



A self-assembled monolayer on gold nanoparticles modified electrode for simultaneous determination of isoproterenol and uric acid



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ABSTRACT

A novel layer-by-layer assembly of gold nanoparticles (AuNPs) and 2-(2,3-dihydroxy phenyl) benzothiazole (DPB) was successfully applied at a glassy carbon electrode (GCE/AuNPs/DPB). At first, AuNPs were deposited on the GCE by chemical and electrochemical methods and then DPB is self-assembled on the Au nanoparticles. This new modified electrode system was characterized by cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS). GCE/AuNPs/DPB was used for simultaneous determination of isoproterenol (IP) and uric acid (UA). It was observed that an electrode with electrochemical deposited of Au nanoparticles has higher electrocatalytic activity for the oxidation of IP in comparison of the electrode with chemical deposited of Au nanoparticles. Also results show the DPB has a key role in electrocatalysis of IP and in simultaneous determination of procedure because the DPB can catalysis the oxidation of IP via EC' mechanism. Differential pulse voltammetry (DPV) exhibited a linear dynamic range over the concentration range of 0.1–900.0 μM and a detection limit (3 s) of 82 nM for IP in the optimum conditions. Finally, DPV was used for simultaneous determination of IP and UA and their detection in real samples.

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1. Introduction

Electroanalytical methods have been proven to be rapid, simple and sensitive and have an impact in the most of fields of science, for example clinical diagnostics, food quality control, security and environmental analysis [1]. A general method of preparation of electroanalytical devices consists the bulk or surface modification of the electrode with species enabling chemical or biological recognition. Therefore, the choice of a suitable electrode is important in electroanalytical research processes. Biocompatibility and capability to incorporate chemical species without their loss in operating medium are of greatest importance. The self-assembly

procedure is a precise modification of the surface structure on a nanometer-scale, which has been employed recently in the fabrication of sensors and biosensors. In particular the self-assembly of organo-sulfur compounds on gold surface have been extensively studied [2–6].

AuNPs have potential applications in the construction of electrochemical sensors and biosensors [7] because of their small dimensional size [8], good stability, biocompatibility [9,10], good conductivity and excellent catalytic activity [8,11]. On the other hand, gold nanoparticle modified electrodes can react with organo-sulfur compounds forming S–Au covalent bonds resulting in monolayer molecular assemblies. These types of modifications can greatly increase the immobilized amount of S-functionalized compounds and enhance the S–Au bond and stability of self-assembled monolayers (SAMs) [12].

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Different methods have been developed to construct AuNPs modified electrodes. These include electroless deposition [13], sol–gel process [14], hydrothermal precipitation [15], assembly by bifunctional chemical linkers [16] and electrodeposition produces [4]. Electrodeposition techniques are simple, fast and inexpensive techniques for the preparation of nanoparticles. In this technique some parameters such as mass, thickness, particle size, crystallographic orientation and morphology of the nanomaterials can be controlled by adjusting the operating conditions.

Isoproterenol (IP) or 4-[1-hydroxy-2-[(1-methylethyl)-amino] ethyl]-1, 2-benzenediol is a drug which used for the treatment of primary pulmonary hypertension and allergic emergencies, bronchitis, cardiac chock and heart attack. But, the excess of the drug may cause heart failure and arrhythmias [17]. The unfavorable effects of IP are related to the drug's cardiovascular effects. IP can produce a tachycardia, which predisposes patients to cardiac dysrhythmias. The chemically modified electrodes have been widely used as sensitive and favorable analytical methods for determination of IP [18–22]. In the electrochemical method, modified electrodes with nanomaterials because of their unique physical and chemical properties have some advantages such as long term stability, sensitivity, reduced overpotential and homogeneity in electroanalysis [23,24].

Uric acid (UA) is the end metabolic product of purine through the liver and is one of the important biomolecules present in physiological fluids. Abnormal UA level is a symptom of several clinical conditions such as kidney damage, gout, and Lesch–Nyan diseases [25]. Also, if UA do not eliminate from the body or there is an increase in production of UA, leads to a condition called hyperuricemia [26]. Several technologies have been reported for UA analysis such as chemiluminescence [27] and electrochemical methods [28,29]. In the other hand, UA strongly interferes with the determination of IP. Therefore a suitable selective, simple, inexpensive, fast, sensitive and accurate detection method is required for determining IP, especially in the presence of UA.

In the present study a novel electrode system was fabricated using a GCE modified by gold nanoparticles. AuNPs were prepared with two methods: the citrate reduction and electrodeposition method. GCE/AuNPs were characterized by scanning electron microscopy (SEM), CV and EIS methods. Then a newly synthesized organo-sulfur, 2-(3,4-dihydroxy phenyl) benzothiazole (abbreviated as DPB), was applied to AuNPs at this electrode forming a self-assembled monolayer modified electrode (GCE/AuNPs/DPB). This is a novel electrode system which used for the first time for determination of IP. This modified electrode was quite effective not only to detect IP, but also in simultaneous determination of IP and UA in mixture. Detection limit and linear range for IP in this work are comparable with other researches.

2. Experimental

2.1. Apparatus and chemical

The electrochemical measurements were performed using a potentiostat/galvanostat μ Autolab Type III (Eco

Chemie B. V.) and electrochemical impedance spectroscopy (EIS) measurements were done by an Autolab potentiostat/galvanostat (PGSTAT-302N, Eco Chemie, Netherlands) with GPES software. A three-electrode cell was used at 25 ± 1 °C. An Ag/AgCl (KCl, sat) electrode, a platinum wire and a modified glassy carbon electrode (Metrohm, 2 mm diameter) with Au nanoparticles and DPB self-assembled monolayer GCE/AuNPs/DPB were used as reference, auxiliary and working electrodes, respectively. The pH measurements were carried out with a Metrohm pH/mV meter model 691. All solutions were prepared with double-distilled water. IP, UA and other reagents were analytical grade (Merck). The buffer solutions were prepared from orthophosphoric acid and its salts in the pH range of 3.0–11.0.

2.2. General procedure for the synthesis of 2-(3,4-dihydroxy phenyl) benzothiazole

A mixture of 2-aminothiophenol (1.2 mmol), 3,4-dihydroxybenzaldehyde (1 mmol) and Al (HSO_4)₃ (0.02 g) was heated at 80 °C for 10 min. The progress of the reaction was monitored by thin layer chromatography (TLC). After completion of the reaction, the mixture was cooled to room temperature, dissolved in acetone followed by addition of water, the solid product was appeared. The product was re-crystallized in hot ethanol. FT-IR (ATR, neat), $\nu = 3478$ (OH), 1623 (C=N), 1589 and 1470 (C=C, aromatic), 1364, 1264 (C=S), 1200 (C–O), 1175, 1071, 1055, 765 (=C–H), 721(=C–H) cm^{-1} . ¹HNMR (400 MHz, Acetone-d₆): δ : 6.84 (t, $J = 8.0$ Hz, 1H), 7.00 (d, $J = 8.0$ Hz, 1H), 7.42 (d, $J = 8.0$ Hz, 1H), 7.48 (t, $J = 8.0$ Hz, 1H), 7.58 (t, $J = 8.0$ Hz, 1H), 8.08 (d, $J = 8.0$ Hz, 1H), 8.15 (d, $J = 8.0$ Hz, 1H), 9.27 (s, 1H), 11.78 (s, 1H).

2.3. Preparation of GCE modified by AuNPs

The GC electrode was first carefully polished with alumina on polishing cloth. The electrode was placed in ethanol container and used bath ultrasonic cleaner in order to remove adsorbed particles. For preparation of GCE modified with AuNPs (GCE/AuNPs) two following methods was used.

2.3.1. Chemical method

In the citrate reduction method the AuNPs were prepared as described by Lee and Meisel [30]. Briefly, 250 mL of 1.0 mM HAuCl₄ was boiled while stirring in a 500 mL round bottom flask. Then 25 mL of 38.8 mM sodium citrate was quickly added. Then the mixture was refluxed for 15 min with continuous stirring. The flask was allowed to cool to room temperature and was stored in the dark at 4 °C until use. Then 20 μ L of AuNPs solution was casted on the surface of GC electrode and dried in air to form an AuNPs film at the electrode surface.

2.3.2. Electrochemical method

In the electrochemical method the GCE/AuNPs was obtained by CV scanning from a solution containing 0.01 M Na₂SO₄, 0.01 M H₂SO₄, and 1.0 mM HAu(III)Cl₄·3H₂O at a scan rate of 60 mV/s⁻¹ for five scans [4]. NaNO₃ was

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