Supporting information

Engineering of self-supported carbon electrode with 2D ultrathin heterostructure of NiCo LDH/NiCoS via a MOF-template for sensitive detection of glucose and H₂O₂

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Preparation of NiCoS/CC: 0.58 g Ni(NO₃)₂·6H₂O, 1.16 g Co(NO₃)₂·6H₂O, 0.17 g urea and 0.21 g NH₄F were dissolved in 80 mL DI water and stirred for 10 min. Then a piece of CC ($1 \times 2 \text{ cm}^2$) and the mixed solution were transferred to 100 mL autoclave and heated at 100°C for 6 h. The sample was rinsed with DI water and dried for 10 h. Finally, 6 mg TAA was dissolved in 40 mL ethanol and stirred. Then the mixed solution and a piece of sample were transferred to a 100 mL autoclave and heated at 120 °C for 4 h.

Preparation of Co MOF/NiCoS/CC: 0.58 g Ni(NO₃)₂·6H₂O, 1.16 g Co(NO₃)₂·6H₂O, 0.17 g urea and 0.21 g NH₄F were dissolved in 80 mL DI water and stirred for 10 min. Then a piece of Co MOF ($1 \times 2 \text{ cm}^2$) and the mixed solution were transferred to 100 mL autoclave and heated at 100°C for 6 h. The sample was rinsed with DI water and dried for 10 h. The sulfurization process is the same as above.



Fig. S1 SEM and TEM images of obtained carbon electrodes with (a, b) Co MOF and (c, d) NiCo LDH.



Fig. S2 XRD patterns of obtained carbon electrodes with Co MOF, NiCo LDH and NiCo LDH/NiCoS



Fig. S3 The full XPS spectra of obtained carbon electrode with NiCo LDH/NiCoS.



Fig. S4 (a), (b) SEM, (c) TEM and (d) HR-TEM images of carbon electrode with NiCo LDH/NiCoS.



Fig. S5 Peak current densities of the NiCo LDH/NiCoS/CC electrode towards glucose oxidation with different concentrations of NaOH.



Fig. S6 SEM images of (a) NiCoS/CC and (b) Co MOF/NiCoS/CC electrodes.



Fig. S7 CV curves of obtained carbon electrodes with NiCoS and Co MOF/NiCoS in the presence (solid line) and absence (dashed line) of 1 mM glucose in 0.5 M NaOH at a scan rate of 20 mV s⁻¹.



Fig. S8 Plots of log I_{pa} vs. log v.



Fig. S9 Plots of concentration vs. current density of NiCo LDH/NiCoS/CC electrode.



Fig. S10 Response time to glucose oxidation of NiCo LDH/NiCoS/CC electrode.



Fig. S11 Sensitivity of the NiCo LDH/NiCoS/CC electrode towards 5 mM H₂O₂ reduction in different NaOH concentrations.



Fig. S12 CV curves of obtained carbon electrodes with NiCoS and Co MOF/NiCoS in the presence (solid line) and absence (dashed line) of 1 mM H_2O_2 in 0.5 M NaOH at a scan rate of 50 mV s⁻¹.



Fig. S13 (a) CV curves of the NiCo LDH/NiCoS/CC electrode to 3 mM H_2O_2 at different scan rates (10 to 80 mV s⁻¹), (b) the fitting the linear relationship between the current density of the reduction peak vs. $v^{1/2}$.



Fig. S14 (a) Responses of H_2O_2 reduction with the obtained NiCo LDH/NiCoS/CC electrodes at different applied potentials (-0.25 V to -0.4 V) and (b) plots of concentration vs. current density.



Fig. S15 Response time to H_2O_2 reduction of NiCo LDH/NiCoS/CC electrode.