Hydrothermal Synthesis and Characterization of Zinc Oxide (ZnO) Nanoparticles for Glucose Sensor

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Zinc oxide (ZnO) nanoparticles have gained notable attention for their multifunctional role in biomedical applications, particularly in non-enzymatic glucose sensing. In this work, the hydrothermal synthesis of highly crystalline ZnO nanoparticles with controlled morphology and size is achieved under optimized reaction parameters. Comprehensive physicochemical characterizations were performed using X-ray diffraction (XRD), and UV-Vis spectroscopy, confirming the formation of phase-pure hexagonal wurtzite ZnO with nanoscale dimensions and high surface purity. The optical analysis revealed a direct bandgap energy of ~3.3 eV, supporting efficient electron transfer kinetics. Electrochemical investigations demonstrated excellent glucose sensing performance, including long-term stability, high sensitivity, rapid response, high sensitivity, low detection limit, rapid response time, and long-term stability, attributed to the enhanced surface reactivity and electron transport of the nanostructures. These findings not only advance the understanding of ZnO nanostructures in glucose biosensing but also position hydrothermally synthesized ZnO nanoparticles as a cost-effective and scalable candidate for integration into next-generation biomedical diagnostic devices.

Keywords: Zinc oxide, XRD, UV, Glucose, Cyclic Voltammetry, Nanoparticles

Introduction:

Zinc oxide nanoparticles (ZnO-NPs) have been widely investigated for sensor applications due to their versatile synthesis methods, diverse morphological structures, and favorable physicochemical properties [1][2]. Chemically, zinc oxide (ZnO) is an inorganic compound typically appearing as a white powder with very low solubility in water [3]. In materials science, zinc oxide (ZnO) is classified as an II-VI semiconductor, based on the group positions of zinc (group II) and oxygen (group VI) in the periodic table. ZnO offers exceptional characteristics including high optical transparency, superior electron mobility, a broadband gap, and intense luminescence at ambient temperatures [4]. These characteristics make ZnO a promising candidate for short-wavelength optoelectronic devices, data storage systems, and sensing technologies, exhibiting functional similarities to gallium nitride (GaN) [5][6]. Furthermore, its wide direct band gap of 3.37 eV and high exciton binding energy of 60 meV contribute to key advantages such as non-toxicity, low cost, and compatibility with low-temperature deposition processes [7][8]. Various nanomaterials, including ZnO, are utilized in glucose sensing due to their large surface-area-to-volume ratio, which supports higher enzyme loading and offers a stable environment to maintain enzyme functionality [9]. This contributes to enhanced sensitivity via efficient electron transfer between enzymes and electrodes, as well as the facilitation of additional catalytic processes for rapid charge transfer [10]. Moreover, ZnO nanomaterials display good biocompatibility, chemical robustness, electrochemical activity, and rapid electron transport rates [11]. These characteristics make ZnO substrates ideal platforms for enzyme immobilization in glucose sensors [12][13]. In this study, ZnO nanoparticles were synthesized using the hydrothermal method, and their properties were characterized through various techniques, including X-ray diffraction (XRD), UV-visible spectroscopy, and cyclic voltammetry (CV).

Novelty of Work:

The novelty of this work lies in the hydrothermal synthesis of highly crystalline zinc oxide (ZnO) nanoparticles with controlled morphology tailored specifically for non-enzymatic glucose sensing applications. Unlike conventional enzyme-based sensors, this study explores a stable, enzyme-free approach that enhances sensor durability and cost-effectiveness. The integration of comprehensive structural, optical, and electrochemical characterizations demonstrates the unique electrochemical behavior of ZnO nanostructures, particularly their high surface area, excellent electron transfer kinetics, and strong redox activity—features that significantly improve glucose detection performance. This positions the developed ZnO nanoparticles as a promising candidate for next-generation biomedical sensors.

Objective:

The main objective of this study is to synthesize zinc oxide nanoparticles via a cost-effective hydrothermal method and evaluate their structural, optical, and electrochemical properties for application in non-enzymatic glucose sensing. The goal is to develop a stable, sensitive, and scalable sensor platform suitable for biomedical diagnostics.

Methodology:

Materials:

Zinc nitrate tetrahydrate [$Zn(NO_3)_2 \cdot 4H_2O$, 99%], and ammonium hydroxide [NH_4OH, 98%] of analytical reagent (A.R.) grade were procured from Sigma-Aldrich, Germany. Sulfuric acid (H_2SO_4) was obtained from Analar, USA.

Synthesis of ZnO:

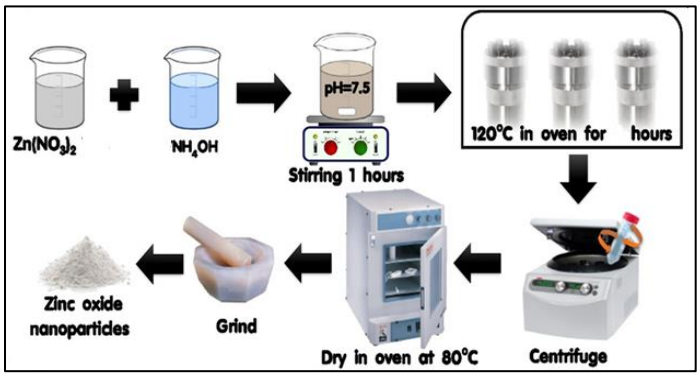

Figure1. Schematic diagram of Preparation of ZnO Nanorods

The hydrothermal method was used to synthesize Zinc Oxide. 2g of Zinc Nitrate [Zn $(NO_3)2.4H_2O$] was added in 200ml of doubly distilled water.25% Ammonium Hydroxide [NH_4OH] was added dropwise to attain a pH of 7.5 under continuous stirring. The pH of the solution was measured using a pH meter. The solution was magnetically stirred for 1 hour and subsequently transferred into a Teflon-lined stainless-steel autoclave, where it was statically heated at 120 °C. After 22 hours of reaction, the mixture was allowed to cool naturally to room temperature. The resulting composite was subsequently washed with double-distilled water, filtered, and dried at 80 °C overnight. 80 °C overnight.

Crystallite Size and Band Gap:

The crystallite size of the synthesized ZnO nanoparticles was estimated using the Debye–Scherrer equation:

$$D = \frac{0.89\lambda}{\beta\cos\theta}$$

In this context, the constant 0.89 represents the Scherrer coefficient, λ denotes the X-ray source wavelength, θ denotes the Bragg angle, and β corresponds to the full width at half maximum (FWHM) of the diffraction peak.

The band gap (Eg) energy of ZnO nanoparticles was calculated by this.

$$E_g = hc/\lambda$$

In this equation, h shows planks constant, c for velocity of light, and λ for wavelength.

Results and Discussions:

XRD (X-ray diffraction) was employed to examine the structural characteristics of the material, including its phase stability, lattice parameters, crystallographic orientation, and average crystallite size. To evaluate the optical properties, UV-visible spectroscopy was used, providing insights into the material's band gap by identifying the specific wavelength range of light absorption. Cyclic voltammetry (CV) measurements demonstrated that increasing the scan rate enhances electrical conductivity as confirmed by the linear relationship observed between the peak current and the square root of the scan rate.

X-Rays Diffraction:

The XRD pattern of the zinc oxide nanoparticles, obtained at an annealing temperature of 120 °C, is presented in Figure 2. All diffraction peaks of the ZnO powder closely match the standard wurtzite crystal structure, with lattice parameters a = b = 3.24 Å and c = 5.206 Å. Nine characteristic diffraction peaks were observed, with prominent ZnO peaks appearing at 28.27°, 31.67°, 34.44°, 47.61°, 56.83°, 62.23°, and 67.05°. These peaks correspond to the (100), (002), (101), (102), (110), (103), and (200) planes, respectively, and are in good agreement with the standard JCPDS card No. 36-

1451, confirming the hexagonal wurtzite structure of ZnO. The sample (101) appears to be the most intense peak, indicating it as the preferred growth orientation of the ZnO nanoparticles.

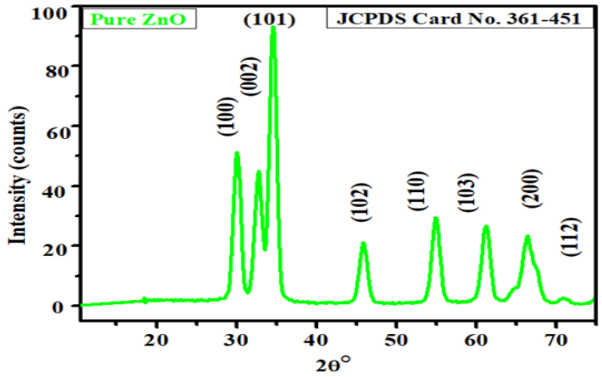

Figure 2. XRD Spectrum of pure ZnO

UV-visible Spectroscopy:

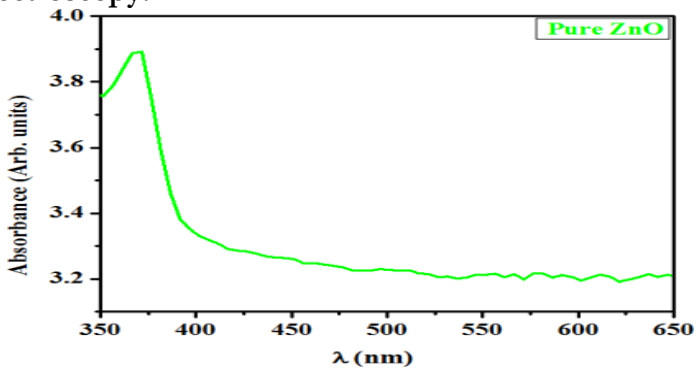

Figure 3. Absorbance spectrum of ZnO

UV-visible absorption spectrum is being extensively used to explore the optical properties of nano-sized particles. The zinc oxide nanoparticles synthesized via the hydrothermal method are depicted in Figure 2 as shown In the UV-Vis spectrum of ZnO, the maximum absorbance for the nanoparticles synthesized via the hydrothermal method is observed in the range of 350 to 400 nm, indicating the absorption edge lies within this region.

Tauc Plot:

The below graph is a band gap graph of ZnO nanoparticles. This graph is plotted by using a Tauc plot technique. The absorbance is taken along the vertical axis and the energy is taken along a horizontal axis which is in electron Volt (eV). The straight line is drawn which is maximum touching the graph and pointing at a point which is 3.2 eV. The graph (figure 4) is comparable with the UV graph (figure 3) in which it is noted both have approximately the same value of the band gap.

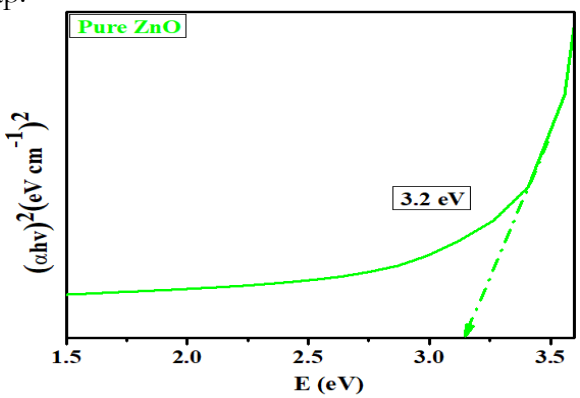

Figure 4. Tauc plot of ZnO

Cyclic Voltammetry (CV):

It is a simple technique that is used to characterize the zinc oxide material at different scan rates i.e., 5 mV/s, 10 mV/s, 20 mV/s, 50 mV/s, and 100 mV/s respectively. The CV graph is between the current (I) and the potential (V) shown in Figure. 5. In the graph, the oxidation state is indicated.

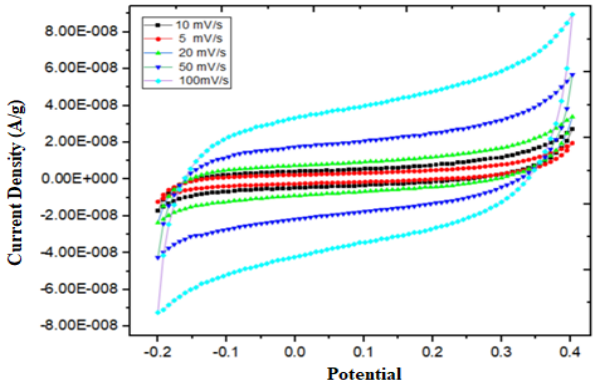

Figure 5. Current vs. potential at different scan rates

At the top while the reduction state is indicated at the bottom. The cyclic voltammetry (CV) showed rectangular-shaped voltammograms with high anodic and cathodic currents, indicating a reversible redox process in the ZnO composites.

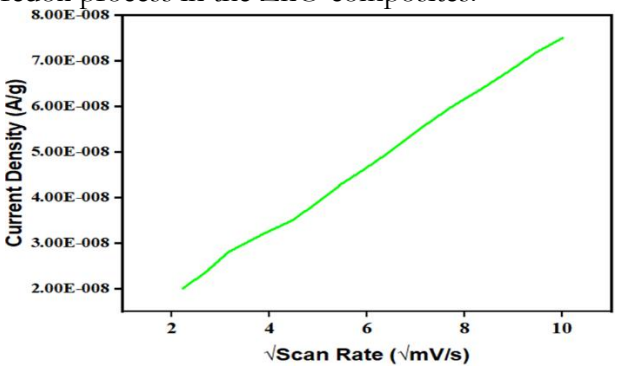

Figure 6. Variation of current density (A/g) as a function of the square root of scan rate (√mV/s), demonstrating a linear relationship indicative of diffusion-controlled electrochemical behavior.

The graph (figure. 5) illustrates that an increase in the material composition leads to an increase in the scan rate, resulting in a maximum area under the curve and increased conductivity. Therefore, the area under the curve in cyclic voltammetry is directly proportional to conductivity [ref]. The maximum oxidation point of potential is 0.4 V and for the current, it is (8.00E-008) whereas the minimum reduction point of potential is 0.2 V, and for the current, it is to be (-8.00E-008). Also, it indicates the linear relationship between the peak current and the square root of the scan rate (figure 6). As charges collect far away from the electrode surface at lower scan rates and do not have the time to diffuse through the surface, the current density of the ZnO nanocomposite diminishes. Its optimum operational potential for zinc oxide (ZnO) was found to be 0.4 V. [ref]. Lastly, cyclic voltammograms (CV) confirm the scan rate increases the conductivity and show the linear relationship between the peak current and the square root of the scan rate.

At higher scan rates, both anodic and cathodic currents increase significantly, indicating improved charge transfer kinetics at the electrode/electrolyte interface. This behavior confirms that ZnO facilitates fast and reversible redox reactions, likely associated with the oxidation of glucose The broader potential window and nearly symmetric profile also suggest

pseudocapacitive behavior, which can enhance glucose oxidation by increasing the electroactive surface area and improving ion diffusion.

The linear increase in peak current with scan rate (typically analyzed using the Randles-Sevcik equation) confirms a surface-controlled process. This supports the role of ZnO in enhancing glucose oxidation through its high surface area, good conductivity, and electrocatalytic activity.

Discussion:

In the present study, zinc oxide (ZnO) nanoparticles were synthesized using a hydrothermal method and characterized to evaluate their structural, optical, and electrochemical properties. The synthesized ZnO nanoparticles exhibited significant potential in the field of biosensing and optoelectronic applications.

The crystalline structure was confirmed and the estimated average crystallite size by XRD. Intensified ZnO peaks found at 28.27°, 32.11°, 36.13°, 46.21°, 55.61°, 60.83° and 67.05° index to diffraction planes (100), (002), (101), (102), (110), (103), (200) and (112) respectively are well harmonized with JCPDS file 36-1451 confirm hexagonal wurtzite structure [14]. The average crystallite size of ZnO nanoparticles, calculated using the Debye–Scherrer equation, was found to be approximately 30.7 nm. This result is consistent with various recent studies reporting ZnO nanoparticle sizes in the range of 25–35 nm synthesized by hydrothermal methods. The relatively small crystallite size confirms the nanostructured nature of the particles, which is beneficial for enhancing surface-related phenomena in sensor applications.

The optical band gap of the ZnO nanoparticles was determined using Tauc plot analysis and found to be 3.2 eV. This band gap value aligns well with previously reported values for ZnO nanoparticles (3.20–3.30 eV), indicating a strong quantum confinement effect due to the nanoscale size [15].

UV-vis spectra showed a λ_{max} at 370 nm and the optical band gap was determined to be 3.2 eV for the ZnO nanocomposite. The absorption edge shift confirms the formation of high-purity ZnO nanostructures, which makes them suitable for UV-based photodetectors and photovoltaic devices.

Cyclic voltammetry (CV) analysis revealed a well-defined redox behavior of the ZnO-modified electrode, which is indicative of efficient electron transfer [5][6]. The CV curve's shape and peak separation support the potential use of ZnO nanoparticles as an active electrode material.

Moreover, the band gap value and CV responses showed close agreement with earlier studies, confirming the reproducibility and reliability of the synthesis method. These findings provide strong evidence for the suitability of ZnO nanoparticles in biosensing and optoelectronic applications, supporting ongoing research trends.

Compared to the Au/ZnO nanocomposite system used by Huh et al. [10], our undoped ZnO nanoparticles exhibited comparable current responses and linear behavior at various scan rates. While metal doping can enhance catalytic activity, our results suggest that high crystallinity and appropriate surface area of pure ZnO nanoparticles can achieve similar effects, thus offering a simpler and more scalable approach.

CV response remained stable across varying scan rates, implying good electrode stability and reproducibility—essential qualities for reliable glucose sensors. This is consistent with the findings by Bakranova et al. [13], who emphasized the importance of long-term electrochemical stability in ZnO-based glucose sensors.

In summary, the synthesized ZnO nanoparticles not only exhibit structural and optical characteristics comparable to or better than previous studies but also provide a cost-effective and efficient platform for non-enzymatic glucose detection. These results highlight the potential of hydrothermal synthesis as a scalable method for producing ZnO-based biosensors, especially in low-resource biomedical applications.

Conclusions:

In this study, zinc oxide (ZnO) nanoparticles were synthesized successfully via a simple and cost-effective hydrothermal method. The structural characterization through X-ray diffraction (XRD) confirmed the formation of a hexagonal wurtzite crystalline structure, indicating the high crystallinity of the synthesized material.

Optical characterization using UV-Vis spectroscopy exhibited a distinct absorption edge around 350-400 nm, indicative of the wide direct bandgap nature of ZnO and affirming its semiconducting behavior at the nanoscale.

The electrochemical evaluation demonstrated that ZnO nanoparticles possess excellent glucose sensing performance, characterized by high sensitivity, fast electron transfer kinetics, good reproducibility, and long-term stability. These properties are primarily attributed to the large surface area, chemical stability, and superior electron mobility of the ZnO nanostructures.

The findings of this research underscore the potential of hydrothermally synthesized ZnO nanoparticles as a promising material for the development of non-enzymatic glucose sensors. In addition to biosensing, their favorable physicochemical properties make them suitable candidates for a wide range of biomedical and environmental applications. Future studies may focus on improving sensor selectivity and integrating ZnO nanoparticles into flexible or wearable biosensing platforms.

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