

OH-compensating non-enzymatic glucose sensor based on a hexagonal metal azolate framework composite for continuous sweat monitoring

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Research Article

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ABSTRACT

Continuous glucose monitoring is transforming analysis by using sweat, a less invasive and more accessible fluid, to provide more accurate readings during exercise. However, obtaining accurate results remains challenging due to its acidity, which can disrupt the alkaline conditions required for non-enzymatic sensing. Herein, we developed a high-surface-area carbon nanofiber (CNFs) composite functionalized with polypyrrole (PPy) and combined with a graphene oxide (GO) composite for non-enzymatic electrochemical glucose sensing in sweat. The composite was fabricated via a sequential drop-casting process using a metal-azolate framework (MAF) for its high electrochemical stability, thereby enabling OH-compensation and ensuring a continuous supply of hydroxyl (OH) groups from GO, compensating for any loss of OH caused by sweat acidity. The CNFs/PPy/GO/MAF composite works efficiently across pH levels 13, 12.5, and 12, with detection limits of 0.033, 0.043, and 0.0145 μM ($\text{S/N} = 3$), respectively. The composite shows high sensitivities of 663, 259.6, and 160.85 $\mu\text{A}/(\text{mM}\cdot\text{cm}^2)$ within the linear ranges of 0.003 to 1.6, 0.003 to 1.4, and 0.003 to 0.7 mM at pH = 13, 12.5, and 12, respectively. Additionally, the composite exhibits high selectivity, stability under decreasing pH, and good repeatability and reproducibility. It yielded sweat analysis results from real samples that closely

matched blood glucose levels measured by a glucometer at all tested pH values of 13, 12.5, and 12. This confirms that the composite offers accurate, reliable, real-time, non-invasive glucose detection under typical alkaline conditions.

KEYWORDS: carbon nanofiber, metal-azolate framework (MAF), graphene oxides, glucose, non-enzymatic, alkaline medium

1 Introduction

Blood glucose levels should remain stable because prolonged high levels can lead to diabetes. Insufficient insulin production or poor cellular response to insulin leads to abnormal levels, making continuous monitoring essential [1, 2]. Once, integrating continuous blood glucose monitors with human activity-tracking technologies opened new possibilities for exploring natural settings. As a result, physical activity is now recognized as a crucial factor in enhancing the accuracy of continuous glucose monitoring during exercise [3-5]. This is most effectively accomplished through sweat-based detection, which offers continuous, non-invasive measurements that provide valuable insights into chemical biomarkers of a person's health [6]. Moreover, wearable features such as flexibility, stretchability, and comfort during exercise enable continuous glucose monitoring, making it less invasive and more convenient than blood draws [7-9]. Non-enzymatic glucose sweat sensors have been developed to address the limitations of enzymatic glucose sensors [10]. These sensors offer advantages over enzymatic sensors, including greater stability, reliability, versatility, freedom from oxygen limitations, cost-effectiveness, and resistance to harsh conditions [11, 12]. Significant advancements have been made in nonenzymatic glucose sensors that use active metals, metal oxides, or nonmetallic materials [13-15]. Since their detection primarily relies on device measurements in an alkaline environment, various techniques to achieve and maintain alkaline conditions are considered [16]. Drop-casting highly concentrated NaOH onto the reaction zone, followed by drying, is used in a non-invasive device that monitors real-time sweat glucose levels [17, 18].

Additionally, a PVA/NaOH solid electrolyte creates an alkaline environment conducive to non-enzymatic glucose detection in sweat [19]. Furthermore, using alkaline solutions inside microcavities allows for their containment with caps, which can replace the alkaline solution and prevent residual sweat interference that might lead to biofouling [20, 21]. Most wearable non-enzymatic sweat glucose sensors incorporate microfluidics to collect sweat, reduce evaporation, and prevent direct human-skin contact with NaOH and the sensing material by channeling sweat.

Many researchers have developed integrated sensor arrays that combine non-enzymatic glucose detection with pH sensing on a single sweat-monitoring platform. Still, this method only measures the pH value and does not assess whether a stable alkaline environment is required for non-enzymatic glucose detection [22, 23]. However, during continuous glucose monitoring, the natural acidity of human sweat (pH 4.5-6.5) interacts with the sensor's initially alkaline environment (pH 13). The high ionic strength of the alkaline solution easily overwhelms the sweat's slight acidity, yet sweat still causes a measurable reduction in local pH to the range 12.5 to 12. Therefore, the material must be specifically designed to compensate for OH⁻ ions and maintain sufficient sensitivity in this range [24, 25]. To address this, we need to solve two issues: first, protect the sensing material from biofouling to ensure its stability and reproducibility. Here, metal-organic frameworks (MOFs), porous materials composed of organic linkers and metal ions like Pt, Ni-Co, Cu, Zn, and Fe, along with cluster-based MOFs and their composites, have attracted attention for electrochemical sweat sensors to detect glucose [26-30]. These metal ions exhibit strong electrochemical redox activity, facilitating the redox reactions of target analytes. However, many MOFs have poor stability in aqueous media (neutral, acidic, and basic) [31], and their performance declines with repeated use due to fragility and low electrical conductivity [32]. The second issue is to ensure a continuous feed of hydroxyl groups (OH) during detection to compensate for any loss of OH. For this OH compensation, we have used a composite containing graphene oxide (GO), which features hydroxyl, phenolic, and epoxy groups on its basal planes, with carboxylic acid groups positioned at the edges [33]. This configuration reduces its electrical conductivity, thereby limiting its use as an electrode material [34]. Nonetheless, the polar oxygen groups confer high hydrophilicity on GO, enhancing dispersibility and facilitating easier processing [35]. These groups also enable chemical modifications, allowing GO to interact with electroactive species and promote at electrodes [36], as well as improving electron transfer in neutral or alkaline electrolytes [37]. Overall, these characteristics position GO as a promising material for advanced electrochemical device applications.

In this contribution, we propose a solution to enhance the sensitivity and stability of non-enzymatic glucose detection in sweat within an alkaline medium. This is achieved by synthesizing high-surface-area CNFsPPy/GO/MAF composite materials to counteract the reduction induced by sweat acidity via a sequential drop-casting process. The CNFs, which serve as a bridge, are functionalized with PPy to enhance the composite's conductivity, thereby

facilitating electron transfer, while the GO provides a continuous flow of OH⁻ ions during detection, compensating for any loss caused by sweat acidity. Metal azolate frameworks (MAF-6-Co^{II}), a subclass of MOFs, are promising due to their higher chemical stability, as demonstrated through the synthesis of MAF-4-Co^{II} and MAF-5-Co^{II}, which exhibit high sensitivity and selectivity for blood glucose detection [38, 39]. The stability of these frameworks stems from inert or hydrophobic imidazolate linkers near the metal centers [40]. Our composite shows enhanced sensitivity and stability even at lower pH values (13, 12.5, and 12).

2 Experimental section

2.1 Materials and reagents

Dimethylformamide DMF solvent, polyacrylonitrile (PAN, $M_w = 150,000$ Da), and absolute ethanol. Ascorbic acid (AA), uric acid (UA), and ethanol (EtOH, 99.5%) were supplied by Shanghai Aladdin Chemical. D (+) glucose was purchased from Damas. Nafion was acquired from Merck Chemical Technology Co., Ltd., Shanghai, China. Potassium chloride (KCl), lactic acid (LA), and other reagents were obtained from Macklin Biochemical Co., Ltd., Shanghai, China. Potassium ferricyanide ($K_3[Fe(CN)_6]$), potassium ferrocyanide ($K_4[Fe(CN)_6]$), and sodium chloride (NaCl) were supplied by Bide Pharmatech Co. Pyrrole (Py) (C₄H₅N, 99%, $M_w = 67.09$ g/mol) and iron trichloride (FeCl₃, 99.99%, $M_w = 162.2$ g/mol, CP) were purchased from Shanghai Aladdin Co., Ltd. Graphite powder was provided by Nanjing XFNANO Materials Tech Co., Ltd., China. Sulfuric acid (H₂SO₄, 98.0%, AR), sodium nitrate (NaNO₃, 99.0%, AR), hydrochloric acid (HCl, 36.0%, AR), hydrogen peroxide solution (H₂O₂, 30.0%, AR) and potassium permanganate (KMnO₄, 99.5%, GR) were brought from Sinopharm Chemical Reagent Co., Ltd., China Ltd. Cobalt(II) acetate [Co(OAc)₂] (99.995% trace metals basis), 2-ethylimidazole (98%) (EIM), benzene (99.9%), cyclohexane (99.5%), were purchased from Sigma-Aldrich. The screen-printed electrode (SPE) was supplied by Poten Technology Co., Ltd., Shandong, China.

2.2 Fabrication of CNFs

A 25 g solution containing 10 wt% PAN in DMF was stirred magnetically at 60 °C for 8 hours. Electrospinning was carried out at a feed rate of 1 mL/h, with an applied voltage of 22 kV and a 15-cm gap between the needle tip and the collector. The composite nanofibrous membranes were produced under controlled relative humidity ($50 \pm 5\%$) and temperature (25 ± 2 °C). The resulting nanofibers were dried in a vacuum oven at 80 °C for 2 h. and the sample was then

stabilized at 280 °C at a rate of 5 °C/min at room temperature. Subsequently, the stabilized nanofibrous samples were annealed at a heating rate of 2 ~ 3 °C/min up to 800 °C in a tube furnace under N₂ flow, then held for 2 hours to complete carbonization.

2.3 Preparation of CNFs/PPy

The CNFs/PPy were produced through oxidative polymerization of pyrrole (Py) [41-43]. First, 1 mL of pyrrole was measured, diluted to 25 mL with distilled water, and 50 mg of CNFs was added under mechanical stirring. Separately, 1 g of FeCl₃ was dissolved in 25 mL of distilled water with mechanical stirring. Both solutions were kept in an ice bath at 0 ~ 5 °C. The FeCl₃ solution was gradually added to the pyrrole solution while stirring in the ice bath for 5 hours, during which a color change signified polymer formation. The mixture was then poured into roughly 250 mL of distilled water to precipitate the CNFs/PPy. The precipitate was collected by centrifugation, washed multiple times with distilled water and acetone to remove unreacted residues, and finally dried overnight at 70 °C under vacuum until a constant weight was reached.

2.4 Preparation of GO

The GO was synthesized by oxidizing graphite powder in concentrated acid using a modified Hummers' method [44]. In brief, the graphitic oxide was produced by stirring 4 g of graphite powder into 89 mL of concentrated H₂SO₄ at room temperature, followed by the addition of 2 g of NaNO₃. The mixture was then cooled to 0 °C, and 12 g was gradually added to the suspension under vigorous agitation [45]. At the same time, the suspension temperature should be maintained at around 20 °C. After the ice bath was removed, the reaction system was transferred to a 36 °C water bath and maintained for 35 minutes. The solution was changed to a viscous mixture (thick paste) and continued to oxidize as 182 mL of water was slowly added. The mixture was transferred to an oil bath, and another 186 mL of water and 20 mL of 30% H₂O₂ solution were slowly stirred into the paste for 17 minutes at 98 °C, changing the mixture's color from brown to yellow. The yellow mixture was centrifuged and washed several times with water and HCl to remove metal ions and acid [46]. Suspensions of GO that are monodisperse in size can also be achieved by density-gradient centrifugation. Finally, the mixture was freeze-dried for 24 hours to obtain GO. Due to the ionization of carboxyl groups, GO can be electrostatically stabilized, forming a colloidal suspension in water [47].

2.5 Synthesis of MAF-6-Co^{II}

According to the literature [39], 0.3 g of Co (OAc)₂ was dissolved in water (25 mL) to form the first solution. A second solution was prepared by dissolving 0.3 g of EIM in an emulsion of water and benzene (65:10 v/v). These solutions were rapidly mixed and stirred at room temperature for 30 min. The resulting blue suspension was centrifuged at 10000 rpm for 15 min. The precipitate was washed several times with water and ethanol by centrifugation. Finally, the blue powder was dried in a vacuum oven at 70 °C for 3 hours and stored for subsequent characterization and applications.

2.6 Preparation of a glucose sensor

Suspension A was prepared by dispersing 1.5 mg of CNFs/PPy and 1 mg of graphene oxide in 5 mL of ethanol and then stirring for 20 minutes. Suspension B was prepared by dispersing MAF (4 mg/mL) in a medium containing ethanol and Nafion®117 (1.5:0.5 v/v) by bath sonication for 20 minutes. Nafion®117 serves as both a binder for MAF and a composite adhesive. Approximately 3 µL of suspension A was drop-cast onto the glassy carbon (GCE) electrode, and then 4 µL of suspension B was drop-cast onto the dried surface of suspension A, followed by drying under an infrared lamp for 15 minutes. The same samples were drop-cast onto the working electrode of the SPE. The as-prepared CNFs/PPy/GO/MAF composites were used directly for glucose detection.

2.7 Characterization and Measurements

The morphology of the synthesized samples was examined using Field Emission Scanning Electron Microscopy (FE-SEM, S-4800, Hitachi Ltd., Japan) at 2 kV and 5 kV. High-Resolution Transmission Electron Microscopy (HR-TEM; JEM-2100F, JEOL Ltd., Japan) was used to characterize the composite's nanostructures, including Selected Area Electron Diffraction (SAED). The phase structures of the samples were analyzed using X-ray Diffraction (XRD, TD-3500, Dandong Tongda Science and Technology Co., China; Cu K α , λ = 1.5406 Å). Additionally, Raman spectra were obtained using a micro-Raman spectroscopy system (in Via-Reflex, Renishaw Co., UK). N₂ adsorption-desorption isotherms were used to evaluate the samples' porous structure and Brunauer-Emmett-Teller (BET) surface area. Al K α (1486.6 eV) was used as the X-ray source in a Kratos Ultra DLD spectrometer for X-ray photoelectron spectroscopy (XPS). The thermal decomposition was characterized using a thermogravimetric analyzer (TGA) and Differential thermogravimetric analysis (DTG) (SDT Q600, USA) from room temperature to 800 °C at a heating rate of 10 °C/min under a nitrogen atmosphere (5 mL min⁻¹).

3 Results and Discussion

3.1 Morphology of the Fabricated Composites

The CNFsPPy/GO/MAF composite is fabricated via a sequential drop-casting method for non-enzymatic glucose detection in sweat. The schematic diagram in **Figure 1a** illustrates the fabrication process of the CNFs/PPy/GO/MAF composites. CNF/PPy forms a highly conductive network that facilitates rapid electron transfer. PPy nanoparticles densely coat and anchor to the CNF surface without destroying the CNFs (Figure 1b). In addition, PPy adheres tightly to the surface of CNFs (Figure S1a, b, Supporting Information), thereby altering the conductive pathways and improving the electrochemical properties. CNFs with an average diameter of 291 nm (Figure S1c), and the PPy nanoparticle size is 203 nm (Figure S1d, Supporting Information). GO nanosheets exhibit a typical crumpled, wrinkled structure with edge folding and dark lines, as clearly observed in the inset of Figure 1c.

Inset in Figure 1d shows FE-SEM and TEM images of MAF-6-Co hexagonal 3D. It clearly reveals the formation of nanocrystals with an average size of approximately 3.7 μm (Figure S1e, Supporting Information). The hydrophobic surface and its members are clearly observed in Figure S1f (Supporting Information) and have thicker walls that maintain the aperture size under electrochemical stress. Thus, selectivity doesn't degrade over repeated use, contributing to long-term stability. CNFs/PPy/GO/MAF composite casting with Nafion on a glassy carbon electrode (Figure 1e). The cross-section shows CNFs/PPy as a bridge between GO and MAF, facilitating continuous electron transfer (Figure 1f, g). XRD curves of MAF, CNFs/PPy, CNFs/PPy/GO, and CNFs/PPy/GO/MAF composites are shown in Figure 1h. The sharp, intense XRD peaks of MAF-6-CoII at (211), (220), (321), and (400) at 2θ values of 7.8, 13.2, 15.6, 18.8, and 18.0 $^\circ$, respectively, indicate high crystallinity. Broad peaks of CNFs/PPy are observed at approximately 25.7 and 42.9, which can be indexed to the (002) and (100) reflections, respectively, indicating a crystalline structure. The relatively weak peak at 43 $^\circ$ can be attributed to low crystallinity. GO shows a sharp diffraction peak at 11.2 $^\circ$, corresponding to the (001) plane. All the XRD peak positions and intensities in CNFs/PPy/GO and CNFs/PPy/GO/MAF indicate successful fabrication of the composite.

The Brunauer-Emmett-Teller (BET) specific surface area and nanoporous structure of CNFs/PPy/GO are shown in Figure 1i. The specific surface areas (SSA) of the MAF, CNFsPPy, GO, and CNFs/PPy/GO/MAF composite samples are approximately 633, 9.3, 34.9, and 153 m^2/g ,

respectively, indicating that CNFs/PPy/GO/MAF exhibits a significantly higher SSA. The high surface area and rough electrode morphology in FE-ESM (Figure 1e) provide more catalytically active sites, thereby improving the adsorption and oxidation efficiency of sweat glucose. The nitrogen adsorption-desorption isotherms of the samples are classified as typical type IV isotherms according to IUPAC standards (Figure S3a, b, Supporting Information), confirming the presence of a mesoporous structure. The 2D-NLDFT pore-size distribution from adsorption-desorption highlights both microporosity and mesoporosity in the samples. The CNFs/PPy/GO/MAF composite can control molecular diffusion through pore size and steric hindrance, thereby capturing glucose molecules and enhancing selectivity and electrocatalytic performance for the anodic glucose oxidation reaction. The size distribution, adsorption-desorption (Figure S4a-d, and Table S1, Supporting Information), which summarizes the surface area, pore volume, pore diameters, and pore size distribution, are provided. The table indicates that the SSA and total pore volume increase with the addition of MAF, thereby further enhancing the composite structure of CNFs/PPy/GO/MAF. The pore-size distribution yields a volume of $0.040 \text{ cm}^3/\text{g}$, a diameter of 3.058 nm , and a total pore volume of $0.1009 \text{ cm}^3/\text{g}$ for pores smaller than 189.1 nm at $P/P_0 = 0.98976$. The resulting pore structure is significantly larger than that in the composite without MAF.

As is well known, XPS is a surface-sensitive technique used to analyze the chemical composition and bonding at the surface. The XPS survey spectrum of the CNFs/PPY/GO/MAF composite (Figure S2a, Supporting Information) shows the presence of Co and N, along with C and O. The C 1s spectrum (Figure S2b, Supporting Information) shows two peaks at 285.2 and 283.70 eV , corresponding to hybridized graphitic carbon ($\text{C}=\text{C}$ or $\text{C}-\text{C}$) and $\text{C}=\text{N}$, respectively. The presence of N is a remnant of PAN during carbonization, as evidenced by N 1s peaks at 398.1 and 399.2 eV , corresponding to $\text{C}=\text{N}$ and $\text{C}-\text{N}$, respectively (Figure S2c, Supporting Information). O 1s binding energies are 530.6 and 532.4 eV , corresponding to $\text{C}-\text{O}$ and $\text{C}=\text{O}$, respectively, due to the atmosphere during the thermal process and the chemical structure of CNFs and GO (Figure S2d, Supporting Information). In Figure 1j, two main Co 2p peaks at 780.6 and 796 eV , separated by 15.4 eV , can be assigned to $\text{Co } 2p^{3/2}$ and $\text{Co } 2p^{1/2}$, respectively. Both $\text{Co } 2p^{3/2}$ and $\text{Co } 2p^{1/2}$ exhibit strong satellite peaks at 785 and 801.6 eV , respectively. This result suggests that the oxidation state of Co in the MAF is $+2$ [38, 48]. The EDS analyses in

Figure 1k, l clearly show peaks for C, N, O, and Co, indicating the presence of these elements with weight percentages of 71, 14.3, 8.09, and 6.61, respectively.

Figure 2a schematically illustrates the plausible mechanism for the non-enzymatic electrochemical oxidation of glucose, which occurs in two steps. First, OH^- ions interact with $\text{Co}^{\text{II}}(\text{OH})_2$ sites in the CNFs/PPy/GO/MAF, producing active $\text{Co}^{\text{II}}\text{OOH}$ species and releasing electrons and water. Concurrently, glucose molecules enter the MAF pores and adsorb onto these active sites. Second, the adsorbed glucose is oxidized to gluconolactone by $\text{Co}^{\text{III}}\text{OOH}$, which is then reduced back to $\text{Co}^{\text{II}}(\text{OH})_2$, thereby regenerating the catalyst and maintaining the $\text{Co}^{\text{II}}/\text{Co}^{\text{III}}$ redox cycle essential for continuous glucose detection. This demonstrates strong redox activity and excellent catalytic performance across all tested pH levels (13, 12.5, and 12), indicating sufficient sensitivity to pH changes and proving that this CNFs/PPy/GO/MAF composite can mitigate OH loss. This makes it suitable for non-enzymatic detection of sweat glucose. Thermogravimetric analysis (TGA) and DTG (Figure 2b, c) were used to evaluate the thermal stability of the materials. The TGA plot showed a two-step weight loss. The first step, observed in both MAF and the CNFs/PPy/GO/MAF composite, occurred between 5% and 190 °C and can be attributed to the removal of guest molecules and unreacted species [49]. The second step, above 495 °C, showed a sharper weight loss of 45% for MAF, which can be ascribed to the decomposition of MAF, and 35% for the CNFs/PPy/GO/MAF composite above 610 °C (Figure 2b). The two loss-weight values for the sample are also shown at the same position in the DTG curves for the MAF and CNFs/PPy/GO/MAF composite, as shown in Figure 2c. This indicates that CNFs/PPy/GO/MAF composites are highly stable and well suited for developing room-temperature sensors.

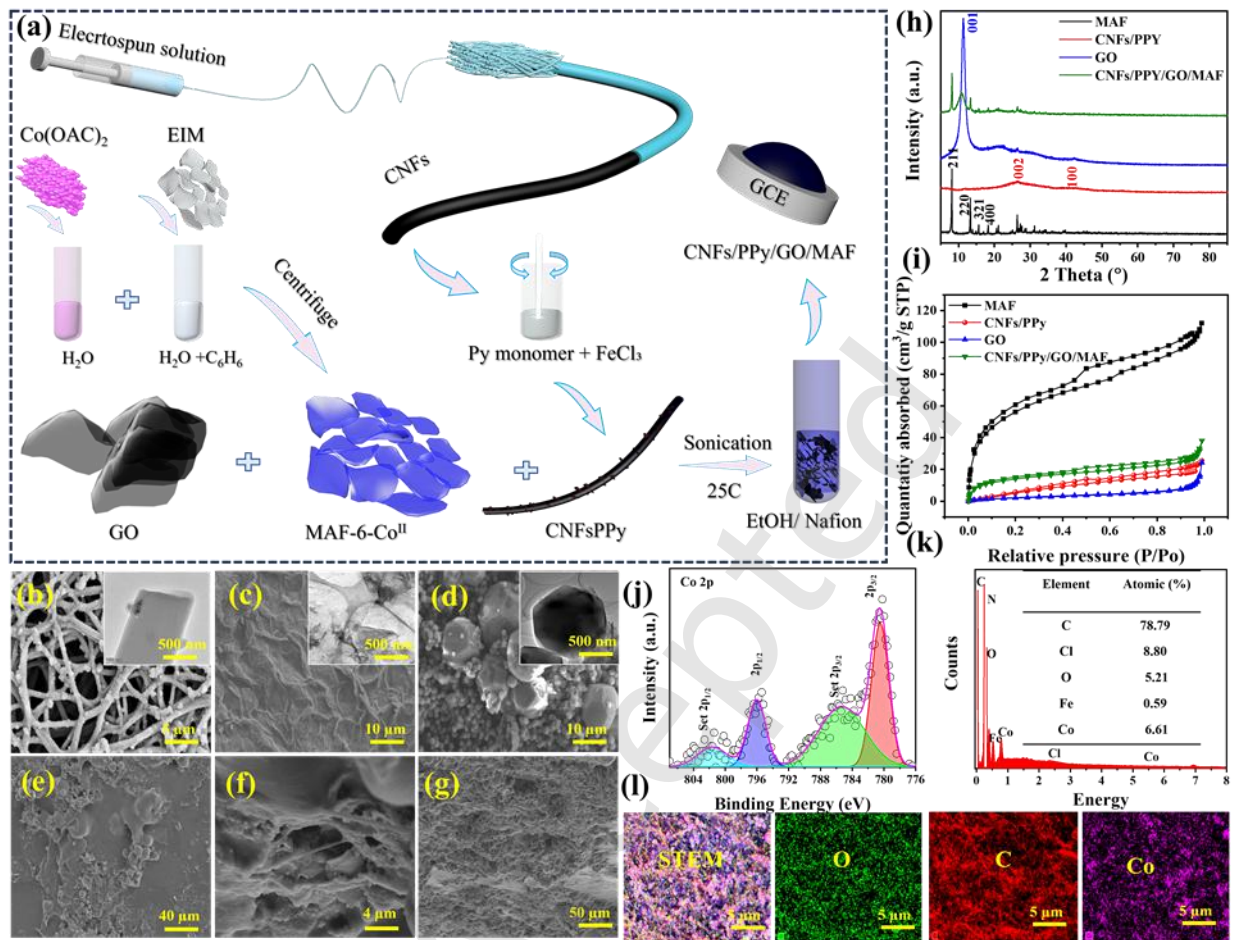


Figure 1 (a) The schematic diagram illustrates the fabrication process of the CNFs/PPy/GO/MAF composites. FE-SEM images of (b) CNFs/PPy, (c) GO, (d) MAF. The inset shows HRTEM. (e-g) CNFs/PPy/GO/MAF composites, magnified image, and cross-section. (h) The XRD pattern of the CNFs/PPy/GO/MAF composite. (i) Nitrogen adsorption and desorption isotherms, (j) high-resolution Co 2p spectrum OF Co 2p in MAF, and (k, l) EDS profile.

3.2 Electrochemical measurements

All electrochemical tests were conducted using a CHI850D workstation (Shanghai, China) at 298 K. CNFs/PPy/GO/MAF composites served as the working electrode (WE), Ag/AgCl as the reference electrode (RE), and a platinum wire as the counter electrode (CE). The electrochemical performance of glucose on the electrode was investigated via cyclic voltammetry (CV) over a potential range of -0.2 to 0.6 V, electrochemical impedance spectroscopy (EIS) was performed in a solution of 0.1 M KCl and 5 mM [Fe(CN)₆]^{3-/4-}, with the frequency range set from 100 kHz to 0.1 Hz and a distribution amplitude of 5 mV, and Amperometric (i-t curve) measurements were

performed to analyze the electrocatalytic properties by continuously adding glucose to alkaline media using 0.1 M NaOH (pH 13), 0.0316 M NaOH (pH 12.5), and 0.01 M NaOH (pH 12). The pH was set using a pH meter (pH100B) with an accuracy of 0.01. Measurements were conducted at the optimal potential of 0.45 V under continuous stirring at 200 rpm.

3.2.1 Cyclic Voltammetry Analysis

The charge-transfer kinetics of CNFs/PPy, CNFs/PPy/GO, and CNFs/PPy/GO/MAF were investigated by CV and EIS, as shown in Figure S5a, b, Supporting Information. CNFs/PPy exhibits a featureless, capacitive-like voltammogram in the $[\text{Fe}(\text{CN})_6]^{3-/4-}$ system, lacking the characteristic oxidation and reduction peaks of the redox probe [50]. The oxidation peak current (I_{pa}) at CNFs/PPy/GO is 70.4 μA , with a peak potential separation (ΔE_p) of 0.271 V. Incorporating GO prevents agglomeration of CNFs/PPy chains, forming an ordered porous network that improves electrolyte penetration and probe accessibility. Additionally, GO sheets act as conductive bridges between CNFs and PPy, reducing charge-transfer resistance and enabling a rapid faradaic response. The I_{pa} in the CNFs/PPy/GO/MAF is 128 μA , with a ΔE_p of 0.416 V, indicating high electrocatalytic activity, an electroactive surface area of 0.1546 cm^2 (calculated details in the supporting information), and a highly porous, high-surface-area architecture that maximizes the number of sites for faster electron-transfer kinetics and enhanced sweat glucose detection. These CV behaviors of CNFs/PPy, CNFs/PPy/GO, and CNFs/PPy/GO/MAF are highly consistent with the EIS spectra (Figure S5b, Supporting Information), with charge-transfer resistance (R_{ct}) at the electrode-electrolyte interface of 46, 36, and 58 Ω , respectively.

Figure S6 in the Supporting Information displays CVs for CNFs/PPy, CNFs/PPy/GO, MAF, and CNFs/PPy/GO/MAF recorded in 0.1 M NaOH (pH = 13) at a scan rate of 45 mV/s, both without and with 0.1 mM glucose. As shown, negligible currents are recorded for both CNFs/PPy and CNFs/PPy/GO. No process due to glucose Oxidation is observed over the potential range examined here. In the MAF's CV with the absence of glucose, anodic and cathodic features are observed at potentials anodic to 0.3 V, likely due to the $\text{Co}^{2+}/\text{Co}^{3+}$ redox couple of the nanostructured material. In the presence of glucose, oxidation begins at about 0.35 V, with a current density of 630 $\mu\text{A}/\text{cm}^2$. These results clearly indicate the high electrocatalytic activity of MAF toward the oxidation of glucose to gluconolactone.

CNFs/PPy/GO/MAF shifts the potential anodically to 0.4 V. Upon addition of glucose, the shift extends to 0.41 V, with an oxidation peak current density of 1000 $\mu\text{A}/\text{cm}^2$. This improvement is attributed to the composite components CNFs/PPy/GO/MAF, CNFs/PPy, which amplify the signal, and GO, which increases the active site density. CVs of CNFs/PPy/GO/MAF were measured at different pH levels (13, 12.5, 12) in the absence and presence of 0.1 mM glucose (Figure 2d, g, j). The CNFs/PPy/GO/MAF displayed clear anodic peak potentials (E_{pa}) and cathodic potentials (E_{pc}) at pH 13 (Figure 2d), which were 0.41 V and 0.15 V, respectively. Consequently, the E_{pa} were 0.42 and 0.43 at pH 12.5 and 12 (Figure 2g, j), respectively. The E_{pc} values were 0.25 and 0.3 at pH 12.5 and 12 (Figure 2g, j), respectively. The oxidation peak current density decreases with decreasing pH, to 65% at pH 12.5 and 36% at pH = 12, relative to pH = 13, due to reduced OH^- availability. This decrease in oxidation peak current density remains acceptable (pH = 12.5: 577 $\mu\text{A}/\text{cm}^2$; pH = 12: 315 $\mu\text{A}/\text{cm}^2$) and compensates for the mild acidity of sweat, ensuring stable and Sensitive detection. CVs were performed at scan rates from 10 to 100 mV/s. The redox peak current density of CNFs/PPy/GO/MAF increases with increasing scan rate (Figure 2e, h, k) at pH levels of 13, 12.5, and 12, respectively. The E_{pa} shifts more positively, and the E_{pc} shifts more negatively, as the scan rate increases, which proposed a quasi-reversible electrochemical reaction mechanism for the CNFs/PPy/GO/MAF composite on the electrode surface at shared activation sites [51]. The oxidation peak current density for glucose increased linearly with the square root of the scan rate at pH = 13, 12.5, and 12 (Figure 2f, i, l). The calibration graphs demonstrated excellent linearity, with R^2 values of 0.999 at all three pH levels, confirming that glucose oxidation is diffusion-controlled [52].

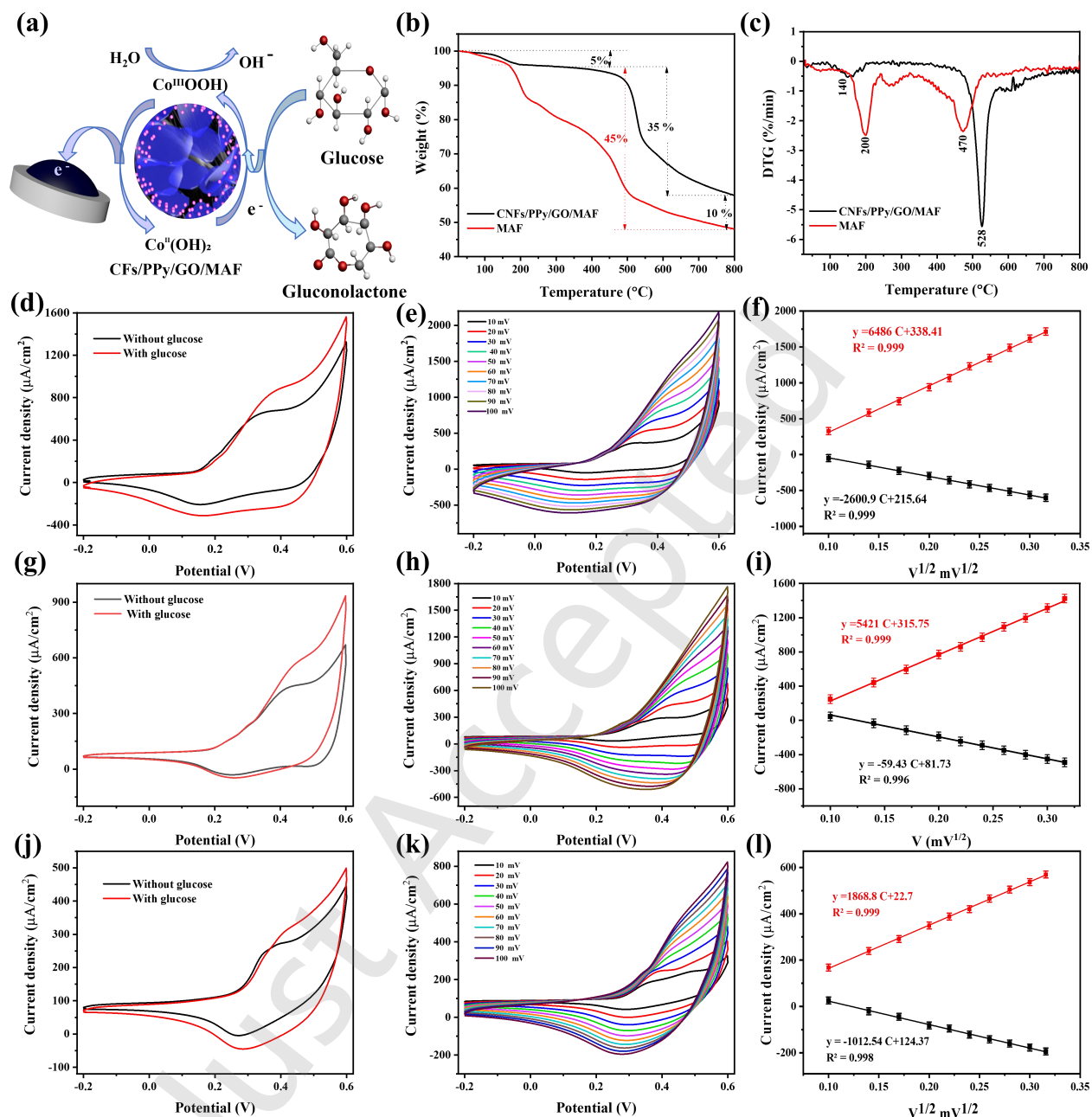


Figure 2 (a) Schematic of a plausible mechanism for the oxidation of glucose at CNFs/PPy/GO/MAF composites. (b, c) TGA and DTG curves. (d, g, j) CVs of CNFs/PPy/GO/MAF at pH = 13, 12.5, and 12 at a scan rate of 45 mV. (e, h, k) CVs at different scan rates, and (f, i, l) their calibration curves.

3.2.2 Amperometry i-t curve Analysis

An amperometric i-t curve was used to investigate the effect of glucose concentration on current density. In this technique, the applied potential significantly affects the method's sensitivity. Initially, the optimal potential was determined at pH = 13, 12.5, and 12 (**Figure 3a ~**

c), respectively, using NaOH solutions containing 0.03 ~ 0.36 mM glucose. The explored potential range was 0.35 ~ 0.45 V. The figure displays typical current-time profiles under different conditions, showing steady-state currents at various potentials. When pH is 13, the steps size gradually increases from 0.35 to 0.45 V as glucose is continuously added. At 0.35 V, the smaller steps lead to decreased sensitivity of the CNFs/PPy/GO/MAF composite. At 0.4 V, the steady-state current density is lower compared to 0.45 V, where the current is higher. Similar studies on related materials report potentials above 0.45 V [38]. Higher applied potentials could affect the sensor's selectivity due to the presence of other oxidizing agents. Therefore, 0.45 V shows the best performance across all three pH levels and is selected as the detection potential for subsequent measurements.

At the same applied potential (pH = 12.5 and 12), the current density is lower than at pH = 13. This decrease in current density, caused by reduced OH⁻ in the electrolyte, remains acceptable for glucose detection in sweat. The calibration curves corresponding to each potential exhibit high linear coefficients (Figure 3d ~ f). At pH = 13, the extent of the step gradually increases to match the further rise in current density. (Figure 3g) is also shown in Figure 3j, confirming a linear relationship between variations in current density and glucose concentration. Two linear regions identified in the calibration plots for glucose oxidation are attributed to the buildup of gluconolactone, the oxidation product, within the material's porous structure. At lower glucose levels, the reaction is diffusion-controlled; molecules diffuse into the CNFs/PPy/GO/MAF composite and are rapidly oxidized at abundant active sites, producing current responses with a high slope in the calibration plot ranging from 0.03 to 1.60 mM. In this measurement, humans sweat a maximum of 1 ~ 200 μ M glucose [53]. The sensitivity, response time, and limit of detection (*LOD*) were calculated using the formula: $LOD = 3.3 \times (\sigma/S)$. In this formula, σ represents the standard error of the intercept, and *S* is the slope of the fitted linear model curve [54], which were determined to be 663 μ A/(mM·cm²), $R^2=0.996$, 3 sec, and 0.033 μ M, respectively. On the other hand, at higher glucose levels, the process shifts to being adsorption-controlled. The buildup of adsorbed oxidation products can form a diffusion barrier, hindering fresh glucose from reaching the active sites and resulting in a lower slope in the current response while maintaining linearity [39]. The sensitivity and R^2 are 53.8 μ A/(mM·cm²) and 0.995, respectively, and over a range of 1.60 ~ 16.3 mM. Although sensor sensitivity slightly decreases at pH 12.5 and 12 (Figure 3h, i) during calibration (Figure 3k, l) due to hydroxide ions (OH⁻), it

remains highly responsive, with values of 259.6 and 6.08 $\mu\text{A}/(\text{mM}\cdot\text{cm}^2)$ across linear ranges of 0.003 to 1.4 mM and 1.4 to 10.9 mM at pH = 12.5. Additionally, sensitivities of 160.85 and 3.27 $\mu\text{A}/(\text{mM}\cdot\text{cm}^2)$ are observed over the ranges of 0.003 ~ 0.7 mM and 0.7 ~ 7.08 mM at pH = 12. However, the CNFs/PPy/GO composite's unique porous structure, characterized by a high surface area and mesopores and nanopores, remains stable. The GO and PPy functional groups help buffer local pH fluctuations and facilitate the transport of OH. This self-compensating mechanism preserves the alkaline microenvironment necessary for effective glucose oxidation, enabling the sensor to maintain high sensitivity and a consistent detection range even in the slightly acidic conditions of sweat. Table S2 in the Supporting Information displays the *LOD* sensitivity, response time, and linear range. The data suggest that the glucose sensor made from the CNFs/PPy/GO/MAF composite performs notably well at low glucose concentrations at pH = 13, 12.5, and 12, compared to all other previously reported sensors. These features improve surface reaction kinetics and lower charge-transfer resistance, resulting in faster electron transfer.

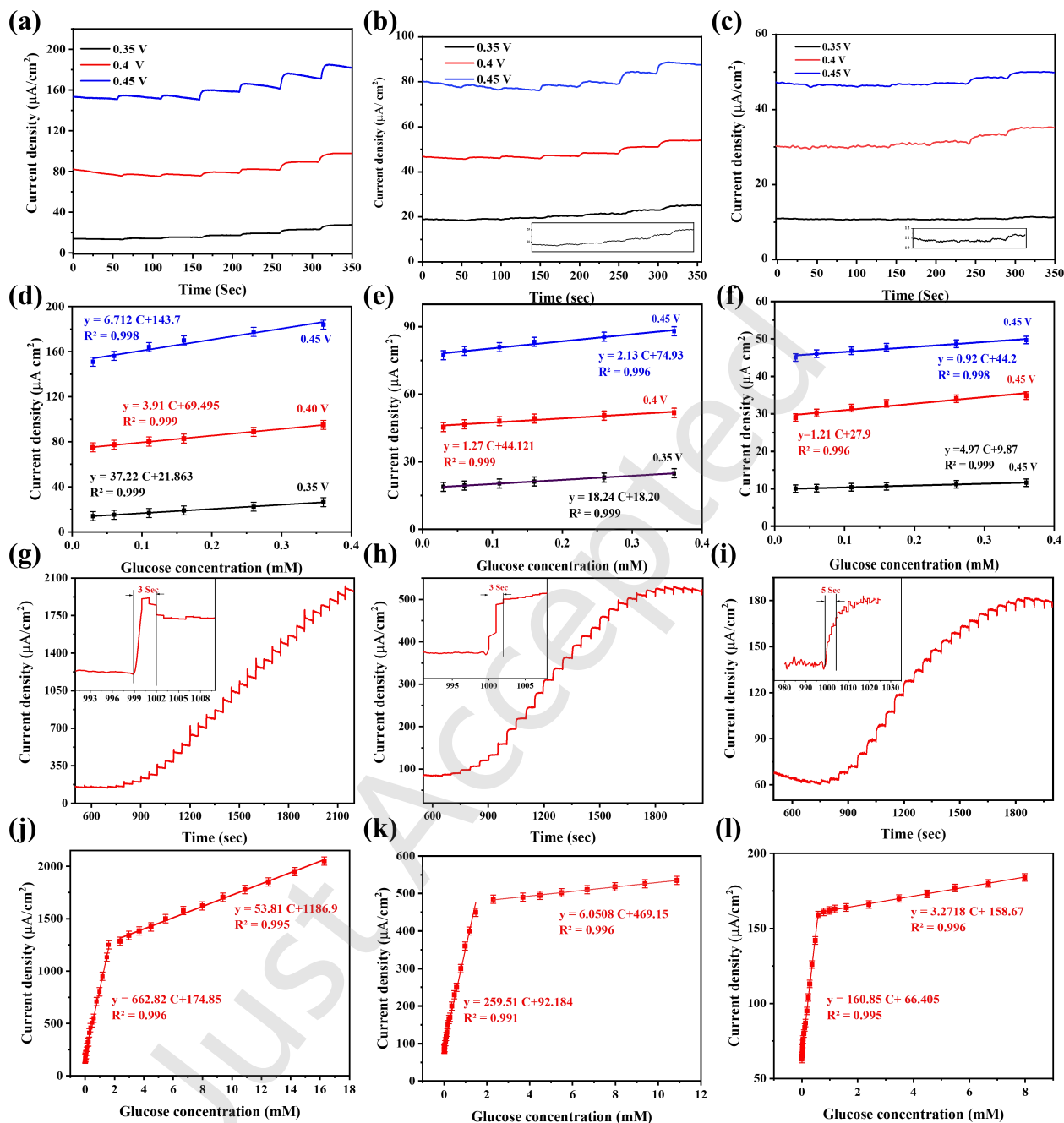


Figure 3 (a ~ c) Amperometric response of the CNFs/PPy/GO/MAF composite to the addition of glucose solutions at 0.03, 0.05, and 0.1 mM and different potentials, with insets showing magnified responses at 0.35 V with pH = 13, 12.5, and 12, respectively, and (d ~ f) corresponding calibration curve for each potential. Amperometric response of the CNFs/PPy/GO/MAF composite upon successive addition of 1 mM glucose at 0.45 V over a glucose concentration range of 0.003 ~ 16.3 mM. (g ~ i) The insets show the response times for

pH = 13, 12.5, and 12, respectively. (j ~ l) The relationships between the steady-state current and glucose concentration at pH = 13, 12.5, and 12.

3.2.3 Stability, Selectivity, Repeatability, and Reproducibility

Operational stability was assessed through consecutive CV (Figure S7a-c, Supporting Information) and stability percentage (Figure S7d, Supporting Information) at pH 13, 12.5, and 12, spiked with 0.1 mM glucose. It was observed that CNFs/PPy/GO/MAF, after 25 cycles, exhibited a decrease in glucose oxidation currents by 3.65%, 6.15%, and 7.46% at pH 13, 12.5, and 12, respectively, indicating that the CNFs/PPy/GO/MAF composite is suitably stable for sweat glucose analysis.

Sweat contains various interferents in addition to glucose, such as sodium chloride (NaCl), lactic acid (LA), uric acid (UA), and ascorbic acid (AA). To evaluate the effect of common interfering substances on the CNFs/PPY/GO/MAF for sweat glucose detection at pH 13, 12.5, and 12, respectively. **Figure 4a ~ c** shows tests at 0.45 V with 0.1 mM glucose and several interferents: NaCl (10 μ M), UA (50 μ M), AA (50 μ M), and LA (100 μ M). At pH = 13 (Figure 4a), the response currents of these interferents are very small compared to the equal concentration of glucose. At pH = 12.5 and 12 (see Figure 4b, c), respectively, the glucose current response is lower than at pH = 13, but it still maintains selectivity. In contrast, in pH = 13, 12.5, and 12, the CNFs/PPY/GO/MAF composite again exhibits a large response current upon glucose introduction, matching the response observed with the initial glucose addition. The selectivity percentage of the glucose response relative to interference is shown in Figure 4d at pH = 13. The interference percentages for NaCl, UA, AA, and LA are 0.78%, 4.1%, 13%, and 7.8%, respectively. These are compared with the current glucose response, which, along with the responses at pH = 12.5 and 12, is evaluated in the presence of interference in pH 12.5 NaCl, UA, AA, and LA, at levels of 2%, 1.7%, 6.8%, and 4.7%, respectively. At pH = 12, NaCl, UA, AA, and LA show interference levels of 3.1%, 3.5%, 9.6%, and 6.4%, respectively. This demonstrates that CNFs/PPY/GO/MAF exhibit strong selectivity for glucose detection, even at lower pH. This is due to the synergistic effect of their mesopores and nanopores, which enhance selective molecular recognition and transport while reducing interference from other species, confirming their robust anti-interference abilities.

The reproducibility of the CNFs/PPY/GO/MAF was tested using four independent electrodes; the amperometric responses at pH = 13, 12.5, and 12 are shown in Figure 4e ~ g across glucose

concentrations of 0.05, 0.1, and 0.2 mM. As glucose concentration increased stepwise, all electrodes displayed consistent, gradual changes in signal. Although the response magnitude at pH = 12.5 and 12 was lower than at pH = 13 due to decreased hydroxide-ion-assisted glucose oxidation kinetics, reproducibility remained satisfactory. The reproducibility percentage is shown in Figure 4h, with relative standard deviations (RSDs) for the oxidation current at 0.2 mM glucose of approximately 5.45%, 5.75%, and 8.4% at pH 13, 12.5, and 12, respectively.

The repeatability of CNFs/PPy/GO/MAF was assessed by testing a single modified electrode six times under identical conditions over a 35-day period. The amperometric responses for 0.1 mM glucose oxidation, where the current gradually decreases at pH = 13, 12.5, and 12, as shown in Figure 4i ~ k, are used to determine the repeatability percentages shown in Figure 4l. The RSD values from day 1 to day 35 are 1.75%, 3.2%, and 2.20% at pH 13, 12.5, and 12, respectively, confirming the CNFs/PPy/GO/MAF composite's potential for continuous monitoring of sweat glucose.

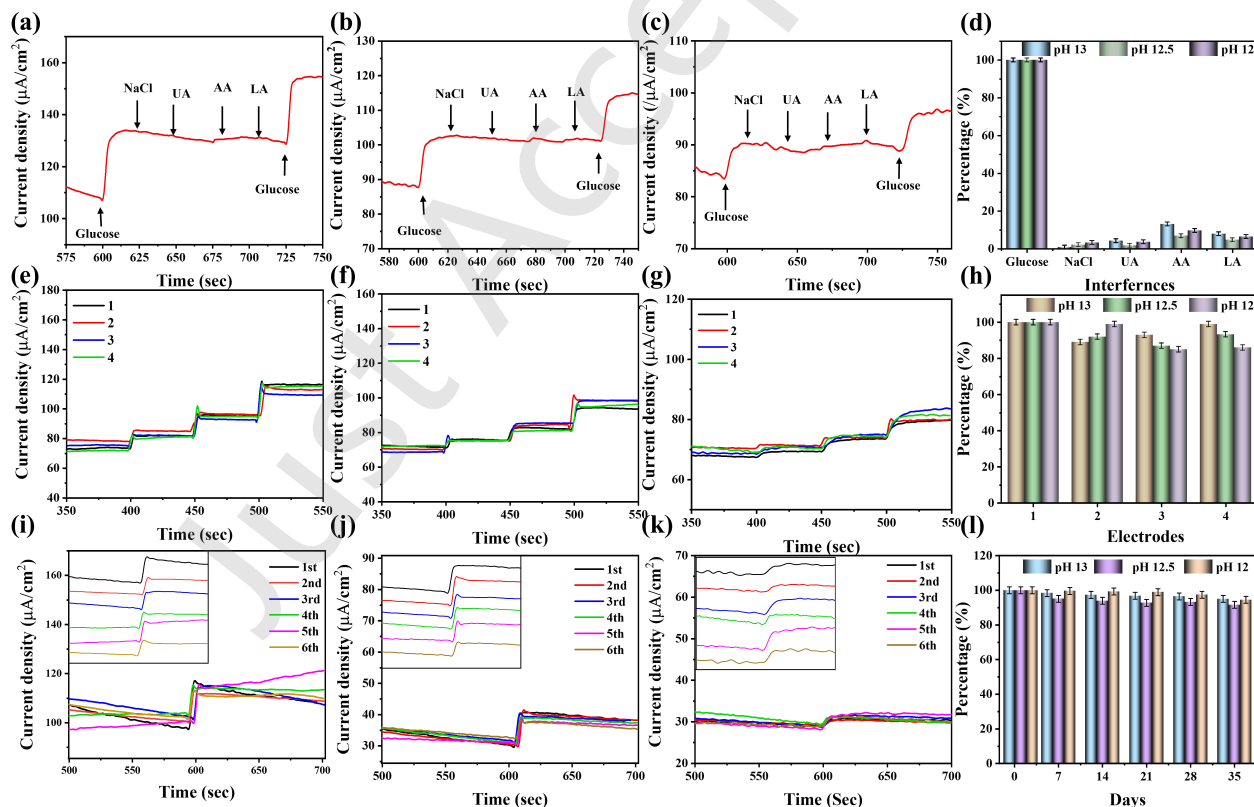


Figure 4 (a ~ c) The selectivity at pH = 13, 12.5, and 12 is shown by current responses in the presence of common interfering species, respectively. (d) The selectivity percentages, (e ~ g) the reproducibility at pH = 13, 12.5, and 12, respectively. (h) Reproducibility percentages. (i ~ k)

The repeatability of the CNFs/PPy/GO/MAF composite at pH = 13, 12.5, and 12, respectively, and (l) Repeatability percentages.

3.3 Detection of glucose in sweat

Patients with diabetes are typically instructed to monitor their blood glucose levels at designated times. According to the American Diabetes Association (ADA) guidelines [55]. This includes measuring fasting levels (after an 8 ~12 hour overnight fast with no food or drink) and checking levels at 1 and 2 hours after a meal. A commercial glucometer was used to measure blood glucose levels during sweat sample collection in healthy volunteers without diabetes. Sweat produced through physical activity can help compensate for low sample volume during rest. Therefore, sweat was collected from the forehead and arms during various physical activities, including walking, moderate exercise, and running. Sweat samples were labeled as fasting, one hour after meals, and two hours after meals. Our composites were applied to the working electrode surface of a flexible, wearable SPE. This platform is ideal for non-enzymatic wearable sweat glucose sensors due to its compact size, enabling precise glucose detection from minimal sweat samples. Because non-enzymatic detection requires an alkaline medium, a 10 μ l drop of NaOH (at concentrations of 0.1, 0.0316, and 0.01 M) was applied to the sensing area, establishing an alkaline environment at pH 13, 12.5, and 12. The electrode was then dried at room temperature, after which 10 μ l of each sweat sample was applied to the same area, as shown in **Figure 5a**. During real sweat testing, the CNFs/PPy/GO/MAF-modified SPE with a dried NaOH layer maintains a stable baseline current. When sweat is applied dropwise, it rapidly rehydrates the solid alkaline layer, immediately activating the sensor and producing a strong amperometric signal. The current then quickly stabilizes to a steady state, demonstrating the sensor's effective design and dependable operation under complete testing conditions. As shown in Figure 5b ~ d, the steady-state current response increased from fasting to one hour after a meal, then decreased again over the next two hours after a meal across pH of 13, 12.5 and 12.

Furthermore, sweat glucose levels (Figure 5e) were measured in parallel (during fasting, one hour after a meal, and two hours after a meal) on the same volunteer using a commercial blood glucometer. From the calibration curves (Figure 3(j ~ l)), the sensitivity (high slope) is used to calculate real-sweat glucose concentration, which recorded as (41, 87, and 64 μ M) at pH = 13 (Figure 5eII), (44, 85, and 65 μ M) at pH = 12.5 (Figure 5eIII), and (43, 88, and 67 μ M) at pH = 12 (Figure 5eIV), respectively, in agreement with the literature [56, 57]. This indicates an

increase in glucose concentration in sweat, which then decreases two hours after meals again. These values are well matched to blood glucose levels measured by the glucometer (Figure 5e). This agreement confirms that the CNFs/PPy/GO/MAF composite provides an accurate, reliable, real-time, non-maintained, stable, and reproducible current signal across all tested conditions. This reliable performance, even at the lower alkalinity typical of physiological variations in sweat, yields high sensitivity, excellent repeatability, and strong pH tolerance.

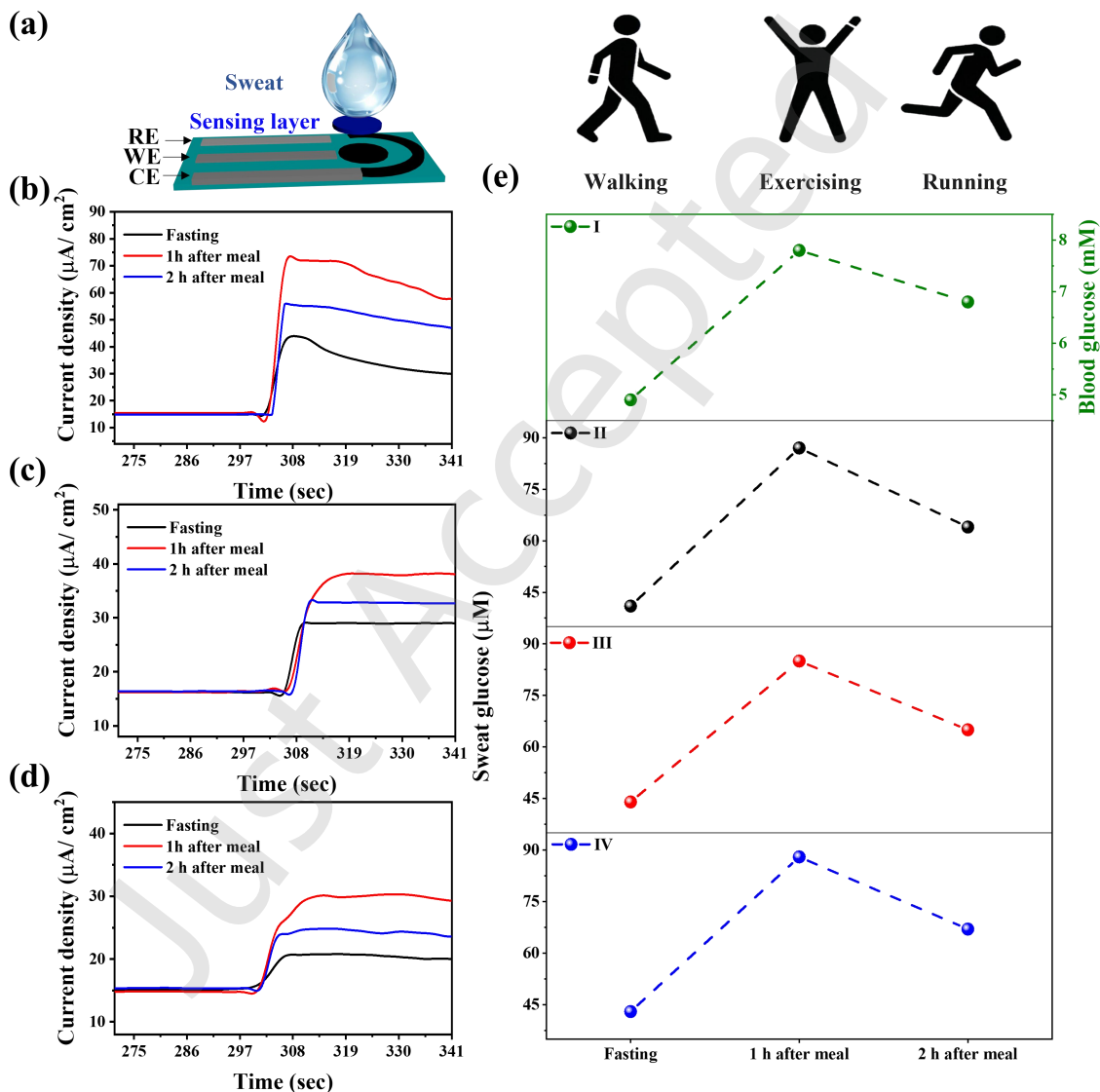


Figure 5 (a) SPE. Amperometric i-t curve for real sweat detection for (b-d) CNFs/PPy/GO/MAF composite using volunteers' sweat at pH = 13, 12.5, and 12, respectively. (e) The response current density of the volunteers' sweat at pH = 13, 12.5, and 12 is shown in (II -IV), respectively, using a CNFs/PPy/GO/MAF composite sensor, and is compared with the volunteers' blood glucose concentrations (I), measured with a commercial glucometer.

4 Conclusions

The findings indicate that the CNFs/PPy/GO/MAF composite performs effectively at pH = 13, 12.5, and 12, which are critical for non-enzymatic glucose detection in alkaline conditions. This composite was fabricated using a sequential drop-casting method, enabling precise assembly of a layered structure comprising a CNFs/PPy conductive layer, GO to enhance electron transfer, and an MAF top layer rich in catalytic sites. The composite maintains high sensitivity, selectivity, a wide linear range, and a low detection limit, even at lower pH levels. It also displays excellent stability across these pH values, with glucose oxidation current decreases of 3.65%, 6.15%, and 7.46%, respectively. Reproducibility tests showed RSDs of 5.45%, 5.75%, and 8.4%, while over 35 days, repeatability resulted in RSDs of 1.75%, 3.2%, and 2.20%. The composite at pH = 13, 12.5, and 12 correlated well with blood glucose levels read by the glucometer. This consistency confirms that the composite provides precise, reliable, real-time, and non-invasive glucose measurements in common alkaline environments.

Supporting Information: The Supporting Information related to this article can be found in the online version at <http://doi.org/10.26599/NR.2026.94909116>.

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Data availability

Data will be made available on request.

Use of AI statement

None

Reference

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Electronic Supplementary Material

OH-compensating non-enzymatic glucose sensor based on a hexagonal metal azolate framework composite for continuous sweat monitoring

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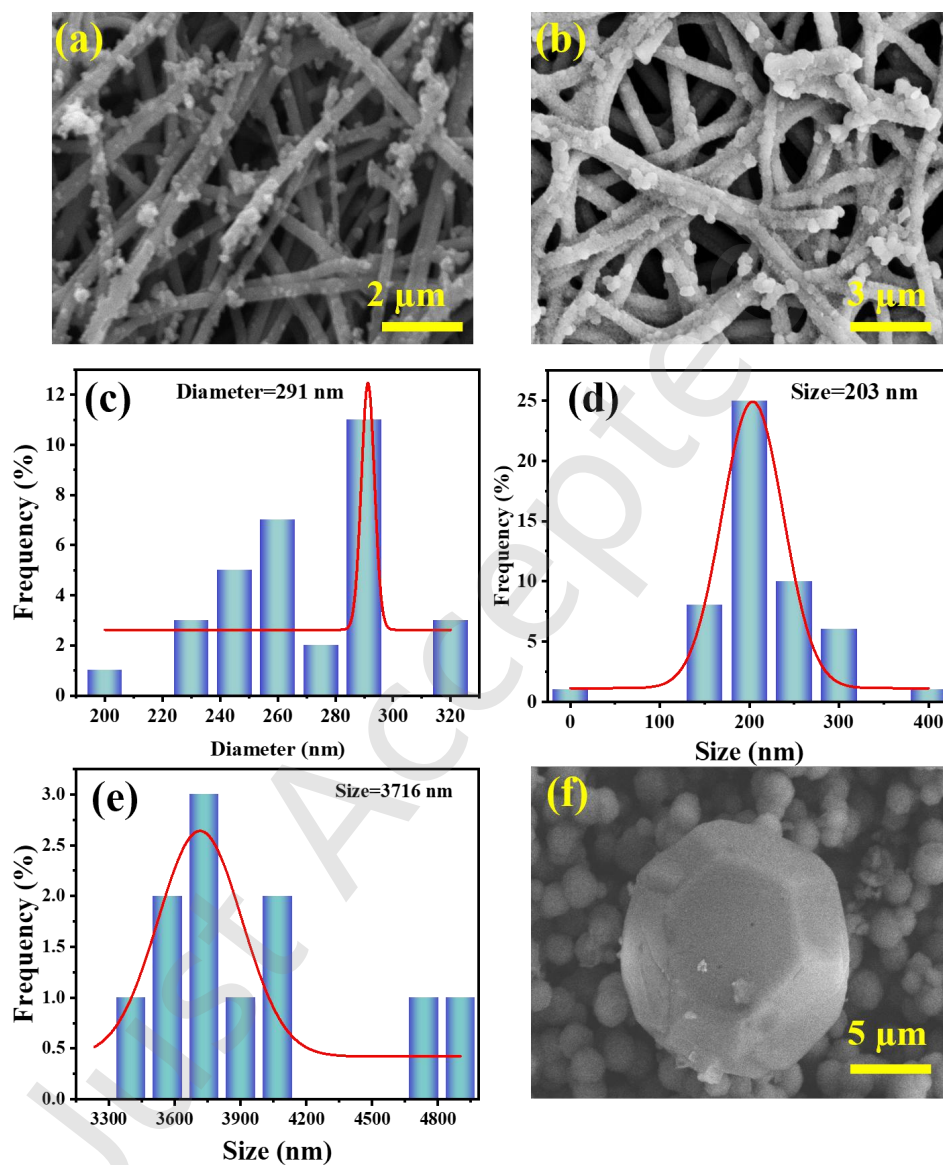


Figure S1 FE-SEM images show (a, b) CNFs/PPy and a magnified image, (c) Statistical histograms of the fiber diameters of CNFs, (d) surface size of PPy on the CNFs, (e) particle size of MAF, and (f) a hexagonal MAF image.

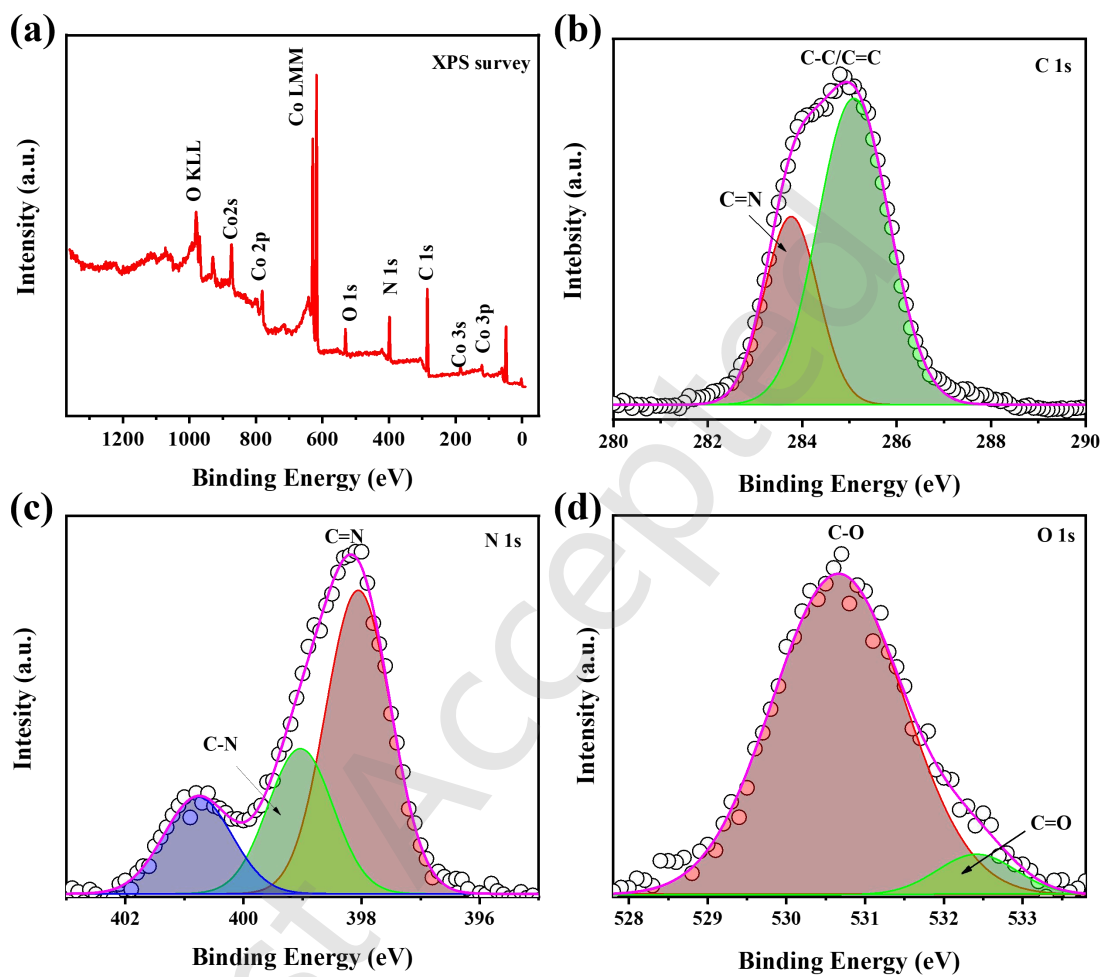


Figure S2 XPS of composites (a) XPS survey spectrum of CNFsPPy/Go/MAF (b-d) high-resolution C 1s, N 1s and O 1s spectrum.

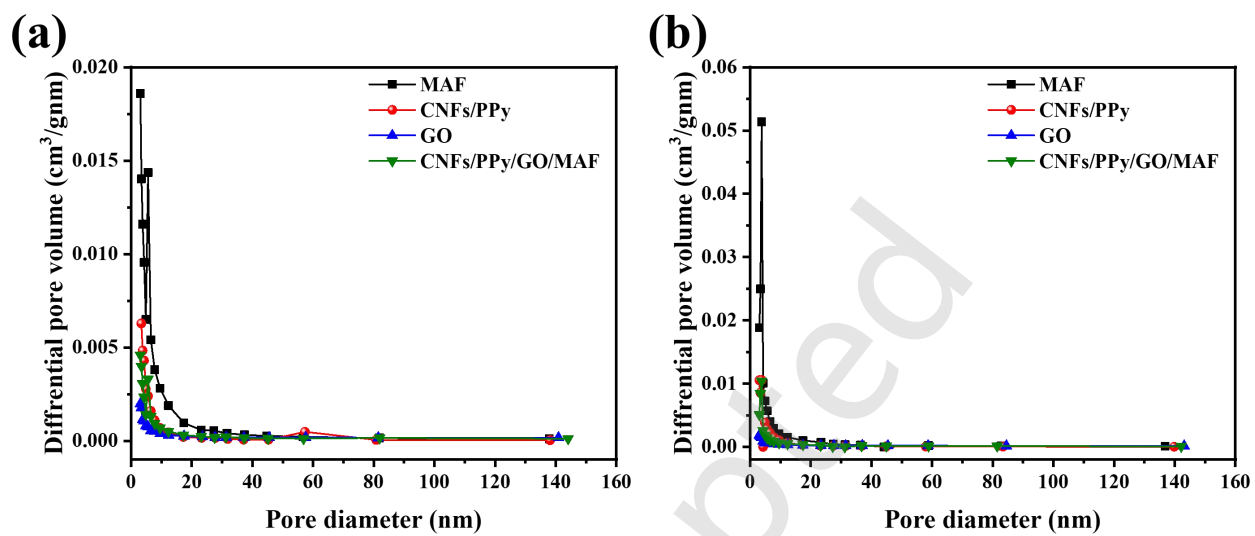


Figure S3 2D-NLDFT pore size distribution desorption, 2D-NLDFT pore size distribution adsorption

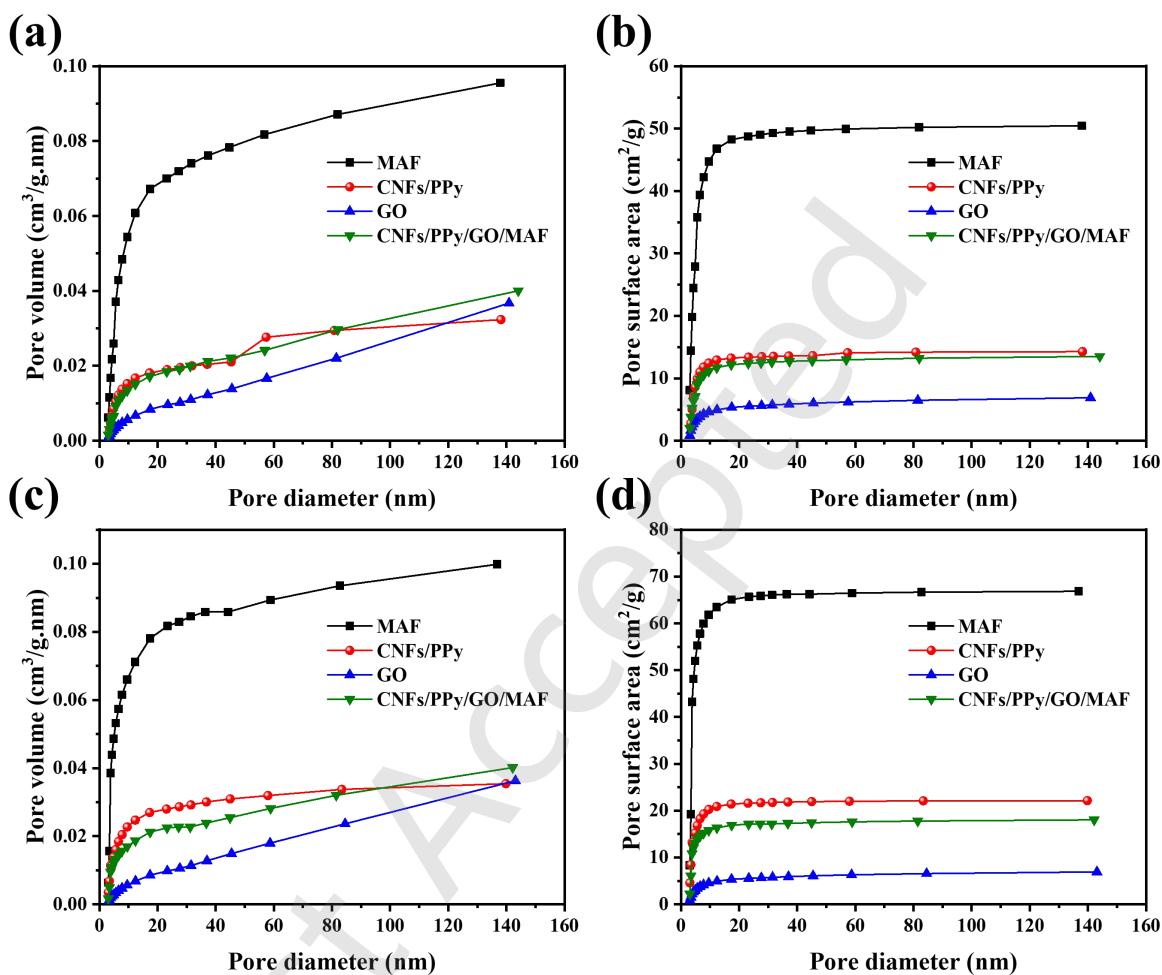


Figure S4 (a, b) Barrett-Joyner-Halenda (BJH) Pore Size distribution adsorption, (c, d) BJH Pore size distribution desorption

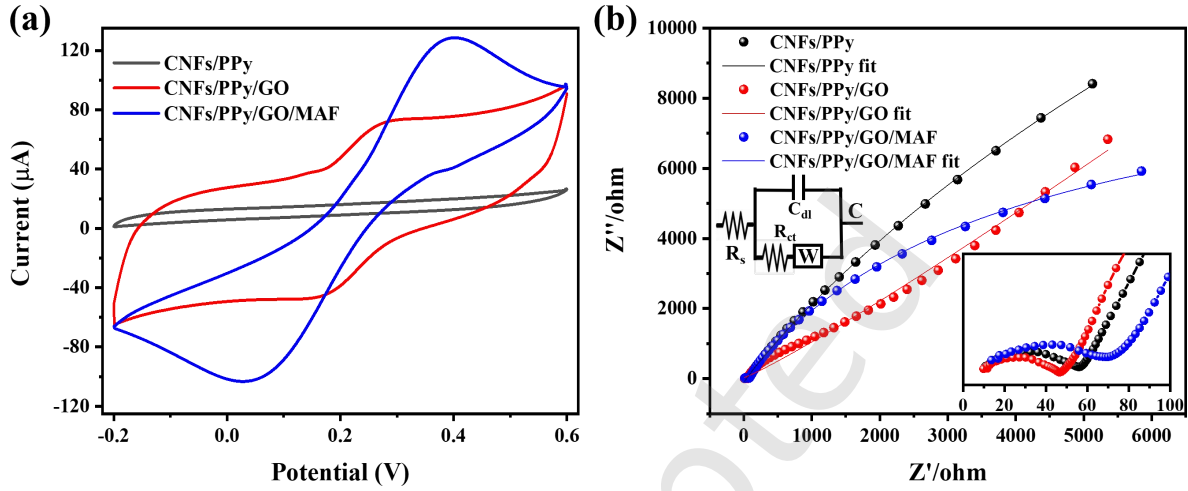


Figure S5 CVs and EIS spectra of CNFs/PPy, CNFs/PPy / GO, and CNFs/PPy / GO /MAF in a 5 mM $K_3[Fe(CN)_6]$ $^{3-/4-}$ solution and 0.1M KCl at a scan rate of 45 mV. (a) The CV shows the Randles equivalent for CNFs/PPy/GO/MAF, and (b) EIS spectra (inset shows the circuit model comprising solution resistance (R_s), Charge Transfer Resistance (R_{ct}), Warburg diffusion resistance (W), and double-layer capacitance (C_{dl})) of CNFs/PPy/GO/MAF.

The electroactive surface area (A_{eff}) of CNFs/PPy / GO /MAF composite was calculated according to the following Randles-Sevcik equation [1].

$$i_{pa} = (2.69 \times 10^5) n^{3/2} D^{1/2} C A_{eff} v^{1/2}$$

where, i_{pa} is the oxidation peak current (A), ($n = 1$) is the number of electron transferred, A_{eff} is the surface effective area of the electrode (cm^2), D is the diffusion coefficient of ferricyanide, which was considered to be $7.60 \times 10^{-6} cm^2/s$ [2], C is the concentration of redox couple (mol/cm^3), and v is the scan rate in V/s.

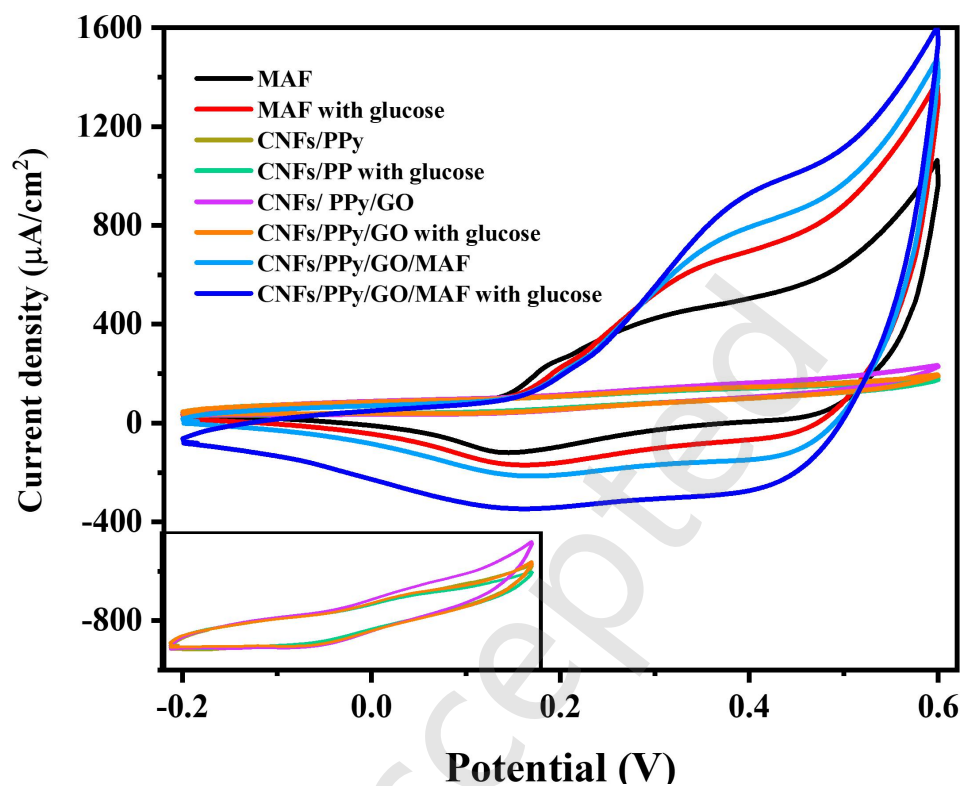


Figure S6 CVs of CNFs/PPy, CNFs/PPy/GO, MAF, and CNFs/PPy/GO/MAF in 0.1M NaOH (pH 13) before and after 0.1mM glucose.

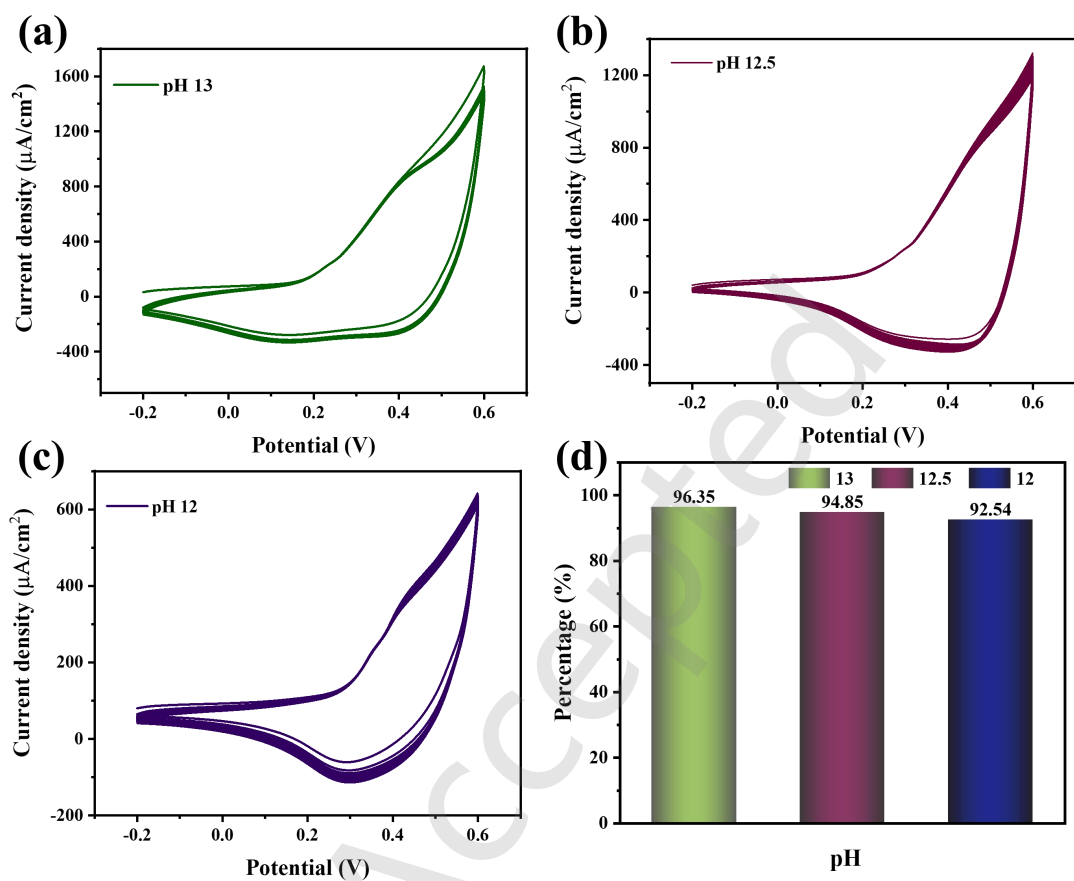


Figure S7 (a-c) Cyclic voltammetry of CNFs/PPy/GO/MAF over 25 cycles to evaluate stability at pH 13, 12.5, and 12 with 0.1 mM glucose, and (d) stability percentage.

Table S1 Structural parameters of composite CNFs/PPy/GO/MAF nanofiber

Samples		Surface Area (m²/g)	Pore Volume (cm³/g)	Pore Diameter (nm)	Total pore volume (cc/g)
MAF	BJH adsorption summary	50.446	0.096	3.058	3.480e-01 pores smaller 176.5 nm (Diameter) at P/Po = 0.98901
	BJH desorption summary	66.854	0.100	3.821	
CNFs/PPy	BJH adsorption summary	14.270	0.032	3.410	4.077e-02 pores smaller than 178.8 nm (Diameter) at P/Po = 0.98915
	BJH desorption summary	22.135	0.035	3.827	
GO	BJH adsorption summary	6.894	0.037	3.053	3.807e-02 pores smaller than 183.8 nm (Diameter) at P/Po = 0.98945
	BJH desorption summary	6.929	0.036	3.65	
CNFs/PPy/GO/MAF	BJH adsorption summary	13.521	0.040	3.058	1.009e-01 pores smaller than 189.1 nm (Diameter) at P/Po = 0.98976
	BJH desorption summary	18.032	0.04	3.830	

Table S2 Analytical performance comparison in the low-range glucose concentration of the CNFs/PPy/GO/MAF composite sweat glucose sensor at pH 13, 12.5, and 12 with those reported previously.

Electrode material	Sensitivity ($\mu\text{AmM}^{-1}\text{cm}^{-2}$)	linear range (mM)	Limit of detection (μM)	pH	Response time (Sec)	long- term	Ref.
CNFs/PPy/GO/MAF	663	0.003-1.6	0.033	13	3	35 days	This work
	53.81	1.6-16.3				98.25%	
	259.6	0.003-1.4	0.043	12.5		35 days	
	6.08	1.4-10.9				96.8%	
	160.85	0.003-0.7	0.0145	12	5	35 days	
	3.27	0.7-7.08				97.8%	
MAF-5-CoII NS	66.50	0.0039-0.125	0.09	13	-	21 days	[2]
	1012.70	0.125-12				95.8%	
Ni/Co-MOF	159.9	0.01-1	2		3.5	11 days	[3]
CFCo2P	409.8	0.0394-0.15	0.97		0.8	-	[4]
ZnO/NiCoO Composite	139.92	0.3-0.075	1.5		1.9	-	[5]
Co@NCTs-800	141.3	0.1-2	2.3		-	-	[6]
NiO-Co3O4/rGO	345	0.00013-10	0.13		-	-	[7]
NiCoFe-LDH/GO	196.18	0.005-7.54	0.41		3	90 days	[8]
CNF@Ni-Co	147	0.001-2	0.03		-	20 days	[9]
NiCo/CNFs	141	0.1-10	20		1.9	-	[10]
Ni2CoS4-CNF	6.201	0.005-	0.00025		3	21 days	[11]

		0.070				84.0%	
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