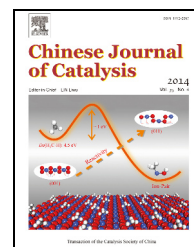


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Article

Simultaneous determination of the concentrations of isoproterenol, uric acid, and folic acid in solution using a novel nanostructure-based electrochemical sensor

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ABSTRACT

A carbon paste electrode modified with 2-((7-(2,5-dihydrobenzylideneamino)heptylimino)methyl) benzene-1,4-diol (DHB) and carbon nanotubes were used to simultaneously determine the concentrations of isoproterenol (IP), uric acid (UA), and folic acid (FA) in solution. First, cyclic voltammetry was used to investigate the redox properties of the modified electrode at various scan rates. Next, the mediated oxidation of IP at the modified electrode is described. At the optimum pH of 7.0, the oxidation of IP occurs at a potential about 90 mV less than that of an unmodified carbon paste electrode. Based on the results of differential pulse voltammetry (DPV), the oxidation of IP showed a dynamic range between 10 and 6000 $\mu\text{mol/L}$, and a detection limit of 1.24 $\mu\text{mol/L}$. Finally, DPV was used to simultaneously determine the concentrations of IP, UA, and FA in solution at the modified electrode.

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1. Introduction

Electrochemical sensors composed of modified electrodes have proven to be an inexpensive and simple analytical tool with remarkable detection sensitivity, reproducibility, and ease of miniaturization compared with other instrumental analysis methods [1–4]. They have been used in a wide range of important applications in clinical, industrial, environmental, and agricultural analyses [1,5,6]. The modification of electrodes with redox modifiers is an interesting field in analytical chemistry. Redox modifiers are electroactive compounds that effectively shuttle electrons between the analyte and the electrode. One of the most important effects of any modifier is the reduction of the overpotential required for electrochemical reaction, which enhances the sensitivity and selectivity of the method.

Because carbon nanotubes (CNTs) possess interesting electrochemical properties owing to the activity of edge-plane-like graphite sites at the CNT ends, they can be used to construct modified electrodes. These electrodes exhibit a low detection limit, high sensitivity, and fast response owing to the signal enhancement provided by the high surface area, low overvoltage, and rapid electrode kinetics [7,8].

Isoproterenol (IP) is widely used for the treatment of primary pulmonary hypertension and allergic emergencies, status asthmaticus, bronchial asthma, ventricular bradycardia, cardiac arrest, and glaucoma [9]. It is also used to treat bronchitis, cardiac shock and heart attack. However, excess of the drug may cause heart failure and arrhythmias [10]. Several methods have been described to determine the concentration of IP in solution, such as spectrofluorometry, chemiluminescence [11], spectro-

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photometry [12], chromatography, and chemiluminescence methods. While these methods are often time consuming, inherently expensive, or complicated, electrochemical methods for the determination of the concentration of IP in solution have advantages such as simplicity, speed, low cost, and ease of use. Amperometric and voltammetric detection are inexpensive and sensitive techniques, and they have been widely used to detect electroactive compounds in pharmaceutical formulations, biological matrices, and medicinal herbs. Because IP contains hydroxyl groups on the aromatic ring and it has an NH group, it is electroactive, making it suitable for electrochemical detection [13–18].

Uric acid (UA) is a primary end product of purine metabolism. Abnormal levels of UA are an indicator of several diseases, such as gout, hyperpiesia, and Lesch-Nyhan syndrome [19]. Hence, monitoring the concentration of UA in biological fluids can be used as an early warning sign of these diseases. Colorimetric, enzymatic, and electrochemical methods are used to determine the concentration of UA [20,21]. The colorimetric method is unreliable for the accurate determination of the concentration of UA. Although determining the concentration of UA by enzymatic methods is promising because of their high level of selectivity, this methodology is inherently expensive and does not have a high detection limit. Electrochemical methods are more selective, less expensive, and less time-consuming than the other methods for determining the concentration of UA [22].

Folic acid (FA) is a water-soluble vitamin that acts as a co-enzyme in the transfer and utilization of one-carbon groups and in the regeneration of methionine from homocysteine [23]. Deficiency of FA is a common cause of anemia and it is thought to increase the likelihood of heart attack and stroke. Many studies suggest that diminished folate status is associated with increased carcinogenesis because FA and vitamin B12 are involved in nucleotide synthesis, cell division, and gene expression [24]. Numerous methods for the measurement of FA are available, including enzyme-linked immunosorbent assays [22], liquid chromatography–tandem mass spectrometry [25], capillary electrophoresis [26], microemulsion electrokinetic chromatography [27], and high-performance liquid chromatography [28]. Because FA is an electroactive component, some electrochemical methods have been reported for its determination [29,30]. Compared with other methods, the electrochemical method is more desirable because of its convenience and low cost.

However, some studies have emphasized the instability of the attached materials on the electrodes as a problem in the use of chemically modified electrodes. It seems that the incorporation of electrocatalysts into the electrode matrix can, even partly, help to solve these problems, and carbon paste electrodes (CPEs) spiked with catalyst may be suitable for this purpose. The ease and speed of preparation for obtaining a new reproducible surface, the low residual current, the porous surface, and low cost are some advantages of chemically modified carbon paste electrodes [31–33].

In this study, we fabricated a novel nanostructure modified carbon paste electrode based on 2-((7-(2,5-dihydrobenzylideneamino)heptylimino)methyl)benzene-1,4-diol (DHB) and

the unique properties of CNTs, and investigated its performance for the electrocatalytic determination of the concentration of IP in aqueous solution. We also evaluated the analytical performance of the modified electrode for quantification of IP in the presence of UA and FA. We found that DHB possesses high stability and good electrocatalytic activity toward the electrocatalytic oxidation of IP. In addition, we discuss the suitability of this modified electrode in the voltammetric determination of the concentration of IP in an aqueous solution by linear sweep voltammetry, differential pulse voltammetry, and chronoamperometry. Finally, the modified electrode was applied to determine the concentration of IP in blood samples.

2. Experimental

2.1. Apparatus and chemicals

The electrochemical measurements were performed with a potentiostat/galvanostat (SAMA 500, Iran). The experimental temperature was controlled at 25 ± 1 °C. A three-electrode cell was constructed, including a platinum wire, a saturated calomel electrode (SCE), and the modified electrode with modifier and nanoparticles (DHB/CNT/CPE) used as the auxiliary, reference, and working electrodes, respectively. A Metrohm 691 pH meter was used to measure the pH of the solutions. IP, UA, FA, and all the other reagents were of analytical grade from Merck (Darmstadt, Germany). Graphite fine powder and paraffin oil (DC 350, density = 0.88 g/cm³, Merck) were used as the binding agent to prepare the pastes. Buffer solutions were prepared from ortho-phosphoric acid and its salts in the pH range 4.0–9.0.

2.2. Synthesis of DHB

1,7-Diaminoheptane (0.18 g, 1.4 mmol) was added to a mixture of 2,5-dihydroxybenzaldehyde (0.35 g, 2.5 mmol) in methanol and stirring for 30 min. The progress of the reaction was monitored by thin-layer chromatography. After the reaction was complete, the yellow solid product was filtered off and washed with cold methanol and pure Schiff base. 2,2'-[1,7-Heptanediyl bis(nitrilomethylidene)]-bis(4-hydroxyphenol) was obtained in 90% yield (mp = 215–217 °C).

The Schiff base product was identified by physical and spectroscopic data as 2-((7-(2,5-dihydrobenzylideneamino)heptylimino)methyl)benzene-1,4-diol: yellow solid; yield 90%; and mp 215–217 °C. Anal. Calcd.: C 68.1, H 7.02, N 7.6; Found: C 68.1, H 6.9, N 7.4. IR (KBr, cm⁻¹): ν 3373–3500 (s, br, 2OH), 1646 (s, C=N), 1503, 1457 (Ar), 1239 (s, C–O). ¹H NMR (400 MHz/DMSO-d₆): δ 12.7 (br, 2OH, intramolecular hydrogen bonding), 8.90 (br, 2OH), 8.42 (s, 2CH imine), 6.78 (d, 2H, Ar, J = 4 Hz), 6.76 (dd, 2H, Ar, J_1 = 8.2 Hz, J_2 = 2.2 Hz), 6.69 (d, 2H, Ar, J = 8.4 Hz), 3.54 (t, 4H, 2CH₂, J = 12.8 Hz), 1.58 (m, 8H, 4CH₂), 1.33 (m, 2H, CH₂). ¹³C NMR (100 MHz/DMSO-d₆): δ 165.77, 153.62, 149.70, 120.12, 119.01, 117.25, 116.90, 59.02, 30.83, 28.88, 27.03. MS: m/z = 370 (M⁺, 4), 235 (6), 220 (22), 206 (28), 193 (90), 179 (32), 150 (68), 137 (92), 126(100). UV/ λ_{\max} : 340 (s), 240 (w).

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