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DIFFERENTIAL PULSE VOLTAMMETRIC DETERMINATION OF CINITAPRIDE USING CARBON PASTE ELECTRODE

D. Sri Vidya, G. Chandra Sekhar*, V. Prabhakara Rao, K. Aruna

*Electroanalytical Lab, Department of Chemistry, Govt. degree & P.G college, Puttur, Chittoor (Dt), Andhra Pradesh, India

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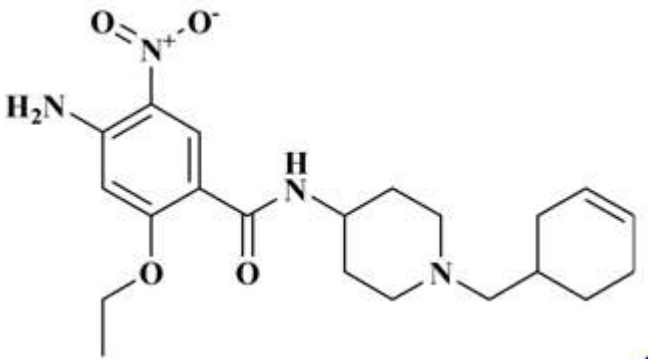
ABSTRACT

The voltammetric performance of cinitapride (CNTP) was investigated at carbon paste electrode using differential pulse voltammetry and cyclic voltammetry. The drug under study exhibited a single, well-defined reduction peak owing to the reduction of NO_2. The electrode and reaction conditions which yielded maximum peak current were established using differential pulse voltammetry. A linear relationship was observed between the peak current and the concentration of CNTP over the range $2.35x10^{-6}$ M to $4.25x10^{-4}$M. The limits of detection and limits of quantitation were found to be $6.55x10^{-9}$ M to $7.42x10^{-8}$M respectively. The proposed method was successfully applied for the determination of CNTP in pharmaceutical formulations without interference from excipients

KEYWORDS: cinitapride; differential pulse voltammetry; cyclic voltammetry; Carbon paste electrodes.

I. INTRODUCTION

Cinitapride (CNTP) (Fig.1) is chemically, 4-Amino-N-[1-(3-cyclohexen-1-ylmethyl)-4- piperidinyl]-2-ethoxy-5-nitrobenzamide and a gastroprokinetic drug[1]. Regarding cinitapride, the determination of this drug can be accomplished using spectrophotometric methods[2]. Previously few methods are reported such as spectrophotometric[3,4] and HPLC[5] methods for determination of cinitapride in pharmaceutical dosage forms. By keeping all this in view, it was thorough worthwhile to develop stability indicating HPLC method**.** Literature survey reveals some chromatographic and polarographic methods have been used for the determination of CNTP[6-10]. Roy et al. reported RP-HPLC Method for the Determination of Cinitapride in the Presence of its Degradation Products in Bulk Drug[11]. Hitesh et al. Development and validation of HPTLC method for simultaneous estimation of cinitapride and omeprazole in combined dosage form[12].



Cinitapride (CNTP) (Figure.1)

Carbon paste electrodes (CPEs) belong to promising electrochemical or bioelectrochemical sensors of wide applicability. In 2008, it was exactly a half century since Ralph Norman Adams from the University of Kansas published a short one-page report[13]. The base of modified carbon pastes is usually a mixture of powdered graphite and nonelectrolytic binder[14-16]. This work aimed to study the reduction of CNTP at carbon paste

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electrode using differential pulse voltammetry and cyclic voltammetric methods and a procedure for the determination of the drug in its pharmaceutical formulation and human was optimized.

II. EXPERIMENTAL

Apparatus

Electrochemical studies were carried out by Autolab PG STAT101 supplied by Metrohm Autolab B.V. The Netherlands. A three electrode system comprising of a glassy carbon electrode modified with polyaniline and multi walled carbon nanotubes (PAN/MWCNT/GCE) as a working electrode. Glassy carbon electrode (GCE-3mm) obtained from Metrohm India Ltd., Chennai. Saturated Ag/AgCl/KCl as a reference electrode and Pt wire as a counter electrode. Electrode surface morphology study was carried out by SEM instrument model OXFORD instrument INCA PENTA FETX3 CARL ZEISS from Japan. An Elico LI-120 pH meter supplied by Elico LTD, Hyderabad, India was used to determine the pH of the buffer solution.

Reagents

Cinitapride were prepared by dissolving methanol and distilled water (from Sigma Mumbai), respectively. Although the stability of the solutions was checked, in this work solutions were freshly prepared and kept in the dark. Solutions of Britton-Robinson (BR) buffer, 0.1 M in acetic, phosphoric and boric acid were prepared from analytical reagents at pH range 2-10 by adding the corresponding amount of NaOH. A solution of 10% KNO_3 in concentrated H_2SO_4 was also prepared.

Procedure

For the voltammetric determination of CNTP, the following procedure was proposed: to 3 ml of standard CNTP solution in the 4.25×10^{-5} M to 1.65×10^{-3} M range was added 5 ml of the nitrating mixture. After 10 min, distilled water was added to make the volume up to 50 ml. A stream of nitrogen was bubbled for 5 min and the voltammograms are recorded at a pulse amplitude of -50 mV, with a scan rate of -50mV/ s.

Preparation of carbon paste electrode

The carbon paste was prepared by thoroughly mixing 5 g of graphite powder with 1.8 ml of paraffin oil in a mortar with pestle. The carbon paste was packed into the hole of the electrode body and smoothed on a clean paper until it had a shiny appearance. The electrode body was constructed by pressing a small rode of stainless steel (diameter 3mm) inside a micropipette tip (1 ml volume capacity) leaving a depression at the surface tip approximately 1 mm for housing the carbon paste, and a thin wire was inserted through the opposite end to establish electrical contact. The carbon paste electrode was immersed in the supporting electrolyte placed in the cell and several sweeps were applied to obtain a low background current [17].

III. RESULTS AND DISCUSSION

Cyclic voltammetric studies

Fig. 1 shows the cyclic voltammograms of 4.25×10^{-5}M CNTP in 0.04 M BR buffer pH 2.0, scan rate -50 mVs-1**,** and accumulation potential 0V. Reduction peak appears at -0.3V, which may be due to reduction of nitro group of the drug, and no oxidation peak is observed in the anodic branch which suggests that the process is irreversible. The cycle 'a' shows the blank solution and cycle 'b'' shows the 4.25×10^{-5}M of CNTP at carbon paste electrode. It shows that the peak current increases in the second cycles, and this behaviour gives an indication of an adsorption character. A plot of logarithm of peak current versus logarithm of the scan rate gave a straight line relation with a slope of 0.72 which is close to the theoretically 1.0 for an ideal relation of surface species The peak potential shifted to more positive values with increasing scan rate.

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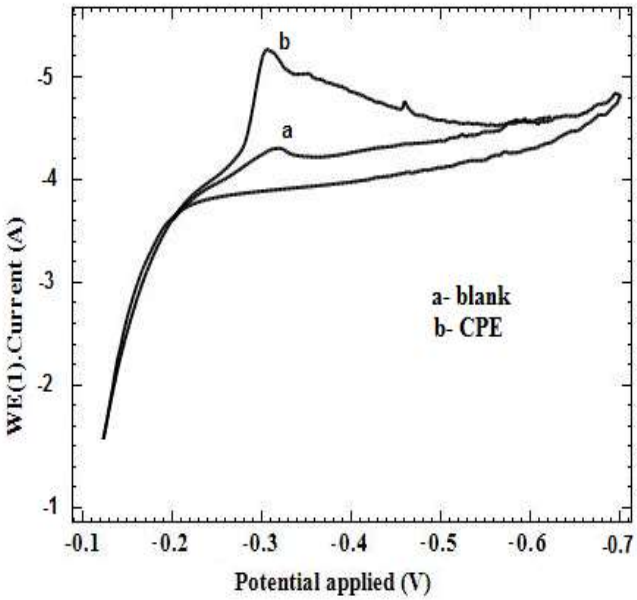

Figure1. Typical cyclic voltammograms of 4.25×10^{-5} M CNTP (a) without CNTP solution in 0.04 M BR buffer pH 2.0 and scan rate of -50 mVs-1

Effect of scan rate

The influence of the scan rate was investigated. The results suggested that differential pulse voltammetric peak current reached maximum value when the scan rate was -50 mV. As for the scan rate; the current response with increasing the scan rate of -25mVs^{-1} to -100 mVs^{-1} gave the maximum response (Fig 5). Accordingly, the optimum conditions for recording a maximum developed and sharper peak for CNTP are t_{acc}: 60 sec., E_{acc}: -0.4, scan rate: 50 mVs^{-1} and pulse amplitude: 50 mV, optimum temperature: 25^{o}C.

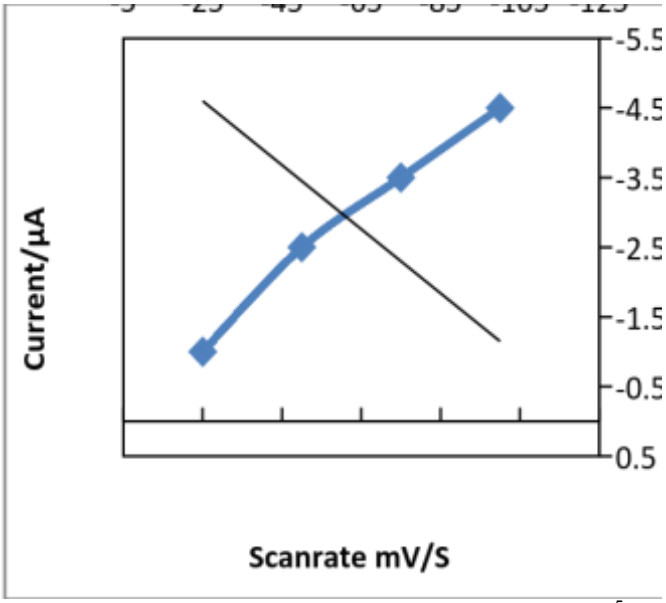

Figure 2. Effect of scan rate on CNTP at pH 2.0 in PB solution at concentration 2.55×10^{-5}M at different scan rates: (a) -25mV/s (b) -50mV/s (c) -75mV/s (d) -100V/s (e) -125mV/s

Effect of pH

The effect of pH, operating with 4.25×10^{-5} M CNTP and adjusting the pH between 2.0-10.0 with B-R buffer was studied. The results showed in Fig. 3, it show that the peak potential varies linearly with pH between 2.0 to

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10.0, with a slope of 0.752mV/pH, close to the theoretical value of 0.682 mV/pH for a reduction process in which the same number of protons as that of electrons is involved. That process, responsible for this behaviour, must be the reduction of the nitro group in the CNTP molecule and the maximum peak was observed at pH 2.0.

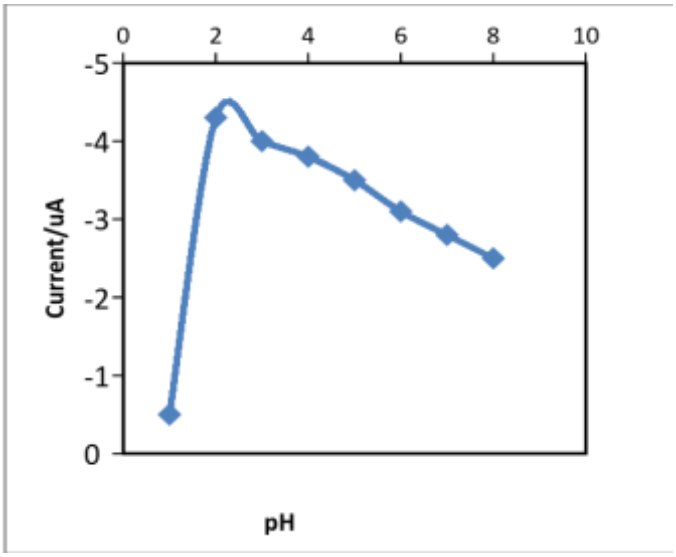

Figure 3.The plot the of current vs pH of CNTP in BR buffer solution, concentration: 4.25×10^{-5}M at glassy carbon electrode in different pH values

Differential pulse voltammetric studies

An accurate volume of the clear supernatant liquor was transferred into the electrochemical cell containing 10 mL of BR buffer of pH 2.0 to yield a final concentration of approximately 4.25×10^{-5}M of CNTP. The DPV was recorded under the optimum experimental conditions at carbon paste electrode. A typical differential pulse voltammogram recorded in various concentration ranges from 4.25×10^{-5}M to 1.65×10^{-3}M at GCE modified with polyaniline and multi walled carbon nanotubes. Fig.6 depicts the differential pulse voltammograms of CNTP in different concentrations.

Calibration graph, limit of detection and limit of quantitation

Calibration curves for standard drug solution under the optimized parameters were obtained. A linear relationship was observed between 4.25×10^{-5} M to 1.65×10^{-3} M of CNTP. Fig. 6 represents the differential pulse voltammograms recorded using the standard addition method. The linear regression equation was I (nA) = 15.32+1.53×10^{-7}C mol/l with a correlation coefficient of 0.962, the limit of detection (LOD = 3(sd)/b) and limit of quantitation (LOQ = 10(sd)/b), were calculated, where sd is the standard deviation of the intercept and b is the slope of the calibration graph. LOD and LOQ were found to be 6.55x10^{-9} and 4.35x10^{-8} CNTP, respectively. The analytical parameters for the calibration graph are summarized in Table 1.

Pharmaceutical samples			
Added(M)	Found(M)	*Recovery (%)	RSD
5.5x10^{-7}	5.45x10^{-7}	99.5	1.45
4.5x10^{-6}	4.35x10^{-6}	98.3	1.52
3.5x10^{-5}	3.33x10^{-5}	98.2	1.85
2x10^{-4}	1.97x10^{-4}	97.5	2.0
1x10^{-4}	0.97x10^{-4}	97.3	2.10
Urine and Serum Samples			
Sample	Spiked (10^{-6} M)	Detected (10^{-6} M)	*Recovery (%)
Urine Sample-1	0.5	0.48	98.0
Urine Sample-2	1.0	0.98	98.4
Serum Sample-1	0.5	0.49	98.3
Serum Sample-2	1.0	0.99	99.0

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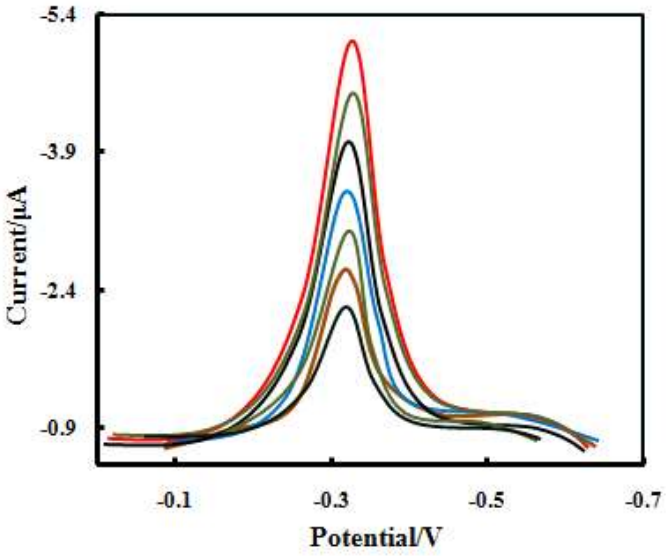

Figure 4. Typical DPV of CNTP in BR buffer solution pH 2.0, concentration: 4.25×10^{-5}M to 1.35×10^{-3}M at Carbon paste electrode

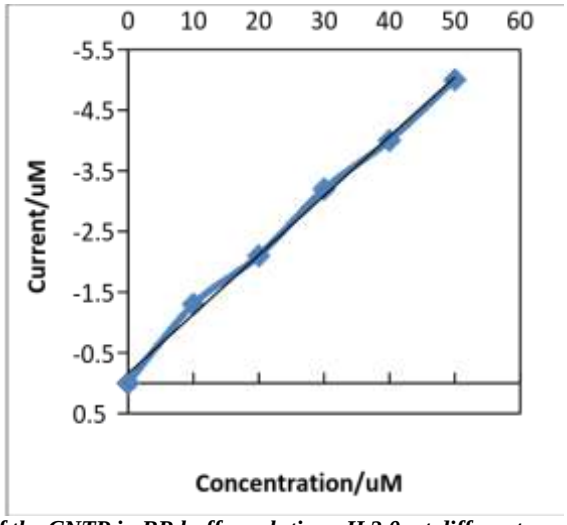

Figure 5. Calibration plot of the CNTP in BR buffer solution pH 2.0, at different concentrations at Carbon paste electrode

IV. CONCLUSIONS

In this paper, the electrochemical behaviour of CNTP on carbon paste electrodes has been investigated by cyclic and differential pulse voltammetric techniques. The proposed procedure showed clear advantages, such as no pre-treatment or time consuming extractions steps were required prior to the analysis, ease of preparation and easy renewable of the electrode surface. The proposed method is less expensive than alternative techniques like HPLC, and hence can be applied to the routine determination of the drug in quality control laboratories

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