

Supporting Information

Three dimensionally ordered mesoporous hydroxylated $\text{Ni}_x\text{Co}_{3-x}\text{O}_4$ spinels for oxygen evolution reaction: on the hydroxyl-induced surface restructuring effect.

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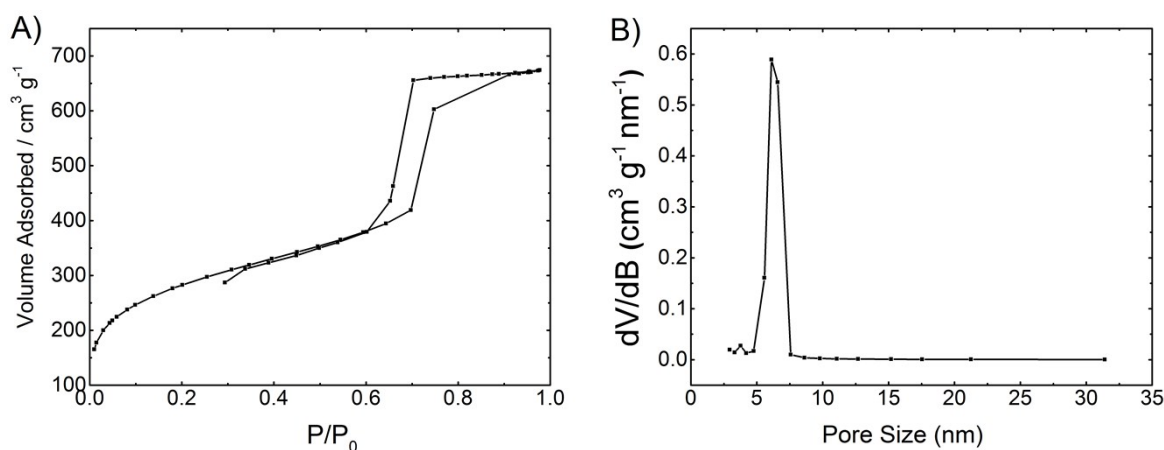


Figure S1. N_2 adsorption-desorption isotherm (A) and corresponding pore size distribution curve of KIT-6 silica template (B).

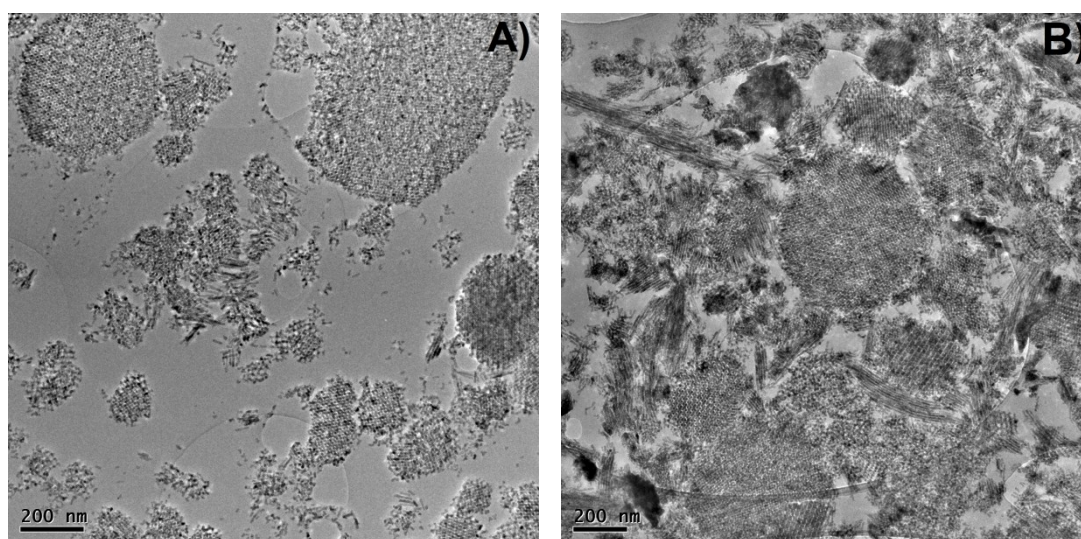


Figure S2. Low-magnification TEM images of as-prepared spinel oxides (A) $\text{Ni}_{0.18}\text{Co}_{2.82}\text{O}_4$, (B) $\text{Ni}_{1.00}\text{Co}_{2.00}\text{O}_4$.

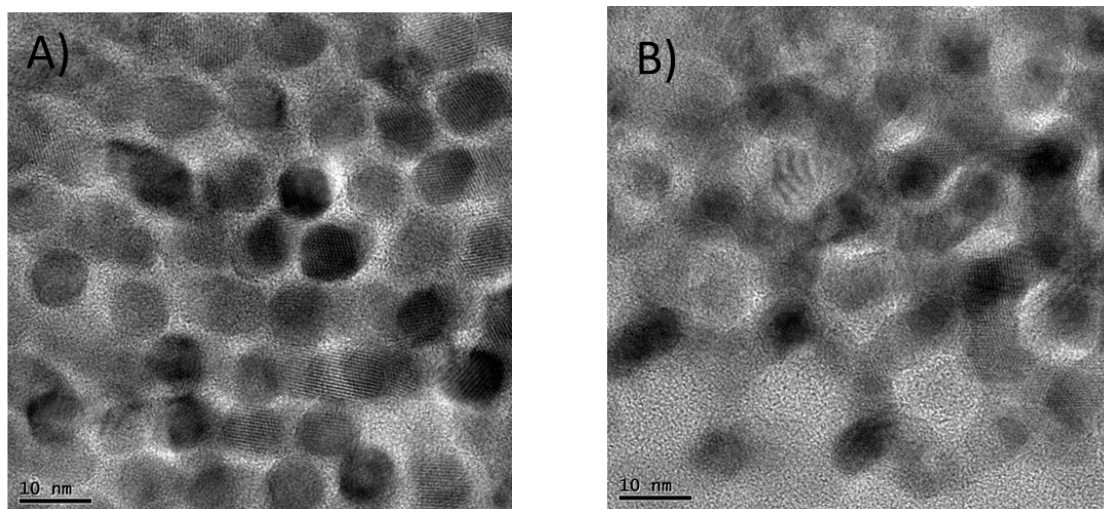


Figure S3. HRTEM images of Co_3O_4 and $\text{Ni}_{0.6}\text{Co}_{2.4}\text{O}_4$ catalysts

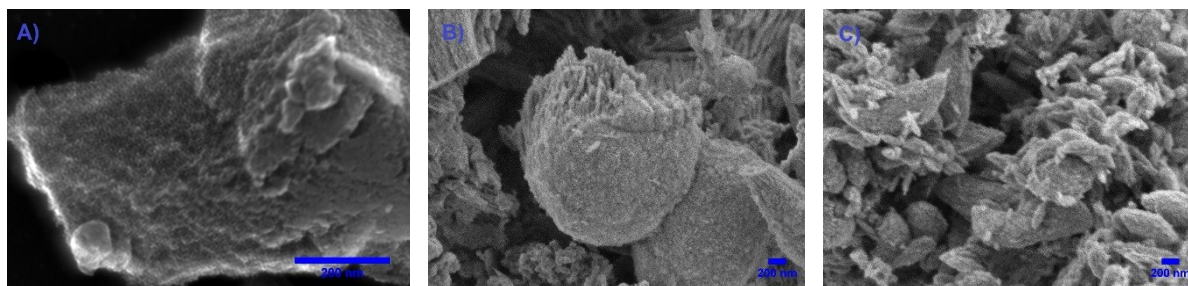


Figure S4. SEM images of (A) KIT-6, (B) Co_3O_4 and (C) $\text{Ni}_{0.6}\text{Co}_{2.4}\text{O}_4$ replicas

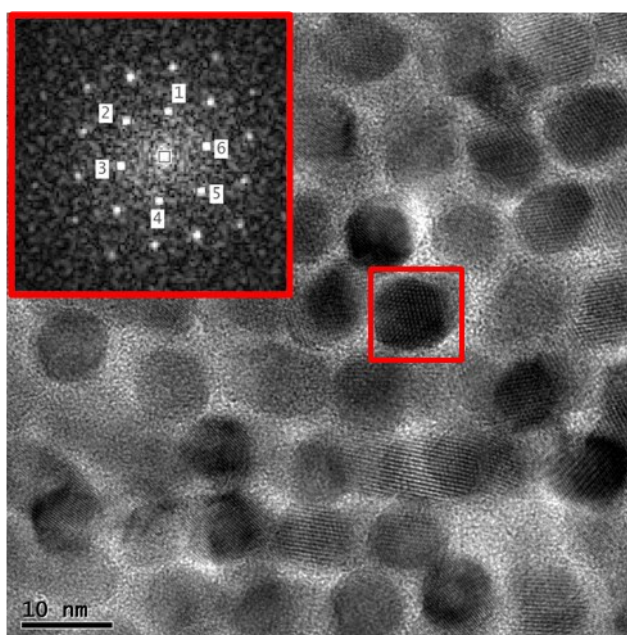


Figure S5: HRTEM image and Fourier transform of a selected area obtained on a Co_3O_4 single crystal. Zone axis : $\langle 0, 1, -1 \rangle$.

Table S6: d spacing corresponding to the spots indexed on Figure S5.

Spot number	d spacing (nm)
1	0.4635
2	0.4017
3	0.4642
4	0.4635
5	0.4017
6	0.4642

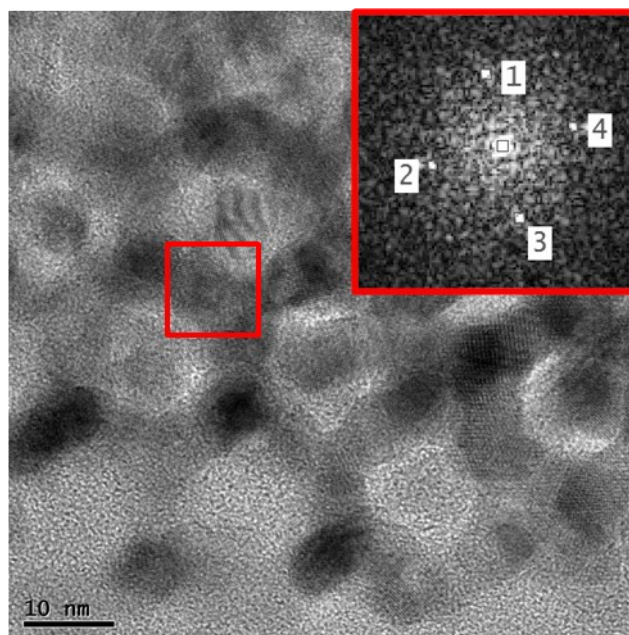


Figure S7: HRTEM image and Fourier transform of a selected area obtained on a NiCo₂O₄ single crystal. Zone axis: $\langle 1, 0, 0 \rangle$.

Table S8: d spacing corresponding to the spots indexed on Figure S6.

Spot number	d spacing (nm)
1	0.2815
2	0.2851
3	0.2815
4	0.2851

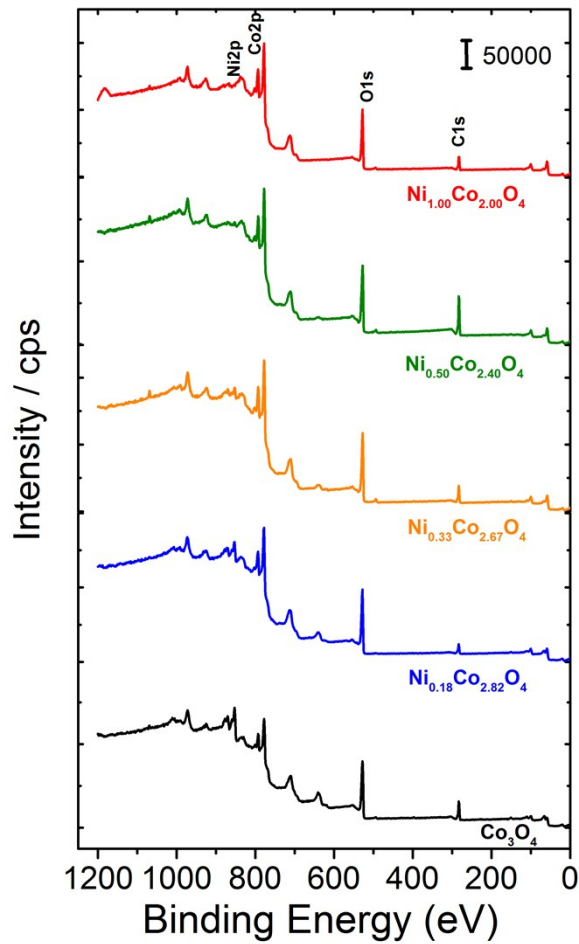


Figure S9. XPS survey spectra for the different catalysts.

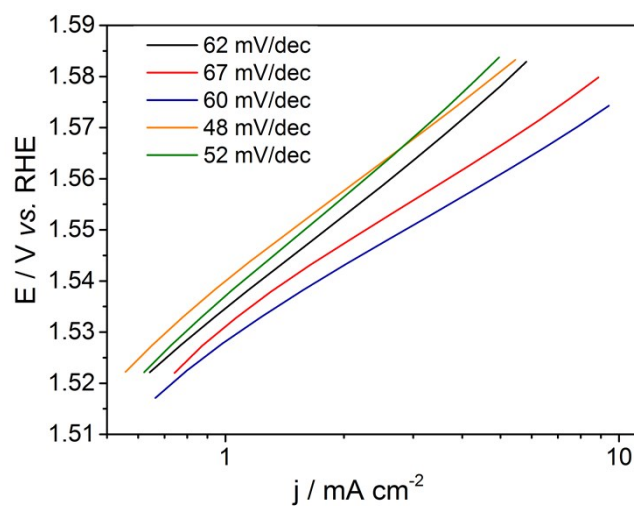


Figure S10. Tafel plots for the different catalysts. Co_3O_4 (black solid line), $\text{Ni}_{0.18}\text{Co}_{2.82}\text{O}_4$ (red), $\text{Ni}_{0.33}\text{Co}_{2.67}\text{O}_4$ (blue), $\text{Ni}_{0.60}\text{Co}_{2.40}\text{O}_4$ (orange), $\text{Ni}_{1.00}\text{Co}_{2.00}\text{O}_4$ (green).

Table S11. EIS fitting parameters for Ni_{0.60}Co_{2.40}O₄ obtained from the equivalent circuit presented on Scheme 1.

at 1.3V vs. RHE	R _{cell} / Ω	C _{dl} / F	p _{dl}	R _{ct} / Ω	C _{ads} / F	P _{ads}	k _{ET} / s ⁻¹	D
Before cycling	7.83	1.13 10 ⁻⁴	0.621	38.6	1.59 10 ⁻³	0.930	8.14	2.61
After cycling	7.48	3.24 10 ⁻⁵	0.693	91.1	2.34 10 ⁻³	0.823	2.34	2.44