

Supplementary Information

CuCoO₂ nanocrystals derived from an isopropanol modified

Cu-BTC using in alkaline water oxidation

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Experimental details

CuCoO₂ derived from Cu-BTC-IPA (Cu-BTC)

All chemicals in the experiment were of analytical grade without further purification. Firstly, 2.61 g Cu(NO₃)₂·3H₂O, 1.26 g H₃BTC and isopropyl alcohol (IPA) were dissolved in 27 mL deionized solution (DI) and 36 mL absolute ethanol (ET), and magnetically stirring for 30 minutes. Secondly, the solution was transferred into a 100 mL Teflon lined autoclave and kept at 120 °C for hydrothermal reaction for 12 h. Afterwards, the resulting blue precipitate was washed three times with ET to remove any by-product impurities. Finally, the Cu-BTC-IPA was dried at 70 °C for 5h for further characterization. For comparison, we prepared Cu-BTC without IPA and named it Cu-BTC.

And then, 0.20 g Cu-BTC-IPA (or Cu-BTC) was used as Cu source, 0.16 g Co(NO₃)₂·6H₂O as Co source, 5.40 g NaOH as the mineralizing agent, dissolving the three in 20 mL DI and 50 mL ET, and magnetically stirring for 30 minutes. Then transfer the solution to a 100 ml Teflon lined autoclave and kept at 140 °C for hydrothermal reaction for 24 h. The obtained precipitate was washed several times with diluted ammonia, DI, and ET, and then dried at 70 °C for 5 h for further characterization. For a better comparison, CuCoO₂ derived from Cu-BTC and Cu-BTC-IPA are named CCO1 and CCO2, respectively.

Structural characterization of CuCoO₂

The crystal structures of these as-obtained Cu-BTC and CuCoO₂ powder were analyzed by X-ray diffraction (XRD D8 Advance). The morphology, microstructure, and chemical composition of the samples were examined by field-emission scanning electron microscopy (SEM Zeiss sigma300) and transmission electron microscopy (TEM, FEI Tecnai G2 F30) equipped with energy-dispersive X-ray spectroscopy (EDX). The Brunauer–Emmett–Teller (BET) specific surface areas and porosity parameters of these samples were measured by N₂ adsorption desorption isothermometry (Micromeritics TriStar II 3020 3.02). Analysis of molecular structure of Cu-

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BTC by Fourier transform infrared spectroscopy (FT-IR Nexus). The structure of CuCoO₂ was investigated by Raman (LABHRev-UV). The surface chemical states of CuCoO₂-based powders and working electrodes were analyzed by X-ray photoelectron spectroscopy (XPS, Thermo Escalab 250Xi).

Electrode preparation and electrochemical measurement

Firstly, 15.0 mg of CuCoO₂ was dissolved in 500 μL DI, 480 μL IPA and 20 μL Nafion to make a suspension, then 20 μL of the suspension was placed on Ni foam (1 cm×1 cm) through drop casting method, and finally the working electrode was obtained by drying at 150 °C for 10 min. The working electrodes prepared with CCO1 and CCO2 were named as Ni@CCO1 and Ni@CCO2, respectively.

The OER performance was tested by cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) in a three-electrode configuration. The electrochemical workstation used was CS2350H Electrochemical workstation (Wuhan Corrtest Instruments Corp., China.), and 1.0 M KOH was used as electrolyte, platinum electrode as counter electrode, and calomel electrode as reference electrode. In the range of 1.05-1.80 V, cyclic voltammetry scan of reversible hydrogen electrode (scan rate: 5 mV·s⁻¹) was performed. In the non-faradaic potential window of -0.05~0.05 V vs. SCE, the electrochemical double-layer capacitance (C_{dl}) was obtained by cyclic voltammetry at different rates (from 20 to 100 mV·s⁻¹). The EIS measurements were performed in the frequency range of 100 kHz-10 mHz under a constant potential of 1.63 V vs. RHE.

All current density values were normalized with respect to the geometrical surface area of the working electrode. All CV curves presented in this work were *iR*-corrected (85%). The correction was done according to the following equation:

$$E_c = E_m - iR_s \quad (1)$$

where E_c is the *iR*-corrected potential, E_m is the experimentally measured potential, and R_s is the equivalent series resistance extracted from the Zero-Input Response (ZIR) measurements. Unless otherwise specified, all potentials were reported versus the RHE by converting the potentials measured vs. SCE according to the following

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formula:

$$E \text{ (RHE)} = E \text{ (SCE)} + 0.241 + 0.059 \text{ pH} \quad (2)$$

Supplementary Figures:

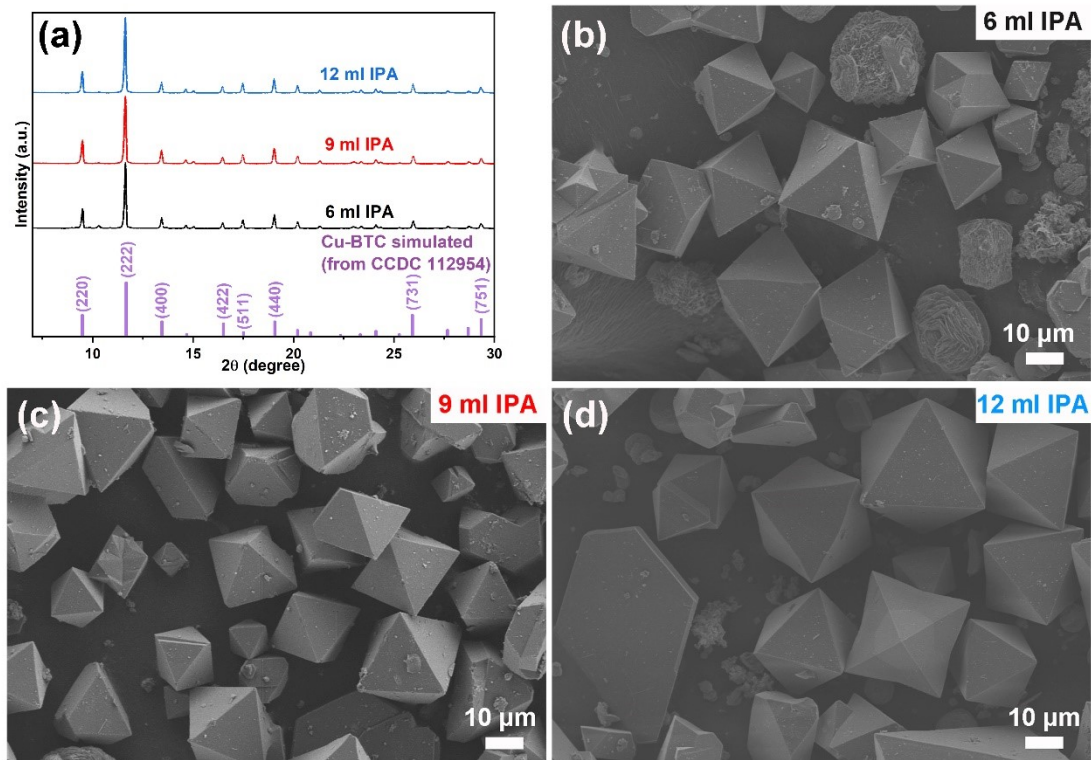


Fig. S1 XRD patterns (a) and SEM images (b-d) of Cu-BTC by adding different amounts of IPA.

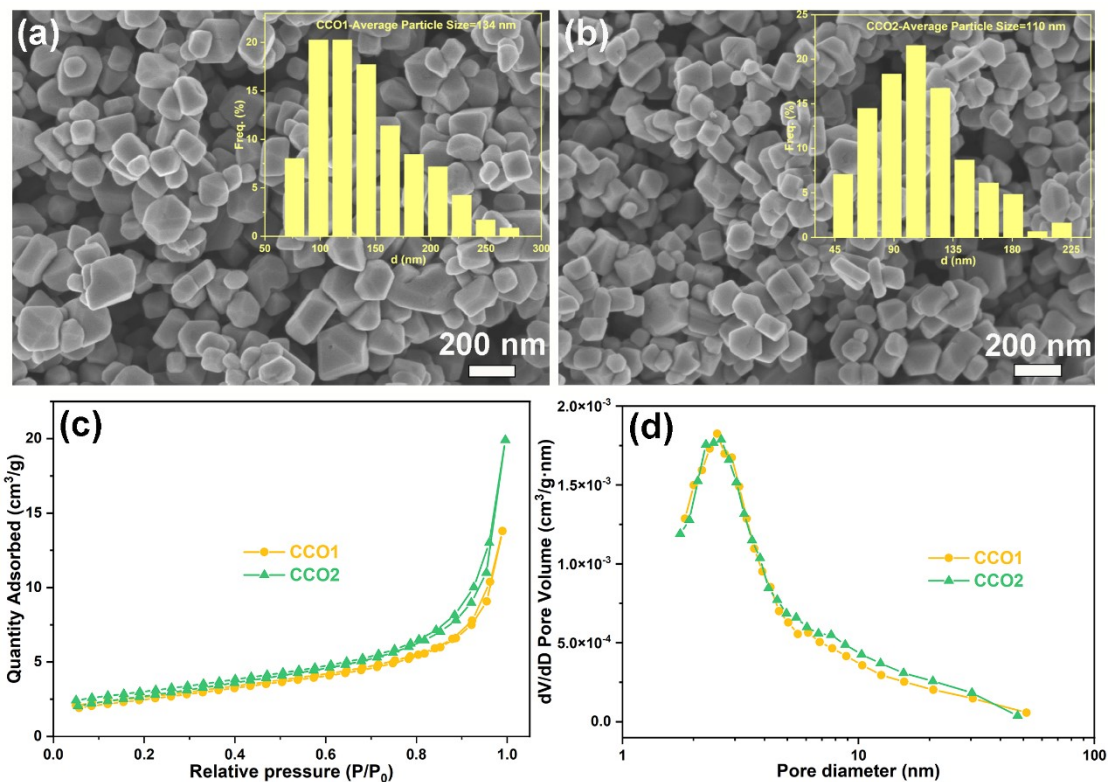


Fig. S2 SEM images and particle size distribution (a, CCO1; b, CCO2), N_2 adsorption-desorption isotherm (c) and BJH pore size distribution curve (d) of CCO1 and CCO2.

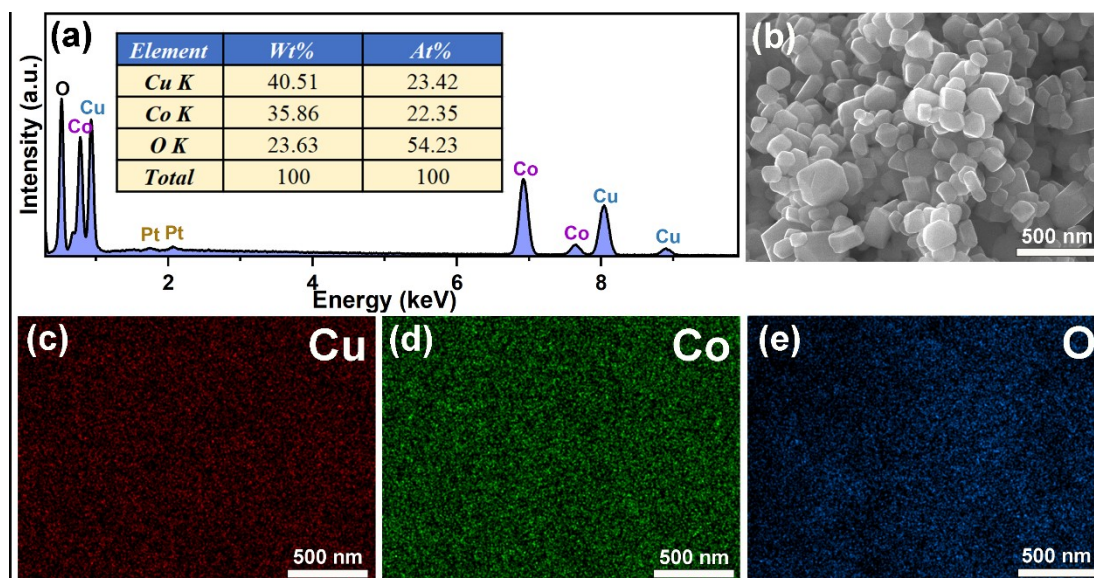


Fig. S3 Elemental analysis report (a), SEM image (b), EDS elemental mappings (c-e) of CCO1.

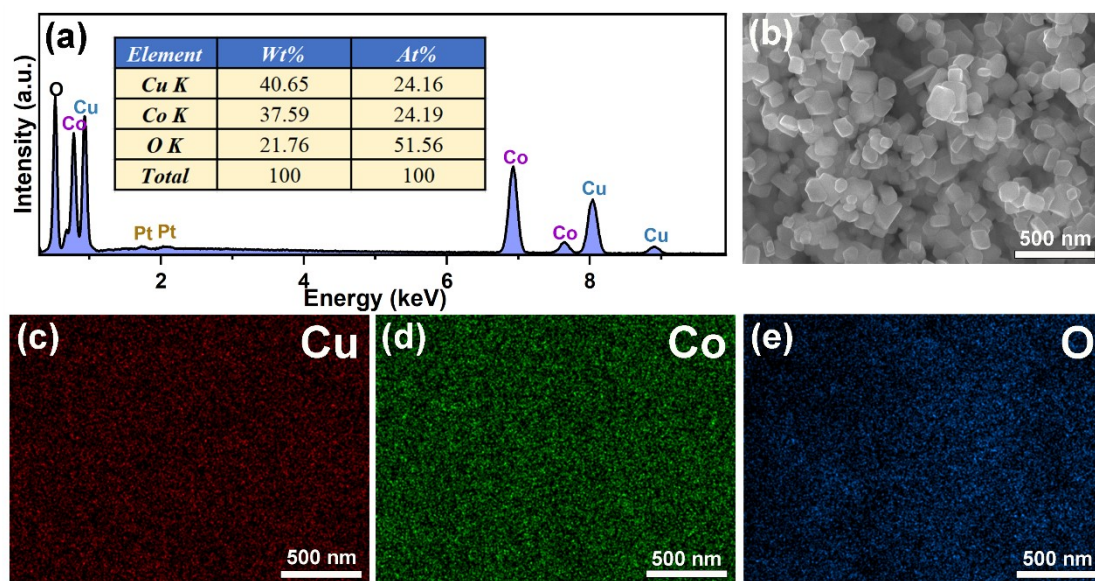


Fig. S4 Elemental analysis report (a), SEM image (b), EDS elemental mappings (c-e) of CCO₂.

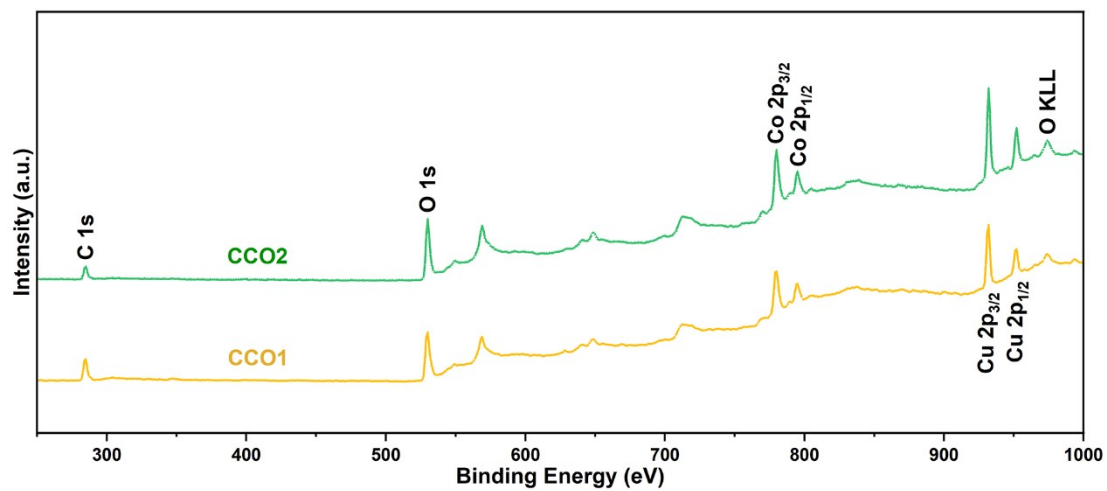


Fig. S5 XPS full spectrograms of CCO1 and CCO2.

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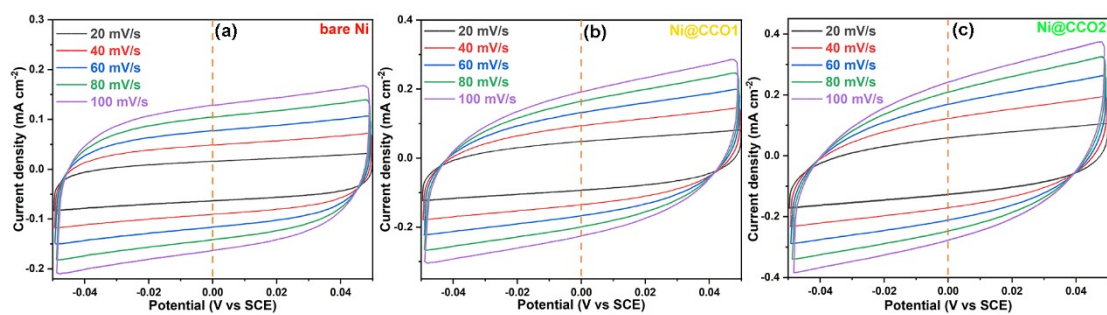


Fig. S6 CV curves of bare Ni, Ni@CCO1 and Ni@CCO2 at different sweep speeds.

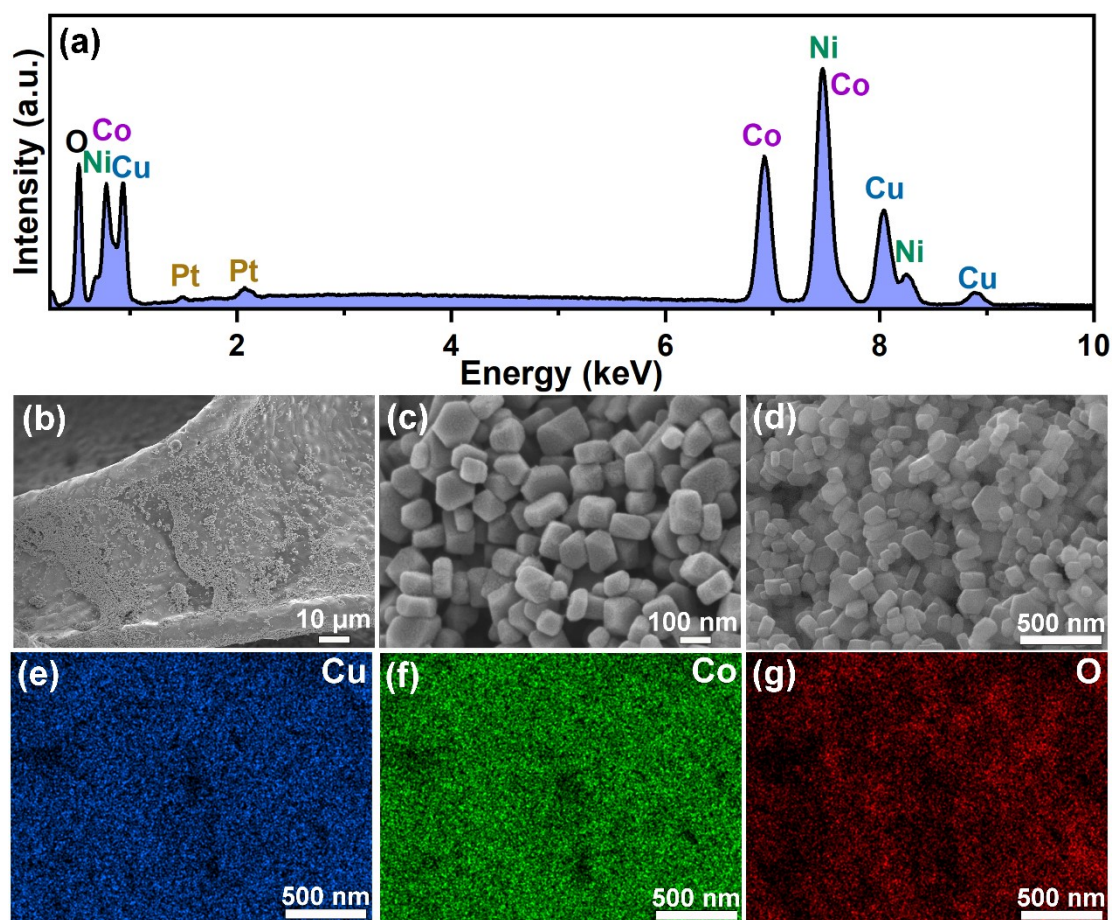


Fig. S7 Elemental analysis report (a), SEM map (b-d) and EDS elemental mappings (e-g) of Ni@CCO2 before OER stability test.

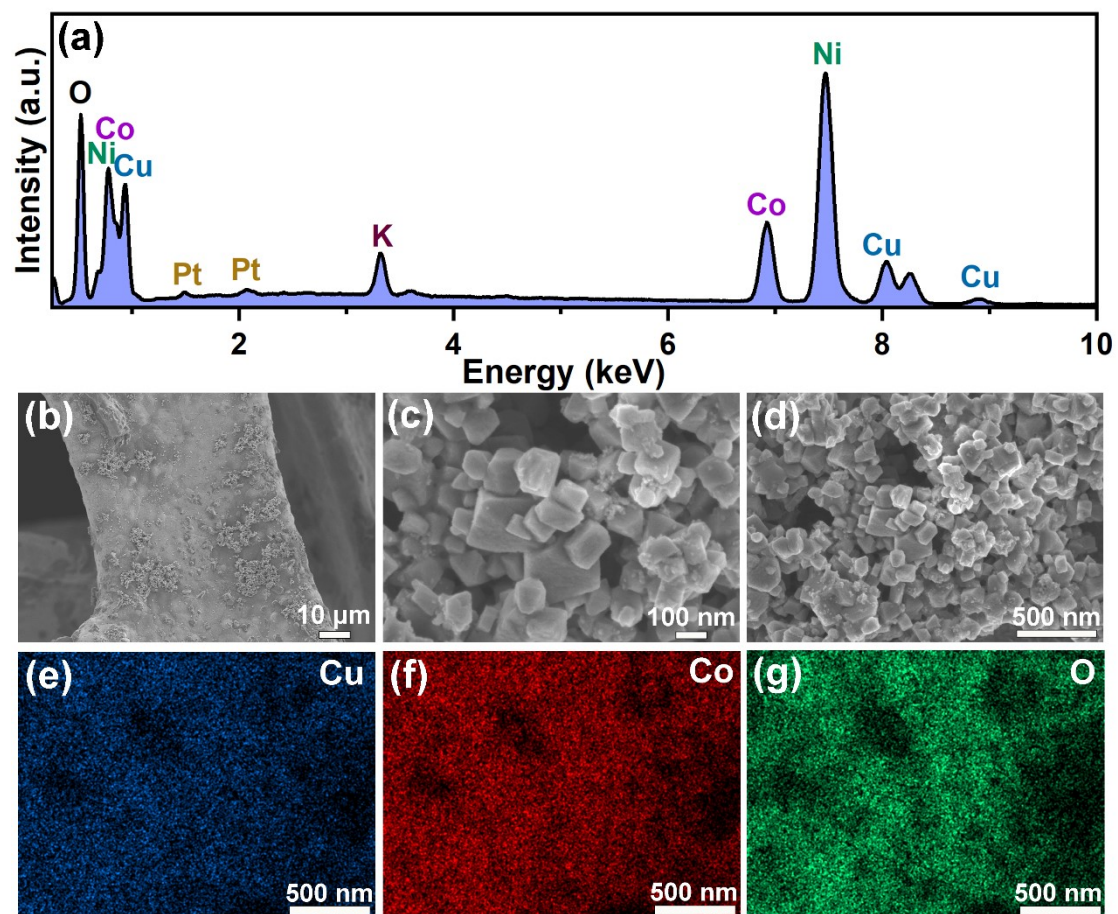


Fig. S8 Elemental analysis report (a), SEM map (b-d) and EDS elemental mappings (e-g) of Ni@CCO₂ after OER stability test.

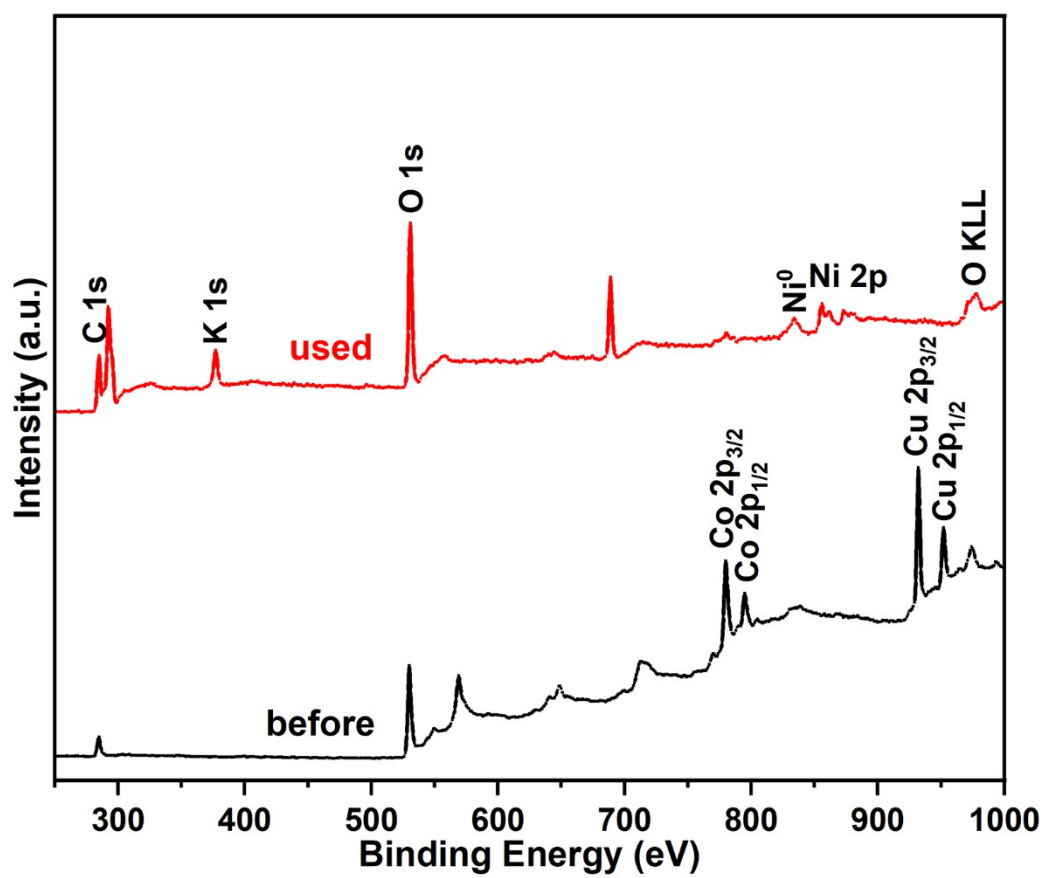


Fig. S9 XPS survey spectra of CCO₂ after 18 hours continuous OER stability test.