Electronic Supplementary Information

Facile sequential ion exchange strategy to synthesize CoSe₂/FeSe₂

double-shelled hollow nanocuboids for highly active and stable

oxygen evolution reaction

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Fig. S1 (a, b) SEM images and (c, d) TEM images of the as-prepared Co-precursor NCs.



Fig. S2 XRD pattern of the as-prepared CoFe-PBA DS-HNCs.



Fig. S3 (a) XRD pattern and (b) EDX spectrum of the as-prepared CoSe₂/FeSe₂ DS-HNCs.



Fig. S4 STEM image and corresponding EDS line scanning of a single CoSe₂/FeSe₂ DS-HNC.



Fig. S5 N_2 adsorption-desorption isotherm and pore size distribution (inset) of the as-prepared CoSe₂/FeSe₂ DS-HNCs.



Fig. S6 (a) SEM image and (b) XRD pattern of the as-prepared CoSe₂ NPs.



Fig. S7 Cyclic voltammogram (CV) curves of $CoSe_2/FeSe_2$ DS-HNCs (a) and $CoSe_2$ NPs (c) in the double layer region at scan rates of 2, 4, 6, 8 and 10 mV s⁻¹ in 1.0 M KOH; (b) and (d) current density as a function of scan rate derived from (a) and (c), respectively.



Fig. S8 LSV curve of RuO₂ for OER.

To achieve the current density of 10 mA cm⁻², RuO₂ catalyst needs an overpotential of 290 mV for OER.



Fig. S9 SEM images of CoFe-PBA samples by using different amount of K₃[Fe(CN)₆]: (a, b) 20 mg, CoFe-PBA DS-HNCs-2, (c, d) 40 mg, CoFe-PBA DS-HNCs, (e, f) 60 mg, CoFe-PBA DS-HNCs-6 and (g, h) 80 mg, CoFe-PBA DS-HNCs-8.



Fig. S10 SEM images, corresponding EDX patterns and the element contents (inset) of different CoSe₂/FeSe₂ DS-HNCs samples: (a, b) CoSe₂/FeSe₂ DS-HNCs-2, (c, d) CoSe₂/FeSe₂ DS-HNCs, (e, f) CoSe₂/FeSe₂ DS-HNCs-6 and (g, h) CoSe₂/FeSe₂ DS-HNCs-8.



Fig. S11 LSV curves of different CoSe₂/FeSe₂ DS-HNCs samples.

| | CoSe ₂ /FeSe ₂ DS- | CoSe ₂ |
|-------------------------------------|--|-------------------|
| | HNCs | NPs |
| $R_{s} \left(\Omega \ cm^{2} ight)$ | 1.56 | 1.06 |
| $R_{ct}(\Omega~cm^2)$ | 2.52 | 7.04 |
| CPE1-T | 0.49 | 0.03 |
| CPE1-P | 0.97 | 0.71 |

Table S1. EIS data of $CoSe_2/FeSe_2$ DS-HNCs and $CoSe_2$ NPs for OER.



Fig. S12 The LSV curves for the CoSe₂/FeSe₂ DS-HNCs before and after 1000 cycles.



Fig. S13 XPS survey spectrum (a) and high-resolution XPS spectra of Co 2p (b), Fe 2p (c) and Se 3d (d) of the as-prepared CoSe₂/FeSe₂ DS-HNCs after OER test.



Fig. S14 SEM images of the as-prepared CoSe₂/FeSe₂ DS-HNCs after OER test.

| | Overpotential (mV) at 10 mA cm ⁻² | References |
|--|---|------------------------------------|
| Catalysts | | |
| CoSo, nonochoota | 320 | J. Am. Chem. Soc. |
| $CoSe_2$ nanosneets | | 2014 , <i>136</i> , 15670. |
| | 324 | Adv. Mater. |
| C0 _{0.85} Se | | 2016 , <i>28</i> , 77. |
| A - C-S- h-H | 320 | Angew. Chem. Int. Ed. |
| Ag-CoSe ₂ -belt | | 2017 , <i>56</i> , 328. |
| | 320 | Chem. Mater. |
| $N1_{0.88}C0_{1.22}Se_4$ | | 2017 , <i>29</i> , 7032. |
| | 250 | ACS Catal. |
| (N1,C0)Se-GA | | 2017 , <i>7</i> , 6394. |
| C- C- ONC | 320 | J. Mater. Chem. A |
| C00.85Se@INC | | 2017 , <i>5</i> , 7001. |
| N.C. | 290 | Adv. Energy Mater. |
| INISE | | 2018 , <i>8</i> , 1702704. |
| E. damed NCC. | 268 | Angew. Chem. Int. Ed. |
| Fe-doped N1Se ₂ | | 2018 , <i>57</i> , 4020. |
| | 260 | ACS Appl. Mater. Interfaces |
| FeSe ₂ (<i>a</i> CoSe ₂ /rGO | | 2018 , <i>10</i> , 19258. |
| | 251 | Adv. Mater. |
| Fe _{0.09} Co _{0.13} -N1Se ₂ | | 2018 , <i>30</i> , 1802121. |
| CoSe ₂ /FeSe ₂ DS-HNCs | 240 | This work |

 Table S2. Comparison of OER performance with recently reported metal selenides catalysts in alkaline medium.