**Oxide/sulfide-based hybrid arrays as robust electrocatalysts for water splitting**

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**Characterization technique**

The same volume of gas sample in the headspace of the electrolytic cell was withdrawn by a SGE gas-tight syringe and analyzed by gas chromatography (GC). The H₂ and O₂ in the sampled gas was separated by passing through a 2 m × 3 mm packed molecular sieve 5A column with an Ar carrier gas and quantified by a Thermal Conductivity Detector (TCD)(Shimadzu GC-9A).

![Graphs](image)

**Fig. S1** CVs of NiCo₂O₄/NF (a), NiCo₂O₄/Ni₃S₂/NF(b) and NF(c) with different scan rates (10-50 mV s⁻¹) in the region of 1.02-1.12V vs RHE.
Fig. S2 CVs of NiCo$_2$O$_4$/NF(a), NiCo$_2$O$_4$/Ni$_3$S$_2$/NF (b) and NF(c) with different scan rates (10-50 mV s$^{-1}$) in the region of -0.08—-0.02 V vs RHE.

Fig. S3 Electrocatalytic efficiency of H$_2$ production over NiCo$_2$O$_4$/Ni$_3$S$_2$/NF at a potential of ca. -0.2 V, measured for 60 min.
**Fig. S4** The OER polarization curves for the NiCo$_2$O$_4$/Ni$_3$S$_2$/NF before and after 500 cycles of the accelerated stability test.

**Fig. S5** O$_2$ production over NiCo$_2$O$_4$/Ni$_3$S$_2$/NF at a potential of ca. 1.43 V, measured for 1h.
Fig. S6 Polarization curve (iR uncorrected) of the NiCo$_2$O$_4$/Ni$_3$S$_2$/NF for water splitting with a scan rate of 5 mV s$^{-1}$ in 1 M KOH.

Fig. S7 H$_2$ and O$_2$ production over NiCo$_2$O$_4$/Ni$_3$S$_2$/NF at a potential of ca. 1.60 V, measured for 8h.