



Electrodeposition of palladium nanoparticles on porous graphitized carbon monolith modified carbon paste electrode for simultaneous enhanced determination of ascorbic acid and uric acid

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ABSTRACT

A novel, simple and highly sensitive electrochemical method is developed for the determination of ascorbic acid (AA) and uric acid (UA) based on a palladium nanoparticles deposited on porous graphitized carbon monolith modified carbon paste electrode (PdNPs/CM/CPE). The chemical modified electrode was characterized by scanning electron microscopy (SEM) and electrochemical impedance spectroscopy (EIS). The PdNPs/CM/CPE displayed excellent electrochemical catalytic activities toward AA and UA with bare CPE, PdNPs/CPE, CM/CPE and PdNPs/CM/CPE by cyclic voltammetry (CV). The oxidation potentials of AA and UA at the PdNPs/CM/CPE shifted negatively and the peak currents were much larger than other electrodes. The amperometric signal of PdNPs/CM/CPE showed a good linearity with correlation coefficient greater than 0.997. The low detection limits ($S/N=3$) for AA and UA were $0.53 \mu\text{mol L}^{-1}$ and $0.66 \mu\text{mol L}^{-1}$, respectively. The proposed method showed easy fabrication, high sensitivity and stability, and good reproducibility. In addition, the modified electrode was applied to determine the AA and UA in human serum sample.

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1. Introduction

Electroanalytical methods have been more attractive in recent years due to their sensitivity, accuracy, lower cost and simplicity [1]. Electrochemically individual and/or simultaneous determinations of the analytes on unmodified electrode are difficult since they have similar oxidation potentials and the electrode fouling results in poor reproducibility [2]. Therefore, the simultaneous determination of small molecules is a major goal in this research field. Various materials including polymer [3–6], carbon-based materials [7–10] and nanocomposite [11–17] have been employed for the modification of the electrode surfaces to improve their analytical

properties. Among them, a new class of carbon materials, porous graphitized carbon monolith (CM), has very interesting physico-chemical properties [18] and high surface area that makes it a potential material for biomolecule immobilization and biosensing applications.

Nowadays, there is an enormous interest in the use of metal nanoparticles to modify the surface of electrode [19–22]. Because of their excellent conductivity and catalytic properties, they could be used as electronic wires to enhance the electron transfer between target analytes and electrode surfaces, and as catalysts to increase electrochemical reactions. Palladium nanoparticles have been used for the modification of electrodes to increase sensitivity and selectivity of target analytes. For example, Gao et al. [23] used palladium electroplated on a carbon nanotube to catalyze the oxidation of formaldehyde and they found that this electrode has a high electrocatalytic activity to formaldehyde oxidation. Wang et al. [24] studied the electrocatalytic oxidation of ascorbic acid (AA), dopamine (DA) and uric acid (UA) using a palladium

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nanoparticles/graphene/chitosan modified electrode. Raof et al. reported the palladium nanoparticles doped mesoporous silica SBA-15 modified in carbon paste electrode for determination of oxalic acid [25]. The basic properties of the catalyst are strongly affected by the microstructure and the surface reactivity [26,27]. Therefore, the morphology and nanostructure of porous graphitized carbon monolith are considered to be the main factors in obtaining high dispersion of palladium nanoparticle catalysis.

AA and UA play an important role in the human body, and often coexist in biological samples. AA is an antioxidant that plays an important role in proper functioning of human metabolism and central nervous system [28]. It is commonly used as an antioxidant agent and easily found in vegetables, fruits and drinks [29,30]. UA is the primary end product of purine degradation metabolism in human being. Its concentration level in body fluids such as human serum and urine is maker of many clinical conditions, including hyperuricaemia, gout and Lesch-Nyan disease [31–33]. Therefore, the development of a sensitive and selective method for their simultaneous determination is needed for analytical applications and researches.

In this work, we reported a palladium nanoparticles deposited on porous graphitized carbon monolith modified carbon paste electrode (PdNPs/CM/CPE) for simultaneous determination of AA and UA. The scanning electron microscopy (SEM) and electrochemical impedance spectroscopy (EIS) were applied to study the modified surfaces. The electrochemical properties of modified electrodes were investigated by voltammetry and amperometry techniques. It was found that the PdNPs/CM/CPE electrode showed two well resolved voltammetric peaks and high potential for simultaneous determination of AA and UA in a mixed solution. Moreover, the modified electrode showed an excellent level of sensitivity and performance in stability and reproducibility. The proposed method was applied to human serum for determination of AA and UA.

2. Experimental

2.1. Chemicals and reagent

All the chemicals used were of analytical grades and aqueous solutions were prepared using deionized water (Millipore, Ireland). Ascorbic acid (AA) and uric acid (UA) were obtained from Sigma. Sodium hydroxide (NaOH), sodium phosphate monobasic dehydrate ($\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$), sodium phosphate dibasic (Na_2HPO_4) and phosphoric acid (H_3PO_4) were obtained from Sigma–Aldrich (Germany). Potassium hexacyanoferrate (III) ($\text{K}_3\text{Fe}(\text{CN})_6$) was purchased from Sigma–Aldrich (Japan). Potassium tetrachloropalladate (II) (K_2PdCl_4) and potassium chloride (KCl) were obtained from Sigma–Aldrich (USA). N,N-dimethylformamide (DMF) was purchased from Sigma–Aldrich (United Kingdom).

Stock solution of AA (50 mmol L^{-1}) was prepared daily by dissolving a suitable amount of the reagent in water. UA (50 mmol L^{-1}) was prepared by dissolving it in a small volume of 0.1 mol L^{-1} NaOH solution and diluted with water.

Phosphate-buffered solutions of different pH were prepared by mixing solutions of 0.1 mol L^{-1} $\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$ and 0.1 mol L^{-1} Na_2HPO_4 at different ratios. The solution pH levels were adjusted by adding 1.0 mol L^{-1} H_3PO_4 solution.

2.2. Apparatus

The electrochemical measurements, cyclic voltammetry (CV) and amperometry, were performed using a CHI 1040A electrochemical workstation (CH Instruments, Austin, TX) at room temperature. A three-electrode system consisted of carbon paste electrode (CPE) modified with CM and deposited PdNPs (PdNPs/

CM/CPE) that was used as working electrodes, a Ag/AgCl (3 M NaCl) as reference electrode (BAS, West Layette, IN), and a platinum wire as counter electrode (Sigma–Aldrich, Dublin, Ireland). The convection transport during the amperometric determination was performed with magnetic stirring.

2.3. Preparation of the PdNPs/CM/CPE

5 mg CM was dispersed in 5 mL DMF, then ultrasonicated for 16 h until a homogenous suspension of CM was obtained. CM dispersion ($5 \mu\text{L}$) was carefully dropped on the top of carbon paste electrode (CPE), allowing the solvent to evaporate at room temperature. The CM/CPE was then obtained.

To prepare PdNPs/CM/CPE, the CM/CPE electrode was immersed in 0.1 mol L^{-1} H_2SO_4 solution containing 1.0 mmol L^{-1} K_2PdCl_4 . The electrochemical deposition of the PdNPs was conducted for 50 s at -1.0 V (versus Ag/AgCl). Finally, the modified electrodes were cleaned by applying a potential scan of -1.0 V to 1.0 V with a scan rate of 0.5 V/s in phosphate buffer solution (pH 4.0) until a steady voltammogram was obtained.

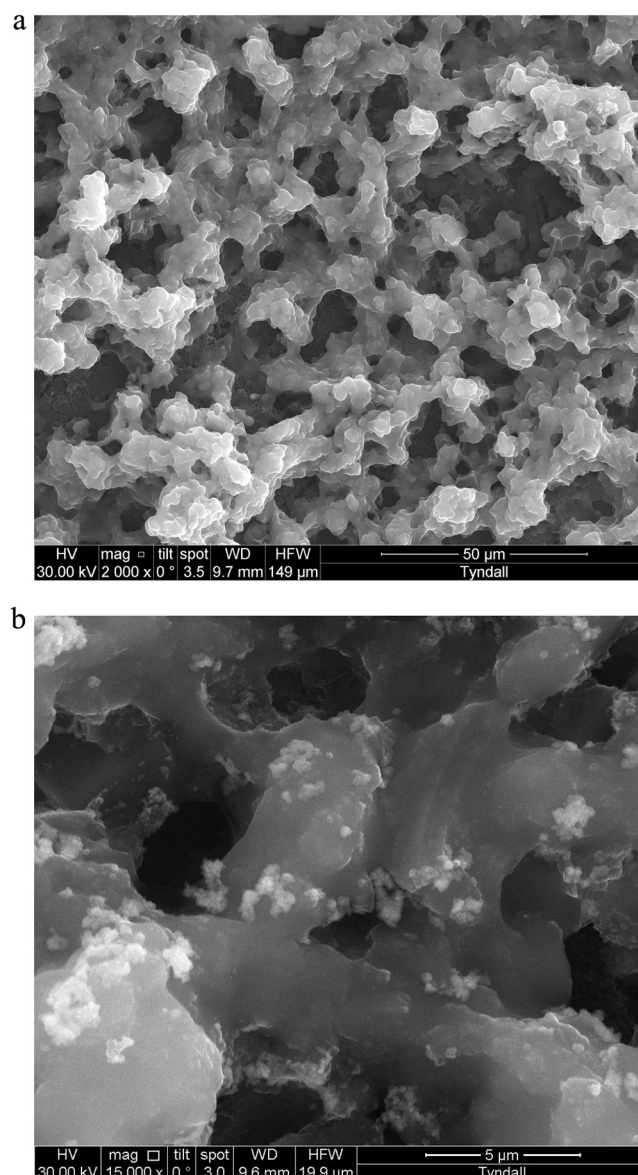


Fig. 1. (A) The SEM image of CM and (B) PdNPs deposited on CM.

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