



Electrocatalytic determination of captopril using a modified carbon nanotube paste electrode: Application to determination of captopril in pharmaceutical and biological samples



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ABSTRACT

A carbon paste electrode modified with carbon nanotube and benzoylferrocene (BF) was fabricated. The electrochemical study of the modified electrode, as well as its efficiency for electrocatalytic oxidation of captopril (CAP), was described. The electrode was employed to study the electrocatalytic oxidation of CAP, using cyclic voltammetry (CV), chronoamperometry (CHA) and square wave voltammetry (SWV) as diagnostic techniques. It has been found that the oxidation of CAP at the surface of modified electrode occurs at a potential of about 85 mV less positive than that of an unmodified CPE. SWV exhibits a linear dynamic range from 1.0×10^{-7} to 3.5×10^{-4} M and a detection limit of 3.0×10^{-8} M for CAP. Finally the modified electrode was used for determination of CAP in CAP tablet and urine sample.

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1. Introduction

S-Captopril (CAP) is a synthetic dipeptide serving as an orally active inhibitor of the angiotensin-converting enzyme (ACE) and has been widely used as antihypertensive drug [1] and to moderate heart failure [2]. It is the only inhibitor of ACE bearing a thiol group and can take up free radicals in living systems and exhibits antioxidant properties [3–5].

CAP is metabolized in liver (it is oxidized into the corresponding disulfide) and is mainly excreted with the urine (40–60% of the excreted drug remain unchanged in the urine) [6]. Therefore, the determination of CAP is important from a physiological point of view as well as for the purposes of quality control. In some real sample analysis, it is important also to discriminate between enantiomers and the enantiopurity tests must follow the general

methods for determination of base molecule. Several methods have already been reported for the determination of CAP in pharmaceutical formulations and clinical samples, including high performance liquid chromatography [7], gas chromatography [8], spectrophotometry [9], fluorimetry [10], radiochemical [11], FT-Raman spectroscopy [12], capillary electrophoresis [13] and chemiluminescence [14]. CAP with its thiol group can oxidize at the surface of various electrodes and different electrochemical methods [15–26] have been used for its determination.

Carbon paste electrode (CPE) is a special kind of heterogeneous carbon electrode consisting of mixture prepared from carbon powder (as graphite, glassy carbon and others carbonaceous materials) and a suitable water-immiscible or non-conducting binder [27–29].

The use of carbon paste as an electrode was initially reported in 1958 by Adams [30]. In afterward researches a wide variety of modifiers including enzymes [31], polymers [32] and nanomaterials [33–36] have been used with these versatile electrodes. CPEs are widely applicable in

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both electrochemical studies and electroanalysis thank to their advantages such as very low background current (compared to solid graphite or noble metal electrodes), facility to prepare, low cost, large potential window, simple surface renewal process and easiness of miniaturization. Besides the advantageous properties and characteristics listed before, the feasibility of incorporation different substances during the paste preparation (which resulting in the so-called modified carbon paste electrode), allow the fabrication of electrodes with desired composition, and hence, with pre-determined properties [36,37].

The electrochemical methods using chemically modified electrodes (CMEs) have been widely used as sensitive and selective analytical methods for the detection of the trace amounts of biologically important compounds. One of the most important properties of CMEs has been their ability to catalyze the electrode process via significant decreasing of overpotential respect to unmodified electrode. With respect to relatively selective interaction of the electron mediator with the target analyte in a coordination fashion, these electrodes are capable to considerably enhance the selectivity in the electroanalytical methods [38–45].

Since the discovery of carbon nanotubes (CNTs) in 1991 [46], numerous investigations were focused on the studies of their properties and applications [47]. Because of the special tube structure, CNTs possess several unique properties such as good electrical conductivity, high chemical stability and extremely high mechanical strength [48]. In addition, the subtle electronic behavior of CNTs reveals that they have the ability to promote electron-transfer reaction and have a high electrocatalytic effect when used as electrode materials [49–53]. All these fascinating properties make CNTs as a suitable candidate for the modification of electrodes [54–57].

Drug analysis is an important branch of analytical chemistry and plays an important role in drug quality control and has a wide impact on public health. Therefore, developing sensitive, simple, rapid, and reliable method for the determination of active ingredient is valuable and necessary [58–80].

Therefore, in the present work, we describe the preparation of a new electrode composed of CNPE modified with benzoylferrocene (BFCNPE) and investigate its performance for the electrocatalytic determination of CAP in aqueous solutions. We also evaluate the analytical performance of the modified electrode for quantification of CAP in some real samples.

2. Experimental

2.1. Apparatus and chemicals

The electrochemical measurements were performed with an Autolab potentiostat/galvanostat (PGSTAT 12, Eco Chemie, the Netherlands). The experimental conditions were controlled with General Purpose Electrochemical System (GPES) software. A conventional three electrode cell was used at 25 ± 1 °C. An Ag/AgCl/KCl (3.0 M) electrode, a platinum wire, and the BFCNPE were used as the reference,

auxiliary and working electrodes, respectively. A Metrohm 710 pH meter was used for pH measurements.

All solutions were freshly prepared with double distilled water. CAP and all other reagents were of analytical grade from Merck (Darmstadt, Germany). The buffer solutions were prepared from orthophosphoric acid and its salts in the pH range of 2.0–11.0. Multiwalled carbon nanotubes (purity more than 95%) with o.d. between 10 and 20 nm, i.d. between 5 and 10 nm, and tube length from 0.5 to 200 μm were prepared from Nanostructured & Amorphous Materials, Inc (Los Alamos, New Mexico, USA). Benzoylferrocene was synthesized in our laboratory as reported previously [33].

2.2. Preparation of the electrode

The BFCNPEs were prepared by hand mixing 0.01 g of BF with 0.89 g graphite powder and 0.1 g CNTs with a mortar and pestle. Then, ~ 0.7 mL of paraffin oil was added to the above mixture and mixed for 20 min until a uniformly-wetted paste was obtained. The paste was then packed into the end of a glass tube (ca. 3.4 mm i.d. and 15 cm long). A copper wire inserted into the carbon paste provided the electrical contact. When necessary, a new surface was obtained by pushing an excess of the paste out of the tube and polishing with a weighing paper.

For comparison, BF modified carbon paste electrode (BF-CPE) without carbon nanotubes, carbon nanotubes paste electrode (CNPE) without BF, and unmodified carbon paste electrode (CPE) in the absence of both BF and carbon nanotubes were also prepared in the same way.

3. Results and discussion

3.1. Electrochemical properties of BFCNPE

BFCNPEs were prepared and their electrochemical properties were studied in a 0.1 M phosphate buffer solution (PBS) (pH 7.0) using CV. The experimental results show well-defined and reproducible anodic and cathodic peaks related to benzoylferrocene/benzoylferricenium redox system, which show a quasi-reversible behavior in an aqueous medium [81]. The electrode capability for the generation of a reproducible surface was examined by cyclic voltammetric data obtained in optimum solution pH 7.0 from five separately prepared BFCNPEs (Table 1).

In addition, the longterm stability of the BFCNPE was tested over a three-week period. When CVs were recorded after the modified electrode was stored in atmosphere at room temperature, the peak potential for CAP oxidation was unchanged and the current signals showed less than 3.0% decrease relative to the initial response. The antifouling properties of the modified electrode toward CAP

Table 1
Cyclic voltammetric data obtained for constructed BFCNPE in 0.1 M PBS (pH 7.0) at 50 mV s^{-1} .

E_{pa} (V)	E_{pc} (V)	$E_{1/2}$ (V)	E_p (V) Δ	I_{pa} (μA)	I_{pc} (μA)
0.645	0.540	0.592	0.105	3.42	-1.97

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