

## Supplementary Materials

### Conjugated polycarboxylate ligands-coordinated NiFe LDH for enhanced oxygen evolution

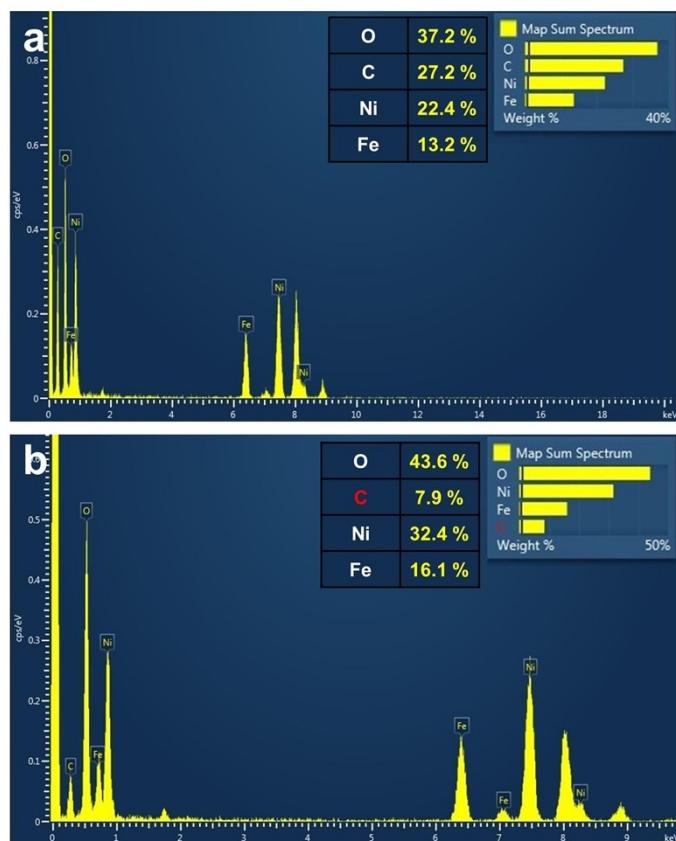
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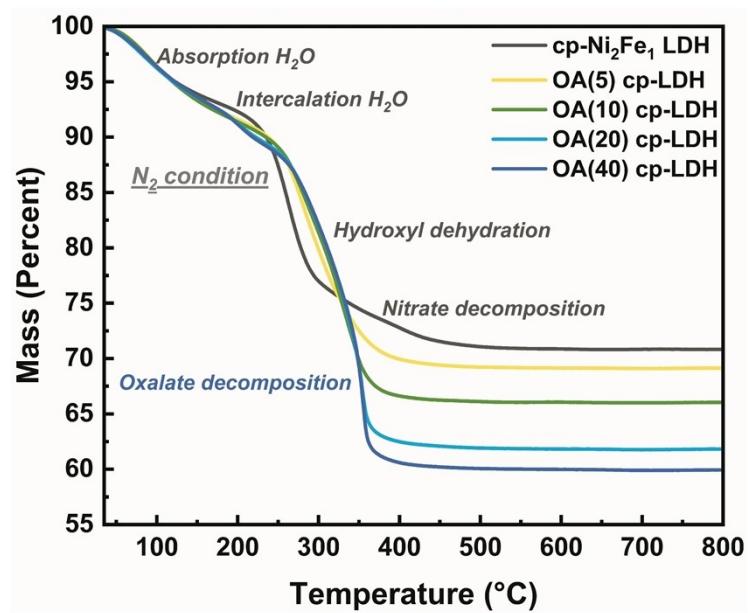
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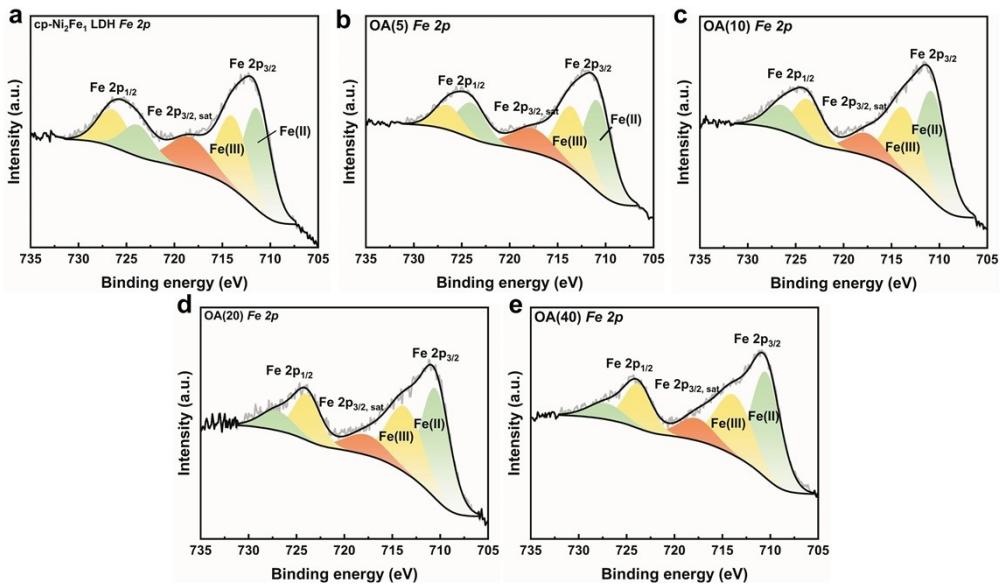
## Supplementary data



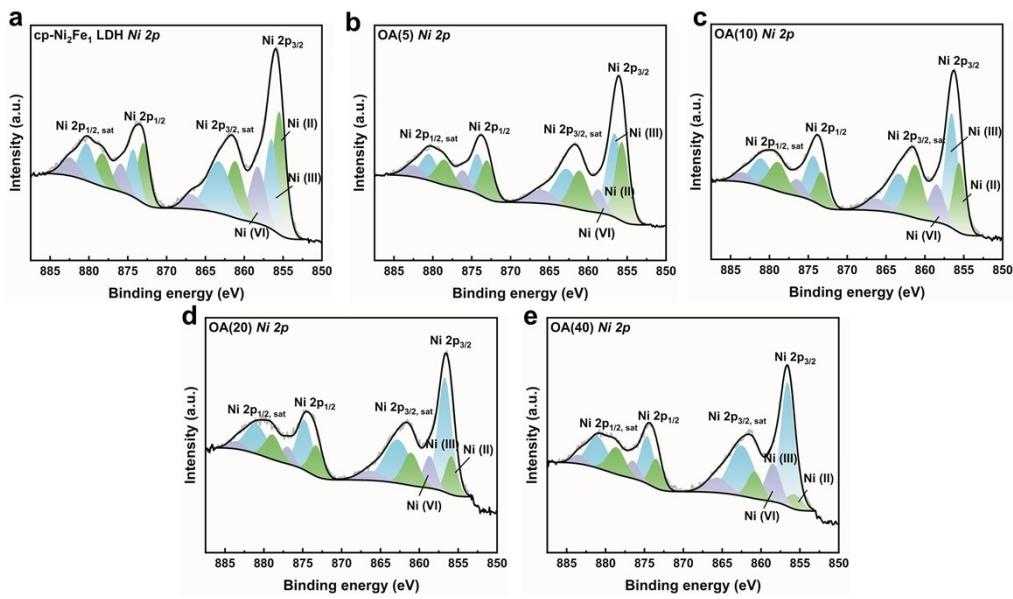
**Figure S1.** EDS results of (a) cp-OA(40) LDH and (b) cp-Ni<sub>2</sub>Fe<sub>1</sub> LDH.



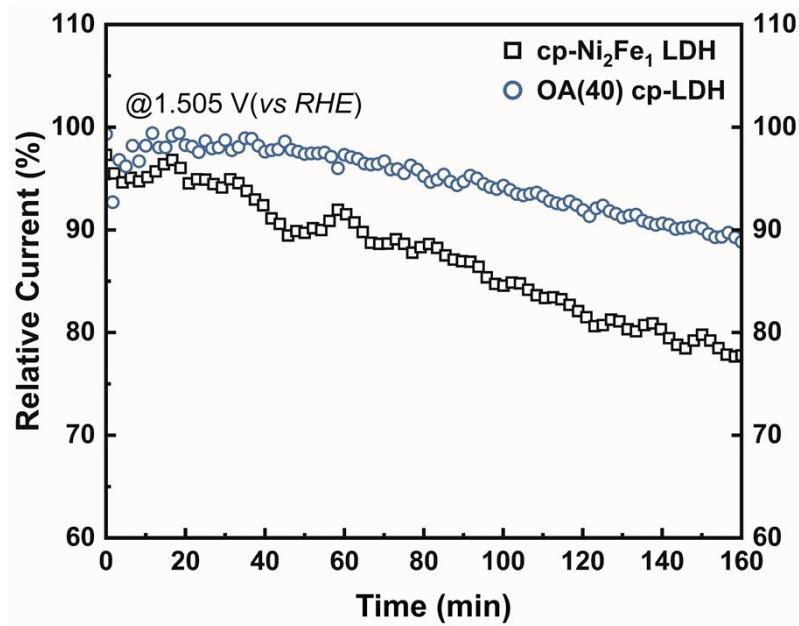
**Figure S2.** Thermogravimetry analysis of cp-Ni<sub>2</sub>Fe<sub>1</sub> LDH and cp-OA(n) NiFe LDH(n= 5, 10, 20, 40)  
in N<sub>2</sub> condition



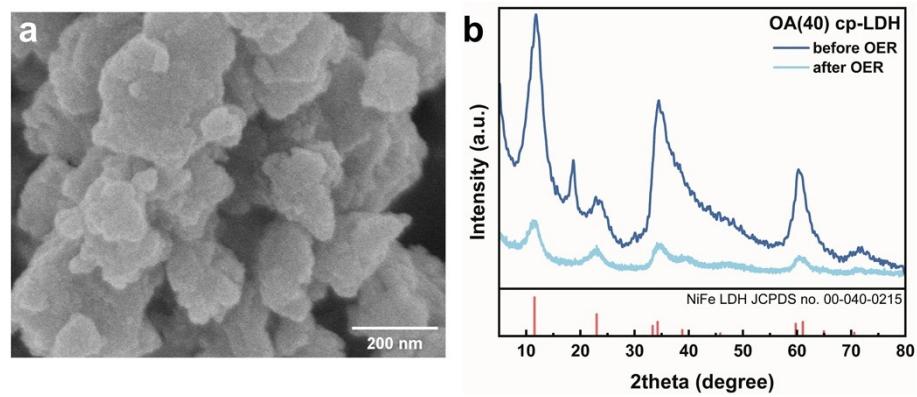
**Figure S3.** Fe 2p XPS peak fitting of cp-Ni<sub>2</sub>Fe<sub>1</sub> LDH and cp-OA(n) NiFe LDH(n= 5, 10, 20, 40).



**Figure S4.** Ni 2p XPS peak fitting of cp-Ni<sub>2</sub>Fe<sub>1</sub> LDH and cp-OA(n) NiFe LDH(n= 5, 10, 20, 40).



**Figure S5.** The chronoamperometry tests of cp-Ni<sub>2</sub>Fe<sub>1</sub> LDH and OA(40) cp-LDH at 1.505 V vs RHE.

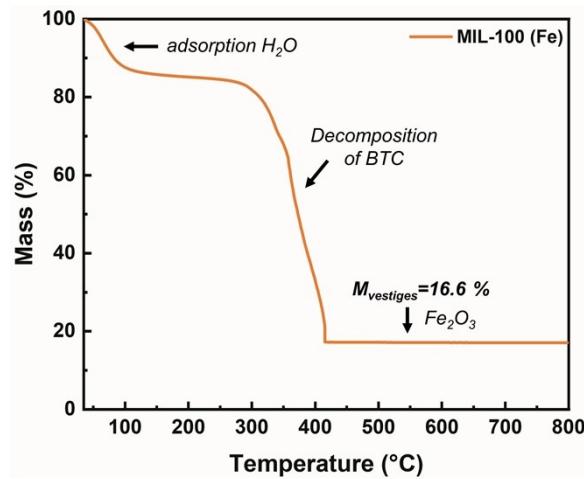


**Figure S6.** (a) SEM image of cp-OA(40) LDH and (b) XRD pattern of cp-OA(40) LDH before and after OER.

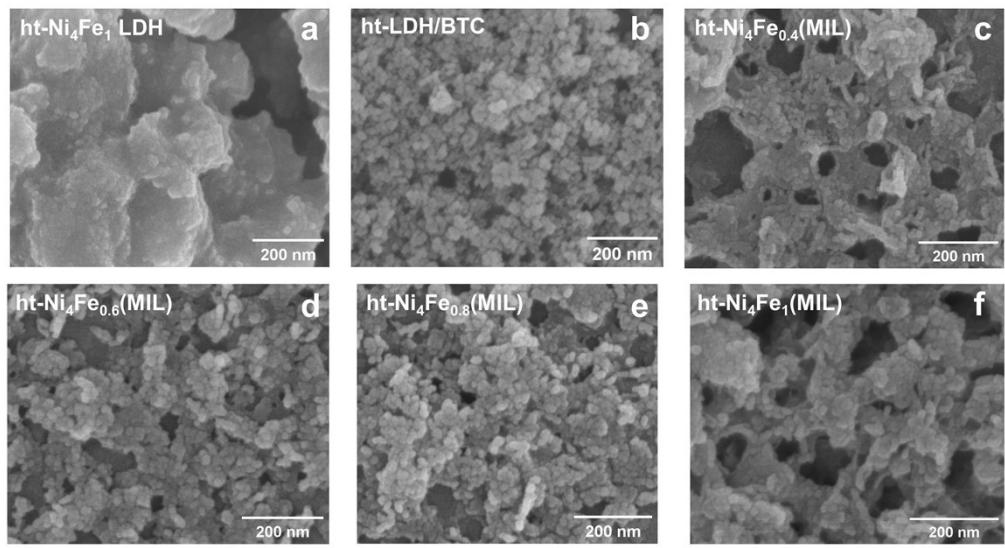
Under the oxygen atmosphere, the BTC of MIL-100 is completely burned to CO<sub>2</sub> and H<sub>2</sub>O, and the residual component is Fe<sub>2</sub>O<sub>3</sub>, according to the proportion calculation:

$$m_{(Fe\%wt)} = m_{vestiges} \times \frac{112}{156},$$

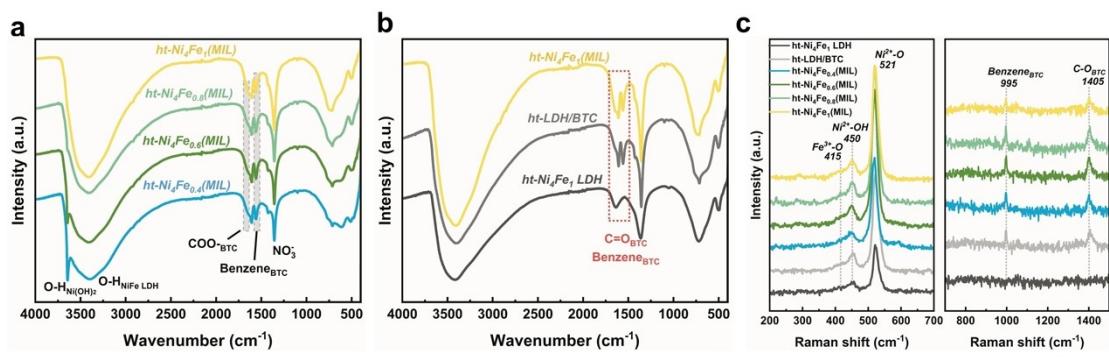
the content of Fe in each portion of MIL-100(Fe) is 11.6%, and BTC is 69.3%. So, 1 g of MIL-100(Fe) powder contains 2.07 mmol of Fe.



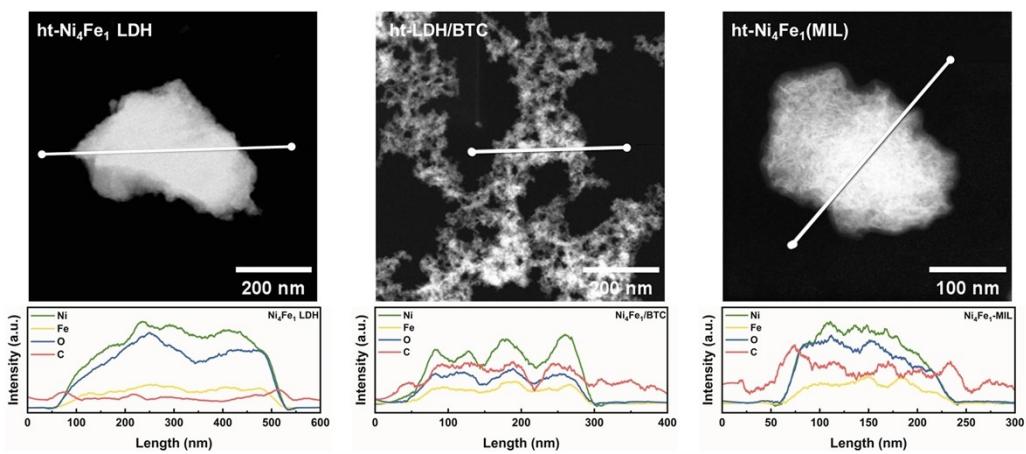
**Figure S7.** Thermogravimetry analysis of MIL-100(Fe)



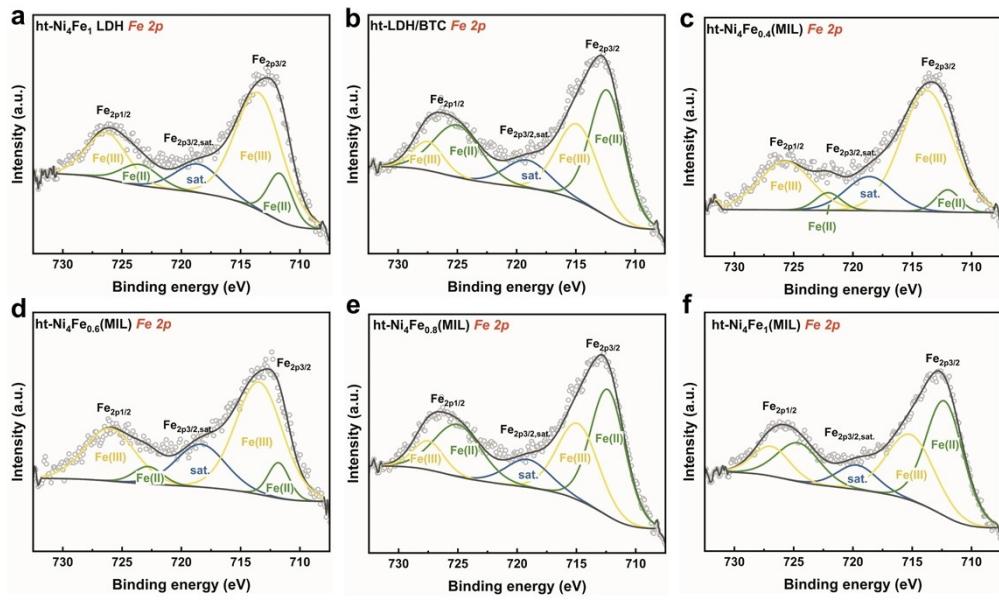
**Figure S8.** SEM images of (a)ht-Ni<sub>4</sub>Fe<sub>1</sub> LDH, (b) ht-LDH/BTC, (c) ht-Ni<sub>4</sub>Fe<sub>0.4</sub>(MIL) (d) ht-Ni<sub>4</sub>Fe<sub>0.6</sub>(MIL), (e) ht-Ni<sub>4</sub>Fe<sub>0.8</sub>(MIL) and (e) ht-Ni<sub>4</sub>Fe<sub>1</sub>(MIL).



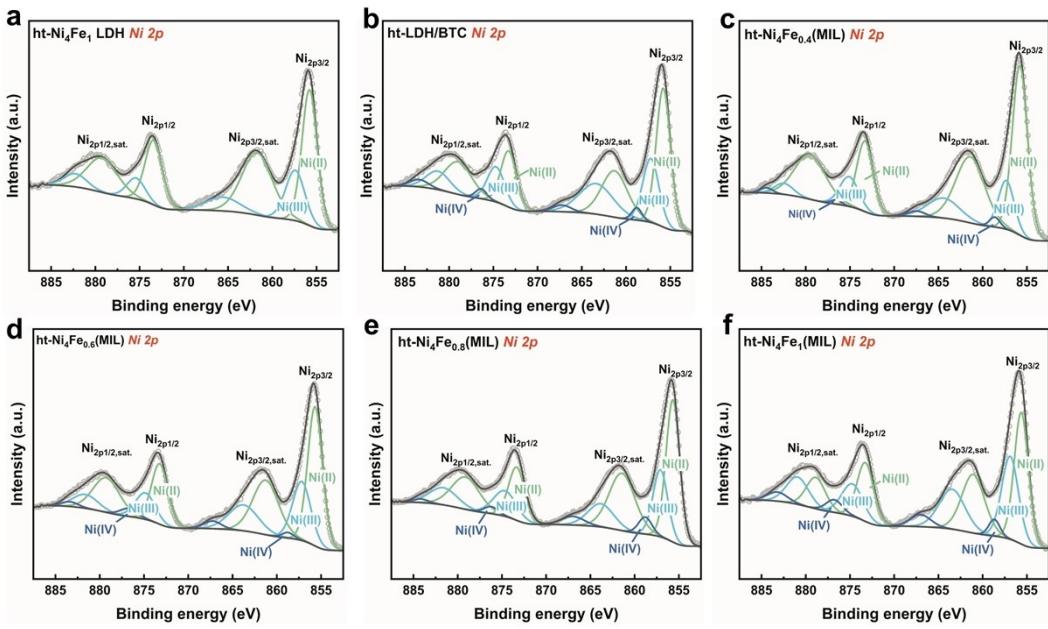
**Figure S9.** (a),(b) FTIR and (c) Raman spectra of *ht-Ni<sub>4</sub>Fe<sub>n</sub>(MIL)* ( $n = 0.4, 0.6, 0.8, 1$ ), *ht-Ni<sub>4</sub>Fe<sub>1</sub>* LDH and *ht-LDH/BTC*.



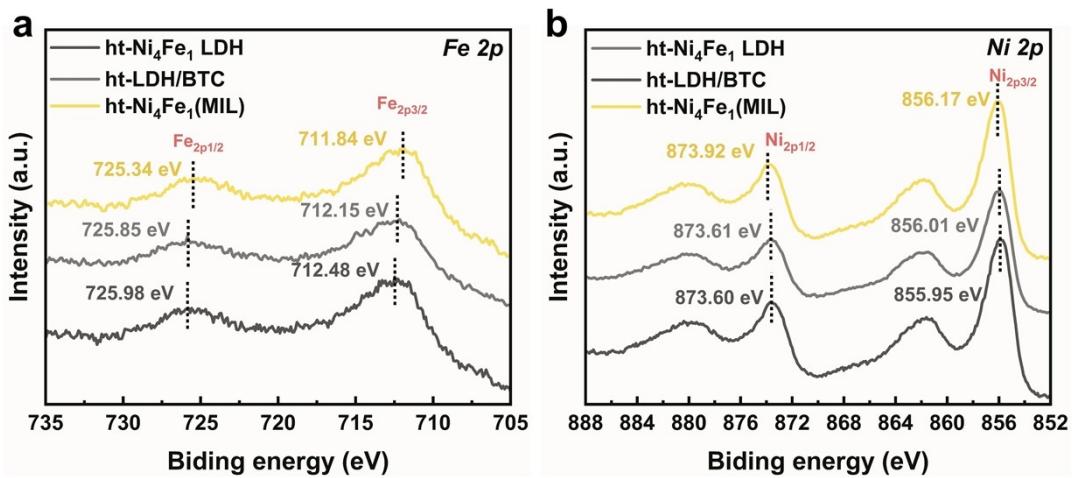
**Figure S10.** HRTEM EDS line-scanning ht-Ni<sub>4</sub>Fe<sub>1</sub>(MIL), ht-Ni<sub>4</sub>Fe<sub>1</sub> LDH and ht-LDH/BTC.



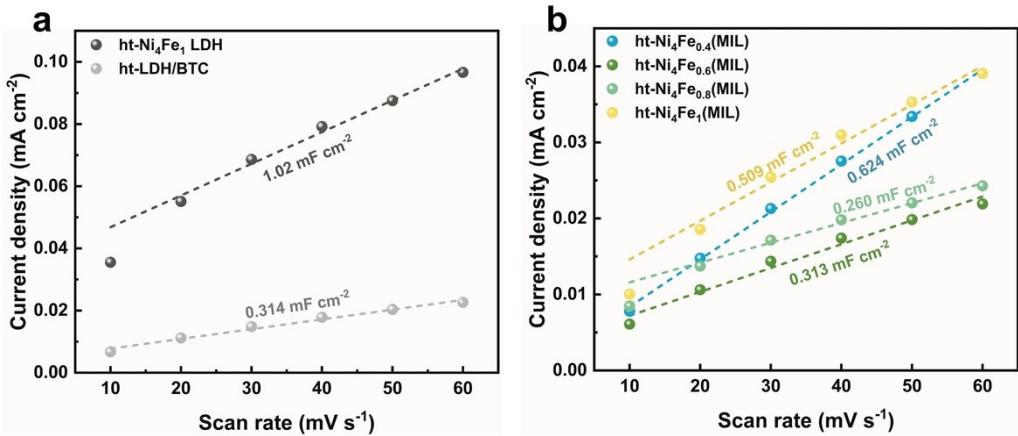
**Figure S11.** Fe 2p XPS peak fitting of ht-Ni<sub>4</sub>Fe<sub>n</sub>(MIL) ( $n=0.4, 0.6, 0.8, 1$ ), ht-Ni<sub>4</sub>Fe<sub>1</sub> LDH and ht-LDH/BTC.



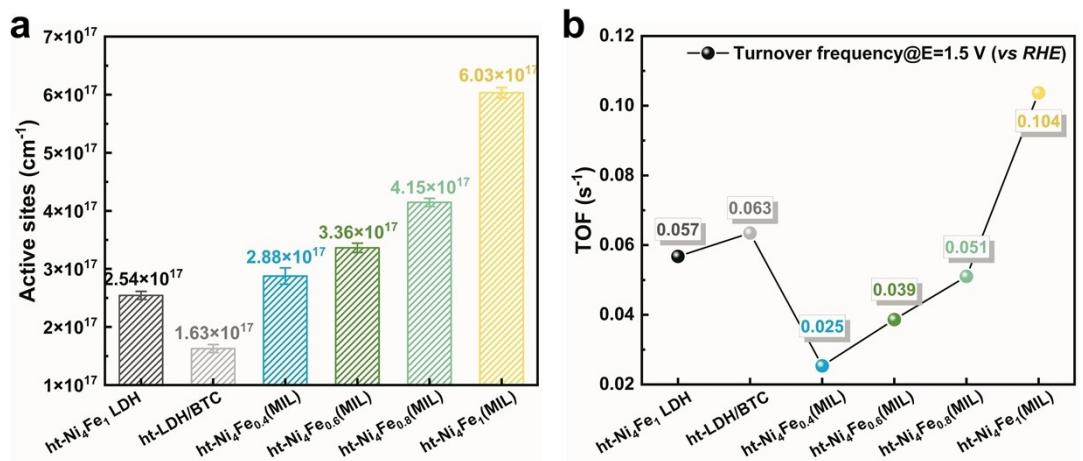
**Figure S12.** Ni 2p XPS peak fitting of ht- $\text{Ni}_4\text{Fe}_n(\text{MIL})$  ( $n = 0.4, 0.6, 0.8, 1$ ), ht- $\text{Ni}_4\text{Fe}_1$  LDH and ht-LDH/BTC.



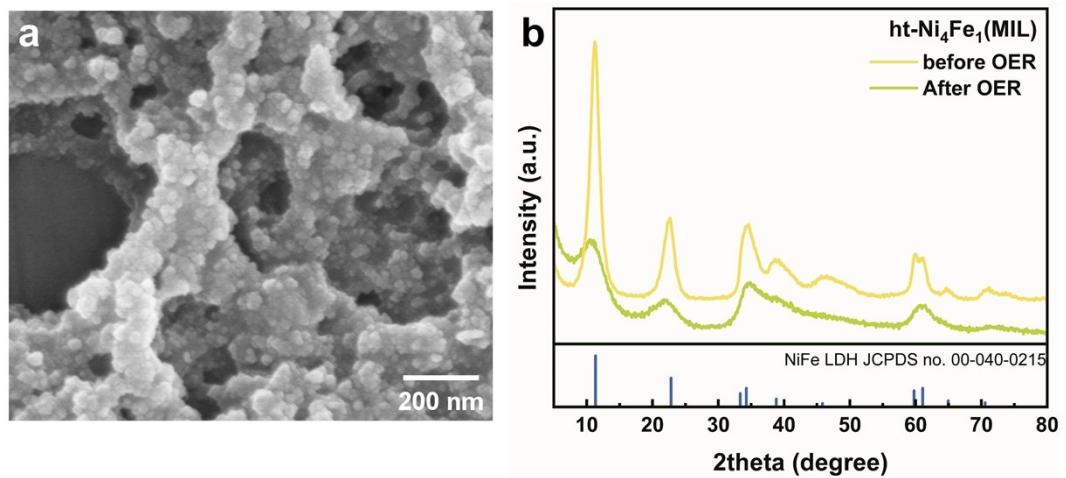
**Figure S13.** (a) Fe 2p and (d) Ni 2p fine XPS spectra ht-Ni<sub>4</sub>Fe<sub>1</sub>(MIL), ht-Ni<sub>4</sub>Fe<sub>1</sub> LDH and ht-LDH/BTC.



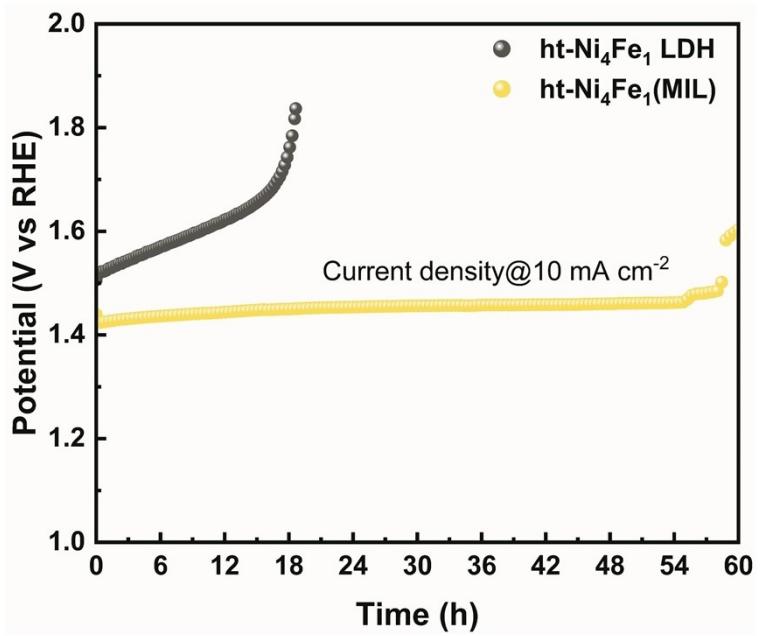
**Figure S14.** Double layer capacitances of (a) ht-Ni<sub>4</sub>Fe<sub>1</sub> LDH, ht-LDH/BTC and (b) ht-Ni<sub>4</sub>Fe<sub>n</sub>(MIL) ( $n=0.4, 0.6, 0.8, 1$ ) estimated by CV at various scan rates.



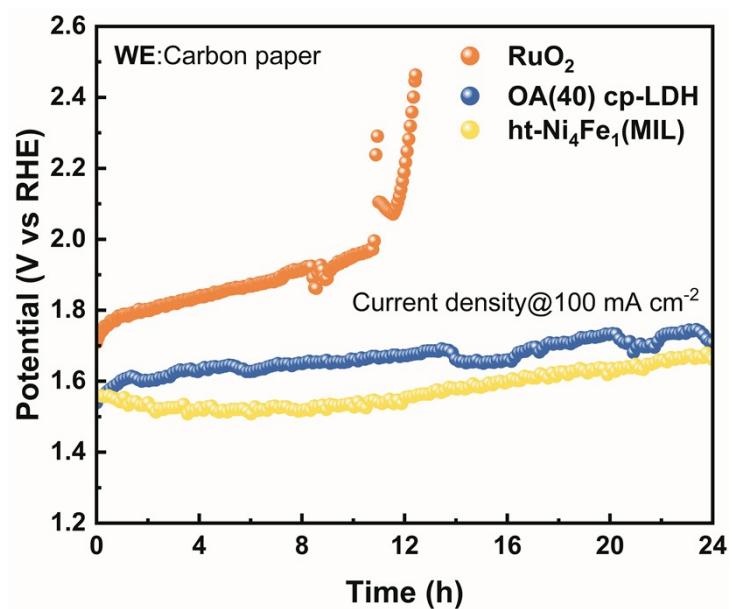
**Figure S15.** (a) Actives sites of prepared catalyst electrodes and (b) Turnover frequency at 1.5 V (vs RHE).



**Figure S16.** (a) SEM image of ht-Ni<sub>4</sub>Fe<sub>1</sub>(MIL) and (b) XRD pattern of ht-Ni<sub>4</sub>Fe<sub>1</sub>(MIL) before and after OER.



**Figure S17.** Chronopotentiometry of ht-Ni<sub>4</sub>Fe<sub>1</sub> LDH and ht-Ni<sub>4</sub>Fe<sub>1</sub>(MIL).



**Figure S18.** Chronopotentiometry at 100 mA cm<sup>-2</sup> of RuO<sub>2</sub>, cp-OA(40) LDH and ht-Ni<sub>4</sub>Fe<sub>1</sub>(MIL).