Supplementary Information (SI) for Materials Chemistry Frontiers. This journal is © the Partner Organisations 2025

Electronic Supplementary Information Feather duster-like CoFe-LDH/CF composite with parallel array structure as an efficient water splitting electrocatalyst

Weiliang Xiong, ab Ting Li, b Xiang Li, a Peng Zhang, b Guosheng Shao, b Yige Zhao ab

^a 713rd Research Institute of China Shipbuilding Industry Corporation, Zhengzhou, 450015, China

b State Center for International Cooperation on Designer Low-Carbon & Environmental Materials (CDLCEM), School of Materials Science and Engineering, Zhengzhou University, Zhengzhou 450001, China. E-mail: zhaoyg@zzu.edu.cn

Experimental Section

Physical Characterizations

The surface morphologies were examined using a ZEISS SIGMA 500 scanning electron microscope (SEM). Transmission electron microscopy (TEM) images was conducted using a FEI Talos F200S microscope. For chemical state analysis, we performed X-ray photoelectron spectroscopy (XPS) analysis using the AXIS Supra equipment, using the C1s peak with binding energy set at 284.8 electron volts as a reference for calibration. Characterization of the material composition and crystal structure was performed by X-ray diffraction (XRD) on a Rigaku Ultima IV system with Cu K α radiation. We used an ASAP 2020 analyzer to determine the specific surface area and pore size distribution through nitrogen adsorption/desorption measurements using the Brunauer-Emmett-Teller (BET) method.

Electrochemical Measurements

A three-electrode system on a CHI 760E electrochemical workstation was used to perform the electrochemical testing under ambient conditions. The reference was a saturated calomel electrode (SCE), while the function of the counter electrode was performed by a graphite electrode. The preparation of the working electrodes involved a systematic polishing process using alumina suspensions with different grain sizes, starting at 1.0 μm, then 0.5 μm, and finally 0.05 μm, to ensure the removal of any surface contaminants. The drop-casting of a catalyst ink onto a 3 mm diameter glassy carbon electrode was done with care, resulting in a catalyst loading of 0.35 mg cm⁻². A mixture of 0.74 mL ethanol, 0.2 mL deionized water, and 0.06 mL of Nafion solution was used to make the ink itself, which was done by ultrasonically dispersing 5 mg of catalyst into this mixture. Calibration of all the potentials reported was done against the reversible hydrogen electrode (RHE) using the relationship specified in equation (1).

$$E_{RHE} = E_{SCE} + 0.0592 \times pH + 0.24 V$$
 (1)

Linear sweep voltammetry (LSV) was performed at a scan rate of 5 mV s⁻¹ with 80% iR compensation to assess oxygen evolution reaction (OER) activity between 1.0 and 2.0 V versus RHE, as well as hydrogen evolution reaction (HER) performance from 0

to -1.0 V versus RHE, all in a 1 M KOH solution saturated with oxygen. The double-layer capacitance (C_{dl}) was estimated by measuring the electrochemically active surface area (ECSA) with cyclic voltammetry (CV) in a 1 M KOH solution saturated with nitrogen. The relationship between C_{dl} and ECSA can be described by the following equation:

$$ECSA=C_{dl}/C_{s}$$
 (2)

where, C_s represent the capacitance of the flat electrode surface ($C_s = 0.04 \text{ mF/cm2}$) The potentials ranged from 0.1 to 0.2 V, and the scan rates varied from 20 to 100 mVs⁻¹. Electrochemical impedance spectroscopy (EIS) was carried out over a frequency range from 0.1 to 10^6 Hz with a perturbation amplitude of 5 mV. To gauge the catalyst durability, long-term stability tests were carried out via chronopotentiometry at 10 mA cm⁻² over 30000 s. Measurements of 20 wt% Pt/C and RuO₂ catalysts were conducted under identical conditions for comparison.

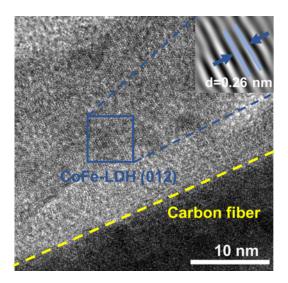


Figure S1. HRTEM image of p-CoFe-LDH/CF

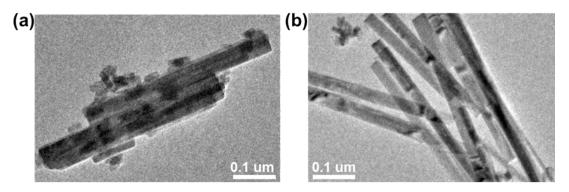


Figure S2. TEM image of (a) p-CoFe-LDH/CF, (b) CoFe-LDH/CF

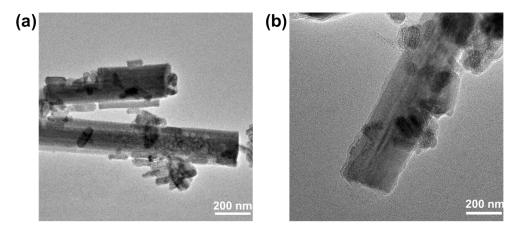


Figure S3. TEM image of (a) p-CoFe-LDH/CF before OER cycling stability test (b) p-CoFe-LDH/CF after OER cycling stability test

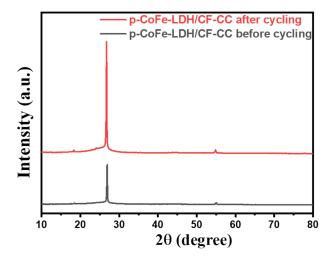


Figure S4. XRD patterns of p-CoFe-LDH/CF-CC before and after OER cyclability testing

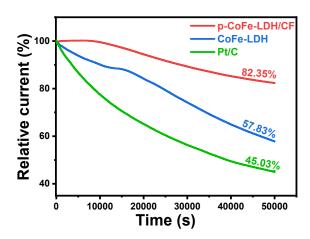


Figure S5. chronoamperometric curves conducted at 10 mA cm-2 for HER

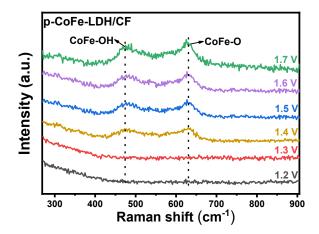


Figure S6. potential-dependent of in situ Raman spectra of p-CoFe-LDH/C

Table S1. Performance comparison of alkaline OER transition-mental-based catalyst in 1 M KOH at large current density.

Catalyst	Overpotentials at 10 mA cm ⁻² (mV)	Overpotentials at 100 mA cm ⁻² (mV)	Reference
p-CoFe-LDH/CF	286	416	This work
LiNiCo-OH	340		10.1021/acs.nanolett.5b00026
Ultrathin CoMn-LDH	350		10.1021/ja5096733
Co ₃ O ₄ @CoO SC	430		10.1038/ncomms9106
Echo-MWCNTs	360		10.1021/ja509879r
Ni _x Co _{3-x} O ₄ nanowire array/Ti foil	370		10.1002/adma.200903896
Co _x P/Cu foil	345		10.1002/anie.201501616.
Ni@N-HCGHF	280	470	10.1002/adma.202003313
Fe ₃ C-Co/NC	371	450	10.1002/adfm.201901949