

## Supporting Information

### Ni<sub>3</sub>Se<sub>4</sub>/Fe(PO<sub>3</sub>)<sub>2</sub>/NF Composites as high-efficiency electrocatalysts with a low overpotential for the oxygen evolution reaction

Ting-Yu Shuai, Qi-Ni Zhan, Hui-Min Xu, Chen-Jin Huang, Zhi-Jie Zhang, Hong-Rui Zhu, Gao-Ren Li\*

College of Materials Science and Engineering, Sichuan University, Chengdu 610065, China

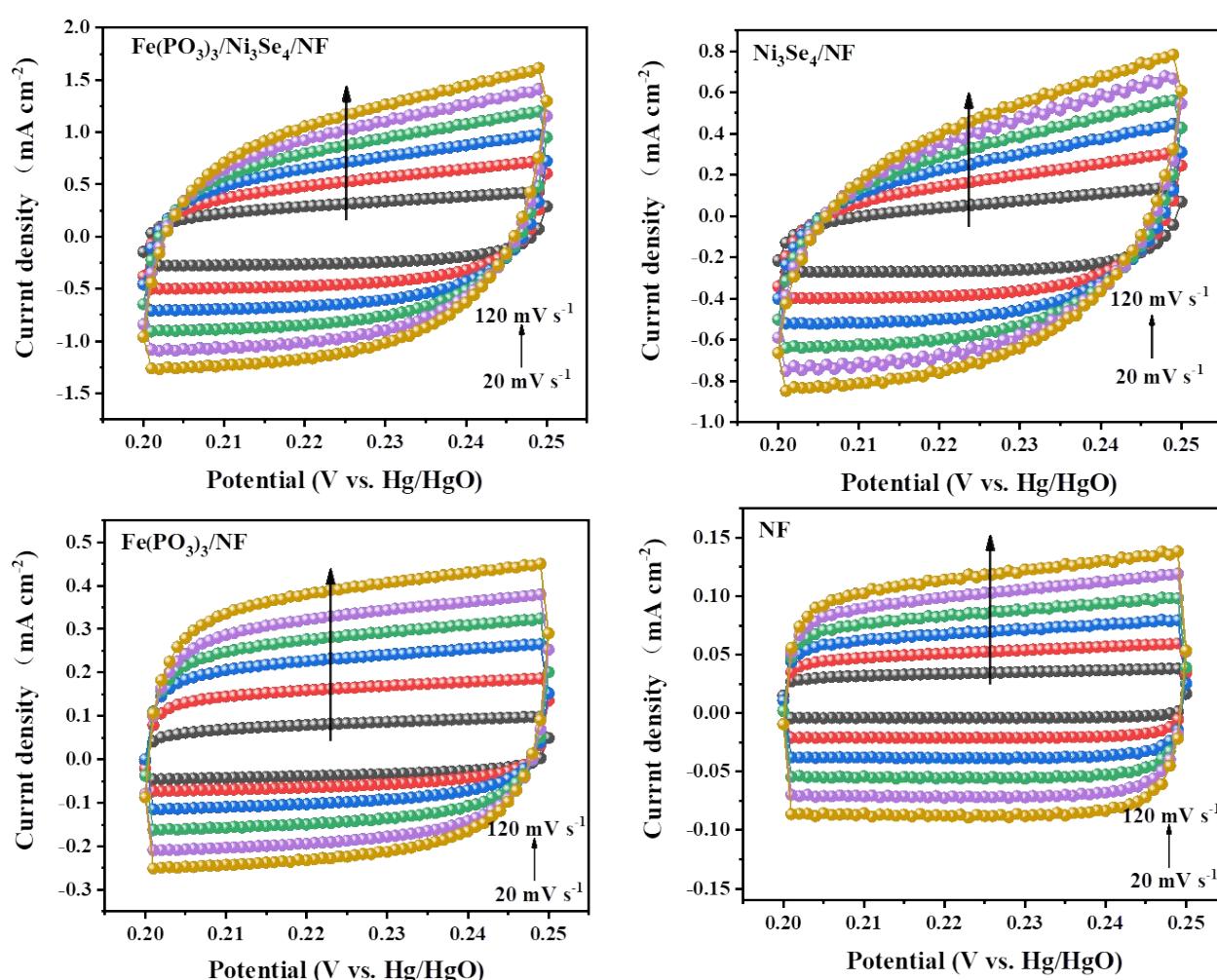
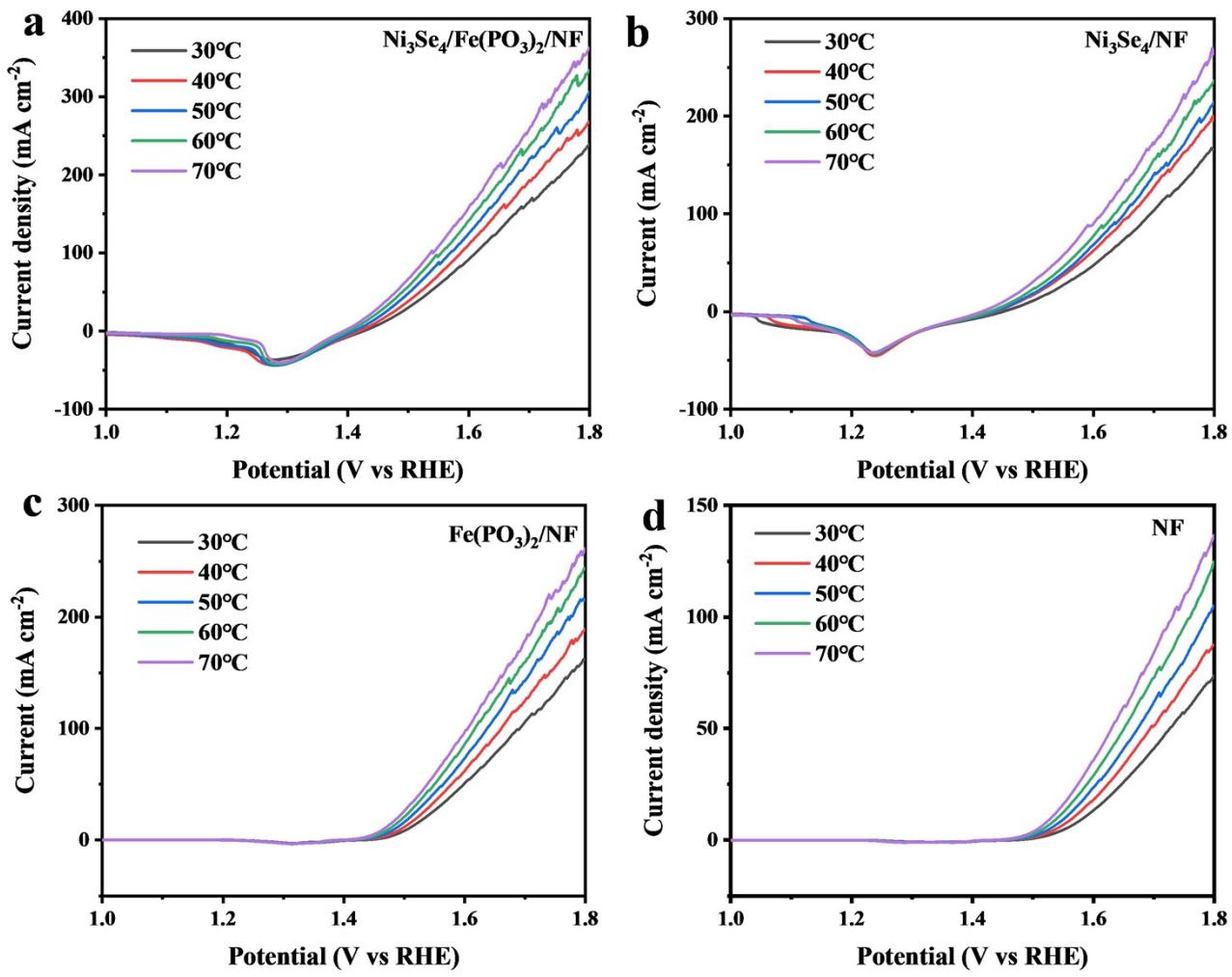
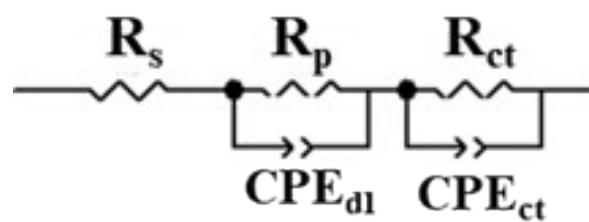


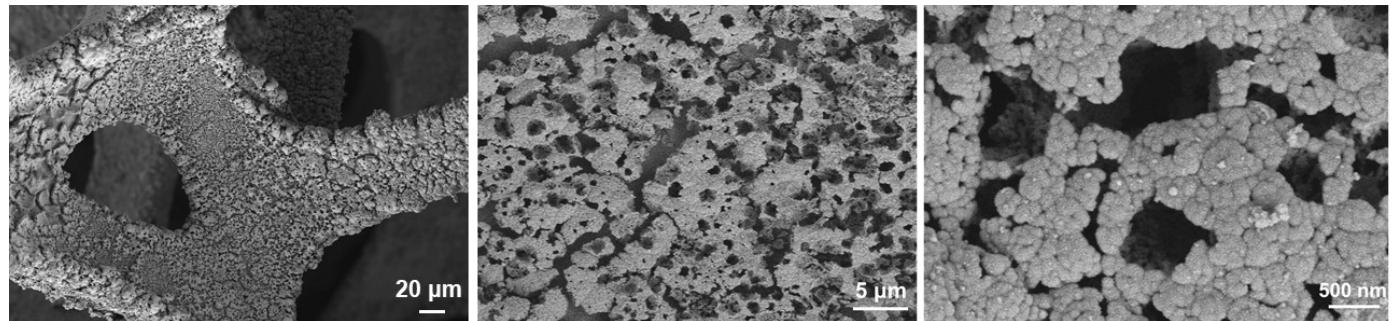
Fig. S1 CVs of (a) Ni<sub>3</sub>Se<sub>4</sub>/Fe(PO<sub>3</sub>)<sub>2</sub>/NF, (b) Ni<sub>3</sub>Se<sub>4</sub>/NF, (c) Fe(PO<sub>3</sub>)<sub>2</sub>/NF, (d) NF from 20 to 120  $\text{mV S}^{-1}$ .



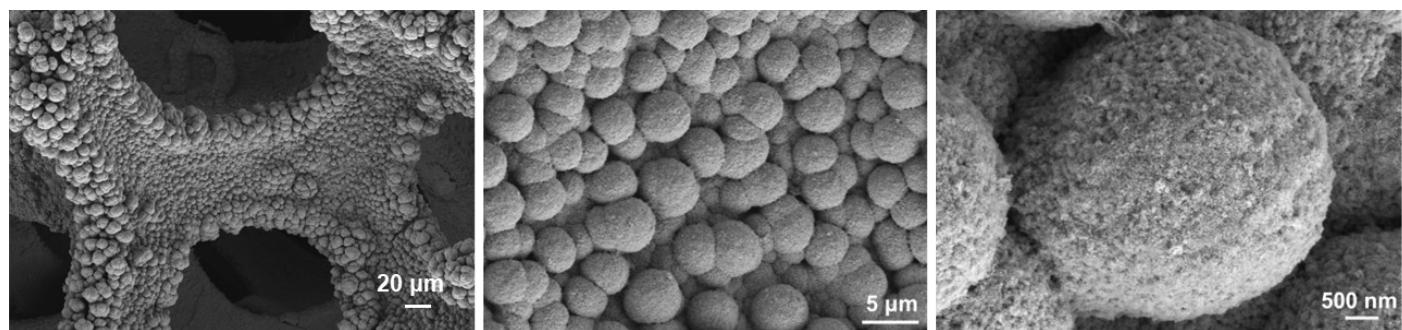
**Fig. S2** Polarization curves of (a) Ni<sub>3</sub>Se<sub>4</sub>/Fe(PO<sub>3</sub>)<sub>2</sub>/NF, (b) Ni<sub>3</sub>Se<sub>4</sub>/NF, (c) Fe(PO<sub>3</sub>)<sub>2</sub>/NF, (d) NF at different temperatures without IR compensation.



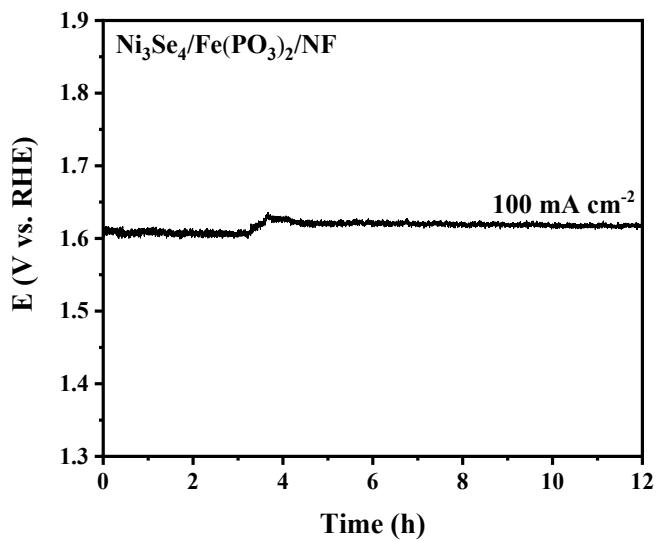
**Fig. S3** The equivalent circuit model for electrochemical impedance spectrum fitting.



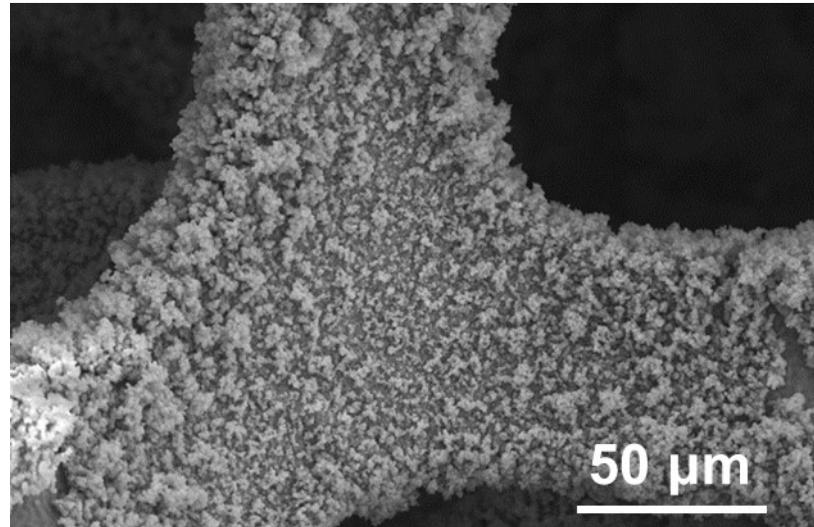
**Fig. S4** SEM images of Ni<sub>3</sub>Se<sub>4</sub>/NF with different magnifications.



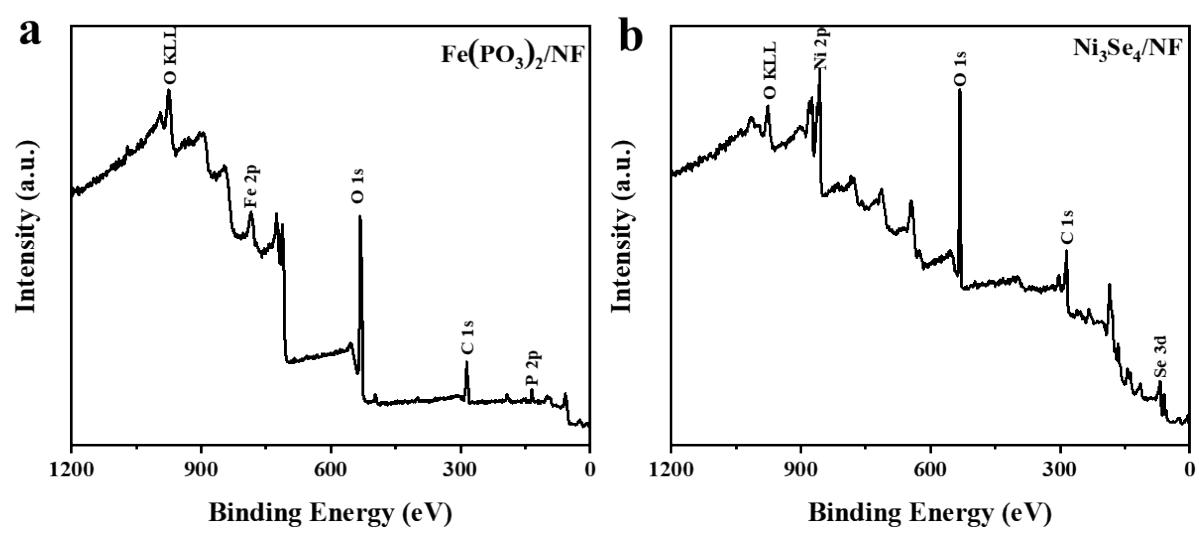
**Fig. S5** SEM images of Fe(PO<sub>3</sub>)<sub>3</sub>/NF with different magnifications.



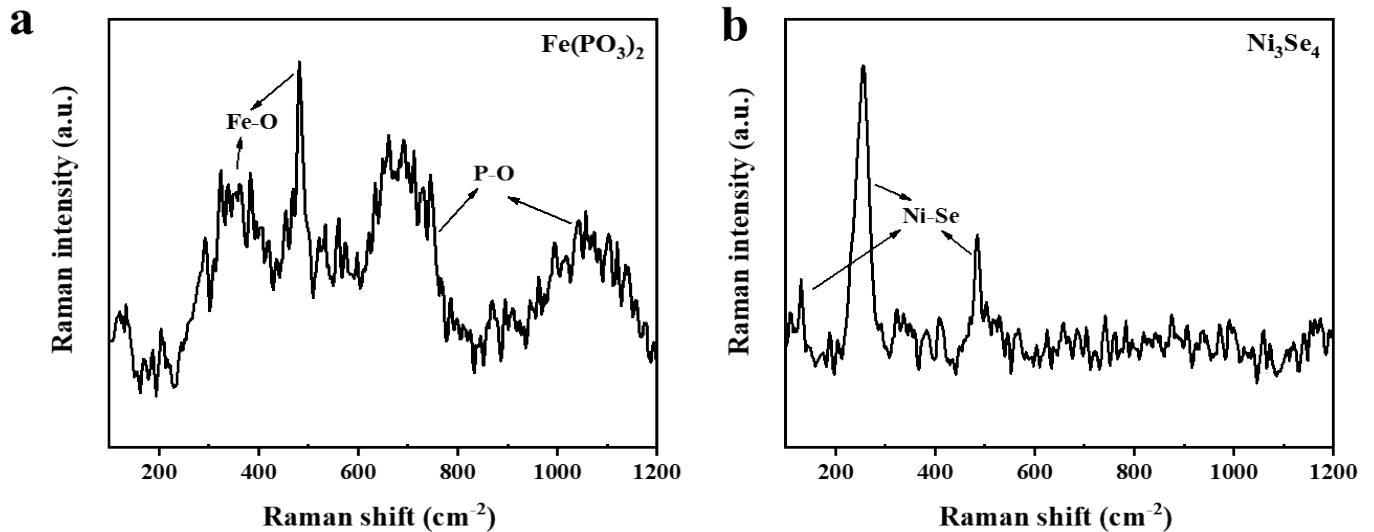
**Fig. S6** Stability test of  $\text{Ni}_3\text{Se}_4/\text{Fe}(\text{PO}_3)_2/\text{NF}$  at the current density of  $100 \text{ mA cm}^{-2}$  for 12 h without IR compensation.



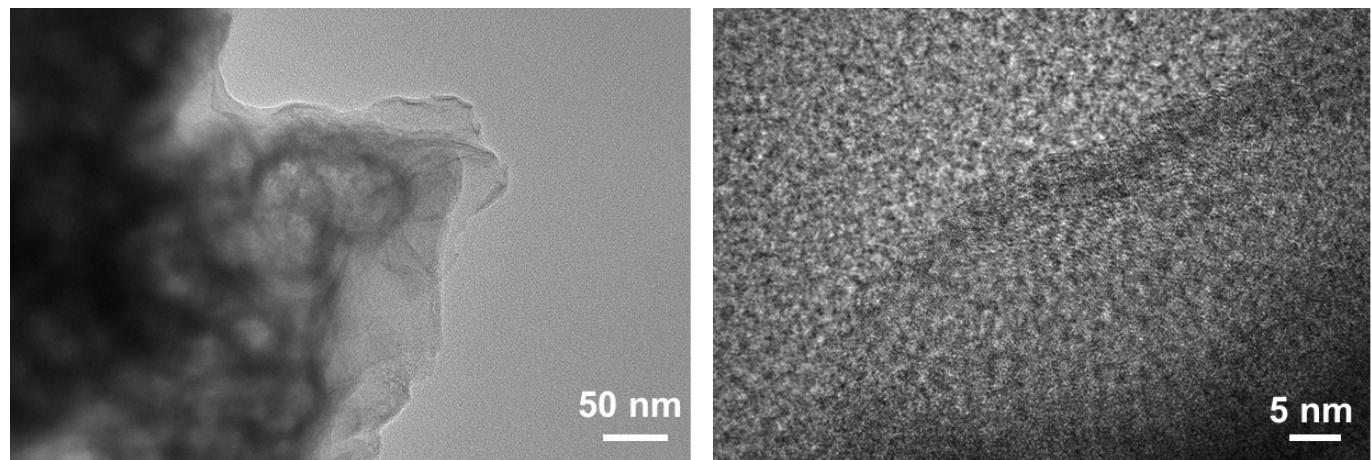
**Fig. S7** SEM images of  $\text{Ni}_3\text{Se}_4/\text{Fe}(\text{PO}_3)_3/\text{NF}$  after 12 h OER stability test at a constant current density of 10  $\text{mA cm}^{-2}$  (the NF skeleton structure was not destroyed).



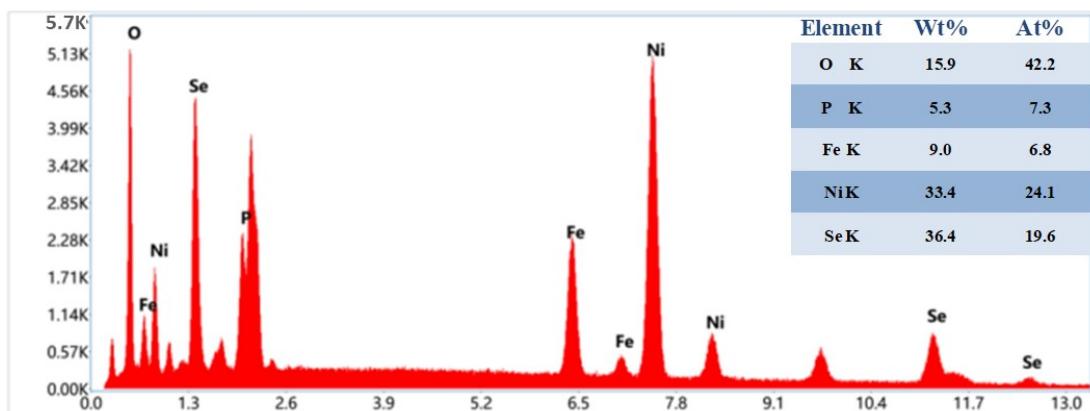
**Fig. S8** XPS elemental survey spectra of (a)  $\text{Fe}(\text{PO}_3)_2/\text{NF}$  and (b)  $\text{Ni}_3\text{Se}_4/\text{NF}$  samples.



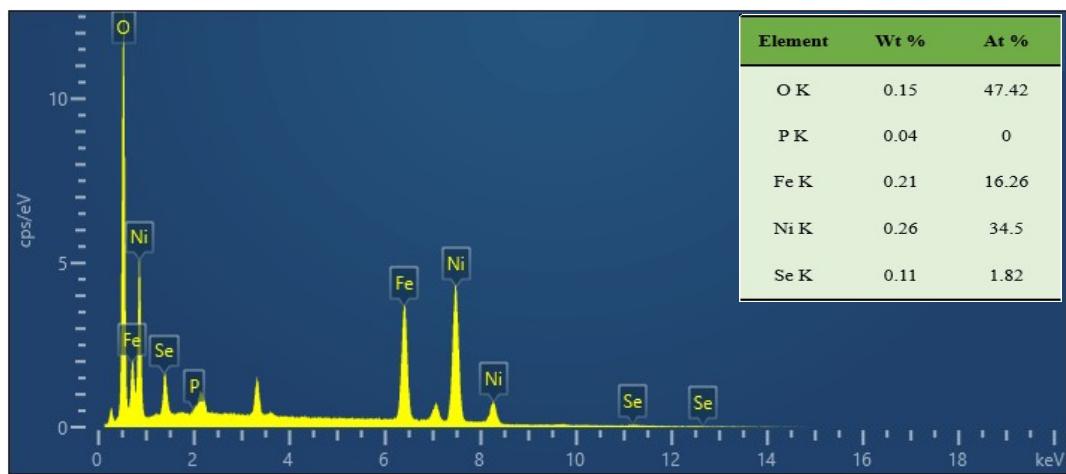
**Fig. S9** Raman shift curves of (a)  $\text{Fe}(\text{PO}_3)_2$  and (b)  $\text{Ni}_3\text{Se}_4$  samples.



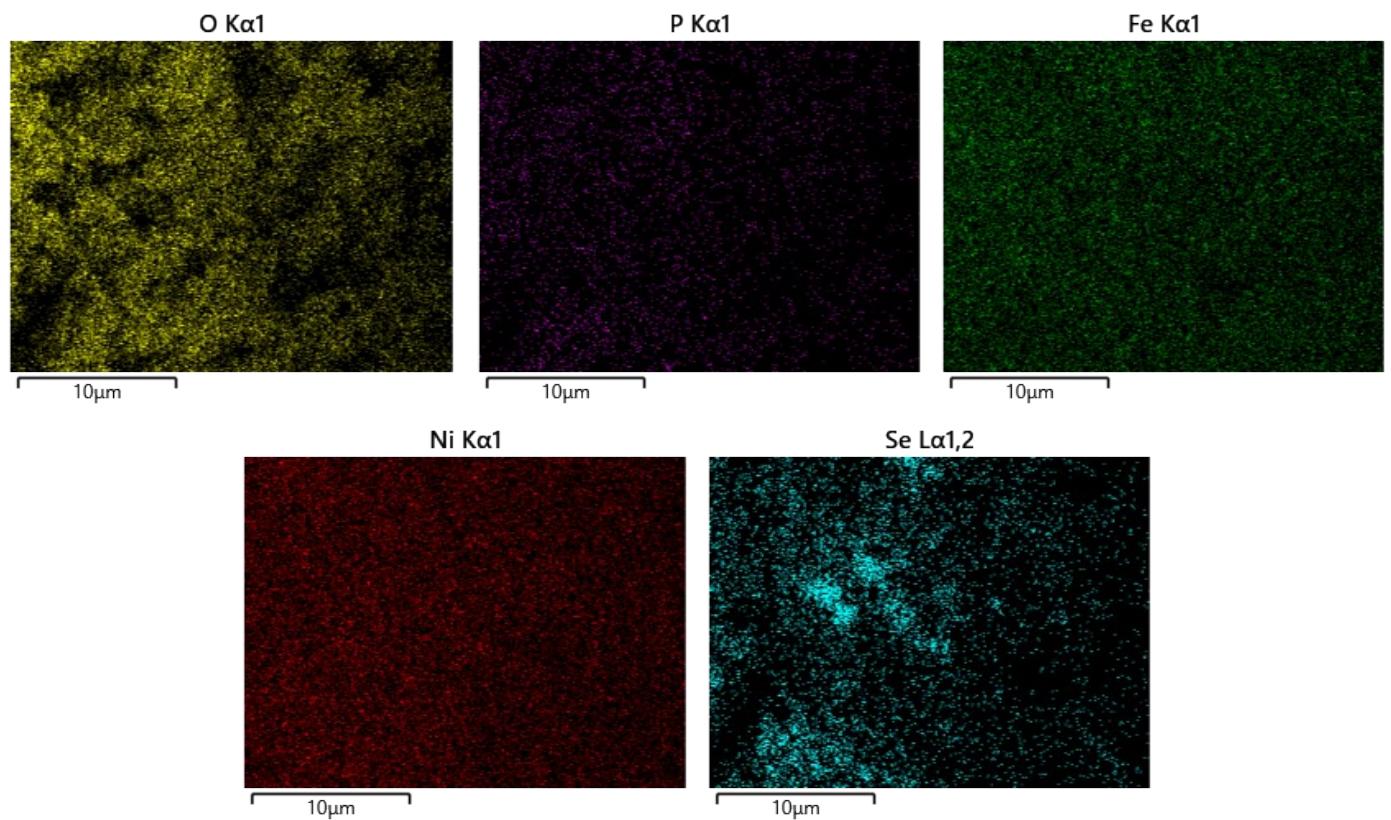
**Fig. S10** TEM images of polycrystalline region of  $\text{Ni}_3\text{Se}_4/\text{Fe}(\text{PO}_3)_2/\text{NF}$  with different magnifications.



**Fig. S11** EDS pattern of the  $\text{Ni}_3\text{Se}_4/\text{Fe}(\text{PO}_3)_2/\text{NF}$ .



**Fig. S12** EDS pattern of the  $\text{Ni}_3\text{Se}_4/\text{Fe}(\text{PO}_3)_2/\text{NF}$  after 12 h stability test at  $10 \text{ mA cm}^{-2}$ .



**Fig. S13** EDS mappings of Ni, Fe, Se, P in  $\text{Ni}_3\text{Se}_4/\text{Fe}(\text{PO}_3)_2/\text{NF}$  after 12 h stability test at  $10 \text{ mA cm}^{-2}$ .

**Table S1.** Summary of EIS fitting results of different catalysts for OER in 1.0 M KOH.

Electrocatalyst	$R_s(\Omega)$	$R_{ct}(\Omega)$	$R_p(\Omega)$
$\text{Ni}_3\text{Se}_4/\text{Fe}(\text{PO}_3)_2/\text{NF}$	1.408	2.283	0.42865
$\text{Ni}_3\text{Se}_4/\text{NF}$	1.084	6.602	0.57529
$\text{Fe}(\text{PO}_3)_2/\text{NF}$	1.298	9.664	0.40404
NF	1.318	44.65	1.724

**Table S2.** BET surface areas, average pore sizes (adsorption) and total pore volumes of various samples.

Sample ID	BET surface area ( $\text{m}^2 \text{ g}^{-1}$ )	Average pore size (nm)	Pore volume ( $\text{cm}^3 \text{ g}^{-1}$ )
$\text{Ni}_3\text{Se}_4/\text{Fe}(\text{PO}_3)_2$	76.765	3.2506	0.076405
$\text{Ni}_3\text{Se}_4$	25.5082	7.5142	0.065105
$\text{Fe}(\text{PO}_3)_2$	17.312	9.7661	0.052543

**Table S3.** The mass and molar ratio of Se and P in Ni<sub>3</sub>Se<sub>4</sub>/Fe(PO<sub>3</sub>)<sub>2</sub>/NF analyzed by ICP-OES.

Element	mass ratio	molar ratio
Se	24.4%	28.4%
P	3.2%	10.4%

**Table S4.** Summary of Electrochemical double-layer capacitance ( $C_{dl}$ ) and ECSA of different samples.

Sample	$C_{dl}$ (mF cm $^{-2}$ )	ECSA (cm $^2$ )
Ni <sub>3</sub> Se <sub>4</sub> /Fe(PO <sub>3</sub> ) <sub>2</sub> /NF	8.5	212.5
Ni <sub>3</sub> Se <sub>4</sub> /NF	4.3	107.5
Fe(PO <sub>3</sub> ) <sub>2</sub> /NF	2.5	62.5
NF	0.8	20.0

**Table S5.** Comparisons of the electrocatalytic performance of NiFe-based catalysts for OER in 1.0 M KOH.

Electrocatalyst	Overpotential (mV)	Tafel slope (mV dec <sup>-1</sup> )	Ref
Ni <sub>3</sub> Se <sub>4</sub> /Fe(PO <sub>3</sub> ) <sub>2</sub> /NF	185( $\eta_{10}$ )	30.4	This work
(Ni,Fe) <sub>3</sub> Se <sub>4</sub>	225( $\eta_{10}$ )	41	<sup>1</sup>
Ni <sub>3</sub> Se <sub>4</sub> /FeOOH	249( $\eta_{10}$ )	46	<sup>2</sup>
Ni <sub>3</sub> Se <sub>4</sub> /NiFe LDH/CFC	223( $\eta_{10}$ )	55.5	<sup>3</sup>
Fe(PO <sub>3</sub> ) <sub>2</sub> /Ni <sub>2</sub> P/NF	177( $\eta_{10}$ )	51.9	<sup>4</sup>
CCS-NiFeP-10	201( $\eta_{10}$ )	41.2	<sup>5</sup>
Fe-18h/NF	220( $\eta_{10}$ )	45.2	<sup>6</sup>
Fe-Ni <sub>2</sub> P@C/NF	255( $\eta_{200}$ )	64	<sup>7</sup>
Fe-Ni <sub>3</sub> S <sub>2</sub>	290( $\eta_{100}$ )	46.9	<sup>8</sup>
FeCoNiS/NF	164( $\eta_{10}$ )	23.2	<sup>9</sup>
Fe-Ni <sub>5</sub> P <sub>4</sub> /NiFeOH-350	221( $\eta_{10}$ )	35.0	<sup>10</sup>
Ni/Fe-MI/OH	229( $\eta_{10}$ )	30.0	<sup>11</sup>
Ni <sub>3</sub> S <sub>2</sub> /MIL-53(Fe)	214( $\eta_{10}$ )	33.8	<sup>12</sup>
d-NiFe-LDH	230( $\eta_{10}$ )	77.0	<sup>13</sup>
NiFe-LDH/NiS/NF	230( $\eta_{10}$ )	60.1	<sup>14</sup>
Ni <sub>3</sub> S <sub>2</sub> @Fe-NiP <sub>x</sub> /NF	240( $\eta_{100}$ )	46.5	<sup>15</sup>
Fe-NiO/NiS <sub>2</sub>	270( $\eta_{10}$ )	40	<sup>16</sup>

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