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Liquid phase determination of isuprel in pharmaceutical and biological samples using a nanostructure modified carbon paste electrode

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ABSTRACT

The electrochemical behavior of isuprel (ISP) on the ionic liquid of 1,3-dipropylimidazolium bromide NiO nanoparticle (NiO/NPs) modified carbon paste electrode (IL/NiO/NPs/CPE) was studied in this paper and further used for ISP detection. ISP showed a diffusion oxidation process on the IL/NiO/NPs/CPE with the oxidation peak potential located at 0.62 V (vs. Ag/AgCl/KCl_{sat}) in a pH 5.0. Compared with that on the traditional carbon paste electrode, small shift of the oxidation peak potentials appeared but with a great increment of the oxidation peak current on the IL/NiO/NPs/CPE, which was due to the presence of ionic liquid and NiO/NPs in the modified electrode. The diffusion coefficient (D) was also determined using electrochemical approaches. The linear response range and detection limit were found to be 0.4–500 μmol L^{-1} and 0.1 μmol L^{-1}, respectively. Other physiological species did not interfere in the determination of ISP at the surface of the proposed sensor in the optimum condition. The proposed sensor was successfully applied for the determination of ISP in real samples.

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1. Introduction

Isuprel is a medication used for the treatment of bradycardia, heart block, and rarely for asthma. It is a non-selective beta-adrenergic agonist and structurally similar to adrenaline [1]. ISP should not be administered to patients with myocardial ischemia. ISP effects on the cardiovascular system (non-selective) relate to its actions on cardiac β_1 receptors and β_2 receptors on smooth muscle within the tunica media of arterioles. Isuprel has positive inotropic and chronotropic effects on the heart. β_2-adrenoceptor stimulation in arteriolar smooth muscle induces vasodilation [2]. Therefore, the determination of ISP is very important in physiological fluids in the diagnosis and treatment of diseases such as gout, hyperuricemia, heavy hepatitis, and Lesch–Nyhan syndrome [3].

In recent time's carbon ionic liquid electrode modified with nanomaterials, which was made by the ionic liquid, nanomaterials and graphite powder had been proposed in the field of electroanalysis [4–9]. As a new kind of chemical modified electrode, ionic liquid electrode modified with nanomaterials had exhibited many excellent properties such as good sensitivity, high conductivity, wide potential window and good anti-fouling ability [10–13].

Conducive nanoparticles and especially metal oxide nanoparticles are a subject of constantly growing interest in many research areas such as biomedical applications [14], photovoltaic systems [15], electrocatalysis [16–20], and lithium-ion batteries [20,21]. Synthesis and investigation of new nanostructure based compounds are very important in nanosensing because variety of shapes, sizes and compositions in analytical application [22–28].

In this paper, we describe the synthesis and application of NiO/NPs modified carbon paste electrode, which utilizes 1,3-dipropylimidazolium bromide as a binder. The electrochemical behavior of ISP at IL/NiO/NPs/CPE, at carbon paste electrode modified with ionic liquid (IL/CPE), at NiO/NPs carbon paste electrode (NiO/NPs/CPE), and at CPE was investigated. The results showed the superiority of IL/NiO/NPs/CPE to the other electrodes in terms of both provision of better reversibility and higher sensitivity. Finally, IL/NiO/NPs/CPE was successfully applied for the determination of ISP in real samples.

2. Experimental

2.1. Chemicals

All chemicals used were of analytical reagent grade purchased from Merck (Darmstadt, Germany) unless otherwise stated. Doubly distilled

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water was used throughout. ISP was from Sigma. Other reagents were used without further purification.

A 1.0×10^{-2} mol L^{-1} ISP solution was prepared daily by dissolving 0.06 g ISP in water in a 25–mL volumetric flask. The solution was kept in a refrigerator at 4 °C in dark. More dilute solutions were prepared by serial dilution with buffer solution.

Phosphate buffer solutions (sodium dihydrogen phosphate and disodium monohydrogen phosphate plus sodium hydroxide, 0.1 mol L^{-1}), PBS, with different pHs were used.

Spectrally pure graphite powder (particle size < 50 μm), and high viscous paraffin oil (density = 0.88 kg L^{-1}) from Merck were used as the substrate for the preparation of the working electrodes.

2.2. Apparatus

Voltammetric and electrochemical impedance spectroscopy studies were performed in an analytical system, Autolab PGSTAT 302N, potentiostat/galvanostat connected to a three electrode cell, Metrohm Model 663 VA stand, linked with a computer (Pentium IV, 1200 MHz) and run with GPES and FRA 4.9 software. Frequency range of 100 kHz to 1.0 Hz was employed for impedance measurements. The AC voltage amplitude used was 5 mV, and the equilibrium time was 20 min. A conventional three-electrode cell assembly consisting of a platinum wire as an auxiliary electrode and an ($Ag/AgCl/KCl_{sat}$) electrode as a reference electrode were used. The working electrode was either an unmodified or modified carbon paste electrode.

2.3. Preparation of the modified electrode

IL/NiO/NPs/CPE was prepared by mixing of 0.2 g of ionic liquid, 0.70 g of the liquid paraffin, 0.3 g of NiO/NPs, and 0.70 g of graphite powder. Then the mixture was mixed well for 50 min until a uniformly wetted paste was obtained. A portion of the paste was filled firmly into one glass tube as described above to prepare IL/NiO/NPs/CPE. Electrical contact was made by pushing a copper wire down the glass tube into the back of the mixture. When necessary, a new surface was obtained by pushing an excess of the paste out of the tube and polishing it on a weighing paper.

2.4. Preparation of real samples

Urine samples were stored in a refrigerator at 4 °C immediately after collection. Ten milliliters of each sample was centrifuged for 15 min at 2500 rpm. The supernatant was filtered using a 0.45 μm filter and then diluted five times with PBS (pH 7.0). The solution was transferred into the voltammetric cell to be analyzed without any further pretreatment. Standard addition method was used for the determination of morphine in real samples.

Ampoule (0.20 mg mL^{-1}) prepared and then 0.10 mL of the solution plus 10 mL of 0.1 mol L^{-1} buffer (pH 5.0) was used for the analysis.

2.5. Synthesis of NiO nanoparticle

0.5 M aqueous solution of Ni $(NO_3)_2.6H_2O$ and 0.5 M aqueous solution of sodium hydroxide (NaOH) were prepared in distilled water. Then, the beaker containing NaOH solution was heated at the temperature of about 60 °C. The Ni $(NO_3)_2.6H_2O$ solutions were added drop wise (slowly for 1.5 h) to the above-heated solution under high-speed stirring. The beaker was sealed at this condition for 2 h. The precipitated NiO/NPs were cleaned with deionized water and ethanol then calcined at 350 °C for 2.0 h. Main reactions occur during the experimental procedure can be written briefly as follows:

$$2NaOH_{(s)} \rightarrow 2Na^{+}{}_{(aq)} + 2OH^{-}{}_{(aq)}, \qquad (1)$$

$$Ni(NO_3)_2 \cdot 6H_2O_{(s)} \rightarrow Ni^{2+}{}_{(aq)} + 2NO_3{}^{-}{}_{(aq)} + 6H_2O_{(aq)}, \qquad (2)$$

$$Ni^{2+}{}_{(aq)} + 2OH^{-}{}_{(aq)} + xH_2O_{(aq)} \rightarrow Zn(OH)_2 \cdot xH_2O_{(s)} \downarrow, \qquad (3)$$

$$Ni(OH)_2 \cdot xH_2O_{(s)} \overset{100}{\rightarrow} Ni(OH)_{2(s)} + xH_2O_{(g)} \uparrow, \qquad (4)$$

$$Ni(OH_2)_{(s)} \overset{300}{\rightarrow} NiO_{(s)} + H_2O_{(g)} \uparrow. \qquad (5)$$

3. Results and discussion

3.1. Nanostructure characterization

The purity and crystallinity of the as-synthesized NiO/NPs were examined by using powder X-ray diffraction (XRD) as shown in Fig. 1. Result shows that the diffraction peaks are low and broad due to the small size effect and incomplete inner structure of the particle. The peaks positions appearing at $2\theta = 37.50°$, 43.10°, 62.57°, 76.50°, and 79.21° can be readily indexed as (111), (200), (220), (311), and (222) crystal planes of the bulk NiO, respectively. All these diffraction peaks can be perfectly indexed to the face-centered cubic (FCC) crystalline structure of NiO. The XRD pattern shows that the samples are single phase and no any other impurities distinct diffraction peak except the characteristic peaks of FCC phase NiO was detected. The average crystallite size is calculated by X-ray diffraction line broadening using the Scherrer formula $d = K\lambda/B \cos\theta$. The grain size of the NiO nanoparticle was 25.0 nm.

3.2. Electrochemical investigation

ISP can be oxidized at positive potential depending on the electrode type and solution pH (see Scheme 1). In order to ascertain this, the voltammetric response of ISP was obtained in solutions with varying pH from 2.0 to 6.0 (Fig. 2, inset) at the surface of IL/NiO/NPs/CPE. The results showed that the peak potential (E) of ISP shifted negatively as the solution pH increased, which indicated that protons were involved in the electrode reaction. A good linear relationship between the peak potential (E) and the solution pH was also established (not shown). The linear regression equation was gotten as E (mV) = −51.0 pH + k ($n = 5$, $r^2 = 0.993$). According to the Nernstian slope ($-59.0x/n$), where x is the hydrogen ion participating the electrode reaction and n is the number

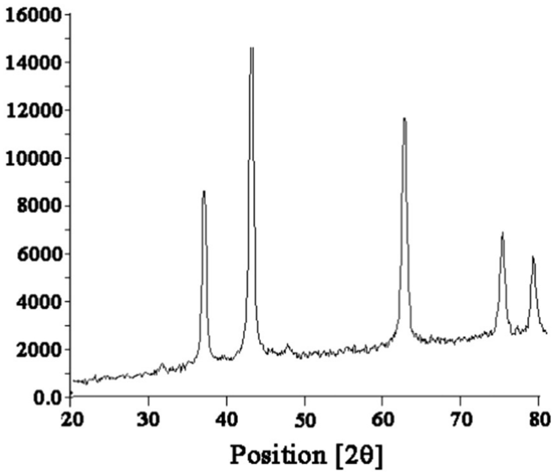

Fig. 1. XRD patterns of as-synthesized NiO/NPs nanoparticles.

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