

Electronic Supplementary Information

Experimental section

Materials: Sodium hydroxide (NaOH, >96%), nickel nitrate ($\text{Ni}(\text{NO}_3)_2$, 99.9%), potassium chloride (KCl, >99.5%), hydrochloric acid (HCl, 12 M), and ethanol (99%) were obtained from Beijing Chemical Reagent Co. Ltd. Glucose (99.99%), ascorbic acid (AA, >99.99%), sodium chloride (NaCl, >99.5%), calcium chloride (CaCl_2 , >99.5%), uric acid (UA, >99.99%), dopamine (DA, >99%), and gibberellic acid (GA, >99%) were acquired from Aladdin Ltd. All reagents are analytical grade. Titanium plate (TP) (0.4 mm thick) was supplied by China Qingyuan Metal Materials Co. Ltd.

Ni@TiO₂/TP synthesis: Initially, TP underwent ultrasonic treatment in HCl, ethanol, and water for 10 min each. Following our previous work¹⁻⁴, the treated TP was then soaked in 5 M NaOH and heated at 180°C in an autoclave for 24 h to obtain sodium titanate/TP. Next, sodium titanate/TP was immersed in 0.05 M $\text{Ni}(\text{NO}_3)_2$ to exchange Na^+ for Ni^{2+} . After rinsing and drying, it was annealed at 500°C under an Ar/H₂ atmosphere for 2 h, resulting in Ni@TiO₂/TP. For comparison, TiO₂/TP was synthesized similarly, using dilute HCl instead of $\text{Ni}(\text{NO}_3)_2$ to exchange Na^+ for H^+ .

Characterizations: X-ray diffraction (XRD) analyses were conducted using a Rigaku D/MAX 2550 diffractometer. X-ray photoelectron spectroscopy (XPS) measurements were performed with a Thermo Scientific K-Alpha spectrometer, utilizing magnesium (Mg) for excitation. Scanning electron microscope (SEM) images were equipped with a Hitachi S-4800 field emission microscope. Transmission electron microscopy was carried out using an FEI Talos F200x microscope.

Electrochemical measurements: The electrochemical performance of Ni@TiO₂/TP and TiO₂/TP (0.2 ~ 0.5 cm) electrodes was studied using a conventional three-electrode system on a Chi660E electrochemical workstation. A platinum wire served as the counter electrode, an Ag/AgCl electrode as the reference, and the prepared Ni@TiO₂/TP electrode as the working electrode. Cyclic voltammetry (CV) scans were performed within a potential range of 0.2 to 0.6 V. Glucose sensing via chronoamperometry (CA) was conducted at 0.50 V, with 0.1 M NaOH as the

electrolyte for both CV and CA measurements. All potentials were referenced to the Ag/AgCl electrode without correction.

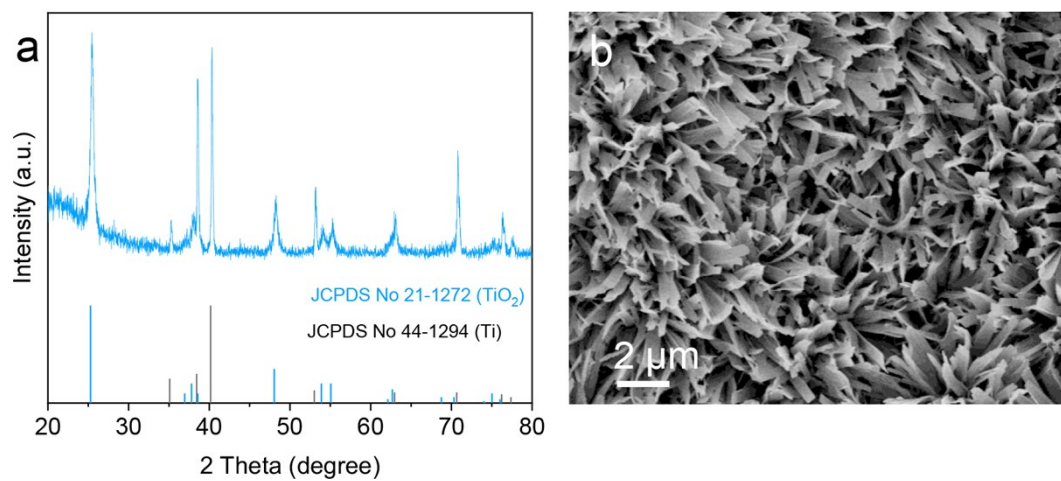


Fig. S1. (a) XRD pattern and (b) SEM image of TiO₂/TP.

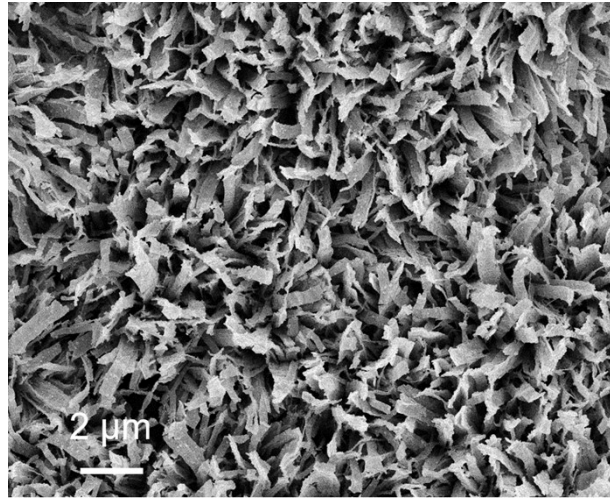


Fig. S2. SEM image of Ni@TiO₂/TP

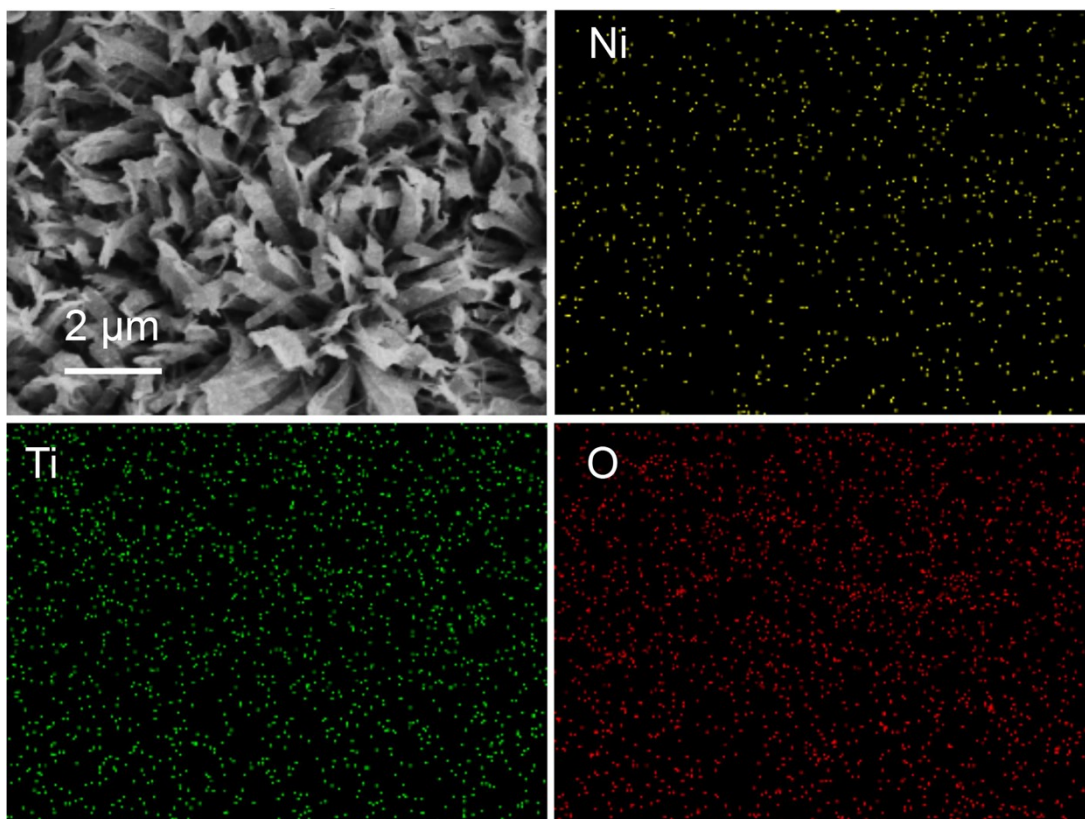


Fig. S3. SEM and corresponding EDX elemental mapping images of Ni@TiO₂.

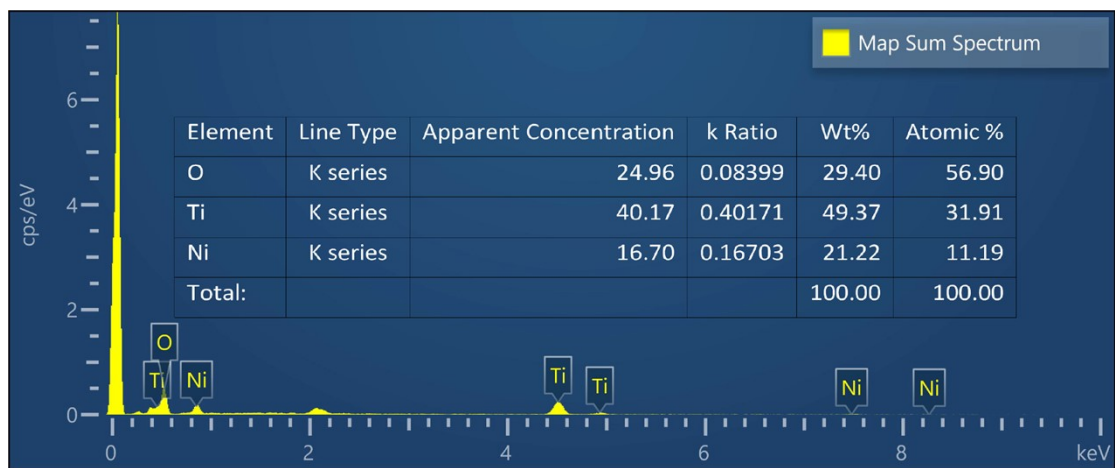


Fig. S4. EDX spectrum of Ni@TiO₂.

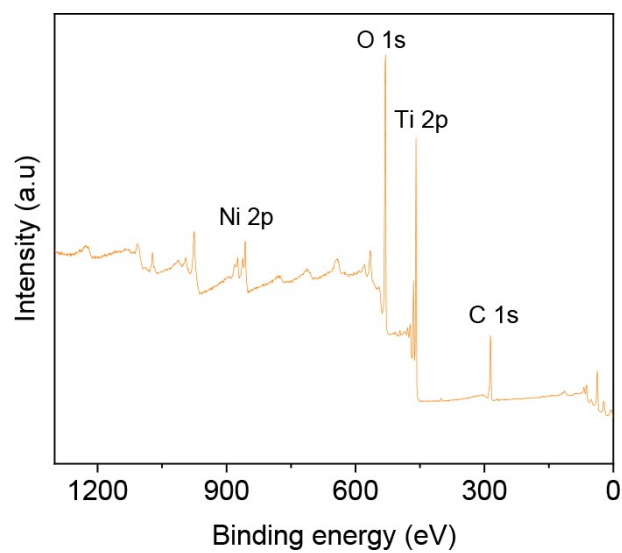


Fig. S5. XPS survey spectrum of Ni@TiO₂.

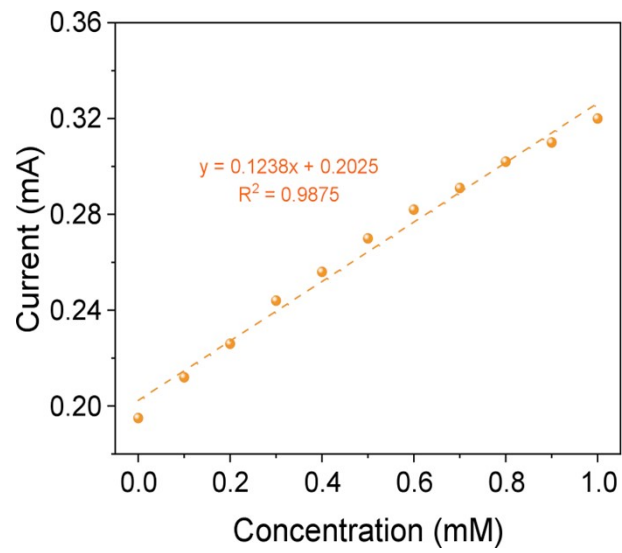


Fig. S6. Plot of the current vs. the concentration of glucose for Ni@TiO₂/TP.

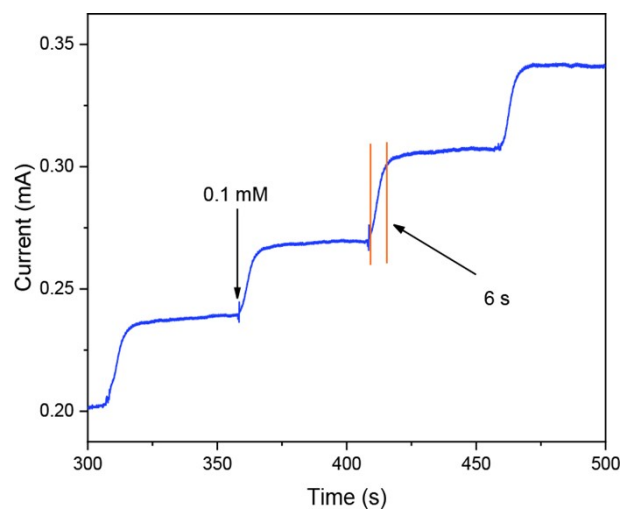


Fig. S7. Current response time of Ni@TiO₂/TP.

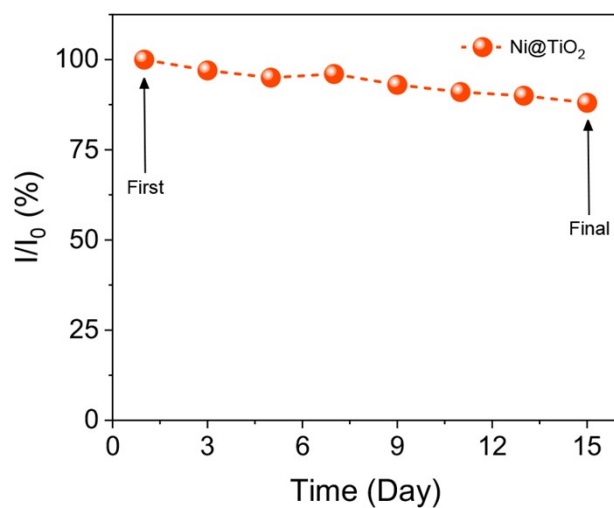


Fig. S8. Relative current response of the Ni@TiO₂/TP electrode towards 0.1 mM glucose after 15 days of storage.

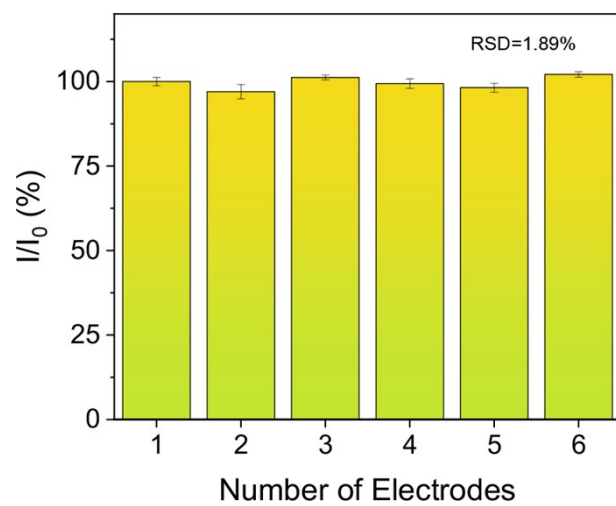


Fig. S9. Inter-electrode repeatability of Ni@TiO₂/TP.

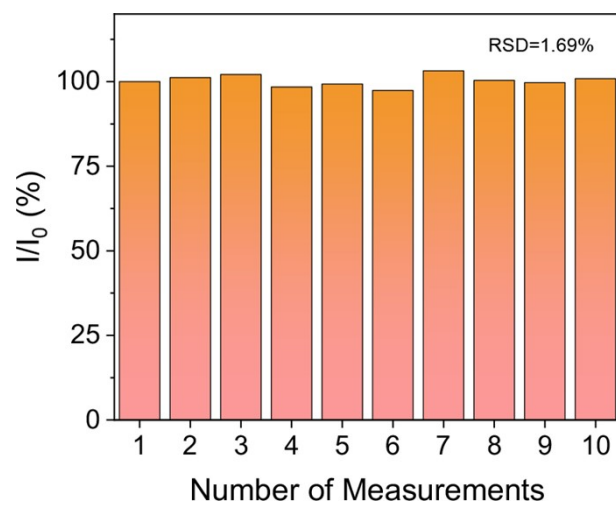


Fig. S10. Intra-electrode repeatability of Ni@TiO₂/TP.

Table S1. Comparison of the performances of Ni@TiO₂/TP with other reported non-enzymatic electrode for glucose detection.

Electrodes	Sensitivity ($\mu\text{A mM}^{-1}\text{cm}^{-2}$)	Linger range (mM)	LOD (μM)	Ref.
Ni@TiO ₂ /TP	10060 3940	0.001-1	0.08	This work
Ni-TiO ₂ /XC72R	3300 273.7	0.05-1 1-20	0.144	5
NiO-TiO ₂ /GCE	24.85	0.002-2	0.7	6
Ni(OH) ₂ /TiO ₂	192	0.03-14	8	7
NPs/TiO ₂ NTs	700	0.004-4.8	2	8
WO ₃ doped TiO ₂ nanotubes with Ni(OH) ₂ nanoparticles	70	/	28	9
Ni-MOF NSAs/CC	13428.89	0.001-7	0.57	10
Conductive Ni-MOF	21744	0.001-8	0.66	11
Ni-DLC/TiO ₂	1063.78	0.99-22.9	0.53	12
Ni/rGO/PP	/	0.0005-1	0.36	13
NiCo-LDH/MWCNTs	2.55 1.15	0.0001-3 3-9.23	0.03	14

Table S2. Recovery tests for Ni@TiO₂/TP glucose biosensor in human blood serum and cell culture fluid.

Sample	Added (mM)	Found (mM)	Recovery (%)	RSD (%) (n=3)
Human blood serum	0.1	0.097	97.4	1.5
	0.3	0.294	98.3	2.7
	0.5	0.508	101.5	2.2
Cell culture fluid	0.1	0.096	96.1	3.4
	0.3	0.307	102.3	2.1
	0.5	0.494	98.8	4.2

References

- 1 X. Fan, D. Zhao, Z. Deng, L. Zhang, J. Li, Z. Li, S. Sun, Y. Luo, D. Zheng, Y. Wang, B. Ying, J. Zhang, A. A. Alshehri, Y. Lin, C. Tang, X. Sun and Y. Zheng, *Small*, 2023, **19**, 2208036.
- 2 K. Dong, Y. Yao, H. Li, H. Li, S. Sun, X. He, Y. Wang, Y. Luo, D. Zheng, Q. Liu, Q. Li, D. Ma, X. Sun and B. Tang, *Nat. Synth.*, 2024, **3**, 763–773.
- 3 J. Liang, P. Liu, Q. Li, T. Li, L. Yue, Y. Luo, Q. Liu, N. Li, B. Tang, A. A. Alshehri, I. Shakir, P. O. Agboola, C. Sun and X. Sun, *Angew. Chem. Int. Edit.*, 2022, **61**, e202202087.
- 4 P. Wu, J. Fan, Y. Tai, X. He, D. Zheng, Y. Yao, S. Sun, B. Ying, Y. Luo, W. Hu, X. Sun and Y. Li, *Food Chem.*, 2024, **447**, 139018.
- 5 J. P. de los Rios, V. Galvan and G. K. S. Prakash, *ECS Adv.*, 2023, **2**, 026502.
- 6 S. Rajendran, D. Manoj, K. Raju, Dionysios. D. Dionysiou, Mu. Naushad, F. Gracia, L. Cornejo, M. A. Gracia-Pinilla and T. Ahamad, *Sensor. Actuat. B-Chem.*, 2018, **264**, 27–37.
- 7 A. Gao, X. Zhang, X. Peng, H. Wu, L. Bai, W. Jin, G. Wu, R. Hang and P. K. Chu, *Sensor. Actuat. B-Chem.*, 2016, **232**, 150–157.
- 8 S. Yu, X. Peng, G. Cao, M. Zhou, L. Qiao, J. Yao and H. He, *Electrochim. Acta*, 2012, **76**, 512–517.
- 9 S. Sharma, S. K. Ganeshan, S. Kundu and K. N. Chappanda, *IEEE T. Nanotechnol.*, 2021, **20**, 185–193.
- 10 Y. Qiao, R. Zhang, F. He, W. Hu, X. Cao, J. Jia, W. Lu and X. Sun, *New J. Chem.*, 2020, **44**, 17849–17853.
- 11 Y. Qiao, Q. Liu, S. Lu, G. Chen, S. Gao, W. Lu and X. Sun, *J. Mater. Chem. B*, 2020, **8**, 5411–5415.
- 12 Y. Kang, X. Ren, Y. Li and Z. Yu, *Molecules*, 2022, **27**, 5815.
- 13 Z. Li, Z. Chen, X. Ji, H. Jin, Y. Si, J. Zhang, C. Chen and D. He, *Nano Res.*, 2024, **17**, 6258–6264.

- 14 Y. Zhu, J. Qian, K. Xu, W. Ouyang, J. Yang and N. Yang, *Chem. Eng. J.*, 2024, **485**, 149795.