Supporting Information

Enhanced oxygen evolution activity on mesoporous cobalt-iron oxides

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Experimental section

Materials

All chemicals were analytical grade and were used as purchased without further purification. Solutions were prepared using high purity water (Millipore Milli-Q purification system, resistivity > 18 MΩ·cm).

Synthesis of SBA-15 and mesoporous cobalt-iron oxides

Synthesis of SBA-15

SBA-15 was prepared according to the literature. In a typical synthesis, Pluronic P123 (2 g) was first dissolved in 15 mL of H₂O and 60 mL of 2 M HCl, and then 4.68 mL (0.02 mol) of TEOS was added under stirring at 40 °C. The molar composition of the mixture, TEOS/P123/HCl/H₂O, was 1/0.017/6/192. Thereafter, the mixture was
maintained under stirring at 40 °C for 24 h and then it was transferred into an autoclave to age for 24 h at 100 °C under static conditions. Finally, the sample was filtered, washed with distilled water thoroughly, air-dried, and calcined at 550 °C to remove the template. The dry white solid was denoted as SBA-15.

**Synthesis of mesoporous cobalt-iron oxides**

0.5 g of SBA-15 was dispersed in 50 mL of toluene at 65 °C. Then, 0.68 g of cobalt nitrate hexahydrate and 0.047 g of iron nitrate nonahydrate were added into this mixture along with forceful stirring for 3 h. After filtrating, the obtained pink powders were calcined at 400 °C for 2 h. The SBA-15 template was removed by washing with 2 M of hot NaOH solution, and mesoporous CoFe_{0.05}O_x without template was finally obtained, which was denoted as meso-CoFe_{0.05}O_x.

For comparison, mesoporous iron-cobalt oxides with different Fe/Co ratios (i.e. 0.01/1, 0.025/1, 0.05/1, 0.075/1, and 0.1/1) were prepared by a similar synthetic procedure with the above synthesis except different Fe/Co ratios.

**Material characterizations**

X-ray diffraction (XRD) was carried out on a RIGAKU D/MAX2550/PC diffractometer at 40 kV and 100 mA with copper filtered Kα radiation (λ = 1.5406 Å). The specific surface areas of the catalysts were measured based on the adsorption isotherms of N_2 at −196 °C using the BET method (Micromeritics ASAP2010). Transmission electron microscope (TEM) images were observed by a Hitachi HT7700.
The valence state of rhodium was determined using XPS recorded on a Thermo ESCALAB 250Xi. The X-ray source selected was monochromatized Al Kα source (15 kV, 10.8 mA). Region scans were collected using 20 eV pass energy.

**Electrochemical activity characterizations**

All electrochemical measurements were performed in a three-electrode system with a glassy carbon electrode (GCE) as the substrate for the working electrode, a graphite rod as the counter electrode and a saturated calomel electrode as the reference electrode. The reference electrode was calibrated with respect to a reversible hydrogen electrode before each experiment. The glassy carbon electrode was pre-polished using 0.05 μm alumina and distilled water. To prepare the working electrode, 2 mg of the catalyst was dispersed in a 0.2 mL mixed solvent of ethanol and Nafion (1 wt%) and sonicated to obtain a homogeneous ink. 8 μL of the catalyst ink was drop-casted on the glassy carbon electrode and dried at room temperature (catalyst loading: 1.127 mg·cm⁻²).

For OER, the working electrode was first activated by steady-state cyclic voltammetry (CV) performed in the potential range from 1.0 to 1.6 V vs RHE at a scan rate of 50 mV s⁻¹ for 50 cycles. Linear scan voltammetry (LSV) curves were then collected at a scan rate of 5 mV s⁻¹. All of the potentials in the LSV polarization curves were with 90% iR compensation unless specifically illustrated. The measurement error was within 10%.
Fig. S1. Raman spectra of meso-Co$_3$O$_4$ and meso-CoFe$_{0.05}$O$_x$. 
Fig. S2. HRTEM image of meso-CoFe$_{0.05}$O$_x$. 
**Fig. S3.** (a) OER polarization curves. (b) Tafel plots.
Fig. S4. (a) CVs of meso-CoFe$_{0.05}$O$_x$ and meso-Co$_3$O$_4$, (b) CVs of meso-CoFe$_n$O$_x$. 
**Fig. S5.** (a) CVs of the meso-CoFe\textsubscript{0.05}O\textsubscript{x} measured in a non-Faradaic region at different scan rate. (b) The cathodic and anodic currents measured as a function of the scan rate.
Fig. S6. (a) CVs of the commercial Co$_3$O$_4$ measured in a non-Faradaic region at different scan rate. (b) The cathodic and anodic currents measured as a function of the scan rate.
Fig. S7. (a) CVs of the meso-Co₃O₄ measured in a non-Faradaic region at different scan rate. (b) The cathodic and anodic currents measured as a function of the scan rate.
Fig. S8. (a) CVs of the meso-CoFe$_{0.01}$O$_x$ measured in a non-Faradaic region at different scan rate. (b) The cathodic and anodic currents measured as a function of the scan rate.

Fig. S9. (a) CVs of the meso-CoFe$_{0.025}$O$_x$ measured in a non-Faradaic region at different scan rate. (b) The cathodic and anodic currents measured as a function of the scan rate.
Fig. S10. (a) CVs of the meso-CoFe$_{0.075}$O$_x$ measured in a non-Faradaic region at different scan rate. (b) The cathodic and anodic currents measured as a function of the scan rate.

Fig. S11. (a) CVs of the meso-CoFe$_{0.1}$O$_x$ measured in a non-Faradaic region at different scan rate. (b) The cathodic and anodic currents measured as a function of the scan rate.
**Fig. S12.** (a) Nyquist plots of the EIS test. (b) The equivalent circuit used for fitting the Nyquist plots.
Fig. S13. OER polarization curves of the meso-CoFe$_{0.05}$O$_x$ before and after stability test in 1 M KOH.
Fig. S14. (a) SEM image and (b) TEM image of meso-CoFe$_{0.05}$O$_x$ after OER.
Fig. S15. XRD patterns of meso-CoFe$_{0.05}$O$_x$ after OER.
Fig. S16. XPS survey spectrum (a), and high-resolution XPS spectra of Fe 2p (b), Co 2p (c), and O 1s (d) of after the OER meso-CoFe\textsubscript{0.05}O\textsubscript{x}. 
Table S1. Comparison of OER performance of meso-CoFe$_{0.05}$O$_x$ with results in recent literature

<table>
<thead>
<tr>
<th>Catalyst</th>
<th>Electrolyte</th>
<th>$\eta@10$ mA cm$^{-2}$ (mV)</th>
<th>Tafel slope (mV dec$^{-1}$)</th>
<th>Ref.</th>
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<tr>
<td>meso-CoFe$_{0.05}$O$_x$</td>
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<td>Fe-Co$_3$O$_4$/CNTs</td>
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<tr>
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<td>10</td>
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<td>FeCo/Co$_2$P@NPCF</td>
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<td>280</td>
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References


11. Q. Shi, Q. Liu, Y. Ma, Z. Fang, Z. Liang, G. Shao, B. Tang, W. Yang, L. Qin


