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Physics

SYNTHESIS AND CHARACTERIZATION OF TIO$_2$ FOR BIOSENSOR APPLICATION

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ABSTRACT This paper presents the synthesis and characterization of Titanium dioxide (TiO2) thin film on Indium tin oxide (ITO) coated glass substrates via electrochemical deposition method. The TiO2 thin film was used for immobilizing glucose oxidase (GOx). Which can provide excellent biocompatibility, good electrical conductivity, low electro-chemical interferences and high signal-to-noise ratio for the development of electrochemical biosensors. The morphology and structure of TiO2 were characterized by X-ray diffraction (XRD). Glucose oxidase (GOX) was successfully immobilized on the surface of indium-tin oxide (ITO) electrode modified by TiO2. For the construction of an electrochemical biosensor. The direct electrochemistry and electro-catalytic performance of the biosensors based on TiO2 ware studied by cyclic voltammetry.

KEYWORDS : Biosensor, cyclic voltammetry, Glucose oxidase, TiO_2.

1. INTRODUCTION

There are growing demand for biomedical applications development through biosensors fabrication [1, 2]. The essential materials, which can be used for fabrication of biosensor, must have good sensitivity, response time and low production cost [3]. Metal oxides can be used as sensing materials for the development of sensors in medical field. They provide fast charge transfer between analyte and sensing material, which gives significant increase in the sensing signal. Metal oxide thin films play an important role to develop electrochemical sensor device due to their better electrocatalytic and electron transfer properties. TiO_2 nan films have high surface to volume ratio, better chemical stability, low isoelectric point and biocompatibility. Due to these properties, it has many applications in tissue engineering and drug delivery, membranes, filters, biosensors, and solar cells. Therefore, TiO_2 nano films as biosensing materials can be used to detect few analytes like glucose and urea [2- 5]. In present research, we have fabricated TiO2 thin film for rapid detection of sensing biomolecules through covalent grafting. TiO2 nan films have better electrochemical, photocatalytic and charge transfer properties with large surface area due to its mesoporous structure, and crystallization [6]. The metastable anatase TiO2 is much stable as compared to various other polymorphs like rutile, brookite and anatase due to its crystal shape with lower average surface energy. The anatase TiO2 nan film is chemically biocompatible material for immobilization of the biomolecules, which may allow increased adsorption of enzyme molecules.

2. Apparatus

The electrochemical measurements were performed by using a CHI 660C (CH Instruments) connected to a personal computer. Which have A three-electrode configuration , consisting of a TiO_2 coated Indium tin oxide (ITO) electrode as a working electrode, whereas Ag/AgCl serving as the reference electrode and platinum wire served and counter electrode. Batch electrochemical experiments were carried out in a 10 mL voltammetric cell at room temperature.

2.1. Immobilization of GOx

For immobilizing of GOx in TiO_2 in the TiO2 / ITO film, 0.25 cm2 was selected as the working electrode. The GOx solution have been prepared by dissolving of 10 mg of GOx in 1 mL phosphate buffer solution (0.1 M and PH 7-8). The TiO_2 / ITO film have been immersed in the GOx solution for 12 hours at room temperature. After that the GOx /TiO_2 / electrode rinsed with PBS (0.1 M and PH 7-8) to remove the free enzyme and dried at room temperature for a few hours. Then the electrode have been stored at 4 ^{0}C when it is not in use.

2.2. Preparation of the glucose solution

The glucose solution have been prepared in 0.1 phosphate buffer saline (PBS) and PH 7-8 and kept in room temperature for 24 hours before use. This is to assert the presence of β-d glucose.

3. Characterization techniques

3.1. Surface characterization and electrochemical measurements

All the electrochemical measurements were carried out with a CHI (600) workstation. A three-electrode configuration was employed for these experiments. A platinum wire was connected as the counter electrode, Ag/AgCl electrode was used as the reference electrode, and TiO_2 deposited in ITO used as working electrodes. For cyclic voltammetry, the working electrode was cycled in a 0.1 M PB solution by applying potential range between 1.0 and -1.0 V vs. Ag/AgCl. And scan rate 50mV/S (Aerometric measurements were carried out in a stirred cell by applying a potential of -0.4 V to the working electrode. A magnetic stirrer was used for this experiment. All measurements were performed at room temperature.

3.1. X-ray diffraction

The particle size of nanomaterial is related to the diffraction peak broadening, so X-ray diffraction spectra of synthesized TiO2 nanoparticles were taken and particle size and phase Composition were determined. Fig. 1 shows XRD pattern for TiO2 nanoparticles. The lattice parameter observed a= b =3.780, c =9.513. Sharp peaks obtained confirmed the Nano crystalline anatase structure corresponding to the planes (101), (004), (200) and (105) indicates the tetragonal structure of TiO2 nanoparticles. The average particle size calculated by using Debye–Scherer Eq. (1) indicated high surface area.

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

Where K known as Scherer's constant ranging from 0.9 to 1.0, k is the wavelength of the X-ray radiation source, which is 1.54056, b is the width of the XRD peak at half height and h is Bragg angle. XRD plot of TiO2 nanoparticles

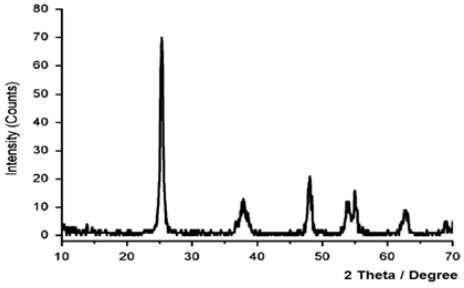

Fig 1 shows XRD pattern for TiO2 nanoparticles

Synthesized at 10 and 14 mA/cm2 showed intense peak at planes. As shown in the Table 1

Table 1 Structural Parameters of TiO_2 Thin Films

Planes	2 Theta in radians	β(Radian)	D (nm)	$D_{average}$(nm)
101	25.316	0.3626	24.23686799	27.55973
004	25.306	0.2463	35.67829053	
200	27.321	0.3725	24.00469662	
105	31.204	0.3529	26.31907614	

2.1. Electrochemical studies

The cyclic voltammetry (CV) was used to study the electrocatalytic activity of glucose biosensor for GOx /TiO_2 / ITO electrodes in 0.1 M (PBS) (PH9.0) over a potential range from -1 V to +1.0 V at scan rate of 50 mVs^{-1}. This method is a fast and convenient tool for characterizing glucose biosensor As depicted in Figure (2) did not show an obvious redox peak, at the TiO2 / ITO electrode (curve A), which indicated that TiO2 was not electrochemically active in the potential range. After GOx assembled into the TiO2/ ITO electrodes, a couple of stable and redox peaks were observed at GOx/TiO2(IO)/ITO electrode and the redox peak height enhanced in presence of 0.1 mM glucose (curve B), indicating the direct electron transfer between GOx and the electrode. Figure (3) is the compare of the two electrodes modified by different structured TiO2. As shown in Figure (3), the cyclic voltammograms of TiO2 modified electrodes exhibit the characteristic peaks with an oxidation peak at 0.4 Vand a reduction peak at 0.٦ V in the reverse scan, whereas no obvious peak appears on the curve of the plain TiO2 modified electrode. And according to Figure (3), the ordinary structured GOx/TiO2/ITO electrode has a poor respond to the glucose concentration change. This indicates that in pores of TiO2, the direct electron transfer between GOx and ITO electrode is greatly enhanced.

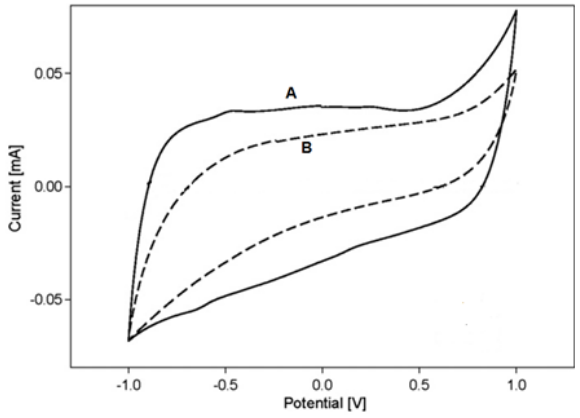

Fig.2 Cyclic voltammograms (CV) of GOx / TiO2 / ITO electrode measured in 0.1 M PBS buffer (Glucose concentration: 2mM, potential range: -1.0V to 1.0V, scan rate: 50mV/s, (a) GO: 0.1wt%, (b) TiO2: 0.1wt%)

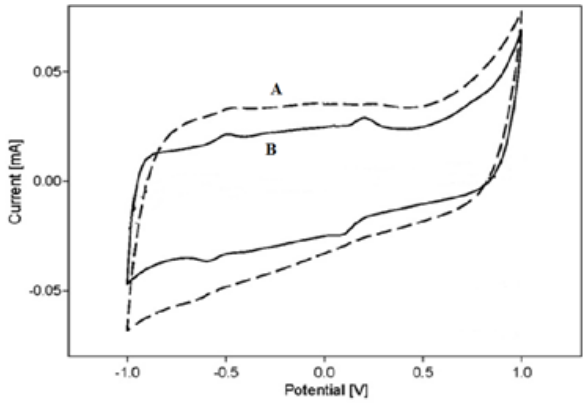

Fig.3. Cyclic voltammograms of the glucose biosensor based on TiO2 modified electrode Prepared at different weight ratios of GO / TiO2 / ITO (a) 0.5, (b) 0.1 (TiO2: 0.1wt%, Glucose conc.:2mM, potential range -1.0V to 1.0Vand scan rate: 50mVs^{-1}).

3.3. Stability of GOx / TiO2 / ITO Electrode

In this study, stability of the GOx/TiO2/ITO electrode was investigated. By measuring potentiometric response of electrode one time every week. Then electrode was kept in 1.0 mM phosphate buffer solution at 4 ^{0}C when not in use. There was slightly decrease of the potentiometric response, indicating that GOx / TiO2/ITO electrode was stable in buffer solution

4. CONCLUSION

In this study we have synthesized of Titanium dioxide (TiO2) thin film on Indium tin oxide (ITO) coated glass substrates via electrochemical deposition method. The TiO2 thin film was used for immobilizing glucose oxidase (Gox). and the electrocatalytic activity for the electro oxidation of glucose on a GOx/TiO2/ITO electrode have been investigated. The cyclic voltammetry and amperometric response of the biosensor give the considerable improvement of the. Such modified electrode gives a higher catalytic activity toward glucose oxidation and a higher sensitivity of glucose detection.

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