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Voltammetric determination of cefpirome at multiwalled carbon nanotube modified glassy carbon sensor based electrode in bulk form and pharmaceutical formulation

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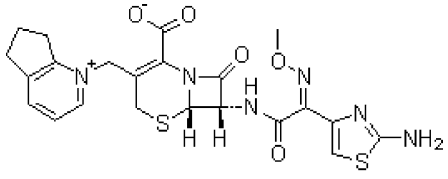
ABSTRACT

A new, simple and low cost voltammetric method for the determination of cefpirome in pharmaceutical preparations has been developed using multiwalled carbon nanotube modified glassy carbon electrode (MWCNT), which showed stable response with enhanced selectivity and sensitivity over the bare glassy carbon electrode. A multiwalled carbon nanotube (MWCNT) modified glassy carbon electrode (GCE) is used for the simultaneous determination of cefpirome by differential pulse voltammetry and square wave voltammetry. Results indicated that cathodic peak of cefpirome is greatly improved at MWCNT modified GC electrode as compared with the bare GC electrode showing excellent electrocatalytic activity towards cefpirome reduction. Linear calibration curves are obtained over the concentration range 100–600 $\mu g\,mL^{-1}$ in Britton Robinson buffer at pH 4.51 with limit of detection (LOD) and limit of quantification (LOQ) are 0.647 $\mu g\,mL^{-1}$ and 2.159 $\mu g\,mL^{-1}$ using SWV and 5.540 $\mu g\,mL^{-1}$ and 18.489 $\mu g\,mL^{-1}$ using DPV, respectively. The described method is rapid and can be successfully applied for the determination of cefpirome in bulk form and pharmaceutical formulations.

1. Introduction

Cefpirome is a new C-3′ quaternary ammonium cephalosporin (A) has been classified as a fourth generation cephalosporin. It is highly active against both gram negative organisms including *Pseudomonas aeruginosa* and gram positive organisms including *staphylococci*. Cefpirome could be effectively used for the treatment of upper and lower urinary tract; lower respiratory tract, skin and soft tissue infections [1,2].



[A]

There are few analytical methods for the identification and quantification of the cefpirome and their metabolites in bulk form, pharmaceutical formulation and biological fluids. These methods include HPLC [3], UV-spectrometry and potentiometry [4,5]. Although spectroscopic and chromatographic methods are widely used for the analysis of various pharmaceutical drugs, most of these methods require separation and pretreatment steps. Electrochemistry has many advantages making it an appealing choice for pharmaceutical analysis. Electrochemical techniques have excellent detection limits with a wide dynamic range and are likely to get preference when low analyte concentrations, small sample volumes or complex sample is to be analyzed.

Electrode surface modification is a field of paramount importance in the modern electrochemistry especially due to the various application possibilities of modified electrodes [6–25]. The interest in developing electrochemical-sensing devices for use in clinical assays is growing rapidly. MWCNTs are now used extensively in the fabrication of novel nanostructure electrochemical sensors. MWCNT-modified electrodes have many advantages over other forms of carbon electrodes due to their small size, high electrical and thermal conductivity, high chemical stability, high mechanical strength and high specific surface area which make them very promising candidates in a wide range of applications. As MWCNTs have the ability to promote electron-transfer reactions and they possess a high electrochemically accessible surface area they can be used as a support material for various catalysts.

In the present work, a MWCNTs film-coated GCE has been developed and tested to take advantage of the convenience of electrochemical methods and characteristics of MWCNTs. The results showed that the sensitivity for the determination of cef-

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pirome using the MWCNTs film coated GCE increased markedly. Some experimental conditions were optimized and a voltammetric method for determination of cefpirome in bulk form and pharmaceutical formulation in the presence of surfactant without any time-consuming extraction or separation steps prior to analysis has been developed.

2. Experimental

2.1. Reagents and materials

Cefpirome was generously provided by Lupin Pharmaceutical Company, Mumbai, India. Injection vials containing cefpirome (*Cef – 4*) labeled 1.0 g cefpirome were obtained from commercial sources. Multiwalled carbon nanotubes with a 95% purity, o.d. = 10–20 nm, i.d. = 5–10 nm and 0.5–50 μm tube length were obtained from Aldrich (USA). For the preparation of standard cefpirome stock solution (10 mg mL^{-1}), 500 mg cefpirome was accurately weighed, dissolved in 1.0% cetyltrimethylammonium bromide and then adjusted to 50 mL with the same surfactant to give the appropriate concentration. Standard working solutions were prepared by appropriate dilutions of the stock solution. Britton Robinson buffers in the pH range 2.0–12.0 were prepared in distilled water by adding suitable amounts of 0.4 M NaOH solution (basic solution) to a stock solution composed of a mixture of 2.14 mL phosphoric acid, 2.472 g boric acid and 2.3 mL of glacial acetic acid (acidic solution). The ionic strength was kept constant by adjusting with 1.0 M KCl. All reagents and solvents were of analytical reagent grade quality and were obtained from Aldrich. The square wave and differential pulse voltammograms were then recorded. The contents of the drug in pharmaceutical preparations were determined using a calibration curve.

2.2. Instrumentation

The voltammetric experiments were performed using μ-autolab type III Potentiostat/Galvanostat (Utrecht, The Netherlands) PGSTAT, Metohm 663 VA stand as electrochemical cell, fitted with a PC provided the appropriate GPES 4.2 software. A three electrode system composed of modified glassy carbon as working, Ag/AgCl as reference and Pt wire as auxiliary electrode was used. Coulometric experiments were performed in the potentiostatic mode using Pt foil with large surface area as working electrode and Pt wire as counter electrode. All solutions examined by electrochemical techniques were purged for 5 min with purified nitrogen gas. All pH-metric measurements were made on a Decible DB-1011 digital pH meter fitted with a glass electrode and a saturated calomel electrode as reference.

2.3. Preparation of MWCNT modified glassy carbon electrode

The GCE was polished on a polishing micro-cloth with 0.5 μM alumina powder then rinsed and sonicated for 5 min in an ultrasonic bath. The electrode was then transferred to the supporting electrolyte and potential in the range of −0.1 to −1.2 V was applied in a regime of cyclic voltammetry for 20 cycles until a stable voltammogram was achieved. The glassy carbon electrode surface was coated with 10 μL MWCNT suspension solution and the solvent was evaporated at room temperature in vacuum drying [26].

2.4. Characterization of the modified electrode

Surface morphological studies of the modified electrode were carried out by scanning electron microscopy (SEM) using Philips SCI quanta 400 instrument. Scanning electron microscopy image of the MWCNTs on the GCE surface is shown in Fig. 1. It can be seen

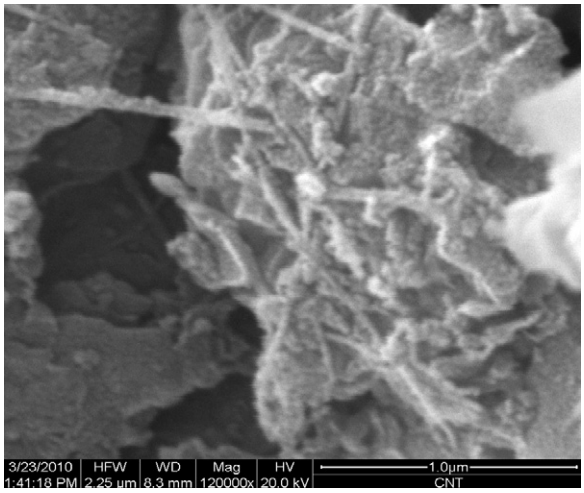

Fig. 1. The scanning electron micrograph of MWCNT-GCE.

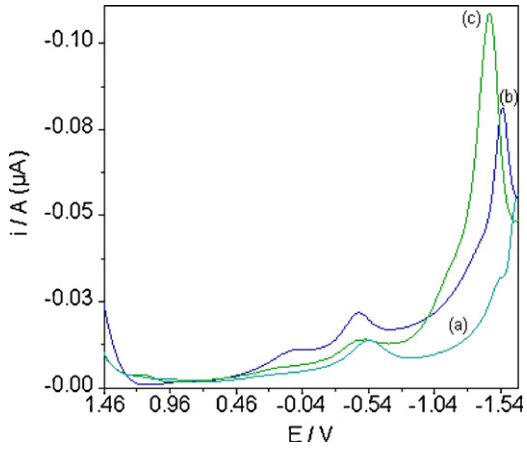

Fig. 2. Differential pulse voltammograms of 500 μg mL^{-1} cefpirome at (a) blank, (b) bare glassy carbon electrode and (c) MWCNT-GCE.

that the MWCNTs were seen in the form of tubes some of which twisted together.

3. Results and discussion

The electrochemical behaviour of cefpirome on multiwalled carbon nanotube modified glassy electrode (MWCNT/GC) was studied by using square wave voltammetry (SWV), differential pulse voltammetry (DPV) and cyclic voltammetry (CV). In all electrochemical methods cefpirome gave one well defined reduction peak in cetyltrimethylammonium bromide (CTAB), which is attributed to the reduction of exocyclic –C=N– bond.

3.1. Comparison of square wave voltammetric behaviour of cefpirome at GCE and MWCNT film-coated GCE

On comparing the voltammetric behaviour of cefpirome in GCE and MWCNTs film-coated GCE, it is observed that cefpirome shows substantial increase in peak current and the limit of detection is also found to be lower in MWCNTs film-coated GCE (Fig. 2).

3.2. Effect of pH and scan rate

Differential pulse (DP) and square wave (SW) voltammetry studies were carried out to investigate the influence of solution pH