



Research paper

Simple and sensitive adsorptive stripping voltammetric assay of granisetron from its dosage form by platinum nanoparticles modified electrodes



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ABSTRACT

Base to the interfacial adsorptive nature of granisetron (GRN) on platinum particles modified glassy carbon electrode (PtPs/GCE). A sensitive analytical method for trace analysis of GRN based on the adsorptive stripping technique has been developed. The application of a square wave mode for the stripping of adsorbed GRN has proved to be much more sensitive compared to differential pulse scan. A study of the variation in the peak current with solution variables such as pH, concentration of granisetron, possible interference, and instrumental variables, such as accumulation time and accumulation potential, has resulted in the optimization of the oxidation signal for analytical purposes. Under the optimized conditions, the square wave peak current showed two linear ranges of GRN concentration: 0.01–0.5 μM and 0.5–25 μM with correlation coefficients of 0.997 and 0.998, respectively. Nanomolar detection limit was obtained as 1.95 nM for the first linear range. Possible interferences by substances usually present in the pharmaceutical formulations were also evaluated. The proposed electrochemical procedure was successfully applied for the determination of GRN in pharmaceutical injection samples (SETRON[®] 3 mg/3 mL) with average recovery of 100.30%.

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1. Introduction

GRN is a selective 5-HT₃ receptor antagonist which is used to prevent nausea and vomiting due to cancer drug treatment and radiation therapy. The chemical structure of GRN is presented as 1-methyl-N-[(1R, 3r, 5S)]-9-methyl-9-azabicyclo [3.3.1] non-3-yl]-1H indazole-3-carboxamide HCl.

The bioavailability, bioequivalence, and pharmacokinetic data have significant role for evaluation and interpretation of drugs and its metabolites. Hence quantitative analysis of these drugs and metabolites has importance. However, their well characterized and fully validated analysis are needed [4]. Analytical methods and instrumentation provide the foundation for all measurements made in a modern clinical chemistry laboratory.

Up to date, various analytical methods have been used for the determination of GRN in bulk and pharmaceutical dosage forms and biological samples. UV spectrophotometry [5,6], liquid chromatography [7–13,18–20], liquid chromatography coupled with tandem mass spectrometry [14–17]. There is one study on GRN with voltammetric methods [21] that was applied with hanging mercury electrode at the potential range –1.3 to –1.5 V. The concentration range of $1.4 \times 10^{-7} \text{ M}$ – $5.7 \times 10^{-7} \text{ M}$ with detection limit $4 \times 10^{-8} \text{ M}$ in pharmaceutical form, whereas in human plasma $1.4 \times 10^{-7} \text{ M}$ – $6.3 \times 10^{-7} \text{ M}$ with detection limit $3 \times 10^{-8} \text{ M}$. Square wave and cyclic voltammetry methods were applied. Solid-phase extraction technique was employed for extraction of granisetron from spiked human plasma. Our study is more sensitive and validate than previous electrochemical study. The published methods were affected from endogenous substances. Mostly, they need some tedious extraction procedure and pre-treatment steps. These steps are involved time consuming preparation and required a sophisticated, expensive equipment.

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So, there is a need to develop simple and original methods for the GRN titration by using electrochemical techniques (potentiometry, amperometry and voltammetry), which have proved their effectiveness in the analytical chemistry. However, the use of conventional electrode materials (C, Pt, and Au) is sometimes not always satisfactory, due to the fouling character of the adsorption [22].

More recently, chemically modified electrodes (CMEs) have attracted much interest in the electrocatalytic oxidation/reduction of important redox systems. Modified electrodes can be prepared by deposition of various compounds such as conducting polymers, metal complexes, transition metals and metal oxides on various electrodes [23–25]. The operation mechanism of such electrodes depends on the properties of the modified materials used to promote selectivity and sensitivity toward the target analytes. This kind of electrode is inexpensive and possesses many advantages such as low background current, wide range of potential windows (in both cathodic and anodic region), easy fabrication, and rapid surface renewal. One of the most important properties of CMEs has been their ability to catalyze the electrode process via significant decrease of the overpotential and increase of the electron transfer kinetics with respect to the unmodified electrode.

Metal nanoparticles have drawn renewed attention in recent years due to their wide range of applications in nanosensor, biosensors and many other fields [1–3]. They have special properties such as large surface area, catalyst, mechanical and electronics. They are a link between larger materials and molecular or atomic structures. Platinum particle (PtPs) applications are successful at electronics, optics, catalysts, and enzyme immobilization fields.

The previous studies indicated that, Pt nanoparticles could increase the surface area and conducive to electron transfer with strong catalytic properties, and have attracted more and more interests as electrode modified materials [26,27]. For example, Wang and Qiu [28] found that, the Pt nanoparticles deposited on the chrome substrate could improve the electrochemical properties of the substrate material and exhibited high catalytic activity towards formic acid electro-oxidation; Chen et al. [29] deposited the Pt nanoparticles on carbon nanotubes by pulsed electrodeposition, the prepared electrode maintained a good catalytic activity to oxygen and hydrogen peroxide.

In this study, glassy carbon electrode (GCE) modified with platinum particles (PtPs/GCE) has been developed for the GRN determination in its injectable dosage form. The electrochemical behavior of GRN has been investigated with sensitive, fast and simple square wave adsorptive stripping voltammetry (SW-AdSV) method. The developed PtPs/GCE sensor exhibits sensitive assay of GRN with rapid response and good stability.

2. Experimental

2.1. Reagents

Platinum hydrogen chloride (H_2PtCl_6) was used for the PtPs modified GCE. It was purchased from Sigma Aldrich. H_2PtCl_6 electrolyte solution is made up of 0.01 M H_2PtCl_6 and 0.1 M H_2SO_4 . GRN was purchased from Zentiva. The stock solution of GRN (1 mM) was dissolved in distilled water and diluted freshly before use. Phosphate buffer solutions (0.1 M) were prepared by mixing stock standard solutions of H_3PO_4 and Na_2HPO_4 , and adjusted with NaOH to various pH values. All chemicals were of analytical grade and distilled water was used for preparing all of the solutions.

2.2. Preparation of modified electrode

GCE electrode ($\Phi = 2$ mm) is polished first with 0.05 μm alumina slurry. After rinsing thoroughly with distilled water, the electrode was sonically cleaned in a 1:1 ethanol/water mixture, for 1 min, to remove any trace of alumina. Afterwards the electrode was cycled between -0.2 and $+1.8$ V in 0.1 M H_2SO_4 with a scan rate of 0.5 V s^{-1} . Pt particles were formed on the glassy carbon by potentiostatic deposition from a solution composed of 0.01 M H_2PtCl_6 + 0.5 M H_2SO_4 where a potential of 0.20 V for 25 s was employed. Following that, the PtPs/GCE was washed with distilled water before use.

2.3. Apparatus

A three-electrode cell is used in the electrochemical measurement, with GCE and PtPs/GCE as working electrode, Ag/AgCl as a reference electrode and platinum wire as a counter electrode; all of the potentials are reported versus Ag/AgCl. Electrochemical measurements were recorded using a computer controlled Autolab potentiostat (PGSTAT 302). CV and SWV experiments were carried out in a static electrochemical cell. The pH measurements were carried out using a Model 538 pH-meter (WTW, Weilheim, Germany) with an accuracy of ± 0.05 pH. The surface morphology of the modified electrodes was observed by scanning electron microscopy (ZEISS, EVO40).

2.4. Square-wave voltammetry parameters

In order to obtain the optimum conditions for the determination of GRN by means of square wave voltammetric (SWV) method, different instrumental parameters such as step potential, amplitude, and frequency were studied and the obtained optimum conditions were: step potential 9 mV; amplitude 65 mV; frequency 50 Hz. The SW-AdSV conditions were also optimized at accumulation potential of 500 mV and accumulation time of 180 s.

2.5. Injectable dosage form and recovery assay procedure

For voltammetric analysis, adequate volume of SETRON[®] (containing 3 mg/3 mL GRN) transfers to a 10 mL of calibrated flask, and completed to the volume with bi-distilled water. The concentration of the prepared solution is equivalent to 1.0×10^{-3} M. Analyzed solutions are prepared by taking aliquots of the GRN solution and diluting with the selected supporting electrolytes. In order to investigate the effects of interferences by the excipients, known amounts of the pure drug is added into the pre-analyzed dosage formulation. The recovery results are determined based on five parallel analyses. The nominal content of amounts is calculated from the corresponding regression equations of previously plotted calibration plots.

3. Results and discussion

3.1. Morphological characterization of the GCE and PtPs/GCE by SEM

The surface morphology of the bare GCE and PtPs/GCE are investigated by SEM with different scales. The GCE surface has a smooth and homogeneous morphology, with some scratches which remain after mechanical grinding and polishing (Fig. 1a). According to the SEM images (Fig. 1b and c), the GCE surface is densely coated with Pt particles. After electrodeposition, a large number of Pt particles is produced on the GCE forming a three-dimensional (3D) structure (Fig. 1b). The Fig. 1c shows SEM image of the sample with a lower magnification. As it can be seen in Fig. 1, the surface of PtPs/GCE was rougher with irregular cubic shape micro particles whose size

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