

Supporting Information

Enhanced electrocatalytic glucose oxidation assisted hydrogen production via interfacial synergistic effect of NiO/NiCo₂O₄ porous nanowire

Zongxi Zhang ^{a,b}, Jiancheng Zhao ^{a,b}, Mei Hong, ^{a,b} Shuai Chen ^{* a,b}, Yan Qiao ^{*a,b}

^a State Key Laboratory of Coal Conversion, Institute of Coal Chemistry, Chinese Academy of Sciences, 27 South Taoyuan Road, Taiyuan 030001, China

^b Center of Materials Science and Optoelectronics Engineering, University of Chinese Academy of Sciences, Beijing 100049, China

Corresponding Author E-mail: chenshuai@sxicc.ac.cn, qiaoy@sxicc.ac.cn

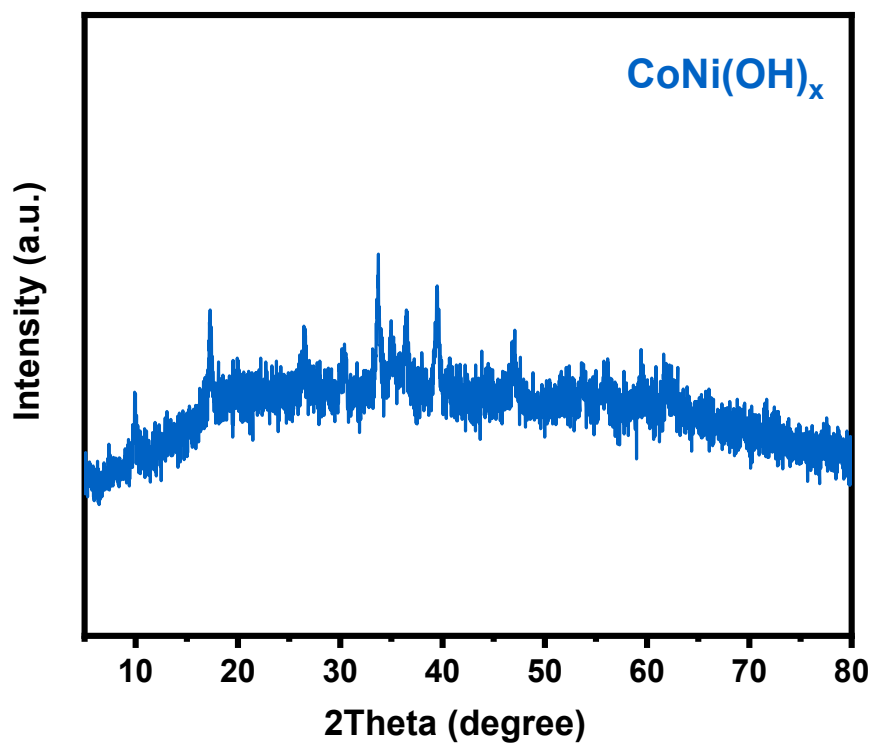


Fig. S1. XRD patterns of CoNi(OH)_x .

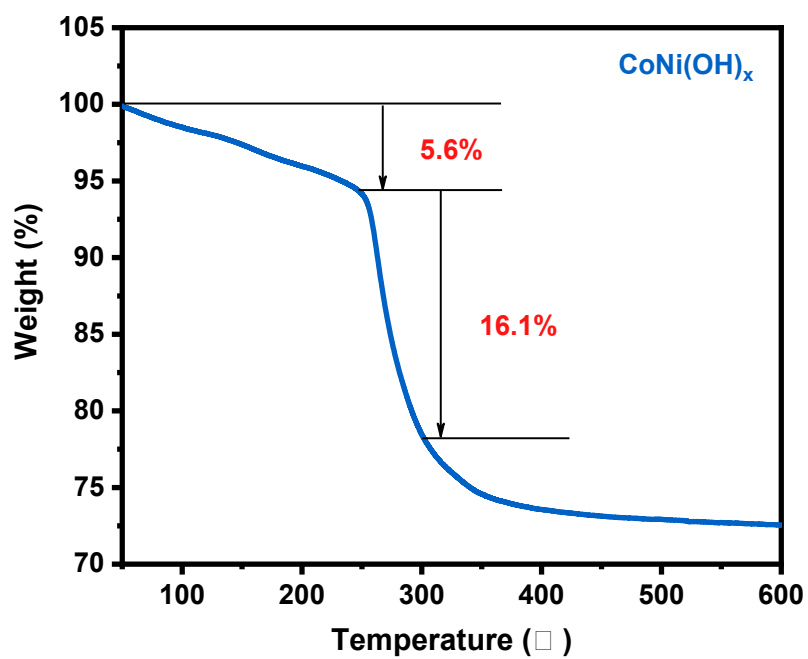


Fig. S2. TG analysis of the CoNi(OH)_x .

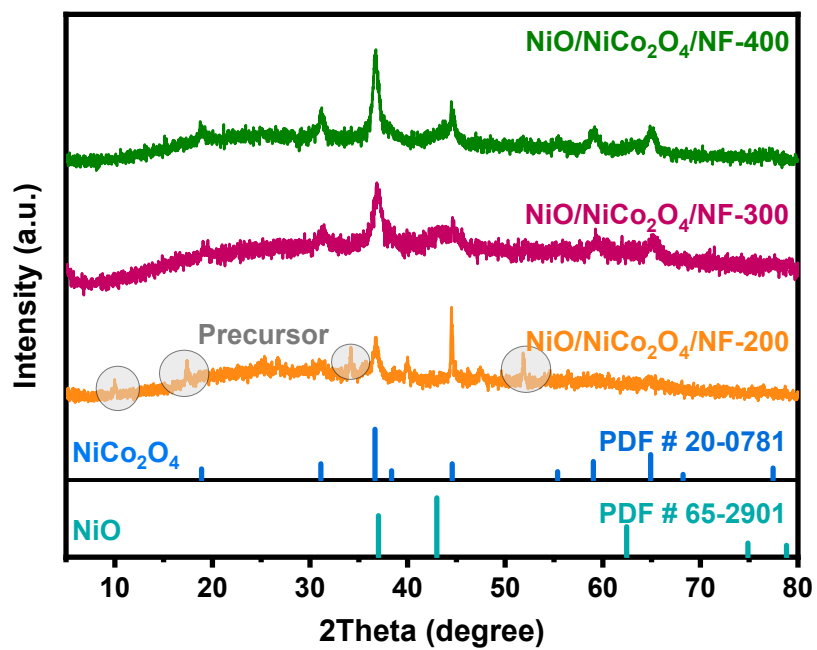


Fig. S3. XRD patterns of NiO/NiCo₂O₄/NF-X (X = 200, 300, 400).

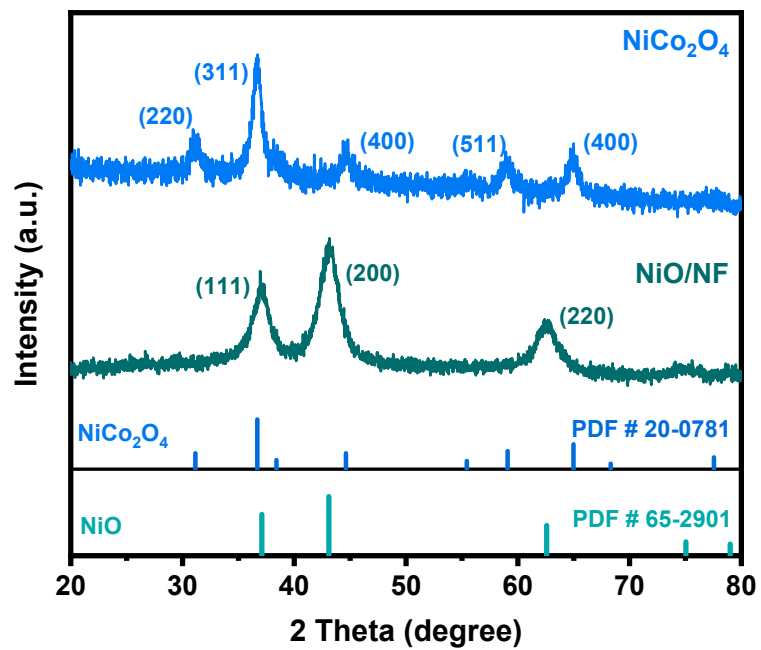


Fig. S4. XRD patterns of NiO/NF and NiCo₂O₄.

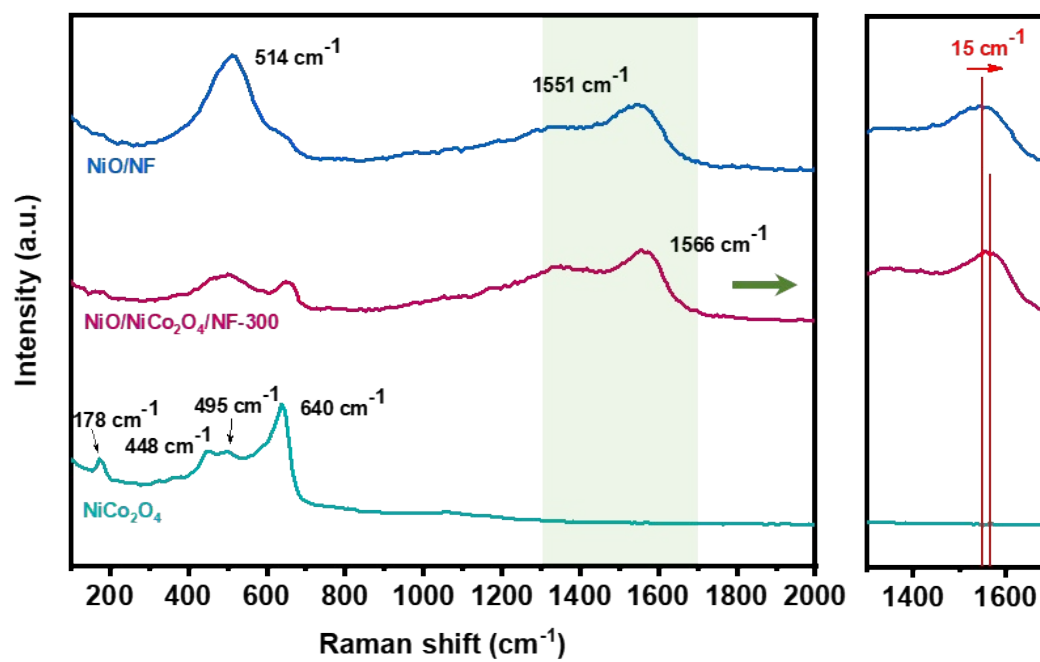


Fig. S5. Raman spectra of NiCo₂O₄, NiO/NiCo₂O₄/NF-300 and NiO.

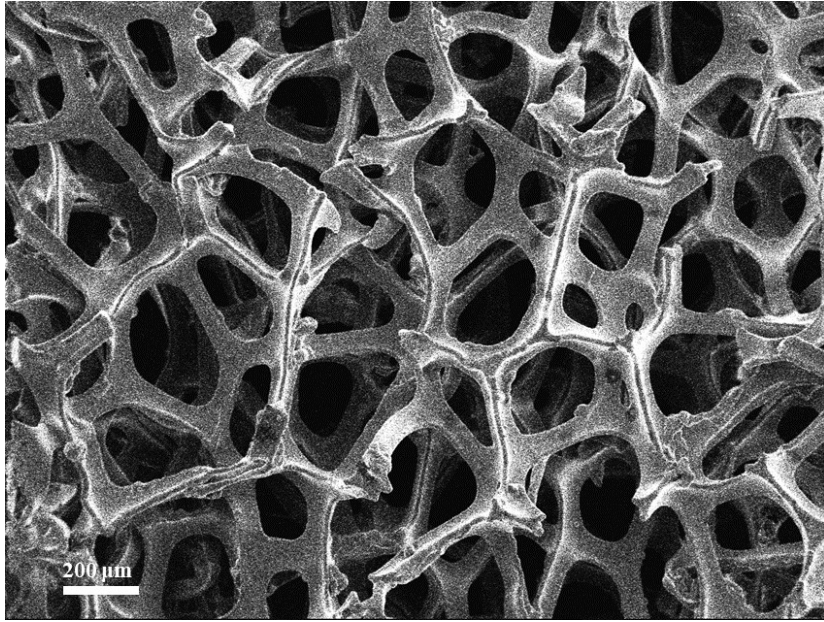


Fig. S6. SEM image of Ni foam.

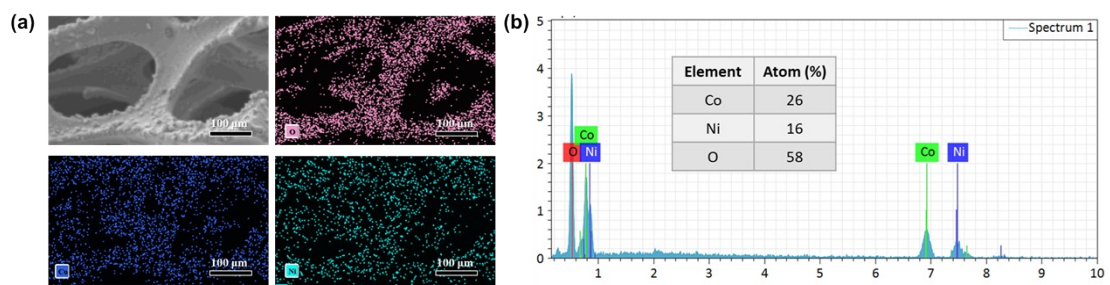


Fig. S7. (a) SEM image, corresponding elements mapping results and (b) the energy dispersive spectroscopy (EDS) of NiO/NiCo₂O₄/NF-300.

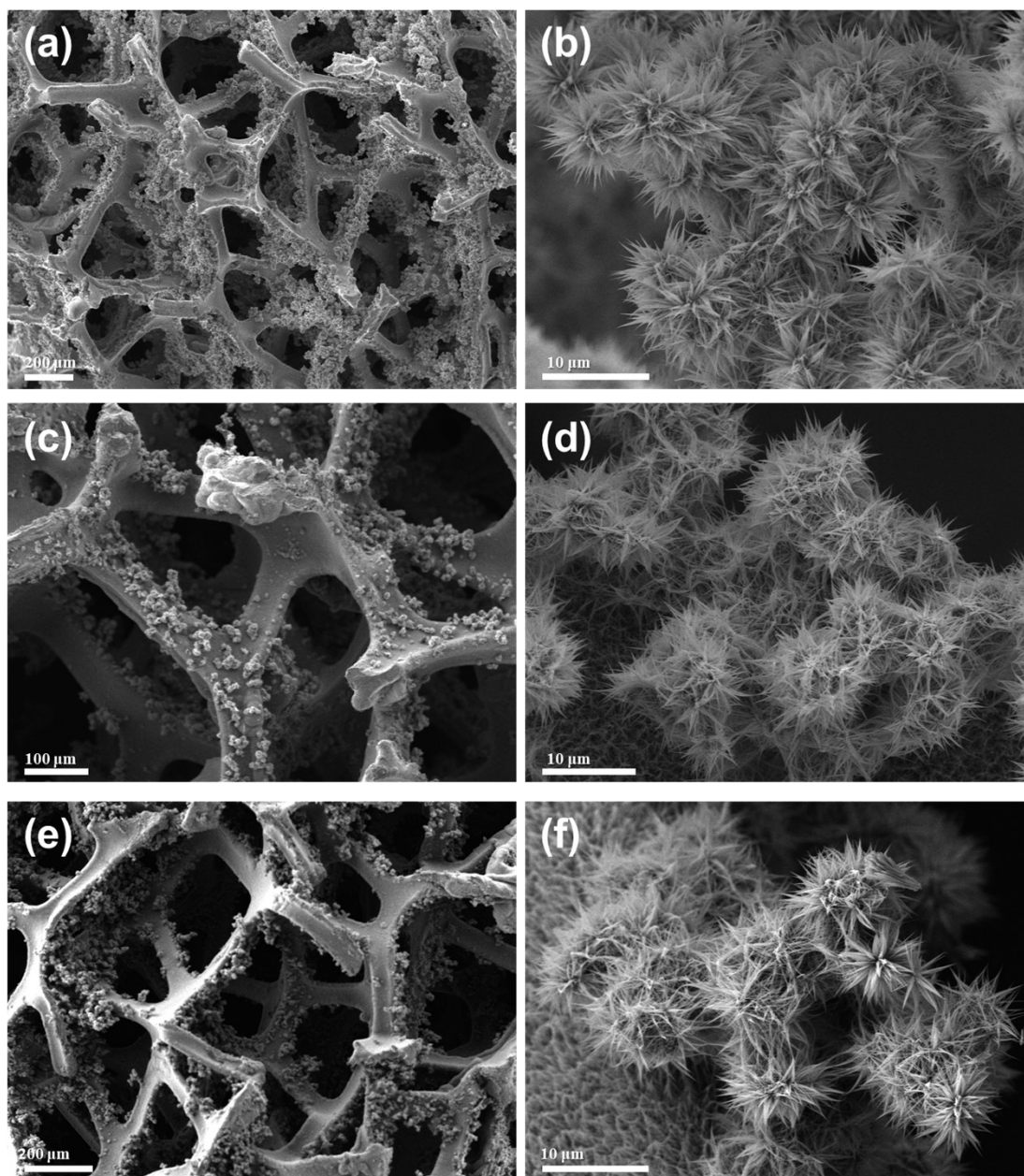


Fig. S8. SEM images of (a, b) $\text{CoNi(OH)}_x/\text{NF}$, (c, d) $\text{NiO/NiCo}_2\text{O}_4/\text{NF-200}$ and (e, f) $\text{NiO/NiCo}_2\text{O}_4/\text{NF-400}$.

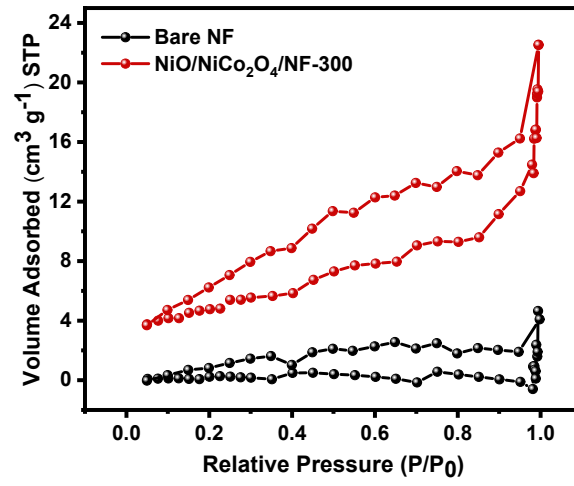


Fig. S9. N₂ adsorption-desorption isotherms of NiO/NiCo₂O₄/NF-300 and bare NF.

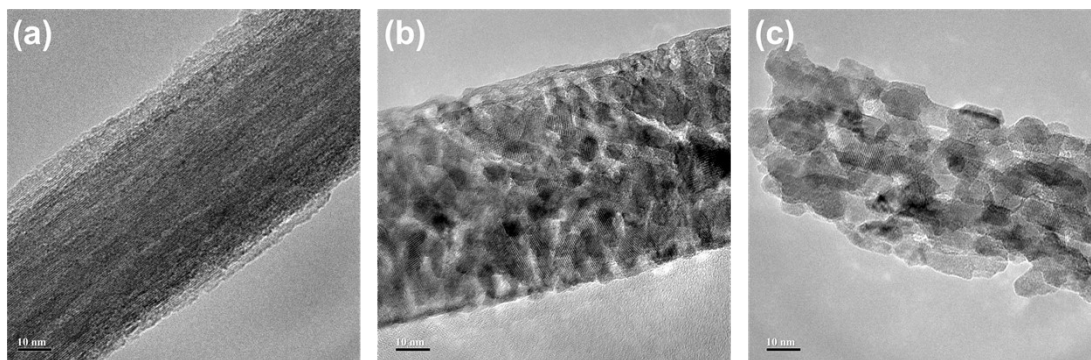


Fig. S10. TEM images of (a) NiO/NiCo₂O₄/NF-200, (b) NiO/NiCo₂O₄/NF-300 and (c) NiO/NiCo₂O₄/NF-400.

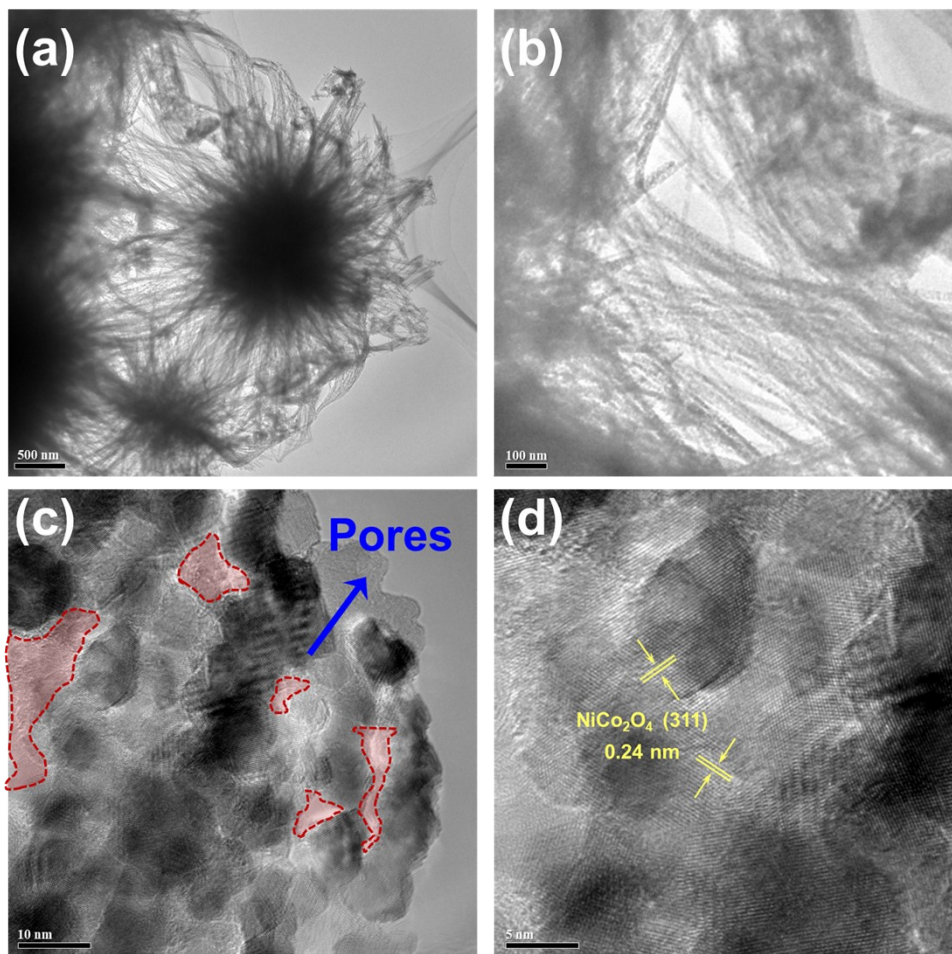


Fig. S11. TEM images of NiCo_2O_4 .

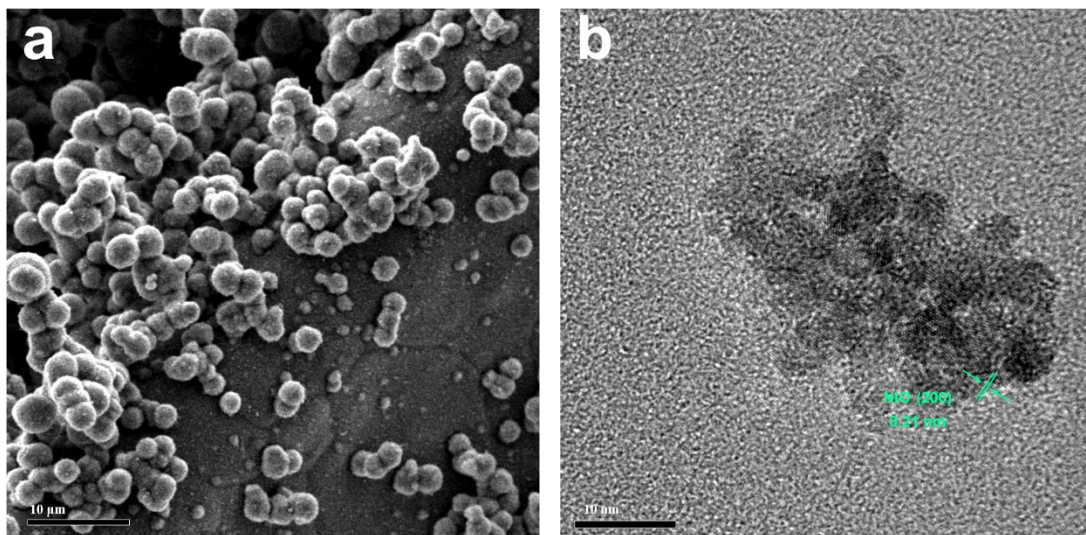


Fig. S12. (a) SEM image and (b) TEM image of NiO/NF.

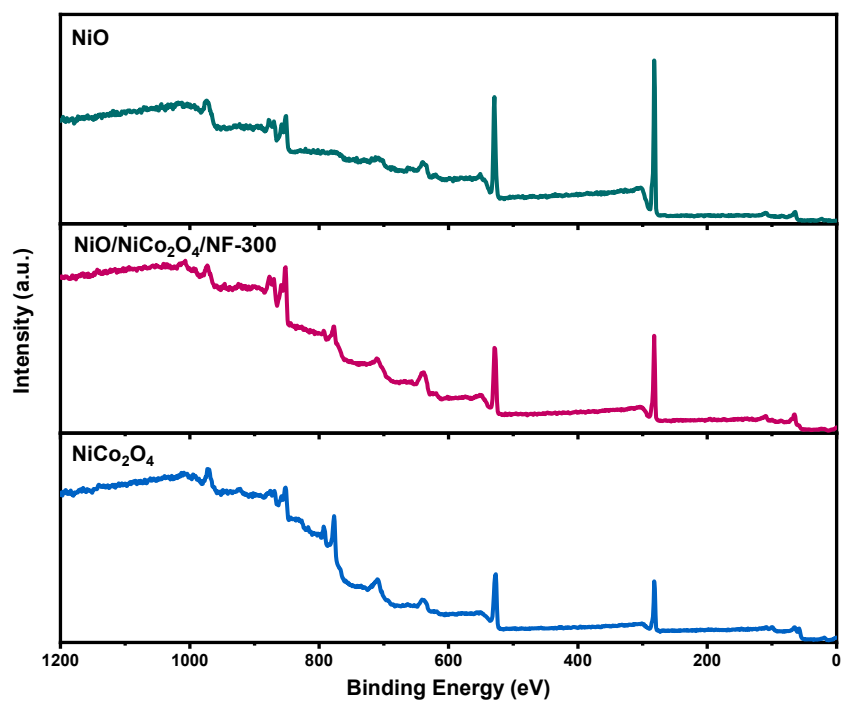


Fig. S13. XPS spectra of NiCo₂O₄, NiO/NiCo₂O₄/NF-300 and NiO.

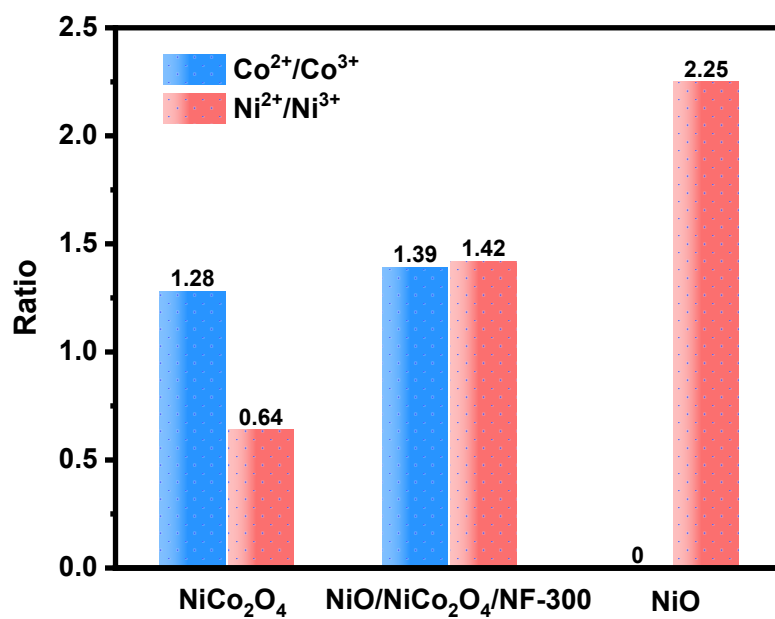


Fig. S14. The $\text{Co}^{2+}/\text{Co}^{3+}$ and $\text{Ni}^{2+}/\text{Ni}^{3+}$ ratios obtained from XPS of the corresponding samples.

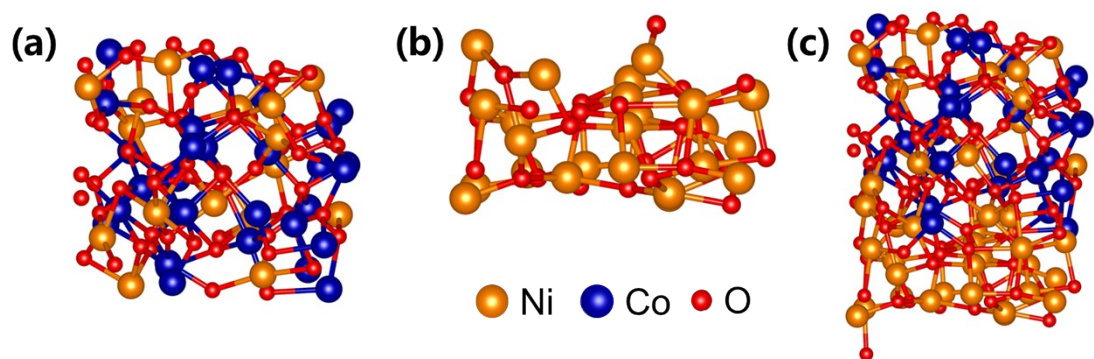


Fig. S15. The theoretical models of the optimized configurations of (a) NiCo_2O_4 , (b) NiO and (c) $\text{NiO/NiCo}_2\text{O}_4$ heterojunction.

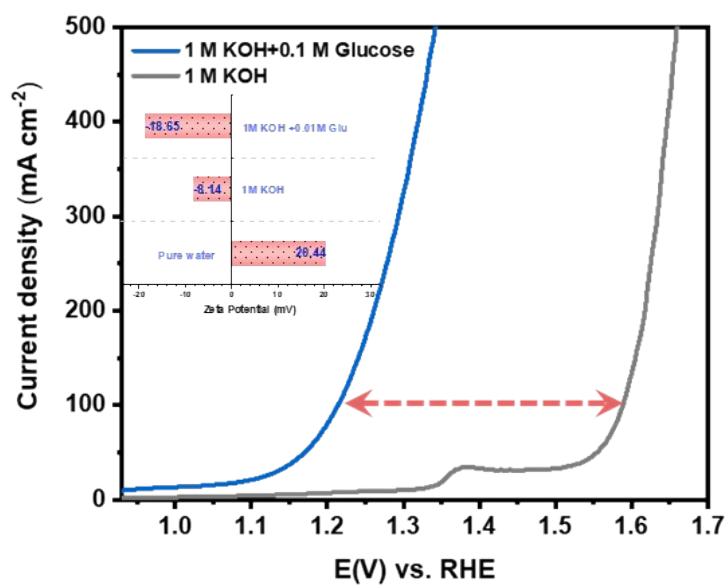


Fig. S16. the corresponding LSV curves of NiO/NiCo₂O₄/NF-300 with and without 0.1 M glucose in 1.0 M KOH (inset shows the corresponding zeta potential in different environments).

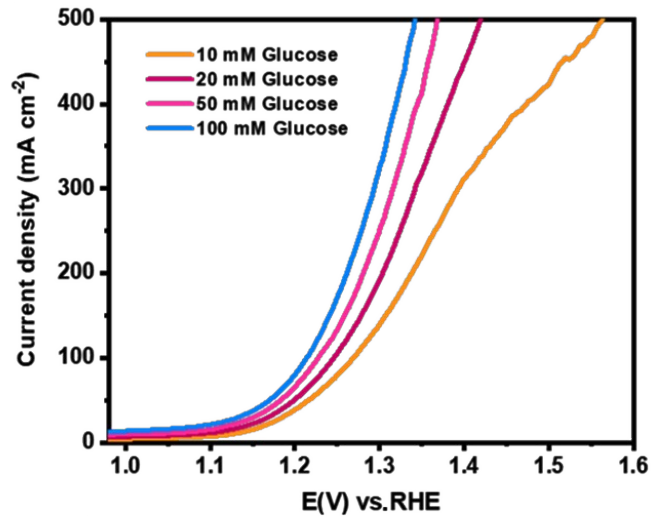


Fig. S17. LSV curves of NiO/NiCo₂O₄/NF-300 at different glucose concentrations.

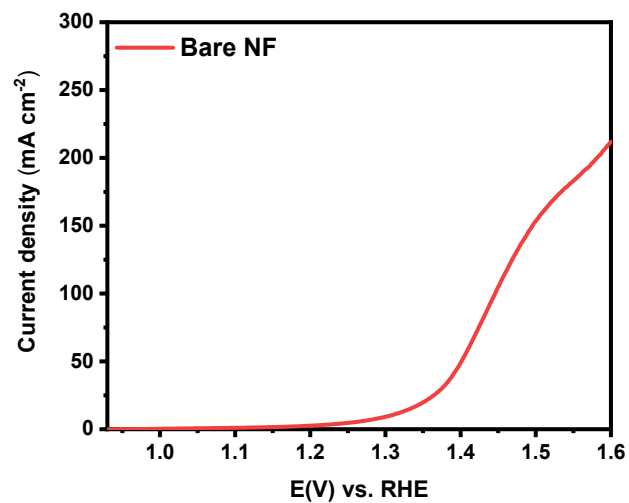


Fig. S18. LSV curves of bare NF with 0.1 M glucose in 1.0 M KOH.

Table S1. Comparison of our catalyst with the reported catalysts for electrochemical saccharides oxidation

| Electrocatalyst | Electrolyte | Potential (V vs. RHE) | j (mA cm ⁻²) | Ref. |
|--|---------------------------------|--------------------------|-------------------------------|----------------------|
| NiFeO _x -NF | 1 M KOH + 50 mM glucose | 1.30 | 61.5 | 1 |
| Fe _{0.1} -CoSe ₂ /CC | 1 M KOH + 500 mM glucose | 1.35 | 100 | 2 |
| Fe ₂ P/SSM | 1 M KOH + 500 mM glucose | 1.33 | 100 | 3 |
| Ru@Ni-B/NF | 1 M KOH + 100 mM glucose | 1.24 | 10 | 4 |
| Co ₉ S ₈ /Ni ₃ S ₂ | 1 M KOH + 50 mM xylose | 1.27 | 100 | 5 |
| NiCoP | 1 M KOH + 100 mM xylose | 1.29 | 100 | 6 |
| Ni-MoS ₂ NPs | 1 M KOH + 300 mM glucose | 1.46 | 10 | 7 |
| NiVP/Pi-VC | 1 M KOH + 100 mM glucose | 1.3 | 10 | 8 |
| CNT@Co/CoP | 1 M KOH + 500 mM glucose | 1.42 | 10 | 9 |
| Co@NPC | 1 M KOH + 100 mM glucose | 1.46 | 10 | 10 |
| CoWO ₄ | 1 M KOH + 100 mM glucose | 1.44 | 10 | 11 |
| NiO/NiCo₂O₄/NF- 300 | 1 M KOH + 100 mM glucose | 0.93 | 10 | This work |
| | | 1.21 | 100 | |
| | | 1.34 | 500 | |

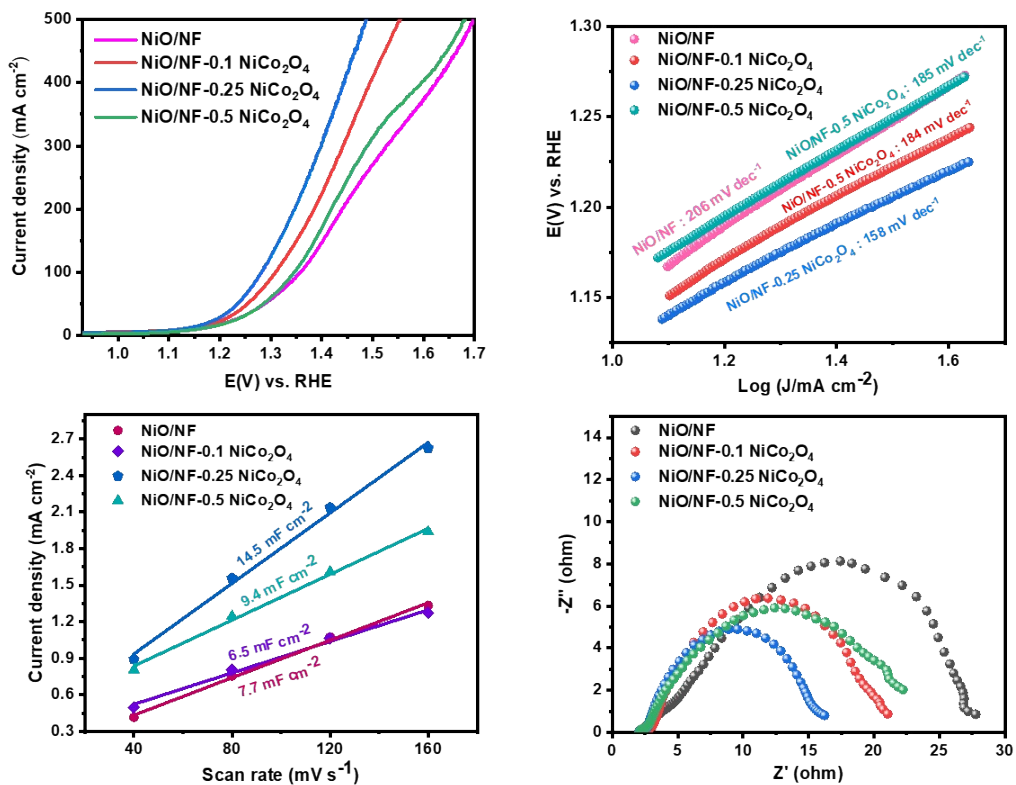


Fig. S19. (a) GOR LSV curves. (b) Tafel plots derived from the LSV curves in (a). (c) Electrochemical double-layer capacitances. (d) Nyquist plots of NiO/NF with different NiCo₂O₄ loadings.

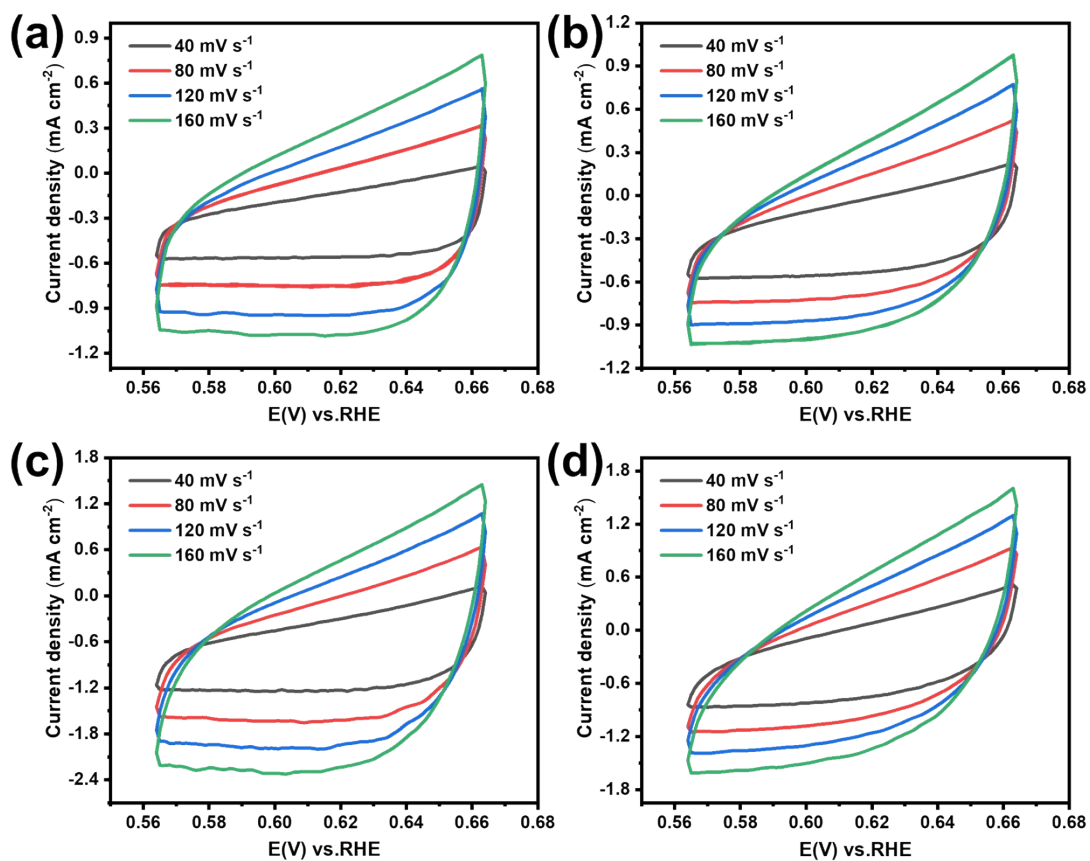


Fig. S20. CV curves of (a) NiO/NF, (b) NiO/NF-0.1 NiCo₂O₄ and (c) NiO/NF-0.25 NiCo₂O₄ (d) NiO/NF-0.5 NiCo₂O₄ at different scan rates.

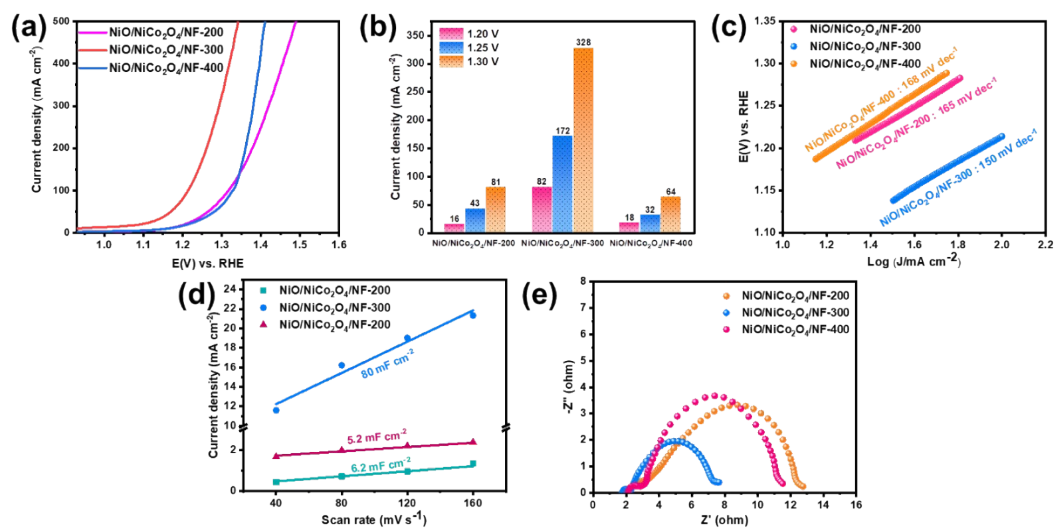


Fig. S21. (a) LSV curves. (b) Corresponding current densities at different potentials. (c) Tafel plots derived from the LSV curves in (a). (d) Electrochemical double-layer capacitances. (e) Nyquist plots of samples obtained at different calcination temperatures.

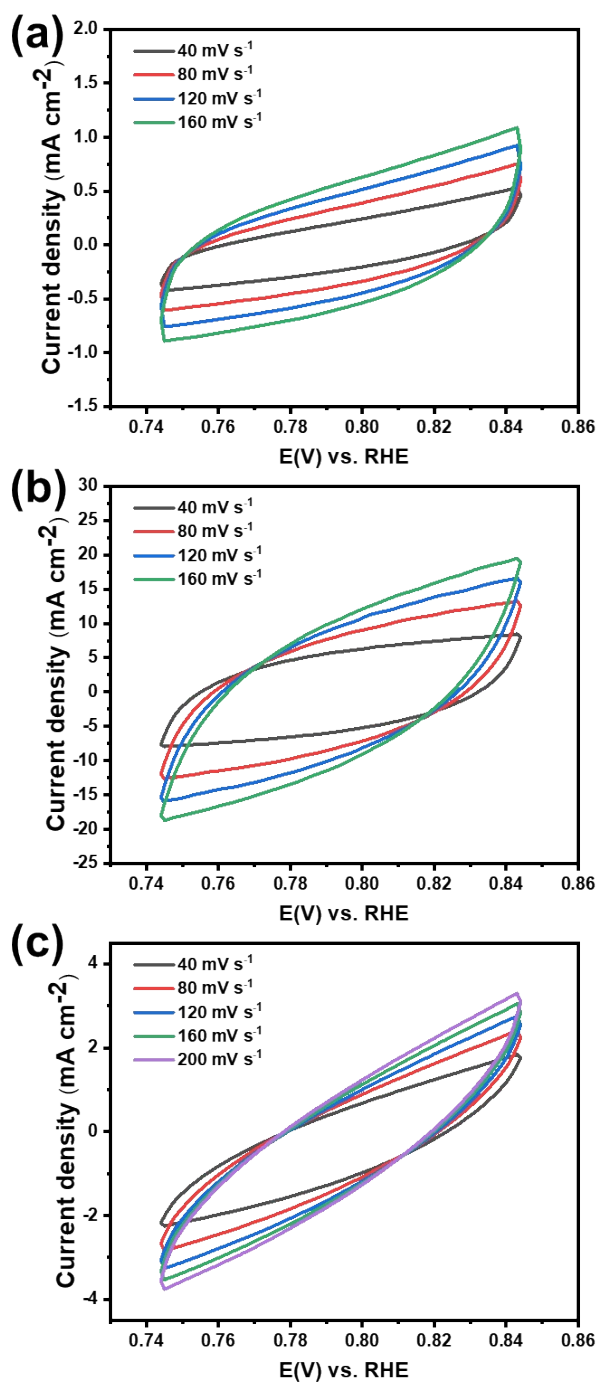


Fig. S22. CV curves of (a) NiO/NiCo₂O₄/NF-200, (b) NiO/NiCo₂O₄/NF-300 and (c) NiO/NiCo₂O₄/NF-400 at different scan rates.

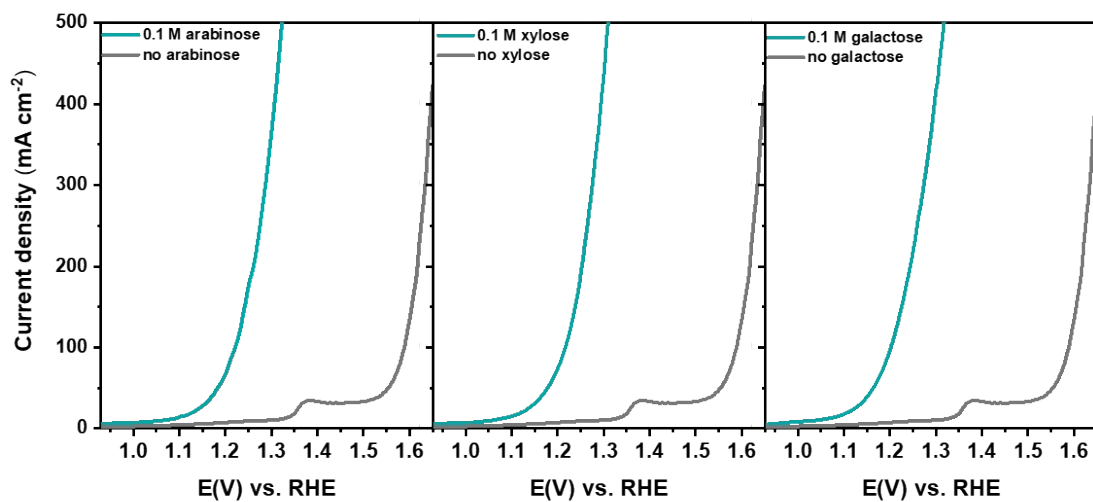


Fig. S23. The corresponding LSV curves of NiO/NiCo₂O₄/NF-300 with and without 0.1 M monosaccharide (galactose, xylose, arabinose) in 1.0 M KOH.

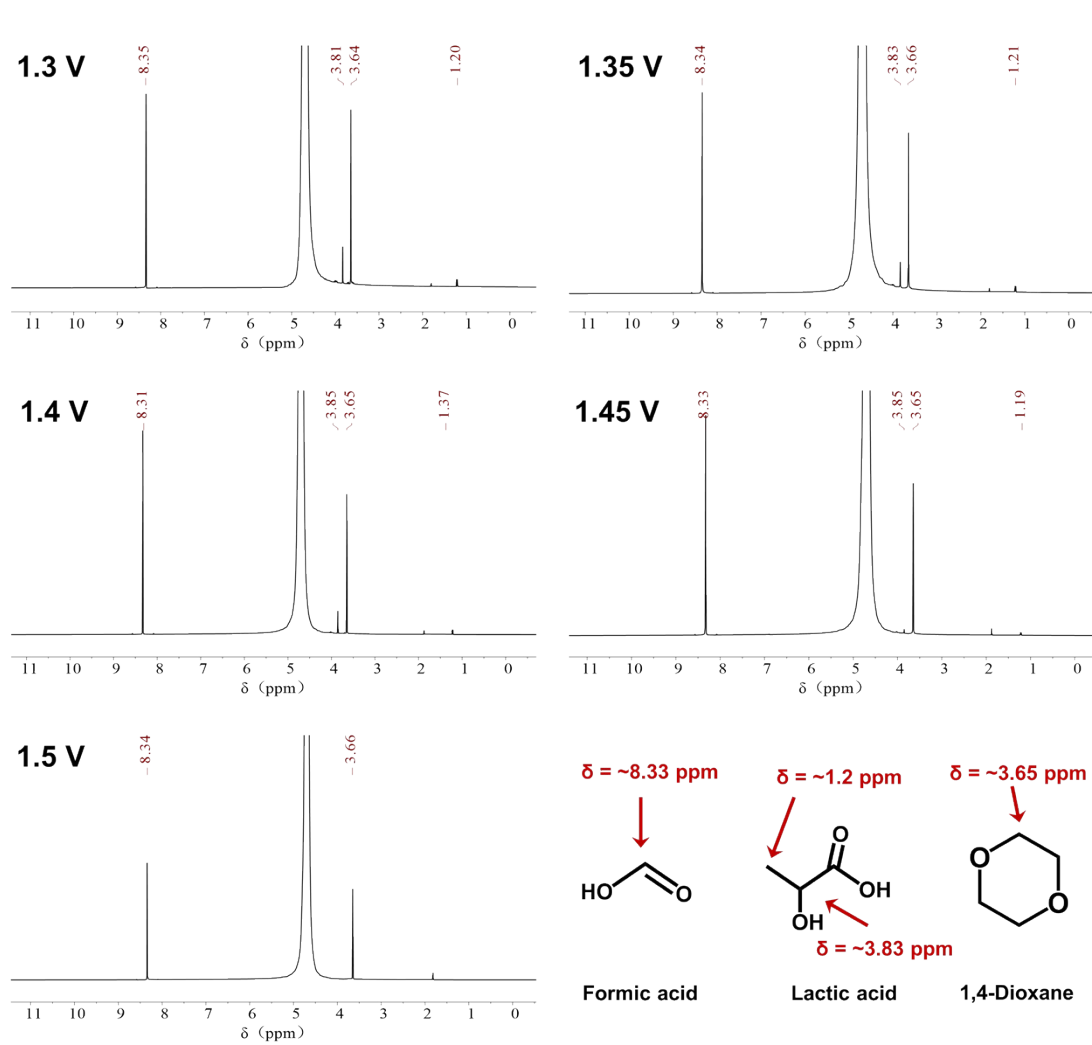


Fig. S24. ^1H qNMR spectra of oxidized products in the electrolyte after 12 h electrolysis of glucose at 1.3 V, 1.35 V, 1.4 V, 1.45 V and 1.5 V.

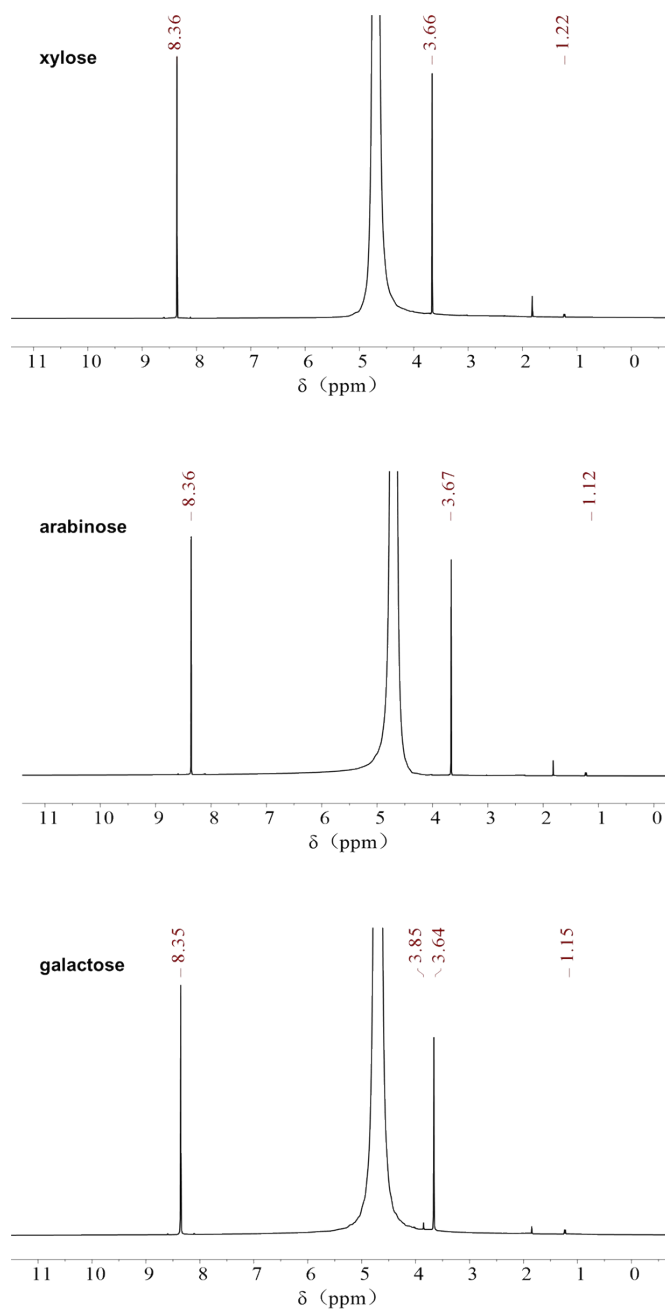


Fig. S25. ¹H qNMR spectra of oxidized products of xylose, galactose and arabinose in the electrolyte after electrolysis at 1.35 V for 12 h.

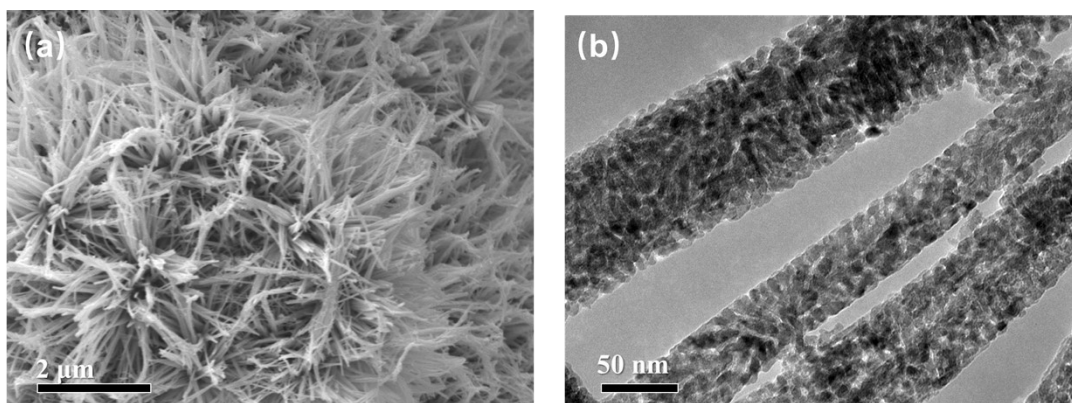


Fig. S26. (a) SEM and (b) TEM image of post-NiO/NiCo₂O₄/NF-300.

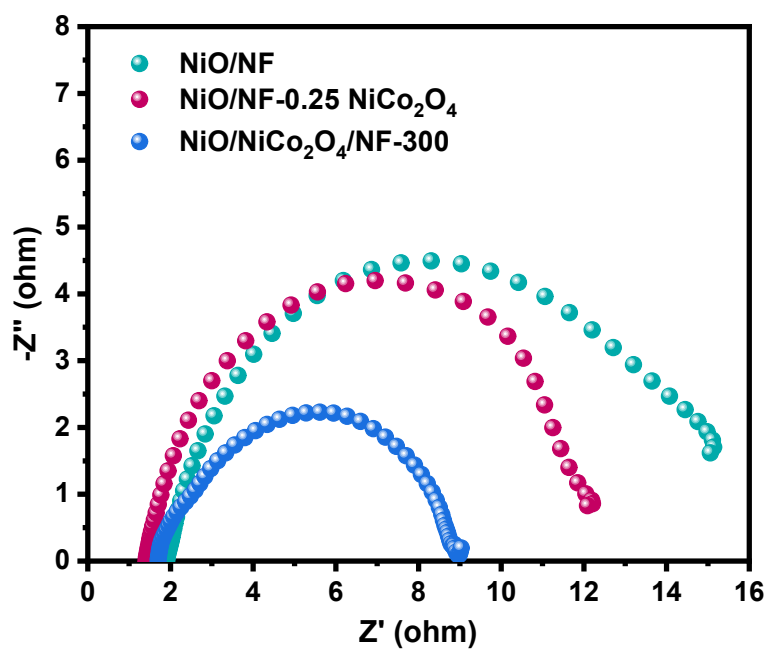


Fig. S27. Nyquist plots of NiO/NF, NiO/NF-0.25 NiCo₂O₄ and NiO/NiCo₂O₄/NF-300.

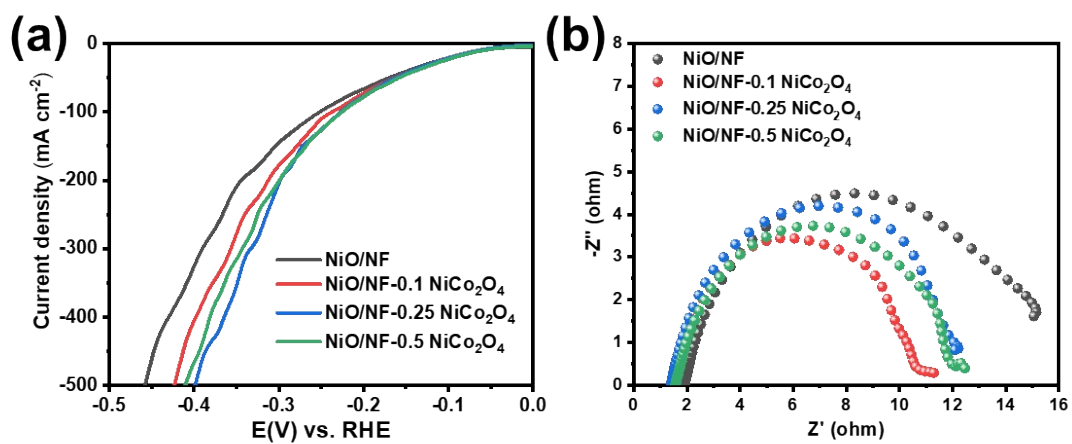


Fig. S28. (a) LSV curves. (b) Nyquist plots of samples with different NiCo₂O₄ loadings.

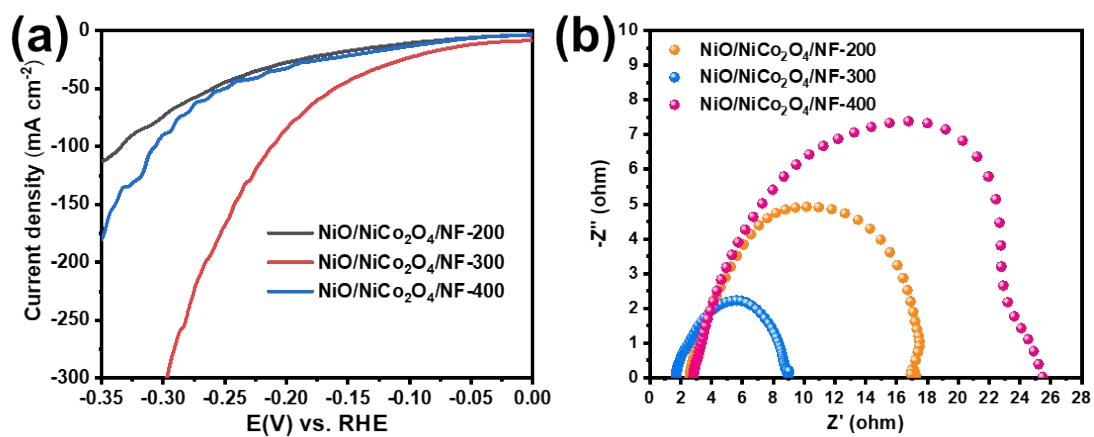


Fig. S29. (a) LSV curves. (b) Nyquist plots of samples obtained at different calcination temperatures.

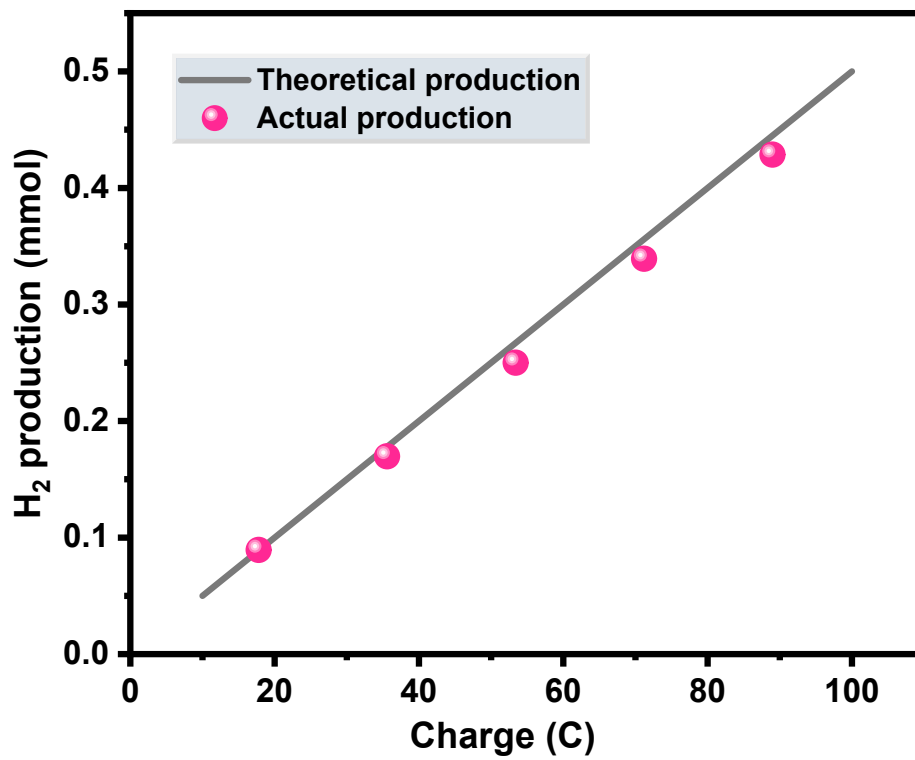


Fig. S30. Actual H₂ production compared with theoretically calculated H₂ production.

References

1. W. J. Liu, Z. Xu, D. Zhao, X. Q. Pan, H. C. Li, X. Hu, Z. Y. Fan, W. K. Wang, G. H. Zhao, S. Jin, G. W. Huber and H. Q. Yu, *Nat. Commun.*, 2020, **11**, 265.
2. D. Zheng, J. Li, S. Ci, P. Cai, Y. Ding, M. Zhang and Z. Wen, *Appl. Catal. B: Environ.*, 2020, **277**, 119178.
3. P. Du, J. Zhang, Y. Liu and M. Huang, *Electrochem. commun.*, 2017, **83**, 11-15.
4. H. Liu, R. Zhang, L. Chen, L. Wang, Y. Guo and Y. Yang, *Adv. Sustain. Syst.*, 2021, **5**, 2000184.
5. Y. Liu, R. Zou, B. Qin, J. Gan and X. Peng, *Chem. Eng. J.*, 2022, **446**, 136950.
6. Y. Yang, R. Zou, J. Gan, Y. Wei, Z. Chen, X. Li, S. Admassie, Y. Liu and X. Peng, *Green Chem.*, 2023, **25**, 4104.
7. X. Liu, P. Cai, G. Wang and Z. Wen, *Int. J. Hydrog. Energy*, 2020, **45**, 32940-32948.
8. N. Thakur, D. Mehta, A. Chaturvedi, D. Mandal and T. C. Nagaiah, *J. Mater. Chem. A*, 2023, **11**, 15868.
9. Y. Zhang, Y. Qiu, Z. Ma, Y. Wang, Y. Zhang, Y. Ying, Y. Jiang, Y. Zhu and S. Liu, *J. Mater. Chem. A*, 2021, **9**, 10893.
10. D. Li, Y. Huang, Z. Li, L. Zhong, C. Liu and X. Peng, *Chem. Eng. J.*, 2022, **430**, 132783.
11. A. Chaturvedi, D. Gupta, S. Kaur, K. Garg and T. C. Nagaiah, *J. Mater. Chem. A*, 2023, **11**, 18280-18290.