Electronic Supplementary Information

2D MOF-derived ultra-thin $CoSe_2$-$NiSe_2$/CN: Dual-phase synergistic effect and abundant defects to enhance the hydrogen evolution reaction

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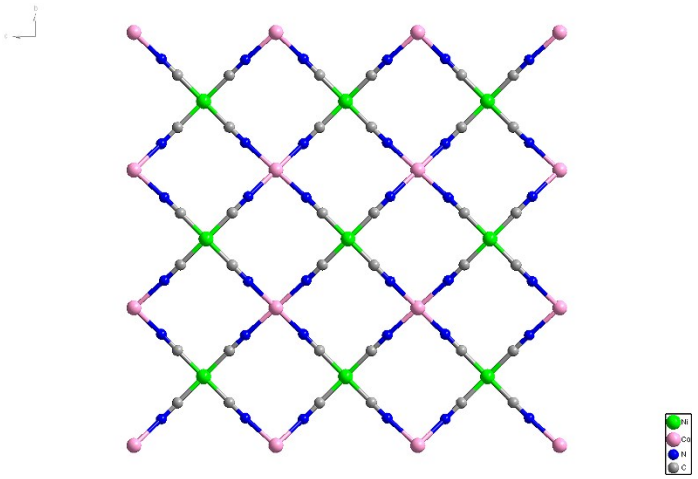
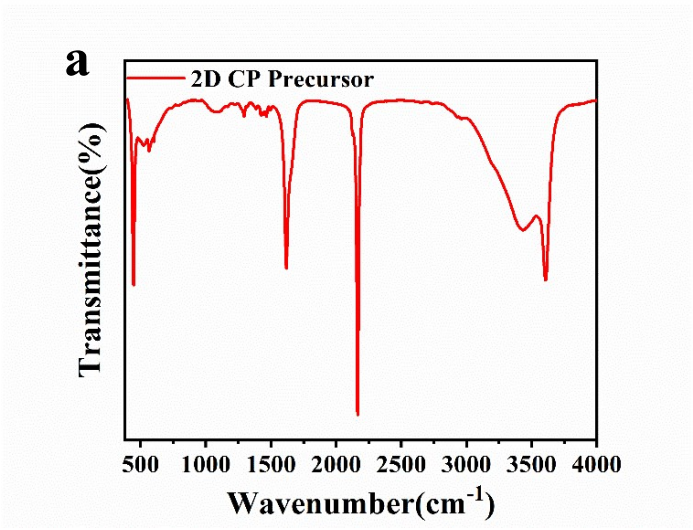
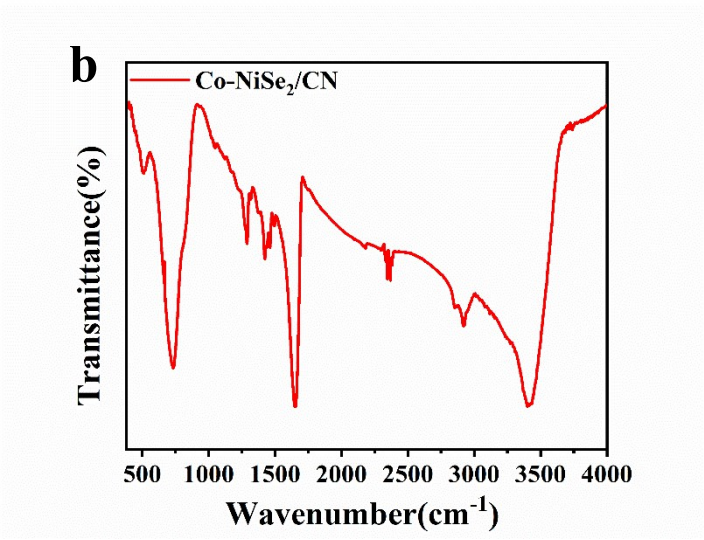


Figure S1 The framework structure of 2D CP ($Co[Ni(CN)_4]$).





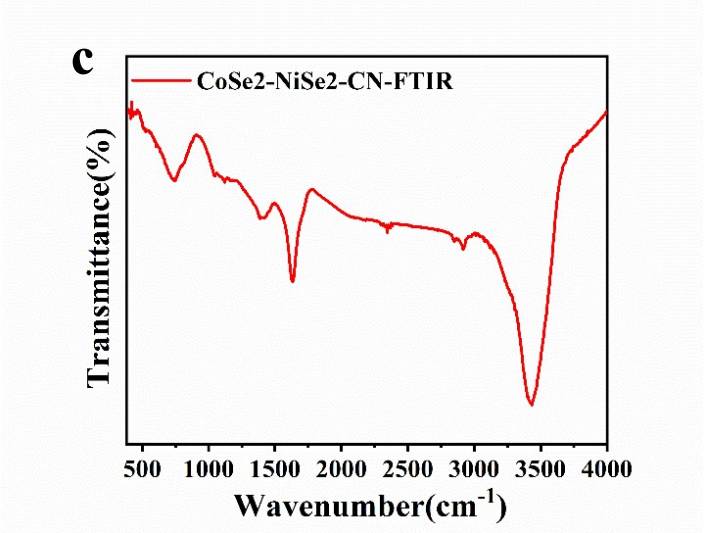


Figure S2 FT-IR spectra of (a) 2D CP Precursor, (b) Co-$NiSe_2$/CN and (c)$CoSe_2$-$NiSe_2$/CN.

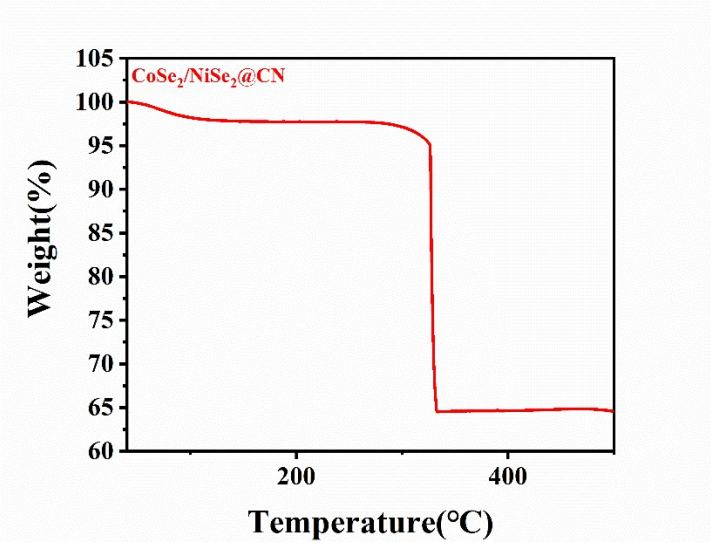


Figure S3 (a) Thermogravimetric analysis (TGA) curves of $CoSe_2$-$NiSe_2$/CN measured under an O_2 atmosphere.

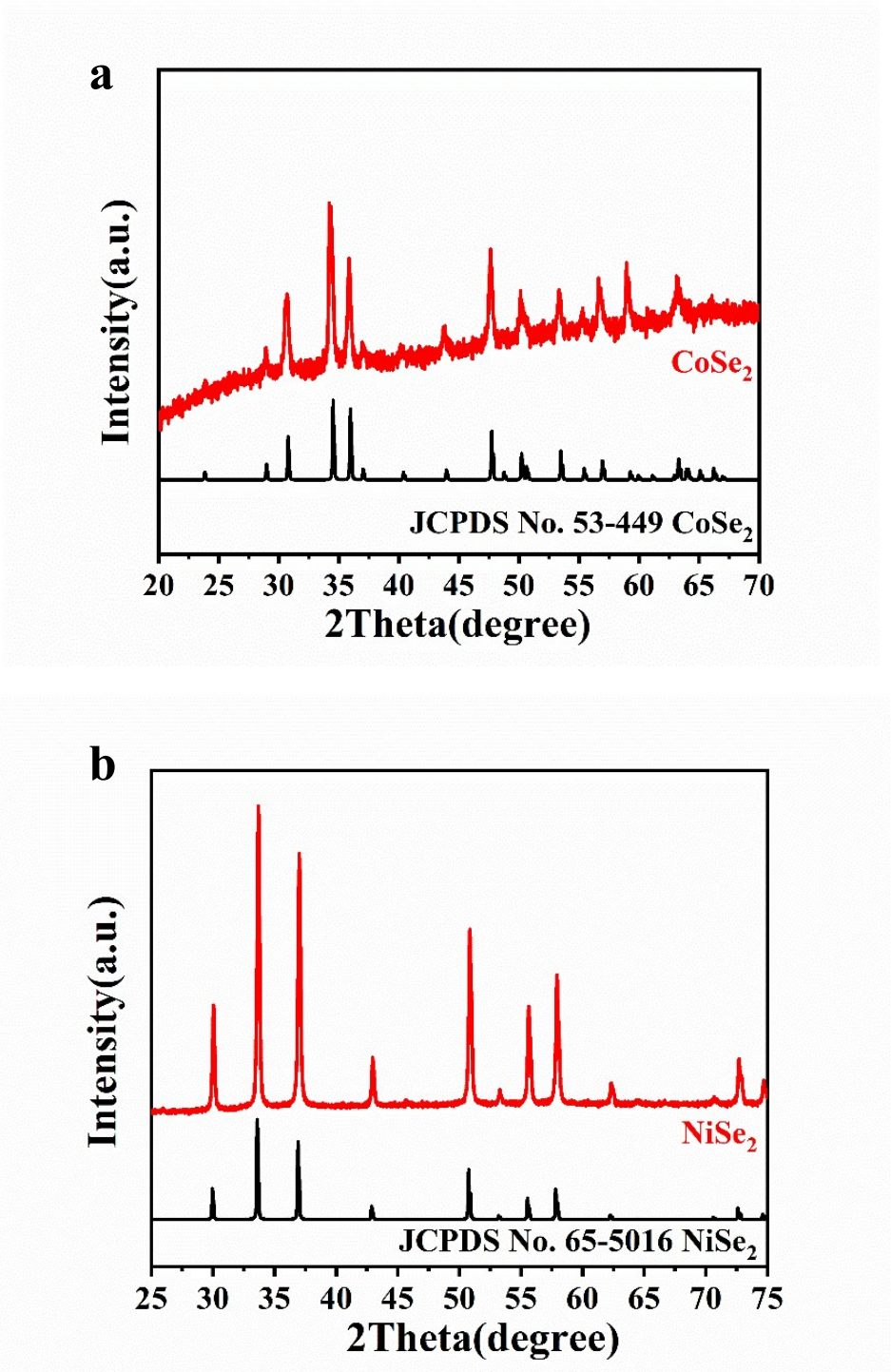


Figure S4 XRD pattern of (a) $CoSe_2$ and (b) $NiSe_2$.

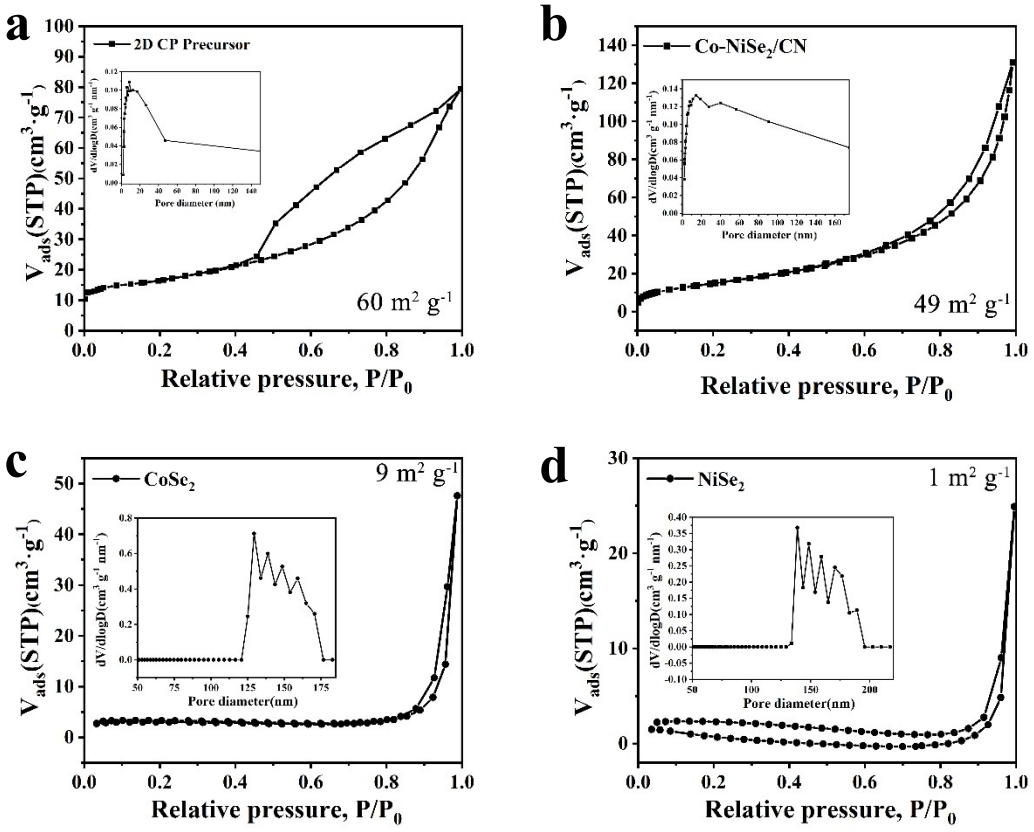


Figure S5 N_2 adsorption-desorption isotherms of (a) 2D CP Precursor, (b) Co-$NiSe_2$/CN, (c) $CoSe_2$ and (d)$NiSe_2$.

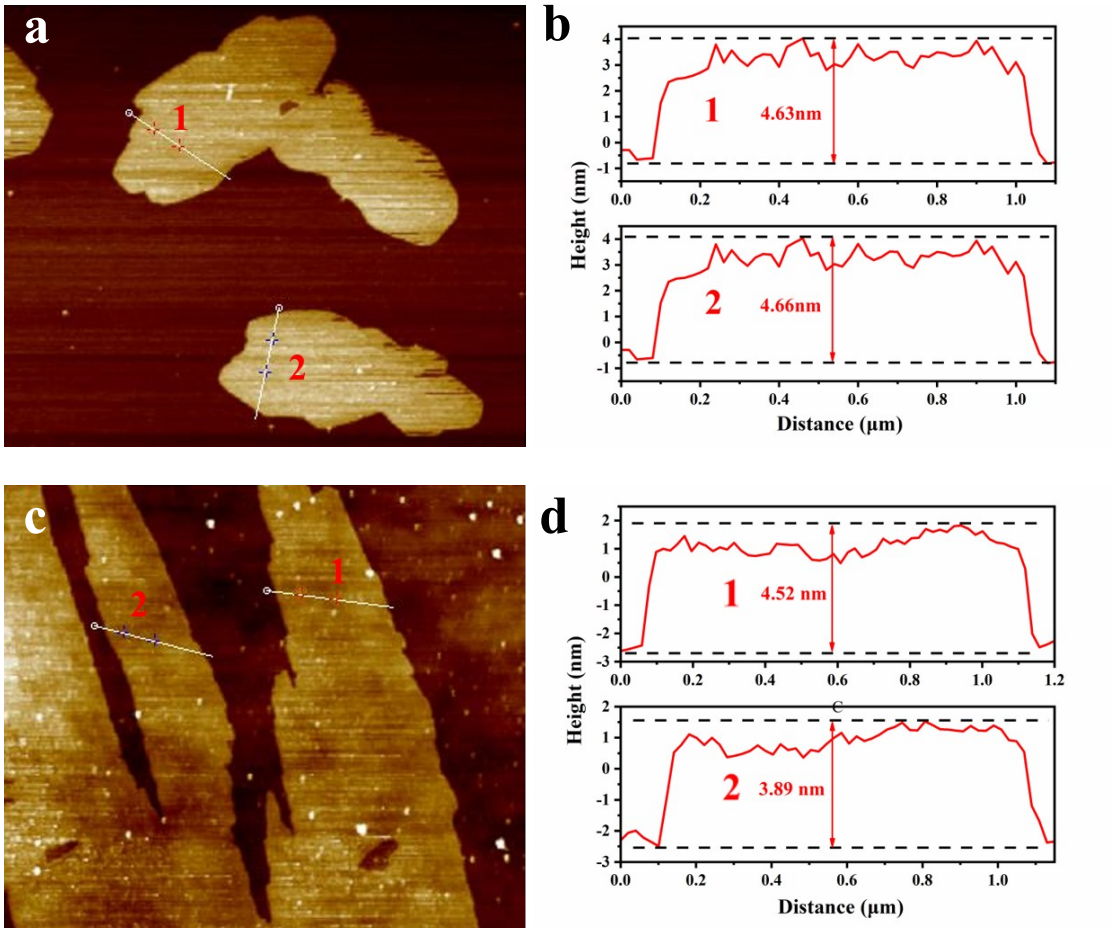


Figure S6 AFM image and the corresponding height profiles of 2D CP Precursor (a) and Co-$NiSe_2$/CN (d).

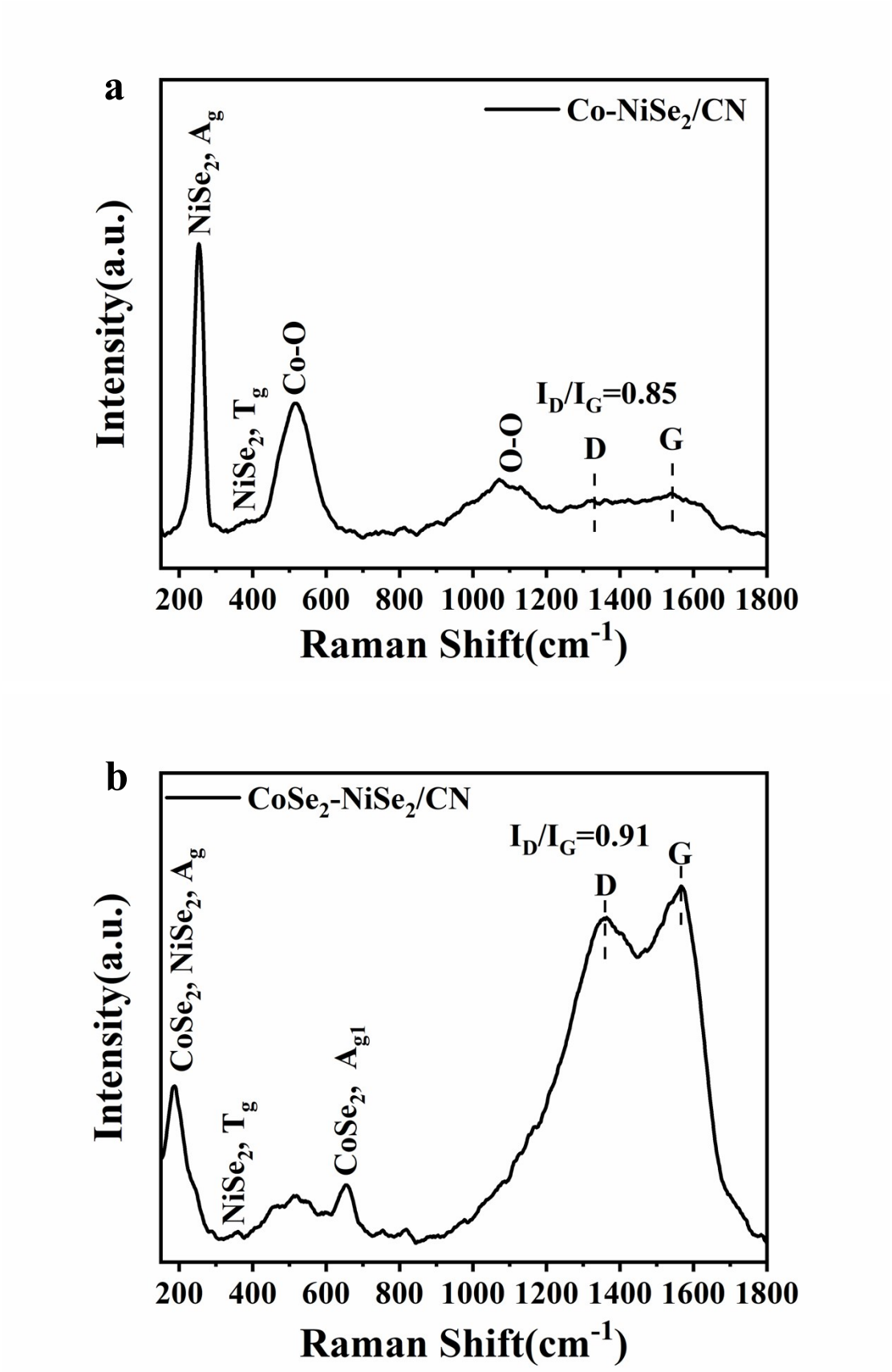


Figure S7 The Raman spectra of Co-$NiSe_2$/CN and $CoSe_2$-$NiSe_2$/CN.

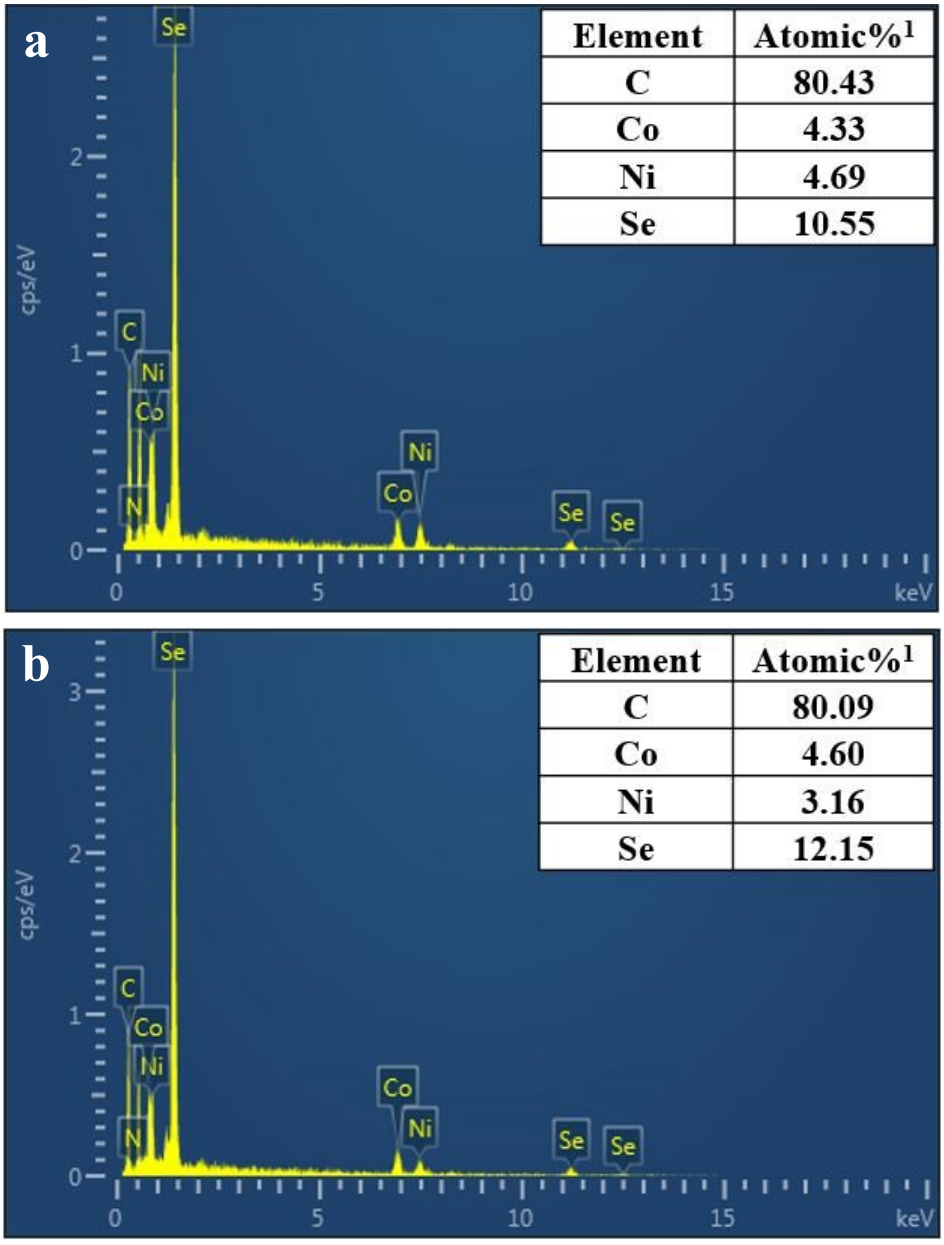


Figure S8 EDS spectra of Co-$NiSe_2$/CN and $CoSe_2$-$NiSe_2$/CN.

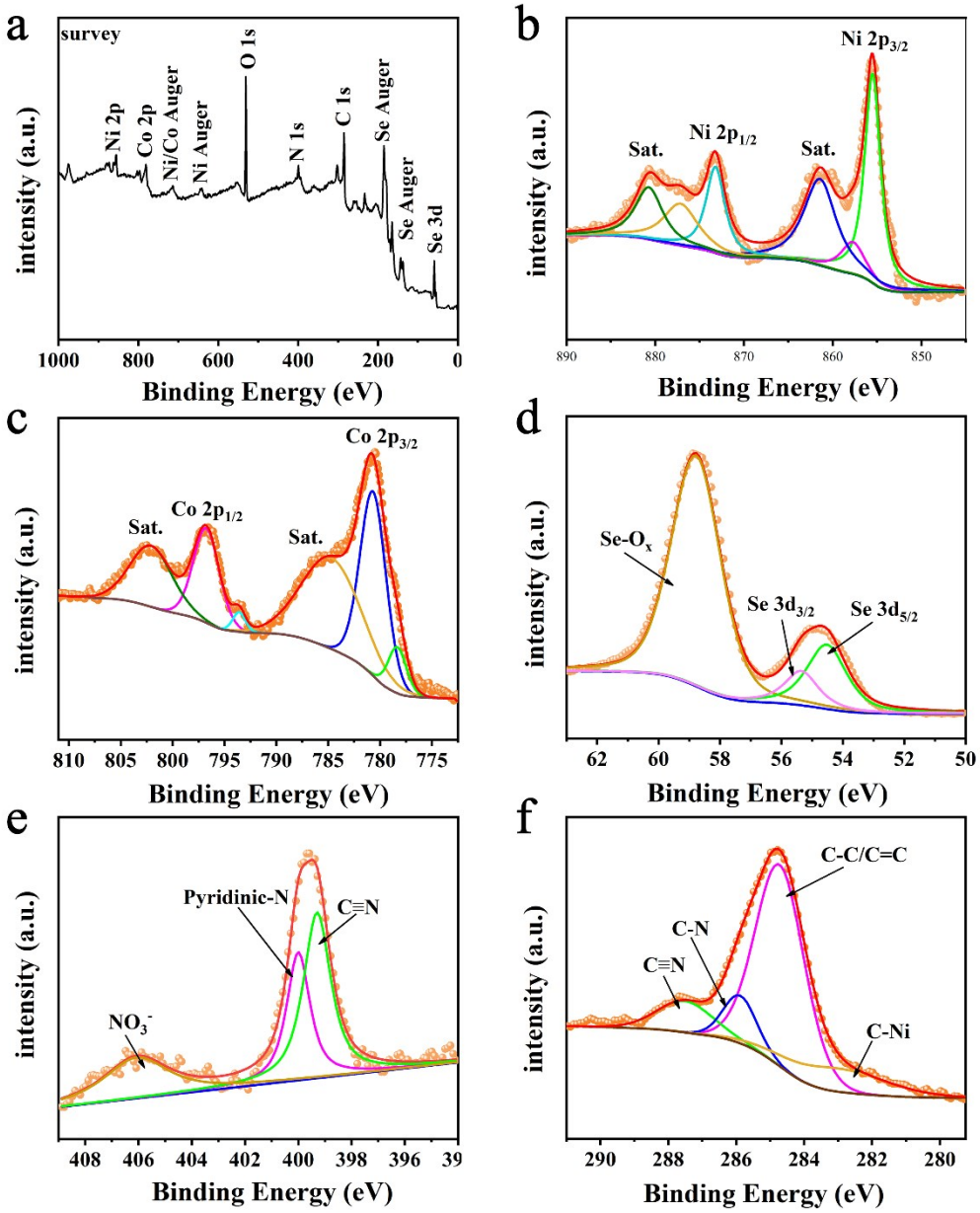


Figure S9 XPS spectra of Co-$NiSe_2$/CN: (a) Full scan spectrum; (b) Ni 2p; (c) Co 2p; (d) Se 3d; (e) N 1s and (f) C 1s.

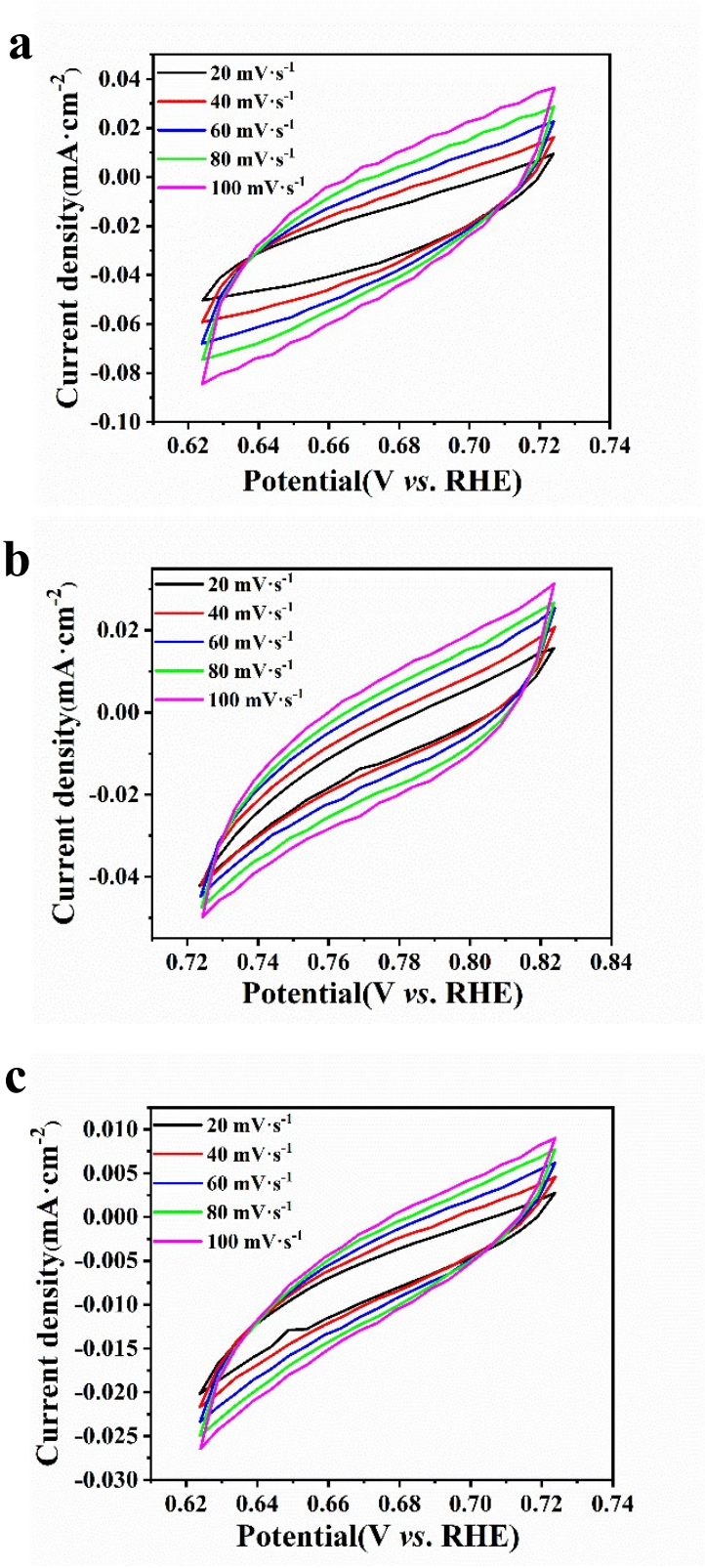


Figure S10 (a-c) CV curves of Co-$NiSe_2$/CN, $CoSe_2$ and $NiSe_2$ in 0.5 M H_2SO_4 respectively at different scan rates.

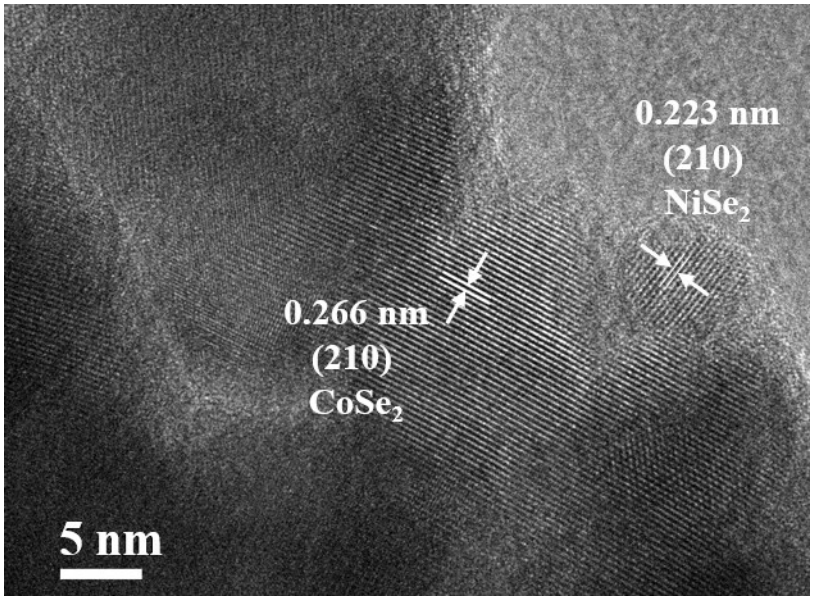


Figure S11 HRTEM image of $CoSe_2$-$NiSe_2$/CN after the electrochemical stability test.

References

1. J.-C. Dong, X.-G. Zhang, V. Briega-Martos, X. Jin, J. Yang, S. Chen, Z.-L. Yang, D.-Y. Wu, J. M. Feliu, C. T. Williams, Z.-Q. Tian and J.-F. Li, In situ Raman spectroscopic evidence for oxygen reduction reaction intermediates at platinum single-crystal surfaces, *Nat. Energy,* 2018**, 4**(1), 60-67.
2. J. Yang, H. Liu, W. N. Martens and R. L. Frost, Synthesis and Characterization of Cobalt Hydroxide, Cobalt Oxyhydroxide, and Cobalt Oxide Nanodiscs, *J. Phys. Chem. C,* 2010**, 114**(1), 111-119.