Construction of MnCo$_2$O$_4$@Ni$_3$M$_x$ (S and P) crosslinked network for efficient electrocatalytic water splitting

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Gas detection

The same volume of gas sample in the headspace of the electrolytic cell was withdrawn by a SGE gas-tight syringe and analyzed by gas chromatography (GC).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 H$_2$ 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).

![Graph showing OER polarization curves for different concentrations of MnCo$_2$(OH)$_6$](image-url)

**Fig. S1** OER polarization curves of MnCo$_2$(OH)$_6$-10mmol, MnCo$_2$(OH)$_6$-15mmol and MnCo$_2$(OH)$_6$-20mmol.
Fig. S2 HER polarization curves of MnCo$_2$(OH)$_6$-10mmol, MnCo$_2$(OH)$_6$-15mmol and MnCo$_2$(OH)$_6$-20mmol.

Fig. S3 OER polarization curves of MnCo$_2$O$_4$@Ni(OH)$_2$-9h, MnCo$_2$O$_4$@Ni(OH)$_2$-12h and MnCo$_2$O$_4$@Ni(OH)$_2$-15h.
Fig. S4 HER polarization curves of MnCo$_2$O$_4$@Ni(OH)$_2$-9h, MnCo$_2$O$_4$@Ni(OH)$_2$-12h and MnCo$_2$O$_4$@Ni(OH)$_2$-15h.

Fig. S5 CV$_S$ of MnCo$_2$(OH)$_6$-15mmol (a), MnCo$_2$O$_4$@Ni(OH)$_2$-12h (b), MnCo$_2$O$_4$@Ni$_3$S$_2$ (c) and MnCo$_2$O$_4$@NiP$_2$ (d) with different scan rates (10-50 mV s$^{-1}$) in the region of 1.02-1.12V vs RHE.
Fig. S6 CVs of MnCo$_2$(OH)$_6$-15mmol (a), MnCo$_2$O$_4$@Ni(OH)$_2$-12h (b), MnCo$_2$O$_4$@Ni$_3$S$_2$ (c) and MnCo$_2$O$_4$@NiP$_2$ (d) with different scan rates (10-50 mV s$^{-1}$) in the region of -0.06 — 0V vs RHE.
Fig. S7 XPS spectra of MnCo$_2$O$_4$@Ni$_3$S$_2$ after electrochemical testing: (a) survey scan, (b) Mn 2p, (c) Co 2p, (d) Ni 2p, (e) S 2p and (f) O 1s.
Fig. S8 A photograph showing generation of $O_2$ bubbles on the MnCo$_2$O$_4$@Ni$_3$S$_2$ electrodes.

Fig. S9 LSV curve of MnCo$_2$O$_4$@Ni$_3$S$_2$ for water splitting before and after reaction.
Fig. S10 (a) Kinetics of O$_2$ and H$_2$ formation in the electrocatalytic system using MnCo$_2$O$_4$@Ni$_3$S$_2$. 