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Titanium Dioxide-Enhanced Carbon Paste Electrodes for Superior Electrochemical Sensors

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In this study, the synthesis and electrochemical oxidation of ferricyanide using a carbon paste electrode (CPE) modified with TiO_2 was examined in detail. The objective was to create an effective and dependable electrochemical sensor for ferricyanide oxidation, a redox probe that is frequently employed in various analytical applications. In addition, the TiO_2-modified CPE was synthesized to enhance the electrocatalytic activity and sensitivity to ferricyanide oxidation. X-ray diffraction (XRD), scanning electron microscopy (SEM), and Fourier-transform infrared spectroscopy (FTIR) were used to characterize the modified sensor. The ferricyanide analysis showed that after TiO_2 modification, the surface area of graphite increased significantly. The impact of scan rates on the electrochemical oxidation process examined at various scan rates showed a strong correlation between the two processes (Correlation coefficient, $R^2 = 0.94$). In comparison to the unmodified electrode, the experimental results showed that the TiO_2-modified CPE showed better electrochemical performance, and higher peak current density but lower oxidation potential. The study provides new insights into the design and optimization of electrode materials for electrochemical sensing applications, suggesting their potential use in various future applications.

Keywords: Carbon Paste Electrode, Electrochemical Oxidation, Titanium dioxide, Ferricyanide oxidation, Electrode materials.

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Introduction

The development of innovative materials has led to remarkable advancements in the design, operation, and performance of electrochemical sensors (ES). One notable material, titanium dioxide (TiO_2), has attracted significant attention due to its exceptional properties such as superior conductivity, high stability, and biocompatibility. The outlined properties make TiO_2 suitable for enhancing the characteristics of ES. Therefore, the application of TiO_2 in carbon paste electrodes (CPE) of ES is a promising approach. However, the promising prospects of TiO_2 integration in CPE require further investigation in order to realize high selectivity, stability, and sensitivity during electrochemical analysis. Likewise, the electrochemical characteristics of the CPE electrode could be enhanced by ferricyanide oxidation. The ferricyanide ($K_3[Fe(CN)_6]$) compound (which contains Ferric (Fe^{3+}) and cyanide (CN^-) ions) can be synthesized and electrochemically oxidised using a CPE modified with titanium dioxide (TiO_2). Therefore, the electrochemical characteristics of the electrode and its ability to detect and analyse ferricyanide ions can be improved by integrating TiO_2 into CPE.

Furthermore, the addition of TiO_2 to CPE is to increase the stability, sensitivity, and selectivity of the electrode for ferricyanide electrochemical oxidation [1].

This is because of its special qualities, including high stability, superior conductivity, and biocompatibility, titanium dioxide (TiO_2) is a material that is used extensively in many different fields. Because TiO_2 can improve the sensitivity, selectivity, and stability of electrochemical sensors and biosensors, it has attracted a lot of interest as a modifier for electrochemical analysis in recent years. The large surface area of TiO_2 offers many active sites for analyte adsorption and speeds up electron transfer processes, which is one of the main benefits of utilising it as a modifier in electrochemical analysis. Improved electrochemical performance can result from TiO_2 ability to mediate electron transport between the analyte and the electrode surface. To improve the selectivity of detection for target analytes, TiO_2 nanoparticles can also be readily functionalised with different chemicals or biomolecules [2]. Because of its special qualities that improve graphite electrode performance, titanium dioxide (TiO_2) is frequently utilised as a modification of graphite for electrochemical analysis. TiO_2 is a material that is well-suited for enhancing the sensitivity and selectivity of graphite electrodes in a variety of electrochemical applications due to its high surface area, exceptional electrical conductivity, and strong chemical stability.

When graphite and TiO_2 are mixed, a composite material is created that has better electrochemical qualities than graphite electrodes alone. By adding TiO_2 nanoparticles to the graphite electrode surface, the active surface area is increased, electron transfer kinetics are accelerated, and analyte adsorption is enhanced, improving electrochemical analysis's sensitivity and detection limits. TiO_2 and graphite work in concert to create a composite material that is ideal for energy storage, environmental monitoring, biosensing, and sensing. The enhanced stability, repeatability, and sensitivity of the modified graphite electrodes make them useful instruments for a range of analytical procedures in both industry and research [3]. Heavy metals, organic pollutants, biomolecules, and medications are just a few of the analytes that have been successfully detected using TiO_2-modified electrodes. TiO_2 special qualities make it an adaptable modifier for a variety of electrochemical sensor types, including potentiometric, voltammetric, amperometric, and impedance sensors.

To create a uniform paste, the preparation procedure usually entails combining carbon powder with a binder (such as paraffin or mineral oil) and TiO_2 nanoparticles. The modified carbon paste electrode is then made by packing this paste into an electrode body. TiO_2 on the electrode surface improves the catalytic activity and electron transfer kinetics for ferricyanide oxidation, which improves analytical performance in terms of sensitivity and detection limits [4]. There are various benefits of using TiO_2 to modify carbon paste electrodes for ferricyanide anodic reaction research. TiO_2 can help electron transfer reactions at the electrode surface because of its high surface area, superior conductivity, and catalytic qualities. Increased sensitivity, selectivity, and stability of the electrode ferricyanide oxidation may result from this alteration. Furthermore, TiO_2-modified electrodes can offer improved control over the electrochemical reactions that lead to ferricyanide oxidation, enabling more precise measurements and analysis [5].

I. Methods

1.1. Reagent

All the reagents used in this research were of Analytical grade. Mortar, pistol, weighing balance, Dichloromethane as a solvent, graphite, TiO_2 nanoparticle, paraffin oil, syringe, and copper wire.

1.2. Instrumentation

µStat-i-400 (Bi)potentiostat/Galvanostat/Impedance Analyzer, DRP-STAT-I-400, Lot: 14091082A-(Metrohm DropSens), Department of Chemistry (Analytical Chemistry Laboratory), Bayero University Kano, Kano State, Nigeria was used for Cyclic Voltammetry.

1.3. Preparation of bared and modified CPE

The preparation of the electrode was performed at an ambient temperature. Hence, 200 mg of composite powder, 800 mg of graphite powder, and 100 µL of paraffin oil were combined to create the CPE composite for 10 min. After moving the mixture to a mortar and pestle for 20 min, 2.0 ml of dichloromethane was added and thoroughly stirred to homogenize it and remove the impurities. For 24 hours, the solvent was allowed to evaporate at room temperature. To create the final composite electrode assembly, the open tip of a plastic syringe with a diameter of 2.4 mm was filled until it was completely packed to make electrical contact with copper wire. The paste was smoothened with paper to generate a reproducible working surface at the tip of the syringe. Each of the three composite compounds (Graphite, TiO_2/Graphite) were subjected to a similar procedure. The incorporation of TiO_2 into CPE was to increase its electrical conductivity. However, this enhancement is highly dependent on the concentration of TiO_2 used. At low concentrations, TiO_2 particles could effectively disperse within the carbon matrix, leading to improved electron transfer rates. Conversely, at higher concentrations, agglomeration occurs, which hinders conductivity due to reduced contact between conductive carbon particles. The surface area available for electrochemical reactions was another critical factor which was influenced by TiO_2 concentration. An optimal amount of TiO_2 (200 mg) created a porous structure that increased the surface area and more active sites for analyte interaction. This is particularly important for sensors that rely on surface reactions for detection. The electrocatalytic properties of the electrode were also affected by TiO_2 concentration. A balanced concentration allows for enhanced catalytic activity towards specific redox reactions, while excessive amounts may lead to diminished performance due to particle aggregation or phase separation [6].

II. Results and Discussion

2.1. Characterization of Composite

2.1.1. FTIR Analysis

Graphite's FTIR data (Figure 1) usually exhibits

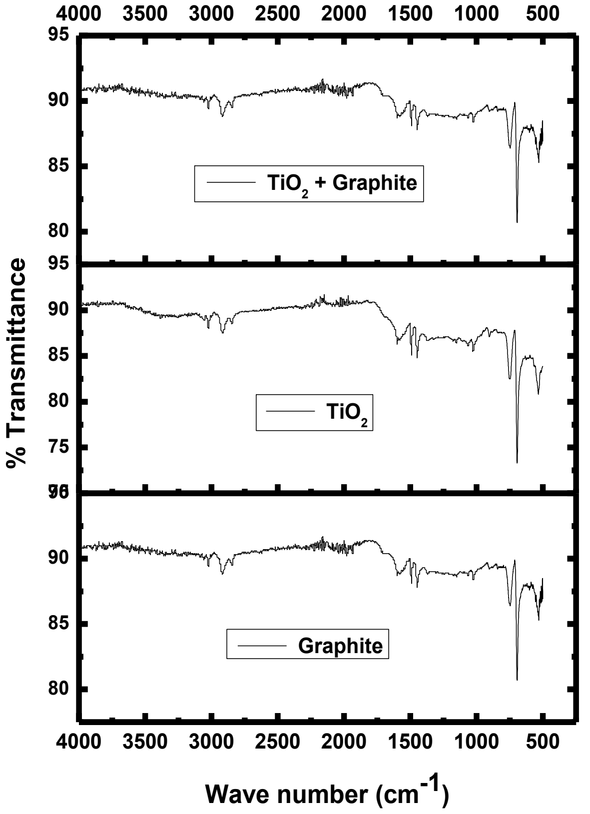


Fig. 1. FTIR spectra of (a)Bare Graphite Graphite (b)TiO_2 (c) Graphite/TiO2 composite.

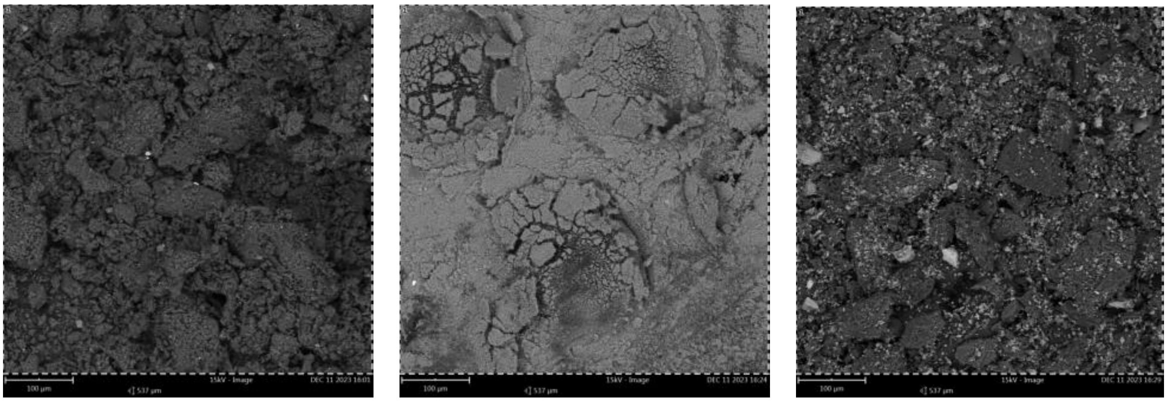
distinctive peaks at different wavenumbers. The stretching vibrations of the sp^2 hybridised carbon atoms in the graphite structure are responsible for the strong peaks at 3060.01 cm^{-1} and 3025.24 cm^{-1} that are seen in the data that has been supplied. The graphite lattice's asymmetric and symmetric stretching vibrations of C-H bonds are shown by the peaks at 2920.76 cm^{-1} and 2849.72 cm^{-1}, respectively. The E_2g mode of vibration, which is linked to the peak at 1600.80 cm^{-1}, indicates that the material is graphitic. Additionally, the peaks at 1492.35 cm^{-1}, 1451.68 cm^{-1}, and 1373.26 cm^{-1} correspond to graphite's D band, G band, and D' band, respectively, and reveal details about the carbon structure's flaws and disorder. While the peaks at 36.81 cm^{-1} correspond to low-frequency modes suggestive of interlayer interactions within the graphite layers, the peaks at 1154.12 cm^{-1}, 1028.02 cm^{-1}, 907.01 cm^{-1}, 748.60 cm^{-1}, and 695.075 cm^{-1} can be attributed to different lattice vibrations and structural properties of graphite. The existence of O-H stretching vibrations is shown by distinctive peaks in the TiO_2 FTIR data at 3023.70 cm^{-1}. In the TiO_2 structure, the peaks at 2918.92 cm^{-1} and 1599.59 cm^{-1} are associated with C-H stretching vibrations. C=C stretching vibrations are responsible for the peaks at 1491.78 cm^{-1} and 1450.71 cm^{-1}, whereas Ti-O-Ti stretching vibrations are seen at 1067.00 cm^{-1}. Ti-O bending vibrations in the TiO_2 structure are linked to the peaks at 694.73 cm^{-1} and 535.00 cm^{-1}, while Ti-O stretching vibrations are linked to the peaks at 1027.11 cm^{-1}, 903.91 cm^{-1}, and 747.26 cm^{-1}. The characteristic peaks at 3024.01 cm-1, 2918.21 cm^{-1}, and 2845.97 cm^{-1} in the TiO_2 / graphite FTIR data are caused by the stretching vibrations of the C-H bonds in the graphite component. The stretching vibration of Ti=O bonds in TiO_2 is represented by the peak at 1979.64 cm^{-1}, whilst the presence of Ti-O bonds in TiO_2 is indicated by the peaks at 1599.49 cm^{-1} and 1491.78 cm^{-1}. While the peaks at 904.39 cm^{-1} and 746.86 cm^{-1} may be linked to lattice vibrations in TiO_2, the peaks at 1450.59 cm^{-1}, 1152.11 cm^{-1}, and 1025.59 cm^{-1} can be attributed to the bending vibrations of C-H bonds in graphite. Other vibrational modes found in the TiO_2/graphite composite material may also be connected to the peaks at 694.77 cm^{-1}, 601.34 cm^{-1}, 555.60 cm^{-1}, and 531.29 cm^{-1} [7].

2.1.2. SEM Analysis of Graphite, TiO_2, and Graphite with TiO_2

A potent method in materials science for examining the surface appearance and structure of different materials at the micro- and nanoscale is scanning electron microscopy or SEM. SEM analysis is essential for describing the morphology and composition of the materials used in the manufacture and electrochemical oxidation of ferricyanide using a carbon paste electrode modified with TiO_2. Because of its conductivity and chemical durability, graphite is a substance that is frequently employed in electrochemistry. Graphite electrodes' surface structure, porosity, and any alterations done to improve their electrochemical qualities can all be seen when they are examined with a scanning electron microscope. Titanium dioxide (TiO_2) is another important material known for its photocatalytic and electrochemical properties. SEM analysis of TiO_2 nanoparticles (Figure 2) can provide insights into their size, shape, and distribution on the electrode surface when used as a modifier in carbon paste electrodes. When graphite is combined with TiO_2 for electrode modification, SEM analysis can elucidate the interaction between these two materials, their dispersion within the carbon paste matrix, and the overall impact on the electrode's performance in electrochemical applications such as the oxidation of ferricyanide [8].

2.1.3. XRD Analysis

One effective method for examining a material's crystal structure is X-ray diffraction (XRD) examination. In the context of the synthesis and electrochemical oxidation of ferricyanide using a carbon paste electrode modified with TiO_2, XRD analysis (Figure 3) can provide valuable information about the composition and structure of the materials involved. When graphite, TiO_2, and a composite of graphite with TiO_2 are used in the preparation of electrodes for electrochemical applications, XRD analysis can be employed to characterize the crystalline phases present in these materials. Graphite is a form of carbon with a layered structure, while TiO_2 is a metal oxide commonly used in various applications due to its unique properties. By combining graphite with TiO_2, it is possible to create a composite material that may exhibit enhanced properties compared to the individual components. In the context of electrochemical oxidation of ferricyanide, the use of a carbon paste electrode modified with TiO_2 can offer advantages such as improved stability, conductivity, and catalytic activity. XRD analysis can help in understanding how the addition of



a b c

Fig. 2. SEM images of (a)Bare Graphite (b)TiO_2 (c) Graphite/TiO_2 composite.

TiO_2 influences the crystal structure of the electrode material and how this, in turn, affects its electrochemical performance [7].

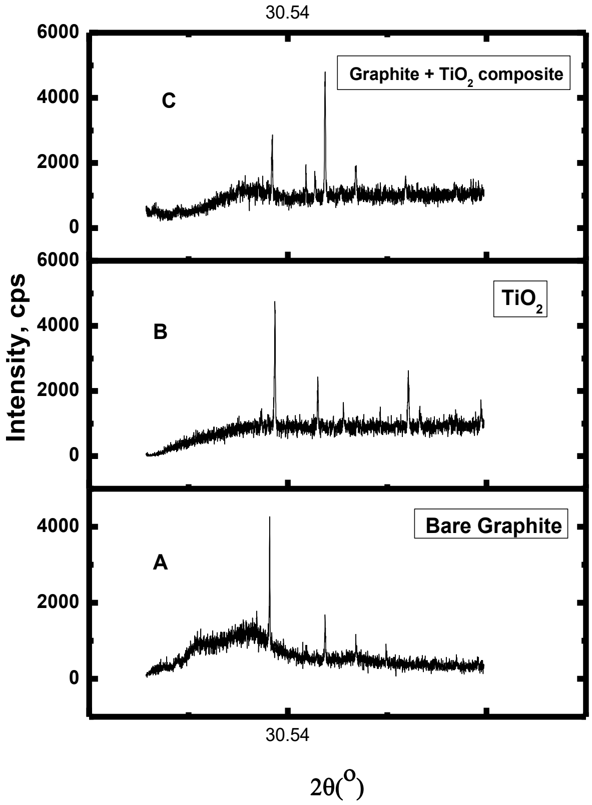


Fig. 3. XRD Patterns of (a)Bare (b)TiO_2 (c) Graphite/TiO_2 composite.

2.2. Mechanism

Titanium dioxide (TiO_2) is a semiconductor with unique electronic properties that can significantly influence the electrochemical behaviour of carbon paste electrodes (CPE). TiO_2 has a wide bandgap (approximately 3.0 to 3.2 eV), which facilitates charge transfer processes when interfaced with conductive materials like carbon. The incorporation of TiO_2 into CPEs enhances their electrical conductivity and provides a larger surface area for electrochemical reactions due to its nanostructured form. In addition, the addition of TiO_2 modifies the surface characteristics of CPE, leading to increased adsorption sites for analytes. This is crucial in sensor applications where the interaction between the electrode surface and target molecules determines sensitivity and selectivity. The hydroxyl groups present on the TiO_2 surface can interact with various chemical species, enhancing the binding affinity of analytes and promoting faster electron transfer kinetics [9]. The enhanced electron transfer rates observed in TiO_2-enhanced CPEs are attributed to several factors. The presence of TiO_2 facilitates better charge carrier mobility within the composite material, allowing for more efficient electron transfer during redox reactions. TiO_2 exhibits photocatalytic properties under UV light, which promotes oxidation-reduction reactions at lower energy thresholds and improves sensor response times. The incorporation of TiO_2 also contributes to the mechanical stability of CPE, making them more durable under operational conditions, when combined with other materials or functional groups in CPE formulations. TiO_2 can create synergistic effects that further enhance sensor performance. According to [10], doping TiO_2 with metal nanoparticles or integration with conducting polymers may lead to improved electrocatalytic activity and broadened detection ranges for various analytes).

2.3. Determination of Oxidation of Ferricyanide

The determination of ferricyanide using graphite and TiO_2/graphite composite involves electrochemical techniques to analyze the presence and concentration of ferricyanide ions in a solution. Ferricyanide is a chemical compound with the formula $[Fe(CN)_6]^{3-}$, which is commonly used in analytical chemistry as a redox indicator. In this context, the use of TiO_2/ graphite composite alongside graphite electrodes enhances the sensitivity and efficiency of the electrochemical detection of ferricyanide ions. When comparing the peak current responses of graphite and TiO_2/graphite composite electrodes, it is observed that the surface area of the TiO_2/graphite composite electrode results in a higher peak current response compared to only graphite electrodes. For instance, if the peak current response of TiO_2/graphite composite (Figure 4) is measured at 12 uA, while graphite alone shows a peak current response of 7 uA, this indicates that the TiO_2/graphite composite electrode has a greater electrochemical activity towards ferricyanide ions. The increased peak current response observed with the TiO_2/graphite composite electrode can be attributed to several factors.

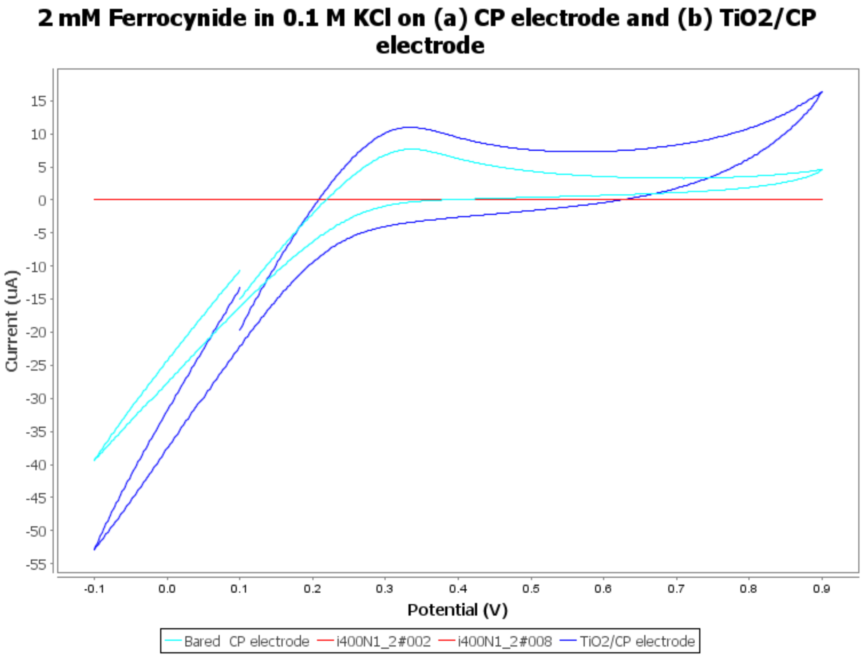


Fig. 4. Figure 4: Cyclic Voltammogram of 2 mM Ferricyanide in a 0.1 M supporting supporting at electrolyte of KCl Graphite and (b) Graphite/TiO_2 composite at a scan rate of 50 mVs^{-1} Ferricyanide at Graphite/TiO_2 rate of 25, 50, 75, 100, 125, and 150 mVs^{-1}.

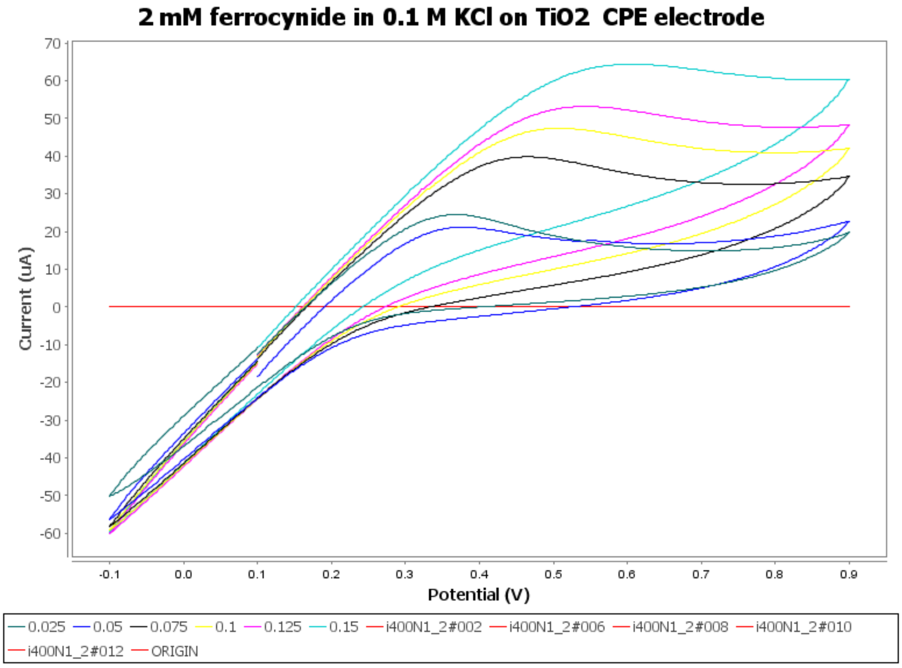


Fig. 5. Cyclic Voltammogram of 2 mM Ferricyanide in a 0.1 M of KCl at compounds scan rate composite at a different scan.

Firstly, the incorporation of titanium dioxide (TiO_2) nanoparticles into the graphite matrix increases the surface area available for electrochemical reactions. The presence of TiO_2 nanoparticles provides more active sites for the oxidation or reduction of ferricyanide ions, leading to a higher current response. Additionally, the synergistic effect between TiO_2 and graphite in the composite material enhances electron transfer kinetics at the electrode-electrolyte interface. This improved electron transfer facilitates faster redox reactions between ferricyanide ions and the electrode surface, resulting in a more pronounced peak current response during electrochemical measurements [11]. Furthermore, the unique properties of TiO_2, such as its high stability, biocompatibility, and photocatalytic activity, contribute to the enhanced performance of the TiO_2/graphite composite electrode in ferricyanide determination. These properties make TiO_2 an excellent candidate for modifying electrode surfaces to achieve sensitive and selective detection of analytes like ferricyanide. Therefore, the utilization of TiO_2/graphite composite electrodes offers significant advantages over conventional graphite electrodes in the determination of ferricyanide due to their increased surface area, improved electron transfer kinetics, and unique properties of TiO_2 nanoparticles. This enhanced electrochemical performance enables more sensitive and precise detection of ferricyanide ions in analytical applications [2].

2.4. Effect of Scan Rate Variation

The Randles-Sevcik plot is a graphical representation used in electrochemistry to analyse the behaviour of an electrochemical system. It is commonly used to determine the kinetics and mechanism of electrode reactions. In this case, the Randles-Sevcik plot was constructed for the square root of scan rate (25, 50, 75, 100, 125, and 150 mV/s) against the anodic peak current at a TiO_2/graphite composite electrode, resulting in an R-squared value of 0.9355 (Figure 6). The square root of the scan rate is plotted on the x-axis, while the anodic peak current is plotted on the y-axis. The Randles-Sevcik equation describes the relationship between these two parameters and can be used to extract important information about the electrochemical system under study. The high value of R-squared (0.9355) indicates a strong correlation between the square root of the scan rate and the anodic peak current, suggesting that the electrode reaction at the TiO_2/graphite composite electrode follows a well-defined kinetic mechanism. This analysis provides valuable insights into the electrochemical behaviour of the TiO_2/graphite composite electrode and can be used to optimize its performance for various applications in energy storage, sensing, or catalysis [13].

2.5. Electrode Comparison

TiO_2-modified electrodes typically exhibit higher specific capacities compared to their unmodified counterparts. For instance, the FeS@TiO_2 nanostructures demonstrated a reversible capacity of 510 mAh g^{-1} after 100 cycles at a current density of 200 mA g^{-1}, which is significantly higher than pure FeS nanostructures. This improvement can be attributed to the anatase phase of TiO_2, which enhances electrical conductivity and facilitates lithium-ion diffusion. The cyclic stability of TiO_2-modified electrodes is another critical advantage (Figure 5). In studies where TiO_2 was integrated into electrode materials, such as FeS or carbon nanofibers, the retention rates were notably high. For example, the FeS@TiO_2 composite maintained a capacity of 456 mAh g^{-1} even after 500 cycles at 400 mA g^{-1}, showcasing its long-term cycling stability. The rate capability of TiO_2-modified electrodes also tends to be superior when compared to similar materials without modification. The introduction of TiO_2 not only improves the overall conductivity but also enhances the kinetics associated with lithium-ion insertion/extraction processes at the electrode/electrolyte interface. In supercapacitor applications, composites like $Fe_{1-x}S$-TiO_2/CNFs have shown promising results with specific capacitance values reaching 138 F/g at a current density of 1 A/g and maintaining about 83% capacitance retention, indicating excellent electrochemical performance. Various studies have highlighted that while transition metal oxides such as Co_3O_4 or RuO_2 are commonly used in supercapacitor applications, TiO_2's combination with conductive carbon materials significantly enhances performance metrics like power density and energy storage capabilities [12].

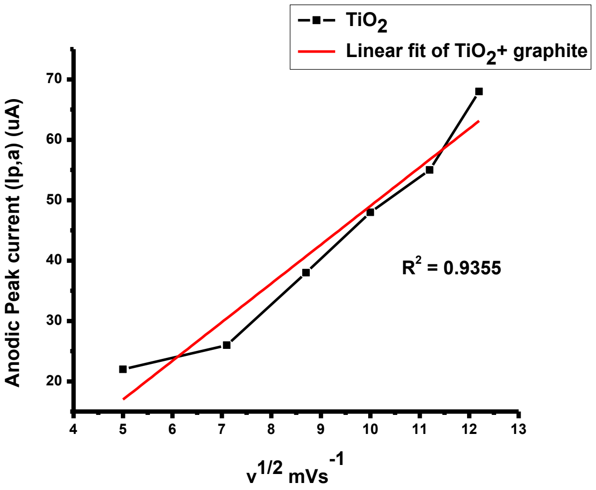


Fig. 6. Graph of Rankles-Servick plot at the electrode at the square root of different scan rates 25, 50, 75, 100, 125, and 150 mVs^{-1}.

Conclusion

The electrochemical oxidation and preparation of ferricyanide using a carbon paste electrode modified with TiO_2 have demonstrated encouraging outcomes in improving the electrode's electrochemical characteristics. Enhancement of the carbon paste electrode's sensitivity, selectivity, and stability for ferricyanide oxidation has been shown through the addition of TiO_2 nanoparticles. Greater electrocatalytic activity towards ferricyanide oxidation results from the presence of TiO_2 on the electrode surface, which increases the surface area available for electron transfer. By lowering the overpotential needed for the oxidation reaction, this modification also contributes to increased electrochemical process efficiency.

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Електроди з вугільною пастою на основі двоокису титану, як перспективні електрохімічні датчики

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У дослідженні детально розглянуто синтез та електрохімічне окислення фериціаніду з використанням електрода з вугільної пасти (CPE), модифікованого TiO_2. Мета полягала у створенні ефективного та надійного електрохімічного датчика окислення фериціаніду, окисно-відновного зонда, який часто використовується в різних аналітичних програмах. Крім того, синтезовано TiO_2-модифікований CPE для підвищення електрокаталітичної активності та чутливості до окислення фериціаніду. Для характеристики модифікованого датчика використовували рентгенівську дифракцію (XRD), скануючу електронну мікроскопію (SEM) та інфрачервону спектроскопію з перетворенням Фур'є (FTIR). Аналіз фериціанідів показав, що після модифікації TiO_2 площа поверхні графіту значно збільшилася. Вплив швидкості сканування на процес електрохімічного окислення, досліджений при різних швидкостях сканування, показав сильну кореляцію між двома процесами (коефіцієнт кореляції, R2 = 0,94). Порівняно з немодифікованим електродом результати експерименту показали, що CPE, модифікований TiO_2, продемонстрував кращі електрохімічні характеристики та вищу пікову густину струму, але нижчий потенціал окислення. Дослідження дає нове уявлення про дизайн та оптимізацію електродних матеріалів для застосування в електрохімічному зондуванні, припускаючи їх потенційне використання в різних майбутніх застосуваннях.

Ключові слова: Електрод на основі вугільної пасти, електрохімічне окислення, діоксид титану, окислення фериціанідом, електродні матеріали.