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Electronic Supplementary Information

2 **Nickel selenide from single molecule electrodeposition**
3 **for efficient electrocatalytic overall water splitting**

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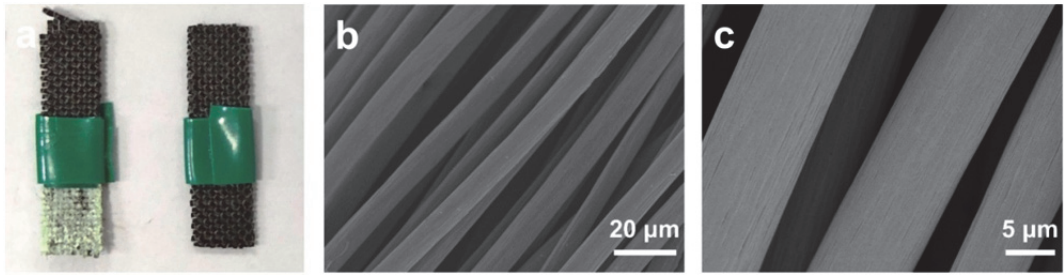
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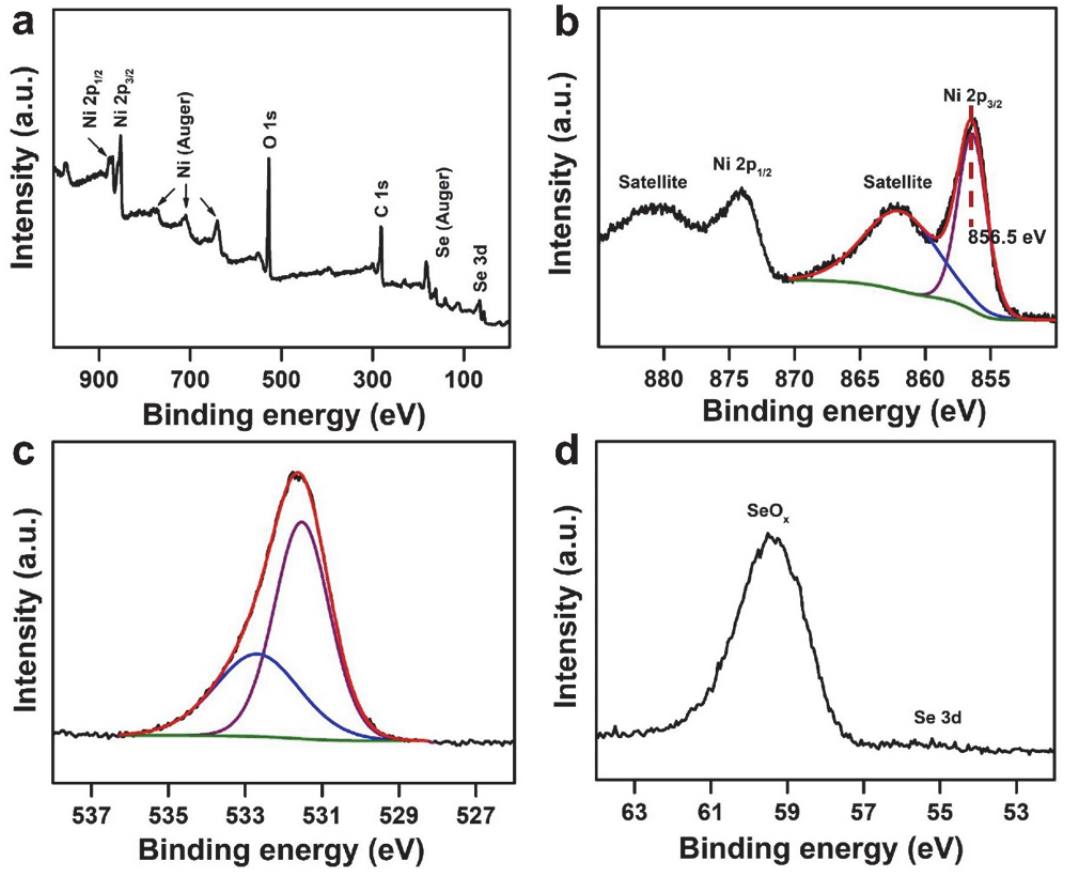
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7 **Figure S1.** (a) Photographs of blank CC substrate (right) and NiSe-TMEDA/CC
8 (left). (b, c) SEM images of blank carbon cloth.

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14 **Figure S2.** (a) XPS survey scan spectrum of NiSe-TMEDA/CC. High-resolution XPS
15 spectra in the (b) Ni 2p, (c) O 1s and (d) Se 3d regions for NiSe-TMEDA/CC.

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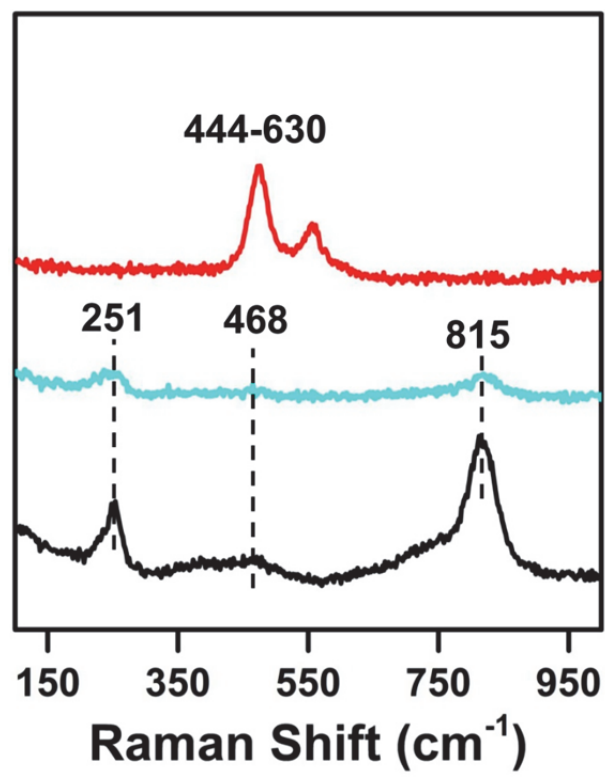


Figure S3. Raman spectra of the NiSe-TMEDA/CC electrode surface before (black lines) and after HER (blue line) or OER (red lines) electrolysis.

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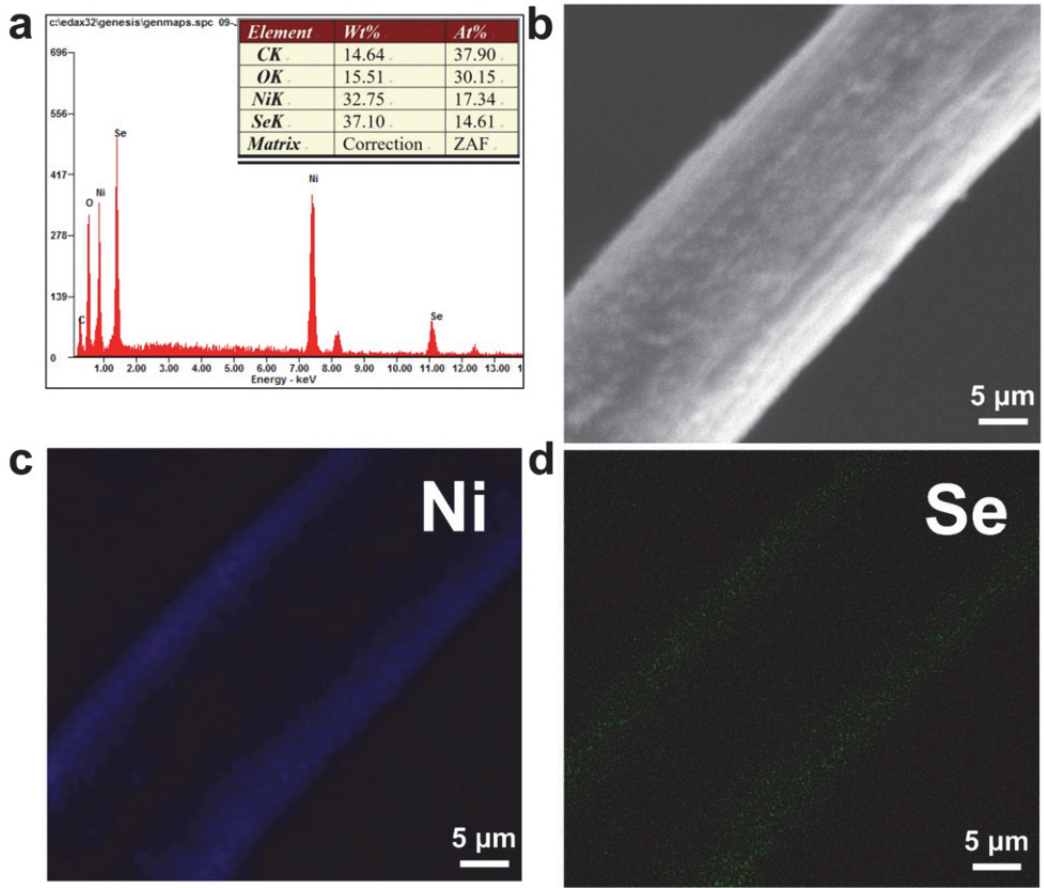
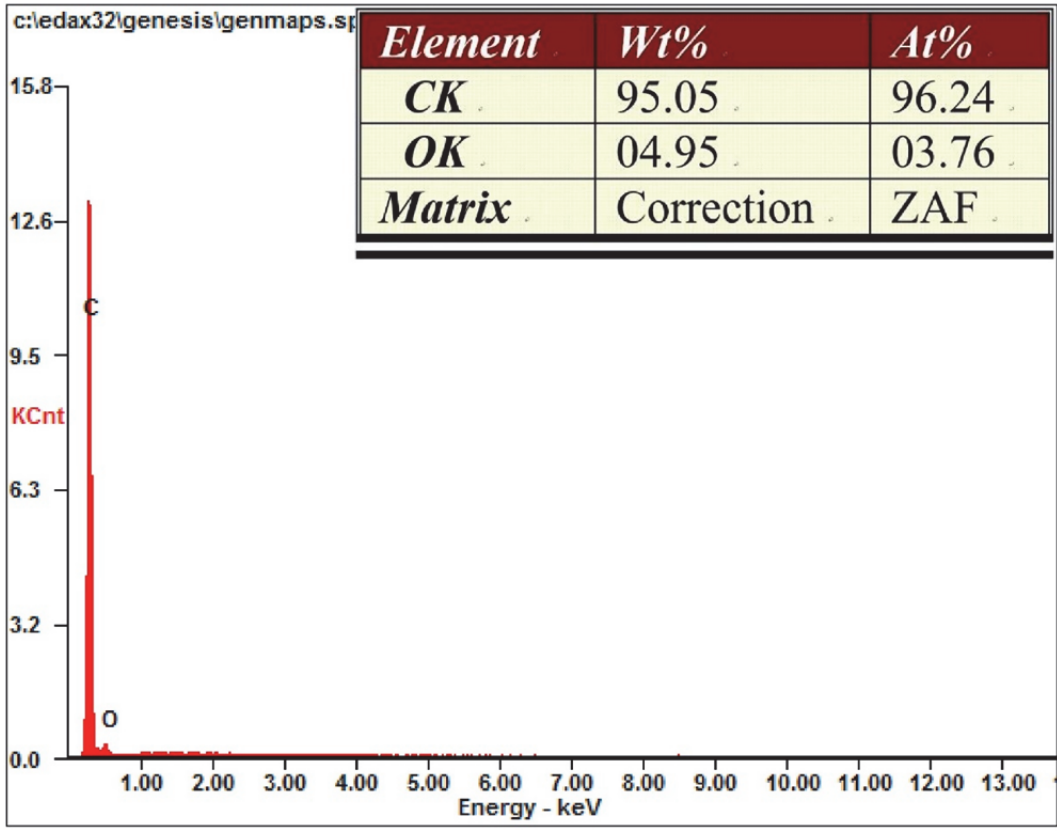


Figure S4. (a) EDX spectrum of the NiSe-TMEDA/CC. (b) SEM image of NiSe-TMEDA/CC and the corresponding element mapping images of Ni (c) and Se (d).

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14 **Figure S5.** EDX spectrum of the blank CC.

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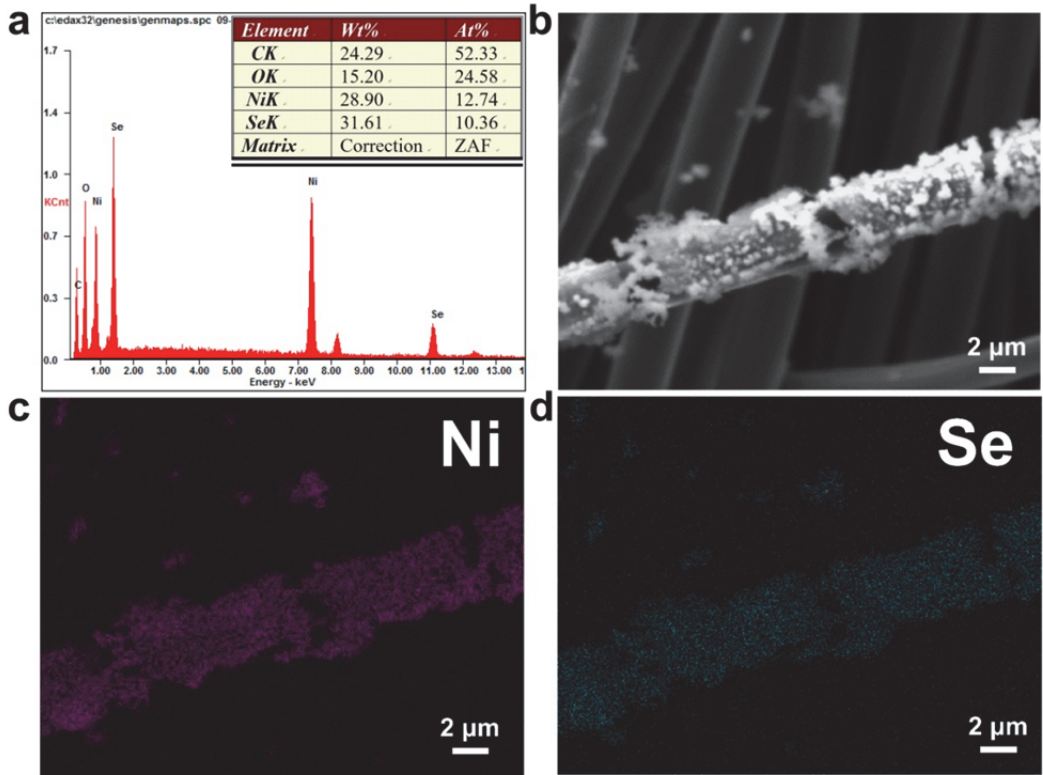


Figure S6. (a) EDX spectrum of NiSe/CC. (b) SEM image of the NiSe/CC electrode and the corresponding element mapping images of Ni (c) and Se (d).

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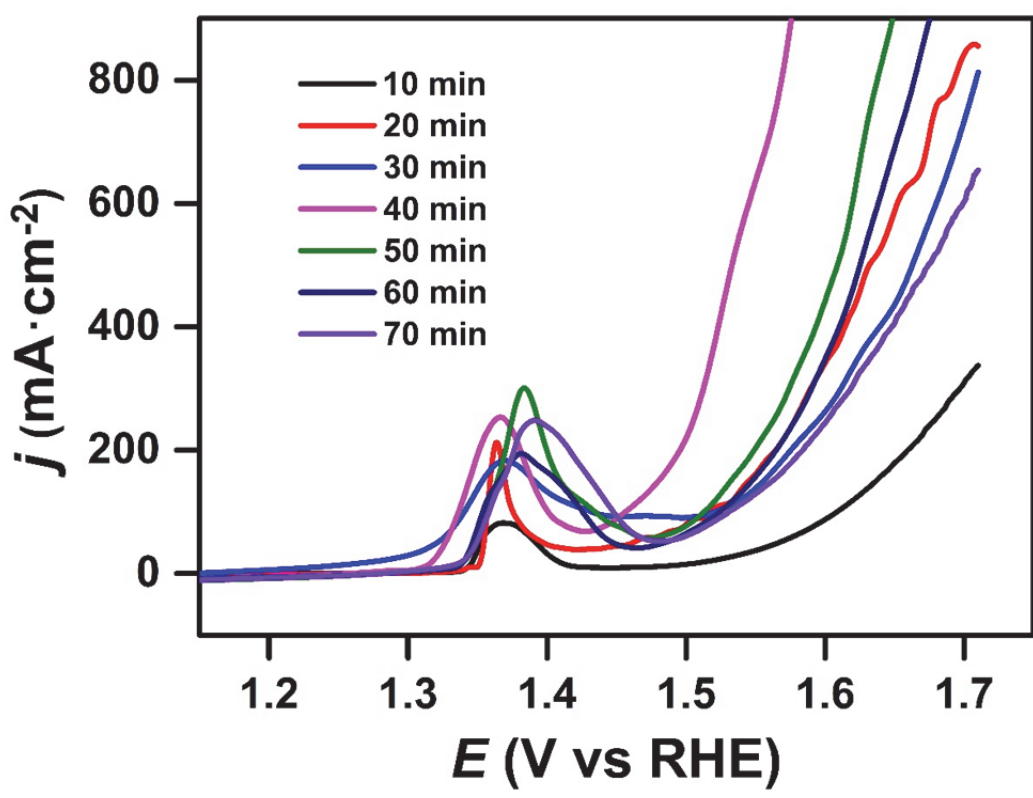


Figure S7. The OER performance of electrodes with different mass loading by adjusting the electrodeposition time during the preparation of the electrodes.

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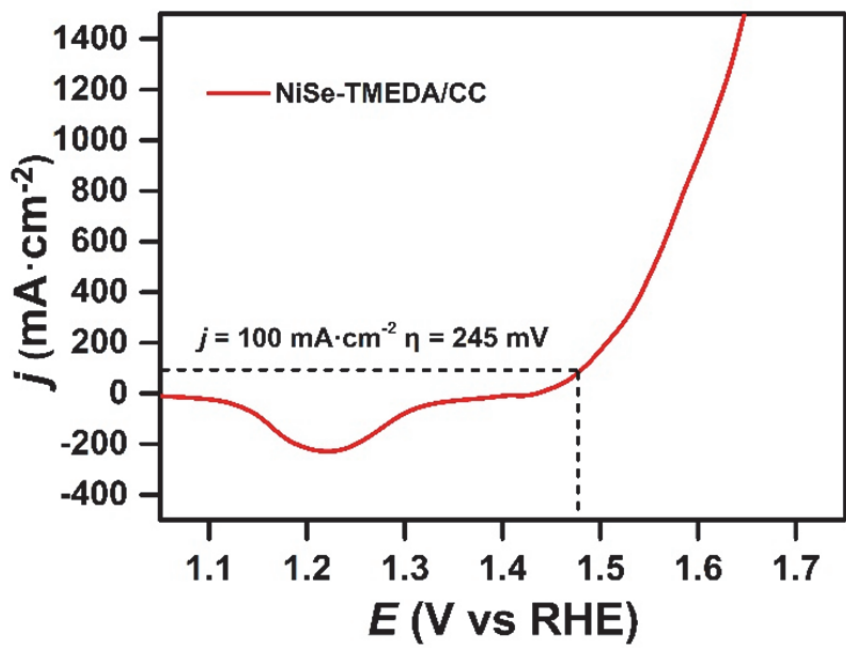
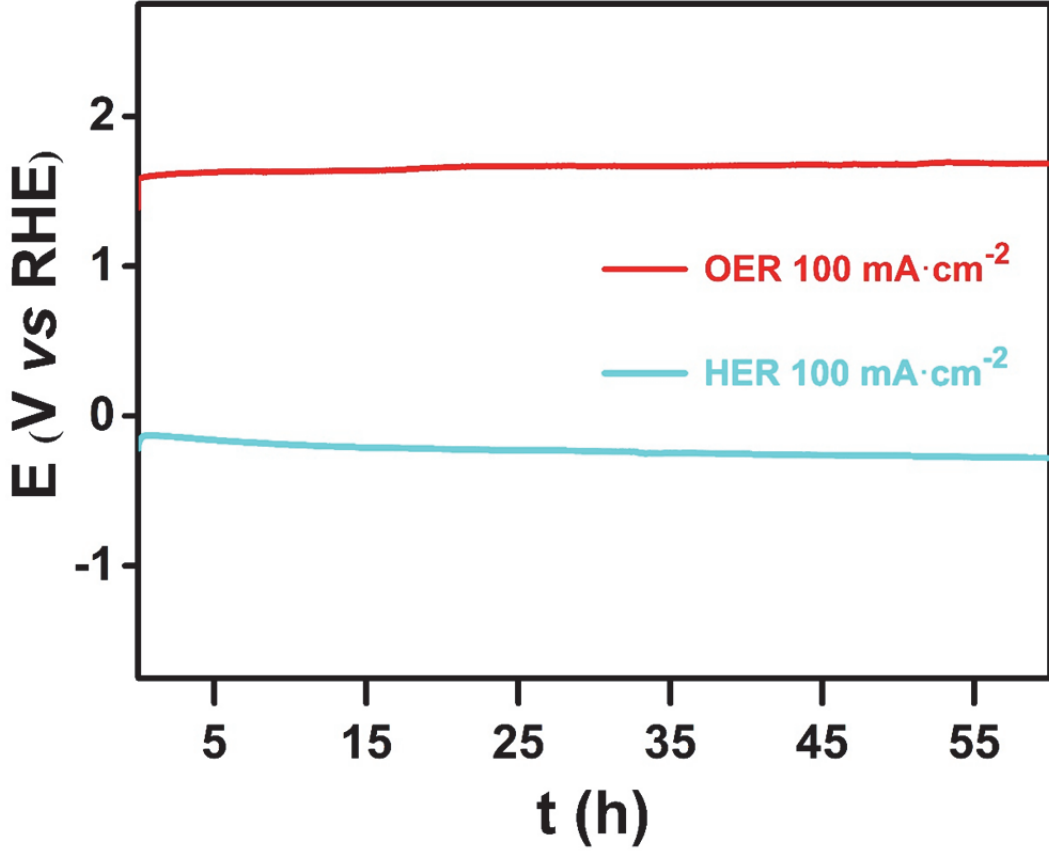


Figure S8. The cathodic sweep curve of OER from the NiSe-TMEDA/CC electrode.

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11 **Figure S9.** The Chronopotentiometry of the NiSe-TMEDA/CC electrode at 100 mA
12 cm^{-2} for both OER and HER without iR compensation.

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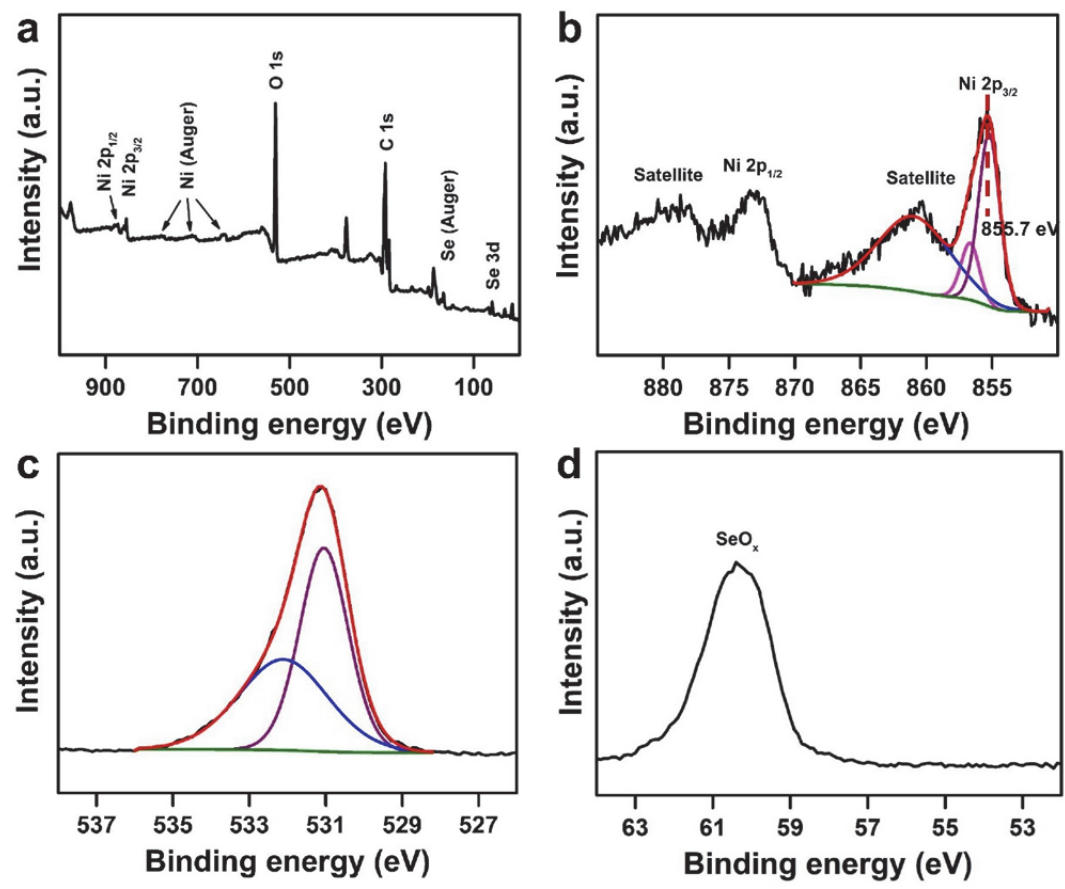


Figure S10. (a) XPS survey scan spectrum of the post-OER NiSe-TMEDA/CC electrode. High-resolution XPS spectra in the (b) Ni 2p, (c) O 1s and (d) Se 3d regions for the post-OER NiSe-TMEDA/CC.

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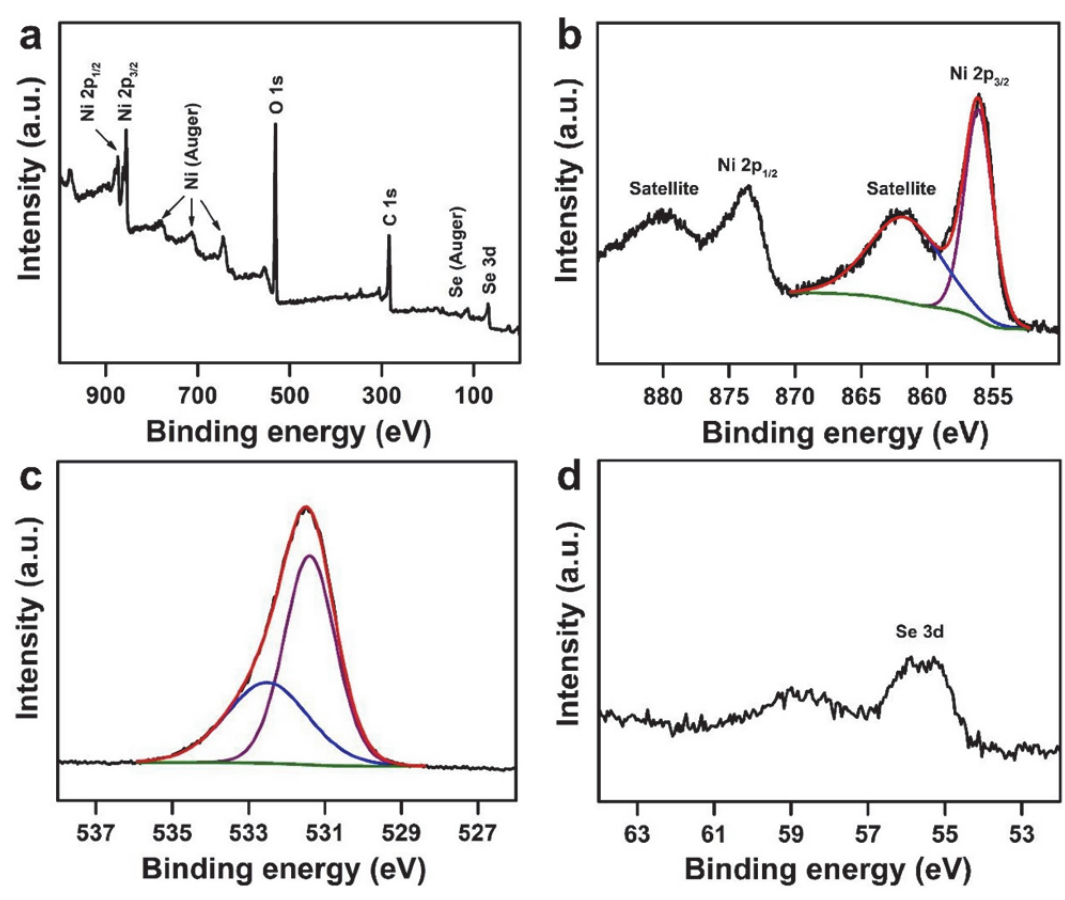


Figure S11. (a) XPS survey scan spectrum of the post-HER NiSe-TMEDA/CC electrode. High-resolution XPS spectra in the (b) Ni 2p, (c) O 1s and (d) Se 3d regions for the post-HER NiSe-TMEDA/CC.

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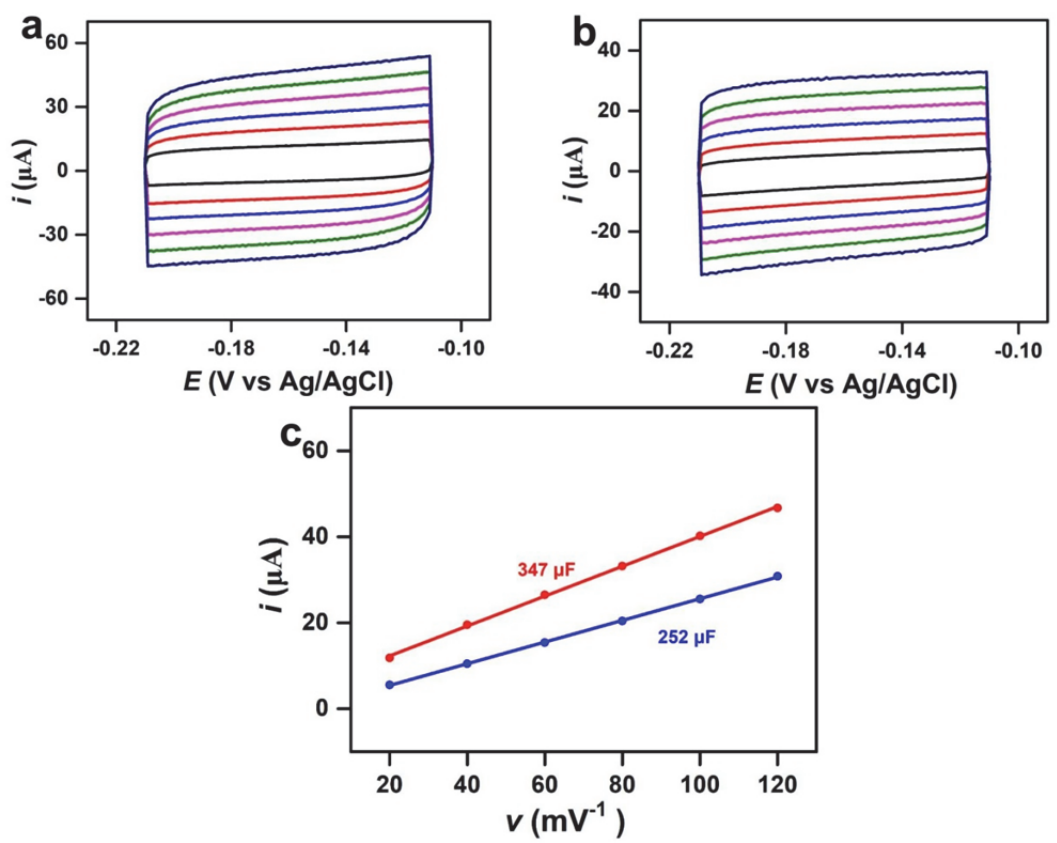
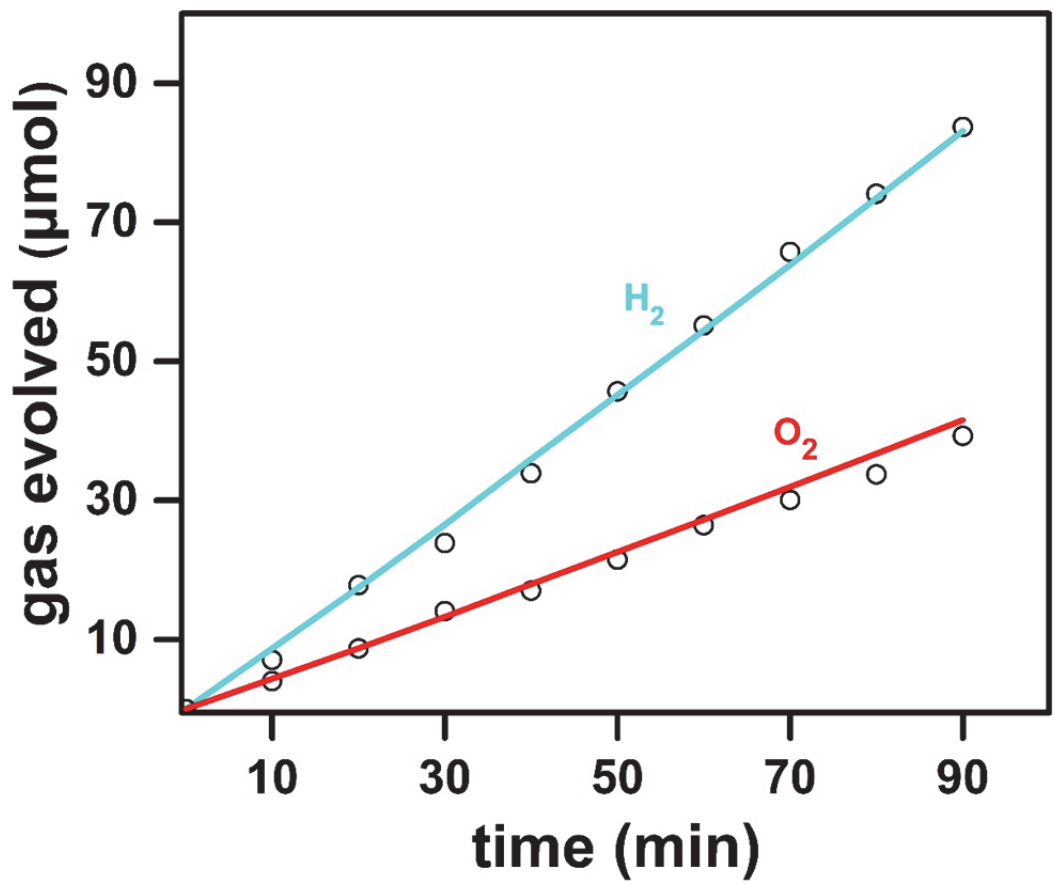


Figure S12. Charging currents of (a) the NiSe-TMEDA/CC and (b) NiSe/CC electrodes recorded in the non-Faradaic potential region at different scan rates from 20 to 120 mV s^{-1} . (c) The anodic charging currents at -0.16 V plotted against the scan rates for the NiSe-TMEDA/CC (red line) and NiSe/CC (blue line).

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11 **Figure S13.** The experimental (open dots) and theoretical (solid lines) amounts of
12 evolved gasses during HER and OER.

1 **Table S1.** Comparison of the overall water splitting performance of the as-prepared
 2 NiSe-TMEDA/CC electrode with literature reports of bifunctional water splitting
 3 electrodes.

| Electrodes | Electro-lyte | Water electrolsis | Ref. |
|--|----------------|--------------------------------------|---|
| NiSe-TMEDA/CC | 1 M KOH | 1.52 V@100 mA cm⁻² | This work |
| NiSe-Ni _{0.85} Se/CP | 1 M KOH | 1.89 V@100 mA cm ⁻² | <i>Small</i> , 2018 , <i>14</i> , 1800763 |
| NiSe ₂ /CFP | 1 M KOH | 1.66 V@10 mA cm ⁻² | <i>Electrochim. Acta</i> , 2019 , 305 37 |
| NiSe ₂ /CC | 1 M KOH | 1.62 V@10 mA cm ⁻² | <i>Electrochim. Acta</i> , 2018 , 279 195 |
| NiSe ₂ nanowires | 1 M KOH | 1.55 V@10 mA cm ⁻² | <i>Small</i> , 2017 , <i>13</i> , 1701487 |
| Se-MnS/NiS | 1 M KOH | 1.47 V@10 mA cm ⁻² | <i>J. Mater. Chem. A</i> , 2019 , <i>7</i> , 26975 |
| Ni ₃ Se ₂ /NiSe | 1 M KOH | 1.61 V@10 mA cm ⁻² | <i>ChemSusChem</i> , 2019 , <i>12</i> , 2008 |
| NiSe/Ni/NC | 1 M KOH | 1.60 V@10 mA cm ⁻² | <i>Electrochim. Acta</i> , 2019 , 300, 93 |
| Fe _{0.09} Co _{0.13} -NiSe ₂ | 1 M KOH | 1.52 V@10 mA cm ⁻² | <i>Adv. Mater.</i> , 2018 , <i>30</i> , 1802121 |
| NiFeMo/NF | 1 M KOH | 1.45 V@10 mA cm ⁻² | <i>ACS Energy Lett.</i> , 2018 , <i>3</i> , 546 |
| Ni-Fe NP | 1 M KOH | 1.47 V@10 mA cm ⁻² | <i>Nature Commun.</i> , 2019 , <i>10</i> , 5599 |
| CoFeZr oxides/NF | 1 M KOH | 1.63 V@10 mA cm ⁻² | <i>Adv. Mater.</i> , 2019 , <i>31</i> , 1901439 |

4 CC: carbon cloth; CP: carbon paper; CFP: carbon fiber paper; NF: nickel foam.