Electronic Supplementary Information

Acidically oxidized carbon cloth: a novel metal-free oxygen evolution electrode with high catalytic activity

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Experimental section

Material: potassium hydroxide (KOH) and nitric acid (HNO₃) were purchased from Beijing Chemical Works and used as received. CC was purchased from Phychemi Hong Kong Company Limited. The water used throughout all experiments was purified through a Millipore system

Characterizations: XPS measurements were performed on an ESCALABMK II X-ray photoelectron spectrometer using Mg as the exciting source. GC analysis was carried out on GC–2014C (Shimadzu Co.) with thermal conductivity detector and nitrogen carrier gas. Pressure data during electrolysis were recorded using a CEM DT-8890 Differential Air Pressure Gauge Manometer Data Logger Meter Tester with a sampling interval of 1 point per second. Nicolet 560 Fourier transform infrared (FTIR) spectrometer was applied to collect the FTIR spectra. A Vario EL elemental analyzer was used to for CHNO elemental analysis.

Preparation of OCC: Typically, 40 mL 70 % HNO₃ was put into a Taflon-lined stainless autoclave (50 mL) and then a piece of CC was immersed into it. The autoclave was sealed and maintained at 90 °C for different time (2, 8, 16 h). After the autoclave cooled down slowly at room temperature, the OCC was taken out and washed with deionized water before dried at room temperature.

Preparation of RuO₂: 0.01 mol of RuCl₃·3H₂O was dissolved in 100 mL of deionized water. The aqueous solution was then heated $(100 \,^{\circ}\text{C})$ and magnetically stirred for 10 min, then 1 mL of sodium hydroxide (1 M) was added to the solution in order to obtain the precursor Ru-hydroxide. The reaction mixture was

maintained under stirring and heated (100 $^{\circ}$ C) for 45 min, then the precipitate was washed several times and calcined at 350 $^{\circ}$ C for 1 h to obtain RuO₂.

Electrochemical measurements: All the electrochemical measurements were conducted in a typical threeelectrode setup with an electrolyte solution of 0.1 M KOH using OCC-8 as the working electrode, a graphite plate as the counter electrode and Ag/AgCl as the reference electrode. In all measurements, the Ag/AgCl reference electrode was calibrated with respect to reversible hydrogen electrode (RHE). Linear sweep voltammetry (LSV) measurements were conducted in 0.1 M KOH with scan rate of 5 mV/s. For comparison, the catalytic performance of RuO₂ powder was tested on a glassy carbon working electrode. All the potentials reported in our work were vs. Ag/AgCl in 0.1 M KOH. $\eta = E$ (RHE) – 1.23 V, E (RHE) = E (Ag/AgCl) + 0.059 pH + 0.197. The calculation of current density was based on the geometrical area.

 Table S1 Comparison of OER performance of OCC-8 with other nonmetal and some metal
 electrocatalysts in alkaline electrolytes.

Catalyst	Electrolyte	Onset η (mV)	η at 10 mA/cm ²	Ref.
OCC-8	0.1 M KOH	328	477	This work
PNG	0.1 M KOH	352	~634	[10]
TRGO/NCNT	0.1 M KOH	~279	~505	[11]
NG-CNT	0.1 M KOH	315	~484	[12]
N/C	0.1 M KOH	290	380	[13]
g-C ₃ N ₄ /G	0.1 M KOH	314	539	[14]
g-C ₃ N ₄ -CNT	0.1 M KOH	300	370	[15]
3D NF/PC/AN	0.1 M KOH	294	~520	[17]
mesoporous Co ₃ O ₄	0.1 M KOH	-	528	[18]
mesoporous Co ₃ O ₄	0.1 M KOH	-	636	[19]
Co ₃ O ₄ /SWNTs	1.0 M KOH	-	593	[20]



Fig. S1 Time-dependent current density curves for another three different electrodes obtained under otherwise identical conditions used for preparing OCC-8 at 0.75V for 24000 s.



Fig. S2 SEM images of (A, B) CC and (C, D) OCC-8.



Fig. S3 The high-resolution O 1s XPS spectra of (A) OCC-8, (B) OCC-2, (C) OCC-16, and (D) CC. (E) the O/C ratios obtained from the XPS data (black dots) and the CHNO elemental analysis (magenta dots) and (F) oxygen functionalities content of CC, OCC-2, OCC-8 and OCC-16.



Fig. S4 FTIR spectrum of OCC-8.



Fig. S5 Nyquist plots of CC, OCC-2, OCC-8 and OCC-16 recorded in 0.1 M KOH solution.



Fig. S6 (A) Low- and (B) high- magnification SEM images of OCC-8 after stability test.



Fig. S7 The high-resolution O 1s XPS spectra and the content of oxygen functionalities (inset) of OCC-8 (A) before and (B) after stability test.