



Simultaneous voltammetric analysis of anti-ulcer and D₂-antagonist agents in binary mixture using redox sensor and their determination in human serum



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ABSTRACT

Pencil graphite electrode was successfully modified with a thin film of poly (eriochrome black T) and applied for the sensitive and selective voltammetric simultaneous determination of pantoprazole sodium and domperidone in a binary mixture. The preparation and basic electrochemical behavior of poly (eriochrome black T) film on the Pencil graphite electrode were investigated. The modified electrode has exhibited very high electro-catalytic activity towards the cited mixture. The anodic peaks of the both species were well defined with enhanced oxidation peak currents. Under the optimum conditions, the linearity ranges were $0.4\text{--}55 \times 10^{-7}$ M and $0.1\text{--}34 \times 10^{-7}$ M for pantoprazole sodium and domperidone, respectively with detection limits of 0.12×10^{-7} M and 0.04×10^{-7} M for pantoprazole sodium and domperidone, respectively. The proposed sensor has been successfully applied in the analysis of pantoprazole sodium and domperidone in synthetic binary mixtures and human serum.

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1. Introduction

Pantoprazole sodium (PAN sodium) is occasionally prescribed with the pro-kinetic drug, domperidone (DOM) (Fig. 1) in binary mixtures. PAN sodium is used to treat peptic ulcers or stomach ulcers via irreversible inhibition of (H^+/K^+)-ATPase enzyme in parietal cells of gastrointestinal tract [1] while DOM prevents accumulation of food in gastrointestinal tract and emesis by blocking dopaminergic receptor in the brain (D_2 -receptor antagonist) [2]. Sometimes, they are administered separately and commonly administered in binary mixture form in capsules. These capsules are commercially available in Indian and European drug markets, e.g. Pan-D[®] and Topdom[®] tablets. A survey of analytical literature concerning the determination of that particular mixture has been done. The methods of its determination include UV-spectrophotometric methods [3,4], spectro-densitometry [5–7], HPLC methods [6,8,9] while these drugs were separately determined using electrochemical methods [10–19]. Polymer films have attracted great attentions due to their good stability, biocompatibility, homogeneity and strong adherence to electrode surface [20,21]. Since the thickness, permeation and charge transport of polymeric films can be controlled by adjusting the electrochemical parameters, polymer modified electrodes have the advantages of improving electro-catalysis, absence of surface fouling and prevention of undesirable reactions competing

kinetically with the desired electrode process [22–24]. It has been demonstrated that polymer modified electrodes, especially those coated with dyes and dyestuffs show excellent stability, reproducibility and homogeneity [25–27]. Most of the redox dyes are artificial electron donors [28,29], and they are able to undergo electro-polymerization to generate stable redox active layers [30].

In this study, the electro-analytical applicability and performance of poly-eriochrome black T as a modifier for pencil graphite electrode (PGE) in the simultaneous determination of PAN sodium and DOM were conducted. Pencil graphite electrode (PGE) was simply modified with a uniform film of electro-active poly-eriochrome black T by means of fast and reproducible electro-polymerization method. The poly-eriochrome black T modified PGE (poly (EBT)/PGE) has exhibited very highly improved electro-chemical responses and allowed sensitive simultaneous determination of the mixture. To the best of our knowledge, this paper is the first report on the simultaneous voltammetric determination of PAN sodium and DOM in a mixture and human serum using modified PGE as a redox sensor.

2. Experimental

2.1. Pharmaceuticals

PAN sodium was supplied as a gift from Sigma, Quesna, El-Menoufia, Egypt. Domperidone was supplied as a gift from 10th Ramadan city, El-

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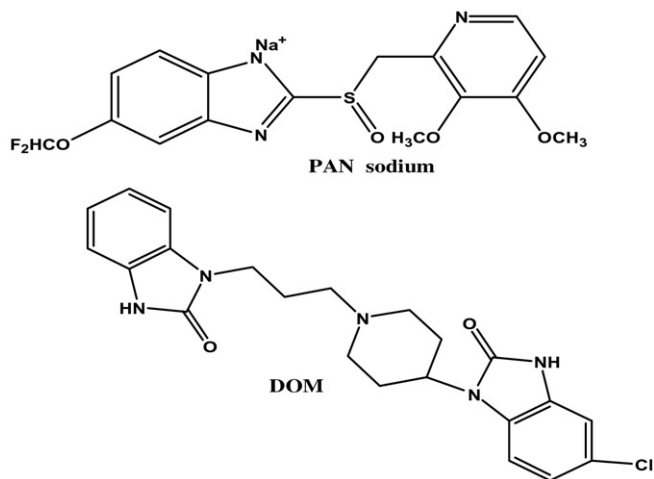


Fig. 1. Chemical structures of PAN sodium and DOM.

Sharqua, Egypt. Aceclofenac, tinidazole, clarithromycin were obtained as gifts from NODCAR, El-Giza, Egypt. Doxycycline was supplied as a gift from CID, Assiut, Egypt.

2.2. Reagents and solvents

Methanol, eriochrome black T, sodium hydroxide, glacial acetic acid, phosphoric acid, sodium acetate, ascorbic acid, ferric chloride, nickel sulphate, copper sulphate, potassium ferricyanide, potassium chloride were purchased from El Nasr Pharmaceutical Chemicals Co., Egypt. Boric acid was purchased from El-Gomhouria Co., Egypt. Uric acid and dopamine were supplied as a gift from department of biochemistry, Assiut University, Assiut, Egypt.

Britton-Robinson buffer (B.R.) as a supporting electrolyte (equal volumes of 0.04 M acetic acid, 0.04 M phosphoric acid and 0.04 M sodium acetate, adjusted to a desired pH by 2 N NaOH).

2.3. Instrumentation

A Princeton VersaSTAT MC (VersaSTAT 3, Model RE-1, Princeton Applied Research, AMETEK, USA) connected to a three-electrode cell was used for the electrochemical measurements. In all measurements, the reference electrode was an Ag/AgCl (3 M KCl), the auxiliary electrode was a platinum wire and PGE as the working electrode. A Pentel pencil, Model P205 (Japan), was used as a holder for the pencil lead. Electrical contact with the lead was achieved by soldering a metallic wire to the metallic part that holds the lead in place inside the pencil. Unless stated otherwise, the pencil was fixed so that about 3 mm of its length is immersed into the solution. Measurements were performed in a 10-mL glass cell containing 6 mL of supporting electrolyte solution. Stirring was achieved with a magnetic stirring bar.

The pH values of solutions were measured using Hanna pH meter (Hanna Instruments Brazil, Saˆo Paulo, Brazil) with a combined electrode. The solutions were sonicated using Branson ultrasonic cleaner, Branson UL Transonics Corporation, Eagle Road, Danbury, CT 06813, USA. Surface morphology studies of the modified electrode were carried out using scanning electron microscope (SEM), JEOL JSM-5400 LV instrument (Oxford, USA). FTIR-studies were carried at faculty of science, Assiut University, Assiut, Egypt.

2.4. Preparation of standard solutions

An accurately weighed amount of 15 mg PAN sodium and 10 mg DOM was transferred into a 100-mL calibrated flask, and dissolved in

about 10-mL methanol. The solution was dissolved in sonicator until clear solution obtained then, completed to the mark with methanol to provide a stock solution containing 3.7×10^{-4} M and 2.3×10^{-4} M for PAN sodium and DOM, respectively. The working standard solutions were prepared by further dilution of the suitable aliquots of the stock solution with Britton-Robinson buffer (pH = 4.0).

2.5. Electro-polymerization of EBT

New PGE was polished on white paper surface until shiny appearance. Then, the electrode was submerged in an NaOH solution (0.1 M) containing EBT (0.1 mM), and the electro-polymerization process was carried out by the positive-going cyclic voltammetry (6 cycles) in the potential range of -0.2 – 0.8 V with a potential scan rate of 100 mV/s. After electro-polymerization, the modified electrode was washed carefully with distilled water and dried in air.

2.6. General procedure

An aliquot of the standard solutions containing PAN sodium and DOM was transferred into the electro-chemical cell containing 6.0 mL of B.R. (pH 4.0). The three-electrode system was placed into the electro-chemical cell and the square wave adsorptive stripping voltammogram (SQWASV) was recorded from -0.2 to 1.4 V. The peak currents were measured for PAN sodium and DOM. Before each measurement, the background voltammogram was recorded. The difference in the background and sample peak currents was considered as the net analytical signal. Cleaning of the modified electrode and providing a fresh surface for the subsequent experiments were simply carried out by five-times potential cycling in the range of -0.3 – 1.4 V in NaOH solution (0.1 M), and subsequently, washing the modified electrode with distilled water to remove any adsorbed species.

2.7. Application of the proposed method

The method was successfully applied for determination of PAN sodium-DOM in synthetic mixtures of different ratios. Also, drug-free human blood plasma was obtained from healthy helpers. In order to remove the serum proteins, acetonitrile (0.75 mL) was added to 1.0 mL of the serum sample and diluted to the mark with B.R. buffer (pH = 4.0) in a 10-mL volumetric flask, and the mixture was centrifuged for 10 min at 5000 rpm. The supernatant was then taken carefully and used for further analysis. The experimental protocol was conducted according to the Egyptian regulations and approved by the Institutional Human

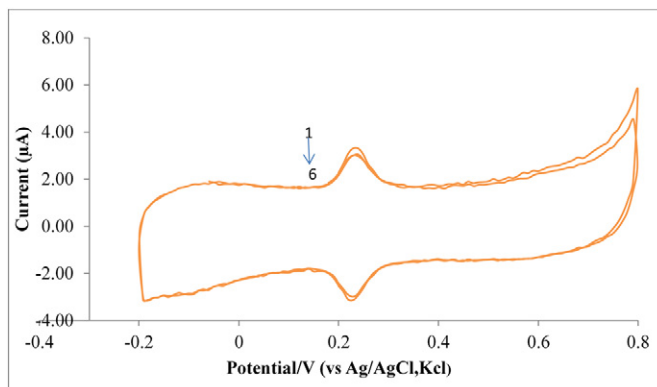


Fig. 2. Successive cyclic voltammograms (6 cycles) for electro-polymerization of EBT on PGE surface. Conditions were 1.0 mM of EBT in 0.1 M NaOH, scan rate of 100 mV/s.

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