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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; ^1H NMR (500 MHz, DMSO-d_6) (δ , 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_4 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°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.

scanning electron microscopy (SEM). Fig. 1 shows SEM images of the bare GCE and the GCE/AuNPs. Fig. 1(b) shows an SEM image of the GCE/AuNPs with many spherical AuNPs on the surface of the GCE, in contrast with the bare GCE in Fig. 1(a), which had a smooth and homogeneous surface. These results demonstrated that AuNPs were deposited on the electrode.

Cyclic voltammograms were measured for the GCE/AuNP-DAT in a phosphate buffer solution (0.1 mol/L) with pH = 7.0 (Fig. 2(a)), using a scan rate of 50 mV/s. The cyclic voltammogram showed an anodic peak at a potential of approximately 0.21 V in the forward potential scan, and a cathodic peak at a potential of approximately 0.07 V in the reverse scan. The ΔE ($E_{pa} - E_{pc}$) value was therefore 0.15 V vs. SCE, greater than the value expected for a reversible system, showing that this electrode process was quasi-reversible [28].

Cyclic voltammograms were measured for the GCE/AuNP-DAT at different scan rates, and the anodic and cathodic peak currents were then plotted as a function of the scan rate, as shown in Fig. 2(b). The anodic and cathodic peak current values were linearly dependent on the scan rate, with a linear relationship between the peak current and the potential scan rate, indicating a surface-confined, controlled redox process at the modified electrode. These results indicated that the DAT was attached to the electrode surface via covalent and thermodynamically favored S–Au bonds [29,30].

The electrochemical behavior of the AuNPs-DAT at the GCE was studied in solutions with different pH values. It was ob-

Fig. 1. SEM images of the surface of (a) the bare GCE and (b) the GCE/AuNPs.

served that the anodic and cathodic peak potentials of the GC/AuNP-DAT shifted to negative values with increases in the pH (Fig. 2(c)). A potential–pH diagram was constructed by plotting the calculated $E_{1/2}$ values for each cyclic voltammogram as a function of pH. As shown in the inset of Fig. 2(c), one straight line was obtained in the pH range of 4.0–11.0 with a slope of 0.053 mV/pH. This result showed that there was an equal transfer of electrons and protons in the redox reaction of DAT in this pH range [28].

3.2. Electrocatalysis of IP oxidation

The electrocatalysis of IP by the DAT molecules in the pro-

Fig. 2. (a) Cyclic voltammogram for the GCE/AuNP-DAT in phosphate buffer solutions (PBS, 0.1 mol/L, pH = 7.0) measured at scan rate of 20 mV/s; (b) Anodic and cathodic peak currents of the GCE/AuNP-DAT as a function of scan rate; (c) Cyclic voltammograms for the GCE/AuNP-DAT measured in PBS (0.1 mol/L) with different pH values (numbers 1 to 8 correspond to pH values of 4.0–11.0, and the inset is a plot of potential vs. pH).

Fig. 3. Cyclic voltammograms obtained in the presence of IP (0.05 mmol/L) at (1) the GCE/AuNP-DAT, (2) the bare GCE, and (4) the GCE/AuNPs; (3) Cyclic voltammogram measured for GCE/AuNP-DAT in the absence of IP. In all cases the supporting solution was PBS (0.1 mol/L, pH = 7.0), and the scan rate was 20 mV/s.

posed GCE/AuNP-DAT electrocatalyst was investigated using CV. Fig. 3 shows cyclic voltammograms for the GCE/AuNP-DAT (curve (1)), the GCE/AuNPs (curve (4)), and the bare GCE (curve (2)) in the buffered aqueous solution (pH = 7.0) containing IP (0.05 mmol/L) measured at scan rate of 20 mV/s. For comparison, curve (3) shows the cyclic voltammogram for the GCE/AuNP-DAT in the phosphate buffer solution without IP measured under the same conditions. Where as the electrochemical signal for the oxidation of IP at the GCE/AuNPs was not very different from that measured using the bare GCE (comparing curves (2) and (4)), the signal for the electro-oxidation of IP was significantly enhanced at the GCE/AuNP-DAT. The voltammograms indicated that the IP could be electro-catalyzed by the redox couple of the DAT molecules. A comparison of curves (1) and (3) in Fig. 3 indicated that the anodic peak current of the modified electrode was greatly increased in the presence of IP, and the cathodic peak

Scheme 2. Electrochemical reactions of DAT, and the electrocatalytic oxidation of IP at the GCE/AuNP-DAT.

disappeared. Based on these results, an EC' catalytic mechanism was proposed to describe the electrochemical oxidation of IP at GCE/AuNP-DAT; the mechanism is illustrated in Scheme 2. In this mechanism, IP is oxidized in the catalytic chemical reaction by the oxidized form of DAT, which is produced via an electrochemical reaction. Therefore, when the DAT was oxidized at a potential of 0.21 V, the IP could also be oxidized at this potential. The IP was thus oxidized at a potential of 0.21 V at the GCE/AuNP-DAT, while it was oxidized at a potential of approximately 0.36 V at the bare GC electrode. These results illustrated the electrocatalytic effect of the proposed electrocatalyst in improving the electro-oxidation of IP.

The effect of changing the scan rate on the electrocatalytic oxidation of IP at the modified electrode was investigated using cyclic voltammetry (Fig. 4(a)). A plot of the peak current (I_p) against the square root of the scan rate ($\nu^{1/2}$) was constructed (Fig. 4(b)); this plot was found to be linear, suggesting that the process was diffusion-controlled, rather than surface-controlled. The variation of the scan rate-normalized current ($I_p/\nu^{1/2}$) with changes in the scan rate, shown in Fig. 4(c), indicated that an electrocatalytic EC' mechanism was responsible for the oxidation of IP by the DAT self-assembled molecules at

Fig. 4. (a) Cyclic voltammograms for the GCE/AuNP-DAT measured in PBS (0.1 mol/L, pH = 7.0) containing IP (50 μ mol/L) at various scan rates (numbers 1–6 correspond to 5, 20, 30, 50, 70, and 100 mV/s); (b) Variation of the anodic peak current as a function of the scan rate; (c) Variation of the normalized current ($I_p/\nu^{1/2}$) as a function of the scan rate.

the electrode.

Chronoamperometric measurements of IP at the GCE/AuNP-DAT were performed for different concentrations of IP, using a potential step of 350 mV. These chronoamperograms were used to evaluate the catalytic rate constant, $k/(\text{L mol}^{-1} \text{s}^{-1})$, for the reaction between IP and DAT at the surface of modified electrode, based on the method of Galus [31]:

$$I_c / I_L = \gamma^{1/2} [\pi^{1/2} \text{erf}(\gamma^{1/2}) + \exp(-\gamma)/\gamma^{1/2}] \quad (3)$$

where I_c is the catalytic current of IP at the GCE/AuNP-DAT, I_L is the limited current in the absence of IP, and $\gamma = kC_b t$ is the argument of the error function (C_b is the bulk concentration of IP, and t is the time elapsed). In cases where γ exceeds a value of 2, the error function is almost equal to 1, and the above equation can therefore be reduced to:

$$I_c / I_L = \pi^{1/2} \gamma^{1/2} = \pi^{1/2} (kC_b t)^{1/2} \quad (4)$$

Therefore, k could be obtained for a given IP concentration based on the slope of the I_c/I_L versus $t^{1/2}$ plot. From the values of the slopes, the average value of k was found to be $373.45 \text{ L mol}^{-1} \text{s}^{-1}$.

3.3. Calibration plot and detection limit

Differential pulse voltammetry (DPV) was used to obtain a calibration plot for the detection and measurement of IP using the GCE/AuNP-DAT. Fig. 5(a) shows DP voltammograms measured for different concentrations of IP. Fig. 5(b) and (c)

show the plot of peak current versus IP concentration (calibration plot) for two linear segments with different slopes. For the concentration range of $1.0\text{--}80.0 \mu\text{mol/L}$ the sensitivity was $0.01221 \mu\text{A} (\mu\text{mol/L})^{-1}$, and for the concentration range of $80.0\text{--}1500.0 \mu\text{mol/L}$ the sensitivity was $0.0059 \mu\text{A} (\mu\text{mol/L})^{-1}$. The detection limit (3σ) for IP in the lower range was calculated as $0.49 \mu\text{mol/L}$. Table 1 shows a comparison between the performance of other modified electrodes and that of the present electrocatalyst for the determination of IP [32–36].

3.4. Elimination of the interferent effects of UA on the determination of IP

UA and IP have overlapping signals on bare electrodes, and the determination of IP in the presence of UA is therefore not possible using bare electrodes. Here, the DP voltammogram for IP was recorded in the presence of UA to investigate the effect of UA on the determination of IP using the GCE/AuNP-DAT. As shown in Fig. 6, the DPV peak for IP was at a potential of approximately 0.15 V and was separated from the DPV peak for UA, which was at a potential of approximately 0.39 V. These results showed that UA, the most interferent substance in biological environments for IP, did not have any interferent effect on the determination of IP; the results also showed that the simultaneous determination of IP and UA could be achieved using the proposed modified electrode.

Fig. 5. (a) Differential pulse voltammograms obtained using the GCE/AuNP-DAT in PBS (0.1 mol/L, pH = 7.0) containing different concentrations of IP (from inner to outer: 1, 10, 40, 60, 80, 100, 300, 500, 700, 900, 1100, 1300, and 1500 $\mu\text{mol/L}$); Plot of the peak currents vs. IP concentration in the range of $1\text{--}80 \mu\text{mol/L}$ (b) and $80\text{--}1500 \mu\text{mol/L}$ (c).

Table 1

Comparison of the performance of modified electrodes for determination of IP.

Electrode	Modifier	Linear range ($\mu\text{mol/L}$)	Detection limit ($\mu\text{mol/L}$)	Ref.
CPE	MWCNT modified with <i>N</i> -(3,4-dihydroxyphenethyl)-3,5-dinitrobenzamide	0.3–125.0	0.10	[32]
GCE	Graphene-L-cystine	4.0–160.0	0.84	[33]
CPE	Copper(II) hexacyanoferrate(III)	196.0–1070	0.80	[34]
GCE	Poly(L-serine acid)	0.5–10.0 and 30.0–120	0.01	[35]
GCE	Poly(1-methylpyrrole)-DNA	2.0–60.0	0.16	[36]
GCE	DAT and AuNPs	1.0–1500.0	0.49	This work

Fig. 6. Differential pulse voltammograms obtained using GCE/AuNP-DAT in PBS solution (0.1 mol/L, pH = 7.0) containing (a) IP and UA with respective concentrations of (1) 10 and 20 $\mu\text{mol/L}$, (2) 40 and 80 $\mu\text{mol/L}$, and (b) only 40 $\mu\text{mol/L}$ of IP (3), and only 80 $\mu\text{mol/L}$ of UA (4).

3.5. Stability of the composite electrocatalyst

The function of the composite GCE/AuNP-DAT was monitored over a one-month period by recording the electrocatalytic cyclic voltammogram for IP. The response of the electrocatalyst did not change significantly in the first two weeks. In this period the peak potential for the oxidation of IP at the modified electrode did not change, and the current signals showed a decrease of less than 4.0% relative to the initial response. After three weeks the current had decreased by more than 10%, relative to its initial value.

4. Conclusions

A glassy carbon electrode modified with gold nanoparticles showed good ability for the immobilization of a self-assembled monolayer of a thiophenol-hydroquinone derivative (DAT). The fabricated GCE/AuNP-DAT electrode exhibited suitable electrochemical properties for an electrocatalytic modified electrode. This electrode displayed electrocatalytic activity for the oxidation of IP and decreased the IP oxidation overpotential. The electrode could be used to determine the concentration of IP in the presence of UA. Good selectivity, a suitable linear range, a suitable detection limit, and its ease of preparation make the proposed modified electrode a useful tool for the accurate determination of IP.

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Graphical Abstract

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

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A nanocomposite electrocatalyst was prepared by immobilizing 3,4-dihydroxyphenyl-azo-2-thiophenol (DAT) on a gold nanoparticle-modified glassy carbon electrode (GCE/AuNPs). The electrocatalyst catalyzed the electrochemical oxidation of isoproterenol via a mediating electrocatalysis mechanism.

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电氧化异丙肾上腺素的纳米复合电催化剂：用作传感器

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摘要: 在金纳米粒子(AuNPs)上经苯硫酚衍生物(3,4-二羟基苯基-偶氮-苯硫酚, DAT)自组装制得了一种新型纳米复合物, 用于修饰玻璃碳电极(GCE/AuNP-DAT)。采用循环伏安法研究了该新型电极的性质, 并将其用作异丙肾上腺素(IP)电催化剂, 考察了该纳米复合物的电催化活性, 从而得到反应机理和催化反应速率常数。由于GCE/AuNP-DAT电极对尿酸氧化没有电催化活性, 因此可将IP的氧化信号从该改进电极中分离出来, 从而排除了尿酸对IP测定的干扰。该电极可作为传感器, 当用于差动脉冲伏安法测定IP时, 线性动态范围为1.0–1500.0 $\mu\text{mol/L}$, 检测极限为0.46 $\mu\text{mol/L}$ 。

关键词: 电催化剂; 金纳米粒子; 自组装; 异丙肾上腺素; 传感器

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