Supplementary Information (SI) for New Journal of Chemistry. This journal is © The Royal Society of Chemistry and the Centre National de la Recherche Scientifique 2025

Supporting Information

Fabrication of 3D Iron modified NiCo₂S₄ structure on Nickel foam as bifunctional

electrocatalyst for Overall Water Splitting



Figure S1. XRD pattern of (a) $NiCo_2S_4/NF$, (b) $Fe_{1.0\%}-NiCo_2S_4/NF$, (c) $Fe_{2.5\%}-NiCo_2S_4/NF$ and (d) $Fe_{5.0\%}-NiCo_2S_4/NF$.



Figure S2. SEM elemental mapping of $Fe_{2.5\%}$ -NiCo₂S₄/NF.



Figure S3. SEM images of (a) $NiCo_2S_4/NF$, (b) $Fe_{1.0\%}-NiCo_2S_4/NF$, (c) $Fe_{2.5\%}-NiCo_2S_4/NF$ and (d) $Fe_{5.0\%}-NiCo_2S_4/NF$.



Figure S4. Cyclic voltammograms of the catalyst in the non-faradaic region recorded at different scan rates in the N₂-saturated 1 M KOH solution of (a) NiCo₂S₄/NF, (b) Fe_{1.0%}-NiCo₂S₄/NF, (c) Fe_{2.5%}-NiCo₂S₄/NF and (d) Fe_{5.0%}-NiCo₂S₄/NF.



Figure S5. Plot of the anodic current against scan rate in the non-faradaic region of the CVs of (a) $NiCo_2S_4/NF$, (b) $Fe_{1.0\%}-NiCo_2S_4/NF$, (c) $Fe_{2.5\%}-NiCo_2S_4/NF$ and (d) $Fe_{5.0\%}-NiCo_2S_4/NF$.



Figure S6. Nyquist plots of bare Ni-foam and $NiCo_2S_4/NF$ for (a) HER polarization (b) OER polarization.



Figure S7. XRD pattern of $Fe_{2.5\%}$ -NiCo₂S₄/NF after HER and OER stability test.



Figure S8. EDS and SEM elemental mapping of Fe_{2.5%}-NiCo₂S₄/NF after HER stability test.



Figure S9. XPS spectrum of Ni 2p, Co 2p, Fe 2p and S 2p for $Fe_{2.5\%}$ -NiCo₂S₄/NF after HER stability test.



Figure S10. EDS and SEM elemental mapping of $Fe_{2.5\%}\text{-}NiCo_2S_4/NF$ after OER stability test.



Figure S11. XPS spectrum of Ni 2p, Co 2p, Fe 2p and S 2p for Fe_{2.5%}-NiCo₂S₄/NF after OER stability test.



Figure S12. Three independent replicates result of Chronopotentiometric of Fe $_{2.5\%}$ -NiCo $_2S_4$ /NF || Fe $_{2.5\%}$ -NiCo $_2S_4$ /NF at 20 mA cm⁻².

Sample	Ni	Со	Fe	S
NiCo ₂ S ₄ /NF	30.5 wt.%	24.3 wt.%	-	8.5 wt.%
Fe _{2.5%} -NiCo ₂ S ₄ /NF	22.3 wt.%	17.1 wt.%	4.3 wt.%	8.2 wt.%

Table S1. Elemental composition of $NiCo_2S_4/NF$ and $Fe_{2.5\%}$ - $NiCo_2S_4/NF$ samples.