Supporting Information

Forest-like NiCoP@Cu₃P Supported on Copper Foam as a bifunctional catalyst for Efficient Water Splitting

Xingxing Ma⁵, Yaqing Chang⁵, Zhang Zhe⁵, Jilin Tang⁵

⁵State Key Laboratory of Electroanalytical Chemistry, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun, Jilin 130022, China.

Characterizations:

The structures of the samples were determined by scanning electron microscopy (SEM) performed on a (Philips XL30 ESEM FEG) scanning electron microscope. Transmission electron microscopy (TEM) images were taken on a HITACHI H-8100 electron microscopy (Hitachi, Tokyo, Japan) operated at 200 kV. Powder X-ray diffraction (XRD) patterns were collected using a RigakuD/MAX 2550 diffractmeter with Cu Kα radiation (λ=1.5418 Å). X-ray photoelectron spectroscopy (XPS) analysis was performed on an ESCALABMK II X-ray photoelectron spectrometer using Mg as the excitation source. Element content analysis was determined through ICP-OES (Thermo iCAP. 6300, DIONEX ICS-1000). The separated NiCoP@Cu₃P was separated from NiCoP@Cu₃P/CF by sonication, dissolved in nitrohydrochloric acid.

Electrochemical measurements

All the experiments were carried out without activation process at ambient temperature. Electrochemical measurements were performed with a CHI 660A electrochemical analyzer (CH Instruments, Inc., Shanghai). A conventional one-component three-electrode cell was used, including a piece of NiCoP@Cu₃P/CF as the working electrode, a saturated calomel electrode (SCE) as the reference.
electrode, and carbon rod as the counter electrode. Potentials were referenced to a reversible hydrogen electrode (RHE): $E_{\text{vs RHE}} = E_{\text{vs SCE}} + 0.242 + 0.059 \text{pH}$.

**Figure S1** EDX spectrum of the NiCoP@Cu3P/CF.

**Figure S2** SEM images of NiCo-LDH@Cu(OH)$_2$/CF: total molar concentration of Co$^{2+}$, Ni$^{2+}$ (a) 0.01 mol/L, (b) 0.005 mol/L
Figure S3 XRD patterns of the NiCo-LDH@Cu(OH)$_2$/CF.

Figure S4 XRD patterns of Ni$_x$Co$_{2-x}$P@Cu$_3$P/CF with various atomic ratio of Co/Ni (2:1, 1:1, 3:1, 1:2, 1:0).
Figure S5 XPS of NiCoP@Cu₃P/CF: (a) XPS survey scan spectrum and (b) high-resolution XPS spectra of Cu.

<table>
<thead>
<tr>
<th>Elements</th>
<th>Weight% (ppm)</th>
<th>Atomic%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni</td>
<td>120700</td>
<td>15.8</td>
</tr>
<tr>
<td>Co</td>
<td>113400</td>
<td>14.9</td>
</tr>
<tr>
<td>Cu</td>
<td>357000</td>
<td>43.3</td>
</tr>
<tr>
<td>P</td>
<td>104100</td>
<td>26.0</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>695200</strong></td>
<td><strong>100</strong></td>
</tr>
</tbody>
</table>

Table 1 Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES) spectra result of NiCoP@Cu₃P/CF

NiCoP : Cu₃P  1.5 : 1.1
Figure S6 HER performance in 1M KOH (a) Electrochemical impedance spectra of the NiCoP@Cu3P/CF catalyst and (b) Tafel plots corresponding to Ni\textsubscript{x}Co\textsubscript{2-x}P@Cu\textsubscript{3}P/CF with various atomic ratio of Co/Ni (2:1, 1:1, 3:1, 1:2, 1:0).

Figure S7 OER performance in 1M KOH: (a) Electrochemical impedance spectra of the NiCoP@Cu3P/CF catalyst and (b) Tafel curves corresponding to Ni\textsubscript{x}Co\textsubscript{2-x}P@Cu\textsubscript{3}P/CF with various atomic ratio of Co/Ni (2:1, 1:1, 3:1, 1:2, 1:0).
Figure S8 OER performance of Ni\textsubscript{x}Co\textsubscript{2-x}P@Cu\textsubscript{3}P/CF: (a) CV curves with scanning rate of 2mV/s, (b) local magnification of (a).

Figure S9 SEM images of NiCoP@Cu\textsubscript{3}P/CF (a) after HER stability test; (b) after HER stability test.