



Electrooxidation study and highly sensitive voltammetric determination of alfuzosin employing multi-walled carbon nanotubes and the ionic liquid 1-hexylpyridinium hexafluorophosphate nanocomposite sensor



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ABSTRACT

By using several electrochemical techniques, the voltammetric behavior of alfuzosin (AFZ) based on the oxidation at the carbon nanocomposite electrode designed by incorporation of multi-walled carbon nanotubes (MWCNT) and the ionic liquid 1-hexylpyridinium hexafluorophosphate (HPFP) has been reported. Various characteristics of the electrode were studied by scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR). Differential pulse voltammetry (DPV) was applied as a sensitive procedure for the quantitative determination of AFZ. Concerning optimized experimental conditions, the proposed electrode exhibited substantial sensitivity toward determination of AFZ. Good linearity in the wide range of 2.0×10^{-8} to 9.0×10^{-5} M was obtained with the detection limit of 4.1×10^{-9} M. Similarly for the amperometric procedure the liner range of 1.0×10^{-5} to 1.0×10^{-4} M with detection limit of 2.1×10^{-6} M was achieved. To acquire more information about the reaction mechanism and estimating some kinetic parameters, cyclic voltammetry (CV) studies was also performed. The oxidation was irreversible and showed adsorption controlled behavior. The method was successfully applied for the determination of AFZ content in plasma samples.

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1. Introduction

Alfuzosin (AFZ) is an alpha 1-selective quinazoline derivative that is appropriate for medication of benign prostatic hyperplasia (BPH) and hypertension. It possesses a bioavailability of about 60%, an elimination half-life of about 5 h, and is broadly metabolized by the liver. It may enhance risk of QT prolongation in susceptible individuals [1–3]. Regardless of its remedial advantages, this alpha-blocker may cause orthostatic hypotension and other blood pressure-related side effects [4]. Hence, the determination of AFZ is essential to acquire optimum medicinal dosages in pharmaceutical formulations. Formerly reported methods which have been applied for analysis of AFZ typically include high-performance liquid chromatography [5–9], mixed micelle cloud point-magnetic dispersive μ -solid phase extraction with fluorescence analysis [10], and spectrophotometric methods [11,12]. Chromatographic techniques require expensive instruments and time consuming purification and pre-concentration procedure. The spectroscopic techniques also usually possess fairly high detection limits [11]. Consequently, rendering an alternative method is significantly preferred. Although electroanalytical methods can be desirable selection because of their good sensitivity, selectivity, simplicity, fast response time, and

low costs, literature review reveals that, electrochemical techniques for AFZ determination were rarely reported [13,14].

For the first time, carbon ionic liquid electrode (CILE) was fabricated in 2006 by Maleki et al. as a new and high-efficiency carbon composite electrode [15]. The basic notion for designing this novel electrode was to replace traditional nonconductive organic binders in carbon paste electrodes (CPEs) with a pyridinium-based ionic liquid. Some remarkable characteristics of CILE are fast electron transfer, widespread potential window in aqueous solutions, renewable surface, small background current and resistivity toward fouling by bio-molecules [16]. Due to such prominent behavior, CILEs can be applied as appropriate sensors in electroanalytical analysis of diverse biological compounds.

Carbon nanotubes (CNTs) have promulgated a large number of substantial applications in electrochemistry, especially in the structure of electrochemical sensors that are reported in the literature. The outstanding properties of carbon nanomaterial-ionic liquid nanocomposites have developed their utilization in the various field of chemistry [17]. The surface of the traditional carbon electrodes have been modified using carbon nanotube/room temperature ionic liquids (CNT/RTIL) nanocomposites. CNTs are outspread in IL pastes owing to their outstanding characteristics such as high electronic conductivity and mechanical resistance combined with an increased electroactive surface area, which effectively improves the electrochemical responses [18,19].

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The present study, describes the usage of MWCNT/CILE as an excellent sensor to investigate the electrochemical oxidation of AFZ as well as the utilization of differential pulse voltammetry (DPV) for highly sensitive quantification of this drug. The results represents that the MWCNT/CILE can be effectively applied for both intentions including the study of electrochemical behavior and the development of new analytical procedures to be applied for the determination of AFZ.

2. Experimental

2.1. Reagents and solutions

Ascorbic acid, uric acid, graphite powder, potassium ferricyanide ($K_3Fe(CN)_6$), potassium dihydrogen phosphate, and dipotassium hydrogen phosphate were purchased from Merck and were used as received. Multi-walled carbon nanotube with a 95% purity, o.d. = 6–9 nm and 5 μ m length was obtained from Sigma-Aldrich (St. Louis, USA). AFZ was kindly provided by Aboureyhan Pharmaceutical Company (Tehran, Iran) and used without prior purification. HPFP was purchased from Merck. AFZ stock solution (0.01 M) was prepared with double-distilled water and stored at 4 °C in the dark. The supporting electrolyte was phosphate buffer (PBS) 0.1 M, pH 7. All solutions were freshly prepared with double distilled water. The drug-free plasma samples were kindly supplied by the Blood Transfusion Organization (Fars, Iran) and belonged to healthy male volunteers. The plasma samples were stored frozen until the assay.

2.2. Electrode preparation

Carbon ionic liquid electrode (CILE) was made by rigorously hand-mixing the graphite powder and HPFP with a ratio of 50/50 (w/w) in a mortar and pestle. The resulting paste was packed stoutly into the electrode cavity (1.8 mm i.d.) of a Teflon holder. To acquire better homogeneity in the composite and to decrease background current, the electrode was heated for 2 min in oven, to a temperature more than the melting point of IL (m.p. ~ 65 °C) before use [20]. The electrical contact was provided by a copper wire. Prior to each measurement, pushing the extra of paste out of the tube and then polishing the freshly exposed paste with weighing paper gained a new surface. The carbon nanotube ionic liquid nanocomposite electrode (MWCNT/CILE) was prepared similarly with the weighted amounts of graphite powder, IL and MWCNT (40%:50%:10%, wt%), respectively. The Multi-walled carbon nanotube paste electrode (MWCNT/CPE) was made by mixing 60% graphite powder, 30% mineral oil and 10% MWCNT. Unmodified carbon paste electrode (CPE) was made in the same way but without adding MWCNTs to the mixture.

2.3. Apparatus

All of the voltammetric studies were performed in an electroanalytical system, Autolab PGSTAT 12, potentiostat/galvanostat and with GPES software. The electrochemical cell was assembled with a conventional three-electrode system: an Ag/AgCl/KCl (3 M) reference electrode (Metrohm) and a platinum disk as a counter electrode. The working electrodes used in this study were CILE, MWCNT/CILE, MWCNT/CPE and CPE. The cell was one compartment cell with an internal volume of 100 ml. All experiments were typically conducted at 25 °C without removing the dissolved oxygen. Scanning electron microscopy (SEM) images were obtained by using a KYKY EM-3200 (China). The FTIR studies were performed using a Perkin-Elmer FT-IR spectrometer RX-1.

3. Results and discussion

3.1. Surface morphology and structural characterization of the electrode

To achieve more assessment into the structure of the fabricated electrode, FTIR spectra of the graphite powder, MWCNT, HPFP and the MWCNT/HPFP were studied and the results has been shown in Fig. 1. The peaks appeared at 2930 cm^{-1} (aliphatic C—H stretching), 1650 cm^{-1} (stretching vibration of C=N), 1490 cm^{-1} (in-plane bending vibration of $-CH_3$, $-CH_2-$) [21,22] and 830 cm^{-1} (PF_6^-) [23] which is in agreement with the literature data. Meanwhile graphite and MWCNT do not support a static dipole moment, their IR absorption peak is very weak [23]. Accordingly, the observed spectral features of the MWCNT/HPFP are only from the ionic liquid. The strong band at 833 cm^{-1} is correlated to the PF_6^- anion and can be seen in both HPFP and the MWCNT/HPFP spectra [23]. Furthermore, the surface morphology of different electrodes including CPE, MWCNT/CPE, CILE and

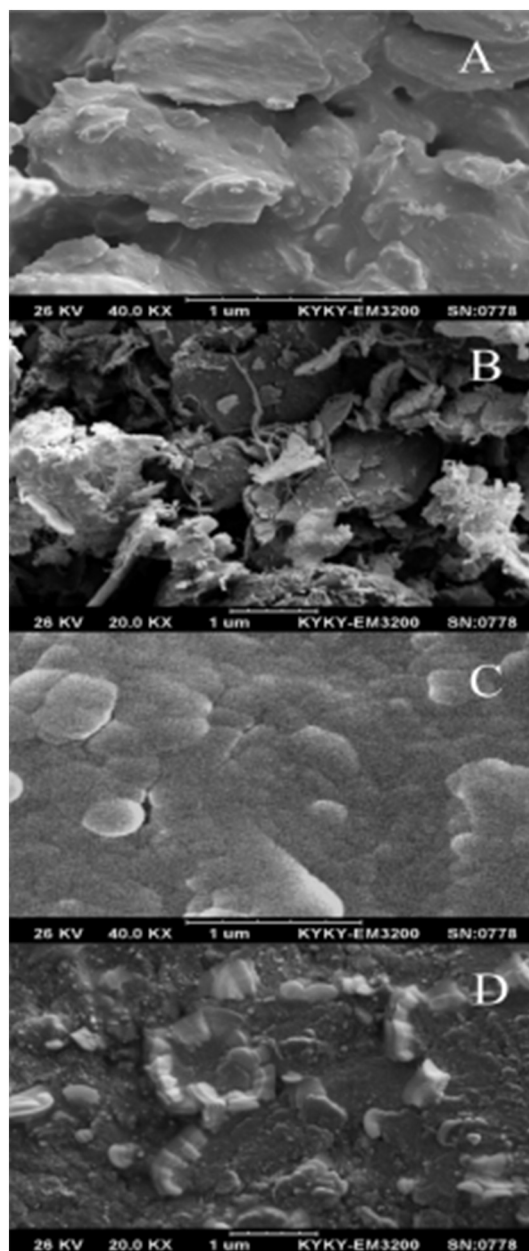


Fig. 1. Scanning electron microscopy of CPE (A), MWCNT/CPE (B), CILE (C) and MWCNT/CILE (D) surface.

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