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ORIGINAL



# Electrochemical Sensors based on Conductive Polymers Incorporate of Nano Material for the Detection of Hydrogen Peroxide (H2O2)

## Sensores electroquímicos basados en polímeros conductores que incorporan nanomateriales para la detección de peróxido de hidrógeno (H2O2)

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#### ABSTRACT

Hydrogen peroxide (H2O2) plays a crucial role in various industries but poses a risk to human health when present in an uncontrolled manner. Hence, it is imperative to develop straightforward, cost-effective, and swift analytical methods for the detection and monitoring of H2O2. This study proposes a detector consisting of polyaniline-doped silver nanoparticles (Ag NPs), utilising a nanostructured okra semiconductor as a sensing material for H2O2 detection. The obtained results indicated that the addition of silver nanoparticles (Ag NPs) (at particle size 30 nm) into the mixture at different concentrations (1, 5, and 10 wt%) and voltages (1,4V-3V) led to good electrochemical performance. The prepared sensor at the Ag nanoparticle weight concentration (10 wt%) proved to have optimal performance. This configuration exhibited a clear and reliable signal response across a broad spectrum of currents at different concentrations of H2O2.

**Keywords:** Polyaniline; Semiconducting (Okra Plant); Silver Nanoparticles; Hydrogen Peroxide Monitoring; Electrochemical.

#### RESUMEN

El peróxido de hidrógeno (H2O2) desempeña un papel crucial en diversas industrias, pero supone un riesgo para la salud humana cuando está presente de forma incontrolada. De ahí que sea imperativo desarrollar métodos analíticos sencillos, rentables y rápidos para la detección y monitorización del H2O2. Este estudio propone un detector consistente en nanopartículas de plata dopadas con polianilina (Ag NPs), utilizando un semiconductor nanoestructurado de okra como material sensor para la detección de H2O2. Los resultados obtenidos indicaron que la adición de nanopartículas de plata (Ag NPs) (con un tamaño de partícula de 30 nm) en la mezcla a diferentes concentraciones (1, 5, y 10 wt%) y voltajes (1,4V-3V) condujo a un buen rendimiento electroquímico. El sensor preparado con la concentración en peso de nanopartículas de Ag (10 % en peso) demostró tener un rendimiento óptimo. Esta configuración mostró una respuesta de señal clara y fiable en un amplio espectro de corrientes a diferentes concentraciones de H2O2.

**Palabras clave:** Polianilina; Semiconductor (Planta De Okra); Nanopartículas de Plata; Monitorización de Peróxido de Hidrógeno; Electroquímica.

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#### **INTRODUCTION**

The exploration of electrochemical sensors has become a highly significant area of research, primarily driven by its potential implications in the development of analytical devices. Advances in the analytical capabilities of amperometric sensors have facilitated the identification of nanostructured materials, particularly inorganic metal nanoparticles (MNPs), and known for their substantial surface area. Silver nanoparticles (AgNPs) have found diverse applications in electrochemistry, including electrocatalysis,<sup>(1)</sup> bioelectroanalysis,<sup>(2)</sup> and electroanalysis. <sup>(3)</sup> AgNPs stand out as preferred nanomaterials due to their ability to serve as a stable substrate, facilitating the effective immobilization of biomolecules and redox mediators. This, in turn, maintains the electrochemical activity of the immobilized molecules.<sup>(4)</sup> The growing interest in AgNP-based biosensors<sup>(5)</sup> is attributed to their notable biocompatibility. Numerous studies have demonstrated that sensors based on AgNPs enhance electron transfer, elevate electrode conductivity, and improve the detection limit of biomolecules.<sup>(6)</sup> An important byproduct resulting from the enzymatic oxidation by various highly selective oxidases is hydrogen peroxide (H2O2). The H2O2 plays a vital role in the metabolism of proteins, carbohydrates, lipids, and other essential substances. Additionally, it contributes to blood sugar regulation and cellular energy production. The robust oxidizing properties of H2O2 and its derivatives have led to their extensive use in synthesizing various organic compounds. Consequently, detecting H2O2 has become crucial in clinical, environmental, and pharmaceutical analyses.<sup>(7)</sup> Several analytical techniques have been developed to detect H2O2, including electrochemistry,<sup>(8)</sup> chemiluminescence,<sup>(9)</sup> spectrophotometry,<sup>(10)</sup> spectrofluorometry,<sup>(11)</sup> and titrimetric measurements.<sup>(12)</sup> Electrochemical methods, due to their rapid detection speed, high selectivity, and sensitivity, are considered more reliable compared to other methods facing challenges such as interference effects and prolonged analysis times.<sup>(13)</sup> Research has indicated that electrodes fabricated using carbon nanotubes,<sup>(14)</sup> metal alloys,<sup>(15)</sup> and metal oxides <sup>(16)</sup> exhibit favorable catalytic performance in the context of direct electrochemistry of H2O2.

S. Palsaniya et al.<sup>(17)</sup> investigated various mechanisms through which intrinsic MoS2 and rGO-MoS2 nanostructures could mitigate H2O2. This amalgamation of sensing materials proves to be effective in discerning H2O2 levels in both raw apple juice and pasteurized milk samples. To the best of our knowledge, this study stands out for its distinctive approach of utilizing concentrated apple juice and pasteurized milk as sensing mediums, without the requirement for additional external electrolytes. This underscores the dependability and practical applicability of the sensing material in real-world sample analyses.

F. Bohlooli et al.<sup>(18)</sup> delved into the prospect of enhancing CNW by incorporating varying levels of oxygen plasma, exploring its applicability as a metal-free electrode in electrocatalysis and sensing applications. The impact of different oxygen flow rates on CNW properties was examined through analytical techniques such as FE-SEM, XPS, Raman spectroscopy, and water contact angle measurements. The sensor capabilities of CNW samples in detecting H2O2 were assessed using cyclic voltammetry and amperometric methods, unveiling notable sensitivity and an extensive linear detection range.

A. Abdelwahab et al.<sup>(19)</sup> presented an electrochemical sensor designed to detect H2O2 through the immobilization of AgNPs on a nanocomposite layer comprising oxidized pTTBA and MWCNT. The electrochemical oxidation of pTTBA into Ox-pTTBA generates active sites characterized by negatively charged carboxylate groups and a polymer backbone abundant in  $\pi$  electrons. This results in an Ox- pTTBA film with enhanced conductivity and cationic permselectivity, promoting robust AgNP adsorption. The inclusion of MWCNT further amplifies sensor conductivity and facilitates efficient electron transfer on the electrode surface.

K. Settue et al.<sup>(20)</sup> undertook this study with the aim of fabricating a laser-induced graphene (LIG) electrode dedicated to H2O2 detection. The methodology involved irradiating a polyimide film using a laser. Subsequently, to augment the electrocatalytic response to H2O2, the LIG electrode underwent modification with MWCNT. The performance of both the unmodified LIG sensors and those incorporating MWCNT was evaluated concerning their efficacy in H2O2 detection.

The aim of this work focuses on preparing H2O2 sensor consists of polyaniline polymer mixed with the semiconductor material derived from the okra plant as matrix enhanced with different concentration (1, 5, and 10 wt. %) of silver nanoparticles (Ag NPs), the impact of the added silver nanoparticles investigated by studying the correlation between the hydrogen peroxide (H2O2) concentration and the current under different applied voltages.

#### **Used Materials**

The materials utilized in this study include polyaniline polymer (PANI) in powder form, Jiangsu Xfnano Materials Tech Co., Ltd. with high purity (99,8 %). The okra plant was sourced from local markets, while the additive silver nanoparticles were prepared in a laboratory (at particle size 30 nm).<sup>(21)</sup> An integrated sensor, specified with dimensions (10 \* 10 \* 0,635 mm), working temperature range of -50°C to 500°C, and constructed with key materials like alumina ceramics and gold, was acquired from CHANGCHUN MEGA BORUI TECHNOLOGY CO., LTD. in China. The main parameters of the sensor include an electrode spacing of (0,09 mm), electrode width of (0,08 mm), carbon film thickness ranging from (9 to 11  $\mu$ m), and a total of 15 electrode pairs. Figure

1 shown the integrated and prepared sensor.



Figure 1. (a) the integrated sensor (b) prepared sensor

## I-V Curve Measurement

The sensor's current and voltage are measured using a Sweep circuit, comprising a power supply, a voltage transformer, a Sweep generator, a variable resistor for controlling the applied voltage, and a combined current and voltage meter (voltmeter). These components are interconnected according to the configuration depicted in figure 2.



Figure 2. Used sweep circuit

## Preparation of samples

Prepare the okra plant (semiconductor) by washing it thoroughly with water, cutting it into pieces, and soaking it overnight in distilled water. Subsequently, extract the gum by filtering it through a cloth, and then dry the extracted gum in an oven at (40 oC). Dissolve (2,5 g) of polyaniline and okra plant powder in dimethyl sulfoxide (DMSO) and mix them through ultrasonication mixer for (15 min) at amplitude (60 %). then, adding the colloidal silver solution (100g silver per 1 liter okra emulsion) to the mixture of polyaniline and okra semiconductor material at a weight concentration of (1, 5 and 10 wt. %). Stirring the mixture using a magnetic stirrer at a temperature of (25 oC) for (6 h). Pour the prepared mixture (2 ml) onto the integrated sensor, distribute it manually, and then dry it at (40  $^{\circ}$ C) for (24 h) to obtain the sensor.

#### **RESULTS AND DISCUSSION**

Due to the unstable nature of peroxide bonds, hydrogen peroxide is a highly reactive molecule. Consequently, the degradation of H2O2 typically proceeds through an intermediate step, resulting in the formation of two hydroxyl ions or an ion and a radical.<sup>(22,23)</sup> Therefore, the interaction between hydrogen peroxide (H2O2) and the (Okra + PANI) mixture sensor leads to the degradation of H2O2 and formation of a complex molecule, accompanied by the release of electrons, thereby inducing conductivity. Subsequently, a SWEP circuit was devised to apply defined voltages (ranging from 3,2 to 2,2 volts) to the sensor while adjusting the concentrations of H2O2. This procedure was conducted to gauge the resulting current at each applied voltage.

Figure 3 clearly illustrates that the current exhibits a proportional increase in response to concentration of H2O2 on the sensor, at different voltage of (2,2-3,2 V). The relationship between the current measured from the electrical circuit and the concentration of H2O2 was observed to be linear at (2,6 v) with the exception of a sudden surge in current. at a concentration of (8×10-4). At this concentration, the recorded current was (99  $\mu$ A), whereas at a concentration of (3,2×10-2), the measured current (312  $\mu$ A). The increasing of the recorded current suggests an anomaly in the expected pattern with varying concentrations of H2O2, which can be attributed to the catalysis of hydrogen peroxide degradation, occurs at the active sites on the sensor surface, leading to the generation of OH radicals. In this process, the radicals acquire electrons from the active material, reverting to hydroxyl anions and desorbing as water molecules. This mechanism is a prevalent detection system in electrochemical sensors.<sup>(24)</sup>



Figure 3. Relationship between the current and concentrations of H2O2 of prepared sensor without the silver nanoparticles

After incorporating silver nanoparticles at three different concentrations (1, 5 and 10 wt. %) with the blend, in figure 4 illustrates that the addition of silver nanoparticles (1 wt. %) to the blend. The results in a discernible sensitivity in the measured currents at voltage (2,6 V). Additionally, a linear correlation between the concentration of H2O2 and the measured currents is observed, but a sudden increase in currents occurs at a concentration of ( $3,2\times10-2$ ) where measured current reached ( $680 \mu A$ ), whereas at a concentration of ( $8\times10-4$ ), the measured current was ( $190 \mu A$ ). The adding of silver nanoparticles led to significant increasing in the recorded current which attributed to the hydroxyl ions and radicals produced by hydrogen peroxide have the potential to undergo chemical reactions with the active materials (Ag nanoparticles) the existing in sensor. In contrast to the sensing mechanism involving noble metals, hydroxyl ions are not only adsorbed but actively engaged with the active sites of transition metal-based structures before being released, usually carrying hydrogen atoms along with them.<sup>(25,26,27)</sup>

When increase silver nanoparticles concentration to (5 wt. %), fig. 5, illustrates the current exhibits a proportional increase in response to concentration of H2O2 on the sensor, at different voltage of (1,4 - 3 V). But there is no evident linear correlation between the concentration of H2O2 with current at different voltages. Therefore, the concentration of silver was raised to 10 %.



Figure 4. Relationship between the current and H2O2 concentrations of prepared sensor with the silver nanoparticles concentration (1 wt. %)



Figure 5. Relationship between the current and H2O2 concentrations of prepared sensor with the silver nanoparticles concentration (5 wt. %)

Figure 6 illustrates a distinct linear correlation between the concentration of H2O2 and the current at a voltage of 2,6 V. where the Current increase gradual with concentration increase of H2O2, at concentration of 1wt % nano silver, there was a breakdown in the material at concentration  $(3,2\times10-2)$  for H2O2, so the conductivity suddenly increased, and the current increased with it but when the percentage of nano silver was increased to 10 wt %, No collapse of the material occurred at in same concentration of H2O2, and this meets with the sensitivity property requirement. This indicates that the sensor from blend (OKRA+PANI) with the addition of silver nanoparticles at concentration (10 wt. %), give both properties linearity and sensitivity which makes it as a proposed sensor for H2O2. Furthermore, when other liquids were added on the sensor, the current reading was (40  $\mu$ A), which is lower than the reading obtained for the lowest concentration of H2O2 (1×10-5). Also, this measurement conducted in the air, did not yield any current reading, this demonstrating the selectivity property.<sup>(28,29)</sup> The reduction of H2O2 is accelerated in the presence of a higher content of silver

nanoparticles (10 wt. %) on the surface of the (PANI+Okra+Ag) sensor. and the reaction between the H2O2 and the Ag nanoparticles goes toward more O2 generation. Subsequently, the generated O2 serves as the detection signal on the prepared modified electrode.<sup>(30,31)</sup> On the other hand, the detection mechanism in the sensor depends on the nature of the conductive material (Ag nanoparticles), where the interaction with the hydrogen peroxide H2O2 leads to change the electrical conductivity.<sup>(32,33)</sup>



Figure 6. Relationship between the current and H2O2 concentrations of prepared sensor with the silver nanoparticles concentration (10 wt. %)

#### CONCLUSION

The results gained from this study lead to the proposal of a composite material that acts as a sensor for hydrogen peroxide (H2O2). This composite material incorporates a semiconducting component Extract it from the okra plant, together with polyaniline. Experimental results demonstrate that using a polyaniline ratio of 50:50 combined with okra extract yields a current ranging from 25 to 312  $\mu$ A at a voltage of 2,6 V, with different concentrations of hydrogen peroxide ranging from 3,2 \* 10-2 to 8 \* 10-4 mmol. Following that, silver nanoparticles were added to the mixture at weight percentages of 1 %, 5 %, and 10 %. The experimental findings indicated that the ideal proportion of silver nanoparticles was discovered to be 10 weight percent, resulting in current ranging from 56 to 315 microamperes at a voltage of 2,6 volts at different levels of hydrogen peroxide concentration, displaying a linear behavior. In addition, this sensor functions most effectively at a voltage of 2,6 volts because it can detect a wide variety of different amounts of hydrogen peroxide. It also possesses the capability to detect lower concentrations of hydrogen peroxide, rendering it an exceptionally effective sensor for detecting hydrogen peroxide.

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#### FINANCING

None.

#### **CONFLICT OF INTEREST**

None.

#### **AUTHORSHIP CONTRIBUTION**

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