



# Sensitive determination of domperidone in biological fluids using a conductive polymer modified glassy carbon electrode



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## ABSTRACT

A simple and sensitive method for domperidone (DP) determination has been developed by electropolymerizing a polymer film on the surface of glassy carbon electrode (GCE) in acidic solution using cyclic voltammetry. The modified sensor was characterized by Field Emission Scanning Electron Microscopy (FE-SEM) and Electrochemical Impedance Spectroscopy (EIS). The electrochemical measurements were carried out using square wave voltammetry and cyclic voltammetry. The modified sensor exhibited an excellent catalytic response towards the oxidation of DP with a well-defined oxidation peak at 840 mV. The modified sensor exhibited linear calibration curve for DP over a concentration range of 0.1  $\mu\text{M}$  to 100  $\mu\text{M}$  in phosphate buffer solution of pH 7.2 with detection limit of 12.0 nM. The sensor was capable to determine DP effectively without any interference from the common metabolites like ascorbic acid, uric acid, xanthine and hypoxanthine. The analytical utilities of the sensor have been demonstrated by determining the DP in human fluids and pharmaceutical samples. Further, the modified sensor displayed voltammetric responses with high sensitivity, good selectivity and reproducibility which make it suitable for clinical diagnosis.

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## 1. Introduction

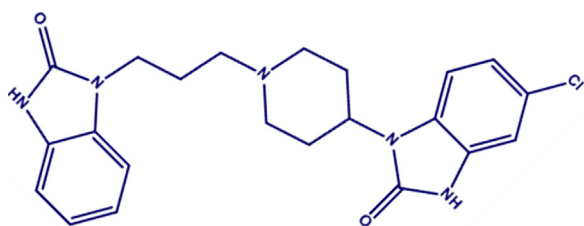
Domperidone, 5-chloro-1-(1-[3-(2,3-dihydro-2-oxo-1H-benzimidazol-1-yl) propyl]-4-piperidinyl)-1,3-dihydro-2H-benzimidazol-2-one, (DP, I) is a dopamine antagonist, which acts as an antiemetic and gastroprokinetic agent [1,2]. As a gastroprokinetic agent, DP facilitates gastric emptying and decreases small bowel transit time by increasing esophageal and gastric peristalsis and by lowering esophageal sphincter pressure. As an antiemetic, it regulates nausea and vomiting by blocking the dopamine receptors at both the chemoreceptor trigger zone and at the gastric level [1,3,4]. Due to its high molecular weight and lower lipid solubility; DP cannot easily cross the blood brain barrier, hence, it can block effectively the peripheral side effects of dopamine agonist agents used in the treatment of Parkinson's disease. DP is effective in improving the symptoms of gastroesophageal reflux, as well as gastrointestinal motility abnormalities [5–9]. DP increases the secretion of prolactin by the pituitary gland as a side effect to its normal uses and thereby augmenting milk supply in the mother's breast. As it cannot easily cross the breast milk and blood-brain barrier or crosses to a very lesser extent, hence the amount of DP

into the milk is insignificant therefore, the side effects of domperidone in the baby are not expected [10–13]. The determination of DP in biological systems has been considered as useful indicator of problem related to hyperprolactinemia, arrhythmias, sudden death and cardiac arrest, hence, the detection of DP concentration in body fluids is considered as of great significance in the field of clinical diagnostics [3,4] Scheme 1.

Conductive polymers have drawn considerable attention during the past decade due to their potential applications in solar cells, energy storage, light-emitting diodes, field-emission and electrochromic displays, conducting adhesives, catalysis, chemical and biosensors [14,15]. Conducting polymers have emerged as one of the most interesting materials for the fabrication of electrochemical sensors as they have many advantages, such as possibility of one step synthesis on different substrates with excellent stability, provide better signal transduction, enhanced sensitivity, reproducibility, biocompatibility and low cost. The better chemical stability of the polymeric film, high sensitivity, more active sites, homogeneity and strong adherence to electrode surface, make conductive polymers a most promising and interesting material for the modification of the sensors [16–19]. Conducting polymers received additional interest due to their combined properties of organic polymer and electronic properties of semiconductors. The great advantage of conducting polymer modified sensors is that the conducting polymers have the potential to exhibit improved response properties and are sensitive

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Scheme 1. Structure of DP.

to small perturbations. Among the different available methods to immobilize a polymer film on the electrode surface, electropolymerization has been demonstrated to be the most convenient because of simplicity and uniform film thickness can be achieved easily by controlling the electrochemical parameters during the electrochemical process [20–24].

Several techniques have been reported for the determination of DP in the last decade, such as high performance liquid chromatography (HPLC) with mass spectrometry, reverse phase HPLC, liquid chromatography-mass spectrometry and UV-spectrophotometric methods [25–36]. As these methods are rather sophisticated, expensive and require time consuming methods prior to analysis and also need organic solvents for separation, few attempts have also been made to use electroanalytical technique for the determination of DP [2,37,38]. As these techniques are less expensive, accurate, convenient and provide quick analysis of various samples they have attracted attention of researchers. In addition, electroanalytical techniques are also considered as ecofriendly [16,39,40].

In this study, we have carried out the electropolymerization of (4-amino-3-hydroxynaphthalene sulfonic acid, AHNSA) on the surface of GCE [41] for the sensitive determination of DP. It was found that the p-(AHNSA)/GCE shows an excellent electrocatalytic activity towards the oxidation of DP. The response of DP in AHNSA polymer was better than other monomers such as melamine, pyrrole etc due to the presence of OH, NH<sub>2</sub> and SO<sub>3</sub>H groups. The quantitative determination of DP in human fluids and pharmacological formulations using the optimized conditions has been carried out and a low detection limit for DP is obtained.

## 2. Experimental

### 2.1. Instrumentation

All electrochemical experiments were performed using Bio-analytical system (BAS, West Lafayette, USA) Epsilon EC-USB voltammetric analyzer equipped with three electrodes in a single compartment glass cell. Glassy carbon electrode (GCE, 3 mm dia.) was used as a working electrode, an Ag/AgCl (3 M NaCl) (BAS Model MF-2052 RB- 5B) and Pt wire were used as reference and counter electrodes, respectively. The pH measurement of buffer solutions was performed using Thermo Fisher Scientific, Singapore Digital pH meter (Eutech Instruments, model pH 700). The characterization of surface morphology of bare and modified sensor was carried out using Field Emission Scanning Electron Microscopy (FE-SEM, model; Zeiss ultra plus 55). Electrochemical impedance spectroscopy (EIS) was performed using a galvanostat (model; Versastat 3, PAR).

### 2.2. Chemicals and reagents

Domperidone, AHNSA, ascorbic acid, uric acid, xanthine and hypoxanthine were purchased from Sigma-Aldrich (USA) and used as received without further purification. All the chemicals used to prepare phosphate buffers in the pH range 2.4–10.0 were obtained from E. Merck (India) Ltd. Mumbai. Phosphate buffers of ionic strength (1.0 M) were prepared according to the reported method

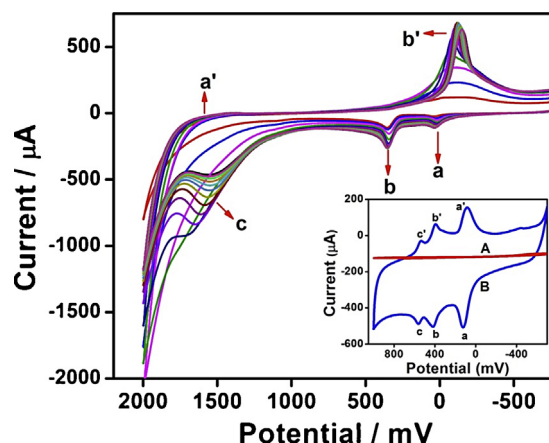


Fig. 1. A series of 15 consecutive cyclic voltammograms showing polymer growth at GCE surface. The electropolymerization was carried out in 0.1 M HNO<sub>3</sub> containing 2 mM AHNSA monomer at scan rate of 0.1 V s<sup>-1</sup>; inset showing cyclic voltammogram of (A) bare GCE and (B) p-(AHNSA)/GCE in 0.5 M H<sub>2</sub>SO<sub>4</sub>.

[42]. Biological samples of human serum and urine were collected from the Institute hospital, I.I.T. Roorkee, after the permission of Ethical Clearance Committee of IIT Roorkee and were used after suitable dilution. All other chemicals and solvents used in the experiment were of analytical grade and double distilled water was used throughout the experiments.

### 2.3. Fabrication of polymeric film

For the fabrication of polymer film of AHNSA on GCE, the surface of the glassy carbon was first polished with a paste of alumina powder (grade I) and ZnO on micro cloth pad to a mirror like finish surface and then it was rinsed with distilled water. The electropolymerization of AHNSA was carried out on the surface of GCE in a solution containing 2 mM of AHNSA in 0.1 M nitric acid solution. The cyclic voltammograms were recorded by scanning the potentials between -0.8 V and +2.0 V at a scan rate of 100 mV s<sup>-1</sup> for 15 scans [41]. The film obtained was rinsed with double distilled water. In the next step modified electrode was cycled between -1.0 and +1.0 V at a scan rate of 100 mV s<sup>-1</sup> in 0.5 M H<sub>2</sub>SO<sub>4</sub> until stable voltammograms were obtained.

### 2.4. Experimental procedure

The stock solution of DP (1 mM) was prepared by dissolving the required amount in the minimum amount of methanol (1.5 ml) and then solution was made to 25 ml with double distilled water. For recording the voltammograms, required amount of this solution was added to the cell containing 2 ml of buffer and total volume was made to 4 ml using double distilled water. Solutions were purged with high purity nitrogen for 15 min to remove oxygen before recording each voltammogram. Phosphate buffer of pH 7.2 was used as supporting electrolyte during the experiments. The optimized operating conditions for square wave voltammetry (SWV) were initial potential (*E*): 400 mV, final potential (*E*): 1200 mV, square wave frequency (*f*): 15 Hz, square wave amplitude (*E*<sub>sw</sub>): 25 mV and potential step (*E*) 4 mV. The step potential was increased in the range 2 to 40 mV, the peak current increased and became constant after 20 mV. In the case of pulse height study (5 to 100 mV), no significant increase was observed in the peak current of DP but peak shape started to broaden >25 mV. The surface of the modified sensor was cleaned after each run using a time base technique by applying a constant potential (-500 mV) for 60 s in buffer. All the potentials reported are with respect to Ag/AgCl electrode at an ambient temperature of 25 ± 2 °C.

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