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Production of Carbon nanoparticle-decorated ZnO Nanostructures and Pure ZnO for Extremely Effective Glucose Sensors.

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ABSTRACT

This work presents a viable method for synthesis carbon nanoparticles decoration zinc oxide nanostructured films and synthesizing pure ZnO films for glucose sensors. Anodization was used to grow zinc oxide nanoparticles on zinc foil with an average particle size of 22.77 nm, and hydrothermal synthesis was used to create carbon nanoparticles. The ultrasonication method was used to decorate ZnO film nanocomposites with carbon nanoparticles. several tests were carried out, including observations of the microstructure, analyses of the crystallinity, and investigations of the electrochemical properties. X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDX), field-emission scanning electron microscopy (FE-SEM), transmission electron microscopy (TEM), and photoluminescence (PL) measurements were used to determine the nanostructures. The electrochemical analysis of the CNPs/ZnO nanostructures greatly improved the sensitivity to glucose compared with pure zinc oxide nanostructures, with a sensitivity of (1975 $\mu A.mM^{-1}.cm^{-2}$), the CNP/ZnO nanostructures improved glucose sensing. The low detection limit is (0.3 mM), and the linear range is 0.3 mM to 0.7 mM.

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إنتاج هياكل نانوية من أكسيد الزنك المزخرفة بجسيمات الكربون النانوية وأكسيد الزنك النقي لأجهزة استشعار الجلوكوز الفعالة للغاية

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الكلمات المفتاحية:

مستشعرات الجلوكوز، مستشعرات حيوية غير إنزيمية، مستشعرات حيوية لأكسيد الزنك، طريقة الأكسدة، الطريقة الحرارية المائية.

الـــخُـــلاصـــة

يقدم هذا العمل طريقة قابلة للتطبيق لتصنيع جسيمات الكربون النانوية لتزيين أغشية أكسيد الزنك النانوية وتصنيع أغشية الزنك النانوية النقية لمستشعرات الجلوكوز، تم استخدام الأكسدة الأنودية لتنمية جسيمات أكسيد الزنك النانوية على رقائق الزنك مع متوسط حجم للجسيمات (nm22.77)، وتم استخدام التخليق الحراري المائي لإنشاء جسيمات نانوية كربونية. تم استخدام طريقة الموجات فوق

الصوتية لتزيين مركبات النانو من فيلم ZnO بجسيمات نانوية كربونية. تم إجراء عدد من الاختبارات، بما في ذلك ملاحظات البنية الدقيقة، وتحليلات التبلور، والتحقيقات في الخصائص الكهروكيميائية. تم استخدام حيود الأشعة السينية XRD , مطيافية الأشعة السينية المشتتة للطاقة (EDX) , والمجهر الإلكتروني الماسح بانبعاث المجال (FE-SEM)، والمجهر الإلكتروني النافذ (TEM) , وقياسات التلألؤ الضوئي (PL) لتحديد هياكل النانو , عند مقارنتها بهياكل نانوية من أكسيد الزنك النقي أدى التحليل الكهروكيميائي لهياكل النانو CNPs/ZnO إلى تحسين حساسية الجلوكوز بشكل كبير مع حساسية مقدارها ($1795\ \mu A\ .\ mM^{-1}\ cm^{-2}$)وحسنت هياكل النانو المزخرفة (CNP/ZnO) من استشعار الجلوكوز، اقل مقدار للكشف كان (0.3 mM)، والنطاق الخطي هو (0.3 mM – 0.7mM)

1. INTRODUCTION

Diabetes is a dangerous medical condition that claims many lives each year. Despite this, diabetic patients still need to have regular blood glucose checks to avoid developing new problems. Thus, it is essential to develop a fast, incredible, accurate, stable, and reliable method of measuring glucose levels [1-5], Glucose monitoring with sensors is a key topic of research because blood glucose imbalance is the main cause of diabetes mellitus, which is a major global public health concern. Studies have revealed that dietary changes and an increase in obesity rates can raise blood glucose levels[6]. Nanotechnology encompasses the study, creation, and use of materials, systems, and devices that typically have sizes smaller than 100 nm. Nanotechnology is becoming an increasingly important component of biosensor development[7, 8]. The word "biosensor" correctly suggests sensing through a biological component, and all biosensors use this concept to some extent[9]. Biosensors are analytical tools that can convert a biological response into an electrical signal[10]. In recent decades, various glucose biosensor types have been studied, such as optical, electrochemical, and acoustic ones. Among them, electrochemical sensors are the most sophisticated and widely used[11]. Electrochemical biosensors are a subclass of chemical sensors that can be characterized by their low detection limits recognition mechanisms[12]. High sensitivity, selectivity, accuracy, and affordability are some of the benefits of electrochemical sensing. Because they are highly sensitive, selective, and user-friendly, electrochemical glucose biosensors are a well-liked option for glucose monitoring[13, 14]. Based on the detection mechanism, electrochemical sensors can be broadly divided into two categories: enzymatic and non-enzymatic[15, 16]. Metal oxides have an excellent stability, inexpensive, and have a high catalytic capacity. They have been seen as very promising alternatives for the electrocatalytic materials of glucose sensors[17]. Materials known as nanostructured metal oxides, or NMOs, have gained attention for their ability to immobilize biomolecules with high biological activity, all of which improve sensing properties[18]. Because of ZnO's unique properties, scientists are very interested in figuring out how it might be used in a variety of applications in electronics and optoelectronics. ZnO is an important technological material whose distinct functional and nano-morphological features have drawn extensive attention in the past ten years[19-22]. Furthermore, because the ZnO nanostructures are biocompatible, they can be easily surface-functionalized and interfaced with chemical and biological materials at various pH and temperature levels[23].Carbon nanoparticles (CNPs) have attracted great attention due to their electrical and physiochemical properties, particularly their optical qualities and electrochemical activity. Because of their high, controlled photoluminescence, nontoxicity, and biocompatibility, CNPs are promising candidates for bio imaging and bio sensing[24, 25]. Because of the hydrothermal method's ease of solution control, high reactivity of the reactants, low energy consumption under hydrothermal conditions, and low air pollution, a wide range of materials, including carbon nanoparticles, have been created[26]. Nanomaterials can be prepared using a variety of techniques. In this study, carbon nanoparticles were prepared via the hydrothermal method, zinc oxide was prepared using the anodization method, and carbon nanoparticles were then decorated onto zinc oxide using the ultrasound method.

2. Experimental part

2.1. Synthesis ZnO NPs by Anodization Method

Every electrochemical experiment was carried out at room temperature. High-purity (99.9%) zinc foil (BDH Company) with a 250 µm thickness was cut into a 1 cm × 1 cm square and cleaned with a solution made from (10ml) pure ethanol (C2H5OH) and (5ml) pure acetone (C3H6O), both were made by ALPHA CHEMIKA. After spending (15 minutes) in an ultrasonic cleaner (JEKEN PS-30, CHINA), the beaker was dried and cleaned using DI water. Using a magnetic stirrer hotplate from STUART Scientific, UK, the anodizing electrolyte was created by dissolving 0.2 M NaOH made in Schlau, Spain, in 50 milliliters of DI water and swirling it for five minutes. Electrochemical anodization was carried out. There are two electrodes in a teflon cylinder. A graphite sheet was used as the counter electrode and zinc foil as the working electrode, and both electrodes were immersed in a NaOH solution. Two centimeters was the adjusted electrode separation. Anodization by electrochemical means was carried out for thirty minutes using two electrodes and an anodizing voltage of 6 V. On the surface of the Zn foil, a light layer formed after the reaction. ZnO nanoparticles were created by annealing the sample to 200 °C for two hours; following this, the Dark Gray was produced on the surface of the Zn foil. A schematic is shown in Figure (1) the setup illustrates the anodization experiment. After being removed from the solution, the Zn foil was cleaned with DI water, dried, and examined

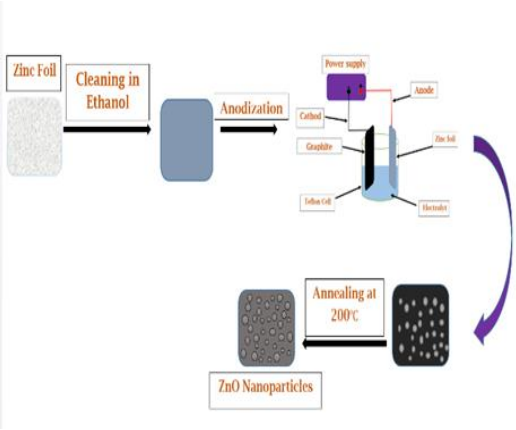


Figure.1 Anodizing System Configuration

2.2. Synthesis of Carbon Nanoparticles (CNPs) by Hydrothermal

The process of creating CNPs involved dissolving 0.1 M of ascorbic acid from China in 160 ml of DI water, stirring it for 10 minutes in a magnetic stirrer hotplate from (Stuart Scientific, UK) , and then applying it in a stainless steel autoclave and applying it in a box furnace (CARBOLITE, ENGLAND) for 6 hours at 180°C. A schematic of the hydrothermal technique used to prepare carbon nanoparticles is shown in Figure (2).

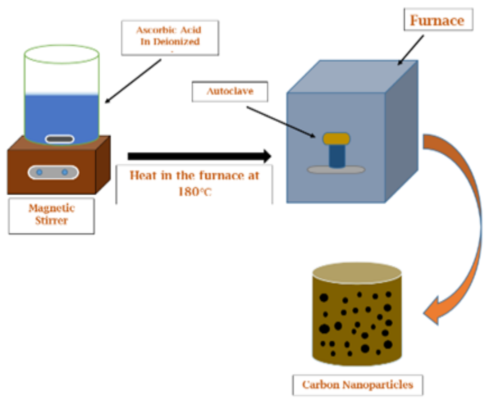


Figure.2 The hydrothermal method for creating CNPs

2.3. Preparation of ZnO/CNP by ultrasonic Method

The ZnO sample should be fully submerged in the liquid after adding the carbon nanoparticle-containing liquid to a petri dish. Once placed inside the ultrasonic machine, the petri dish was left there for five minutes. The preparation process for applying carbon nanoparticles to ZnO foil's surface using the ultrasonic method is shown in Figure 3.

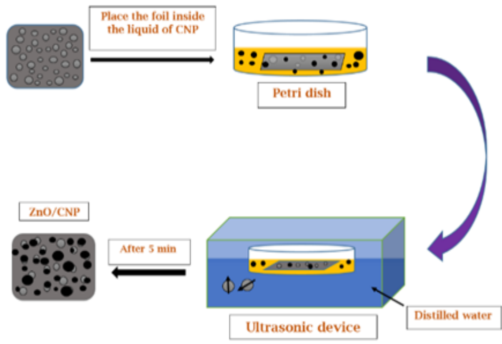


Figure.3 Schematic of the preparation of ZnO foil surface decoration with carbon nanoparticles using the ultrasonic method

2.4. Characterization process

A set of instruments was used to characterize the properties of the ZnO films that were produced, and XRD patterns from the deposited films were obtained with a Bruker D8 X-ray device, origin (λ= 1.5418 Å), and a CuKα radiation source. Furthermore, the structural properties and morphology were examined using the Field-Emission Scanning Electron Microscopy (FESEM) model Mira3 Tescan from (Czechia), and the elemental composition of the materials was characterized using Energy Dispersive Spectroscopy (EDX). The device used to analyze the morphology of CNP formations is the TEM model AB LEO912, manufactured by ZEISS in Germany.

2.5. Electrochemical measurements

The three-electrode system was used for all experiments. The electrochemical measurements were performed using a Keithley 2430-C Source Meter (SMU) device with a contact check/GPIB interface and 1 kW pulse mode (A Tektronix Company) shown in Figure (4). Ag/AgCl served as the reference electrode, while the working electrode represented ZnO nanoparticles and the counter electrode represented a graphite sheet. The ZnO and CNP/ZnO chronoamperometric responses were tested at 1 V. Cycle voltammetry (CV) and IV were used to characterize ZnO and CNP/ZnO films in 0.1M (NaOH) (basic electrolyte) at a scan rate of 100 mV with varying glucose concentrations (0.3, 0.5, 0.7) mM at 1 V, the responses of ZnO NPs and CNP/ZnO were measured by chronoamperometry

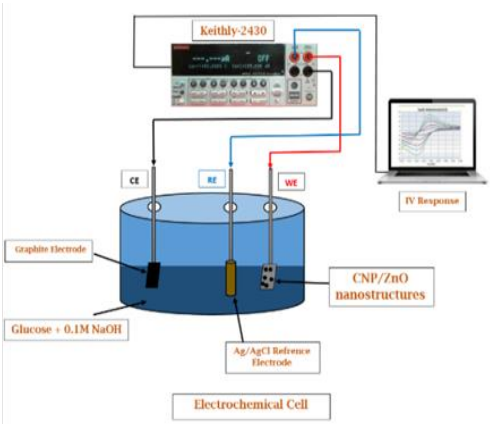


Figure.4 Diagram illustrating the glucose biosensor electrode measurement system

3. Results and discussion

The findings are presented and discussed in this section.

3.1. X-ray Diffraction (XRD) Analyses

The ZnOs and CNP/ZnO structural characteristics were studied, and XRD patterns were used for phase identification. Comparison between patterns were made in the experiment. Utilizing the Joint Committee on Powder Diffraction Standard database (JCPDS), ZnO's diffraction peaks are precisely indexed and have a hexagon shape. ZnO (36–1451) revealed the presence of seven peaks in ZnO nanostructures at (34.75°, 36.84°, 47.88°, 57.00°, 63.09°, 68.44°, 77.88°) matching the hkl Miller indices of crystallographic ZnO planes of (002), (101), (102), (110), (103), (112), (202), respectively. Scherer equation [27] was employed to evaluate the crystallite size (D) of the prepared films

$$D=\frac{0.9\,\lambda}{\beta\cos\theta} \qquad (1)$$

Carbon nanoparticles. It can be observed from the above XRD pattern that the average of crystallite size of ZnO nanostructures was 9.53 nm.

Table 1: XRD peak position (**2*θ***), diffraction planyes (h k l), lattice parameters (d), FWHM, the crystallite size (D), dislocation density (***δ***), and the strain (***ε***) of ZnO NPs

Mat	2θ Exp	2θ JCPDS	θ radians	d (A°)	hkl	FWHM radians	ε (10^{-3})	δcm^{-2} (10^{10})	D nm
ZnO	34.75	34.42	0.303	2.603	002	0.0114	2.82	66.4	12.27
	36.84	36.25	0.321	2.428	101	0.0104	2.59	55.1	13.46
	47.88	47.53	0.417	1.911	102	0.0175	4.37	157	7.96
	56.85	56.60	0.497	1.624	110	0.0187	4.67	180.1	7.45
	62.89	62.86	0.550	1.477	103	0.0185	4.62	176.3	7.53
	67.99	67.96	0.596	1.378	112	0.0286	6.76	423.3	4.86
	77.76	76.95	0.679	1.238	202	0.0106	2.59	57.3	13.20

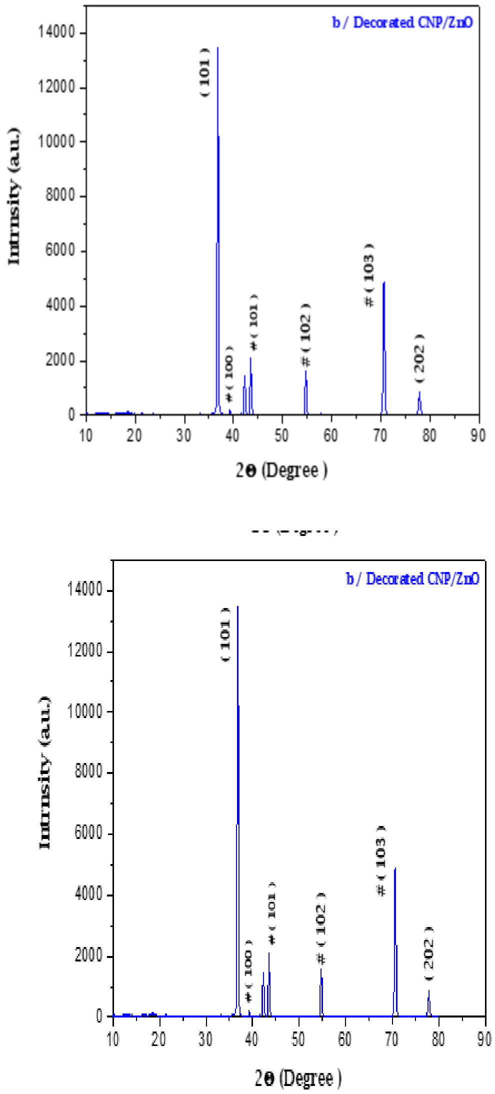


Figure.5 XRD patterns of the a- Pure ZnO b- Decorated CNP/ZnO

3.2. The EDX analysis of ZnO Nanoparticles

Figure (6) displays the results of the analysis of the energy-dispersing X-ray spectrum indicating the presence of zinc and oxygen as well as the weight percentages of Zn and O and C, which are 57.15 % and 19.13 % and 23.72 %, respectively, and the proportions of the weight of Zn and O were 78.01 % and 21.99 %, respectively. This can verify the carbon nanoparticles' adsorption onto zinc oxide nanoparticles' surface.

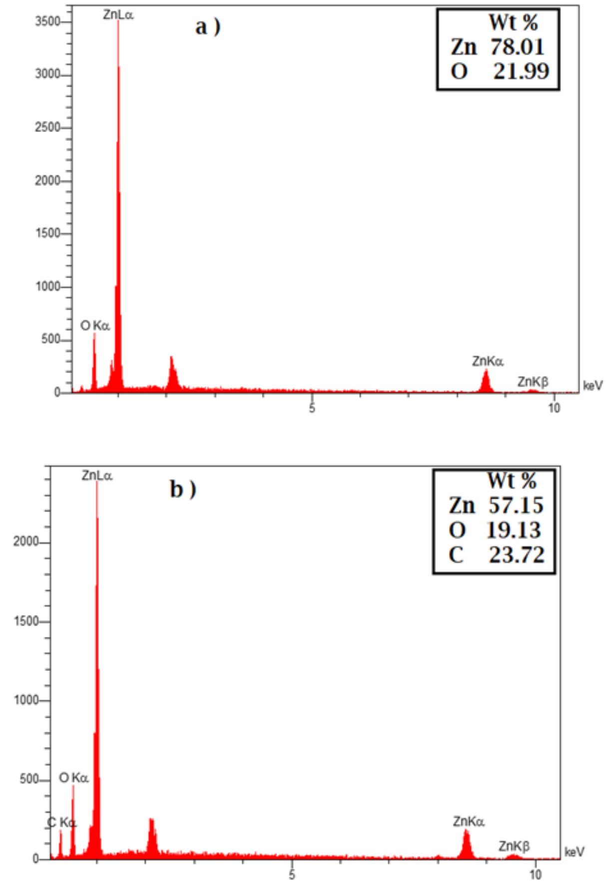
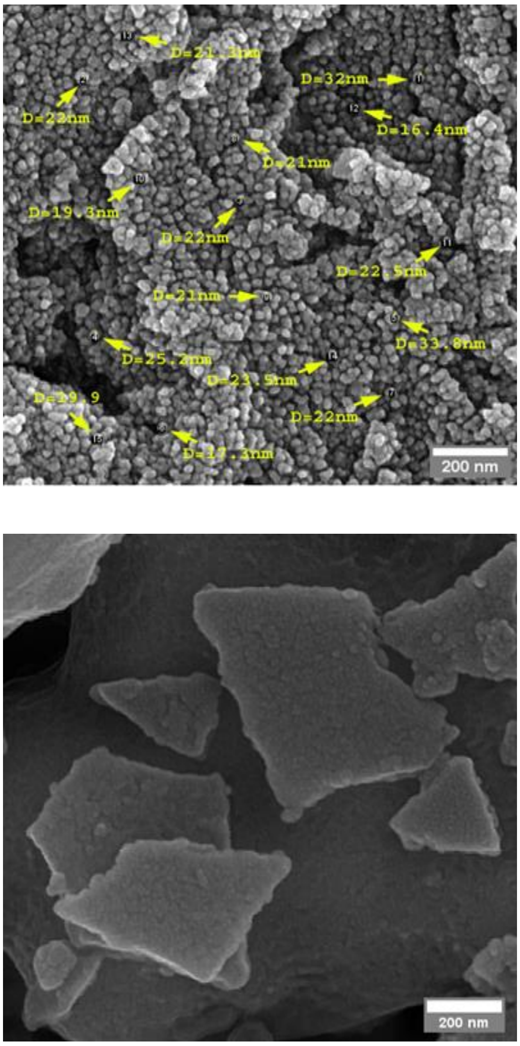


Figure.6 The EDX analysis a- Pure ZnO b - Decorated CNP/ZnO

3.3. Morphological Analysis

Figure 7 displays FE-SEM pictures. At a 200 nm magnification, it is observed that the diameters of the zinc oxide nanoparticles ranged from 15 to 40 nm. Figure (b) illustrates the transformation of the zinc oxide layer with images magnified by 200 nm following the addition of carbon nanoparticles. The images in Figure (b), which are magnified to 200 nm, show how the zinc oxide layer changed after being decorated with carbon nanoparticles. It is important to note that the carbon nanoparticle sheet's pH value of (3) indicates that it is acidic because it has reduced to a single, featureless layer above the zinc oxide layer. It was found that the majority of zinc foil's surface is covered in carbon nanoparticles.



A carbon nanoparticles sheet decoration a layer of zinc oxide nanoparticles

Figure.7 The morphology images of a- Pure ZnO nanoparticles b- Decorated CNP/ZnO

3.4. TEM (Transmission electron microscopy) Morphological Analysis

Figure (8) shows TEM images at magnification almost from (25 nm to 80 nm) it is noted after examination that Carbon Nanoparticles were formed.

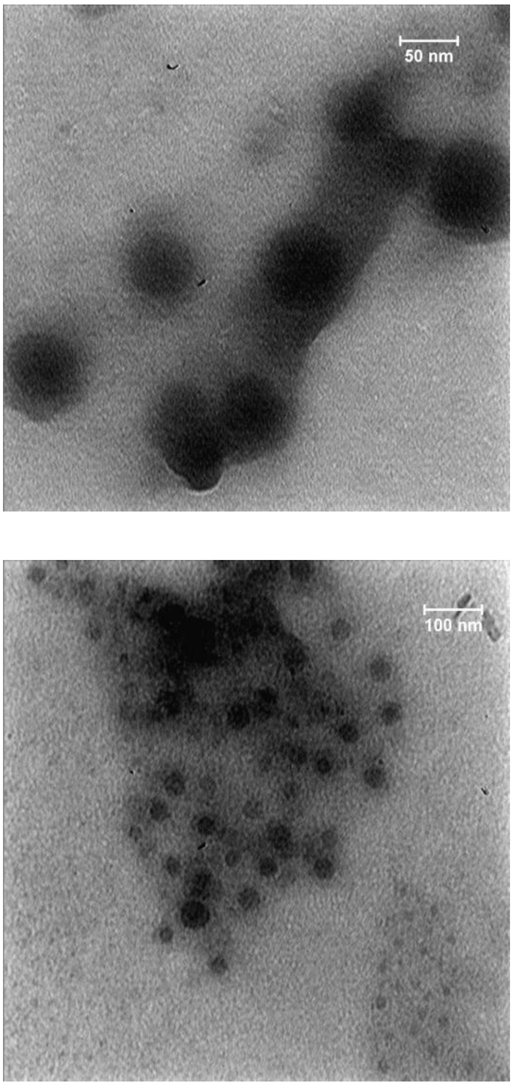


Figure.8 TEM images at magnification almost from (25 nm to 80 nm)

3.5. The Glucose biosensor's Electrochemical Characterization

By looking at and analyzing the glucose biosensor's electrical response, the main features and functionalities of the suggested biosensor were assessed to see how well it could perform high-accuracy measurements. Also, by analyzing their IV characteristics, electrode sensors' electrical responsiveness can be increased, making it possible to estimate their sensitivity. IV characteristic curves are typically used to electrically and mathematically characterize the behavior of various biosensor components inside an electronic circuit; the range of glucose molarities was chosen because the sensor's sensitivity increases with concentration. The effective conductivity of the working electrode ZnO NPs and CNP/ZnO Nanostructures with different concentrations is shown in Figure (9), which illustrates low glucose molarity can be used to determine the sensor's sensitivity at low concentrations. In the different concentrations within the range of (0.3 – 0.5 – 0.7) mM dissolved in the NaOH aqueous solution, biosensors' current responsiveness can be recorded via the applied voltage (0 – 1) V with a scan rate of 100 mV/s. All measurements were taken at room temperature. The manufactured glucose sensor was found to exhibit a significant increase in current when exposed to a voltage range between 0 and 1 volts. Additionally, an increase in current was observed with an increase in glucose concentration. There are no peaks and just a slight background current at zero glucose concentration. On the other hand, the maximum glucose biosensor response, the highest current, and the 1 mM glucose concentration were all reached by increasing the concentration. By using the slope of the linear line drawn for the measured response current, the sensitivities of the bare ZnO NPs and CNP-decorated ZnO NPs sensors were assessed. It is clear that the sensor reacts well to lower glucose concentrations. Reusability, reproducibility, and stability are other important aspects to consider when evaluating the efficacy of sensing apparatus.

Carbon nanoparticles directly decorated upon the surface of the electrode gave the sensor strong mechanical stability and contributed to the exceptional stability of the electrode as well, and an increase in sensitivity was observed.

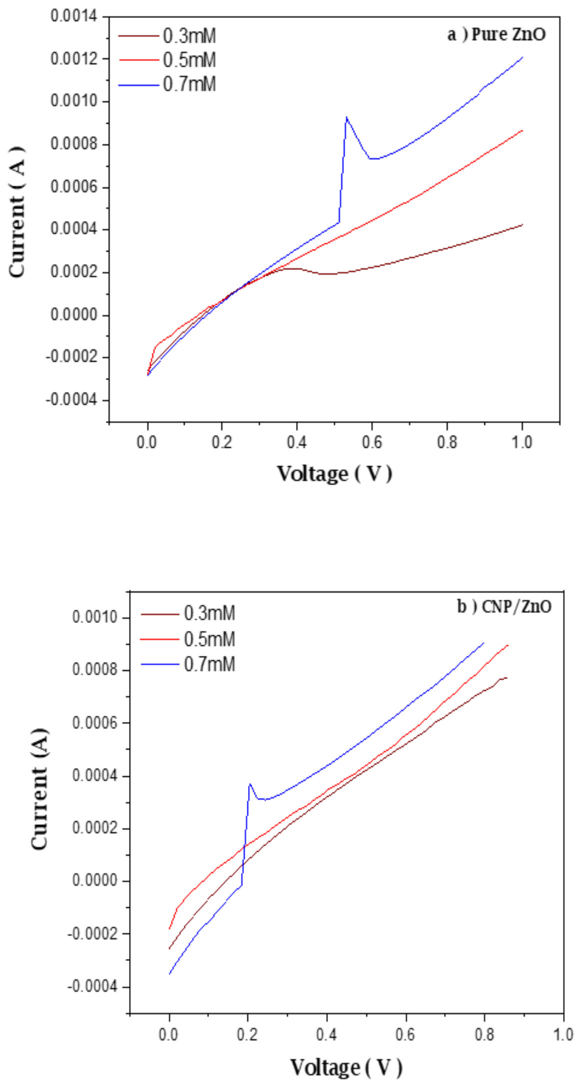


Figure.9 (a – b) IV Characterization of ZnO and CNP/ZnO with different glucose

The quantity of electrons released into the electrolyte during the electrochemical reaction determines the sensor's sensitivity. The curve's gradient and intercept in Figure (10) demonstrate a sensitivity of (1776 μA. mM^{-1}. cm^{-2}) when glucose levels rise linearly, and the sensitivity is (1975 μA. mM^{-1}. cm^{-2}) when employing CNP/ZnO nanostructures as an electrode as opposed to a ZnO nanoparticles sheet. Low limits of detection of 0.3 and 0.8 mM were demonstrated, respectively. The enhanced ZnO/CNP electrode accelerates the glucose electrochemical reaction, which is the cause of the higher sensitivity value of ZnO/CNP due to the synergistic effect of ZnO and CNP.

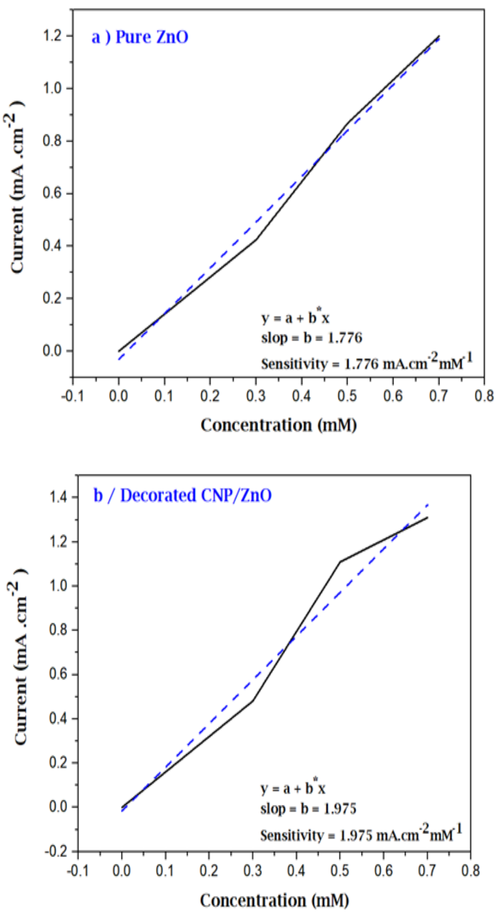


Figure.10 A calibration curve with electrode response a- Pure ZnO b- CNP/ZnO

The comparisons between the current study's sensitivity, detection limit, and linear working range and those of previous ZnO and related support nanostructure-based glucose biosensors are summarized in Table 2.

4. Conclusions

For non-enzymatic glucose sensing, the anodization process proved to be successful in producing pure ZnO and Carbon Decorated ZnO (CNP/ZnO). Using the ZnO material as a glucose biosensor, it showed good electrocatalytic performance and sensitivity (1776 μA. mM^{-1}. cm^{-2}). In contrast, a higher sensitivity value of (1975 μA. $mM^{-1}cm^{-2}$) was revealed when using the as-prepared CNP/ZnO electrode. High stability, repeatability, and selectivity with a linear range of 0.3 mM to 0.7 mM and a limit of detection of 0.3 mM, ZnO nanoparticle surface decorated with carbon nanoparticles holds potential for the development of highly sensitive, inexpensive electrochemical glucose biosensors. This result shows that the sensor has a promising application due to its high-performance non-enzymatic glucose and its creative, low-cost architecture

Table 2: compares different ZnO electrode-based free-enzymatic electrochemical sensors

Electrode	Sensitivity	Linear Range	Lower Limit	Reference
ZnO/MWCNT/GCE	64.29	1 – 10 mM	0.82 mM	[30]

GCE/ZnO@NDCS/GO_x	231.7	0.2 – 12 mM	6.3 μM	[31]
GOD-ZnO/CRG/Pt	17.64	0.2 – 1.6 mM	Not Mentioned	[32]
GR-CNT-ZnO-GO_x	5.36	0.1 – 6.5 mM	4.5 μM	[33]
CNP\ZnO	1975	0.3 – 0.7 mM	0.3 mM	This Work

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