

Supporting Information

Phase-controllable synthesis of cobalt hydroxide for electrocatalytic oxygen evolution

Fenglei Lyu,^{ab} Yaocai bai,^b Qingfa Wang,^{a†} Li Wang,^a Xiangwen Zhang,^a and Yadong Yin^{b†}

a. School of Chemical Engineering and Technology, Key Laboratory for Green Chemical Technology of the Ministry of Education, Tianjin University, Tianjin 300350, China.

b. Department of Chemistry, Materials Science and Engineering program, and UCR Center for Catalysis, University of California, Riverside, CA 92521, USA

†Corresponding Author: Email Address: qfwang@tju.edu.cn, yadong.yin@ucr.edu, Fax: +1-951-827-4713

Experiment

Synthesis of Co(OH)₂-Cl

In a typical synthesis, CoCl₂ (2 mmol) and HMT (4 mmol) are dissolved in water (72 mL) and ethanol (8 mL) into a three-neck flask (100 mL). The above solution is refluxed h at 95 °C for 1 h. The product is collected by centrifugation and washed three times by water and ethanol, further dried at 60 °C for 12 h.

Synthesis of Co(OH)₂-NO₃ and Co(OH)₂-Ac

The synthetic procedures of Co(OH)₂-NO₃ and Co(OH)₂-Ac are similar with that of Co(OH)₂-Cl, except the addition of Co(NO₃)₂ and Co(Ac)₂, instead of CoCl₂.

Characterization

The morphology of the samples were investigated with transmission electron microscopy (TEM, Philips Tecnai 12, 200 kV). Powder XRD patterns were obtained with a Bruker D8 Advance Powder X-ray diffractometer using Cu K α radiation. UV-Vis absorption spectra were recorded with a Hitachi U-3010 spectrometer equipped with a 60 mm diameter integrating sphere using BaSO₄ as the reference.

Electrochemical characterization. All electrochemical measurements were carried out in a three-electrode cell at room temperature, with a Pt foil, a saturated calomel electrode (SCE), and 1.0 M KOH as the counter electrode, reference electrode, and electrolyte, respectively. For the preparation of working electrode, 5 mg catalysts is dispersed in 750 μL water, 250 μL isopropanol and 50 μL Nafion solution (5%) with the assistance of sonication for at least 30min. Then the dispersion is drop-casted on a polished glassy carbon electrode (3 mm in diameter) with loading amount of 0.28 mg/cm^2 . After drying at 60 $^\circ\text{C}$ for 5min, catalysts covered glassy carbon electrode is applied as working electrode. Before the electrochemical tests, cyclic voltammetry is conducted at a scan rate of 50 mV/s for at least 20 cycles with the potential range from 0 V to 0.5 V to activate the catalysts. Linear sweep voltammetry curves are collected at a scan rate of 5 mV/s from 0 V to 0.6 V on a Autolab 302N electrochemical workstation. Chronopotentiometry is performed at a current density of 5 mA/cm^2 for hours. All the linear sweep voltammetry and chronopotentiometry curves are not iR -corrected. In operando electrochemical impedance spectroscopy (EIS) measurements were performed under 0.5 V VS SCE with amplitude of 10 mV and frequency range from 0.01 Hz to 100 kHz. Electrochemically active surface area was estimated by CV method. CV curves are measured at the potential range from 0.15 V to 0.25 V vs SCE at scan rate of 5, 10, 15, 20, 25 mV/s , respectively. For the conversion of potential against Reversible Hydrogen Electrode (RHE), the following equation is applied:

$$E_{\text{RHE}} = E_{\text{SCE}} + 0.244 + 0.059 \times \text{pH}$$

Supporting Figures

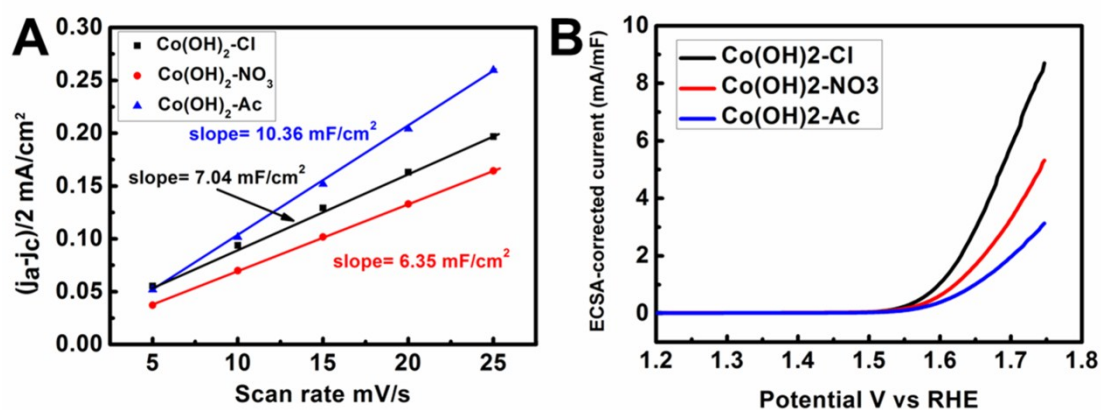


Figure S1. (A) Electrochemically active surface area (ECSA) of Co(OH)₂-Cl, Co(OH)₂-NO₃ and Co(OH)₂-Ac and (B) Corresponding ECSA-corrected LSV curves.

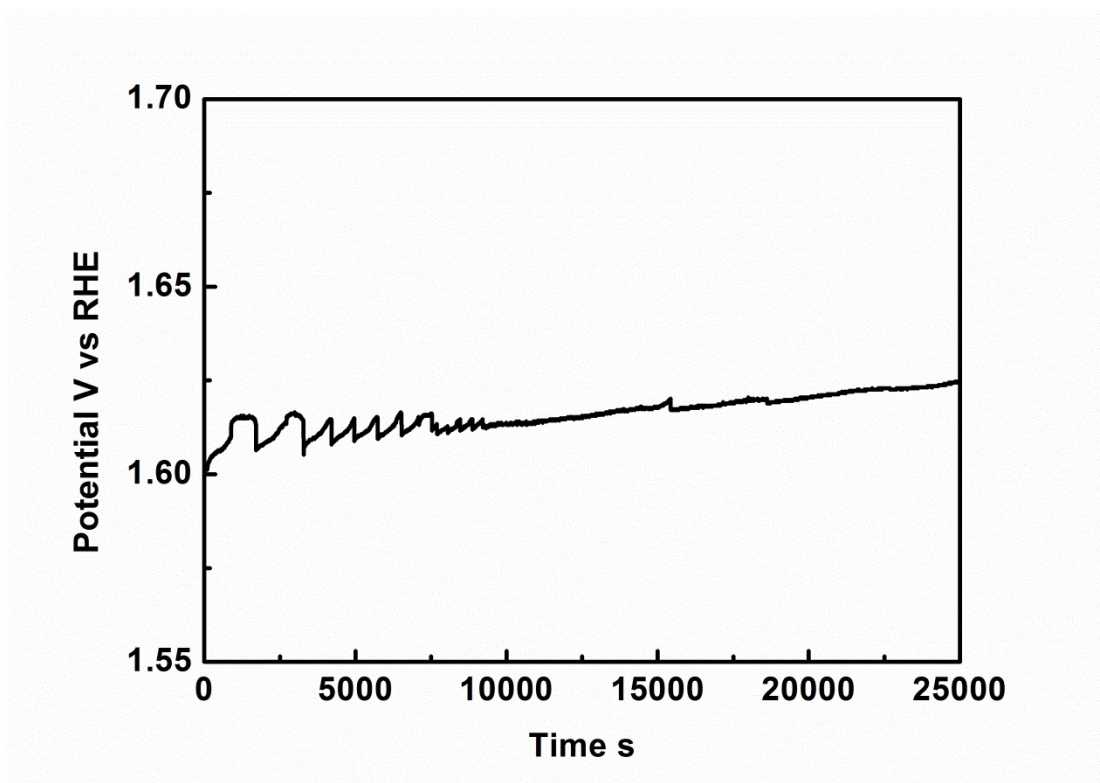


Figure S2. Chronopotentiometry curve of Co(OH)₂-Cl at 5 mA/cm².

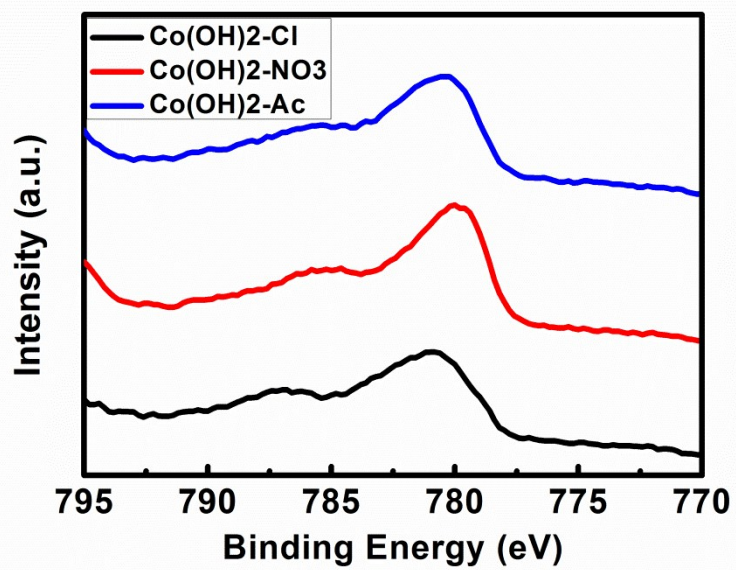


Figure S3. Co 2p XPS spectra of Co(OH)₂-Cl, Co(OH)₂-NO₃ and Co(OH)₂-Ac.