Electrochimica Acta 127 (2014) 20–26

Contents lists available at ScienceDirect

Electrochimica Acta

journal homepage: www.elsevier.com/locate/electacta

Sensitive Voltammetric Determination of Niclosamide at a disposable pencil graphite electrode

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ARTICLE INFO

Article history:
Received 15 December 2013
Received in revised form 17 January 2014
Accepted 26 January 2014
Available online 7 February 2014

Keywords:
Niclosamide
Pencil graphite electrode
Differential pulse voltammetry
Cyclic voltammetry

ABSTRACT

Niclosamide (NA), an effective drug against tapeworm infections, was electrochemically studied using a pencil graphite electrode (PGE). A low-cost sensitive and selective procedure was developed for determination of NA by recording differential pulse voltammograms of NA in pH 7.0 Britton-Robinson buffer solution containing 0.1 M KCl and 30% DMF at the PGE. The PGE displayed very good electrochemical behavior with significant enhancement of the peak current compared to a glassy carbon electrode. Moreover, results obtained from the differential pulse voltammograms show that PGE offers a disposable, low cost, selective and sensitive electrochemical sensor for determination of NA. Under experimental conditions, the PGE had a linear response range from 0.05 μM to 10 μM NA with a detection limit of 0.015 μM (based on 3_{sb}). A relative standard deviation of 0.57% was obtained for five successive determinations of 5 μM NA, which indicate acceptable repeatability. This voltammetric method was successfully applied to the direct determination of NA in tablets. No interference from the tablet excipients was encountered.
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1. Introduction

An important ethanolamine salt, Niclosamide (2′,5-dichloro-4′-nitrosalicylanilide, NA, Scheme 1), is a teniacide of the anthelmintic family that is especially effective against cestodes that infect humans [1]. Concerning the form of dosage, it is usually taken in chewable tablets depending on the type of worm and patient's age and/or weight. NA is used as an effective drug against tapeworm infections due to its lethal effect on tapeworms upon contact and is not effective against other worms such as pinworms or roundworms [2,3]. Its activity against worms interferes with the electron transport linked to oxidative phosphorylation and suppresses the glucose uptake [4]. It is also used as a piscicide, which is a chemical substance that is poisonous to fish. In spite of its wide applications, NA appears to be toxic to some aquatic organisms [5,6] and its long-term administration causes problems to terrestrial and aquatic species or life [5]. Therefore, achieving sensitive and selective determination of NA has attracted a lot of attention and a number of qualitative and quantitative methods have already been developed for NA determination. With this aim, spectroscopic techniques such as spectrophotometry and fluorimetry on derivatives or complexes of NA [7–11], chromatographic techniques such as liquid chromatography–tandem mass spectrometry (LC-MS-MS) [12], high performance liquid chromatography (HPLC) [13–15], gas-liquid chromatography [16,17], and electrochemical techniques such as polarography [18] and voltammetry [19–24] have been proposed. However, spectrophotometric methods that involve complex formation or derivatization are nonselective and subject to high interference [7–11]. Chromatographic methods suffer from disadvantages such as the laborious derivatization procedure involved in the modification of NA by various reagents, high cost, and their being time-consuming [12–17]. The polarographic method based on the reduction of NA has a very narrow linear range [18]. Therefore, development of an alternative analytical methodology for determination of NA has become necessary; in particular, a method that is both sensitive and simple.

One of the most efficient approaches in pharmaceutical analysis is the use of modern electroanalytical methods, which have the advantage of easy application, high sensitivity, accuracy and selectivity, simplicity and fast (less time-consuming) detection and low cost. Therefore, differential pulse and square wave voltammetric techniques have been reported for electrochemical determination of NA using a bare glassy carbon electrode (GCE) [20–22]. However, it was reported that the use of an unmodified GCE suffers from sluggish electron transfer and fouling of surface which result in poor sensitivity and selectivity. In order to improve sensitivity and avoid fouling, modified GCEs such as carbon nanoparticle/Chitosan composite/GCE [23] and poly(3,4-ethylenedioxythiophene) modified GCEs [24] have been proposed for sensitive and selective determination of NA.

In this study, a pencil graphite electrode (PGE) was used for sensitive and selective voltammetric determination of NA. When compared with other carbon-based electrodes, PGEs have the same advantages, such as high electrochemical reactivity, commercial

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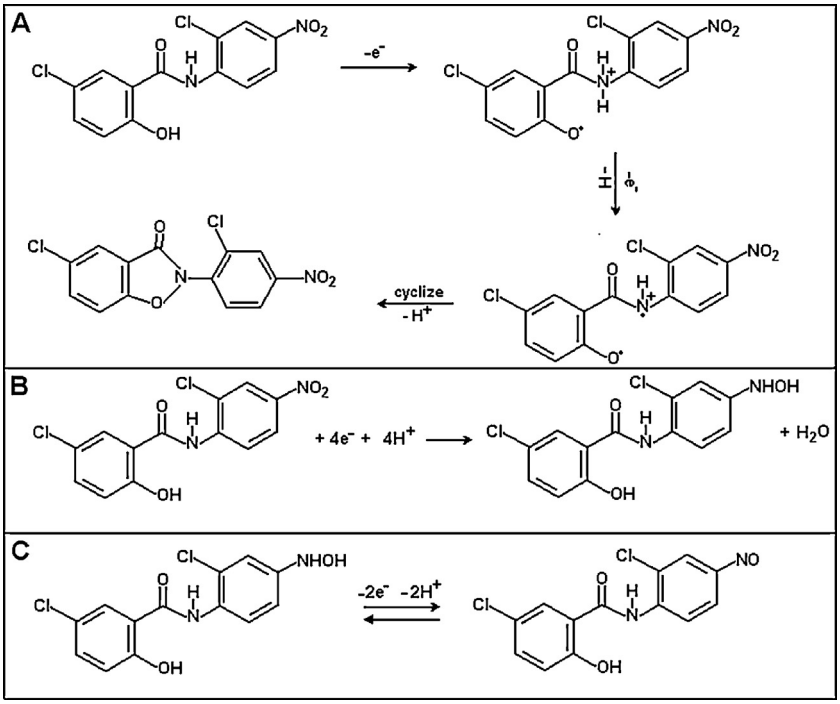
0013-4686/$ – see front matter © 2014 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.electacta.2014.01.153



Scheme 1. Proposed mechanism for electrochemical behavior of NA.

availability, good mechanical rigidity, disposability, low cost, low technology and ease of modification [25]. In addition, it was reported that pencil lead electrodes offer a renewal surface which is simpler and faster than polishing procedures, common with solid electrodes, and result in good reproducibility for individual surfaces [25]. Thus, many scientists have recently focused on the use of these electrodes in various electroanalytical applications due to the useful properties of PGEs [25–34]. Although the above-mentioned modified electrodes demonstrated very good sensitivity and a low detection limit, these methods are time-consuming due to preparation of the modified electrodes and the electrode materials are more expensive than PGEs.

Taking into account the good properties of PGEs in electroanalysis, in this work we used PGE for NA determination using the differential pulse voltammetric technique with improved qualities such as easy availability, simplicity, disposability and low cost of electrode, wider linear range, low detection limit, and high selectivity.

2. Material and Methods

2.1. Apparatus and Chemicals

H_3PO_4 (85%, d: 1.71 g mL^{-1}), CH_3COOH (96%, d: 1.05 g mL^{-1}), H_3BO_3, NaOH and KCl were purchased from Merck (USA) and Niclosamide, Dimethyl formamide (DMF) were purchased from Sigma (USA). A stock standard solution of NA (10^{-2} M) was prepared in DMF and kept in the dark. The required concentration of NA in aqueous buffer solutions was then prepared from the stock standard solution. Britton-Robinson (BR) buffer solutions in the pH range 7–10 were prepared from 0.04 M H_3PO_4, 0.04 M H_3BO_3 and 0.04 M CH_3COOH containing 0.1 KCl in deionized water. The pH of the solutions was adjusted by adding 0.2 M NaOH.

Cyclic and differential pulse voltammetric experiments were performed in a traditional three-electrode system using a platinum wire as the counter electrode, an $Ag/AgCl/KCl_{sat}$ as the reference electrode, and a PGE as the working electrode. A pencil lead with a diameter of 0.5 mm (Ultra-Polymer, 2B) and a total length of 60 mm (Tombow, Japan), and a mechanical pencil Model T 0.5 (Rotring, Germany), which was used as the holder for the pencil lead, were purchased from a local bookstore. Electrical contact to the lead was obtained by wrapping a metallic wire to the metallic part of the holder. For each measurement, a total of 10 mm of lead was immersed into the solution. The length of pencil lead was measured with a ruler as 10 mm into the supporting electrolyte and then was dipped into a tip of the electrochemical cell.

All electrochemical experiments were carried out using a Compactstat Electrochemical Interface (Ivium Technologies, Eindhoven, The Netherlands). A HI 221 Hanna pH-meter with a combined glass electrode (Hanna HI 1332) was used to follow the pH values of the solutions. The solutions throughout this work were all prepared using deionized water from a Milli-Q (Millipore, Bedford, USA) device.

2.2. Procedure

In the electrochemical measurements, PGE was used directly while GCE was used after it was polished with alumina slurry on the polishing cloth, washed with deionized water and sonicated with DMF and water in an ultrasonic bath, respectively. Firstly, the electrochemical behavior of NA at PGE and also GCE was investigated by recording cyclic voltammograms in the BR buffer solution in the pH range of 7.0-10.0. For this, 10 mL of supporting electrolyte was placed in the electrochemical cell and after a total of 10 mm of pencil graphite was immersed into the supporting electrolyte, the cyclic voltammograms were recorded at a scan rate of 50 mV s^{-1} using a potential applied profile with two-stop crossing, in which the potential was initially scanned from +0.1 V to +0.8 V, followed by +0.8 V to -1.0 V, and finally from -1.0 to +0.1 V. Then, the cyclic voltammograms of 1×10^{-4} M NA were recorded under the same conditions after the required volume of NA solution had been added to the cell. Similar experiments were repeated for the GCE. Highly pure argon was purged from the supporting electrolyte for 5 min before all electrochemical experiments.

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