Supplementary material

Oxidative Thermal Conversion of Hydrothermal Derived Precursors toward the Mixed-Metal Cobaltite Spinel Oxides ($ZnCo_2O_4$ and $NiCo_2O_4$): In-Situ Investigation by Synchrotron-Radiation XRD and XAS techniques

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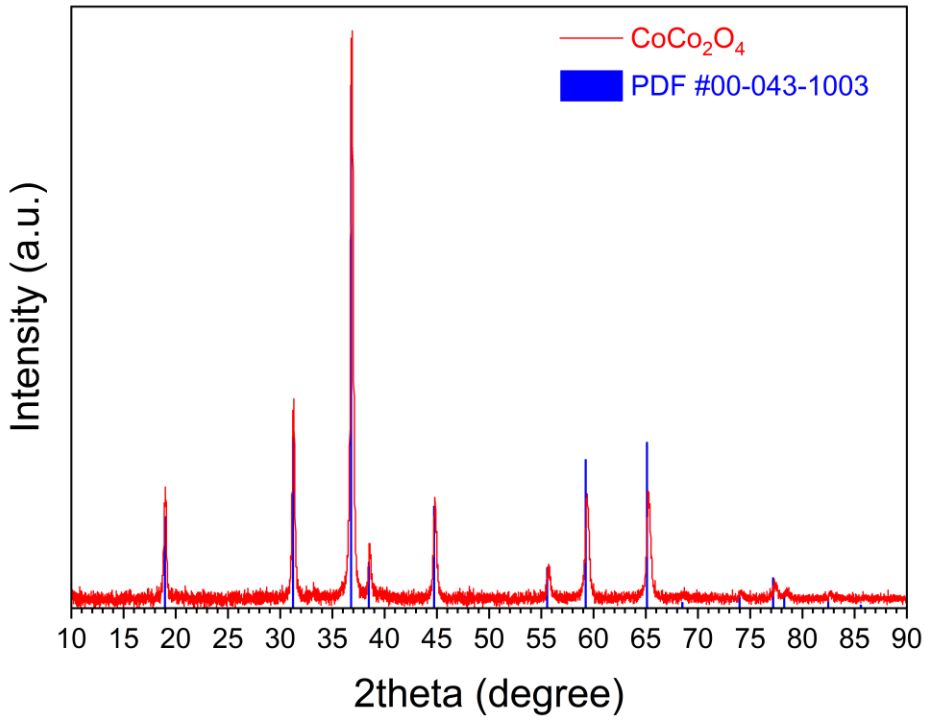

Figure S1. XRD profile of $CoCo_2O_4$ spinel oxide sample prepared by calcination of the CCO precursor at 480 °C, 10 hours, under air atmosphere (heating rate = 1 °C·min^{-1}).

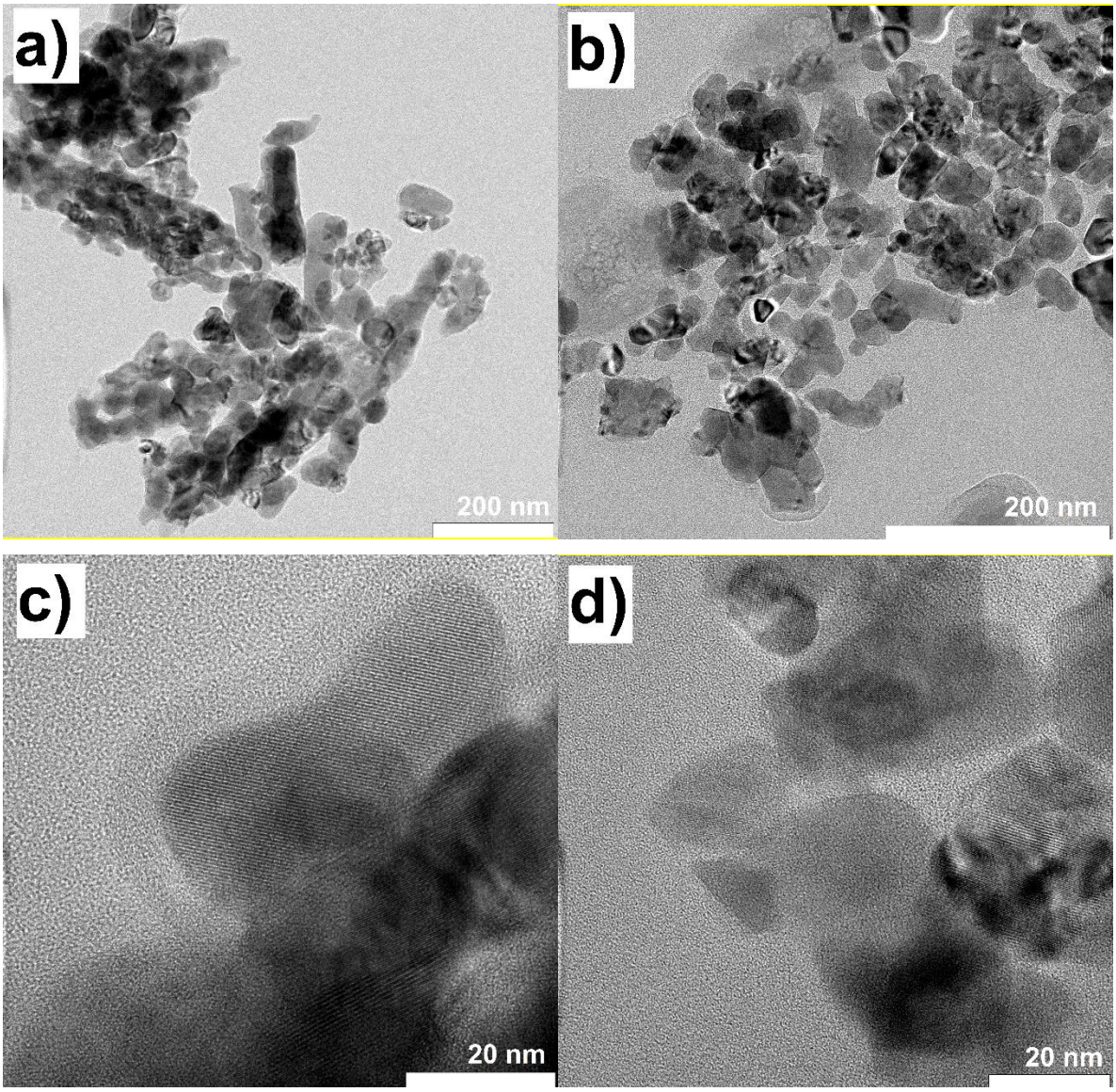

Figure S2. TEM images of the (a, c) $ZnCo_2O_4$ and (b, d) $NiCo_2O_4$ spinel oxides obtained by field-emission transmission electron microscope (FETEM, JEOL-JEM-3100F, operating voltage = 300 kV). The spinel oxide sample was prepared by calcining the prepared hydrothermal derived ZCO and NCO precursors at 480 °C in muffle furnace, under air flow for 10 hours (heating rate = 1 $°C \cdot min^{-1}$).

Table S1. XRD peak positions of the obtained product (at room temperature) after the thermal conversion of the CCO precursor.

2θ (°)	Lattice spacing (Å)	h	k	l	Space group	Crystalline phase
12.71	4.6661	1	1	1	Fd-3m	[a]$CoCo_2O_4$
20.84	2.8569	2	2	0	Fd-3m	[a]$CoCo_2O_4$
24.29	2.4551	1	1	1	Fm-3m	[b]CoO
24.50	2.4349	3	1	1	Fd-3m	[a]$CoCo_2O_4$
25.61	2.3311	2	2	2	Fd-3m	[a]$CoCo_2O_4$
28.10	2.1283	2	0	0	Fm-3m	[b]CoO
29.66	2.0186	4	0	0	Fd-3m	[a]$CoCo_2O_4$
36.56	1.6470	4	2	2	Fd-3m	[a]$CoCo_2O_4$
38.85	1.5535	5	1	1	Fd-3m	[a]$CoCo_2O_4$
40.17	1.5042	2	2	0	Fm-3m	[b]CoO
42.46	1.4267	4	4	0	Fd-3m	[a]$CoCo_2O_4$

[a]PDF #00-043-1003, [b]PDF #00-048-1719

Table S2. XRD peak positions of the obtained product (at room temperature) after the thermal conversion of the ZCO precursor.

2θ (°)	Lattice spacing (Å)	h	k	l	Space group	Crystalline phase
12.71	4.6661	1	1	1	Fd-3m	[c]$ZnCo_2O_4$
20.84	2.8563	2	2	0	Fd-3m	[c]$ZnCo_2O_4$
24.49	2.4361	3	1	1	Fd-3m	[c]$ZnCo_2O_4$
25.59	2.3331	2	2	2	Fd-3m	[c]$ZnCo_2O_4$
29.64	2.0200	4	0	0	Fd-3m	[c]$ZnCo_2O_4$
36.56	1.6471	4	2	2	Fd-3m	[c]$ZnCo_2O_4$
38.84	1.5538	5	1	1	Fd-3m	[c]$ZnCo_2O_4$
42.45	1.4269	4	4	0	Fd-3m	[c]$ZnCo_2O_4$

[c]PDF 00-023-1390

Table S3. XRD peak positions of the obtained product (at room temperature) after the thermal conversion of the NCO precursor.

2θ (°)	Lattice spacing (Å)	h	k	l	Space group	Crystalline phase
12.72	4.6635	1	1	1	Fd-3m	[d]$NiCo_2O_4$
20.83	2.8583	2	2	0	Fd-3m	[d]$NiCo_2O_4$
24.46	2.4389	3	1	1	Fd-3m	[d]$NiCo_2O_4$
25.61	2.3310	2	2	2	Fd-3m	[d]$NiCo_2O_4$
29.63	2.0204	4	0	0	Fd-3m	[d]$NiCo_2O_4$
36.50	1.6497	4	2	2	Fd-3m	[d]$NiCo_2O_4$
38.84	1.5538	5	1	1	Fd-3m	[d]$NiCo_2O_4$
42.38	1.4292	4	4	0	Fd-3m	[d]$NiCo_2O_4$

[d]PDF 01-073-1702