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Novel phosphomolybdic acid/single-walled carbon nanohorn-based modified electrode for non-enzyme glucose sensing

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ABSTRACT

A novel phosphomolybdic acid/single-walled carbon nanohorn based amperometric sensing platform was successfully constructed for determination of glucose. Single-walled carbon nanohorn acted as an active matrix and was used to immobilize phosphomolybdic acid. As-prepared phosphomolybdic acid/single-walled carbon nanohorn composite was characterized by SEM, EDX and XRD. Electrochemical behaviors of modified electrodes were characterized by cyclic voltammetry and chronoamperometry. Electrochemical investigations of phosphomolybdic acid/single-walled carbon nanohorn electrode showed three well-defined pairs of redox peaks and rapid electron transfer between electrode and electrolyte. Excellent electrocatalytic performance was obtained due to the synergistic effect of phosphomolybdic acid and single-walled carbon nanohorn. It was concluded that the linear amperometric response to glucose was achieved in the concentration range of 0.035 mM to 4.0 mM, and the detection limit was calculated as 2.4 μM. Moreover, as-prepared phosphomolybdic acid/single-walled carbon nanohorn electrode exhibited prominent selectivity, stability and reproducibility, making it a promising tool for sensitive non-enzymatic determination of glucose.

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1. Introduction

Diabetes mellitus, which is commonly known as diabetes, has become a universal epidemic. Tight regulation of blood glucose level is an efficient way to control diabetes morbidity and mortality [1]. Therefore, it is imperative to develop various methods for monitoring glucose, such as colorimetric methods [2], optical techniques [3] and electrochemical methods [4,5]. Among these methods, electrochemical method is a demonstrated effective approach for determination of glucose, not only due to the simple instrumentation and easy operation, but also the fast response, excellent selectivity, high sensitivity and low cost.

Currently, many glucose sensors on the market are mainly based on glucose oxidase-assisted electrochemical oxidation. However, due to inherent flaws of enzyme molecules themselves, such as instability, poor selectivity, high cost of enzymes and complicate immobilization procedures, the applications of enzymatic sensors are restricted. To avoid the described disadvantages, great efforts have been made to develop non-enzymatic electrochemical sensors [6].

As a kind of nanostructured carbon materials of high purity, single-walled carbon nanohorn (SWCNH) has horn-shaped sheaths composed of graphene sheets and a conical structure with a particularly sharp apical angle. SWCNH provides excellent electrical conductivity, high specific surface area and internal space, and these significant features have attracted great attention for potential applications, such as adsorption [7], aptamer techniques [8], drug delivery [9], fuel cells [10] and supercapacitors [11]. Zhu et al. built SWCNH-modified GCE and employed for the simultaneous determination of uric acid, dopamine and ascorbic acid [12]. Zhang et al. used a gold nanoparticles-carbon nanohorns hybrid for amperometric response of hypoxanthine and xanthine [13]. During electrocatalytic reactions, SWCNH acts as an excellent support on which to immobilize other particles and a conducting pathway to assist a fast electrochemical kinetics.

Polyoxometalates, well-known anionic molecular metal-oxygen clusters, are capable of delivering reversible multi-electron transfer reactions, making them interesting for many fields, such as fuel cells [14], batteries [15], supercapacitors [16] and electrochemical sensors [17,18]. Phosphomolybdic acid (denoted as PMo_{12}) has been used for construction of chemical modified electrodes, acting as a mediator in multiple and consecutive electron transfer reactions [19]. The attachment of polyoxometalates to electrode surfaces was achieved by adsorption, Langmuir-Blodgett technique, layer-by-layer self-assembly, entrapment as dopants into polymers and other strategies [20–23]. However, the disadvantages of poor conductivity and considerable leakage have seriously hampered the use of polyoxometalates as electrocatalyst [24]. It is essential to explore effective materials and

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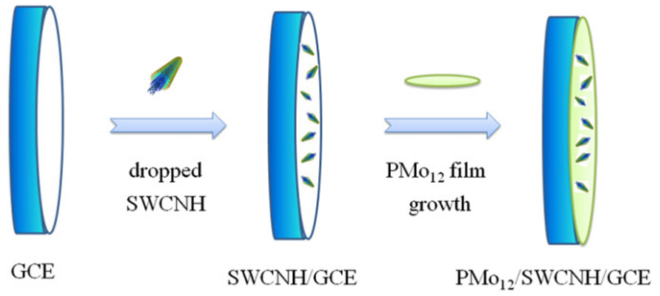

Fig. 1. Schematic illustration of fabrication process of PMo_{12}/SWCNH/GCE.

methods to immobilize polyoxometalates in order to obtain expected electrocatalyst.

In our work, we successfully fabricated a novel PMo_{12}/SWCNH based amperometric sensing platform for determination of glucose. SWCNH acted as an active matrix and exhibited excellent electrochemical performance, which not only stabilized the structure of PMo_{12} but also enhanced their catalytic activity. Due to the synergistic effect of PMo_{12} and SWCNH, PMo_{12}/SWCNH/GCE electrode exhibited excellent catalytic performance for glucose oxidation. Moreover, as-prepared electrode also presented prominent selectivity, stability and reproducibility, demonstrating the potential application in electrochemical sensing devices.

2. Materials and methods

2.1. Reagents and materials

SWCNH was purchased from Jiansin Scientific & Trading Co. (Beijing, China). Sodium phosphomolybdate ($Mo_{12}Na_3O_{40}P$), NaOH, Na_2SO_4, Na_2HPO_4, NaH_2PO_4, *N*,*N*-dimethylformamide (DMF), ethanol and H_2SO_4 were analytical grade and purchased from Chengdu Chemical Reagent Factory (Chengdu, China). Glucose, ascorbic acid (AA), dopamine (DA) and uric acid (UA) were obtained from Sigma-Aldrich (St. Louis, USA). Phosphate buffer solution (PBS) was prepared by mixed stock solutions of Na_2HPO_4 and NaH_2PO_4 (0.10 M). Doubly distilled water was used throughout the whole experiment.

2.2. Electrodes preparation and characterization

Prior to use, GCE ($\Phi = 3.0$ mm) was polished with 500 and 50 nm aluminum oxide powders and then washed successively with doubly distilled water and ethanol. A homogeneous mixture was formed by adding 1.0 mg SWCNH into 1.0 mL DMF, and sonicated for at least 30 min. The preparation procedures of PMo_{12}/SWCNH/GCE were as follows: 5.0 μL of mixture was dropped on the surface of GCE and allowed to dry in ambient air, then immobilized PMo_{12} on SWCNH/GCE was carried out by cyclic voltammetry between −0.20–0.60 V at scan rate of 50 mV s^{-1} for 25 cycles in the solution containing 50 mM $Mo_{12}Na_3O_{40}P$ and 0.50 M H_2SO_4. The as-prepared electrode was thoroughly washed with doubly distilled water and dried in air. The process was briefly illustrated in Fig. 1. For comparison, the preparation procedures of SWCNH/GCE and PMo_{12}/GCE were similar to that of PMo_{12}/SWCNH/GCE just with dropping and using bare GCE as matrix instead of SWCNH/GCE. Before the electrochemical measurements, electrolytes were purged with N_2 for 1 h to remove oxygen completely.

The morphologies of SWCNH and PMo_{12}/SWCNH composite were characterized by transmission electron microscope (TEM, Libra 200FE, Carl Zeiss) and scanning electron microscope (SEM, Ultra 55, Carl Zeiss), respectively. The elemental analysis of the composite was characterized by energy-dispersive X-ray spectroscopy (EDX, Ultra 55, Carl Zeiss). The structure of PMo_{12}/SWCNH was investigated by X-ray diffractometer (XRD, X' Pert PRO, Netherlands) with Cu K_α radiation

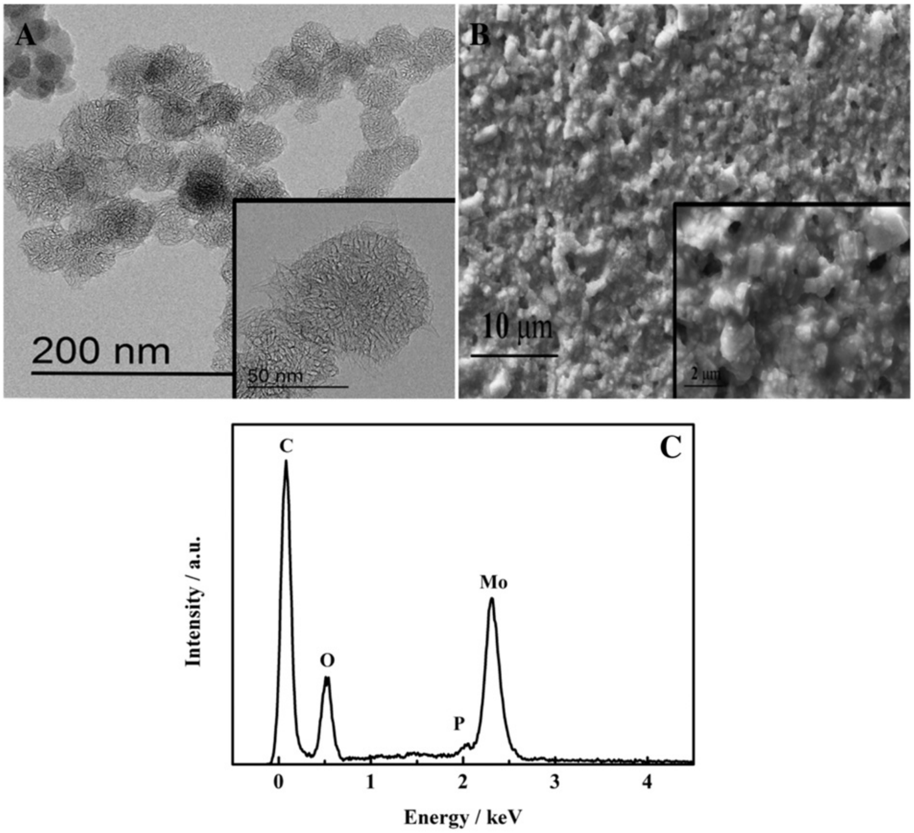

Fig. 2. TEM image of SWCNH (A), SEM image (B) and EDX spectrum (C) of PMo_{12}/SWCNH composite.

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