# ELECTROCHEMICAL SENSOR FOR SWEAT MONITORING

SENSOR ELETROQUÍMICO PARA MONITORAMENTO DE SUOR

# SENSOR ELECTROQUÍMICO PARA EL CONTROL DEL SUDOR

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## ABSTRACT

Introduction: Attention is given to developing electrochemical sensors for the rapid and real-time measurement of lactate levels. The synthesis of electrochemical sensors is based on an electrode modified with a nanocomposite. Objective: Analyze an electrochemical sensor's feasibility for sports monitoring sweat in lactate. The Au@CNTs were the main focus of this study. Methods: The Au@CNTs composite was synthesized on the GCE surface and tested under pre-established protocols as a sensor. Results: The shape and structure of the modified electrodes were analyzed using SEM. The results showed that the Au@CNTs nanoparticles in the Au@CNTs nanocomposite were evenly distributed throughout the porous CNTs network. The performance of the developed sensor was measured using cyclic voltammetry and amperometry. The electrochemical biosensor responded linearly to lactate over phosphate buffer solution with a low detection limit and sensitivity. Conclusion: The experiment of this sensor evaluated lactate concentrations in real sweat samples that were exceptionally close to the injection amount, enabling it as an effective biosensor for the detection of lactate in sweat samples. *Level of Evidence: Therapeutic Studies - Outcome Investigation*.

Keywords: Biosensing Techniques; Nanoparticles; Lactate; Electrochemistry.

# RESUMO

Introdução: É dada atenção ao desenvolvimento de sensores eletroquímicos para a medição rápida e em tempo real dos níveis de lactato. A síntese de sensores eletroquímicos é baseada em um eletrodo modificado com um nanocomposto. Objetivo: Analisar a viabilidade de um sensor eletroquímico para monitoramento esportivo de suor em lactato. O Au@CNTs foi o foco principal deste estudo. Métodos: O composto Au@CNTs foi sintetizado na superfície GCE, e testado sob protocolos preestabelecidos como sensor. Resultados: A forma e estrutura dos eletrodos modificados foram analisadas usando SEM, e os resultados mostraram que as nanopartículas de Au@CNTs no nanocomposto Au@CNTs foi medido usando voltametria cíclica e amperometria. O biosensor eletroquímico respondeu linearmente ao lactato sobre solução tampão fosfato com um limite de detecção e sensibilidade reduzidos. Conclusão: O experimento deste sensor avaliou as concentrações de lactato em amostras de suor real que estavam excepcionalmente próximas à quantidade de injeção, habilitando-o como um biosensor efetivo para detecção de lactato em amostras de suor. **Nível de evidência: Estudos Terapêuticos - Investigação dos Resultados.** 

Descritores: Técnicas Biossensoriais; Nanopartículas; Lactato; Eletroquímica.

### RESUMEN

Introducción: Se presta atención al desarrollo de sensores electroquímicos para la medición rápida y en tiempo real de los niveles de lactato. La síntesis de los sensores electroquímicos se basa en un electrodo modificado con un nanocompuesto. Objetivo: Analizar la viabilidad de un sensor electroquímico para la monitorización esporádica del sudor en el lactato. Los Au@CNTs fueron el objetivo principal de este estudio. Métodos: El compuesto de Au@ CNTs se sintetizó sobre la superficie de GCE, y se probó bajo protocolos preestablecidos como sensor. Resultados: La forma y la estructura de los electrodos modificados se analizaron mediante SEM, y los resultados mostraron que las nanopartículas de Au@CNTs en el nanocompuesto de Au@CNTs estaban distribuidas uniformemente en la red porosa de CNTs. El rendimiento del sensor desarrollado se midió mediante voltamperometría cíclica y amperometría. El biosensor electroquímico respondió linealmente al lactato sobre la solución tampón de fosfato con un bajo límite de detección y sensibilidad. Conclusión: El experimento de este sensor evaluó las concentraciones de lactato en muestras reales de sudor que eran excepcionalmente cercanas a la cantidad inyectada, lo que le permite ser un biosensor eficaz para la detección de lactato en muestras de sudor. **Nivel de evidencia: Estudios terapéuticos - Investigación de resultados.** 



Descriptores: Técnicas Biosensibles; Nanopartículas; Lactato; Electroquímica.

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#### INTRODUCTION

Clinical diagnosis of frequently evaluated critical parameter, lactate concentration, is used in surgery, exercise science, acute mental/physical discomfort, and the food industry to examine a person's health, pathology, and routine health inspection.<sup>1</sup> Based on the scale of activity, the level can reach 25 mmol/L.<sup>2</sup> In addition to being widely employed in the food and health management industries, lactate monitoring is essential in the treatment of diseases such liver disease, tissue hypoxia, bleeding, sepsis, and respiratory failure.<sup>3,4</sup> There is a need for a sensor having good sensitivity and high inputs screen of lactate in varied samples because the conventional methods for measuring lactate levels are unreliable and cumbersome.<sup>5</sup> Therefore, excessive focus has been paid to developing low-cost moveable devices, notably electrochemical sensors have been developed for quick, real-time measurement of the lactate levels.<sup>6</sup>

Particularly, 1-dimensional nanostructures have been discovered to be on the rise used in sensors, photonic, actuators, and optoelectronic devices due to 1-dimensional systems that enable them to possess alluring properties, including but not limited to piezoelectric, semiconducting, and pyroelectric, as noted in earlier studies.<sup>7,8</sup> Because of their electro-catalytic effectiveness, high aspect ratios, and rapid electron communication, they are suitable for increased immobilization and the necessary transducing phenomena.<sup>9,10</sup> Of all the transition elements, Au is the most conductive and reactive, and it has lately been employed with promising properties like improved mechanical strength, excellent dispersion, and low coefficient of friction.<sup>11</sup> Due to their antibacterial qualities, Au nanoparticles have lately grown in favor in biosensor applications.<sup>12</sup> However, it is necessary to increase the effectiveness of biosensors. In the current study, an efficient method that combined an electrochemical procedure was used to create an Au NPs@CNT composite on a glass carbon electrode (GCE).

#### MATERIALS AND METHOD

Before making any modifications, the GCE surfaces was cleaned for 25 minutes using alumina slurries (0.4 $\mu$ m, Meck, Germany) and a polishing cloth. It was then cleaned for 5 minutes in ultrasonic soak and washed with DI water. The 0.4 HAuCl<sub>4</sub>·3H<sub>2</sub>O aqueous solution, which contains 0.2 M H<sub>2</sub>SO<sub>4</sub>, was used to scatter the 10g CNTs. The resulting mixture was processed continuously for 6 minutes at a potential of 0.1 V. Using an Autolab potentiostat-galvanostat and an electrochemical cell with an Ag/AgCl electrode material (3 M KCl), Pt disk as that of the auxiliary electrode, and GCE as the working electrode, Au@CNTs nanostructures was electrodeposited. Electrodeposition was carried out using the cyclic voltammetry (CV) method for 10 cycles at a scan rate of 10 mV/s and a potential range of -1 to 1 V.<sup>13</sup> Following electrodeposition, ethanol, 40 mM -aminothiophenol, 40 mM tetrabutylammonium perchlorate (TBAP), and 20 mM 2-oxindole were combined to create the solution in which the Au@CNTs/GCE was submerged for 50 minutes.

Using a cell with three compartments, a functional Au@CNTs/GCE electrode, and a Pt wire in instead of the counter electrode, electrochemical investigations were carried out. We observed and recorded the potentials of Ag/AgCl reference electrode. A Lactate solution was created by combining 5.0 mg of Lactate with 1.0 mL of PBS solution. In order to dry in atmospheric circumstances, 20 µL of a solution was allowed to drip onto the electrodes. A scanning electron microscope was used to analyze the Au@CNTs/GCE electrode. With cycling a working electrode from -0.1 and -0.4 V at a scan rate mVs<sup>-1</sup>, cyclic voltammetry (CV) tests were performed. Several athletes' perspiration was actually collected, and the samples were immediately evaluated after their response. To perform a sensor regaining analysis, a tiny piece of the 950 liter total sample was combined with 100µL of lactate solution (0.1M). The study was conducted in accordance with the Declaration of Helsinki principle. The participants signed the Free and Informed Consent Form (EHIC).

#### **RESULTS AND DISCUSSION**

The surface morphologies of CNTs/GCE and AuNPs@CNTs/GCE are shown in Figure 1. As seen in the SEM image of the CNTs/GCE (Figure 1a), a electrodeposited CNTs were arranged in bundles with a diameter of about 50 nm, adhering to one another to form a porous structure. According to Figure 1b's SEM picture of Au@CNTs/GCE, the porous structure confirms the deposition of Au NPs on CNTs/GCE surface.

The CV plots of GCE, CNTs/GCE, and Au@CNTs/GCE in 0.1M PBS at a scan rate of 20mV/s in the potential range of 0.1 to 0.9V are shown in Figure 1c. As shown, the CV plots of GCE and CNTs/GCE do not exhibit a redox peak, but Au@CNTs/GCE has a redox peak around 0.54 V and 0.43 V, respectively, showing the oxidation of Au<sup>0</sup> into Au<sup>+</sup> and the reduction of Au<sup>+</sup> onto Au<sup>+</sup>.<sup>14-16</sup>

To the best of our knowledge, no Au@CNTs/GCE nanostructure-containing electrochemical biosensor to measure lactate had been created before to this work. Due to this circumstance, the reaction is reliant on the lactate-catalyzed oxidation from lactate to pyruvate. Figure 2a depicts the Au@CNTs/GCE electrode's CV response both with and without accumulated lactate concentrations in a 0.1M phosphate buffer. The parameters of the investigation—reproducibility, sensitivity, repeatability, linear-range of level, and limit of detection—are discovered.<sup>17</sup> Figure 2b shows a linear calibration curve with concentration ranges from 0.01 to 0.6mM (R<sup>2</sup>= 0.9987). The calculated sensitivity was determined to be 48.02µA/mM based on the gradient of the figure.

The LOD was  $1.4\mu$ M, or the limit of detection. Due to their larger aspect ratio and longer carrier lifespan than others, this LOD is comparable to those obtained from other methods generally employed to measure lactate. The relative standard deviation (RSD), which was obtained from 9 different measurements of a 0.5mM lactate concentration using a constant biosensor, was used to evaluate the repeatability. 5 percent was given as the RSD value. After evaluating the storage stability, it was determined



Figure 1. Surface morphology of (A) CNTs/GCE, (B) Au@CNTs/GCE, (C) CV plots of GCE, CNTs/GCE, and Au@CNTs/GCE with 0.1M PBS at 10mV/s scan rate into potential ranges of 0.1to 0.9V.



Figure 2. (A) CV plots of Au@CNTs/GCE sensor in interaction by a 0.1 M PBS with and without 0.0 to 0.5mM of lactate, (B) Calibration plot from the stated CV results.

those 10 days later, it retains 50% of its initial reactions. Based on these findings, it can be concluded that using Au@CNT nanocomposites to create lactate biosensors enables the acquisition of an analytical response that is comparable to those obtained by other nanostructures.<sup>18</sup> Additionally, the biosensor created for this study has advantages like a simple production technique and the utilization of affordable nanomaterial.<sup>19</sup>

The sensor is quite stable, making it possible to use it to accurately measure the lactate concentration in sweat samples. Sweat was tested for lactate concentration in order to examine the Au@CNTs/ GCE electrodes for measuring lactate in real samples. The amount of lactate in the sweat sample was calculated using a standard injection of lactate. Figure 3a displays the amperometrogram that was observed after injecting multiple doses of 0.1 mM lactate solutions into a sweet sample made with Au@CNTs/GCE. The calibration curve is displayed in Figure 3b with a coefficient of correlation of 0.9878. As a result, the calculated lactate concentrations in 0.1M PBS and pure sweat were 0.18mM and 0.25mM, respectively. The findings indicate that Au@CNTs/ GCE may be used as a biosensor to assess lactate in sweat samples.<sup>20</sup>



Figure 3. (A) The amperometrogram of Au@CNTs/GCE into 0.1M PBS of various levels of lactate into real samples; (B) graph of the calibration curve.

#### CONCLUSIONS

One of the most crucial factors evaluated when evaluating an athlete's performance is sweat lactate concentration. In this study, a simple method for fabricating a sensitive electrochemical sensors to detect lactate was used. The sensor was created using an Au@CNTs/GCE coated GCE substrate. Using FESEM analysis, the morphological characteristics of Au@CNTs/GCE were identified. The distribution of Ag NPs in CNTs nanomaterials was validated by FESEM data. Amperometry and CV were used to assess the sensor's performance. With a low limit of detection and sensitivity of 1.4µM and 48.02 mAcm<sup>-2</sup> mM<sup>-1</sup>, respectively, the electrochemical biosensor demonstrated a linear relation to the lactate into phosphate buffer solution. This sensor can be thought of as a biosensor for lactate detection in sweat samples because the level of lactate it estimated in actual sweat samples was extremely similar to the amount of injection.

The author declare no potential conflict of interest related to this article

AUTHORS' CONTRIBUTIONS: This work and its knowledge content and the drafting of the manuscript is completed by Yanling Zhou. The author completed its execution and writing of this manuscript.

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