Sustainable non-enzymatic glucose sensors from residual biomass-derived carbon materials

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Introduction

Glucose is the most abundant monosaccharide in nature and the primary source of cellular energy. It plays a crucial role in carbohydrate metabolism, and abnormal blood glucose levels are associated with diabetes mellitus, a leading cause of mortality worldwide. According to the World Health Organization (WHO), the global prevalence of diabetes is projected to increase to 783 million by 2045. This growing prevalence has spurred the development of accurate glucose sensing technologies.

Electrochemical glucose sensors are typically classified as enzymatic or non-enzymatic. While enzymatic sensors offer high selectivity and sensitivity, their performance can be affected by environmental factors such as temperature and pH. Non-enzymatic sensors provide improved stability, reproducibility, and cost-effectiveness, making them suitable for miniaturized or wearable devices (Yan, T *et al* 2025). Transition metals, particularly Ni, are attractive electrocatalysts due to their abundance, low cost, and favourable redox behaviour. Incorporating Ni into carbon-based materials enhances electron transfer, increases active site density, and improves glucose oxidation efficiency, while carbon supports derived from residual biomass provide high conductivity and large surface area. Biomass can be converted into porous carbons via hydrothermal carbonization (HTC) and chemical activation, with performance further enhanced through Ni doping (Mahieddine, A *et al* 2023).

In this study, *Typha domingensis* (TD), *Cladium mariscus* (CM), and *Phragmites australis* (PA) were selected as biomass sources due to their abundance and invasive nature in wetlands. The materials were transformed into electroactive carbons via HTC, KOH chemical activation, Ni doping, and thermal treatment at 600 ºC. The resulting materials were characterized and tested as non-enzymatic glucose sensors.

Materials and Methods

Biomass samples (TD, CM, PA) were collected from the *Tablas de Daimiel National Park*, Spain. The raw materials were rinsed, dried at 60 ºC for 48 h, and ground. Ground biomass was dispersed in water (130 g/L), stirred 30 min, and hydrothermally treated at 200 ºC for 2 h. The hydrochar was filtered, washed, and dried at 90 ºC. Hydrochars were labeled TD/hydro, CM/hydro, and PA/hydro. Hydrochars were mixed with 50% KOH solution, stirred at 80 ºC for 2 h, dried, and thermally activated at 600 ºC under N_2. Ni doping was achieved by adding 0.5 M $NiSO_4 \cdot 6H_2O$ to the KOH mixture prior to thermal treatment. Final materials were labelled TD/KOH, CM/KOH, PA/KOH and TD/KOH-Ni, CM/KOH-Ni, PA/KOH-Ni.

Electrochemical measurements were conducted in a three-electrode cell using a rotating glassy carbon electrode as the working electrode, a Hg/HgO reference electrode, and a graphite counter electrode. The electrolyte consisted of 1 M NaOH at ambient temperature. Cyclic Voltammetry (CV) (-100 to 100 mV for ECSA; 100 to 600 mV for glucose detection, at 20 mV/s) and Chronoamperometry (CA) (320 to 390 mV, stepwise glucose addition) were performed. Selectivity was evaluated using Ascorbic acid (AA), Uric acid (UA), Acetaminophen (AP), and NaCl at 0.1 mM vs 1 mM glucose.

Results and Discussion

Electrochemical tests revealed that TD/KOH-Ni exhibited the best glucose sensing performance among all biomass-derived materials. CV measurements showed pronounced glucose oxidation peaks, and CA experiments demonstrated clear stepwise current increases with successive glucose additions.

The sensor achieved a sensitivity of 85.86 μA/(cm^2·mM), a LOD of 45.13 μM, and a linear range up to 6 mM, highlighting its suitability for physiological glucose monitoring.

Selectivity tests confirmed the sensors robustness: endogenous interferents produced negligible current, whereas glucose generated a significant amperometry response at 370 mV vs Hg/HgO (see Figure 1). These results indicate high selectivity toward glucose even in the presence of physiologically relevant interfering compounds (AA, UA, AP or NaCl).

The enhanced performance of TD/KOH-Ni is attributed to several factors: high ECSA (41.28 cm^2), defect-rich carbon structure from KOH activation, Ni nanoparticles improving conductivity and catalytic activity, and surface functionalities (C=O, C=N) facilitating glucose oxidation. Morphological and compositional analyses using high-resolution scanning electron microscopy (HR-SEM), energy-dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD), and X-ray photoelectron spectroscopy (XPS) confirmed porous, nickel-decorated structures with electroactive nickel species, explaining the superior electrocatalytic behaviour.

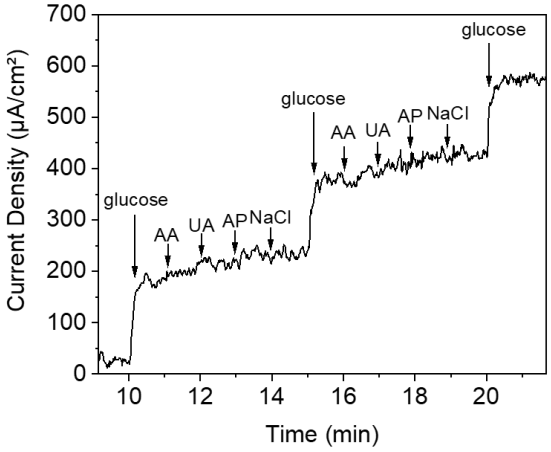

Figure 1. Current response of TD/KOH-Ni electrode in 1 M NaOH at 370 mV upon successive addition of glucose, AA, UA, AP and NaCl.

Conclusions

Non-enzymatic glucose sensors were successfully developed from invasive wetland biomass (TD, CM, PA). HTC, KOH activation, Ni doping, and thermal treatment yielded electroactive carbons suitable for alkaline media. The TD/KOH-Ni sensor exhibited high sensitivity (85.86 μA/(cm^2·mM)), low LOD (45.13 μM), wide linear range (up to 6 mM), and excellent selectivity. Despite slight ECSA reduction due to Ni, conductivity and electrocatalytic activity were enhanced. Morphological (HR-SEM), compositional (EDX, XRD) and surface analyses (XPS) confirmed the structural and chemical features responsible for improved performance. This work demonstrates a cost-effective, environmentally friendly strategy for developing high-performance glucose sensors from residual biomass, valorizing organic waste for sustainable electrochemical applications.

References

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