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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 $HAuCl_4$ using jengkol (*Archidendron pauciflorum*) extract as a reductor. The synthesized gold nanoparticles were characterized by 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) mV to 600 mV, scan rate of 100 mV/s for 5 cycles. The surface plasmon resonance (SPR) band of UV– Vis spectrum at 530.7 nm confirmed the presence of gold nanoparticles. The results of Au nanoparticle characterization using PSA show that the size of the Au-NPs formed is 33.5 nm with an optimal $HAuCl_4$ concentration of 0.20 mM. Characterization of gold nano-deposited SPCE was carried out by measuring the peak current of the 1 mM $K_3Fe(CN)_6/K_4Fe(CN)_6$ system in KCl electrolyte solution (0.1 M) using cyclic voltammetry over a potential range of (-500) mV to 1000 mV, scan rate 100 mV/s for 5 cycles. Gold nanoparticles deposited by differential pulse voltammetry showed a higher current response compared to 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 at the electrode surface [1]. One of the environmentally friendly approaches for synthesizing Au-NPs is through green synthesis using natural materials as reducing and stabilizing agents. In this context, jengkol (*Archidendron pauciflorum*) extract 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, 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 main 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. DPV, in particular, 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 [5].

The electrochemical method for measuring heavy metal is a technique that has several advantages, such as fast analysis time, high sensitivity, ease of use, and low cost. Several studies have been conducted on the electrochemical measurement of heavy metal using various electrodes. Some electrodes reported to have been used for electrochemical measurement of heavy metal: Au electrode modified with nanoparticles [6]; gold nanoparticles (AuNPs)-modified glassy carbon electrode (GCE) [7]; glassy carbon electrode (GCE) coated with Ni nanoparticles [8]; Be/Be^{2+} electrode pair [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 (SPE) at low cost and with easy availability. The use of SPE 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 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], HAuCl4, $K_3Fe(CN)_6/K_4Fe(CN)_6$, 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 begins with the synthesis of gold nanoparticles (AuNP), characterization of AuNP, deposition of SPCE using AuNP, 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 characterization of AuNP was conducted by a UV-visible spectrophotometer and particle size analyzer (PSA). The AuNP deposition process on the SPCE surface followed two steps, firstly drop casting deposition and secondly, 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 mV to 1000 mV, with a scan rate of 100 mV/s for 5 cycles.

2.4. Deposition of SPCE/AuNP by Drop Casting Method

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

2.5. 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 scan rate of 100 mV/s for 5 cycles. Characterization was also performed for the 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 HAuCl4 into AuNP can be seen in **Fig. 1.**

Fig. 1 illustrates the synthesis of gold nanoparticles (AuNPs) via a green reduction approach, in which *Archidendron pauciflorum* (jengkol) leaves extract serves as a natural reducing agent. Initially, the 0.20 mM $HAuCl_4$ solution appeared colorless. However, 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)—an optical property characteristic of noble metal nanoparticles like gold [13].

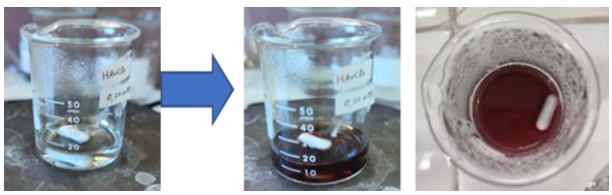

Fig. 1. Synthesis of AuNP by $HAuCl_4$ reduction method

In contrast, 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 absence of the reddish-purple tone in this control

demonstrates that the observed color change is not caused by the extract or heat alone, but rather by the specific reduction of Au^{3+} ions to elemental gold (Au^0) facilitated by bioactive compounds in the extract [14].

Phytochemicals such as phenolic acids, flavonoids, ascorbic acid, and sulfur-containing molecules present in the jengkol extract 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 prevent aggregation. The efficiency of this reduction pathway is evidenced 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 AuNP using a UV-visible spectrophotometer was carried out by measuring the absorbance of the synthesized AuNP at wavelengths ranging from 400 to 600 nm. The measurement results can be seen in **Fig. 2 (inset figure).** The characterization results using the UV-visible spectrophotometer show that the maximum wavelength with the highest absorbance is for the 0.4 mM $HAuCl_4$ concentration, at a wavelength of 535.20 nm with an absorbance of 1.867. The characterization of AuNP using a particle size analyzer (PSA). The measurement results of AuNP using PSA can be seen in **Fig. 2**.

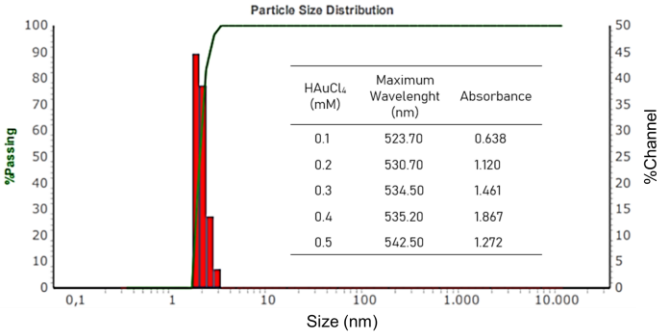

Fig. 2. Characterization of gold nanoparticles size using a particle size analyzer. **Inset Figures**: characterization results of the $HAuCl_4$ using the UV-visible spectrophotometer

The analysis reveals 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 particles 1.94 nm in diameter were produced at a concentration of 0.5 mM $HAuCl_4$. This observation suggests that jengkol leaves extract is highly effective as a reducing agent, facilitating the conversion of Au^{3+} to Au^0 under mild, environmental 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 have been shown to act both as electron donors and particle-stabilizing agents [2-13].

The small and uniform size of the 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, enhancing both the sensitivity and the response time of the sensor [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 not only validates its capability as a green reducing agent but also highlights its promise for low-cost, sustainable production of nanomaterials tailored for biosensors and analytical chemistry applications [15].

Based on the UV–Vis spectrophotometric analysis, an absorption peak observed at approximately 530.7 nm confirms 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 on 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 nanoparticle size, shape, and surface environment [17].

The sharpness and intensity of the SPR peak further reflect the uniformity and stability of the nanoparticles in colloidal form. A narrow, symmetric 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, which indicated that 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].

UV–Vis spectroscopy, as employed in this work, serves as a critical preliminary tool for assessing the optical quality of the synthesized nanoparticles before their integration into modified SPCEs (screen-printed carbon electrodes). The technique's sensitivity to subtle surface changes also makes it ideal for monitoring functionalization processes and 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) leaves 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 HAuCl4 using the differential pulse voltammetry technique at potentials ranging from (-1500) to 600 mV show higher oxidation and reduction current responses compared to the unmodified SPCE electrode. This indicates that the Au nanoparticles deposited on the SPCE electrode can enhance the potential current response of the working electrode. The characterization of the $K_3Fe(CN)_6$ NAu-SPCE electrode using the drop casting vs DPV technique can be seen in **Fig. 3.**

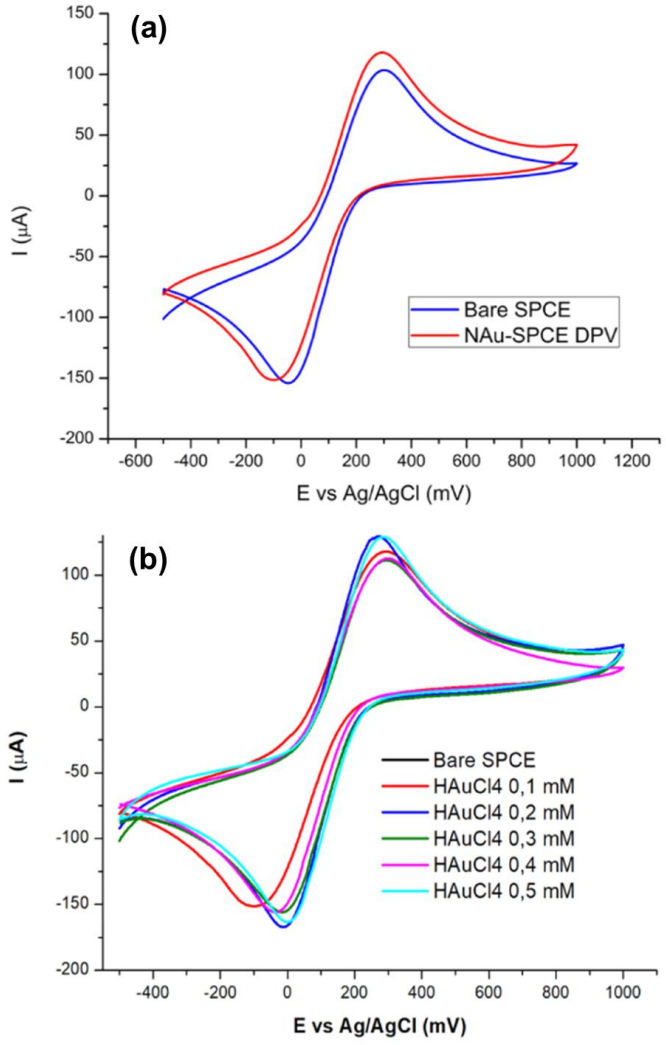

Fig. 3. Characterization result of the $K_3Fe(CN)_6$ NAu-SPCE electrode using (a) the drop casting technique, and (b) DPV technique

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

The working electrode based on screen-printed carbon electrodes (SPCE) modified with gold nanoparticles (AuNPs) exhibited a significant enhancement in redox current response following the deposition of gold from an $HAuCl_4$ 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].

A comparative analysis of 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, suggesting that AuNPs were 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] which demonstrated that SPCE electrodes electrochemically modified with AuNPs showed more than double the redox current response compared to those modified through physical casting.

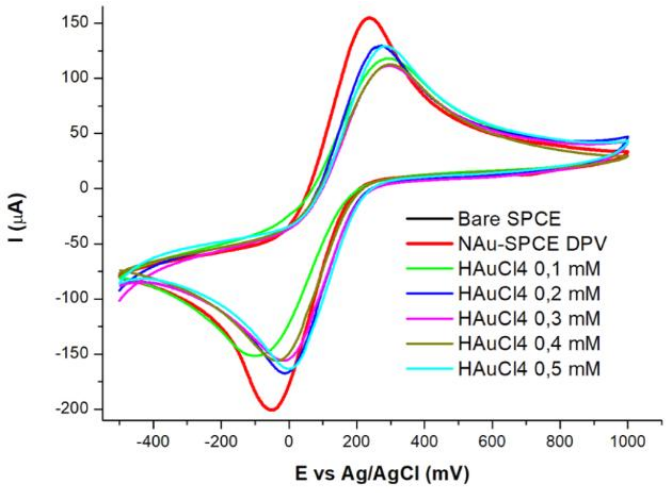

Fig. 4. The characterization results of the bare SPCE electrode vs. Nano-Au SPCE

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.**

As shown in **Fig. 4,** the screen-printed carbon electrode (SPCE) modified with gold nanoparticles (AuNPs) using a drop-casting method with $HAuCl_4$ and *Archidendron pauciflorum* 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: the increase in the electroactive surface area (ECSA) and the formation of homogeneous, well-dispersed active sites on the electrode surface [23].

A similar trend was reported by Attia et al. [24], who observed that increasing $HAuCl_4$ concentration from 2 mM to 10 mM during electrodeposition enhanced the anodic peak current up to 64 µA and expanded the ECSA to 21 mm² before reaching a saturation point. This 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 provide more active sites, promoting better charge transfer and improving the sensitivity of the electrode, especially in the detection of analytes like nitrite, dopamine, or ascorbic acid [25-26].

The use of electrochemical techniques like Differential Pulse Voltammetry (DPV) or Constant Potential Amperometry (CPA) has also been shown to offer superior control over nanoparticle deposition. This enables fine-tuning of particle distribution, density, and adhesion, which contributes to stable and reproducible electrochemical behavior [27]. These improvements are critical for applications in electrochemical sensors, where a high signal-to-noise ratio and low detection limits are essential.

Modification of electrodes with nanomaterials enhances the performance of electroanalytical sensors by increasing the active surface area and providing additional reaction sites [28]. Deposited nanoparticles improve electrical conductivity and accelerate electron transfer rates, thereby increasing 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 extract 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) leaves 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 both 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, as confirmed by electrochemical characterization with the $K_3Fe(CN)_6$ redox probe. These findings highlight the potential of AuNP-modified SPCEs, particularly those prepared by 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 related to the publication of this article. The data supporting the findings of this study are available upon request from the corresponding author (YD).

AUTHOR CONTRIBUTIONS

The experimental work was carried out 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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