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A high-efficiency oxygen evolution electrocatalyst based on a $Co_3[Co\ (CN)_6]_2$@NiFe LDH composite material

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Experimental Section

Materials

All of the chemicals are of analytical degree and can be used without any special treatment. Cobalt acetate tetrahydrate $(CH_3COO)_2Co\cdot4H_2O$, AR), sodium citrate dihydrate ($Na_3C_6H_5O_7.2H_2O$, AR), potassium cobalt cyanide ($K_3Co(CN)_6]$, AR), nickel nitrate hexahydrate ($Ni(NO_3)_2\cdot6H_2O$, 98%), ferric nitrate (III) nonahydrate ($Fe(NO_3)_3\cdot9H_2O$，99%), urea ($CO(NH_2)_2$) were bought from Sinopharm Chemical Reagent Co. Ltd. Nafion (5 wt%) was purchased from Alfa Aesar Chemical Co. Ltd, anhydrous ethanol (CH_3CH_2OH, GR) was bought from Beijing Tongguang Fine Chemical Co. Ltd.

Materials Characterization.

A scanning electron microscope (SEM, ZEISS Gemini-SEM300) and a transmission electron microscope (TEM, JEOL JEM-F200) were used to examine the micro-nanostructures and morphologies of the composites. Meanwhile, energy dispersive X-ray spectroscopy (EDS, ZEISS Gemini-SEM300) was equipped for elemental distribution analysis. The crystalline phase structure of as-prepared materials was determined by Ultima IV X-ray diffractometer (40 kV, 40 mA, Cu-Kα radiation) at a scanning rate of 10° min^{-1} from 5 to 80°. The catalysts were analyzed for elemental composition and valence states using X-ray photoelectron spectroscopy (XPS Thermo Scientific K-Alpha).

Electrochemical testing

All electrochemical tests were carried out using an electrochemical workstation (CHI760E). Firstly, the preparation of catalyst electrodes was carried out using the coating technique. 5 mg of as-prepared catalyst and 5 mg of Super-P were added to a mixed solution formed by 100 μL of Nafion (5 wt%) solution and 900 μL of anhydrous ethanol. The mixture was then sonicated for 45 min to obtain a homogeneous catalyst ink. Afterward, electrochemical tests were carried out with a three-electrode system (1 M KOH alkaline solution). To prepare the working electrode, 10 μL of catalyst ink was dropped onto a glassy-carbon electrode（GCE）with a diameter of 5 mm. A graphite rod was used as the auxiliary electrode, and a Hg/HgO electrode (0.098 vs. RHE) was used as the reference electrode. The potentials were converted to a reversible hydrogen electrode (RHE) according to the Nernst equation of $E_{RHE} = E_{Hg/HgO} + 0.098 + 0.059$❿PH. Meanwhile, all tested potentials were calibrated at 95% iR compensation, with the value of R being the ohmic resistance of the electrolyte. The linear scanning voltammogram (LSV) of the OER from 0.2 V-0.8 V relative to the RHE was determined with the GCE electrode rotating at a constant rate of 1600 rpm and a potential scanning rate of 5 mV s^{-1}. Electrochemical impedance spectra (EIS) were measured between 0.01 Hz and 100 kHz with an amplitude of

5 mV for cases where the applied potential was sufficient to trigger the OER. The accelerated stability was tested by 1000 cycles of CV (scanning rate 100 mV s^{-1}) in 1 M KOH electrolyte, and the catalyst stability was tested for 10 h by a constant current density of 10 mA cm^{-2}. Finally, the electrochemical double layer capacitance (C_{dl}) of the various samples was evaluated using CV in the non-Faraday region at different scan rates (v = 20, 40, 60, 80, and 100 mV s^{-1}) to determine the electrochemically active surface area (ESCA) accurately.

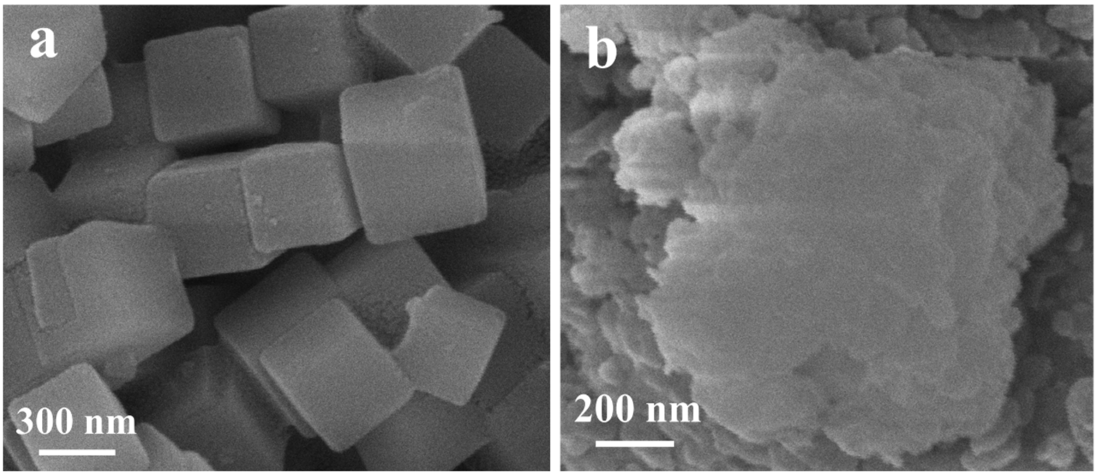

Fig. S1 SEM images of (a) Co-PBA and (b) NiFe-LDH.

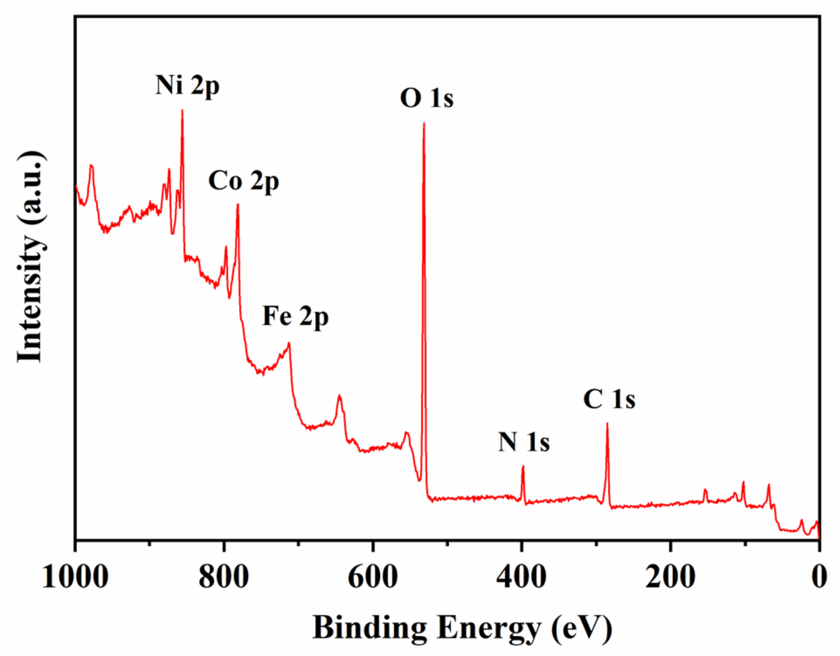

Fig. S3 XPS overall spectrum of Co-PBA@NiFe-LDH-30.

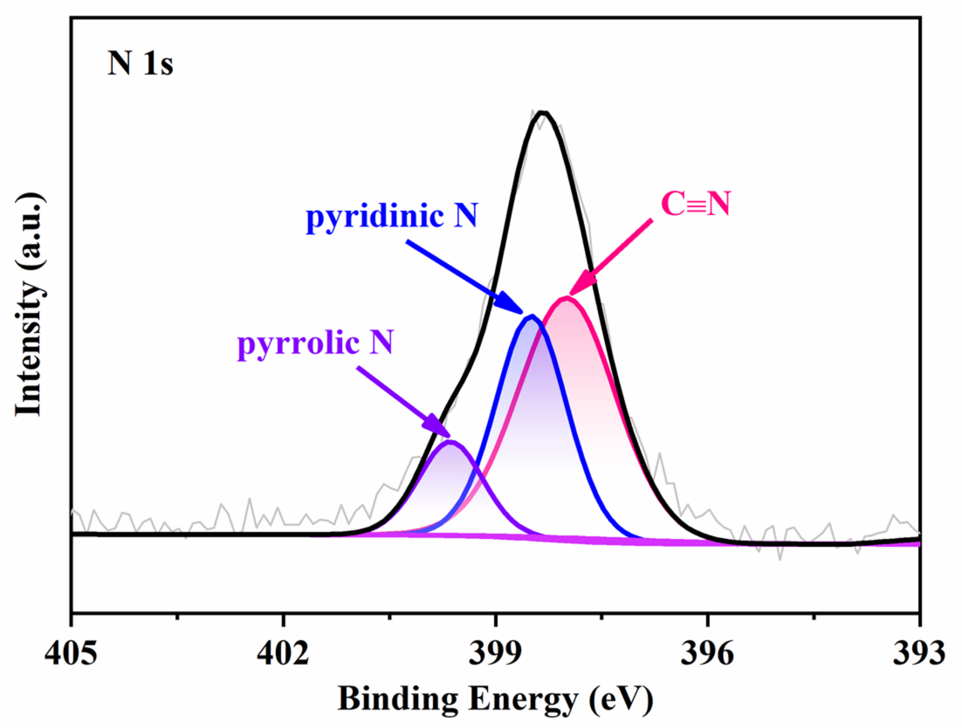

Fig. S3 High-resolution XPS spectra of N 1s of Co-PBA@NiFe-LDH-30.

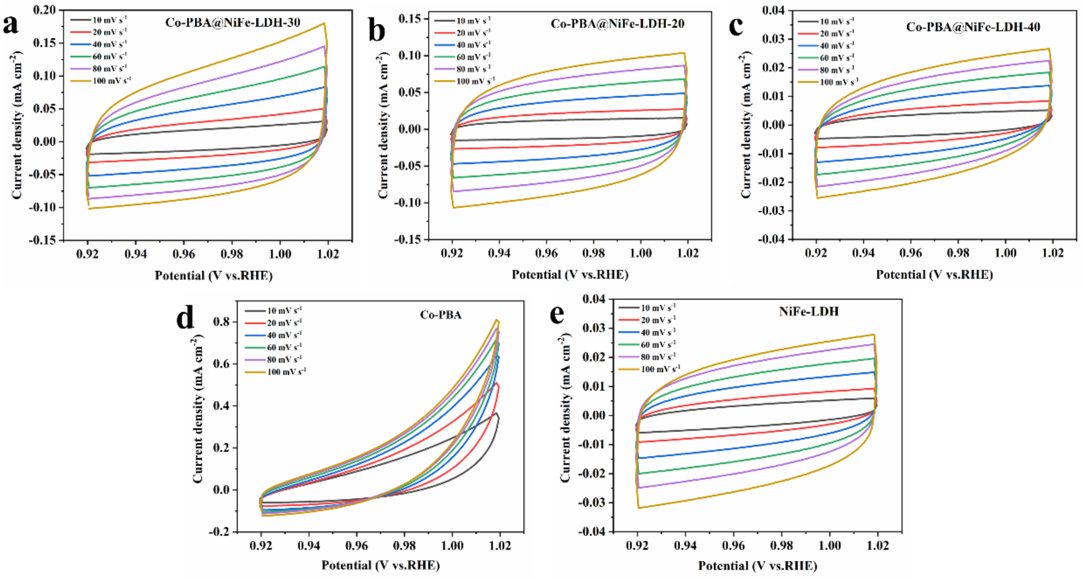

Fig. S4 CV measurement with various scan rates for (a) Co-PBA@NiFe-LDH-30, (b) Co-PBA@NiFe-LDH-30, (c) Co-PBA@NiFe-LDH-30, (d) Co-PBA and (e) NiFe-LDH in 1 M KOH at 0.92-1.02 V (vs. RHE)

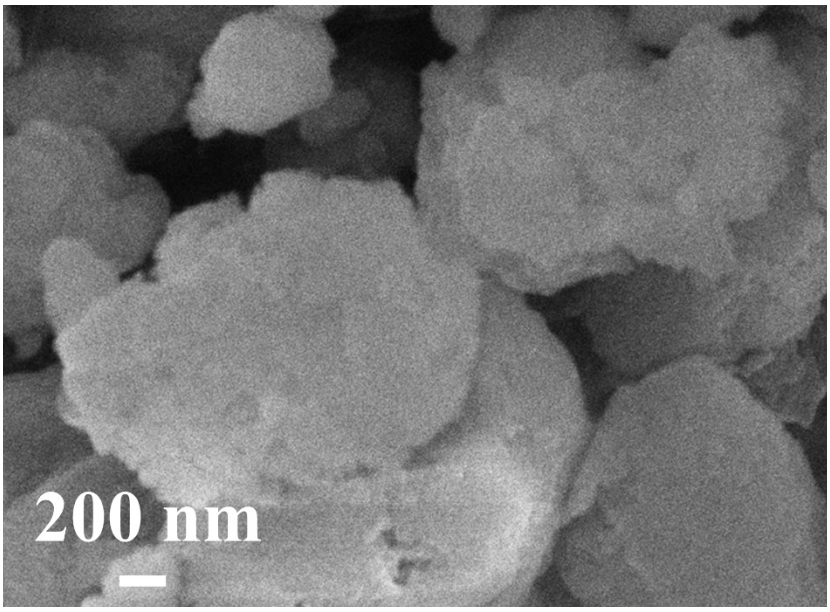

Fig. S5 SEM images of Co-PBA@NiFe-LDH-30 after stability test

Tab.S1 The element ratio of Co, Ni, Fe, C, N, and O in Co-PBA@NiFe-LDH-30 by EDS semi-quantitative Analysis

Element	Mass (%)	Atomic (%)
Co K	25.60	8.13
Ni K	9.18	2.93
Fe K	3.37	1.13
C K	29.57	46.10
N K	23.61	31.55
O K	8.68	10.16