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ORIGINAL ARTICLE

Synthesis of carbon nanosheet from barley and its use as non-enzymatic glucose biosensor

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Abstract In this work, carbon nanosheet (CNS) based electrode was designed for electrochemical biosensing of glucose. CNS has been obtained by the pyrolysis of barley at 600–750 °C in a muffle furnace; it was then purified and functionalized. The CNS has been characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD) and Raman spectroscopic techniques. The electrochemical activity of CNS-based electrode was investigated by linear sweep voltammetry (LSV) and square wave voltammetry (SWV), for the oxidation of glucose in 0.001 M H_2SO_4 (pH 6.0). The linear range of the sensor was found to be 10^{-4}–10^{-6} M (1–100 μM) within the response time of 4 s. Interestingly, its sensitivity reached as high as ~26.002±0.01 μA/μM cm^2. Electrochemical experiments revealed that the proposed electrode offered an excellent electrochemical activity towards the oxidation of glucose and could be applied for the construction of non-enzymatic glucose biosensors.

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1. Introduction

The continuous development of new nanomaterials for their applications in electroanalytical techniques has been associated with the necessity of the improvement of the quality of efficient electrochemical devices and bio-devices. Nowadays, designing of more sensitive and selective electrochemical devices to recognize the small quantity of analytes has received more and more attention [1]. Carbon-based nanomaterials, especially carbon nanotubes and graphene, are extremely attractive in the bio-analytical area for electrode design as they can combine properties of the high surface area, acceptable biocompatibility, chemical and electrochemical stability and good electrical conductivity [2,3]. Furthermore, many works have shown that the application of different kinds of carbon nanomaterials is receiving more interest in electroanalytical chemistry, because of their effective surface area.

Diabetes has remained a major cause of death and serious vascular and neuropathy diseases. The diagnosis and management of diabetic patients require precise monitoring and control of the glucose level in the body. Therefore, frequent testing of the physiological glucose level is critical in confirming treatment efficiency, preventing long-term complications and avoiding a diabetic emergency, such as hypoglycemic (low blood sugar, <3 mM). This urgency has led to fascinating research and innovative detection strategies in glucose biosensor field. Electrochemical glucose biosensors had their beginning in 1962

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reported by Clark and Lyons, who described the measurement of glucose in blood plasma using the enzyme glucose oxidase coupled with a potentiometric electrochemical biosensor. Electrochemical methods based on glucose oxidase (GOx) have played a vital role in simple, easy-to-use blood sugar testing and they have been widely studied over the last few decades for continuous glucose monitoring [4–12].

Enzyme-based sensors show good selectivity and high sensitivity, but their activity is affected by temperature, pH, and toxic chemicals [13,14]. In order to overcome these drawbacks of enzymatic glucose sensors, non-enzymatic glucose sensors have been designed and fabricated, which have several attractive advantages, such as good stability, selectivity and sensitivity and lower detection limit [15–28].

In continuation of our earlier research work of synthesis of carbon nanoparticles from different natural sources [29–32], we have now used barley, which is rich in complex carbohydrates, mainly starch (76%). Interestingly, it gives another nanoform of carbon, i.e., carbon nano-sheet (CNS), after pyrolysis at 600–750 °C in a muffle furnace. A non-enzymatic glucose biosensor was fabricated from CNS and electrochemical studies have been carried out using linear sweep voltammetry (LSV) and square wave voltammetry (SWV). The results showed that the simple preparation procedure coupled with the low cost and high electrochemical activity unfolds a new pathway for economic, reliable and sensitive detection of glucose.

2. Experimental

2.1. Reagents

β-D glucose (99.5%) and H_2SO_4 were purchased from Sigma. All solutions were prepared with deionized water. Pyrolysis of barley was carried out in a muffle furnace (Tanco, PL Tandon & Company, India). The surface morphology of CNS was studied by a scanning electron microscope (FEI Quanta 200 Hv) and X-ray diffraction (XRD) patterns were recorded with JSO ISO DEBYEFLEX 2002 Model X-ray powder diffractometer. Raman spectra were recorded using a Raman spectrometer; WITEC MODEL with 514 nm excitation. Electrochemical studies were performed using a minipotentiostat (Dropsens μstat 100, Spain).

2.2. Synthesis and functionalization of CNS

CNS was synthesized by pyrolysing a cheap and readily available raw material, barley, at 600–750 °C for 2 h under insufficient flow of air in a muffle furnace. The black carbon soot obtained was collected in a thimble and placed into a soxhlet extractor for sequential purification with petroleum ether, acetone, ethyl alcohol and finally with water followed by functionalization with nitric acid. 250 mL of 2 M HNO_3 was stirred with the carbon powder for several minutes and then kept overnight. Excess nitrate was then removed by repeatedly dissolving the black particles in water and then the black particles were evaporated to dryness. The powdered CNS was then characterized by SEM, XRD and Raman spectroscopy.

2.3. Fabrication of CNS electrode

The CNS electrode was fabricated following our reported method [29], just like the commercially available standard electrode DS110 (DRP 110CNT). It was fabricated on an insulating Teflon material containing three silver wires. Both working electrode and counter electrode were made of CNS while reference electrode and electric contacts were made of silver (Fig. 1). Its dimensions is 3.5 cm × 1.0 cm × 0.5 cm (length × width × height) and it is ideal for working with 50 μL volume like the standard electrode.

CNS was first stirred with polystyrene solution, prepared in chloroform (9:1 ratio) followed by sonication. A drop of the slurry was then deposited as a very fine thin film on the Teflon substrate covering two silver wires, serving as working and counter electrodes. The third silver wire was used as a reference electrode.

2.4. Non-enzymatic detection of β-D glucose with fabricated CNS electrode

For non-enzymatic detection of β-D glucose, LSV and SWV studies were carried out at CNS electrode using different concentrations of glucose in 0.001 M H_2SO_4 and maintaining the pH at 6.0. Then, 50 μL of the glucose solution was taken by a micropipette and dropped on the surface of the electrode.

3. Results and discussions

3.1. Characterizations of CNS

SEM images shown in Fig. 2 clearly indicate the formation of carbon nanoparticles arranged in a sheet-like pattern, having width below 50 nm, and Fig. 3 shows the XRD patterns of CNS. Two predominant peaks were observed at around 25.69° and 41.65°, which were assigned for (002) and (111) reflections. Fig. 4 shows the Raman spectra of CNS with two prominent peaks at around 1310 cm^{-1} and 1620 cm^{-1}. The peak having smaller intensity is known as the D band and the peak having greater intensity is known as the G band.

3.2. Mechanism of non-enzymatic glucose detection at CNS electrode

It has been observed that the functionalization of CNS with 2 M HNO_3 leads to the generation of functional groups like hydroxyl (–OH) and carboxyl groups (–COOH) which were suitable for ongoing derivatization reactions [33]. These groups were responsible for the adsorption of glucose molecules on the surface of carbon nanoparticles. The probable reactions are described in Scheme 1 [34]. During the reaction, when β-D glucose reacts with H_2SO_4, it liberates two protons and two electrons, converting into gluconolactone and hydrogen peroxide. On further hydrolysis, the ring structure of gluconolactone breaks into an open structure of gluconic acid.

3.3. Effect of scan rate on the peak current and peak potential of β-D glucose at CNS electrode

LSV was employed to determine the effect of scan rate on the electrochemical detection of β-D glucose at the CNS electrode. Fig. 5 displays 2D and 3D plots of LSV, which show the overlapping of voltagrams of 10^{-5} M β-D glucose (pH ~ 6.0) at various scan rates. The current versus scan rate plot shown in the inset exhibits a linear relationship:

$$Y = -1.3677 + 0.08491X, \quad R^2 = 0.99579,$$

which indicates that the electrochemical kinetics reaction is adsorption-controlled as the adsorption-controlled process should result in a linear plot of current I versus scan rate (ν). The linearity

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