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Preparation and Characterization of Chitosan/Titanium Dioxide Film for Electrochemical Sensing Applications

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ABSTRACT

Chitosan/TiO_2-modified electrodes were characterized using the cyclic voltammetry method. The electrodes were fabricated through an electrodeposition technique to produce a coating on the substrate surface. TiO_2 concentrations of 50, 100, 150, 200, and 250 mg/L were used. The chemical structure and functional group interactions of the films were characterized using Fourier Transform Infrared (FTIR) spectroscopy, which revealed characteristic chitosan functional groups and Ti–O–Ti stretching bands, indicating the dispersion of TiO_2 within the chitosan matrix. The electrochemical performance of the electrodes was evaluated in phosphate-buffered saline (PBS) solution at pH 7. The results showed that the chitosan/TiO_2 concentration variation of 150 mg/L produced the highest anodic current response, while the optimum scan rate was obtained at 75 mV/s. A linear relationship between peak current and scan rate was obtained, with a regression equation of $y = 0.0014x + 0.3788$ and a coefficient of determination (R^2) of 0.9869, indicating a strong linear correlation. Based on these results, the chitosan/TiO_2 material exhibited electrochemical responses with potential for further development in electrochemical sensing applications.

Keywords: Chitosan, Titanium Dioxide, Sensing Material, Cyclic Voltammetry

ABSTRAK

Elektroda termodifikasi kitosan/TiO_2 dikarakterisasi menggunakan metode voltammetri siklik. Elektroda difabrikasi melalui metode elektrodeposisi untuk menghasilkan lapisan pada permukaan substrat. Variasi konsentrasi TiO_2 yang digunakan adalah 50, 100, 150, 200, dan 250 mg/L. Karakterisasi struktur kimia dan interaksi gugus fungsional pada film menggunakan Fourier Transform Infrared (FTIR), yang menunjukkan keberadaan gugus khas kitosan serta pita regangan Ti–O–Ti sebagai indikasi terdispersinya TiO_2 dalam matriks kitosan. Kinerja elektrokimia elektroda diuji dalam larutan penyangga fosfat (PBS) pH 7. Hasil pengujian menunjukkan bahwa variasi konsentrasi kitosan/TiO_2 150 mg/L memberikan respons arus anodik paling tinggi, sedangkan laju pemindaian optimum diperoleh pada 75 mV/s. Hubungan linear antara arus puncak dan laju pemindaian menghasilkan persamaan regresi $y = 0{,}0014x + 0{,}3788$ dengan koefisien determinasi $R^2 = 0{,}9869$, yang menunjukkan adanya korelasi linear yang kuat. Berdasarkan hasil tersebut, material kitosan/TiO_2 menunjukkan respons elektrokimia yang berpotensi untuk dikembangkan lebih lanjut pada aplikasi penginderaan elektrokimia

Kata kunci: Kitosan, Titanium Dioksida, Material Penginderaan, Voltammetri Siklik

1. Introduction

An electrochemical sensor device is defined as an analytical system capable of enabling the quantification and identification of chemical species in solution based on electrochemical signal responses, such as variations in current and potential [1]. This method is widely used due to its various advantages, including relatively low cost, fast response, ease of operation, high sensitivity, low detection limits, and its capability to be integrated into miniaturized devices [2]. The performance of an electrochemical sensor is strongly influenced by the properties of the electrode material, which ideally should possess high electrical conductivity, a large active surface area, and good electrocatalytic activity to support efficient electron transfer processes [3].

Chitosan is a natural polymer derived from the deacetylation of chitin and has been widely studied in electrochemical sensor applications due to its biocompatibility, film-forming ability, and the presence of reactive functional groups such as amino and hydroxyl groups [4] [5]. The amino groups impart hydrophilic properties that facilitate biomolecular interactions and can be protonated under acidic conditions, forming a positively charged cationic polyelectrolyte. This characteristic enables interaction with anionic analytes and contributes to the enhancement of current response in electrochemical sensors [6]. In addition, chitosan is capable of forming stable films on solid substrates, even under dry conditions, and remains intact when immersed in acidic solutions [7]. Due to its unique properties and application flexibility, chitosan has great potential as a sensing material, and its performance continues to be widely developed. However, this material also has several limitations, such as low mechanical strength, limited stability, and poor electrical conductivity [8]. Therefore, the incorporation of additional materials is required to improve the performance of chitosan in electrochemical sensing applications. One such material that can be introduced is titanium dioxide (TiO_2).

Titanium dioxide (TiO_2) is one of the metal oxides that has attracted significant attention due to its biocompatibility, electrocatalytic properties, high photosensitivity, non-toxicity, high surface reactivity, low cost, and abundant availability [9]. As an n-type semiconductor with a band gap energy of approximately 3.0 eV, it can function as a charge carrier in electrical conduction. Therefore, TiO_2 has great potential in electrochemical sensor applications [10]. The combination of chitosan and TiO_2 is expected to produce a synergistic effect, in which TiO_2 enhances electron transfer kinetics and increases the active surface area, while chitosan acts as a flexible matrix for film formation and interaction with analytes.

In a previous study, Nainggolan et al. (2024) reported that chitosan/TiO_2 films exhibited a good response for ammonia detection using the amperometric method. The results showed that sensors based on chitosan–TiO_2 films produced a higher maximum output voltage compared to sensors based on pure chitosan films when exposed to ammonia. Although this method is effective for real-time current measurements, it has limitations in providing comprehensive information regarding the electron transfer mechanism and the overall electrochemical behavior of the material [11]. In contrast, the present study employs cyclic voltammetry (CV), which allows for a more comprehensive analysis of oxidation–reduction processes as well as the interaction between the electrode material and the analyte [12]. Therefore, this approach is expected to provide a deeper understanding of the electrochemical performance of chitosan/TiO_2 films.

In this study, the mixing of the two materials was carried out using the ionic gelation method, which is widely used due to its simplicity, ease of control, ability to produce more homogeneous results, and enhancement of electron affinity during the mixing process [13]. This method was combined with stirring and sonication using an ultrasonic bath. It is expected that through the application of these methods, TiO_2 can be homogeneously distributed within the chitosan matrix.

The fabrication of the working electrode was carried out using the electrodeposition method, with a copper screen-printed electrode (SPE) as the substrate, coated with a chitosan/TiO_2 solution serving as the sensitive layer. The electrodeposition method is a simple, rapid, and cost-effective technique, while also enabling the formation of controlled thin films [14]. The resulting thin film was then analyzed to determine its structural properties and sensing capability using FTIR spectroscopy and cyclic voltammetry measurements.

2. Method

The materials used in this study included commercial chitosan with medium molecular weight (Sigma-Aldrich), titanium dioxide (TiO_2) (NRE), glacial acetic acid (CH_3COOH), anhydrous oxalic acid ($C_2H_2O_4$), phosphate buffer solution (PBS) at pH 7, and deionized water, copper-printed circuit board (PCB) substrate. The equipment used consisted of an analytical balance (Mettler), an oven (Memmert), a hotplate stirrer (Cimarex), a magnetic stir bar, an ultrasonic bath (Kerry Pulsatron), a power supply (OOCU), an FTIR spectrometer (Shimadzu IR Prestige-21), and a potentiostat (CorrTest).

The initial preparation stage involved the dissolution of chitosan and TiO_2. The dissolution of chitosan in acetic acid was carried out using an ionic gelation method combined with stirring. A certain amount of chitosan with a concentration of 1.5% (w/v) was weighed and dissolved in 2% (v/v) acetic acid. The mixture was stirred

at room temperature at a speed of 300 rpm for 24 hours until a homogeneous solution was obtained [15]. For the preparation of a 50 mg/L TiO_2 solution, 5 mg of TiO_2 was weighed and dissolved in 1 N oxalic acid. The mixing process was conducted using a hotplate stirrer at 80 °C with a stirring speed of 200 rpm under low-light conditions for 4 hours until a homogeneous solution was achieved. The TiO_2 solution was then transferred into a 100 mL volumetric flask and diluted with 1 N oxalic acid up to the calibration mark. The same procedure was applied for other TiO_2 concentrations of 100, 150, 200, and 250 mg/L.

The next stage involved mixing the two materials for the preparation of thin films used in FTIR characterization and working electrode fabrication. A chitosan solution with a concentration of 1.5% (w/v) and TiO_2 solutions with varying concentrations (50, 100, 150, 200, and 250 mg/L) were used. A total of 20 mL of 1.5% (w/v) chitosan solution was mixed with 10 mL of TiO_2 solution (50 mg/L) at a ratio of 2:1 under constant stirring at 200–300 rpm for 24 hours until a homogeneous mixture was obtained. The mixture was then subjected to ultrasonication for 1 hour [15]. The ratio of chitosan to TiO_2 was selected based on previous studies and preliminary observations. It was found that a 1:1 ratio resulted in difficulty in forming a uniform thin film, likely due to an insufficient polymer matrix. In contrast, the 2:1 ratio provided better film-forming ability and improved structural stability.

The fabrication of the chitosan/TiO_2 film on copper-printed circuit board (PCB) substrate was carried out using the electrodeposition technique. This method is effective for producing thin material layers on the working electrode surface through electrochemical reactions controlled by an applied electric current [16]. The electrodeposition process facilitates the migration of protonated amino groups (NH_3^+) from chitosan toward the cathodic substrate, resulting in the formation of a uniformly deposited film layer. A voltage of 3.6 V was applied for 5 minutes, after which the chitosan/TiO_2-modified SPE substrate was dried in an oven at 60 °C for 60 minutes. Fig 1 shows the chitosan/TiO_2-modified electrode fabricated using the electrodeposition method.

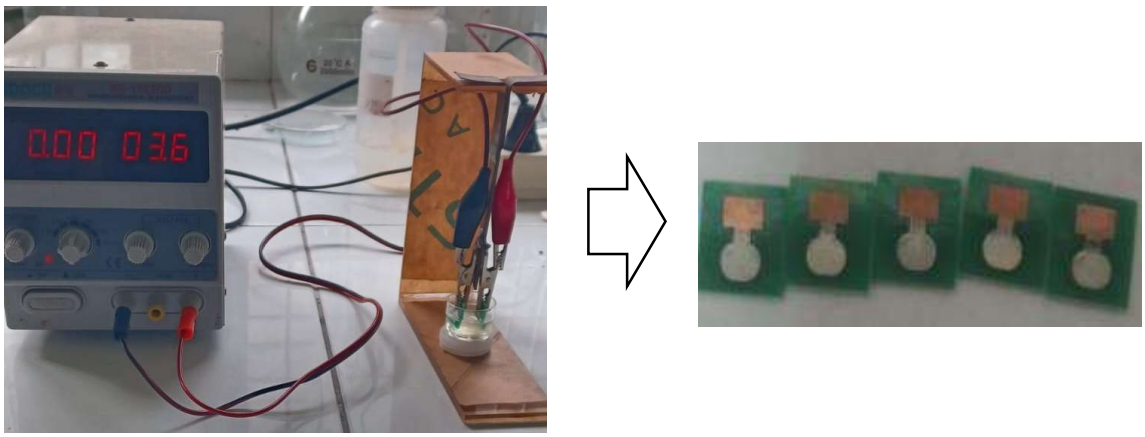

Figure 1. Electrodeposition process of chitosan/TiO2 modified copper electrode

Chitosan acts as a cationic polyelectrolyte matrix that binds and stabilizes TiO_2 particles through electrostatic interactions and hydrogen bonding. When a negative potential is applied to the copper electrode, water reduction generates OH^- ions, leading to an increase in local pH near the cathode surface. This alkaline condition triggers the deprotonation of NH_3^+ groups into neutral $–NH_2$ groups, causing chitosan to lose its solubility and precipitate together with TiO_2 particles. As a result, a chitosan/TiO_2 thin film is formed on the surface of the working electrode [17].

The characterization of chitosan/TiO_2 films was performed using Fourier Transform Infrared Spectroscopy (FTIR) to identify the functional groups and investigate the chemical interactions within the composite material. The FTIR technique is based on the absorption of infrared radiation by molecular bonds, which induces vibrational transitions at specific wavelengths and produces characteristic spectral patterns corresponding to the chemical structure of the material. This characterization aim to evaluate potential physical or chemical interactions between the chitosan matrix dan TiO_2 particles. The FTIR analysis was carried out using Shimadzu IR Prestige-21. The functional groups present in the chitosan/TiO_2 film were identified using an FTIR spectrophotometer within the wavenumber range of 500-4000 cm^{-1}. The obtained functional group assignments were subsequently analyzed by comparing the observed absorption peaks with those reported in the literature.

The electrochemical properties of the chitosan/TiO_2-based electrode were evaluated using cyclic voltammetry (CV). Measurements were performed using a potentiostat in a three-electrode system consisting of a working electrode, a reference electrode, and a counter electrode. The chitosan/TiO_2 film coated on a copper-printed circuit board (PCB) substrate served as the working electrode, an Ag/AgCl electrode was used as the reference electrode, and a graphite electrode functioned as the counter electrode. The electrochemical response of the chitosan/TiO_2 film deposited on the copper-PCB substrate was tested in phosphate buffer solution (PBS) (Himedia) as the supporting electrolyte. The potential range applied was from −1 V to +1 V.

3. Results and Discussion

3.1. Fourier Transform Infrared Spectroscopy (FTIR) Analysis

The functional groups of the chitosan/titanium dioxide (TiO_2) film were investigated using Fourier Transform Infrared (FTIR) spectroscopy. Thin films of chitosan and chitosan/TiO_2 were used as the tested samples. During FTIR characterization, distinct absorption bands observed in the transmittance spectra signifity the interaction of infrared radiation with molecular components at particular wavelenghts. Such bands are associated with the vibrational behavior of chemical bonds within the material.

Chitosan and TiO_2 were successfully blended to form a homogeneous and stable solution at room temperature. The resulting chitosan/TiO_2 matrix was then cast into an acrylic container and allowed to dry, leading to the formation of a thin film layer. The FTIR spectra of chitosan and chitosan/TiO_2 thin films are presented in Fig. 2.

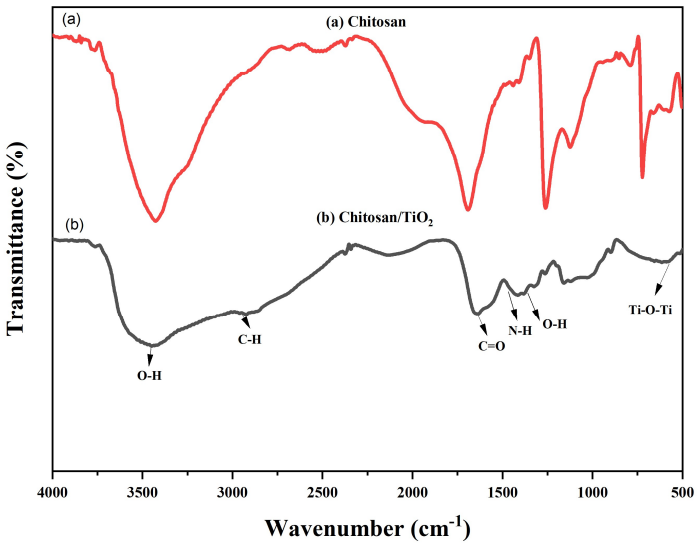

Figure 2. FTIR spectra of (a) chitosan thin film, (b) chitosan/TiO2 thin film

FTIR spectrum of chitosan shows a strong OH stretching band at 3448.72 cm^{-1}. Absorption peaks at 1633.71 cm^{-1} and 1413.82 cm^{-1} are assigned to C=O stretching in the –NHCO– group and N–H vibrations of $–NH_2$ [18], while bands at 1382.96 cm^{-1} and 2922.16 cm^{-1} are characteristic of O–H bending and C–H stretching vibrations [19].

Comparable findings were obtained for the chitosan/TiO_2 thin film, as the FTIR spectrum revealed overlapping characteristics associated with each constituent material. The FTIR spectrum of chitosan/TiO_2 shows a band at 572.86 cm^{-1} related to Ti–O–Ti stretching vibrations, confirming the presence and interaction of TiO_2 in the mixture. An O–H stretching band remains visible at 3427.51 cm^{-1} but with lower intensity, indicating interaction with the TiO_2 surface. The C–H stretching vibration occurs at 2924.0 cm^{-1}, while a slight increase in the C=O stretching band at 1691.57 cm^{-1} reflects chitosan–TiO_2 interactions. The O–H deformation peak of chitosan is observed at 1350.17 cm^{-1}. The functional group wavenumbers identified from FTIR analysis are listed in Table 1.

Table 1. FTIR spectrum data of chitosan and chitosan/TiO2

Functional group	Wavenumber (cm^{-1})	
	Chitosan	Chitosan/TiO_2
O-H stretching	3448.72	3427.51
C-H stretching	2922.16	2924.0
C=O stretching	1633.71	1691.57
N-H bending	1413.82	1409.96
O-H bending	1382.96	1350.17
Ti-O-Ti	-	572.86

The FTIR spectra of chitosan and chitosan/TiO_2 are generally similar, but the peak intensities of the chitosan/TiO_2 thin film are lower than those of chitosan. Figure 1 shows changes in the transmittance intensity and slight shifts in the O–H and C=O stretching bands, indicating interactions between TiO_2 and the hydroxyl groups of chitosan. This suggests successful filler–matrix interaction without significant alteration of the chitosan chemical structure as TiO_2 content increases.

3.2. Cyclic Voltammetry (CV) Analysis

The determination of the optimum concentration for the modified electrode was carried out using cyclic voltammetry (CV) in phosphate buffer solution (PBS) at pH 7. The sample tested was the chitosan/TiO_2 solution that had been applied onto the surface of a copper-printed circuit board (PCB) substrate as the working electrode, with an Ag/AgCl electrode as the reference electrode and a graphite electrode as the counter electrode. These three electrodes functioned together in determining the optimum concentration of the chitosan/TiO_2 solution.

The voltammogram provides two types of information: peak current height (quantitative) and peak potential (qualitative). The sharpness of the peak affects the accuracy of current measurement. The peak current reflects the periodic electron transfer resulting from electrochemical reactions at the electrode surface. To determine the optimum concentration of the chitosan/TiO_2-modified electrode, the optimum oxidation and reduction potentials were obtained from the voltammogram. The current was measured within a potential range of −1 V to +1 V at a scan rate of 75 mV/s to obtain both anodic (oxidation) and cathodic (reduction) currents. Figure 3 shows the optimum concentration at various TiO_2-to-chitosan ratios in PBS solution at pH 7.

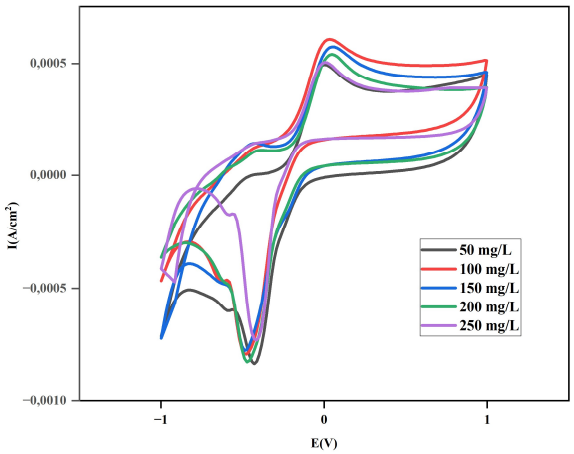

Figure 3. Voltamogram of (a) Chitosan/TiO2 modified electrode with variation of TiO2

Figure 3 shows the presence of distinct oxidation and reduction peak currents at each chitosan/TiO_2 concentration. The variation of TiO_2 in the electrode contributes to the improvement of both mechanical and electrical properties. In evaluating the optimum modified electrode, the analysis was based on the anodic peak current (IpA). The anodic peak current reflects the oxidation process occurring at the electrode surface, which is closely related to the efficiency of electron transfer and the active surface area of the modified electrode.

The optimum anodic peak current was observed in phosphate buffer solution (PBS) at pH 7, obtained using the chitosan/TiO_2-modified electrode with a TiO_2 content of 150 mg/L, as shown in Fig 3 and Table 2.

Table 2. Optimum Electrode in Phosphate Buffer Solution

TiO_2 (mg/L)	EpA (V)	EpC (V)	IpA (mA)	IpC (mA)
50	0,001	-0,430	0,3509	-0,5875
100	0,031	-0,476	0,4149	-0,9087
150	0,055	-0,482	0,4553	-0,7212
200	0,061	-0,469	0,4433	-0,9721
250	0,007	-0,415	0,4276	-0,3135

Based on Table 2, the anodic peak current (IpA) increases with increasing TiO_2 concentration up to 150 mg/L, indicating enhanced electrochemical activity. This increase can be attributed to the higher active surface area and improved electron transfer resulting from the incorporation of TiO_2 into the chitosan matrix. The presence of TiO_2 provides additional active sites and facilitates charge transfer, leading to a higher current response. However, further increases in TiO_2 concentration (200–250 mg/L) result in a decrease in the anodic peak current. This behavior is likely due to particle agglomeration at higher concentrations, which reduces the effective surface area and hinders electron transfer. Therefore, a concentration of 150 mg/L is considered the optimum condition for achieving maximum electrochemical performance.

The optimum chitosan/TiO_2-modified electrode was used to determine the optimum scan rate using cyclic voltammetry in phosphate buffer solution (PBS) at pH 7. The scan rate variations applied were 50, 75, 100, 125, and 150 mV/s. The scan rate represents the rate of change of the applied potential per unit time. Variations in scan rate affect the rate of electron transfer in the electrolyte solution according to the oxidation–reduction potential of the analyte. This analysis is important for determining the reaction mechanism and identifying the optimum condition that provides a stable current response. The results of the scan rate measurements for the chitosan/TiO_2-modified electrode are presented in Fig 4.

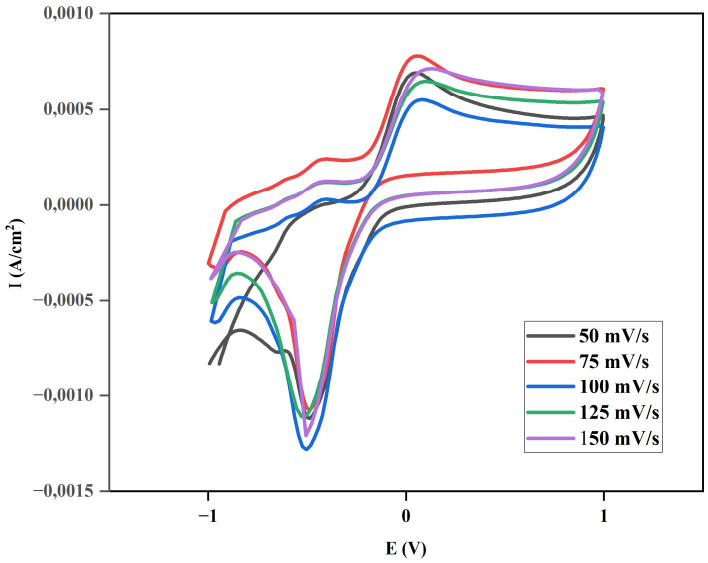

Figure 4. Scan rate variation of chitosan/TiO2 modified electrode with concentration TiO2 150 mg/L

The effect of scan rate on the electrochemical response was evaluated based on both anodic (IpA) and cathodic (IpC) peak currents. As the scan rate increased, both IpA and IpC values also increased, which is consistent with general electrochemical behavior due to enhanced electron transfer kinetics and reduced diffusion time. However, despite the increase in peak current at higher scan rates, the voltammogram at 75 mV/s exhibited the most well-defined and stable oxidation and reduction peaks. The relationship between the

anodic peak current (IpA) and the variation in scan rate is shown in Fig 5.

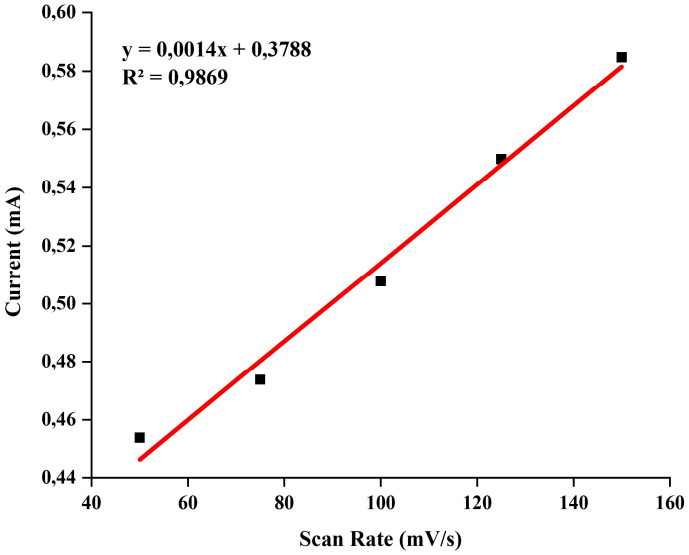

Figure 5. Plot of anodic peak current (IpA) versus scan rate

Table 3. Results of Scan Rate Variation on Anodic and Cathodic Peak Currents.

Scanrate (mV/s)	IpA (mA)	IpC (mA)
50	0,4539	-0,7530
75	0,4737	-1,2107
100	0,5078	-1,3442
125	0,5498	-1,4808
150	0,5848	-1,7285

Based on Table 3, it can be observed that increasing the scan rate in the electrochemical process results in higher anodic peak current (IpA) and cathodic peak current (IpC) values. The increase in current is attributed to the faster electron transfer occurring at the electrode surface. Therefore, as the scan rate increases, the resulting peak current also increases due to the accelerated electrochemical reaction process.

The scan rate also influences the thickness of the diffusion layer, which is formed due to the concentration gradient between the analyte and the electrode surface. A higher scan rate results in a thinner diffusion layer (i.e., the diffusion layer forms closer to the electrode surface), thereby facilitating more efficient electron transfer at the working electrode. Conversely, a lower scan rate leads to a thicker diffusion layer at the electrode surface, which hinders electron transfer and results in a lower current response [20]. However, excessively high scan rates may lead to incomplete electrochemical reactions due to the limited interaction time with the analyte. On the other hand, very low scan rates may promote side reactions within the chitosan matrix, which can affect both the anodic and cathodic peak currents of the analyte.

The linear relationship between peak current (Ip) and scan rate was evaluated to investigate the electrochemical behavior of the modified electrode. The regression equation ($y = 0.0014x + 0.3788$) with a coefficient of determination ($R^2 = 0.9869$) indicates a strong linear correlation, suggesting that the electrochemical process is likely diffusion-controlled. It should be noted that this analysis does not represent sensor sensitivity, which requires a calibration curve based on analyte concentration. Instead, these results provide insight into the electron transfer mechanism of the system.

The FTIR analysis confirms the presence of functional groups such as –OH and $–NH_2$ from chitosan, as well as Ti–O–Ti bonds from TiO_2. These functional groups play an important role in the electrochemical performance of the material. The –OH and $–NH_2$ groups can enhance the interaction between the electrode surface and the analyte through hydrogen bonding and electrostatic interactions, thereby facilitating charge transfer. Meanwhile, the presence of TiO_2 contributes to improved electron transfer due to its semiconducting properties. This synergistic interaction between the functional groups and TiO_2 is believed to be responsible for the enhanced current response observed in the cyclic voltammetry analysis.

Conclusion

The chitosan/TiO_2-modified electrode has been successfully prepared and characterized using FTIR and cyclic voltammetry methods. The results showed that the incorporation of TiO_2 improved the electrochemical performance through enhanced electron transfer and increased active surface area of the electrode. The optimum condition was obtained at a TiO_2 concentration of 150 mg/L and a scan rate of 75 mV/s, which produced a stable current response. These findings indicate that the interaction between chitosan and TiO_2 plays an important role in improving electrochemical behavior. Although parameters such as the limit of detection (LOD), long-term stability, and selectivity were not evaluated in this study, these aspects are important for future investigations. Overall, the results suggest that the chitosan/TiO_2 material exhibits promising electrochemical responses.

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