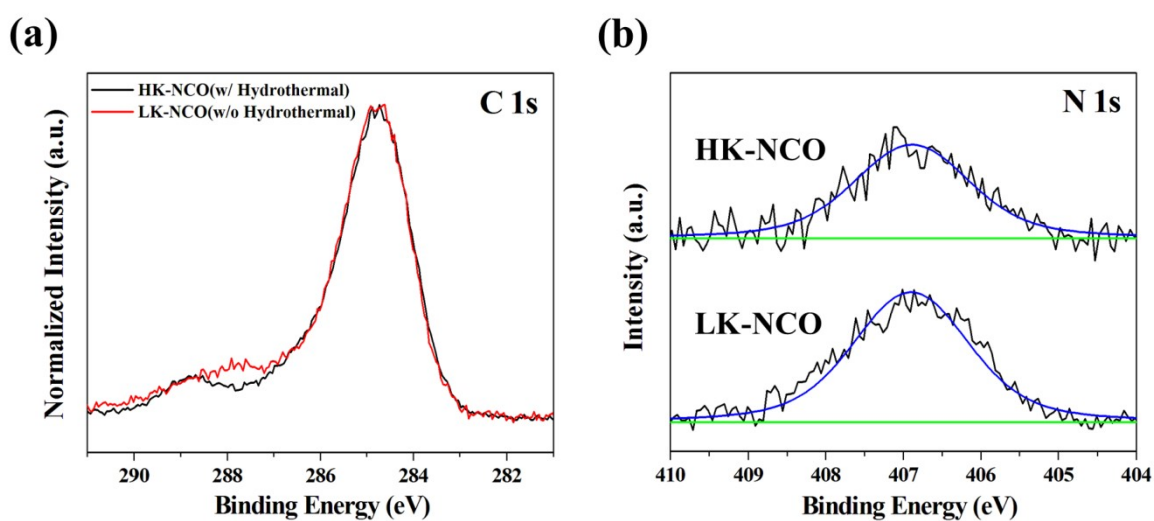


Figure S4. XRD pattern of hydrothermally synthesized K-Ni-Co-PBA. Inset shows an expansion of the region of interest, confirming the absence of crystalline KNO₃.



$$(c) \frac{N_{(area)}}{N_{(ASF)}} = \frac{K_{KNO_3}(area)}{K_{(ASF)}} = \frac{\frac{1}{3}O_{KNO_3}(area)}{O_{(ASF)}}$$

Measured in pure KNO_3

$$N_{(ASF)} = 4.506, K_{(ASF)} = 1.466, O_{(ASF)} = 3.023$$

ASF: Atomic Sensitivity Factor

Figure S5. (a) C 1s and (b) N 1s spectra of the synthesized catalysts (calibrated using the C 1s peak of adventitious carbon at 284.4 eV) (c) area calculation of elements derived from KNO_3 .

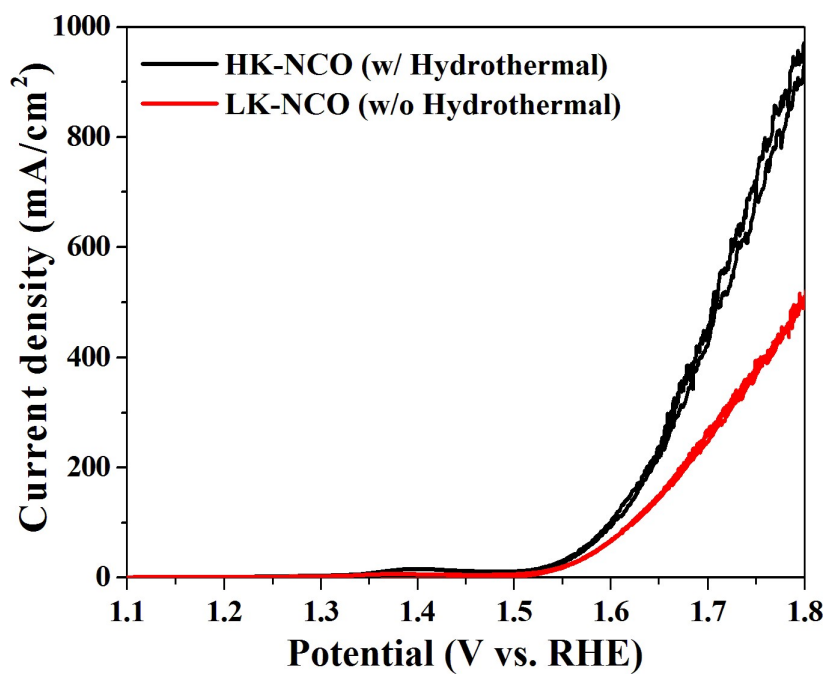


Figure S6. CV curves of the prepared catalysts recorded at a scan rate of 1 mV s^{-1} to extract overpotentials at 10 mA cm^{-2} and Tafel slopes.

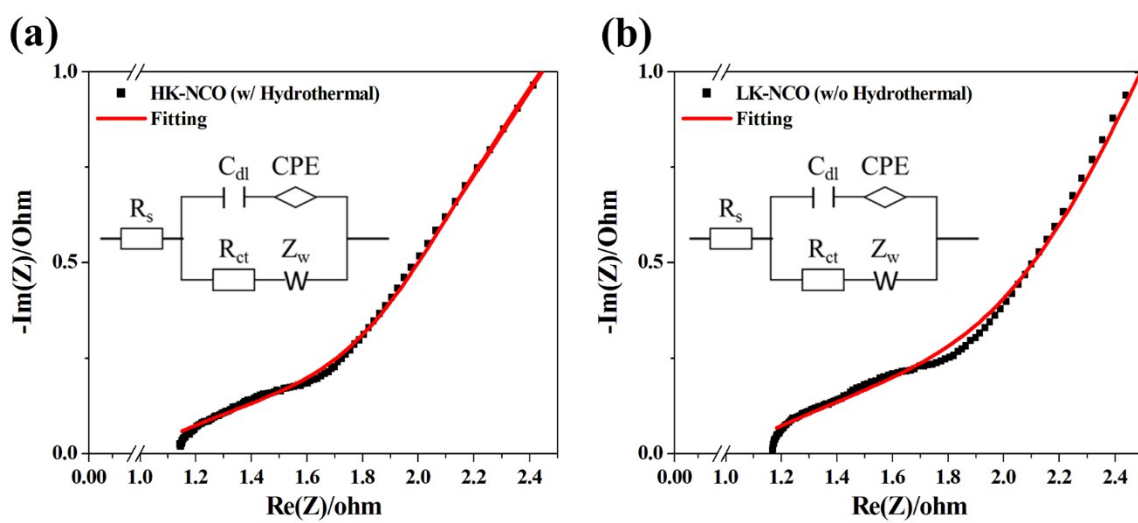


Figure S7. Nyquist plot fitting results for (a) HK-NCO and (b) LK-NCO.

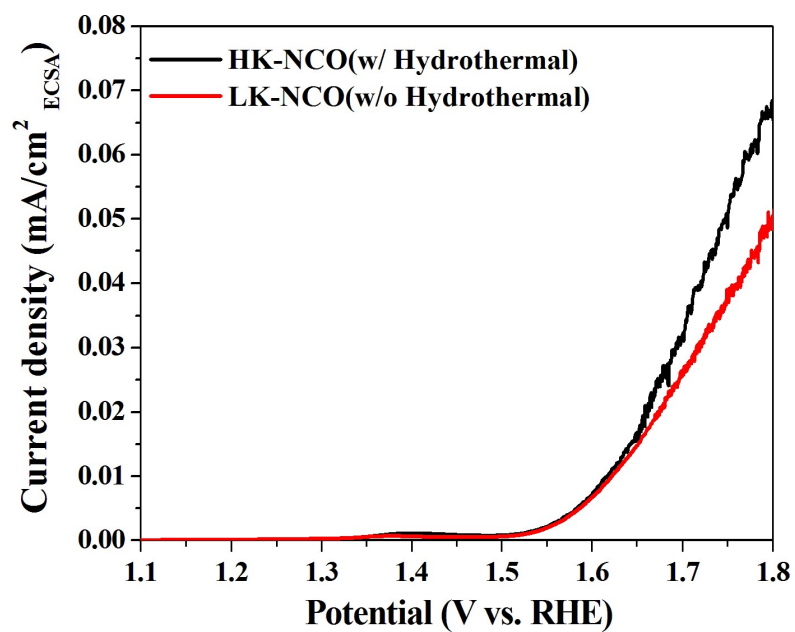


Figure S8. ECSA-normalized LSV polarization curves of the prepared catalysts recorded at a scan rate of 1 mV s^{-1} .

Table S1. Area of peaks corresponding to different chemical states in the X-ray photoelectron spectra of the synthesized catalysts.

Catalyst	Ni^{2+}	Ni^{3+}	Ni^{0}	Ni^{+}	O1	O2	O3	Ni^{2+}	Ni^{3+}	Ni^{0}	N
HK-NCO	15003.5	14218.2	11226.4	33370.5	23319.6	18842.2	24618.5	2713.2	2255.2	438.6	1348.1
LK-NCO	37949.5	26733.4	30128.4	79380.8	47448.7	24842.6	9111.2	3976.4	1663.3	642.8	1975.7

Table S2. Comparative overpotentials and Tafel slope values of electrocatalysts in this work and other literature.

Catalyst	Substrate	η (mV) at 10 mA cm ⁻²	Tafel slope (mV dec ⁻¹)	Electrolyte	Ref
K-doped NiCo ₂ O ₄ (HK-NCO)	Ni foam	292 (reverse current)	49.9	1 M KOH	This work
Mesoporous NiCo ₂ O ₄	KIT-6 (mesoporous silica molecular sieve)	350	43	1 M KOH	1
P-doped NiCo ₂ O ₄ Nanowire	Ni foam	300	120	1 M KOH	2
Ir-doped (10 at%) NiCo ₂ O ₄ Nanostructure	Glassy carbon electrode	303	78	1 M KOH	3
MOF-derived NiCo ₂ O ₄ /NiO-rGO	Glassy carbon electrode	340	66	1 M KOH	4
NiMn LDH nanosheets/NiCo ₂ O ₄ nanowires	Ni foam	310	99	1 M KOH	5
NiCo ₂ O ₄ 3-D nanoflowers	Graphene nanosheets	383	137	1 M KOH	6
3-D core-shell structured NiCo ₂ O ₄ @CoS	Ni foam	290	92	1 M KOH	7
Hierarchical NiCo ₂ O ₄ nanosheet-CNTs	Ni foam	390	68.1	1 M KOH	8
Co ₃ O ₄ / NiCo ₂ O ₄	Ni foam	320	84	0.1 M KOH	9
Hierarchical NiCo ₂ S ₄ nanosheets/rGO	Glassy carbon electrode	366	65	1 M KOH	10
Ni _{0.75} Cu _{0.25} Co ₂ O ₄	Graphite felt	509	119	1 M KOH	11

MOF (metal organic frameworks), 3-D (Three-dimensional), LDH (layered double hydroxide), CNT (carbon nanotubes), rGO (reduced graphene oxide)

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