



Selective and sensitive determination of tannic acid using a 1-benzoyl-3-(pyrrolidine) thiourea film modified glassy carbon electrode



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ARTICLE INFO

Article history:

Received 25 March 2016

Received in revised form 27 June 2016

Accepted 27 June 2016

Available online 28 June 2016

Keywords:

Surface modification

Voltammetry

Tannic acid

ABSTRACT

In this study the selective determination of tannic acid (TA) was performed using a glassy carbon (GC) electrode modified with 1-benzoyl-3-(pyrrolidine) thiourea (PrTu). PrTu was deposited on the GC electrode in nonaqueous media by cyclic voltammetry. PrTu-GC electrode demonstrated an electrocatalytic effect on the oxidation of TA using differential pulse voltammetry (DPV) method in 0.1 M Britton Robinson (BR) buffer solution of pH 2. Under optimum conditions, linear calibration graphs were obtained over the TA concentration range 2.0×10^{-6} to 4.2×10^{-5} M. The limit of detection (LOD) was found as 0.6 μ M. The proposed electrode was applied to the determination of spiked TA in tea. The proposed method was also applied for the determination of TA in orange juice samples and the results of the experiments were in agreement with that obtained by the spectrophotometric comparison method.

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1. Introduction

Tannic acid (TA), a water soluble polyphenol compound has been investigated for many years, especially to cure many diseases [1–4]. Due to its wide range of applications, analysis of TA is important not only in food but also in the medical and environmental fields. TA is present commonly in the human diet including fruits and different kinds of vegetables and can be found in several beverages, including wine, beer, coffee, black tea and white tea. It is used as a food additive as clarifying agent, flavor adjunct and flavoring agent [5] as well as additive in medical and veterinary fields due to its antimicrobial activity, anticarcinogenic and antimutagenic potentials and also antioxidant nature protect cellular oxidative damage [6,7]. Although TA has useful properties, it may cause serious problems because TA can form carcinogenic disinfection byproducts such as trihalomethanes during chlorination of water. Moreover, TA has toxicity for species which are aquatic such as algae, phytoplankton and fish [8–10]. The content of TA in fruits, tea and beer influences their taste and also it is an important standard used to evaluate the quality of the products [11].

Several methods including spectrophotometry [12], colorimetry [13], liquid chromatography [14] and chemiluminescence [15–17] have been applied for the determination of TA content in the waters, pharmaceuticals and foods. Each method has its advantages and

drawbacks. For example, chromatographic methods allow the determination of TA, but they are time consuming and expensive. Compared to these methods, the electrochemical method has several advantages such as its low cost, the options of analysis without extraction or pre-concentration, highly selective and sensitive and the short time required for analysis. But, only very few reports were accessible in the literature for the electrochemical determination of TA [18,19]. Electroanalytical methods with different types of electrodes were used for TA determination, but the presence of ascorbic acid limits the use of some of these methods, or sample pretreatments are needed to remove ascorbic acid before analysis [5,20,21]. These examples show that rapid, sensitive and selective TA determination is still a problem.

In this study, a modified surface was prepared by covering the GC electrode with PrTu. The modified surface was characterized by cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS) and X-ray photoelectron spectroscopy (XPS). The prepared modified electrode was used as a sensor for TA determination by applying the standard addition method in tea and orange juice samples. The interfering effect of some ions and biological species were also studied at pH 2 Britton-Robinson (B-R) buffer.

2. Experimental

2.1. Reagents and chemicals

The reagents and solutions in the study are all of reagent grade. TA was purchased from Merck and a primary stock solution of 1×10^{-3} M TA was prepared in distilled water. Working solutions

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were then prepared by diluting the stock solution with distilled water. Tetraethylammonium tetrafluoroborate (TEATFB) used for the supporting electrolyte, were of the highest purity available from Merck and Acetonitrile (ACN) as a solvent were of available from Sigma-Aldrich. A stock solution of ferrocene (1×10^{-3} M) was prepared by dissolving it in ACN containing 0.1 M TEATFB.

Britton-Robinson (B-R) buffer solution was prepared in such a way that 2.3 mL of glacial acetic acid, 2.7 mL of phosphoric acid (85%), and 2.47 g of boric acid were dissolved by dilution with water to 1.0 L; 50 mL portions of this solution were taken, and the pH was adjusted by the addition of an appropriate amount of 2.0 M NaOH to the desired value.

2.2. Apparatus

A BAS model electrochemical analyzer (Bioanalytical Systems, Epsilon Basic Plus Potentiostat/Galvanostat, USA) was used for differential pulse voltammetry (DPV) and cyclic voltammetry (CV) measurements. A conventional three-electrode system was used. Glassy carbon (GC) electrode (BAS Model) was used as working electrode. The reference electrode was an Ag/AgCl/KCl (sat.) used in aqueous media and an Ag/AgNO₃ (0.01 M) used in ACN nonaqueous media. The counter electrode was Pt wire. pH values were measured using a Thermo Scientific Orion 4-Star Plus pH/Conductivity Meter with combined glass electrode was used to measure pH of all the solutions. Absorption spectra and absorbances were recorded using SHIMADZU UVmini-1240 UV-visible spectrophotometer.

2.2.1. CV measurements

EIS measurements were performed in the presence of a 1.0 mM ferrocyanide/1.0 mM ferricyanide (1/1) mixture with 0.1 M KCl as supporting electrolyte. EIS data were performed in the frequency range from 100 kHz to 0.05 Hz by applying an AC voltage with 5 mV wave amplitude and at an electrode potential of 0.215 V, the formal potential of ferrocyanide/ferricyanide redox couple.

XPS determination was carried out at Thermo K-Alpha spectrometer. The X-ray source is Al K α X-rays (monochromatic) which is carried out at 90° electron take-off angle. The experiments were conducted at pressures below 10^{-7} mbar. All samples were scanned without ion beam surface cleaning procedure.

A Jeol (Jeol JSM-6060LV) Scanning Electron Microscope (SEM) was used to observe the surface morphology on GC surface. Olympus SZX7 stereomicroscope was used for optical measurement.

2.3. Cleaning and modification of GC electrode surfaces

GC electrode was mechanically polished first with fine wet emery papers with grain size of 4000 (Buehler) and then with 0.3 μ m and 0.05 μ m alumina slurries (Baikowski Int. Corp.) on a microcloth pad (Buehler). Polished GC electrode was sonicated in ultra-pure water and then with ACN for 10 min. for removal of trace alumina from the surface. After these procedures, GC electrode was rinsed with ACN.

Anodically surface modification of cleaned GC electrode was performed in a solution of 1 mM PrTu (synthesized by the reported procedure [22]), in ACN containing 0.1 M TEATFB as a supporting electrolyte vs. Ag/AgNO₃ (0.01 M) reference electrode using CV with a scan rate of 100 mV s⁻¹ for 25 cycles between -2 V and 2 V. The electrode was then rinsed with ACN to remove any physisorbed or unreacted materials from the electrode surface.

2.4. Preparation of tea sample

The solution sample of tea was prepared by adding deionized water to 3 g dry tea in an Erlenmeyer flask, stirring on a hot plate at 90 °C for 1 h, cooling to room temperature, filtering and then TA is diluted with filtrate as its concentration will be 1×10^{-3} M.

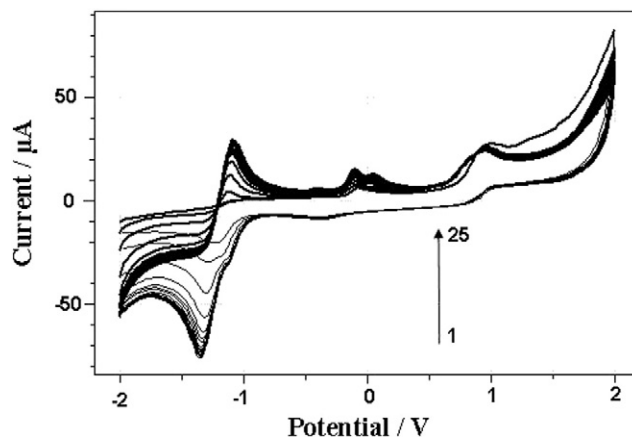


Fig. 1. The CVs of 1 mM PrTu solution on GC electrode vs. Ag/Ag⁺, supporting electrolyte is 0.1 M TEATFB in acetonitrile, scan rate is 100 mV s⁻¹.

3. Results and discussion

3.1. Electrochemical preparation of PrTu modified electrode

Fig. 1 shows cyclic voltammograms (CVs) of GC electrode in 1 mM PrTu solution in ACN containing 0.1 M TEATFB. The parameters for CV were as follows: initial E = -2 V, final E = 2 V, Scan rate = 100 mV s⁻¹, sensitivity = 1×10^{-5} A/V. As seen in Fig. 1, the peak

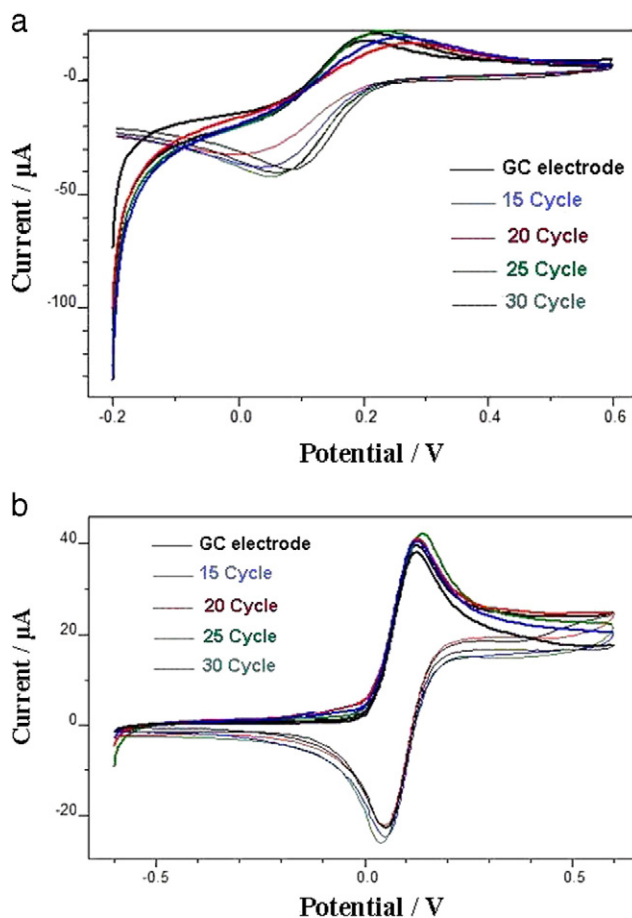


Fig. 2. Cyclic voltammograms of 1 mM (a) ferricyanide (in 0.1 M KCl) vs. Ag/AgCl/KCl (sat.) reference electrode and (b) ferrocene (in acetonitrile containing 0.1 M TEATFB) vs. Ag/Ag⁺ reference electrode on PrTu-GC electrodes with different (15, 20, 25, 30) scan cycles, scan rate is 100 mV s⁻¹.

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