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Electrodeposited Gold Nanoparticles on Graphene Oxide-Modified Carbon Sheets for Enhanced Electrocatalysis and Hydrogen Peroxide Sensing

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ABSTRACT

Non-enzymatic electrochemical sensors for hydrogen peroxide (H_2O_2) are attractive due to their simplicity and stability relying on intrinsic electrocatalytic activity of functional electrode materials. In this work, electrochemical metal deposition was employed fabricate gold nanoparticles (AuNPs) directly on conductive substrate namely bare carbon sheets (CS) and graphene oxide-modified carbon sheets (GO/CS) using the chronoamperometry method. Scanning Electron Microscopy (SEM) confirmed the successful formation of AuNPs on both substrates, with a 57.99% reduction in nanoparticle size observed on GO/CS. The electrocatalytic performance of CS, GO/CS, AuNPs/CS, and AuNPs/GO/CS was evaluated in ferricyanide solution, where the AuNPs/GO/CS electrode exhibited the highest current density and fastest electron transfer. Furthermore, hydrogen evolution reaction (HER) studies in acidic medium demonstrated the enhanced catalytic performance of AuNPs/CS and AuNPs/GO/CS, with current increases of 283.74% and 363.37%, respectively, indicating efficient proton reduction to hydrogen. Additionally, only AuNPs/CS and AuNPs/GO/CS electrodes detected a distinct hydrogen peroxide (H_2O_2) oxidation peak at 0.98 V, with the AuNPs/GO/CS electrode showing a 12.11% higher peak current density. A linear correlation between charge and H_2O_2 concentration was observed over a range of 0.005–10 mM with limit of detection (LOD) 5 μM, highlighting the potential of AuNPs/GO/CS electrodes for sensitive electrochemical sensing applications.

Keywords: *Electrodeposition, Gold Nanoparticles, Graphene Oxide, Hydrogen Peroxide, Electrochemical Sensing*

1. INTRODUCTION

Hydrogen peroxide (H_2O_2) is a reactive inorganic compound and strong oxidizing agent widely used in industries and naturally present in various biological environments, as well as in water and air. Depending on applications, the hazardous and toxic concentration of H_2O_2 varies in food, health and environment [1]. In human body, H_2O_2 is generated as by-product of various enzymatic and metabolic reactions and abnormal concentration is associated with metabolic disorders such as diabetes, pulmonary diseases, or other health conditions [2]. Therefore, precise sensing of H_2O_2 is very important and H_2O_2 sensors have been a forefront research for decades. Although numerous transduction elements have been explore for H_2O_2 detection, electrochemical sensors remain as the favorable method owing to their superior sensitivity and selectivity, cost efficiency, rapid response, and ease of miniaturization [3]. In order to improve the catalytic properties of non-enzymatic H_2O_2 electrochemical sensors, many research has employ nanomaterials as sensing material.

Carbon-based nanomaterials have attracted considerable interest due to their unique characteristics, including low cost, chemical stability, inert electrochemistry, and excellent electrocatalytic activity toward a wide range of redox reactions [4-5]. These advantages have led to their extensive application in electroanalysis and electrocatalysis. The emergence of graphene, a two-dimensional (2D) material with remarkable electrical, mechanical, and surface properties, has greatly stimulated research in this field [6-7]. Among its derivatives, graphene oxide (GO) has received particular attention because the presence of oxygen-containing functional groups improves its dispersibility in aqueous media and provides abundant active sites for chemical modification [8-9]. Consequently, GO-based hybrid materials therefore offer a promising strategy to enhance the functionality of nanomaterials and open new opportunities for developing advanced catalytic, magnetic, and optoelectronic systems [10-11]. In combination with gold nanoparticles (AuNPs), GO acts as a conductive and chemically active support that prevents nanoparticle aggregation, improves dispersion, and facilitates electron transfer, thereby maximizing the catalytic performance of the hybrid nanostructure [12-13]. Owing to their distinctive physicochemical and electronic properties, gold nanoparticles (AuNPs) have been widely explored for broad applications in catalysis, fuel cells, and biosensing. Among the numerous synthesis approaches,

electrodeposition has emerged as a particularly attractive strategy. This technique allows the direct formation of AuNPs on conductive substrates in a rapid, scalable, and controllable manner, thereby improving electrode conductivity, promoting electron transfer, and enhancing analytical sensitivity and selectivity [14]. However, relationship between the crystal growth kinetics and the mass transport during electrocrystallization can be systematically controlled via various electrochemical and precursor parameters that strongly influenced particle size, morphology, and distribution. These outcome will subsequently effect on the catalytic performance of AuNP-modified electrodes [15-16].

In our previous work, we investigated the effect of electrodeposition duration on the morphology, electrocatalytic activity, and electrochemiluminescence (ECL) performance of nitrogen-doped carbon dots (NCDs) on screen-printed electrodes (SPEs) [17]. Building on this foundation, the present study focuses on evaluating the electrocatalytic performance and sensing capabilities of electrodeposited AuNPs, with and without GO as a supporting matrix, to better understand the synergistic contributions of AuNP–GO hybrid structures in electrochemical applications specifically for non-enzymatic H_2O_2 detection.

2. MATERIALS AND METHOD

Potassium chloride (KCl), potassium ferricyanide ($K_3[Fe(CN)_6]$), potassium phosphate dibasic (K_2HPO_4) and potassium phosphate monobasic (KH_2PO_4) were obtained from HmbG Trading Corporation, Malaysia. Tetrachloroauric(III) acid trihydrate ($HAuCl_4.3H_2O$) was purchased from Acros Organics, Belgium while hydrochloric acid (HCl) was acquired from Fisher Scientific (M) Sdn. Bhd. Graphene Oxide (GO) purchased from GO Advanced Solutions Sdn. Bhd. Phosphate buffer solution (PBS, 0.1 M, pH 7.4) was prepared using K_2HPO_4 and KH_2PO_4. All the chemicals were used as received.

Scanning Electron Microscopy (Tescan Vega, Czech Republic) was used to observe surface morphology of the prepared electrodes. All electrochemical measurements were carried out using a USB-powered portable Metrohm DropSens potentiostat/galvanostat/impedance analyzer (μStat-i-400s, Metrohm DropSens, S.L, Spain) and data were collected using the DropView 8400 software. A three-electrode setup for electrochemical measurement was applied, where a platinum wire and Silver-Silver Chloride (Ag/AgCl) were used as counter and reference electrodes, respectively.

2.1. Preparation of Electrodes

The substrate of the all electrodes are made of carbon sheet (CS) with electrode surface of 1 cm^2. Graphene oxide modified CS (GO/CS) electrode was made by drop casting 50 μL of 1 mg/mL GO in ethanol. After that, the GO/CS electrode was dried at 60 °C for 1 hour in convection oven. Meanwhile, gold nanoparticles (AuNPs) modified CS and GO/CS were prepared by using electrochemical deposition method. Firstly, 1 mM $HAuCl_4$ solution was made in 0.1 M HCl (pH 1.2). Then, the electrodeposition of AuNPs was performed through the chronoamperometry (CA) method at -0.4 V (vs Ag/AgCl) in 5 mL of $HAuCl_4$ solution for the duration of 100 seconds [16-17]. Finally, the AuNPs/CS and AuNPs/GO/CS electrodes were rinsed using distilled water and blot-dried. All electrodes were kept in desiccator at room temperature until used.

2.2. Electrocatalytic Evaluation and Hydrogen Peroxide Detection

The electrocatalytic performance of all electrodes was evaluated in 5 mM ferricyanide ($Fe(CN)_6^{3-/4-}$) prepared in 0.1 M KCl using cyclic voltammetry (CV) within a potential range of –0.6 to 0.8 V. The hydrogen evolution reaction (HER) activity was further investigated by CV in 0.1 M HCl over a potential range of –0.4 to –1.2 V. For sensing studies, the electrochemical detection of 1 mM hydrogen peroxide (H_2O_2) in 0.1 M PBS was conducted using CV from 0 to 1.4 V. The sensitivity of the electrodes toward H_2O_2 was examined across a concentration range of 5 μM to 10 mM. All electrochemical measurements were performed at room temperature with a scan rate of 100 mV/s.

3. RESULTS AND DISCUSSION

3.1. Surface Morphology of Electrodes

Figure 1(a) presents the SEM image of the carbon sheet (CS), where the surface of the substrate is visibly rough and flaky. Upon drop-casting with GO, as shown in Figure 1(b), multiple overlapping GO layers are observed, forming a continuous coating that fully covers the CS surface. This modification alters the surface morphology, creating a more uniform platform for subsequent electrochemical processes. In Figure 1(c) and 1(d), circular bright spots are clearly visible on the CS and GO/CS substrates, serve as direct evidence that electrochemical deposition via the chronoamperometry method successfully generated AuNPs on the electrode surfaces. On the bare CS substrate, the AuNPs appear densely distributed with relatively uniform particle size. In contrast, on the GO/CS substrate, the deposition yields a greater density of much smaller particles, interspersed with a few larger nanocrystals. These results shows that nucleation occurs simultaneously at many sites of oxygen-containing functional groups GO that serve as reactive anchoring sites for Au^{3+} precursors and subsequent reduction to Au^0 [18]. This leads to the formation of a large number of nuclei and restricts subsequent particle growth, resulting in smaller and more uniformly distributed AuNPs. Without GO, few nuclei form on CS and have excessive growth that produce large AuNPs. To further quantify these observations, ImageJ software was employed to analyze the SEM images of AuNPs/CS and AuNPs/GO/CS electrodes to calculate the AuNPs diameter. Figure 2 shows the histograms of AuNPs diameter distribution on CS and GO/CS substrates. From a total of 395 particles, AuNPs exhibit an average particle size of 254.2 ± 175.0 nm on CS substrate. Meanwhile, a total of 783 AuNPs

particles were measured on GO/CS substrate, yielding an average particle size of 106.8 ± 92.0 nm. The presence of larger AuNPs suggests partial agglomeration on CS substrate during the deposition process. In comparison, AuNPs with a dominant population of small nanoparticles and a minor fraction of larger aggregates on GO/CS substrate, indicating effective dispersion and nucleation of AuNPs on the graphene oxide support [19]. This observation confirms the role of graphene oxide as an effective support that promotes AuNP nucleation and suppresses excessive particle growth and agglomeration with 57.99% reduction in particle size.

Figure 1. SEM images of (a) CS, (b) GO/CS, (c) AuNPs/CS and (d) AuNPs/GO/CS.

Figure 2. AuNPs diameter distribution on (a) CS and (b) GO/CS.

3.2. Electrocatalytic Evaluation

The electrocatalytic characteristics of CS, GO/CS, AuNPs/CS, and AuNPs/GO/CS electrodes were examined by cyclic voltammetry (CV) in a ferricyanide solution, as shown in Figure 3. The CV curves exhibit an oxidation potential (E_{ox}) for the conversion of ferrocyanide to ferricyanide at approximately 0.31 V, and a reduction potential (E_{red}) for the reverse process at approximately 0.12 V, with distinct peak currents (I_p) observed for each electrode. The peak-to-peak separation ($\Delta E_{p-p} = E_{ox} - E_{red}$) was further evaluated to assess the electron-transfer kinetics between the electrodes and the analyte.

Figure 3. CV and of CS electrode modified with GO and electrodeposited AuNPs in 5 mM ferricyanide + 0.1 M KCl at scan rate of 100 mV/s.

Table 1 Electrocatalytic evaluation of various electrode in ferricyanide solution

Electrode	I_p (mA cm^{-2})	E_{ox} (V)	E_{red} (V)	ΔE_{p-p} (V)
CS	2.683	0.318	0.126	0.192
GO/CS	2.612	0.312	0.124	0.188
AuNPs/CS	2.877	0.318	0.126	0.192
AuNPs/GO/CS	3.030	0.310	0.126	0.184

Quantitative analysis in Table 1 revealed that the CS electrode produced a ΔE_{p-p} of 0.190 V with an I_p of 2.683 mA cm^{-2}. The GO/CS electrode yielded a slightly smaller ΔE_{p-p} of 0.188 V but a reduced I_p of 2.612 mA cm^{-2}, representing a 2.69% decrease compared to bare CS due to insulating properties of GO that hinder the electron transfer. In contrast, the AuNPs/CS electrode exhibited an I_p of 2.877 mA cm^{-2}, corresponding to a 7.23% increase relative to CS, although the ΔE_{p-p} increased slightly to 0.192 V, indicating a trade-off between current response and electron-transfer kinetics. Notably, the AuNPs/GO/CS hybrid electrode achieved the highest I_p of 3.030 mA cm^{-2} and the lowest ΔE_{p-p} of 0.184 V. This reflects a 12.92% increase in I_p together with a 3.16% reduction in ΔE_{p-p} compared to bare CS, confirming that the synergistic combination of GO and AuNPs significantly enhanced the electrocatalytic activity.

Since all electrodes show reversable reaction towards $Fe(CN)_6^{3-/4-}$ reaction, the catalytically active sites available for electrochemical reactions known as electrochemically active surface area (ECSA) of the electrode can be determined through Randles–Sevcik equation (1).

$$I_p = (2.69 \times 10^5) n^{3/2} A C D^{1/2} v^{1/2} \quad (1)$$

where I_p is peak current (A), n is number of electron transferred in the redox process, A is electrode area (cm^2), C is concentration of electroactive species (mol/cm^3), D is diffusion coefficient of ferricyanide (7.6×10^{-6} cm^2/s) and v is scan rate (V/s) [19]. The calculated ECSA are 0.362 cm^2, 0.352 cm^2, 0.388 cm^2 and 0.409 cm^2 for CS, GO/CS, AuNPs/GO and AuNPs/GO/CS electrodes, respectively. Herein, the ECSA of electrode modified by AuNPs increase 7.18% and 16.19% from CS and GO/CS substrate, respectively. Hence the superior electrocatalytic performance can be attributed to the smaller particle size of AuNPs on the GO/CS substrate, which provides a higher surface-to-volume ratio and increases the electroactive surface area. These properties are well established to improve catalytic activity and facilitate faster electron transfer, thereby enhancing the sensitivity and overall efficiency of electrochemical sensors [20].

Figure 4. CV of CS electrode modified with GO and electrodeposited AuNPs in 0.1 M HCl.

The hydrogen evolution reaction (HER) performance of the different electrode substrates was evaluated by examining the catalytic reduction of protons to hydrogen in an acidic medium. As illustrated in Figure 4, this process was examined through CV in 0.1 M HCl, where the measured current corresponds to the catalytic reduction activity. Compared to bare CS, the current increased by 11% for the GO/CS electrode, 327% for the AuNPs/CS electrode, and 415% for the AuNPs/GO/CS electrode. These findings demonstrate that the incorporation of AuNPs significantly enhances HER performance, while the combination of AuNPs with GO provides an even greater improvement. The superior performance of the AuNPs/GO/CS hybrid electrode can be attributed to the synergistic effect of AuNPs and GO. The AuNPs provide abundant active sites and excellent catalytic activity, whereas the GO layer increases surface roughness, facilitates proton adsorption, and promotes efficient electron transport. Collectively, these factors accelerates proton reduction, leading to enhanced electrocatalytic activity and overall HER performance [16, 18].

3.3. Hydrogen Peroxide Detection

Figure 5(a) presents the CV response of 1 mM H_2O_2 in PBS on the different electrode substrates. It can be observed that AuNP-modified electrodes exhibit clear electrocatalytic activity toward H_2O_2 oxidation, whereas the bare CS and GO/CS electrodes show negligible electrochemical response. The onset oxidation potential of H_2O_2 occurs at approximately +0.8 V. Notably, the AuNPs/GO/CS electrode demonstrates an increase of 13.8% in oxidation current density compared to the AuNPs/CS electrode, indicating its superior catalytic activity and suitability as an efficient H_2O_2 sensor. GO layer provides abundant adsorption sites for H_2O_2 molecules due to the presence of numerous surface defects and oxygen-containing functional groups. Although these defects are beneficial for the adsorption, they also deteriorate the electrical conductivity of GO, resulting in weak electrocatalytic response of the GO/CS electrode toward H_2O_2. On the other hand, AuNPs prove to be a good catalysts for H_2O_2 reaction by improving electric conductivity and electron transfer. Combining the adsorption ability of GO and catalytic properties of AuNPs have synergistically enhances H_2O_2 detection performance of AuNPs/GO/CS electrode [20, 21, 22, 23].

Figure 5. (a) CV of CS electrode modified with GO and electrodeposited AuNPs in 1 mM H_2O_2 + 0.1 M PBS (pH 7.4). (b) CV of AuNPs/GO/CS electrode in various concentrations of H_2O_2 from 5 μM to 10 mM in 0.1 M PBS (pH 7.4) and its calibration curve (inset).

Figure 5(b) shows the CV responses of the AuNPs/GO/CS electrode toward varying concentrations of H_2O_2, with the inset displaying the corresponding calibration plot. A linear relationship between charge and H_2O_2 concentration is observed over a wide range from 5 μM to 10 mM, with a sensitivity of 776.9 $\mu AmM^{-1}cm^{-2}$ and an excellent correlation coefficient (R^2 = 0.9947). The limit of detection (LOD) was determined to be 5 μM, confirming the high sensitivity and reliability of the AuNPs/GO/CS electrode for H_2O_2 detection. Table 2 shows the comparison of AuNPs/GO/CS electrode with other reported non-enzymatic H_2O_2 electrochemical sensors utilizing oxidation reaction potential. AuNPs/GO/CS electrode shows a sensitive detection at wide linear range compared to other develop electrode.

Table 2 Performance of non-enzymatic H_2O_2 electrochemical sensor

Electrode	LOD (μM)	Sensitivity ($\mu AmM^{-1}cm^{-2}$)	Linear range (mM)	Ref
AuNPs/GO/CS	5	776.9	0.005–10	This work
Pt-coated Au NPs core–shell structure	0.18	56.59	0.001-0.45	[24]
Au nanorods (1:5)	9.38	292.55	0.009-0.14	[25]
Graphene wrapped Cu2O nanocubes	3.3	47	0.3-3.3	[26]

4. CONCLUSION

In this study, gold nanoparticles (AuNPs) were successfully electrodeposited onto bare carbon sheets (CS) and graphene oxide-modified carbon sheets (GO/CS), resulting in significantly reduced nanoparticle size and enhanced surface properties, particularly on the GO/CS substrate. The AuNPs/GO/CS electrode demonstrated high electrocatalytic activity, achieving the highest current density and fastest electron transfer in ferricyanide solution. Its improved proton reduction efficiency in the hydrogen evolution reaction (HER) and high sensitivity toward hydrogen peroxide (H_2O_2) detection confirm its versatility as an advanced electrochemical platform. Furthermore, this electrode presents strong potential for

applications in biosensing, portable point-of-care devices, and renewable energy conversion systems.

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