

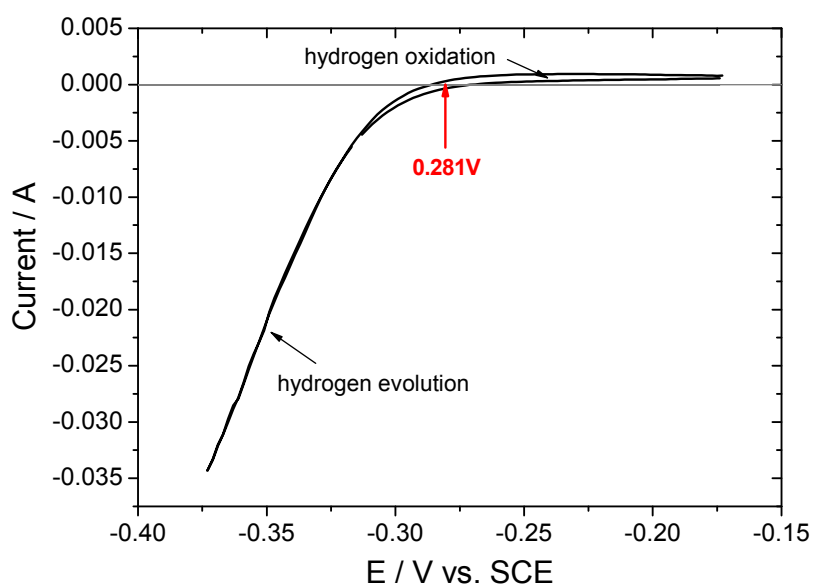
Mixed-Solution Synthesis of Sea Urchin-Like NiSe Nanofiber Assemblies as Economical Pt-Free Catalysts for Electrochemical H₂ Production

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RHE Calibration. In all measurements, we used SCE as the reference electrode. It was calibrated with respect to RHE. The calibration was performed in the high purity hydrogen saturated electrolyte with a Pt foil as the working electrode. Cyclic voltammetry (CV) was run at a scan rate of 1 mV s⁻¹, and the average of the two potentials at which the current crossed zero was taken to be the thermodynamic potential for the hydrogen electrode reaction. The CV result was shown below:



Therefore, in 0.5 M H₂SO₄ solution, E(RHE) = E(SCE) + 0.281 V.

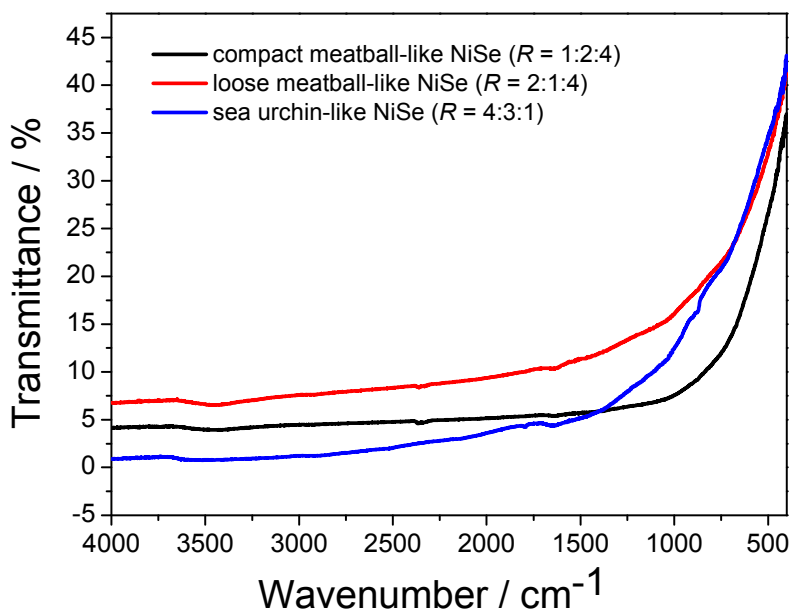


Fig. S1 FT-IR spectra of the samples prepared at 180 °C for 12 h in a ternary solution with different volume ratios of $V_{\text{DETA}}/V_{\text{Ni}^{2+}\text{H}^{+}\text{H}_2\text{O}}/V_{\text{DIW}}$.

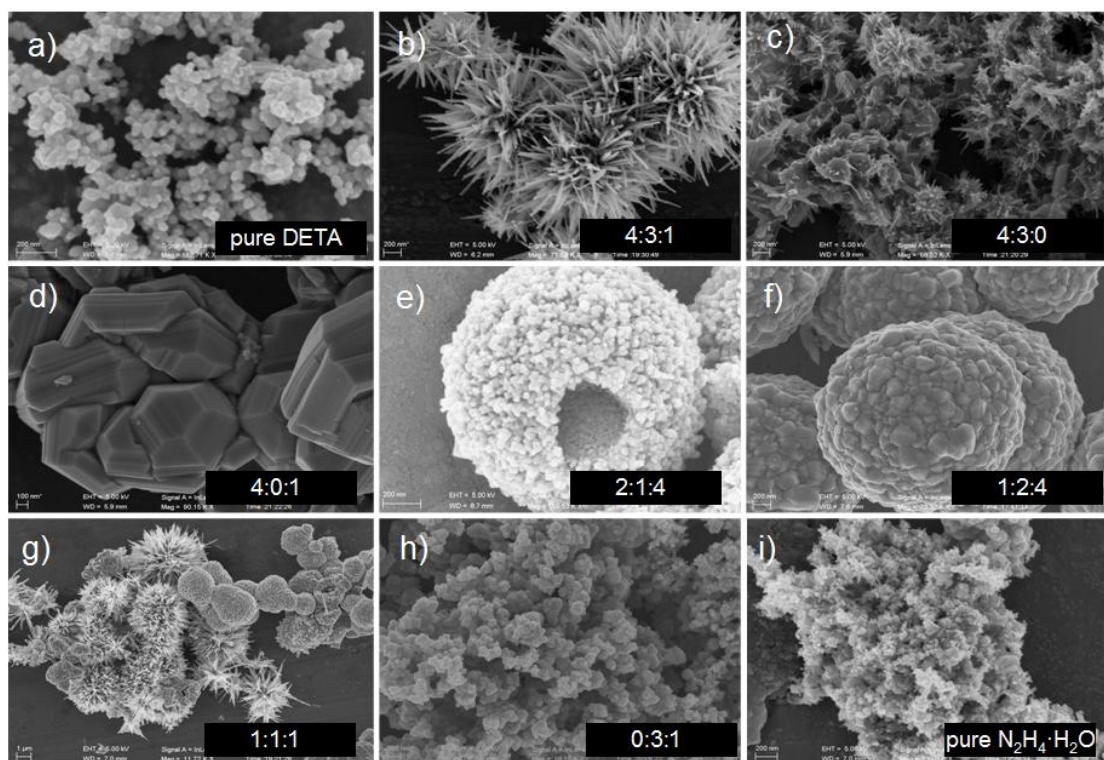


Fig. S2 SEM images of the products prepared at 180 °C for 12 h in a ternary solution with different volume ratios of $V_{\text{DETA}}/V_{\text{N}_2\text{H}_4\cdot\text{H}_2\text{O}}/V_{\text{DIW}}$.

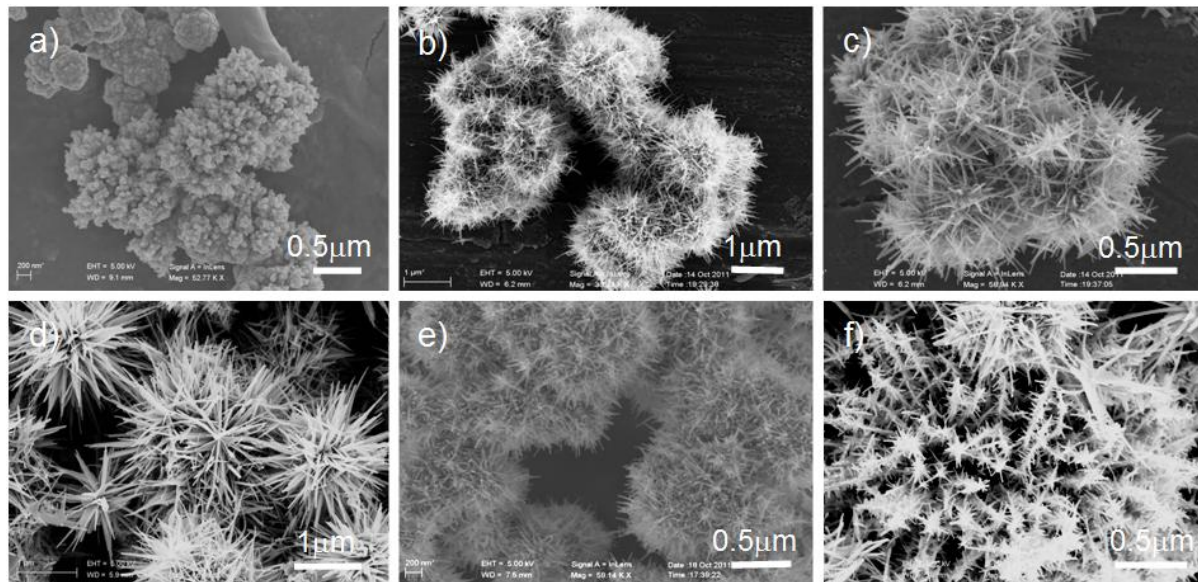


Fig. S3 SEM images of the samples prepared at 180 °C in a ternary solution ($V_{\text{DETA}}/V_{\text{N}_2\text{H}_4\cdot\text{H}_2\text{O}}/V_{\text{DIW}} = 4:3:1$) for different reaction times. (a) 1 h; (b) 4 h; (c) 8 h; (d) 12 h; (e) 16 h; (f) 20 h.