Supporting Information

Ultra-fast preparation of Ni-Fe nanoclusters/graphene heterojunction catalyst for oxygen evolution reaction and urea oxidation reaction

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1. Supplementary Figures



Fig. S1. The optical photograph of NiFe NCs/GO.



Fig. S2. The TEM image of pure GO.



Fig. S3. (a) TEM of NiFe NCs/GO, (b) the grain diameter distribution map of NiFe NCs/GO obtained from (a).



Fig. S5. (a) Nitrogen adsorption-desorption isotherms and (b) Pore sizes distribution

of NiFe NCs/GO and NiFe-LDH.



Fig. S6. Raman spectra of GO and NiFe NCs/GO.



Fig. S7. The survey spectrum of NiFe NCs/GO. The only found of Ni, Fe, C and O elements.



Fig. S8. LSV curves of NiFe NCs/GO-1:2, NiFe NCs/GO, NiFe NCs/GO-2:1, NiFe

NCs/GO-3:1 and NiFe NCs/GO-4:1.



Fig. S9. LSV curves of NiFe NCs/GO/NF, NiFe-LDH/EC-600JD/NF and NiFe-LDH/XC-72/NF.



Fig. S10. TEM images of NiFe-LDH/EC-600JD and NiFe-LDH/XC-72.



Fig. S11. Cyclic voltammograms of (a) NiFe NCs/GO/NF, (b) RuO₂/NF, (c)GO/NF at different scan rates in 1 M KOH. (d) The LSV polarization curve normalized by the Electrochemical surface area.



Fig. S12. The TEM image of NiFe NCs/GO (a) before and (b) after the 12h i-t test.

Catalysts	Electrolyt e	η ₁₀ , OER	Tafel (mV·dec⁻¹)	Source
NiFeCo-LDH/CF	1 М КОН	249	42	[1]
Au@Ni(Fe)OOH	1 М КОН	258	56.78	[2]
NF-Na-Fe-P	1 М КОН	261	39.68	[3]
H-CoSx @NiFe LDH/NF	1 М КОН	250	49	[4]
Co@NiFe-LDH	1 М КОН	253	44	[5]
CoO-Co4N@NiFe-LDH/NF	1 М КОН	231	39	[6]
NiFe-MOF/G	1 М КОН	258	49	[7]
NiFe-LDH/NF-3.5	1 М КОН	249	49.8	[8]
NiFe-25	1 М КОН	299	48.7	[9]
Fe ²⁺ -NiFe-LDHs	1 М КОН	249	40	[10]
NF-LDH-21	1 М КОН	242	25.9	[11]
Ni _{0.5} Fe _{0.5} /C	1 М КОН	270	40.4	[12]
(Ni ₇ Fe ₃)OOH-S	1 М КОН	238	42.7	[13]
NiFe NCs/GO	1 M KOH	231	41	This work

Table S1. Comparison of the OER performance of NiFe NCs/GO/NF with the most recently reported OER catalysts at 10 mA·cm⁻² in 1 M KOH.

References

- [1] Lin Y P, Wang H, Peng C K, Bu L M. Co-induced electronic optimization of hierarchical NiFe LDH for oxygen evolution. *Small*, **2020**, 16, 2002426.
- [2] Liu X, Jing S, Ban C. Dynamic active sites in NiFe oxyhydroxide upon Au nanoparticles decoration for highly efficient electrochemical water oxidation. *Nano Energy*, 2022, 98: 107328.
- [3] Zhao Y, Gao Y, Chen Z. Trifle Pt coupled with NiFe hydroxide synthesized via corrosion engineering to boost the cleavage of water molecule for alkaline water-splitting. *Applied Catalysis B: Environmental*, **2021**, 297: 120395.
- [4] Lee Y J, Park S K. Metal–Organic Framework-Derived Hollow CoSx Nanoarray Coupled with NiFe Layered Double Hydroxides as Efficient Bifunctional Electrocatalyst for Overall Water Splitting. Small, 2022, 18(16): 2200586.
- [5] Liu S L, Wan R D, Lin Z S, Liu Z, Liu Y, Tian Y, Qin D D. Probing the Co role in promoting the OER and Zn–air battery performance of NiFe-LDH: a combined experimental and theoretical study. *J. Mater. Chem. A*, **2022**, 10, 5244 6.
- [6] Chen B, Humayun M, Li Y. Constructing Hierarchical Fluffy CoO-Co₄N@ NiFe-LDH Nanorod Arrays for Highly Effective Overall Water Splitting and Urea Electrolysis. ACS Sustainable Chem. Eng. 2021, 9, 42, 14180–14192.
- [7] Wang Y, Liu B, Shen X. Engineering the Activity and Stability of MOF-Nanocomposites for Efficient Water Oxidation. Advanced Energy Materials, **2021**, 11(16): 2003759.
- [8] Huang Y, Liu X, Li X. Interfacial oxidation using potassium ferrate to fabricate

self-supported hydrophilic NiFe-LDH nanoarrays for overall water splitting at high current density. *Sustainable Materials and Technologies*, **2022**, 34: e00508.

- [9] Rinawati M, Wang Y. X.; Chen K. Y. Designing a spontaneously deriving NiFe-LDH from bimetallic MOF-74 as an electrocatalyst for oxygen evolution reaction in alkaline solution. *Chemical Engineering Journal*, **2021**, 423, 130204.
- [10] Cai, Z.; Zhou, D. J.; Wang, M. Y.; Bark, S. M.; Wu, Y. S.; Wu, Z. S.; Tian, Y.; Xiong, X. Y.; Li, Y. P.; Liu, W. et al. Introducing Fe²⁺ into nickel-iron layered double hydroxide: local structure modulated water oxidation activity. *Angew. Chem. Int. Ed.* **2018**, 130, 9536-9540.
- [11]Kyoung Ryeol Park, Jaeeun Jeon, Heechae Choi, Junho Lee, Dong-Ha Lim, Nuri Oh, Hyuksu Han, Chisung Ahn, Baejung Kim, and Sungwook Mhin. NiFe Layered Double Hydroxide Electrocatalysts for an Efficient Oxygen Evolution Reaction. ACS Applied Energy Materials, 2022, 5 (7), 8592-8600.
- [12] Wang Z, Wang Y, Zhang N. Highly efficient oxygen evolution catalysis achieved by NiFe oxyhydroxide clusters anchored on carbon black. *Journal of Materials Chemistry A*, **2022**, 10(19): 10342-10349.
- [13]Liu W, Wang X, Wang F. Electrochemical hydroxidation of sulfide for preparing sulfur-doped NiFe (oxy) hydroxide towards efficient oxygen evolution reaction. *Chemical Engineering Journal*, **2023**, 454: 140030.