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Evaluation of Scan Rate Influence on Cyclic Voltammograms of Copper Electrodes Coated with PS-CS-GA/ZnO Nanocomposite

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Abstract

Copper (Cu) is widely utilized as an electrode material due to its high conductivity and relatively low cost. This study aims to develop nanocomposite-modified Cu electrodes using polysulfone (PS), chitosan (CS), glutaraldehyde (GA), and zinc oxide (ZnO) to form PS-CS-GA/ZnO-NP modified Cu electrodes. The nanocomposite was prepared in the proportions of 48.8% PS, 24.4% CS, 24.4% ZnO, and 2.4% GA using the phase inversion method. The characterization of the PS-CS-GA/ZnO nanocomposite-modified Cu electrode was conducted using cyclic voltammetry with a 0.01 M $K_3[Fe(CN)_6]$ *in 0.1 M KCl. The cyclic voltammogram profile was obtained at a scan rate of 25 mV/s, exhibiting an ipa/ipc ratio of 0.93 within a potential of -1.5 to +0.4 V vs Ag/AgCl over 15 cycles. The FTIR spectrum of the PS-CS-GA/ZnO nanocomposite thin film displayed a peak at 1668* cm^{-1}*, indicating the presence of C=N groups, which suggests a cross-linking interaction between CS and GA.*

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Keywords: *chitosan, cyclic voltammetry, modified electrode, nanocomposite, polysulfone*

1 Introduction

These sensors are widely employed in various industries, including the food and pharmaceutical sectors, due to their simple preparation and operation compared to spectrophotometric (UV-Vis, MS) and chromatographic (GC, HPLC) [1].

Numerous studies on electrochemical sensors have utilized inert metals such as Au, Ag, and Pt; however, these metals are relatively expensive. Copper (Cu) is an alternative metal that offers high conductivity at a lower cost, making it a suitable candidate for modification [2].

Chitosan (CS), an abundant and environmentally friendly biopolymer, is hydrophilic but exhibits unstable mechanical properties, necessitating its modification [3]. In this study, ZnO nanoparticles (NPs) are utilized to modify the Cu electrode, enhancing its sensitivity [4]. In this research, the composite material that will be modified on the Cu electrode is ZnO NP which can increase the sensitivity of the modified electrode. Glutaraldehyde (GA) is employed to cross-link with the amine groups in chitosan [5]. while the incorporation of polysulfone (PS) polymer improves the composite's mechanical strength and structural stability [6]. while polysulfone (PS) is added to improve mechanical strength and structural stability [7].

Therefore, the preparation of nanocomposite-modified Cu electrodes, specifically PS-CS-GA/ZnO is anticipated to serve as effective electrochemical sensors for various applications.

The development of this biosensor is closely related to our previous research, including the development of chitosan as a membrane and the isolation of polyphenol oxidase from food materials, as a continuous innovation in biosensor development research for various applications [8–11].

2 Method

2.1 Material

The chemicals utilized in this research, all of pro-analysis grade (p.a) and sourced from Sigma Aldrich, played a pivotal role in facilitating the experimental procedures. Chitosan extracted from blue crab shells (*Rajungan/ P. pelagicus*) with a particle size of 200-300 mesh, ZnO (~88%) synthesized in situ using the sol-gel method, glutaraldehyde ($OHC(CH_2)_3CHO$), glacial acetic acid (CH_3COOH), N-methyl-2-pyrrolidone, NMP (C_5H_9NO), potassium chloride (KCl), potassium ferricyanide ($K_3[Fe(CN)_6]$), and double-distilled water (aquabidest) constituted the core components for the research endeavors. Additionally, the polysulfone Polysulfone ($[C_6H_4\text{-}4\text{-}C(CH_3)2C_6H_4\text{-}4\text{-}OC_6H_4\text{-}4\text{-}SO_2C_6H_4\text{-}4\text{-}O]n$) (U-DEL®P-1700 NT LCD) were obtain from Solvay served as a crucial material for conducting the experimental investigations outlined in this study.

2.2 Preparation of PS-CS-GA/ZnO Nanocomposite

Solution A (CS-GA-ZnO): A 2% CS solution and a 0.6% ZnO solution in 1% acetic acid are mixed and heated to 50-69°C with stirring (100-300 rpm) until homogeneous. Subsequently, 1 mL of 25% GA solution is added and heating continues until fully mixed.

Solution B (10% PS in NMP): Dissolve 2 g of PS in 10 mL of NMP at 80°C, stirring at 100-300 rpm for 1-1.5 hours until homogeneous.

Solution C (PS/CS-GA-ZnO): Solution A and Solution B are combined at 80°C and stirred for 5-7 hours until homogeneous, resulting in a PS/CS-GA-ZnO paste.

The final composition of the nanocomposite remains at 48.7% PS, 24.4% CS, 24.4% ZnO, and 2.4% GA, achieved through the mixture of Solution A (CS, ZnO, GA) with Solution B (PS).

2.3 Instrumentation

Standard laboratory glassware was used throughout the experiments. Key equipment included a commercial copper rod electrode, polished with 1000 grit sandpaper, with a length of 10 cm and a diameter of 2 mm. Subsequently, it was inserted into a capillary tube measuring 8 cm in length and 5 mm in diameter.

2.4 Measurement using cyclic voltammetry technique

Cyclic voltammetry was conducted using a 25 mL mini cell with a three-electrode system, consisting of a working electrode (WE) of PS-CS-GA/ZnO-modified copper (Figure 1), a platinum wire rod as the auxiliary electrode (AE), and an Ag/AgCl reference electrode (RE). These components were connected to an Electrochemical workstation type-1030 potentiostat/galvanostat Merck INGSENS-TM, controlled by a computer running the cyclic voltammetry software [12,13]. Figure 2 showed the ilustration of this mini cell.

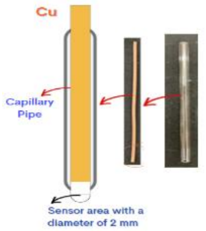

Figure 1. The construction of the PS-CS-GA/ZnO film modified electrode

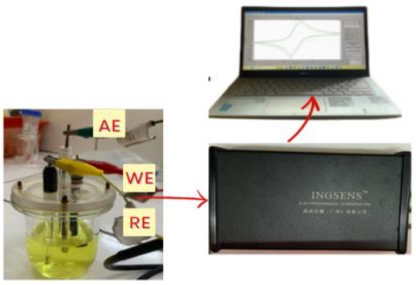

Figure 2. Cyclic voltammetry was conducted using a 25 mL mini cell with a three-electrode system. These components are crucial for analyzing the PS-CS-GA/ZnO nanocomposite-modified copper electrodes as the working electrode

2.5 Modification of PS-CS-GA/ZnO Nanocomposite on Cu Electrode

In this study, a PS-CS-GA/ZnO paste, designated as solution C, was synthesized by mixing solutions A and B. The synthesis process involved stirring the PS-CS-GA/ZnO nanocomposite at a rotation speed of 300-400 rpm for 5-7 hours until achieving a homogeneous mixture. The resulting solution appeared clear, yellowish, and thick. Subsequently, a thin film layer was applied onto a cleaned Cu wire using the drop method, employing the phase inversion technique, where PS-CS-GA/ZnO nanocomposite paste was deposited onto the sensor electrode area. The thin film layer was allowed to dry on the sensor area for a few minutes. The visual form of the modified Cu electrode with PS-CS-GA/ZnO nanocomposite is shown in Figure 3.

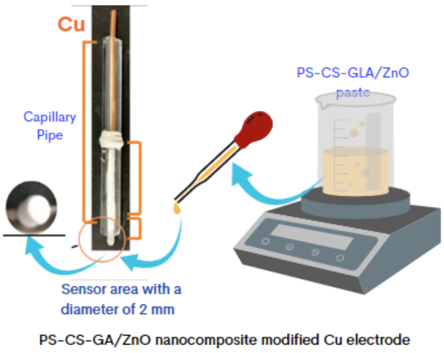

Figure 3. The PS/CS-GA-ZnO film-modified Cu electrode was prepared via the phase inversion method

2.6 *Characterization of PS-CS-GA-ZnO*

Characterization of the PS-CS-GA-ZnO modified Cu electrode via cyclic voltammetry was conducted using a 0.01 M $K_3[Fe(CN)_6]$ in 0.1 M KCl as the supporting electrolyte. The cyclic voltammogram profile of the PS-CS-GA-ZnO nanocomposite modified Cu electrode was evaluated in a 0.01 M $K_3[Fe(CN)_6]$ in 0.1 M KCl across a potential range of -1.5 to +0.4 V, employing scan rates from 5 to 25 mV/s.

Functional groups in the PS-CS-GA/ZnO nanocomposite thin films were characterized using ATR-FTIR. The IR spectrum of the PS-CS-GA/ZnO film was obtained using an FTIR Prestige 21 Shimadzu instrument.

3 Result and Discussion

3.1 *Cu electrode modified with PS-CS-GA/ZnO nanocomposite*

The CS polymer and zinc oxide (ZnO) are known to readily dissolve in 1-2% acetic acid [14]. Unmodified CS is highly sensitive to environmental changes and exhibits poor mechanical properties. To address these limitations and improve the properties of CS, the addition of supplementary materials becomes essential [15]. In this study, supplementary materials such as ZnO nanoparticles (NPs), glutaraldehyde (GA), and polysulfone (PS) were incorporated. The possible reaction between ZnO nanoparticles and CS is shown in Figure 4.

The incorporation of glutaraldehyde (GA) into the CS-ZnO nanocomposite is facilitated by its role as a cross-linking agent within chitosan (CS). The cross-linking mechanism involves the amino groups (NH_2) in CS reacting with the carbon atoms in the aldehyde groups of GA, replacing the oxygen to form C=N bonds, commonly known as Schiff bases. This reaction also releases H_2O molecules as byproducts [16]. The resulting covalent bonds between the amino and aldehyde groups are characterized by their strength, high efficiency, low toxicity, substantial binding capacity, and ease of formation [17]. Moreover, the cross-linking of CS with GA enhances the hydrophobicity of CS, thereby modifying its mechanical properties [18,19].

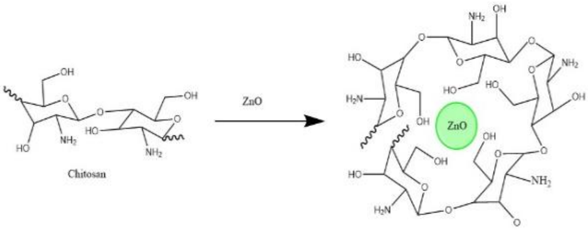

Figure 4. Chitosan-ZnO nanocomposite presented in simplified form [16]

The addition of polystyrene (PS) to the CS-GA/ZnO nanocomposite is anticipated to enhance the electrode's performance as a sensor. PS is a polymer known for its mechanical strength, thermal stability, and inertness [20].

3.2 *Characterization of Working Electrodes using Cyclic Voltammetry*

The characterization of PS-CS-GA/ZnO nanocomposite electrodes was conducted using the cyclic voltammetry method. Cyclic voltammetry involves three main components: a potentiostat, a computer, and an electrochemical cell. During the process, the potentiostat applies a potential to the WE, which in this case is the Cu-modified PS/CS-GA/ZnO nanocomposite, and this potential is compared with the potential at the Ag/AgCl. As a result, a current flows through the electrolyte solution in the mini electrolysis cell, and a redox reaction takes place on the surface of the working electrode with the assistance of the Pt as AE [21].

The cyclic voltammogram illustrates the relationship between current (I) and potential (E), where the x-axis is the potential (E) and the y-axis is the response to the potential, namely current (I) [22]. This graphical representation provides insight into the electrochemical behavior of the PS-CS-GA/ZnO nanocomposite electrodes. The current response is indicative of the redox processes occurring at the electrode surface, offering valuable information regarding the electrochemical properties and performance of the modified electrodes.

The electrochemical cell utilized in cyclic voltammetry comprises an electrolyte solution, an analyte solution, and three electrodes [23]. In this study, the redox reaction mechanism of the WE in the analyte solution, specifically 0.01 M $K_3[Fe(CN)_6]$ in 0.1 M KCl, was investigated. The measurement potential was adjustable within the range of -2.0 to 1.0 V vs Ag/AgCl. Optimal conditions were determined within the potential range of -1.5 to +0.4 V vs Ag/AgCl, with a scan rate of 25 mV/s, as evidenced by the resulting I_{pa}/I_{pc} ratio approaching 1 (Table 1).

Table 1. The values of I and E of the cyclic voltammogram of the PS-CS-GA/ZnO nanocomposite modified Cu electrode in 0.01 M $K_3[Fe(CN)_6]$ in 0.1 M KCl at varying scan rates

Scan rate (mV/s)	I_{pa} (μA)	I_{pc} (μA)	E_{pa} (V)	E_{pc} (V)	E_{pa}-E_{pc} (V)	$\|I_{pa}/I_{pc}\|$
5	40.70	-29.08	-0.36	-0.67	0.31	1.40
10	58.10	-49.95	-0.32	-0.73	0.41	1.16
15	54.10	-67.30	-0.28	-0.73	0.45	0.80
20	63.00	-70.95	-0.29	-0.74	0.45	0.89
25	75.50	-80.80	-0.27	-0.78	0.51	0.93

In cyclic voltammetry, the Ag/AgCl maintains a fixed potential, ensuring stable reference conditions throughout the measurement process. Meanwhile, the diffusion current observed in the cyclic voltammogram is facilitated by a Pt electrode serving as an AE. This Pt electrode conducts electric current from the signal source through the solution to the WE [24]. The current response can be depicted on graphs of current versus potential and current versus time. The current versus potential graph is called a cyclic voltammogram which has 2 peaks, namely the anodic peak current (I_{pa}) where oxidation occurs, and the cathodic current peak (I_{pc}) where reduction occurs. The reaction that occurs is written in Equation 1 and 2.

Red.: $[Fe(CN)_6]^{3-} + e^- \rightarrow [Fe(CN)_6]^{4-}$ (1)
Ox.: $[Fe(CN)_6]^{4-} \rightarrow [Fe(CN)_6]^{3-} + e^-$ (2)

The peak current in the voltammogram is governed by the Nernst Equation (Equation 1), which relates the cell potential (E) to the standard potential (E_0) and the analyte's redox activities, involving constants like F (Faraday constant), R (gas constant), n (number of electrons), and T (temperature) [22]. In this study, the reduction of Fe^{2+} to Fe^{3+}involves the transfer of one electron.

$$E = E^0 + \frac{RT}{nF} ln \frac{(Ox)}{(Red)} \qquad (3)$$

The cyclic voltammogram (Figure 5) illustrate the response of the PS-CS-GA/ZnO nanocomposite-modified Cu electrode to 0.01 M $K_3[Fe(CN)_6]$ in 0.1 M KCl, demonstrating typical I_{pa} and I_{pc}. An upward arrow denotes an anodic current, reflecting an oxidation reaction, whereas a downward arrow denotes a cathodic current, indicating a reduction reaction.

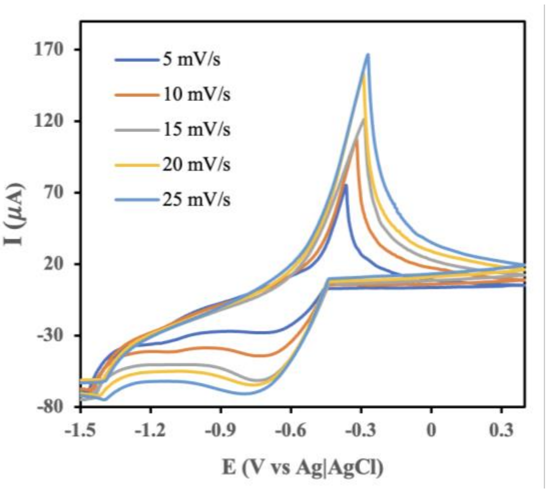

Figure 5. Cyclic voltammogram of PS-CS-GA/ZnO nanocomposite modified electrode at varying scan rates in 0.01 M $K_3[Fe(CN)_6]$ in 0.1 M KCl

Based on Table 1 and Figure 5, the increase in scan rate correlates with higher peak reduction currents, with values of -29.08, -49.95, -67.30, -70.95, and -80.80 μA, respectively. Since the electrochemical reaction involves the transfer of one electron, a scan rate with an $I_{pa}/I_{pc} = 1$ indicates a relatively stable electrode response. In this experiment, a scan rate of 25 mV/s exhibited a relatively stable electrode response, with an $I_{pa}/I_{pc} = 0.93$, compared to other scan rates of 5, 10, 15, and 20 mV/s.

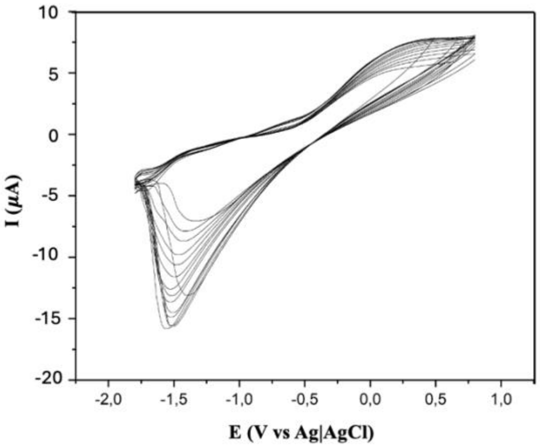

Figure 6. The cyclic voltammogram profile shows how cycle number affects the PS-CS-GA/ZnO modified Cu electrode over 15 cycles, from 1.8 to +0.8 V vs Ag|AgCl at a 5 mV/s scan rate. This was analyzed in 0.01M $K_3[Fe(CN)_6]$ in 0.1 M KCl

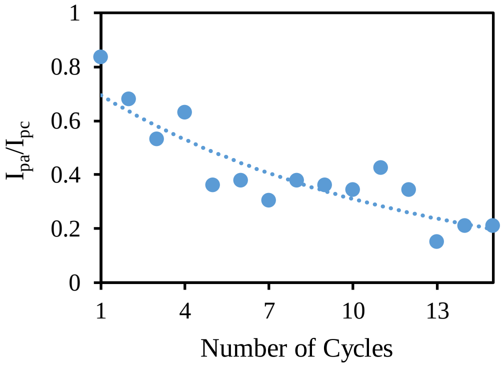

Figure 7. The relationship between the IPA/IPC ratio decreases as the number of cycles increases

The determination of the number of cycles for the PS-CS-GA/ZnO nanocomposite-modified Cu working electrode involved cyclic voltammetric scanning of 15 cycles within the potential range of -1.8 to +0.8 V vs Ag|AgCl, with a scan rate of 5 mV/s, as depicted in Figures 6 and 7. This analysis aimed to evaluate the electrode's performance in detecting 0.01 M $K_3[Fe(CN)_6]$ in 0.1 M KCl by assessing the generated current within the specified potential range, providing insights into the electrode's sensitivity and suitability for electrochemical sensing applications.

Observation of the cyclic voltammogram indicates that the ipa/ipc ratio tends to decrease from 1 as the number of cycles increases (Figure 9). This decline is attributed to the reduction in Fe concentration in the $K_3[Fe(CN)_6]$ in 0.1 M KCl solution, leading to a decrease in current production. In cyclic voltammetry, the generated current is directly proportional to the chemical concentration in the solution [23]. Moreover, increasing the number of cycles may also induce damage to the electrode, a factor influenced by the electrode's characteristics [25].

3.3 *FTIR spectra of PS-CS-GA/ZnO nanocomposite thin films*

Figure 8 shows the FTIR spectrum of the PS-CS-GA/ZnO nanocomposite Film, indicating specific absorption characteristics within the film.

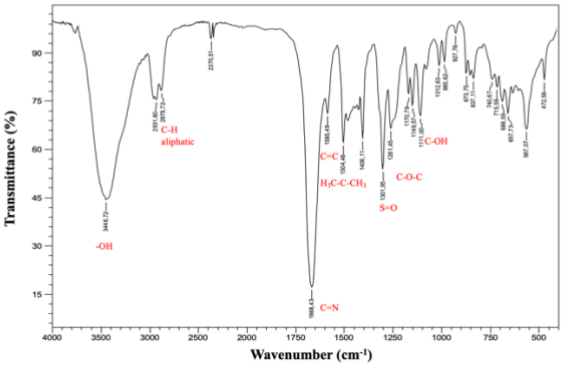

Figure 8. FTIR spectra of PS-CS-GA/ZnO nanocomposite thin films

FTIR analysis within the wavenumber range of 4000-400 cm^{-1} was conducted to explore potential interactions following the addition of PS polymer to the CS-GA-ZnO nanocomposite mixture. Kumar et al. [26] elaborated on CS characteristics, highlighting a peak at 3200-3400 cm^{-1} (-OH strain), contrasting with our study's observation of a peak at 3448 cm^{-1}. Moreover, a peak at 2879 $cm^{-1}$was identified for aliphatic C-H strain, slightly deviating from the anticipated 2872 cm^{-1}. Furthermore, the presence of a C-O-C bending group was indicated by absorption at 1261 cm^{-1}, while C-OH stretching was detected at 1111 cm^{-1} [27].

At a wavenumber of 2931 cm^{-1} (aromatic C-H stretch), our findings correspond to those reported by Kumar et al. [26] . Similarly, the peak at 1261 cm^{-1}, indicating the stretch of the C-O-C ether group, exhibited consistent values with our study. Furthermore, the peak at 1302 cm^{-1}, representing asymmetric S=O strain, closely resembled the value obtained in our investigation, recorded at 1301 cm^{-1}. Additionally, the peak at 1261 cm^{-1}, representing the stretch of the C-O-C ether group, exhibits a consistent peak value with our research.

Additionally, Lusiana et al [28] examined the FTIR spectrum of the PS membrane, identifying a peak at 1490 cm^{-1}, while our research observed a peak at 1504 cm^{-1} corresponding to the CH_3-C-CH_3 group. Moreover, the peak at 1588 cm^{-1} signifies absorption of the C=C aromatic benzene group, whereas our study recorded a peak at 1585 cm^{-1}. A peak at 1654 cm^{-1} was identified, indicating the presence of C=N stretching (Schiff base), confirming cross-linking between CS and GA. In our research, a peak at 1668 cm^{-1} was observed [29].

4 Conclusion

The PS-CS-GA/ZnO nanocomposite modified Cu electrode was prepared via phase inversion, resulting in a clear yellowish viscous film. Cyclic voltammetry revealed an optimum scan rate of 25 mV/s within the potential range of -1.5 to +0.4V, with an ipa/ipc ratio of 0.93. The number of electrode cycles decreases with increasing cycle number. FTIR analysis confirmed the presence of the C=N group and crosslinking between CS and GA, indicated by a peak at 1668 cm^{-1}.

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