



Simultaneous determination of ascorbic acid, uric acid and glucose using glassy carbon electrode modified by nickel nanoparticles at poly 1, 8-diaminonaphthalene in basic medium



K.M. Hassan^{a,*}, G.M. Elhaddad^b, M. Abdel Azzem^b

^a Physics and Mathematics Engineering Department, Faculty of Electronic Engineering, El-Menoufia University, Egypt

^b Chemistry Department, Faculty of Science, El-Menoufia University, Egypt

ARTICLE INFO

Article history:

Received 14 December 2013

Received in revised form 23 April 2014

Accepted 3 June 2014

Available online 28 June 2014

Keywords:

Modified poly/1,8-diaminonaphthalene electrode

Nickel nanoparticles

Ascorbic acid

Uric acid

Glucose

Simultaneous determination

ABSTRACT

Poly 1,8-diaminonaphthalene (PDAN) modified electrode based on glassy carbon (GC) electrode was prepared in sulfuric acid (H₂SO₄) and acetonitrile (ACN) [4.5 M H₂SO₄ in ACN] mixed solvent using cyclic voltammetry technique (CV). The optimum conditions for the film formation was obtained by sweeping the electrode potential between +0.2 V and +1.2 V with scan rate of 0.1 V s⁻¹ for 20 cycles using 1.0 mM of 1,8-diaminonaphthalene monomer (DAN). Poly 1,8-diaminonaphthalene/GC (PDAN/GC) modified electrode was found to be active in ACN containing 0.1 M lithium perchlorate (LiClO₄) and inactive in 0.1 M sodium hydroxide (NaOH). Nickel ions (Ni(II)) were incorporated into the polymer backbone. The obtained new modified electrode with dispersed nickel nanoparticles (Ni/PDAN/GC) exhibits stable redox behavior of Ni(II)/Ni(III) couple in aqueous 0.1 M NaOH solution. Ni/PDAN/GC modified electrode was characterized by CV and scanning electron microscope (SEM). The electro-oxidation of single ascorbic acid (AA), uric acid (UA) and glucose (Glu) at Ni/PDAN/GC modified electrode in 0.1 M NaOH was studied using square wave voltammetry (SWV) technique. Simultaneous determination of AA, UA and Glu at Ni/PDAN/GC electrode in 0.1 M NaOH was achieved where Ni/PDAN/GC electrode showed the capability of potential splitting of oxidation peaks of the present biological compounds. The modified electrode was applied for the determination of UA, AA and Glu in real samples.

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1. Introduction

Due to the pivotal role of Glu in physiological processes, its concentration determination has become a very important issue in clinical, biological and food matrices [1–3]. Therefore creation of sensitive, selective, reliable and low-cost Glu sensors to take regular measurements of blood Glu levels is of great demand [4].

Recent studies disclosed that the concentration of ascorbate in biological fluids can be used to quantify the amount of oxidative stress in human metabolism [5,6]. AA is very popular due to its antioxidant properties and its presence in the human diet as a vital vitamin [7,8]. Excessive oxidative stress has been attributed to diabetes, cancer and hepatic disease. Therefore, great efforts have been paid for the development of reliable methods to quantify ascorbate in biological systems [9].

UA, an important biomolecule present in blood serum and urine is the primary end product of purine metabolism. For a healthy person, abnormal levels of UA in blood serum and urine lead to

several diseases; namely, gout hyperuricemia, pneumonia, kidney damage, cardiovascular diseases and Lesch-Nyhan syndrome [10,11], therefore detection of UA in human fluids can be utilized as a powerful indicator for early detection of diseases. Colorimetric, enzymatic and electrochemical methods are used to determine the concentration of UA [12].

AA and UA usually coexist with Glu in physiological samples [13], and the oxidation potentials of AA and UA are too close to be determined separately at bare electrodes.

Modified electrodes have obvious advantages in the detection of analytes. For example, electropolymerization can accelerate transmission of electrons on the surface of the electrode, it is highly selective and sensitive due to the film homogeneity in electrochemical deposition, and it has strong adherence to the electrode surface with large surface area [14,15].

Owing to porous structure and high surface area of many conducting polymers and the possibility of dispersing metallic particles into the polymers, considerable attention has been paid to these systems [16]. Moreover, the electro-catalytic activities of such modified electrodes are higher than pure metals. In this regard, it has been reported that various polymeric electrodes

* Corresponding author. Tel.: +20 1001303945.

E-mail address: khalidha_306@yahoo.com (K.M. Hassan).

modified by dispersed metals show catalytic properties for organic compounds oxidation [17,18].

In this study, PDAN/GC modified electrode will be fabricated by electropolymerization method in which nickel nanoparticles will be incorporated into the film by chemisorption [19]. The obtained Ni/PDAN/GC modified electrode will be examined for the oxidation of Glu, AA or UA together with their simultaneously determination.

2. Experimental

2.1. Materials

1,8-Diaminonaphthalene of analytical grade was purchased from Aldrich, kept in the dark and stored in a refrigerator before used. Sulfuric acid (H_2SO_4 98%) (Merck), acetonitrile (99.9%) HPLC (LAB-SCAN) and ethanol (ADWIC) (96%) were used without further purification. Sodium hydroxide (NaOH) and nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$) were analytical grade chemicals. Glucose (Merck), ascorbic acid and uric acids (Aldrich) were used as received. Diamond paste (Presi) 2 μm , and freshly distilled water was always used.

2.2. Instruments

Electrochemical measurements were recorded using a potentiostat voltammetric analyzer from BAS with CV50 W software. All voltammograms were obtained in a conventional three-electrode cell which contains a 3 mm GC as working electrode (WE), platinum wire as an auxiliary electrode and Ag/AgCl as a reference one. The WE was cleaned by polishing with diamond paste, followed by rinsing with ethanol and then with the used solvents.

2.3. Experimental procedure

2.3.1. Electrochemical preparation of PDAN/GC modified electrode

Typical cyclic voltammogram of PDAN film formation in a mixed solvent of [4.5 M H_2SO_4 in ACN] in the presence of 1.0 mM of DAN monomer at GC electrode using CV technique is shown in Fig. 1. The electrode potential was swept at a rate of 0.1 V s^{-1} between 0.2 and 1.2 V for 20 cycles. The monomer is electro-oxidized in one irreversible anodic peak at 0.98 V (peak a) during the first scan. This anodic peak could be attributed to the oxidation of the monomer to the radical cation then the dication [20]. No reversible cathodic peak corresponding to the anodic one was detected indicating a fast consumption of the electro generated mono radicals by follow up chemical reaction to form the dimer [21].

A reduction peak for the product formed during the anodic oxidation is observed at 0.58 V (peak b). A new anodic peak appeared at potential of 0.68 V (peak c) with a semireversible cathodic one at

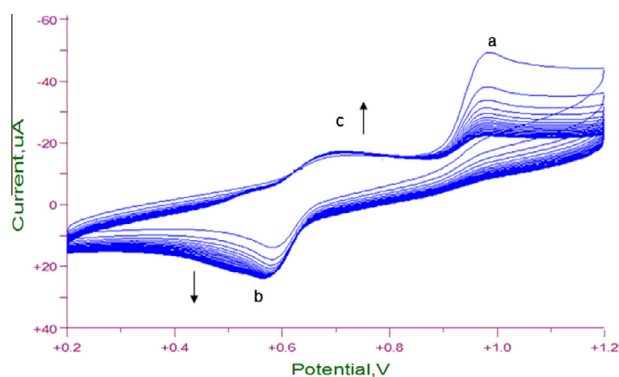


Fig. 1. Cyclic voltammograms of the electropolymerization of 1.0 mM DAN in a mixed solvent of [4.5 M H_2SO_4 in ACN] at GC electrode using scan rate of 0.1 V s^{-1} for 20 cycles.

0.58 V (peak b) starting from the second scan which may be due to the redox behavior of the formed product. On continuous cycling, peak (a) diminished in current while that of peaks (b and c) increased giving evidences for PDAN film formation and its progress on the surface of the GC electrode [20].

2.3.2. Optimum conditions for PDAN film formation

In order to achieve better electroactive redox response of the prepared PDAN/GC modified electrode, different factors affecting the electroactivity of the prepared PDAN film such as potential limits, scan rate, number of cycles and H_2SO_4 concentration in the mixed solvent were examined (figures not shown) by comparing the anodic peak currents of the electrochemical responses of the prepared PDAN films in an ACN solution containing 0.1 M LiClO_4 .

The effect of the potential limits on the anodic peak currents of the electrochemical responses of the prepared PDAN/GC modified electrodes were examined in the range between -0.2 V and 1.2 V . Results showed that the film formed between 0.2 V and 1.2 V gives the highest anodic peak current. The effect of potential scan rate was examined by sweeping the electrode potential from 0.01 V s^{-1} to 0.12 V s^{-1} . The current response of PDAN/GC modified electrode increases with potential scan rates between 0.01 V s^{-1} and 0.1 V s^{-1} and starts to decrease until 0.12 V s^{-1} revealing the best scan rate is 0.1 V s^{-1} .

The effect of film's thickness (determined by the number of polymerization scans) on the electrochemical activity of PDAN/GC modified electrode was studied by scanning the electrode potential in a range from 5 to 35 cycles. The current intensity of the film response increases gradually as the number of cycles increases from 5 to 20 cycles. The deterioration of the electroactivity begins as the number of cycles increases over 20 cycles.

It was important to give a close view for the solvent used by changing the H_2SO_4 concentration in the mixed solvent from 1.0 M to 6.0 M which indicated that the best concentration to be used is 4.5 M. Finally, the obtained PDAN/GC modified electrode prepared with the optimum conditions was taken from the polymerization medium, rinsed with ACN followed by distilled water to remove any traces of DAN monomer. The prepared PDAN/GC modified electrode was found to be inactive in alkaline 0.1 M NaOH aqueous solution in the potential range between 0.0 and 0.7 V at scan rate of 0.1 V s^{-1} as could be seen from Fig. 2a.

2.3.3. Incorporation of nickel nanoparticles into PDAN film

In order to incorporate Ni nanoparticles ions into PDAN film, a freshly prepared PDAN/GC modified electrode was placed in an open circuit of a well stirred aqueous solution of different concentrations of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (0.1, 0.3, 0.5, 0.7, 1.0 and 2 M) using different periods of time (1, 5, 10, 15, 30, 45, 60 and 120 min).

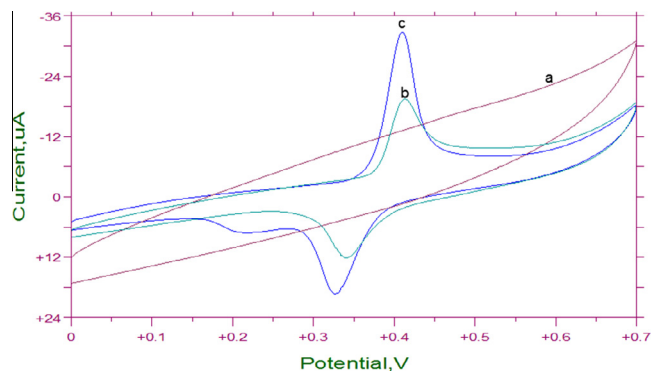


Fig. 2. Cyclic voltammograms of the electrochemical responses of: (a) PDAN/GC modified electrode, and Ni/PDAN/GC modified electrodes, (b) before and (c) after anodic polarization in 0.1 M NaOH solution using CV technique at scan rate of 0.1 V s^{-1} .

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