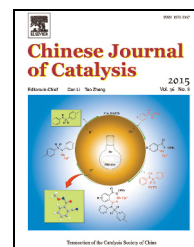


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## Article

# A nanocomposite electrocatalyst for the electro-oxidation of isoproterenol and its application as a sensor



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## ABSTRACT

A novel nanocomposite (GCE/AuNP-DAT) was fabricated via the self-assembly of a thiophenol derivative (3,4-dihydroxyphenyl-azo-2-thiophenol, DAT) on a gold nanoparticle (AuNP)-modified glassy carbon electrode (GCE). This novel modified electrode was investigated using cyclic voltammetry, and the electrode was applied for the electrocatalysis of isoproterenol (IP). The electrocatalytic activity of this nanocomposite was studied, and the reaction mechanism and catalytic rate constant were determined for IP. The GCE/AuNP-DAT did not show any electrocatalytic activity for the oxidation of uric acid, and the oxidation signal for IP was therefore not affected by the presence of uric acid at this electrode; any interference from uric acid in the detection of IP was eliminated by the modified electrode. This electrode was applied as a sensor, and differential pulse voltammetry data obtained using this sensor exhibited a linear dynamic range of 1–1500  $\mu\text{mol/L}$  and a detection limit of 0.46  $\mu\text{mol/L}$  for IP.

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## 1. Introduction

Chemically modified electrodes can be prepared using two general methods, namely bulk or surface modification of the electrode using materials with suitable electrocatalytic activity. Such electrodes are electro-catalysts that can improve a desired electrochemical reaction [1] and are suitable for application as electrochemical sensors because the electrode surfaces can be customized to achieve the required selectivity or sensitivity [2]. Self-assembly is a modification procedure that can be used to prepare self-assembled monolayers (SAMs), which can be used to modify the surface of electrodes [3].

Gold nanoparticles (AuNPs) are important nanomaterials for the fabrication of modified electrodes because of their good stability and biocompatibility [4,5], their good conductivity, and their excellent catalytic activity [6,7]. AuNP-modified elec-

trodes can react with organo-sulfur compounds to create SAMs via the formation of S-Au covalent bonds at the electrode surface. Several sulfur-containing compounds with different S atom positions have been reported for the preparation of SAM-modified electrodes; in some of these compounds the S atom is a simple aliphatic thiol [8], and in others the S atom is a thiophenol [9], or is in a heterocyclic ring [3].

Isoprenaline (IP) is an important neurotransmitter used for the treatment of neural disorders such as Parkinson's disease [10]. The cardiovascular effects of IP, which are typically compared with those of adrenaline and noradrenaline, show that IP can successfully relieve nervous tension from almost every kind of smooth musculature [11]. Such effects are more conspicuous in the musculature of the bronchus and gastrointestinal tract. IP is more effective when taken via inhalation, and it can be used effectively for bronchitis, cardiac shock, and heart

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attacks [10,11]. However, the excessive use of IP can lead to heart failure and arrhythmias. To date, various methods for determining IP concentration have been suggested and employed, including spectrofluorimetry [12], spectrophotometry [13], liquid and gas chromatographic methods based on fluorimetry [14], electrochemical detection (for HPLC) [15], and chemiluminescence [16]. Chemically modified electrodes have also been widely applied as sensitive analytical tools for the determination of IP [17,18].

Abnormal levels of uric acid (UA) are a symptom of several diseases, including gout, hyperpiesia, and Lesch-Nyhan disease [19]. The concentration of UA in biological fluids can therefore be monitored to provide an early warning of the presence of these diseases. Several techniques have been reported for the analysis of UA, including the detection of chemiluminescence [20], chromatography [21], and electrochemical methods [22–25]; UA is a significant interferent for the determination of other biological substances such as IP.

In this study, a novel nanocomposite electrode system was fabricated using a glassy carbon electrode (GCE) modified with AuNPs, on which a novel synthesized organo-sulfur compound (3,4-dihydroxyphenyl-azo-2-thiophenol, abbreviated as DAT) was self-assembled to produce the final GCE/AuNP-DAT electrode. We reported previously the construction of a similar electrode that used a different organo-sulfur compound as the head element of the electrode [26]. In that work, 2-(2,3-dihydroxyphenyl)benzothiazole (DPB) was used to form a SAM on an AuNP-modified GCE; DPB has an S atom in its heterocyclic ring, but the DAT used in the present study is a thiophenolic derivative. One of the aims of the present study was to build on our previous work to investigate the effect of S atoms on the formation of electro-active self-assembled layers on a gold modified-electrode. Here, we showed that this novel compound (DAT) could form electro-active SAMs on a GCE/AuNPs. This novel modified electrode was used for the first time as an electrocatalyst for the oxidation of IP, and as an electrochemical sensor for the detection of IP. The chemical hydroquinone structure of DAT in this electrocatalyst provided the ability to catalyze the oxidation of IP via an electrocatalytic mechanism that is explained in this report. The electrocatalyst was effective in eliminating any interference from UA in the determination of IP. The simultaneous determination of IP and UA was carried out at the electrode.

## 2. Experimental

### 2.1. Instruments and reagents

The electrochemical measurements were performed using a potentiostat/galvanostat (SAMA 500 Electroanalyzer System, I.R. Iran). A three-electrode cell was operated at  $25 \pm 1^\circ\text{C}$ ; the cell included a modified glassy carbon electrode (Metrohm) as the working electrode, a saturated calomel electrode (SCE) as the reference electrode, and a platinum wire electrode as the auxiliary electrode. All potentials are reported here with respect to the SCE. The pH measurements were performed using a Metrohm Model 691 pH/mV meter.

All solutions were prepared using double-distilled water. IP, UA, and the other reagents were of analytical grade (Merck). The buffer solutions were prepared from orthophosphoric acid and its salts. DAT was synthesized at our laboratory. The spectroscopic data for the synthesized compound were as follows. FT-IR (ATR)  $\bar{\nu}/\text{cm}^{-1}$ : 3430–3000, 1616, 1559, 1507, 1450, 1284, 1200, 1102, 816, 756;  $^1\text{H}$  NMR (500 MHz, DMSO- $d_6$ ) ( $\delta$ , ppm) ( $J$ , Hz): 7.9 (s, 1H), 7.64 (d,  $J = 6.6$  Hz, 1H), 7.58 (s, 1H), 7.5–7.45 (m, 2H), 7.24 (brs, 1H), 6.96 (m, 1H), 6.86 (m, 1H), 6.59 (m, 1H), 6.4 (m, 1H).

### 2.2. Preparation of AuNPs

The AuNPs were prepared using the citrate reduction method described by Lee and Meisel [27]. Briefly, 250 mL of HAuCl<sub>4</sub> solution (1 mmol/L) was boiled in a 500-mL round-bottomed flask under stirring, and 25 mL of sodium citrate solution (38.8 mmol/L) was added quickly to this solution. The mixture was then refluxed for 15 min under continuous stirring. The flask was allowed to cool to room temperature and was stored in the dark at  $4^\circ\text{C}$  until use.

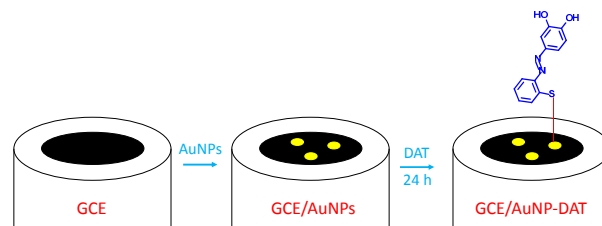
### 2.3. Preparation of the electrocatalyst

The procedure used to fabricate the proposed GCE/AuNP-DAT electrocatalyst is illustrated schematically in Scheme 1. The GC electrode was first carefully polished with alumina on a polishing cloth, and was then subjected to ultrasonication in deionized water, and subsequently ethanol, to remove any adsorbed particles. Twenty microliters of a dispersion of AuNPs (prepared as described above) was then cast on the surface of the GC electrode, and the resulting sample was dried in air to form an AuNP film on the electrode surface (the resulting sample is denoted here as GCE/AuNPs). The GCE/AuNP-DAT was prepared by immersing the GCE/AuNPs in DAT-ethanol solution (5 mmol/L) for 24 h at room temperature. Upon removal from this solution, the electrode was thoroughly rinsed with twice-distilled water (to remove the physically adsorbed species) and was then used for the electrochemical experiments.

## 3. Results and discussion

### 3.1. Electrochemical investigation of the GCE/AuNP-DAT

Before electrochemical studies were performed using the prepared electrocatalyst, its surface was characterized using



**Scheme 1.** Procedure used to fabricate the GCE/AuNP-DAT electrocatalyst.

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