

Carbon-supported Nickel/Nickel Oxide Nanohybrid Composite as a High-Performance Sensor for Electrochemical Non-Enzymatic Glucose Detection

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Synthesis of graphitic carbon nitride (g-C₃N₄)

Bulk G-C₃N₄ was prepared according to a previously reported approach¹. In this process, 3 g of melamine was grinded for 10 minutes. Then, the resulting compound was transferred to a silica crucible for calcination at 550 °C (heating rate of 5 °C/min), and holding the sample at the same temperature for 3 h until a yellow powder of bulk g-C₃N₄.

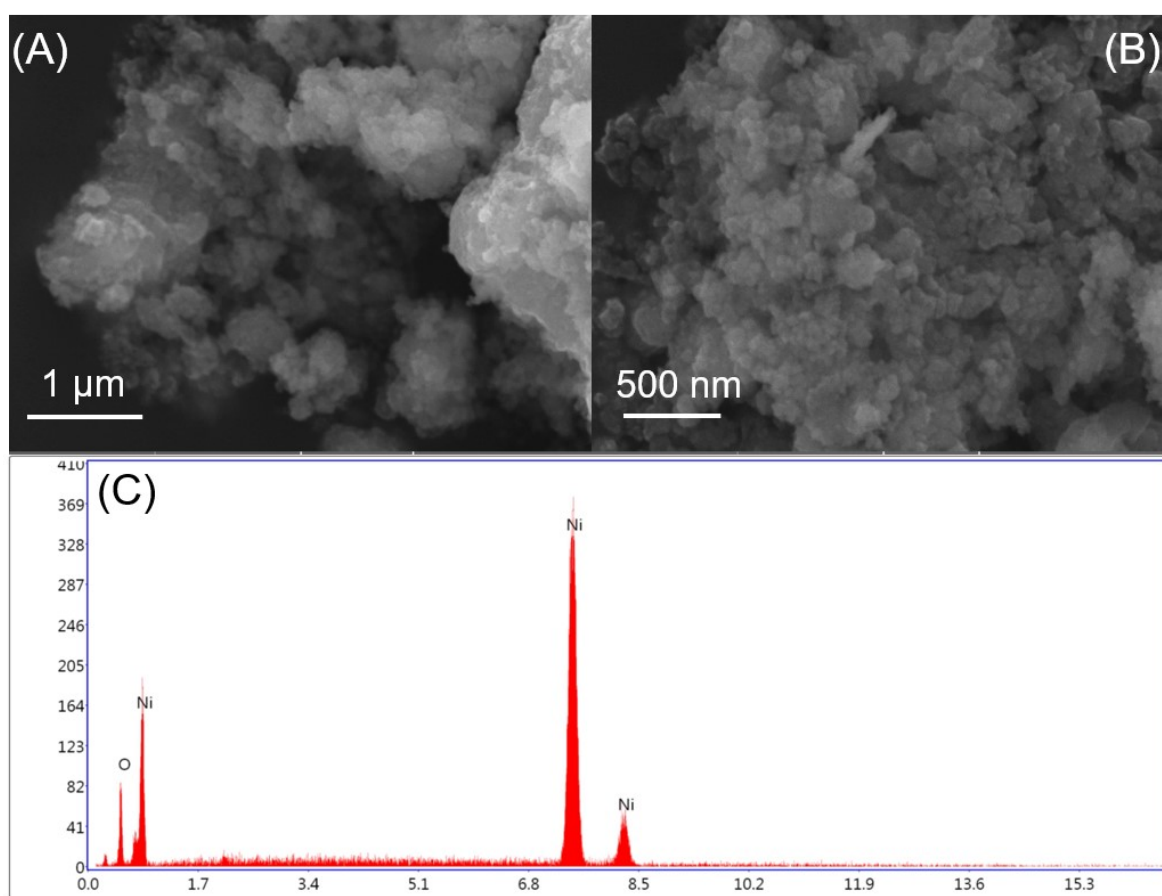


Figure S1 (A)-(B) SEM micrographs and **(C)** Energy dispersive spectrum of Ni/NiO sample.

Table S1. The weight percentage of each element in Ni/NiO and Ni/NiO@C samples

Compound	Element	Weight (%)	Atomic (%)	Intensity	Error (%)	K ratio
Ni/NiO	Ni	89.86	70.72	393.30	2.43	0.9065
	O	10.14	29.28	29.35	11.62	0.0418
Ni/NiO@C	Ni	50.48	19.30	321.48	10.93	0.4642
	O	25.48	35.75	73.39	2.91	0.4642
	C	24.04	44.94	27.25	12.59	0.0668

was obtained from EDS.

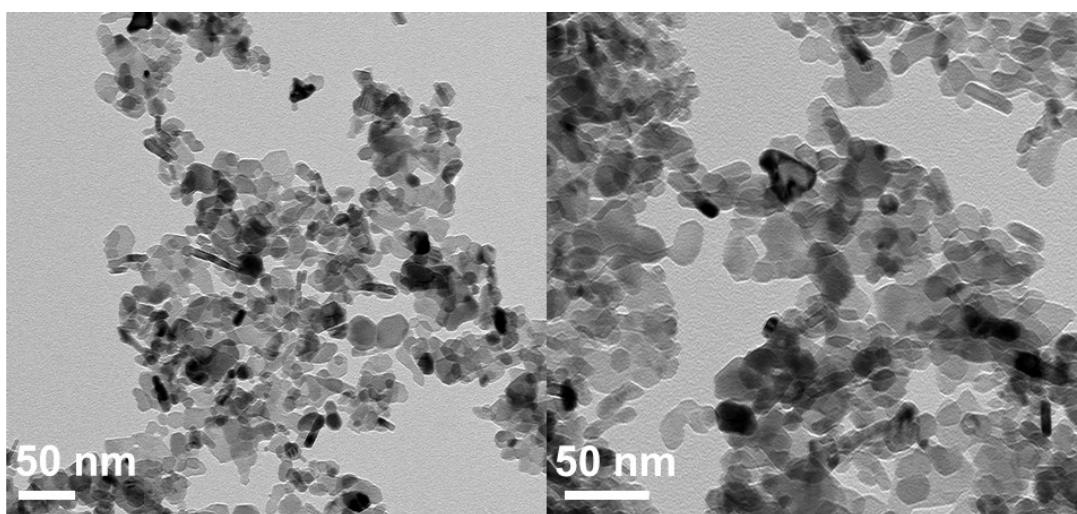


Figure S2. TEM images of Ni/NiO sample.

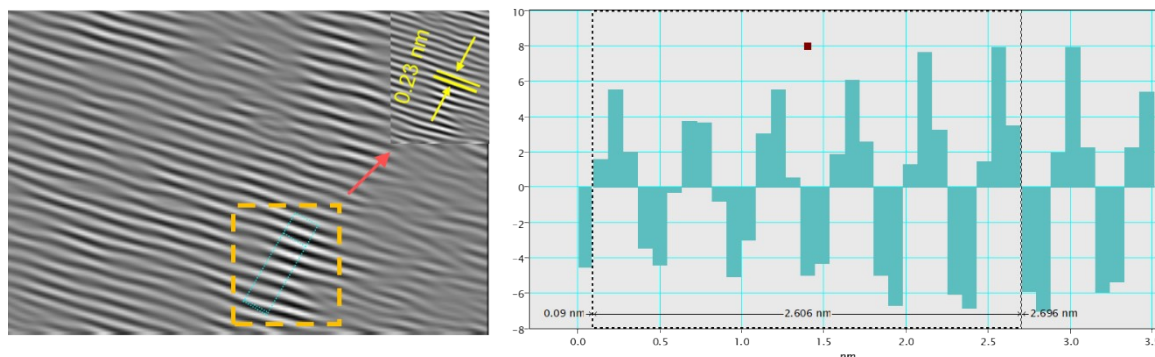


Figure S3. The determination of d-spacing from HR-TEM images

Electrochemical Characterization of Ni/NiO and Ni/NiO@C material

Before the electrochemical measurements, the electrode surface properties of Ni/NiO and Ni/NiO@C modified GCE were examined by using electrochemical impedance on benchmark ferricyanide $[\text{Fe}(\text{CN})_6]^{3-/4-}$ redox system. In the Nyquist diagram, the semicircle signifies the charge transfer resistance (R_{ct}), which is related to the kinetics of electron transfer at the electrode surface, while the linear portion denotes diffusion^{2, 3}. **Figure S4** represents the Nyquist plots of 0.1 M KCl containing 5 mM $[\text{Fe}(\text{CN})_6]^{3-/4-}$ at GCE [**Curve (iii)**], Ni/NiO/GCE [**Curve (ii)**] and Ni/NiO@C/GCE [**Curve (i)**]. From **Figure S4**, a semicircle with a larger diameter is attained for bare GCE while a smaller semicircle was observed in Ni/NiO/GCE and Ni/NiO@C/GCE in the frequency range of 0.01 Hz to 100 kHz. For bare GCE, Ni/NiO/GCE, and Ni/NiO@C/GCE, the R_{ct} values of 26.3, 6.1, and 2.3 k Ω , respectively. Compared with bare GCE and Ni/NiO/GCE, Ni/NiO@C/GCE showed a very smaller semicircle with lower R_{ct} which indicates the higher electrocatalytic activity. The higher electrocatalytic activity of Ni/NiO@C is observed due to the presence of conductive carbon, oxygen vacancies and surface defects on the crystal lattice system which accelerate the charge transfer

process on the electrode-electrolyte interface. This could be the reason for the improved electrochemical response at the electrode-electrolyte interface.

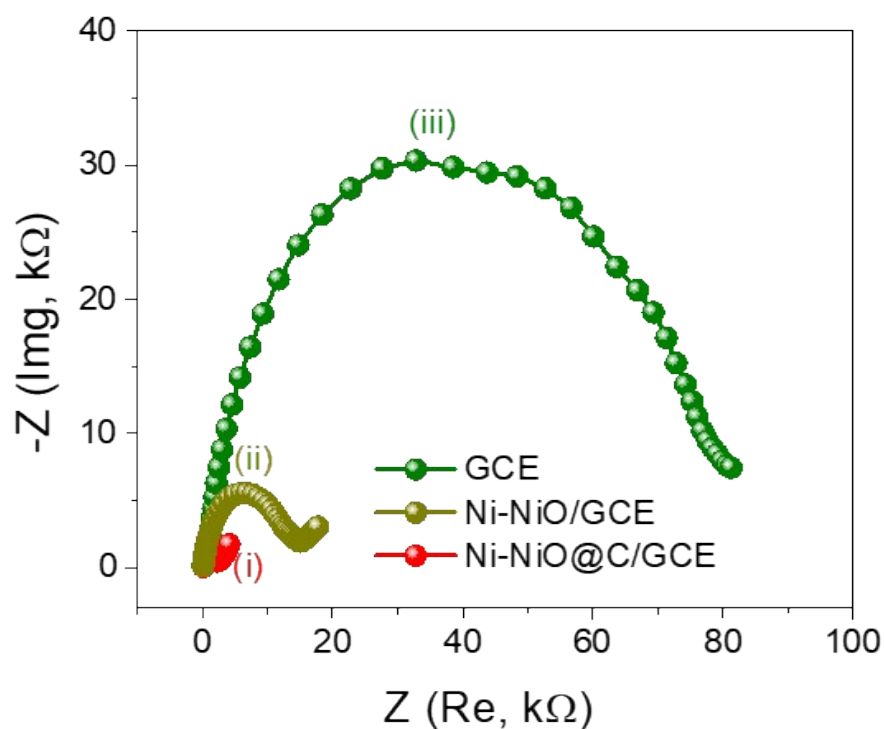


Figure S4. Electrochemical impedance performance of unmodified GCE [Curve (iii)], Ni/NiO/GCE [Curve (ii)], and Ni/NiO@C/GCE [Curve (i)] in 0.1M KCl containing 5 mM $[\text{Fe}(\text{CN})_6]^{3-/4-}$.

References

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2. K. K. Tadi and R. V. Motghare, *Journal of The Electrochemical Society*, 2016, **163**, B286.
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