



Electrochemical investigation of the voltammetric determination of hydrochlorothiazide using a nickel hydroxide modified nickel electrode

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ABSTRACT

The preparation and electrochemical characterization of a nickel hydroxide modified nickel electrode as well as its behavior as electrocatalyst toward the oxidation of hydrochlorothiazide (HCTZ) were investigated. The electrochemical behavior of the modified electrode and the electrooxidation of HCTZ were explored using cyclic voltammetry. The voltammetric response of the modified electrode in the detection of HCTZ is based on the electrochemical oxidation of the Ni(II)/Ni(III) and a chemical redox process. The analytical parameters for the electrooxidation of HCTZ by the nickel hydroxide modified nickel electrode were obtained in NaOH solution, in which the linear voltammetric response was in the concentration range from 1.39×10^{-5} to 1.67×10^{-4} mol L⁻¹ with a limit of detection of 7.92×10^{-6} mol L⁻¹ and a sensitivity of $0.138 \mu\text{A L mmol}^{-1}$. Tafel analysis was used to elucidate the kinetics and mechanism of HCTZ oxidation by the modified electrode.

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1. Introduction

Electrochemical methods comprise a collection of extremely useful and versatile measurement tools in biological [1–5], food [6–9] and environmental [10–13] analysis due the high sensitivity, selectivity, accuracy and precision, as well as wide linear dynamic range, with relatively low-cost instrumentation. In the pharmaceutical area [14], the use of electrochemistry is an important approach in drug discovery and research as well as quality control, drug stability, determination of physiological activity and especially in their dosage forms in biological samples.

The diuretics are substances used to increase the production of urine and sodium excretion in order to adjust the volume and composition of body fluids or to eliminate excess of fluids [15]. As therapeutic agents, they are used in the treatment of congestive heart failure, edemas, hypertension, kidney, liver cirrhosis, lung diseases, and prophylaxis of renal failure [15]. According to the World Anti-doping Agency (WADA) the diuretics are on the list of prohibited substances in sports [16] due to their potent ability to remove water from the body and to mask the administration of other doping agents, conditions which promote rapid weight loss that can be required to meet a weight category and reducing their concentration in urine primarily because of an increase in urine volume, respectively [17]. In this manner, it was expected that these substances were not found in biological samples in athletes. Despite this, some athletes misuse diuretics in sports in order to give them competitive advantage.

Among the diuretics, the most common drug detected in the year 2008 by WADA laboratories [17] was hydrochlorothiazide (6-chloro-1,1-dioxo-3,4-dihydro-2H-1,2,4-benzothiadiazine-7-sulfonamide — HCTZ). The HCTZ is a benzothiazide diuretic that acts directly on the kidney by increasing the excretion of sodium chloride and water and, to a lesser extent, that of potassium ion [18]. Also, it is a antihypertensive drug which improves the action of other hypotensive substances.

Thus, the development of convenient, rapid, selective and sensitive analytical methods for the determination of illicit diuretics, such as HCTZ, in human urine samples, especially in the urine samples of athletes has gained great importance. The official method recommended by the United States Pharmacopeia [19] for the determination of HCTZ involves the use of high performance liquid chromatography (HPLC). Still, in recent years, different methods have been reported for the detection of HCTZ in pharmaceutical formulations or biological samples, such as capillary electrophoresis [20–22], chemiluminescence [23,24], chemometry [25], conductimetry [26], polarography [27], liquid chromatography/tandem mass spectrometry [28–31] and spectrophotometry [32–34].

Although there are several methods, most of these require large amounts of sample, several time consuming manipulation steps, sophisticated instruments and special training. To minimize these problems and achieve better sensitivity and selectivity, there is the possibility of using electrochemical techniques applying electrochemical sensors. In this way, several electrochemical sensors for HCTZ detection based on unmodified and modified electrodes have been developed (Table 1).

Among the wide range of chemical modifier compounds, the nickel hydroxide has gained notoriety due its ease in construction of modified

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Table 1

A brief review of electrochemical sensors for HCTZ.

Material	Ref.
5-Amino-20-ethyl-biphenyl-2-ol modified carbon paste electrode	[41]
Benzoylferrocene-modified carbon nanotube paste electrode	[37]
Boron-doped diamond electrode	[38]
Carbon nanotube/polypyrrole film	[42]
Carbon nanotubes/silicone rubber composite electrode	[43]
Ferrocenedicarboxylic acid modified carbon paste electrode	[44]
Glassy carbon electrode	[39]
Graphene/ferrocene composite carbon paste electrode	[45]
Multiwalled carbon nanotubes modified electrode	[46]

electrodes both in the form of thin films and micro/macro-scale, in addition to the presence of sites of Ni(II), especially in its oxidized form Ni(III), which have excellent properties in the electrochemical oxidation of various organic molecules [35]. Therefore, in this manuscript, the preparation and electrochemical characterization of a nickel hydroxide modified nickel electrode, as well as, its behavior as electrocatalyst toward the oxidation of hydrochlorothiazide were investigated.

2. Material and methods

2.1. Apparatus

Cyclic voltammetry measurements were carried out with a micro-Autolab (Metrohm) controlled by a microcomputer using de GPES 4.9 software in a thermostatic electrochemical cell containing three electrodes: nickel hydroxide modified nickel electrode (NMN) as the working electrode, platinum wire as the auxiliary electrode and saturated calomel electrode (SCE) as the reference electrode.

2.2. Reagents and solutions

All the chemicals were of analytical high-purity and all solutions were prepared using Millipore Milli-Q deionized water. The supporting electrolyte used for all experiments was 0.5 mol L⁻¹ NaOH (Sigma-Aldrich). The analyte solution was prepared by the addition of HCTZ (Sigma-Aldrich) in 0.5 mol L⁻¹ NaOH.

2.3. Preparation of nickel and nickel hydroxide-modified nickel (NMN) electrode

A nickel (100% purity) rod with a diameter of 6.24 mm and length of 10.0 cm was introduced in a glass cylinder with inner diameter of 8.0 mm and was wrapped with epoxy resin. This electrode stayed for 24 h in room temperature to dry. The electrode surface was polished using 400 and 600 mesh emery paper and thoroughly rinsed with distilled water. The modification of the working electrode was carried out by cyclic voltammetry in a range of potential from 0.25 V to 0.50 V with fifty potential cycles in 0.5 mol L⁻¹ NaOH solution.

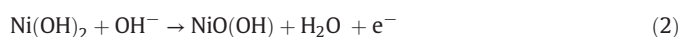
2.4. Performance of the modified nickel electrode as electrochemical sensor for HCTZ

The HCTZ determination was made by the application of potentials via linear voltammetry using a potential range from 0.25 V to 0.45 V vs. SCE at a scan rate of 25 mV s⁻¹. The modified nickel electrode was submitted to potential scans in 25 mL of 0.5 mol L⁻¹ NaOH. The evaluation of the electrochemical performance of sensor for HCTZ was conducted by the addition of the analyte (1.0 mmol L⁻¹) in the electrochemical cell.

3. Results and discussion

3.1. Activation of nickel modified electrode

The chemical modification on the nickel electrode surface was made applying 50 potential cycles in a potential range from 0.25 V to 0.50 V vs. SCE at a scan rate of 25 mV s⁻¹ in 0.5 mol L⁻¹ NaOH solution. Cyclic voltammograms (Fig. 1) obtained show that consecutive potential scans lead to a progressive increase of anodic and cathodic peak current values, indicating the formation of the Ni(OH)₂ in surface electrode. The electrode becomes derivatized to the oxide species quantitatively in surface. The electrochemical reactions may be described as follows [35]:



The shift of the peaks due to the number of scan is due changes in crystal structures of the nickel hydroxide and nickel oxyhydroxide on electrode surface.

3.2. Electrochemical properties of the nickel hydroxide modified nickel electrode

After the activation of the electrode, the electrochemical behavior of the modified nickel electrode was studied by cyclic voltammetry in 0.5 mol L⁻¹ NaOH solution. The electrochemical processes related to the modified electrode were stabilized after fifteen potential cycles. It is further observed an increase the reversibility of the redox system. From the second potential scan, both currents slightly increased and the distance between the peak potentials decreased to stabilize after fifteen cycles (Supplementary material).

The cyclic voltammogram obtained after stabilization for the modified electrode (Supplementary material) revealed a reversible system with only one redox couple ($E_{pc} = 0.330$ and $E_{pa} = 0.396$ V vs. SCE for scan rate of 25 mV s⁻¹) which is attributed to the redox process of Ni(II)/Ni(III), peak-to-peak separation (ΔE_p) of 0.066 V and half potential ($E_{p/2}$) of 0.363 V versus SCE, according to Eq. (2).

The effect of the potential scan rates (5 to 150 mV s⁻¹) on the voltammetric response for the modified electrode in 0.5 mol L⁻¹ NaOH solution was investigated. The recorded cyclic voltammograms

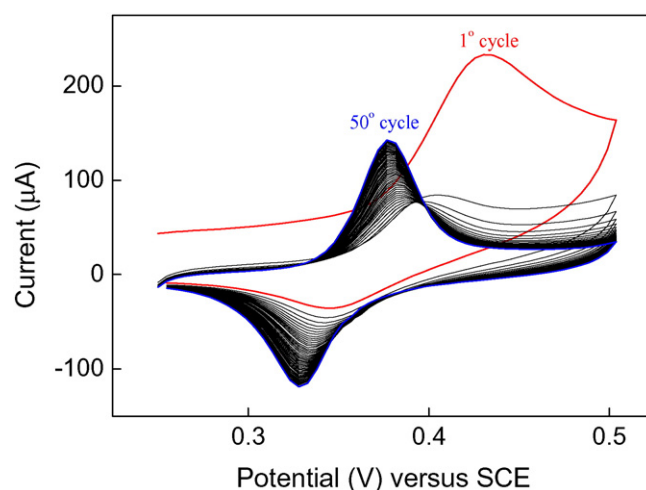


Fig. 1. Cyclic voltammograms for deposition of nickel hydroxide on the nickel electrode surface applying 50 potential cycles in a potential range from 0.25 to 0.50 (V) vs. SCE at scan rate of 25 mV s⁻¹ in 0.5 mol L⁻¹ NaOH solution. Red line = 1st cycle; blue line = 50th cycle.

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