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Full Paper

ZnO QDs: Synthesis, Electrochemical, and Spectroscopic Characterizations, and Application as an Electrode Modifier in a Voltammetric Study

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Abstract- Zinc oxide quantum dots (ZnO QDs) possess unique optical, electronic, and chemical properties due to the quantum confinement effect, making them ideal for sensing applications. Also, advances in sol-gel synthesis allow precise control over size, shape, and surface chemistry, improving functionality. By adjusting synthesis parameters, their effectiveness in optical and electrochemical applications has been increased. However, further research is needed to understand the relationship between synthesis parameters and performance fully. In this paper, ZnO QDs were synthesized using sol-gel techniques. The synthesis parameters, such as precursor concentrations, pH, temperature, and reaction time, were optimized to control the size, morphology, and properties of the ZnO QDs. The electrochemical characterization of the ZnO QDs was then studied by cyclic voltammetry utilizing $K_4[Fe(CN)_6]$, as a known electrochemical probe in biosensing, in 0.1 M KCl solution. Also, a blood sample was used to find the electrochemical behavior of the ZnO QDs modified on a glassy carbon electrode (GCE) in a biological matrix. It was found that the modified working electrode, ZnO QDs/GCE, acts as an anti-oxidative in an alkaline blood medium and oxidative in the electrolyte of KCl.

Keywords- ZnO QDs; Cyclic voltammetry; Electrode Modifier; Redox reaction; Biosensor

1. INTRODUCTION

Zinc oxide (ZnO) quantum dots (QDs) are nanoscale semiconducting particles that exhibit exceptional optical and electronic characteristics due to their diminutive size. Typically measuring less than 10 nanometers in diameter, these nanomaterials display properties that are influenced by both their size and surface chemistry, which enhances their versatility across a wide array of applications. ZnO QDs are characterized by high fluorescence, remarkable stability, and significant biocompatibility, making them ideal candidates for various biological uses, such as bioimaging, drug delivery, and biosensing. Furthermore, ZnO QDs have gained attention as effective platforms for biosensors, attributed to their sensitivity, selectivity, and ability to undergo functionalization [1].

ZnO QDs are particularly appealing in comparison to other quantum dots due to their composition of metals with lower toxicity, while still retaining desirable photochemical and optical properties. However, their potential toxicity remains a critical consideration [2]. Synthesis of ZnO QDs is typically achieved via wet chemical methods, such as alkaline-activated hydrolysis and condensation of zinc acetate solutions. For instance, ZnO QDs with diameters of 2.5–4.5 nm have demonstrated selective sensitivity for NO detection at low operating temperatures (200°C) with concentrations as low as 2 ppm. At higher temperatures (above 350 °C), the QDs exhibit greater responses to acetone and methanol, illustrating their potential for developing ZnO-based gas sensors [3].

ZnO, as one of the most widely utilized metal oxides, has been incorporated into green synthesis methods to produce starch-coated ZnO (ZnO/starch) QDs. These nanoparticles exhibit a spherical morphology with sizes ranging from 5 to 10 nm, as revealed by transmission electron microscopy (TEM). Their solar photocatalytic activity was evaluated using Rhodamine B (RhB) dye, where ZnO/starch QDs demonstrated superior decomposition of RhB compared to unmodified ZnO QDs after 120 minutes of exposure [4].

In the context of biosensing, ZnO QDs synthesized through deposition methods have been characterized using techniques such as TEM, UV-vis spectroscopy, and Raman spectroscopy. These nanoparticles have been employed to stabilize acidic enzymes, such as uricase, on nanostructured guiding layers for detecting uric acid [5]. Furthermore, ZnO nanoparticles (NPs) in combination with multi-walled carbon nanotubes (MWCNTs) have been used to modify carbon paste electrodes (CPEs) for the electrochemical detection of pharmaceuticals, such as naproxen, at biological pH levels. The ZnO/MWCNT-modified electrodes demonstrated high sensitivity and selectivity in voltammetric determinations, emphasizing the role of ZnO QDs in pharmaceutical analysis [6].

Electrochemical techniques leveraging the properties of QDs offer numerous advantages, including high accuracy, sensitivity, low cost, and rapid analysis. For instance, electrochemical sensors based on ZnO QDs have been tested using cyclic voltammetry, differential pulse voltammetry, and electrochemical impedance spectroscopy. These sensors exhibited excellent

electrocatalytic activity for the detection of thiourea at a high pH of 12.0 and showed selectivity even in the presence of organic compounds and anions. Their application in real samples, such as farmland water and fruit juice, demonstrated excellent recovery values, showcasing their potential for environmental and food safety monitoring [7,8].

This study aims to explore and expand the applications of ZnO QDs in developing highly efficient electrochemical sensors, focusing on their structural and functional versatility, to address challenges in the biological, environmental, and pharmaceutical fields.

ZnO QDs were synthesized through a sol-gel method, then characterized with various techniques, and finally, their electrochemical behavior was studied using cyclic voltammetry in $K_4[Fe(CN)_6]$ and 0.1 M KCl solution, as a known electrochemical probe in biosensing.

2. EXPERIMENTAL SECTION

2.1. Chemicals

Zinc nitrate ($Zn(NO_3)_2.6H_2O$ 98%) and NaOH 99%) were received from Central Drag House CDH company (India). Potassium chloride (KCl 98%) and $K_4[Fe(CN)_6]$ were obtained from Sinopharm Chemical Reagent Co, Ltd. (SCRC), (China), HCl from BDH Company. For this work, deionized water was used; healthy human blood from Iraq-Baghdad Imamen Kadhmiyan Teaching Hospital was received.

2.2. Instruments

UV/Vis Spectrophotometer (UV-199I): this was used to characterize the level energy of quantum resulting from quantum confinement [Manufacturer: Shimadzu]. Transmission Electron Microscope: this was used to calculate the size of nanoparticles, measured in nm [Manufacture: JOEL-2100]. FE-SEM: it provided robustness of the synthesis method employed and the quality of the resulting nanostructures, [Manufacturer: Shimadzu]. XRD (PW1730): was used to characterize (002), (004), and (006) diffraction peaks at 2θ values of 32^o and 34.6^o, and 36^o respectively, [Manufacturer: Philips]. Cyclic voltammetry: was used as a potential station of type EZST 12051401 to show the cyclic voltages of the results in the personal computer [Manufacturer: NuVant Systems, Inc USA].

2.3. Synthesis of ZnO QDs

Nanoparticle zinc oxide was prepared by the sol-gel method, where 15 grams of zinc nitrate were weighed, dissolved in 300 mL of ion-free distilled water, and placed on an esterified until complete dissolution for 15 minutes at a temperature of 60°C. In another bowl, 8 grams of sodium hydroxide was dissolved in 100 mL of ethylene alcohol and placed on the sterilizer until it dissolved for 10 minutes without heat. Next, sodium hydroxide dissolved in alcohol was added to zinc nitrate dissolved in water using a syringe. The mixture was distilled for one hour,

during which the formation of molecules occurred. Quantum dots for nano-zinc were observed, with the solution displaying a bright yellow color under a UV transilluminator, as illustrated in Figure S1 [9-11].

2.2. Preparation of Modified Working Electrode ZnO QDs/GCE

The glass carbon electrode (GCE) with a diameter of 3 mm was polished. The working electrode was washed for 10 minutes in an ultrasonic bath and dried by air [12]. The mechanical entrapment method has been used to modify the GCE electrode with ZnO QDs.

2.3. Electrochemical Characterization

The electrochemical analysis of the platform was performed utilizing cyclic voltammetry with the EZstat potentiostat manufactured by NuVant Systems Company (USA). This potentiostat was interfaced with a 15 mL quartz cell that housed three electrodes: a glassy carbon electrode (GCE) serving as the working electrode, a silver/silver chloride electrode (Ag/AgCl) functioning as the reference electrode, and a platinum wire acting as the auxiliary electrode. Each of these electrodes was connected to the potential terminal and integrated with the user's personal computer, as depicted in Figure S2 [13].

3. RESULTS AND DISCUSSION

3.1. Spectroscopic Characterization (UV-analysis)

ZnO QDs exhibit sharp excitation peaks in their UV absorption spectra at 360 nm, which are characteristic of quantum energy levels resulting from quantum confinement. The position and intensity of these interesting peaks can provide information about the size, size distribution, and crystallinity of ZnO QDs (Figure 1A).

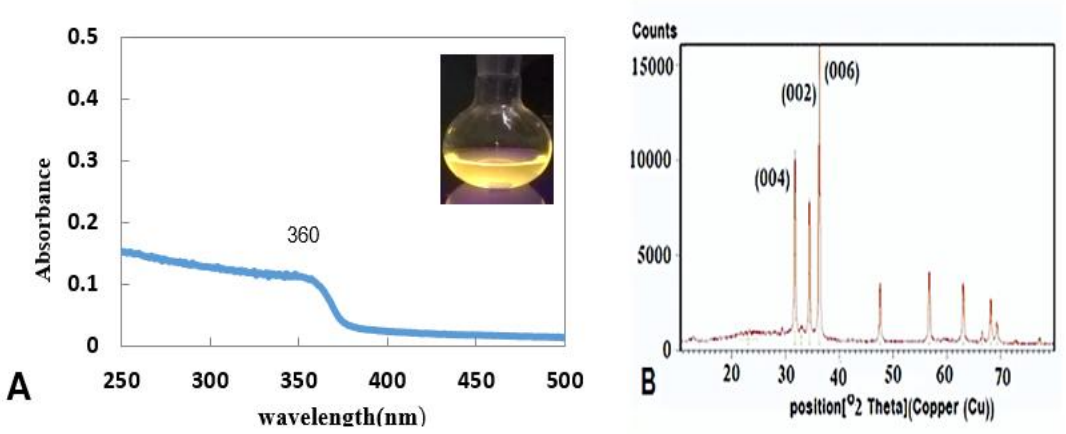

Figure 1. (A) Zinc oxide quantum dots under 360 nm ultraviolet light; and (B) UV spectroscopy of ZnO QDs B: XRD analysis of ZnO QDs

3.2. X-Ray Diffraction (XRD) Analysis

The XRD spectrum of the prepared ZnO QDs shown in Figure 1B exhibited characteristic (002), (004), and (006) diffraction peaks at 2θ values of 32º and 34.6º, and 36º respectively, indicating the crystalline nature of the ZnO QDs. This is due to the intercalation process but still keeping the planar stacking structure [14].

3.3. Field Emission Scanning Electron Microscope (FESEM) and Transmission Electron Microscope (TEM) Analysis

Figures 2A and B provide a detailed visual representation of the spherical morphology exhibited by zinc oxide quantum nanoparticles, as revealed through Field Emission Scanning Electron Microscopy (FE-SEM) analysis. The images clearly depict the uniform shape and size distribution of the nanoparticles, highlighting their distinct spherical characteristics. Furthermore, the average particle size obtained from the FE-SEM analysis is consistent with the measurements derived from Transmission Electron Microscopy (TEM) analysis. This correlation between the two analytical techniques reinforces the reliability of the observed particle size and morphology, suggesting that the zinc oxide quantum nanoparticles possess a well-defined structure that is crucial for their potential applications in various fields, such as electronics, photonics, and biomedical engineering. The agreement in particle size measurements from both FE-SEM and TEM underscores the robustness of the synthesis method employed and the quality of the resulting nanostructures.

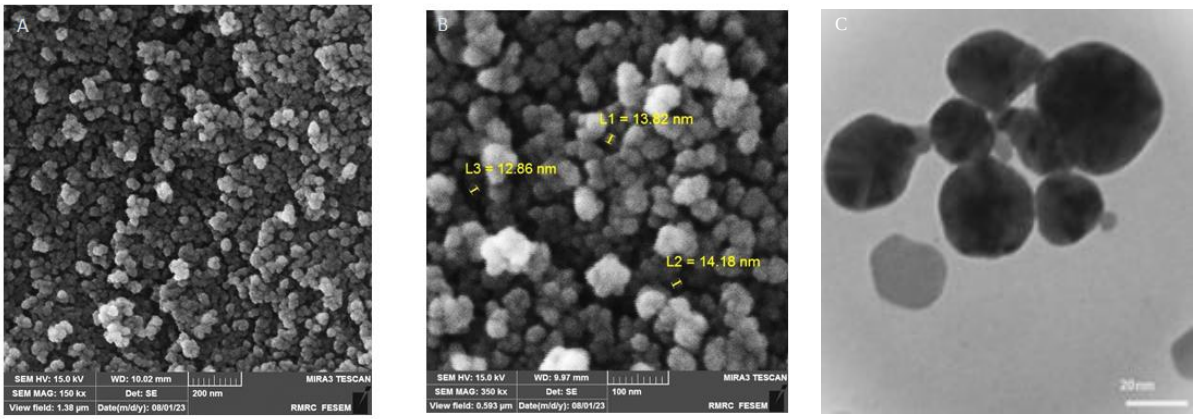

Figure 2. (A) and (B) FE-SEM of zinc oxide quantum dot nanoparticles; Scale bar in Figure A and B are 200 and 100 nM, respectively; (C) TEM of ZnO QDs

Figure 2C presents characteristic transmission electron microscopy (TEM) images of zinc oxide nanoparticles (ZnO NPs) synthesized via the sol-gel method. The TEM images reveal that the morphology of the ZnO NPs is predominantly spherical, with sizes measuring 10 nm and 5 nm. The analysis suggests that the employed method is effective for the production of

zinc oxide nanoparticles in either spherical or semi-spherical forms, with size variations contingent upon the concentrations of the precursor materials. Additionally, there is an observed tendency for these particles to aggregate, resulting in larger dimensions that can reach the microscopic scale when influenced by water-polarized electrodes.

3.4. Electrochemical Characterization

Cyclic voltammetry (CV) is a common electrochemical technique used to investigate the redox behavior and electrochemical properties of materials, including semiconductor quantum dots (QDs) such as ZnO QDs. This study fucoses on the redox peak currents in cyclic voltammogram, different studies determined the electrochemical properties of ZnO QDs as in the following:

ZnO QDs as modified nanomaterials on the glassy carbon electrode were used to develop a new sensor to study the electrochemical properties of the modified electrode (GCE/ ZnO QDs) using $K_4[Fe(CN)_6]$ in 0.1 M KCl solution at different concentrations, pH, scan rate, and reliability with stability study.

In this investigation, two types of working electrodes were utilized: the first a glassy carbon electrode (GCE), and the second a modified electrode composed of zinc oxide quantum dots (ZnO QDs) on GCE. The cyclic voltammogram depicting the behavior of iron ions in a 0.1 M KCl solution is presented in Figure 3 for both the GCE and the modified ZnO QDs/GCE electrode. The results indicated that the redox current peaks associated with the reversible Fe(II) – Fe(III) reaction were significantly enhanced when using the modified ZnO QDs/GCE compared to the standard GCE. This suggests that ZnO QDs nanoparticles serve as highly sensitive materials, suitable for various applications.

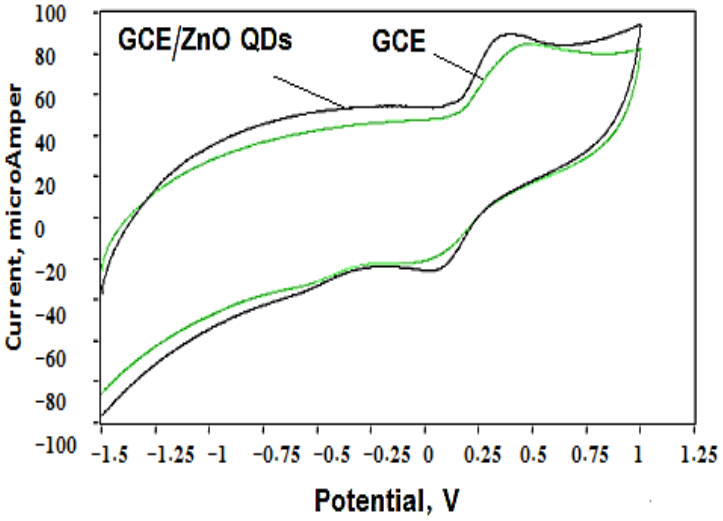

Figure 3. Cyclic voltammogram of 0.1 M $K_4Fe(CN)_6$ in 0.1M KCl on each of GCE and GCE/ZnO QDs with a Ag/AgCl reference electrode at scan rate of 0.1 $Vsec^{-1}$

3.4.1. Effect of Varying $K_3[Fe(CN)_6]$ Concentration on ZnO QDs / GCE

The modified electrode, which consists of zinc oxide (ZnO) quantum dots deposited on a glassy carbon electrode (GCE), was utilized in aqueous solutions for the detection and quantification of trace amounts of iron ions in both the ferrous (Fe^{2+}) and ferric (Fe^{3+}) states. This detection was achieved through the technique of cyclic voltammetry, a powerful electrochemical method that allows for the analysis of redox reactions.

The ZnO quantum dots on the GCE exhibited remarkable electrochemical activity towards both Fe(II) and Fe(III) ions. This was assessed by monitoring the oxidation-reduction current peaks associated with the redox couple of potassium ferricyanide ($K_4[Fe(CN)_6]$) at low concentrations. The results of these experiments are visually represented in Figure 4A, which illustrates the distinct current peaks corresponding to the oxidation and reduction processes of the iron ions.

To further quantify the relationship between the concentration of $K_4[Fe(CN)_6]$ and the observed redox currents, a calibration curve was constructed. This curve was generated by analyzing the oxidation-reduction behavior of $K_4[Fe(CN)_6]$ across a range of concentrations from 0.1 mM to 0.45 mM in a 0.1 M potassium chloride (KCl) solution, as shown in Figure 4B. The calibration data yielded a linear relationship described by the equation y = 83.81x + 38.452, where y represents the current response and x denotes the concentration of $K_4[Fe(CN)_6]$. This relationship exhibited a high degree of correlation, with a coefficient of determination (R^2) of 0.986, indicating excellent sensitivity and reliability of the sensor for detecting low concentrations of iron ions.

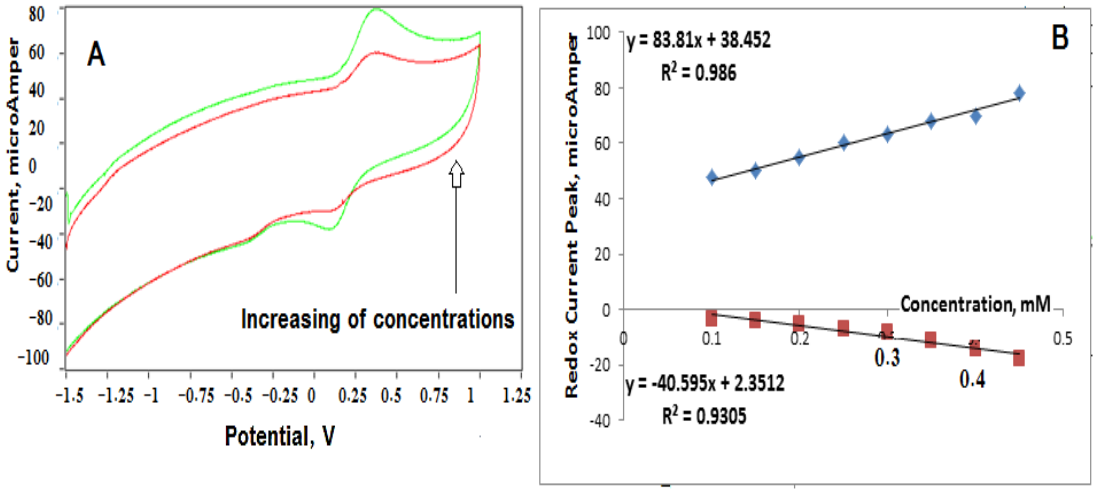

Figure 4. (A) Cyclic voltammogram of 0.1 M $K_4Fe(CN)_6$ at different concentrations in 0.1 M KCl on GCE/ZnO QDs as working electrode against Ag/AgCl as reference electrode at a scan rate of 0.1 $Vsec^{-1}$; (B) Relationship between oxidation-reduction current peaks against different concentrations of 0.1 M $K_4Fe(CN)_6$ (0.1–0.45 mM) in 0.1 M KCl on GCE/ZnO QDs as working electrode against Ag/AgCl as reference electrode at scan rate of 0.1 $Vsec^{-1}$

Conversely, the reverse reaction, which corresponds to the reduction of $K_4[Fe(CN)_6]$ back to $K_3[Fe(CN)_6]$, was characterized by the equation y = -40.595x + 2.3512, with a sensitivity represented by an R^2 value of 0.9305. Although slightly lower than the forward reaction, this value still indicates a strong correlation, affirming the sensor's capability to accurately measure the redox behavior of iron ions. The identified relationship between the redox current of iron ions and their differing concentrations indicates that an increase in the concentration of iron ions is associated with a notable improvement in conductivity [15].

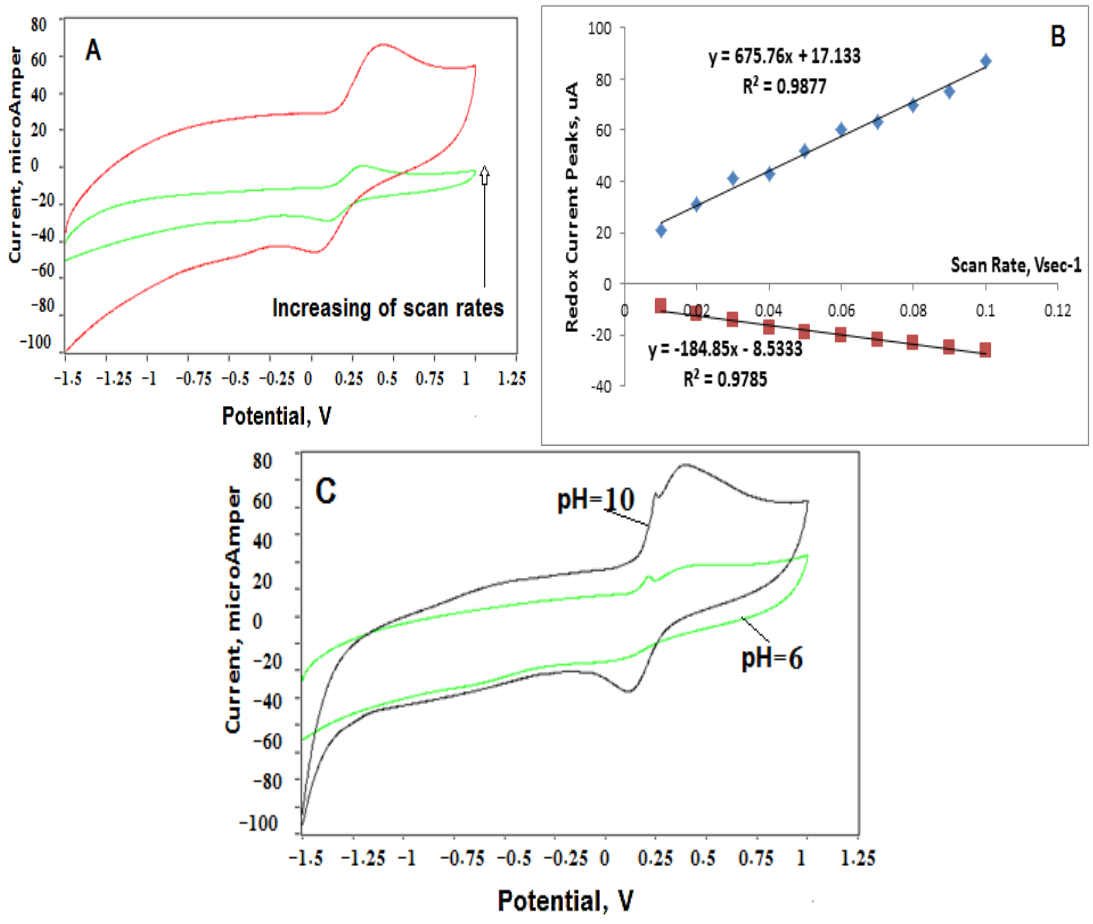

Figure 5. (A) Cyclic voltammogram of 0.1 M $K_4Fe(CN)_6$ in 0.1M KCl on ZnO QDs/GCE as working electrode against Ag/AgCl as reference electrode at different scan rates of 0.01–0.1 $Vsec^{-1}$; (B) relationship between oxidation-reduction current peaks against different scan rates (0.01– 0.1 $Vsec^{-1}$) 0.45 M $K_4Fe(CN)_6$ in 0.1M KCL on ZnO QDs / GCE as working electrode against Ag/AgCl as reference electrode at scan rate of 0.1 $Vsec^{-1}$; (C) The cyclic voltammogram of 0.1 M $K_4Fe(CN)_6$ was obtained at various pH levels in a 0.1 M KCl solution, utilizing ZnO quantum dots (QDs) on a glassy carbon electrode (GCE) as the working electrode, with an Ag/AgCl electrode serving as the reference electrode, at a scan rate of 0.1 $Vsec^{-1}$

3.4.2. Effect of varying scan rates and pH on ZnO QDs / GCE

The investigation focused on the influence of varying scan rates on the cyclic voltammograms of a 0.1 M $K_3[Fe(CN)_6]$ solution in a 0.1 M $KClO_4$ supporting electrolyte, utilizing a modified ZnO quantum dots/glassy carbon electrode (GCE) as the working electrode. The scan rates examined ranged from 0.01 to 0.1 V/s. It was observed that the oxidation and reduction currents associated with the Fe(III)/Fe(II) redox couple increased with the scan rate, attributed to heterogeneous kinetics and an ohmic IR drop, as illustrated in Figure 5A.

A linear relationship was established between the oxidation-reduction current peaks and the scan rate, as depicted in Figure 5B, conforming to the equations $y = 675.76x + 17.133$ ($R^2 = 0.9977$) and $y = -184.85x - 8.5333$ ($R^2 = 0.9785$), respectively. The redox current peaks for the Fe(II)/Fe(III) couple exhibited an increase with higher scan rates. In contrast, at slower scan rates, the diffusion layer extends further from the electrode, resulting in reduced diffusion at the electrode surface compared to faster scan rates. Consequently, the current is diminished at slower scan rates and elevated at faster rates, as the flow to the electrode is directly proportional to the current. [16].

The effect of varying pH on the cyclic voltammogram of 0.1 M $K_3[Fe(CN)_6]$ in a 0.1 M KCl solution was studied on ZnO QDs/GCE as shown in Figure 5C. It was found that the redox current of $K_3[Fe(CN)_6]$ increases at solution pH of high acidic value (6) and peaks separation decreases indicating increasing reversibility with decreasing pH. For the electrolyte solution with pH 10, redox peak separation increases with decreasing redox current due to the formation of hydrolyzed Fe(III) species which are rather electro-inactive and irreversible [17].

3.5. Cyclic Voltammetric Study of ZnO QDs / GCE in Blood Medium

Cyclic voltammetry serves as a valuable technique for examining the redox properties of ZnO quantum dots (QDs) on a glassy carbon electrode (GCE) within a blood medium. The characteristics, including the location and magnitude of the oxidation and reduction current peaks, yield insights into the electrochemical behavior of the ZnO QDs, as well as any alterations brought about by the presence of the blood medium. By comparing the cyclic voltammograms obtained in the blood medium with those from a control electrolyte, it becomes possible to discern any matrix effects or the influence of interfering species [18].

3.5.1. Effect of varying pH in Blood Medium on ZnO QDs / GCE

The pH of the blood medium is an important factor that can affect the performance of ZnO QDs when used as a sensing platform in GCE. To improve the performance of the ZnO QDs/GCE system in blood media, it is important to carefully evaluate the pH dependence and determine the optimal pH range that provides the best stability, electrochemical activity, and analyte reactions.

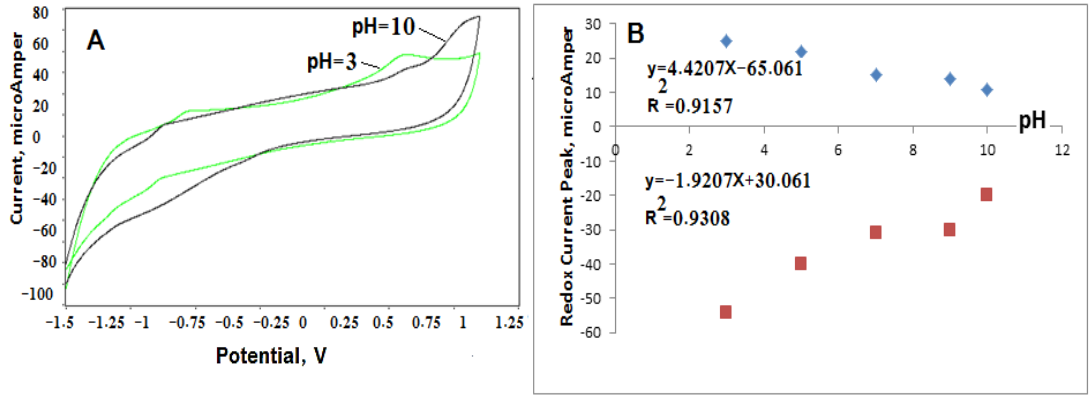

Figure 6. (A) Cyclic voltammograms obtained for varying pH levels in a blood medium using ZnO quantum dots (QDs) on a glassy carbon electrode (GCE) as the working electrode, with an Ag/AgCl electrode serving as the reference electrode, at a scan rate of 0.1 $Vsec^{-1}$; (B) The correlation between the oxidation and reduction current peaks and the varying pH levels of blood on the GCE/ZnO QDs working electrode, referenced against the Ag/AgCl electrode at the same scan rate of 0.1 $Vsec^{-1}$

In general, the physiological pH range of blood (about 7.35–7.45) is an important consideration when designing and evaluating ZnO QDs/GCE sensors for biomedical applications. Strategies such as surface modification or the use of pH buffering agents can be used to maintain optimal pH conditions and enhance sensor performance in blood media. Figure 6A illustrates the cyclic voltammogram of the blood sample at varying pH on the modified working electrode ZnO QDs/GCE, the oxidation current peak of the blood sample was enhanced in acidic pH=3, while decreased in alkaline pH=10 as shown in the relationship between the oxidation current peak against to the varying pH at the range from 3 to 10 in Figure 6B.

3.6. Reliability and stability study

In a cyclic voltammetry study, the results obtained in experiments can be evaluated and the sensitivity of the working electrode is checked by scanning the cyclic voltammogram of the reactant about ten times as shown in Figure S3, which appears to be a good overlap for the CV scan, and then calculate the relative standard deviation (RSD) of the oxidation-reduction peaks have an acceptable value of ±2.5 [19-21].

4. CONCLUSION

The study investigated the synthesis of zinc oxide quantum dots (ZnO QDs) and their electrochemical and spectroscopic properties when used to modify a glassy carbon electrode (GCE). The use of cyclic voltammetry likely provided insight into the redox behavior and

electrochemical activity of ZnO QDs on the modified electrode. The results, such as the successful synthesis of ZnO QDs, their stable immobilization on GCE, the observed electrochemical of ZnO QDs has two oxidation current peaks and one reduction peak in an electrolyte, and possibly any applications or implications for the characterization of the ZnO QDs/GCE system. It was found that the modified working electrode ZnO QDs/GCE acts anti-oxidative in an alkaline blood medium and oxidative in an electrolyte of KCl. ZnO QDs can also be considered as a substance used in electrochemistry applications to determine the levels of oxidizing substances in the blood, such as blood pollutants of heavy metals.

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Declarations of interest

The authors declare no conflict of interest in this reported work.

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