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Development and Characterization of Gold Nanoparticle-Modified SPCEs for the Electrochemical Sensing

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Abstract— Gold nanoparticles were successfully synthesized by reducing HAuCl4 using jengkol extract as a reductant. The synthesized gold nanoparticles were characterized using UV-vis spectroscopy and particle size analyzer (PSA). The synthesized gold nanoparticles were deposited on the screen-printed carbon electrode (SPCE) substrate using 2 methods: drop casting and differential pulse voltammetry over a potential range of -1500 to 600 mV, at a scan rate of 100 mV/s for 5 cycles. The surface plasmon resonance (SPR) band of the UV- Vis spectrum at 530.7 nm confirmed the presence of gold nanoparticles. The results of Au nanoparticle characterization using PSA showed that the size of the Au-NPs formed was 33.5 nm with an optimal HAuCl4 concentration of 0.20 mM. Characterization of gold nano-deposited SPCE was carried out by measuring the peak current of the 1 mM K3Fe(CN)6/K4Fe(CN)6 system in a KCl electrolyte solution (0.1 M) using cyclic voltammetry over a potential range of -500 to 1000 mV, with a scan rate of 100 mV/s for 5 cycles. The gold nanoparticles deposited by differential pulse voltammetry exhibited a higher current response than drop-casting deposition.

Keywords— *Cyclic voltammetry; Differential pulse voltammetry; Drop casting; Gold nanoparticles; Screen printed carbon electrode*

1. INTRODUCTION

Gold nanoparticles (Au-NPs) have received widespread attention in the development of sensor technology due to their unique properties, such as high conductivity, chemical stability, and the ability to enhance electron transfer on the electrode surfaces [1]. One environmentally friendly approach to synthesizing Au-NPs is through green synthesis, using natural materials as reducing and stabilizing agents. In this context, jengkol extract (*Archidendron pauciflorum*) becomes an interesting candidate because it contains phenolic compounds and organic sulfur that have the potential to reduce metal ions into nanoparticle forms [2].

In electrochemical applications, Au-NPs are often utilized to modify the surface of electrodes, one of which is the Screen-Printed Carbon Electrode (SPCE). SPCE is an economical and portable sensor platform that is highly suitable for in-situ analysis because it can be mass-produced and used once. Modification of SPCE with Au-NPs has been proven to enhance the sensitivity and selectivity of electrochemical sensors due to the increase in active surface area and electron transfer rate [3-4].

Voltammetry techniques, especially cyclic voltammetry (CV) and differential pulse voltammetry (DPV), are the primary methods in the characterization of modified electrodes. Voltammetry allows the measurement of current in response to an applied voltage, thus providing information about redox reactions, electron transfer rates, and the surface properties of the electrode. In particular, DPV is more sensitive than CV and is very useful in trace compound analysis because it can reduce capacitive current and improve the signal-to-noise ratio (SNR) [5].

The electrochemical method for measuring heavy metals offers several advantages, such as fast analysis times, high sensitivity, ease of use, and low cost. Several studies have been conducted on the electrochemical measurement of heavy metals using various electrodes. Some electrodes reported to have been used for electrochemical measurement of heavy metal include: Au electrode modified with nanoparticles [6]; gold nanoparticles (AuNPs)-modified with glassy carbon electrode (GCE) [7]; glassy carbon electrode (GCE) coated with Ni nanoparticles [8]; Be/Be^{2+} electrode pairs [9]; determine the speciation of chromium [10].

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However, in recent years, advancements in screen-printed microfabrication technology have enabled the mass production of various highly reproducible screen-printed electrodes (SPEs) at low cost and easy availability. The use of SPEs with working electrode surfaces modified with nanomaterials, such as carbon nanofibers (CNFs), carbon nanotubes (CNTs), and graphene (GPH), can increase the electrode surface area and enhance electron transfer. This positively impacts analytical performance. Some studies on the modification of SPCE with metal nanoparticles include the modification of SPCE with AuNP to detect glycated hemoglobin [11]. Most studies on the modification of SPE with metal nanoparticles have been conducted to detect organic compounds. The detection of heavy metals using SPE modified with metal nanoparticles is still not widely done. Therefore, combining electroanalytical methods with SPE-modified with nanomaterials is an interesting and innovative option for measuring hexavalent chromium in wastewater samples.

This study aims to develop and characterize SPCE electrodes modified with biosynthesized Au-NPs using jengkol leave extract. Additionally, this study compares the effectiveness of two nanoparticle deposition techniques on the SPCE surface, namely the drop casting method and electrochemical deposition through differential pulse voltammetry. The evaluation was conducted based on the redox current response of the $K_3Fe(CN)_6/K_4Fe(CN)_6$ system, using voltammetry as the main characterization method. The results of this research are expected to contribute to the development of more sensitive, efficient, and environmentally friendly electrochemical sensors for analytical applications.

2. EXPERIMENTAL SECTION

2.1. Materials

The materials used in this study were jengkol leaves extract from previous research [12], $HAuCl_4$, $K_3Fe(CN)_6/K_4Fe(CN)_6$, and KCl from Merck.

2.2. Instrumentation

The instruments used in this study were analytical balance (Mettler Toledo), UV-visible spectrophotometer (Shimadzu) , particle size analyzer (Mictotrac), Potensiostat (eDAQ).

2.3. Procedure

The research method started with the synthesis of gold nanoparticles (AuNPs), characterization of AuNPs, deposition of SPCE using AuNPs, and the study of the electrochemical behavior of AuNP-deposited SPCE. Gold nanoparticles were synthesized using a 10% (w/v) jengkol leaf extract in distilled water as a natural reducing agent. The AuNP was characterized using a UV-visible spectrophotometer and particle size analyzer (PSA). The AuNP deposition process on the SPCE surface used two methods that were drop casting deposition and electrodeposition. The study of the electrochemical behavior of AuNP-modified SPCE (AuNP/SPCE) was conducted by measuring the peak current of the $K_3Fe(CN)_6/K_4Fe(CN)_6$ 1 mM system in KCl electrolyte solution (0.1 M) using cyclic voltammetry in the potential range of -500 to 1000 mV, with a scan rate of 100 mV/s for 5 cycles.

2.4. Preparation of 50.76 mM HAuCl4 Solution

The 50.76 mM $HAuCl_4$ solution was prepared by dissolving 1 g of noble metal in 100 mL of aqua regia with the aid of heating, followed by the addition of distilled water to 100 mL and homogenization.

2.5. Characterization of AuNP

Each of the Au nanoparticle solutions was then characterized for its maximum wavelength using a UV-vis spectrophotometer, with a scanning wavelength of 400 to 600 nm. The particle size of the formed nanoparticles was characterized using a particle size analyzer (PSA).

2.6. Deposition of SPCE/AuNP by Chronoamperometry

In a 50 mL volumetric flask, a 10 mM $HAuCl_4$ solution was prepared to deposit Au nanoparticles on the SPCE electrode. The SPCE electrode was coated with 40 µL of the 10 mM $HAuCl_4$ solution. Then, using differential pulse voltammetry, Au nanoparticles were electrochemically deposited at a potential range of (-1500)–600 mV, with a scanning rate of 100 mV/s for five cycles.

2.7. Deposition of SPCE/AuNP by Drop Casting Method

A total of 40 µL of 0.2 mM nano-Au solution was dropped onto the SPCE electrode and then left to dry. This process was repeated 5 times.

2.8. Study of Electrochemical Behavior of SPCE/AuNP

Each SPCE electrode modified with Nano Au using drop casting and differential pulse voltammetry techniques was then characterized using a 1.0 mM $K_3Fe(CN)_6$ solution in 0.10 M KCl with the cyclic voltammetry technique at a potential range of (-500)–1000 mV, with a scanning rate of 100 mV/s for 5 cycles. Characterization was also carried out for unmodified SPCE electrode (bare SPCE).

3. RESULT AND DISCUSSION

3.1. Synthesis of Gold Nanoparticles (AuNP)

The synthesis of gold nanoparticles (AuNP) was carried out using the reduction method of $HAuCl_4$ with

jengkol extract as the reducer. The synthesis process of $HAuCl_4$ into AuNP is presented in **Fig. 1.**

Fig. 1 depicts the green synthesis process of gold nanoparticles (AuNPs) utilizing *Archidendron pauciflorum* (jengkol) leaf extract as a natural reducing agent. Initially, a 0.20 mM $HAuCl_4$ solution appeared colorless. Upon heating and the subsequent addition of the plant extract, the solution developed a reddish-purple color. This color shift is a preliminary visual indication of gold nanoparticle formation, attributed to surface plasmon resonance (SPR) phenomenon—an optical property characteristic of noble metal nanoparticles such as gold [13].

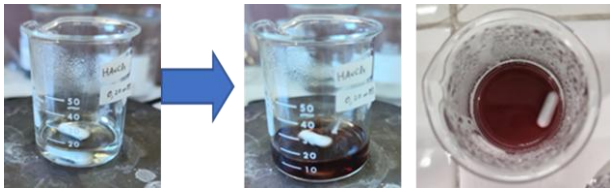

Fig. 1. Synthesis of AuNP by $HAuCl_4$ reduction method

Conversely, the control sample—prepared by mixing heated distilled water with jengkol extract in the absence of $HAuCl_4$—displayed a yellowish-brown hue that resembled the native color of the extract itself. The nonappearance of the reddish-purple tone in this control indicates that the observed color change is not solely caused by the extract or thermal treatment. Instead, it indicates that the formation of gold nanoparticles is specifically induced by the reduction of Au^{3+} ions to elemental gold (Au^0), facilitated by the bioactive compounds in the extract [14].

Phytochemicals present in the jengkol extract, such as phenolic acids, flavonoids, ascorbic acid, and sulfur-containing molecules, are known to act as electron donors capable of reducing metal ions. In addition to initiating the reduction process, these compounds also serve as capping agents that bind to the nanoparticle surface and preventing aggregation. The efficiency of this reduction pathway is reflected by the rapid appearance of the SPR-associated color, indicating that jengkol extract enables a fast and environmentally friendly route for the synthesis of stable AuNPs.

3.2. Characterization of Structure and Morphology

The characterization of the synthesized AuNPs was performed using a UV-visible spectrophotometer by measuring the absorbance within the wavelength range from 400 to 600 nm. As presented in **Fig. 2**, the highest absorbance was observed at a concentration of 0.4 mM HAuCl4, with a maximum absorption peak at 535.20 nm and an absorbance value of 1.867, indicating the characteristic surface plasmon resonance (SPR) of gold nanoparticles. Further characterization was conducted using a particle size analyzer (PSA) to determine the hydrodynamic diameter of the synthesized AuNPs. The PSA measurement results are in **Fig. 2**.

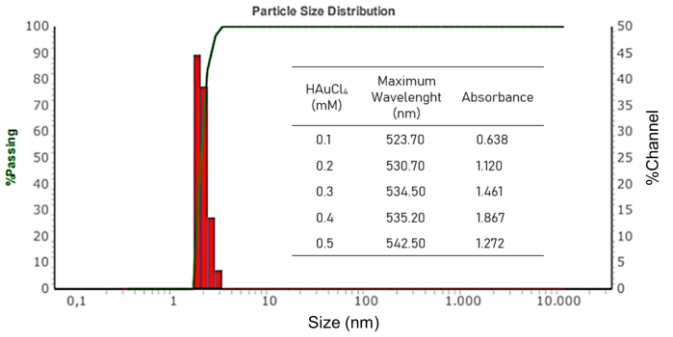

Fig. 2. Characterization of gold nanoparticles size using a particle size analyzer. **Inset Figures**: characterization results of the $HAuCl_4$ using the UV-visible spectrophotometer

The analysis revealed that particle size varied significantly depending on the concentration of the gold precursor ($HAuCl_4$), ranging from 1.94 nm to 169 nm. Remarkably, the smallest particle, with an average diameter of 1.94 nm, was obtained at a concentration of 0.5 mM $HAuCl_4$. This observation suggests that jengkol leaf extract is highly effective as a reducing agent, facilitating the conversion of Au^{3+} to Au^0 under mild and environmentally friendly conditions. The efficiency of this bioreduction process is closely linked to the phytochemical components present in the extract, such as phenolic compounds, flavonoids, ascorbic acid, and sulfur-containing molecules, which serve both as electron donors and particle-stabilizing agents [2,13].

The small and uniform size of the synthesized nanoparticles is particularly advantageous for electrochemical sensing applications. In sensor systems based on Screen-Printed Carbon Electrodes (SPCE), smaller AuNPs offer a larger effective surface area and improved electron transfer properties, thereby enhancing both the sensitivity and the response time of the sensor system [1]. Furthermore, well-dispersed AuNPs ensure consistent electrode modification, a critical factor for reproducibility in sensor performance [13]. From an electrochemical perspective, AuNPs below 10 nm are often preferred due to their superior catalytic activity and ability to promote redox reactions at lower overpotentials [4].

The successful synthesis of such small particles using jengkol extract validates its capability as a green reducing agent. Furthermore, this finding highlights its promise for low-cost and sustainable production of nanomaterials tailored for biosensors and analytical chemistry applications [15].

The UV–Vis spectrophotometric analysis revealed an absorption peak observed at approximately 530.7 nm, confirming the successful formation of well-dispersed and stable gold nanoparticles (AuNPs). This peak corresponds to the characteristic surface plasmon resonance (SPR) phenomenon, which arises from the collective oscillation of free electrons at the surface of gold nanoparticles when exposed to incident light [16]. SPR is not only a signature feature indicating the

presence of AuNPs, but also provides valuable insight into the nanoparticles' size, shape, and surface environment [17].

The sharpness and intensity of the SPR peak further reflect the uniformity and colloidal stability of the synthesized nanoparticles (AuNPs). A narrow, symmetrical SPR band suggests that the synthesized AuNPs have a relatively consistent size and shape and, are well suspended without significant aggregation. In this study, the appearance of a sharp and well-defined SPR peak is consistent with the PSA results, indicating the particle sizes fall within the nanometer range. Generally, smaller and more uniform particles result in a blue-shifted SPR peak, while larger or aggregated particles cause a red shift and broader absorption bands due to increased scattering and polydispersity [18].

In this study, UV–Vis spectroscopy was employed as a critical preliminary tool to evaluate the optical quality of the synthesized nanoparticles before their integration into modified SPCEs (screen-printed carbon electrodes). The technique's sensitivity to subtle surface alterations also makes it ideal for monitoring functionalization processes and relevant surface interactions relevant in nanoparticle-based electrochemical sensors [19].

In summary, the UV–Vis data not only validate the successful synthesis of AuNPs but also indicate that the green reduction system using *Archidendron pauciflorum* (jengkol) leaf extract produces nanoparticles with suitable optical and morphological properties. These characteristics are essential for their further application in SPCE-based chemical sensing, particularly for detecting target analytes that rely on surface charge transfer mechanisms.

3.3. Study of Electrochemical Behavior of SPCE/AuNP

The characterization results of the SPCE working electrode modified with Au nanoparticles deposited from $HAuCl_4$ using the differential pulse voltammetry technique in the potential range (-1500) to 600 mV showed higher oxidation and reduction current responses compared to the unmodified SPCE electrode. This finding indicates that Au nanoparticles deposited on the SPCE electrode can enhance the potential current response of the working electrode. Furhtermore, the characterization of the $K_3Fe(CN)_6$ NAu-SPCE electrode using the drop casting vs DPV technique is presented in **Fig. 3.**

The characterization results of the working SPCE electrode modified with Au nanoparticles showed that the highest current response is obtained through Au deposition using the Differential Pulse Voltammetry technique at potentials ranging from (-1500) to 600 mV for 5 cycles.

The working electrode based on a screen-printed carbon electrodes (SPCEs) modified with gold nanoparticles (AuNPs) exhibited a significant enhancement of redox current response following the deposition of gold from $HAuCl_4$ precursor solution using the Differential Pulse Voltammetry (DPV) technique, within a potential range of −1500 to 600 mV. Compared to the unmodified SPCE, the AuNP-modified electrode generated substantially higher oxidation and reduction peak currents. This notable improvement can be attributed to the increased electroactive surface area and enhanced surface conductivity imparted by the presence of AuNPs, which together facilitate more efficient electron transfer processes [20].

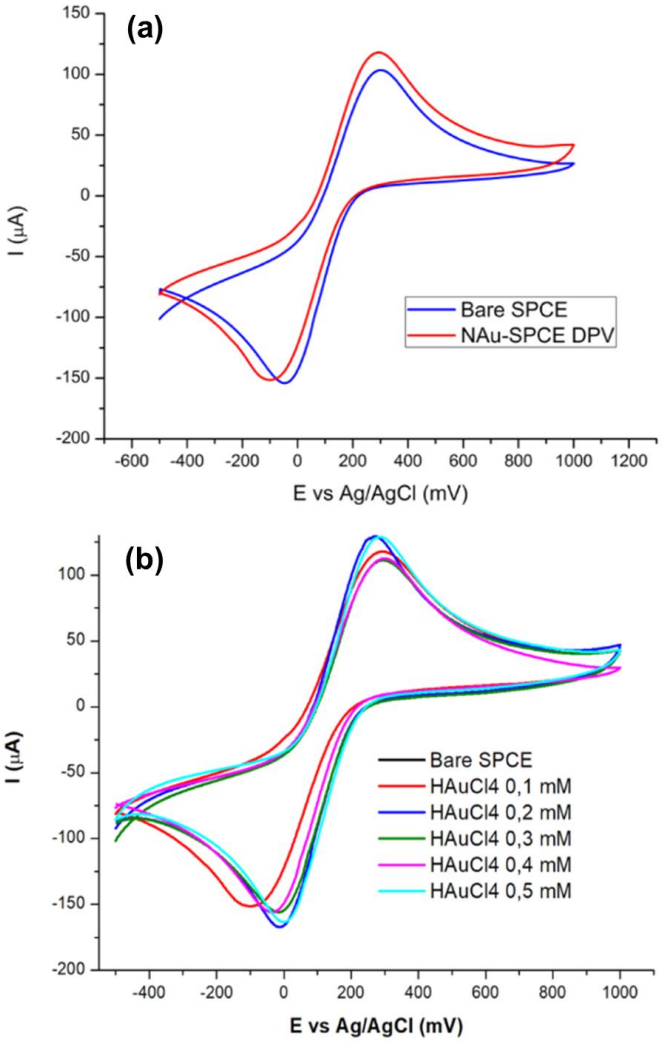

Fig. 3. Characterization result of the $K_3Fe(CN)_6$ NAu-SPCE electrode using (a) the drop casting technique, and (b) DPV technique

A comparative analysis of the two deposition methods—simple drop casting and electrochemical deposition via DPV—revealed that the DPV technique produced a superior current response. As illustrated in **Fig. 3**, the electrode modified through DPV yielded the highest peak current. These results sugggest that AuNPs are more uniformly distributed and more strongly adhered to the carbon surface of the electrode compared to the drop casting method. This finding is in

agreement with a study by Sharif et al. [21] who demonstrated that SPCE electrodes electrochemically modified with AuNPs showed more than double the redox current response compared to those modified through physical casting.

Gold nanoparticles enhance the kinetics of electrochemical reactions by providing additional active sites and reducing interfacial resistance between the electrode and the electrolyte. These advantages make AuNP-modified SPCEs a promising platform for high-performance electrochemical sensing, especially for the detection of analytes such as glucose, dopamine, heavy metals, and various environmental contaminants like pesticides [22].

The characterization results of the bare SPCE electrode and the Nano-Au SPCE are depicted in **Fig. 4.** The screen-printed carbon electrode (SPCE) modified with gold nanoparticles (AuNPs) using the drop-casting method with $HAuCl_4$ and *Archidendron pauciflorum* (jengkol) extract demonstrated a significant increase in redox current compared to the bare SPCE. The highest anodic and cathodic peak currents were observed at a precursor concentration of 0.2 mM $HAuCl_4$. This improvement is primarily attributed to two key mechanisms: an increase in the electroactive surface area (ECSA) and the formation of homogeneous, well-dispersed active sites on the electrode surface [23].

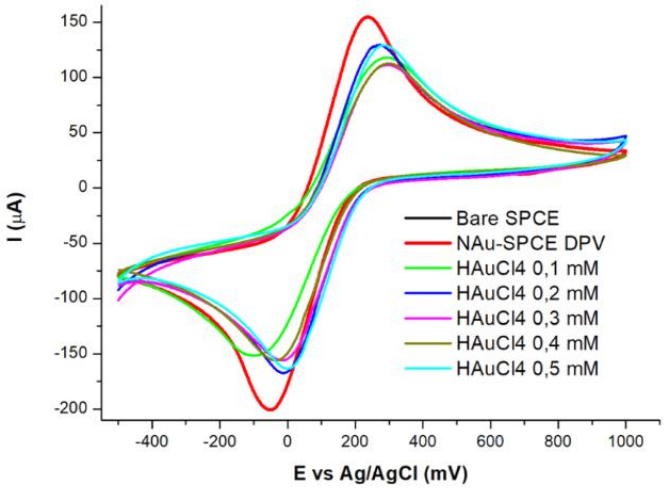

Fig. 4. The characterization results of the bare SPCE electrode vs. Nano-Au SPCE

A similar trend was reported by Attia et al. [24], who observed that increasing the $HAuCl_4$ concentration from 2 mM to 10 mM during electrodeposition enhanced the anodic peak current to 64 µA and expanded the ECSA to 21 mm² before reaching a saturation point. This result is consistent with the optimal response at 0.2 mM in our study, which may represent a threshold for maximal surface coverage and minimal aggregation of AuNPs.

Moreover, the morphology of the synthesized nanoparticles—particularly the formation of nanostructures such as spikes or dendrites—plays a significant role in determining their electrocatalytic behavior. AuNPs with spiky or branched surfaces offer more active sites, thereby promoting better charge transfer and improving the sensitivity of the electrode, particularly in the detection of analytes such as nitrite, dopamine, or ascorbic acid [25,26].

The application of electrochemical techniques like Differential Pulse Voltammetry (DPV) or Constant Potential Amperometry (CPA) has demonstrated superior control over nanoparticle deposition. These techniques enable fine-tuning of particle distribution, density, and adhesion, which contributes to stable and reproducible electrochemical behavior [27]. Such improvements are critical for applications in electrochemical sensors, as they demand a high signal-to-noise ratio and low detection limits.

Modification of electrodes with nanomaterials enhances the performance of electroanalytical sensors by increasing the active surface area and providing additional reaction sites [28]. The deposited nanoparticles improve electrical conductivity and accelerate the electron transfer rates, thereby increasing the charge storage capacity. These changes result in sharper and more sensitive voltammetric responses [29], enhancing the sensor's selectivity toward target analytes. Therefore, the increase in redox current observed at 0.2 mM $HAuCl_4$ supports the conclusion that this concentration yields AuNPs with ideal surface characteristics and electrochemical properties for SPCE modification. The combination of green synthesis using plant extracts and a controlled deposition strategy provides a robust platform for developing sensitive and reliable electrochemical sensors.

CONCLUSION

Gold nanoparticles (AuNPs) were successfully synthesized using *Archidendron pauciflorum* (jengkol) leaf extract as a green reducing agent, yielding particles within the nanometer range of 1–168 nm. These AuNPs were effectively deposited onto screen-printed carbon electrodes (SPCE) using two methods: physical (drop casting) and electrochemical (differential pulse voltammetry, DPV) methods. Among the tested approaches, electrochemical deposition via DPV at potentials between −1500 and 600 mV for five cycles provided the most significant enhancement in redox current response. This was confirmed by electrochemical characterization with the $K_3Fe(CN)_6$ redox probe. These findings highlighted the potential of AuNP-modified SPCEs, particularly those prepared via DPV, as a promising platform for the development of high-performance electrochemical sensors.

SUPPORTING INFORMATION

This article does not include any supplementary materials.

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CONFLICT OF INTEREST

The authors confirm the absence of any conflicts of interest regarding the publication of this article. The data supporting the findings of this study are available upon request from the corresponding author (APT).

AUTHOR CONTRIBUTIONS

The experimental work was performed by YD, APT, H, RM, MH, and UA. YD and APT were responsible for drafting and revising the manuscript. All authors have reviewed and approved the final version of the manuscript.

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