

## Supplementary Information

### Controllable synthesis of nickel sulfides integrated with carbon fibers towards enhanced hydrogen evolution reaction kinetic

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#### Characterization

The scanning electron microscopy (SEM) characterization was carried out via a FEI Quanta 250 FEG. X-ray power diffraction (XRD) patterns were obtained with a Cu K $\alpha$  radiation on a Bruker D8 ADVANCE diffractometer ( $\lambda = 1.5604 \text{ \AA}$ ). The Raman spectra was performed via Aramis confocal Raman microscope ( $\lambda = 532 \text{ nm}$ ). The elemental valence states of the samples were tested by X-ray photoelectron spectroscopy (XPS) with Al K $\alpha$  as the X-ray source on a Thermo-VG Scientific Escalab 250Xi spectrometer. The nitrogen adsorption-desorption characterization was investigated with an ASAP2460 Micromeritics equipment at 77 K. High-resolution transmission electron microscopy (HRTEM) and elemental mapping analysis were collected via America FEI Talos F200i.

#### Electrochemical measurements

Electrochemical measurements were conducted on a CHI 760E electrochemical workstation at 25 °C. The as-prepared catalyst directly was used as a working electrode, a

carbon rod as the counter electrode and a mercury/mercuric oxide electrode (Hg/HgO, filled with 1.0 M KOH) as the reference electrode. All potentials were referenced to the reversible hydrogen electrode (RHE) according to the equation:  $E_{\text{RHE}} = E_{\text{Hg/HgO}} + 0.059 \times \text{pH} + 0.098 \text{ V}$ . Linear sweep voltammetry (LSV) was carried out at a scan rate of  $2 \text{ mV s}^{-1}$ . The electrochemically active surface area (ECSA) of the catalyst was evaluated by measuring the double-layer capacitance ( $C_{\text{dl}}$ ) with cyclic voltammetry. The stability performance was measured by chronopotentiometry method. EIS was measured within the voltage range of 0.1-1.0 V at frequency ranging from  $10^5 \text{ Hz}$  to 1 Hz to analyze reaction kinetic of HER.

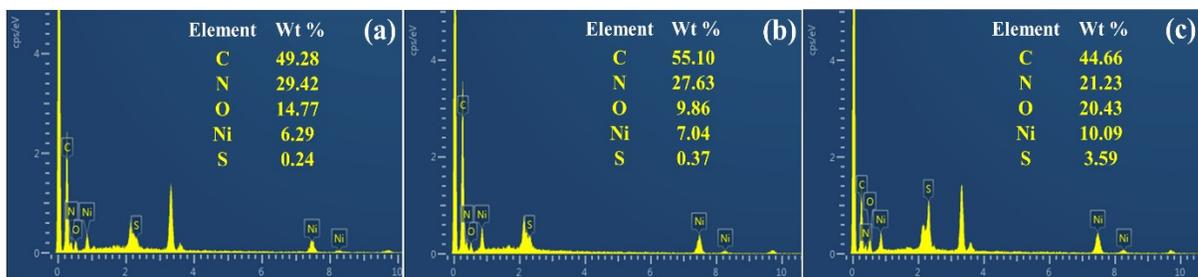


Fig. S1 EDX spectra of (a) NiS-0@CNFs, (b) NiS-1@CNFs and (c) NiS-2@CNFs.

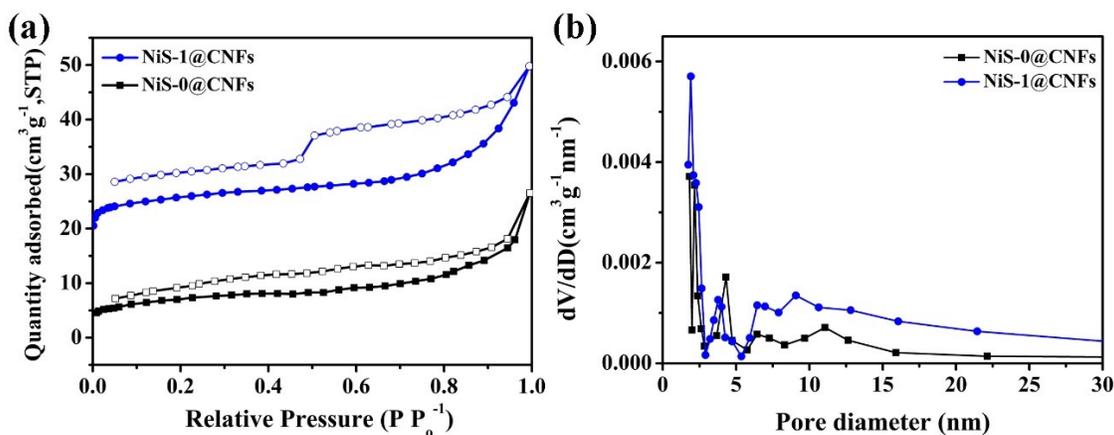


Fig. S2  $\text{N}_2$  adsorption/desorption isotherms (a) and pore size distribution (b) of NiS-y@CNFs

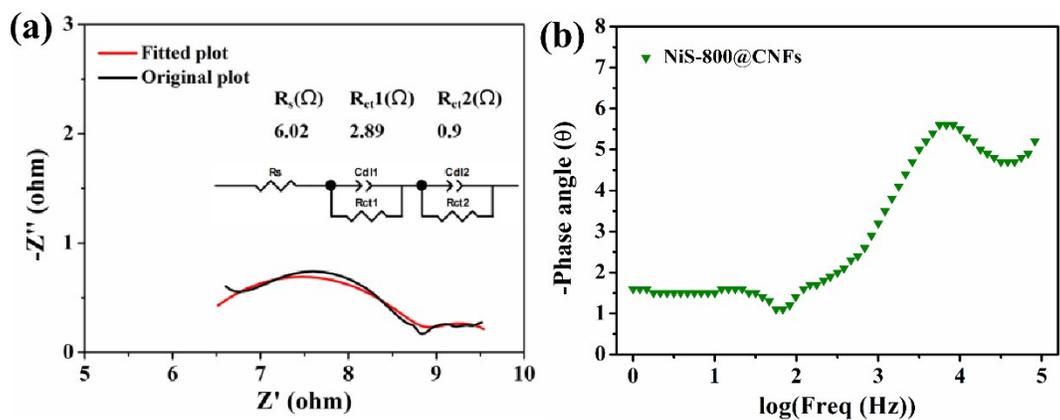


Fig. S3 Nyquist plot and Bode images of NiS-800@CNFs

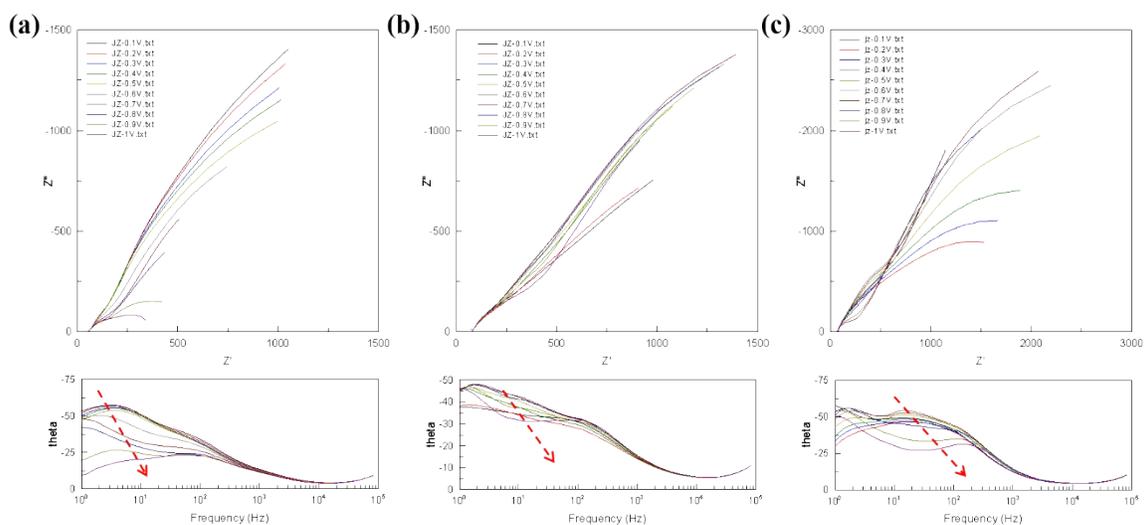


Fig. S4 Nyquist and Bode images of NiS-x@CNFs at various overpotentials

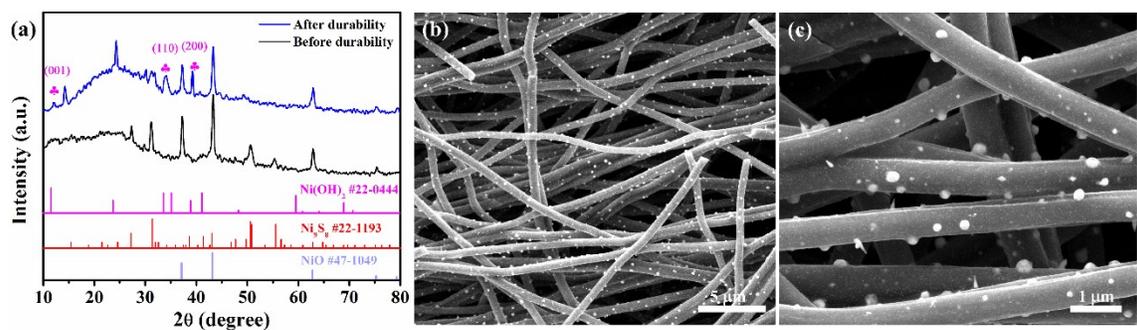


Fig. S5 (a) XRD pattern and (b, c) SEM images of NiS-800@CNFs after durability at high current density of  $200 \text{ mA cm}^{-2}$ .

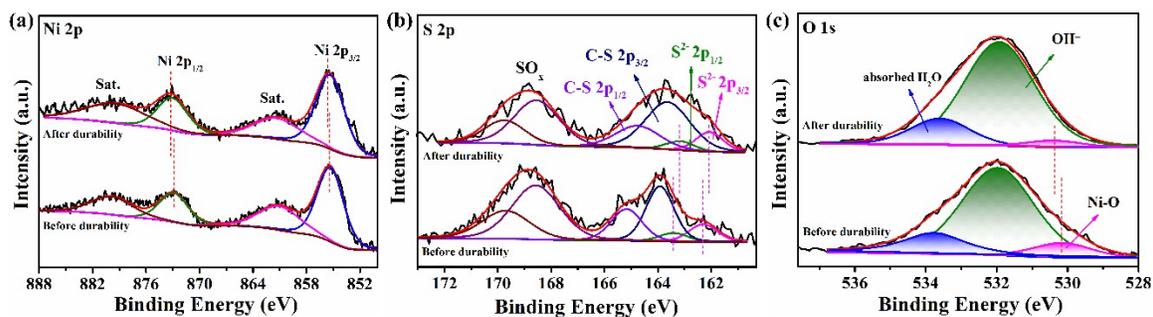


Fig. S6 XPS spectra of Ni 2p (a), S 2p (b) and O 1s (c) for NiS-800@CNFs after durability.

Table S1 Properties comparison of recent reported electrocatalysts for alkaline HER.

Catalyst	Overpotential at 10 mA cm <sup>-2</sup> (mV)	Tafel slope (mV dec <sup>-1</sup> )	Testing substrate	Ref
NiS-800@CNFs	119	102.1	No-substrate	This work
NiS-ZnS/rGO-ST/NF	161	133	Silica template	1
CoS <sub>x</sub> /Ni <sub>3</sub> S <sub>2</sub> @NF	204	133.32	Nickel foam	2
NiS-Ni <sub>2</sub> P <sub>2</sub>	147	68	GCE	3
Zn-NiS-3	208	113	Nickel foam	4
ZCS@rGO	135	47	Reduced graphene oxide	5
NiS/Mo <sub>2</sub> CT <sub>x</sub>	157	77	GCE	6
VS/Ni <sub>x</sub> S <sub>y</sub> /NF	125	113	Nickel foam	7
NiFeVS <sub>x</sub> @NF	127	121	Nickel foam	8
MoS <sub>2</sub> /CoS <sub>2</sub> /CC	147	90.5	Carbon cloth	9
Mo-NiS <sub>x</sub> /NF	155	88	Nickel foam	10

a. GCE: glassy carbon electrode

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