Supplementary information

Ni, Co hydroxide triggers electrocatalytic production of high-purity benzoic acid over 400 mA cm^{-2}

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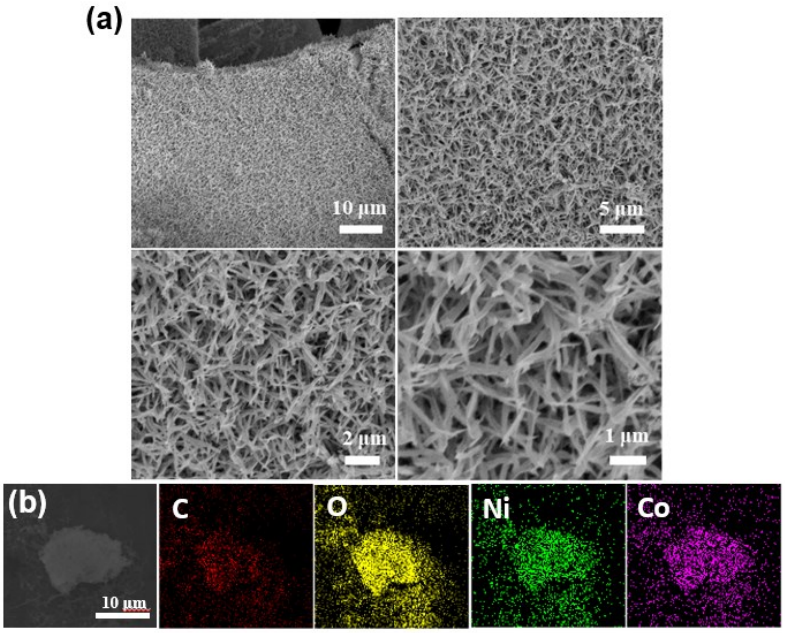

Figure S1. (a) The SEM images of Ni-Co-CH/NF precursor. (b) SEM-EDX mapping of Ni-Co-CH/NF precursor. The powders on the surface of Ni-Co-CH/NF were obtained by ultrasonic treatment in the alcohol, and drop-casted on the aluminum foil to prevent the influence of the Ni atom from NF.

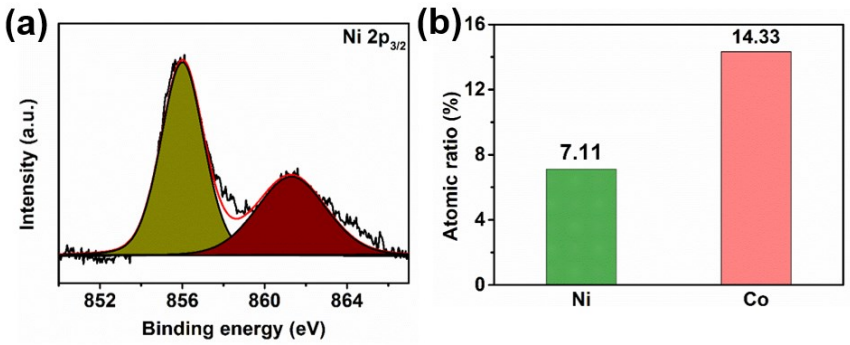

Figure S2. (a) The high-resolution XPS spectrum of the bottom products after the hydrothermal synthesis. (b) The atomic content of Ni and Co in the bottom products.

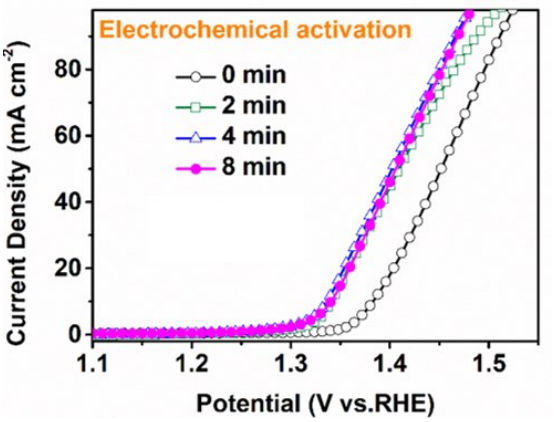

Figure S3. The LSV curves of the activated materials prepared by electrochemical activating the Ni-Co-CH/NF at a constant potential of 1.5 V vs. RHE for 0 min, 2 min, 4 min, and 8 min in the mixed 1 M KOH and 0.1 M BA solutions.

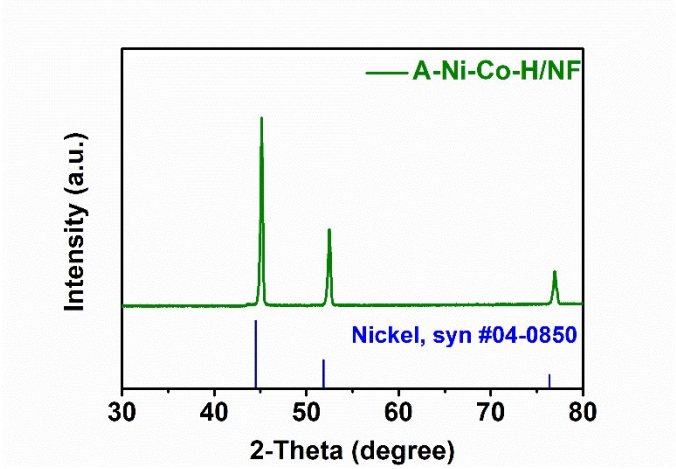

Figure S4. The XRD pattern of A-Ni-Co-H/NF.

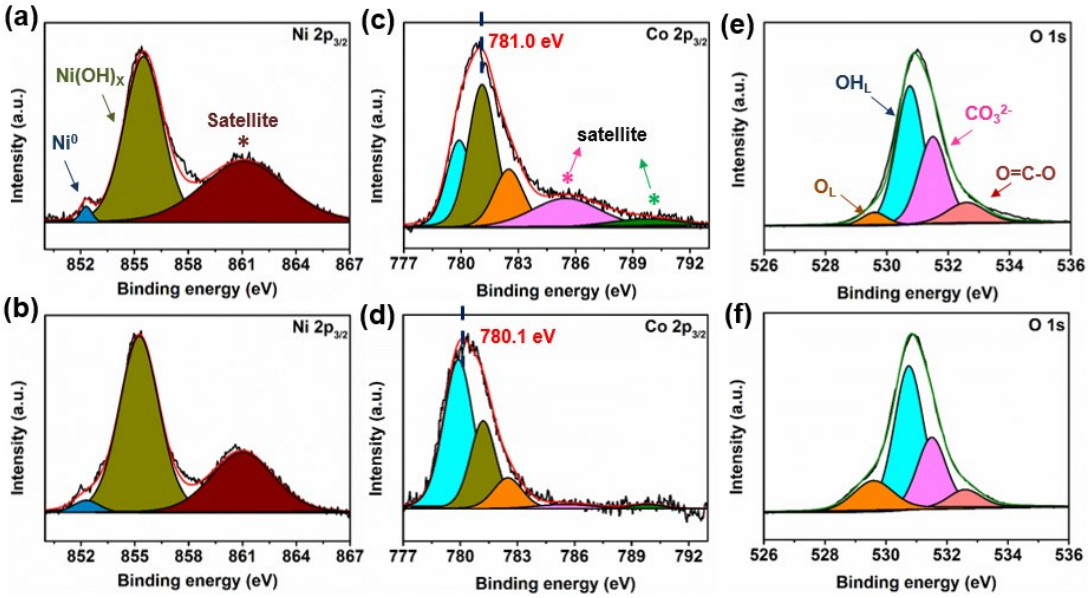

Figure S5. The high-resolution XPS spectra of Ni 2p, Co 2p and O 1s for Ni-Co-CH/NF (a, c, e) and A-Ni-Co-H/NF (b, d, f).

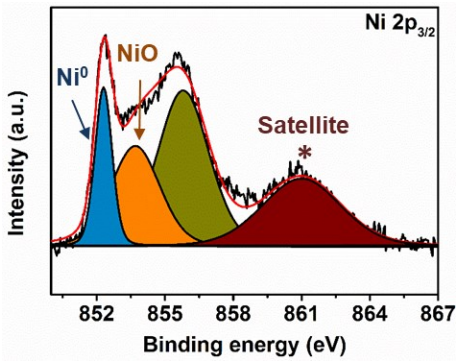

Figure S6. The high-resolution XPS spectrum of Ni 2p for NF.

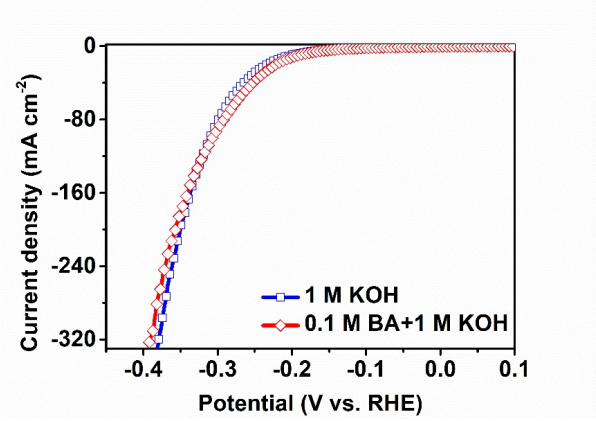

Figure S7. The LSV curves of the A-Ni-Co-H/NF as HER catalyst at the electrolyte of 1 M KOH with or without the adding of 0.1 M BA.

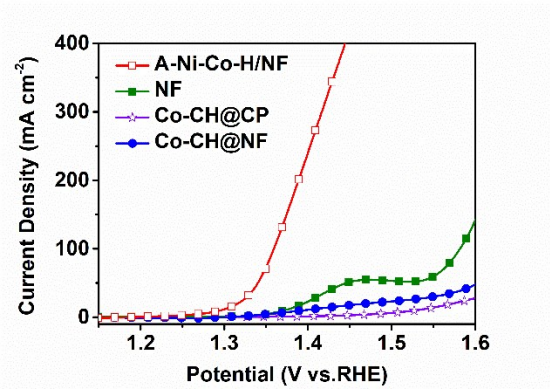

Figure S8. LSV curves of A-Ni-Co-H/NF, Co-CH@CP, Co-CH@NF, and NF in 1 M KOH mixed with 0.1 M BA solutions.

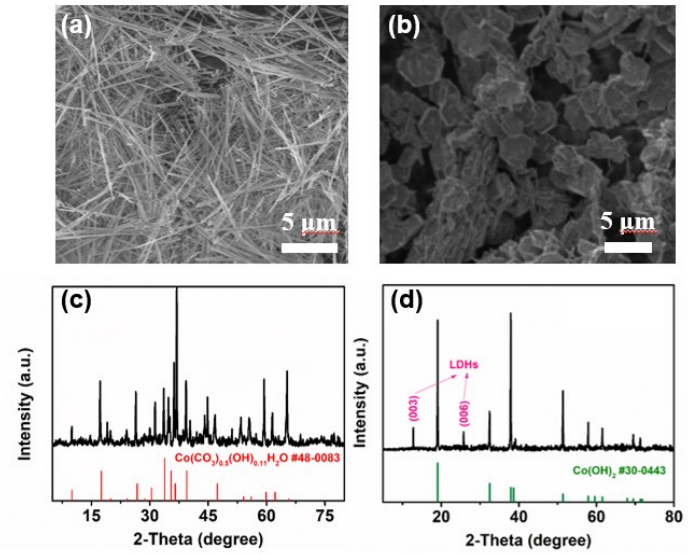

Figure S9. The SEM images of Co-CH powders (a) and (b) Co-CH after being soaked in 1 M KOH for 30 min. The XRD patterns of the (c) Co-CH and (d) Co-CH after being soaked in 1 M KOH for 30 min.

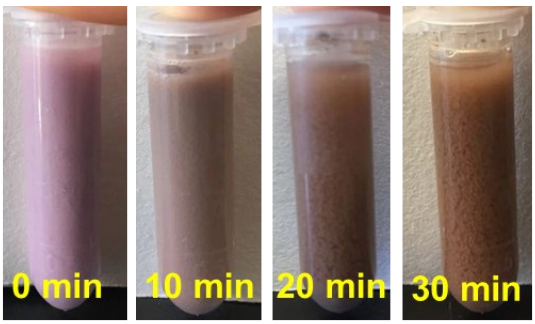

Figure S10. The digital images of Co-CH after being soaked in the 1 M KOH solution for 0 min, 10 min, 20 min and 30 min.

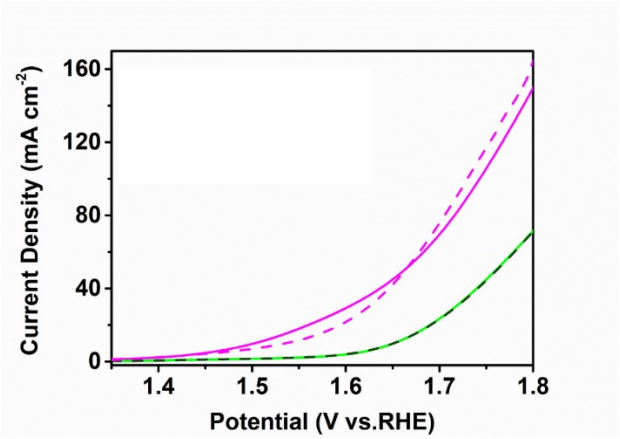

Figure S11. The LSV curves of Co-CH (pink solid line) and Co-CH after being soaked in 1 M KOH (green solid line) at a scan rate of 5 mV s^{-1}. Dash line represents electrochemical activated samples for 10 min in 1 M KOH containing 0.1 M BA electrolyte.

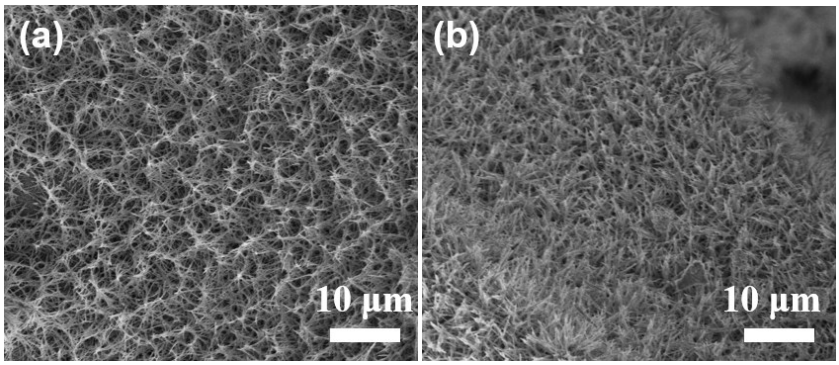

Figure S12. The images of (a) the Ni-Co-CH/NF and (b) the Ni-Co-CH/NF soaked in 1 M KOH solution for 30 min.

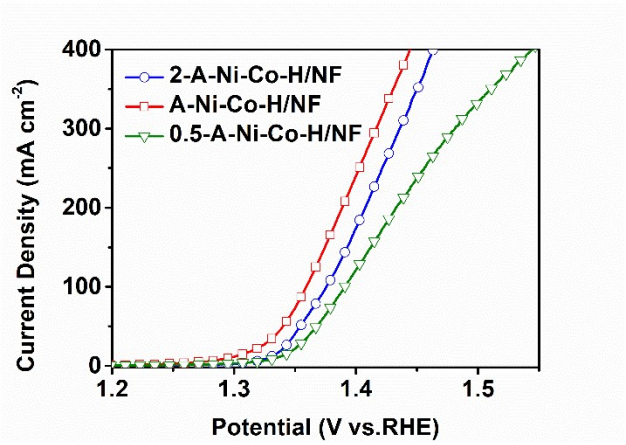

Figure S13. The LSV curves of the series of A-Ni-Co-H/NF eletrocatalysts synthesized with different concentrations of Co^{2+}. Red line represents the core/target electrocatalyst, the blue and green lines represent the corresponding electrocatalysts prepared by 2-fold and 0.5-fold Co^{2+} source, respectively.

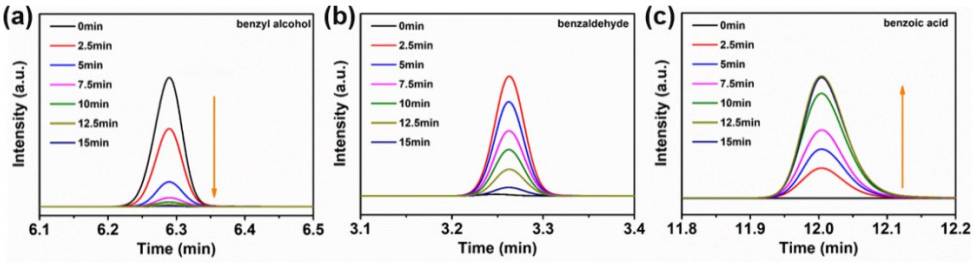

Figure S14. (a) GC chromatogram traces of the BA (a), Ph-CHO (b), and Ph-COOH (c) at different reaction time following the BA electrochemical oxidation reaction, in 1 M KOH with 0.1 M BA electrolyte, catalyzed by A-Ni-Co-H/NF.

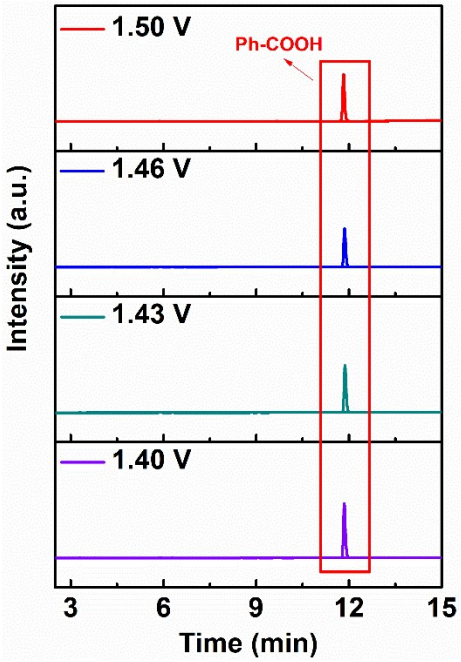

Figure S15. GC chromatograms of electrocatalytic products on the basis of the EBA reaction at different applied potentials ranging from 1.40 to 1.50 V vs.RHE.

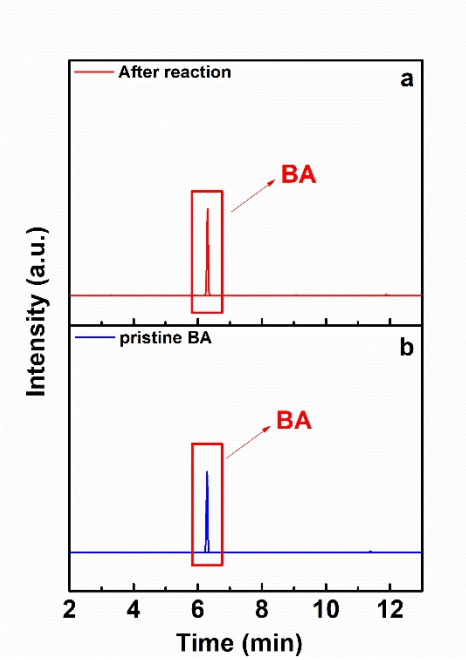

Figure S16. (a) GC chromatogram traces of the products for the benzyl alcohol oxidation reaction catalyzed by A-Ni-Co-H/NF without the input of electricity, in 1 M KOH with 0.1 M BA electrolyte. (b) The GC chromatogram of pristine 0.1 M BA.

The energy balance calculation

$$I = jS \tag{1}$$

Where I is the electrolysis current (A), j is the current density (A cm^{-2}), S is the area of anodic electrode (cm^2), which is 1 cm^2 here.

$$Q=It \tag{2}$$

$$n_e = \frac{Q}{N_A q_e} \tag{3}$$

Where n_e is the electron-transfer numbers and t is the time of electrolysis (1 h), N_A is Avogadro constant, and q_e is the charge number of single electron.

$$m_{H_2} = \frac{1}{2} n_e M_{H_2} \tag{4}$$

$$W_{H_2} = m_{H_2} E_{H_2} \tag{5}$$

Where m_{H_2} is the mass of produced H_2, M_{H_2} is the relative molecular mass and E_{H_2} is the energy density of H_2 (33500 Wh kg^{-1})

$$W_E = IUt \quad (6)$$

Where U is the applied voltage that is extracted from the polarization curves in Fig. 3a.

Table S1. The ICP measurement results of the reaction solution in hydrothermal reactor.

Element	Mean
Co	7.142 mg/L
Ni	62.00 mg/L

Table S2. The comparison for the value of i_M between A-Ni-Co-H/NF and the other ECO electrocatalysts reported recently.

Electrocatalyst	**Reactant**	**i_M (mA cm^{-2})**	**Reference**
Co_3O_4 NWs/Ti	10 mM BA	2	9
Nanocrystalline Cu foam	5 mM HMF	4	31
NC@$CuCo_2N_x$/CF	15 mM BA	25	8
M-$Ni(OH)_2$ nanosheet	0.33 M urea	35	4
Ni-Mo-N/CFC	0.1 M glycerol	100	3
Ni_2P porous nanosheet	0.5 mmol THIQs	135	23
Ni_3N@C	10 mM HMF	200	22
$CuCo_2O_4$	50 mM HMF	220	30
NiClO-D	0.33 M urea	105	12
NiFe-LDH	10 mM HMF	40	19
A-Ni-Co-H/NF	**0.1 M BA**	**>400**	**our work**

Table S3. Electrocatalytic results for oxidation of BA reaction at different applied potentials using A-Ni-Co-H/NF as electrocatalyst.

Potential	Conversion (%)	Selectivity (%)	
(V vs.RHE)	BA	Ph-CHO	Ph-COOH
1.40	99.7	0.1	99.6
1.43	99.8	0.1	99.7
1.46	99.8	0.2	99.6
1.50	99.7	0.1	99.6