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Original scientific paper

Electroanalysis of tert-butylhydroquinone in food products using a paste electrode enlarged with single wall carbon nanotubes as catalyst

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Abstract

In this study, an electrochemical sensor was introduced as a simple and fast electro-analytical tool to monitor and sensing of tert-butylhydroquinone (TBHQ) in food products. The suggested electrochemical sensor is fabricated by modification of paste electrode (PE) by single wall carbon nanotubes (SWCNTs) as nanocatalyst. The oxidation current of TBHQ was improved by about 2.62 times and its oxidation potential was reduced by about 50 mV after using SWCNTs as conductive catalyst on a carbon paste matrix. The oxidation current of TBHQ showed a linear dynamic range of 0.05 to 390 µM in the sensing process using SWCNTs/PE as the electroanalytical sensor. On the other hand, SWCNTs/PE successfully monitored TBHQ with a detection limit of 10 nM at optimum conditions. The real sample analysis data clearly showed a recovery range of 97.2 to 104.3 %, which is very interesting for a new analytical tool in the food-sensing process.

Keywords

Food analysis; electroanalysis; modified electrode; carbon paste; voltammetry

Introduction

Monitoring food additives is one of the best methods for investigating quality food products and checking the presence of prohibited substances [1-7]. For this purpose, different measurement methods have been reported in food samples [8-13]. In the meantime, attention to electrochemical methods has grown significantly due to many advantages and low cost [14-18]. By presenting various solutions for modification in electrochemical sensors, various reports have been presented for the use of these sensors in the construction of measuring instruments [19-24]. High overpotentials and low redox signals of electroactive materials and, especially, food additives are the main problems to trace level monitoring of them in real samples [25-29]. Therefore, conductive catalysts were suggested for the fabrication of highly conductive and sensitive electrochemical sensors [30-33].

It is important to check the concentration of food additives and especially antioxidants widely used in the food industry to determine the food quality [34]. The tert-butylhydroquinone (TBHQ) is one of the phenolic-type antioxidants with a wide range of applications in many edible animal fats and vegetable oils [35]. High concentrations of TBHQ can be harmful to the human body and create some problems, such as vision disturbances and neurotoxic effects [35]. Therefore, many research works focused on monitoring them in food products [36,37]. However, efforts are still being made to provide simpler and more sensitive solutions.

Carbon nanotubes (CNT) are one of the main and useful carbon nanocatalysts widely used in different branches of science, especially in sensors and energy majors [38-40]. Easy modification and high surface area with good electrical conductivity have introduced them as a unique catalyst [41,42]. On the other hand, according to the literature, single wall carbon nanotubes (SWCNTs) showed more advantages than other carbon-based nanomaterials as catalysts for the fabrication of electrochemical sensors due to high electrical conductivity and good surface area and were selected for this work [43].

In this research work, a carbon paste electrode (PE) was modified with single-walled CNTs (SWCNTs) as conductive catalysts and a fabricated sensor was used to determine TBHQ in food products. The results clearly showed the powerful ability of SWCNTs/PE in trace analysis of TBHQ with acceptable recovery data in real sample analysis.

Experimental

Instrument and materials

A potentiostat/galvanostat (Metrohm Company) was used to record redox signals of TBHQ in an aqueous solution. All potentials were recorded using Ag/AgCl/KCl_{sat} as a reference electrode. Pt wire was used as a counter electrode and SWCNTs/PE as the working electrode. The TBHQ (99 %) was purchased from ACROS Company and a stock solution of TBHQ was prepared by dissolving 0.0166 g TBHQ in PBS (pH 7.0) + ethanol (1:1 v/v) (10 mL) under ultrasonication to 10 min. SWCNTs were purchased from Sigma-Aldrich Company and used to fabricate SWCNTs/PE electrodes. The orthophosphoric acid (85 %) was purchased from Merck Company and used to prepare phosphate buffer solution (PBS, 0.1 M).

Fabrication of SWCNTs/PE

The ratio of SWCNTs was prepared by mixing 0.15 g SWCNTs + 0.85 g graphite powder in the presence of paraffin oil as a binder. The powder dissolved in 10 mL diethyl ether solution and then stirred at 25 °C to evaporate the solvent. Then paraffin oil was added dropwise and the sample was hand-mixed for 45 min. The SWCNTs/PE was added to the end of the glass tube and an electrical connection was established by a copper wire.

Real sample preparation

Orange and apple juices and soybean oil were selected as real samples to study the capability of SWCNTs/PE in monitoring TBHQ. The orange and apple juices were centrifuged for 20 min at 4000 rpm and then the solution was filtered to preparation of pure sample and diluted by PBS (pH 7.0). The soybean oil was extracted with 50 mL ethanol for 1 h and then was filtered to prepare a pure sample and diluted by PBS (pH 7.0). The standard addition was used as an analytical method.

Results and discussion

Optimization of SWCNTs

The ratio of SWCNTs catalyst to graphite powder is one of the important factors in the fabrication of SWCNTs/PE as a new sensor. Therefore, the oxidation signal of 200 µM TBHQ was recorded at the surface of the carbon paste electrode (CPE) modified with 0, 5, 10, 15 and 20 wt.% SWCNTs. The results showed maximum sensitivity in the presence of 15 wt.% of SWCNTs in SWCNTs/PE matrix (Figure 1).

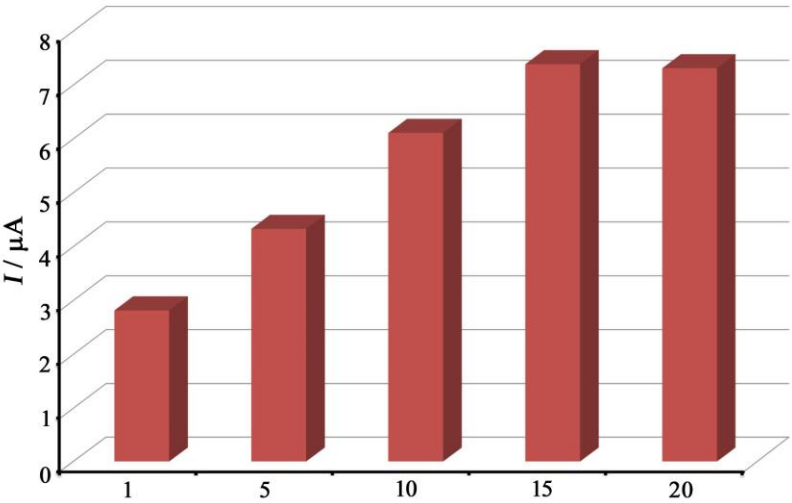


Figure 1. *Recorded oxidation current of TBHQ at surface of paste electrode modified with different percentage of catalyst*

Electrochemical investigation

The oxidation signal of TBHQ was recorded at a pH range of 5.0 to 9.0 and results showed in Figure 2 inset. The plot of the oxidation current of TBHQ *vs.* pH is shown in Figure 2 and clearly confirms maximum sensitivity at neutral conditions. On the other hand, the plot of the oxidation peak potential vs. pH follows the linear equation E = -0.057 pH + 0.784 (R^2 =0.9942) (not shown). Therefore, the reaction mechanism shown in Scheme 1 was suggested for the redox reaction of TBHQ at the surface of SWCNTs/PE [44].

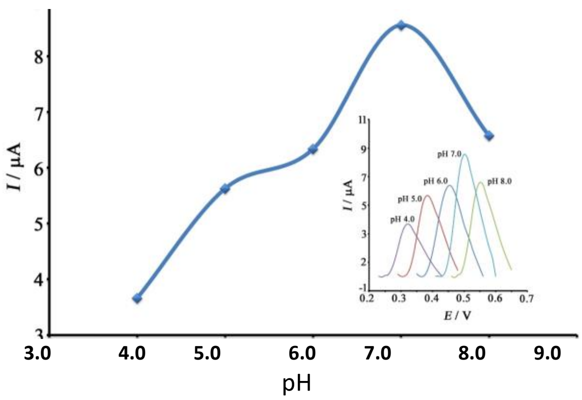
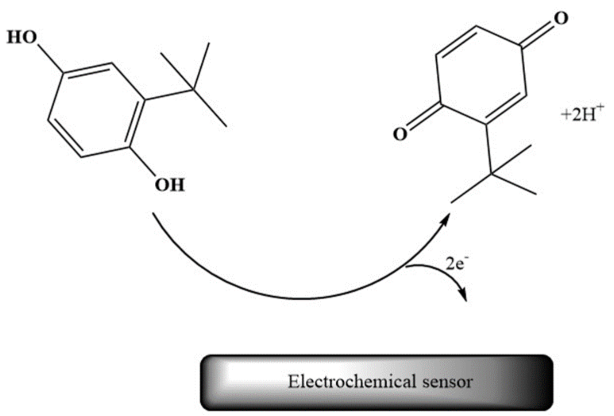


Figure 2. *I-pH curve for electrooxidation of 220 µM TBHQ at surface of SWCNTs/PE. Inset: DPV of 220 µM TBHQ at surface of SWCNTs/PE in pH range 4.0 to 9.0*



Scheme 1. *TBHQ electrooxidation mechanism*

The oxidation signal of 200 µM TBHQ was recorded at the surface of PE (Figure 3 curve a) and SWCNTs/PE (Figure 3 curve b), respectively. The increase in current from 2.816 to 7.386 µA and the decrease in potential from 550 mV to 500 mV are the main advantages of SWCNTs as catalysts at the surface of the paste electrode. On the other hand, the active surface area of PE and SWCNTs/PE were calculated at about 0.13 and 0.19 cm^2, respectively. This issue clearly confirms the high electrical conductivity of SWCNTs as a catalyst for the electrooxidation of TBHQ.

A positive shift was observed in the oxidation potential of TBHQ with the increase in the scan rate (see Figure 4 inset), revealing kinetic limitations and quasi-reversible behavior in the redox reaction of TBHQ at the surface of SWCNTs/PE. In addition, a linear dependence of the current peak height and the square root of the scan rate was observed, $I = 1.5580\,\nu^{1/2} + 2.6282$, confirming a diffusion-controlled electrooxidation process (Figure 4) [45].

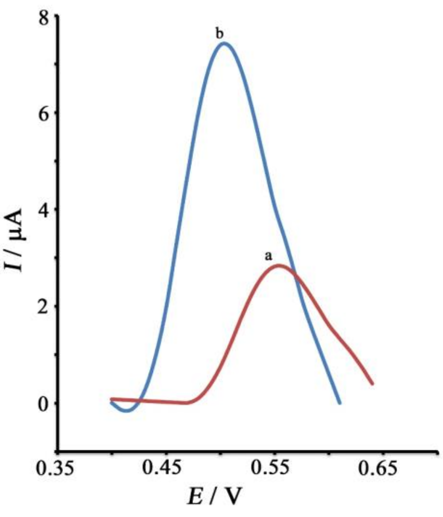


Figure 3. *DPV 200 µM TBHQ at surface of PE (a) and SWCNTs/PE (b)*

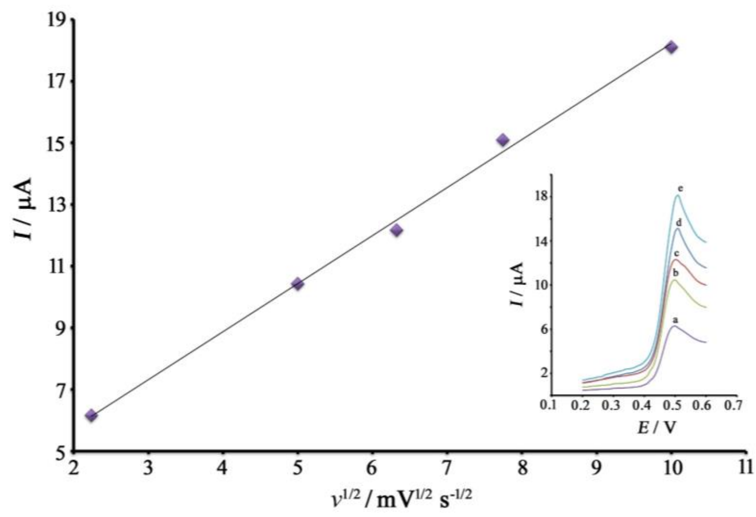


Figure 4. *I-*$\nu^{1/2}$ *curve to redox reaction of TBHQ at the surface of SWCNTs/PE. Inset: linear sweep voltammograms TBHQ at scan rates a) 5; b) 25; c) 40; d) 60 and e) 100 mV* s^{-1}

The stability of SWCNTs/PE in monitoring 200 µM TBHQ was investigated over 70 days. Results are shown in Figure 5 and data showed good stability of SWCNTs/PE in monitoring TBHQ in an aqueous solution after 60 days.

The differential pulse voltammograms (DPV) of TBHQ in the concentration range 0.05 to 390 µM were recorded at the surface of SWCNTs/PE and results are shown in Figure 6 inset. Results displayed a linear equation $I = 0.0325C + 0.8047$ ($R^2 = 0.9974$) where C is concentration. The limit of detection 10 nM ($Y_{LOD} = 3S_b/m$ where S_b is the standard deviation of blank solution and m is sensitivity or slope of LDR plot) was calculated for SWCNTs/PE as a new sensor.

In the final step, the standard addition results relative to monitoring of TBHQ using SWCNTs/PE are shown in Table 1. The experiments were repeated three times and mean values were displayed in the table. Results clearly confirm the powerful ability of SWCNTs/PE in sensing TBHQ in orange juice and soybean oil samples.

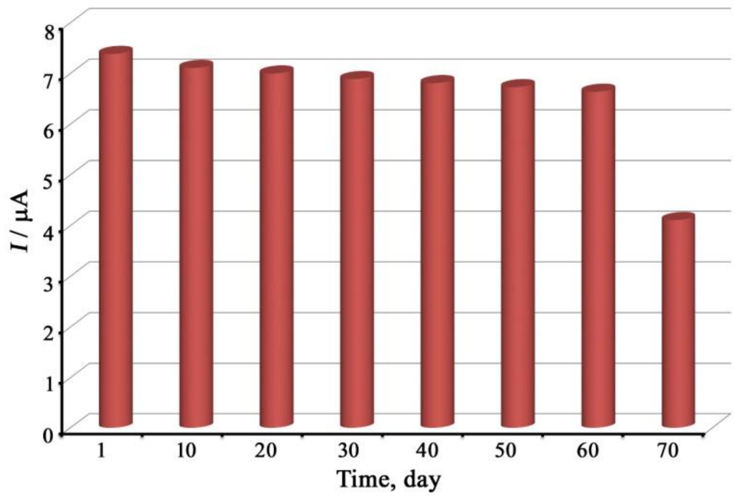


Figure 5. *Diagram of oxidation current of 200 µM TBHQ in period of 70 days at the surface of SWCNTs/PE*

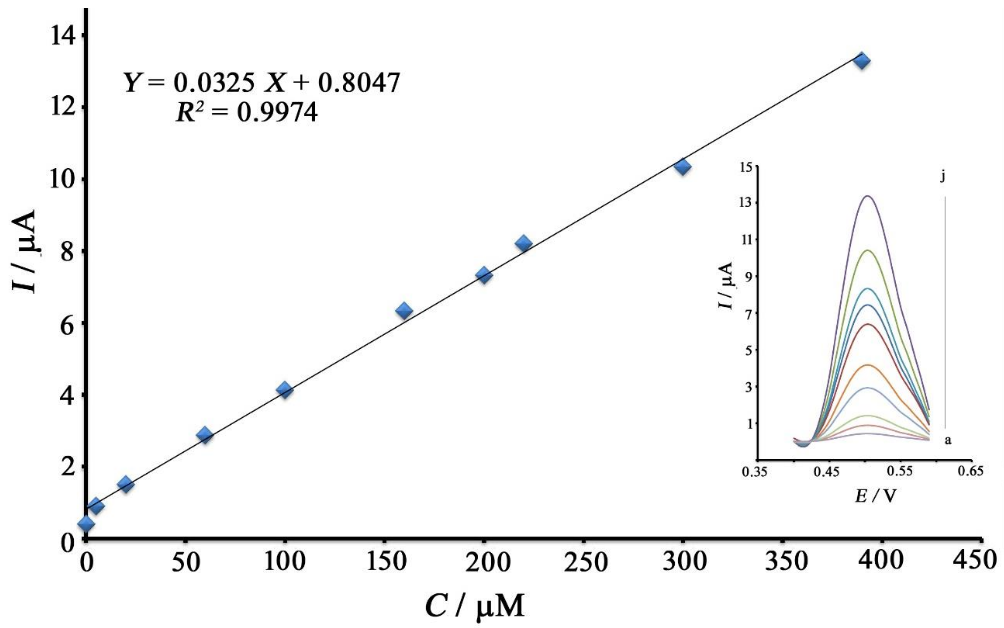


Figure 6. *LDR plot to sensing of TBHQ using SWCNTs/PE as sensor. Inset: DP voltammograms TBHQ in the concentration range 0.05 to 390 µM (from a to j)*

Table 1. *Monitoring of TBHQ in food samples*

Sample	Amount of TBHQ, µM			Recovery, %
	Added	Expected	Found by proposed method	
Soybean oil	---	---	1.86 ± 0.22	---
	5.00	6.86	6.98±0.44	101.74
Orange juice	---	---	<LOD	---
	10.00	10.00	9.72±0.73	97.2
Apple juice	---	---	<LOD	---
	20.00	20.00	20.86±0.99	104.3

Conclusions

The presence study suggested a very attractive and capable electroanalytical sensor to monitor TBHQ in food products. The new sensor was fabricated using the modification of a paste electrode with SWCNTs as a conductive and powerful catalyst. The SWCNTs/PE showed catalytic activity to sensing of TBHQ by reducing 50 mV oxidation potential and increasing the 2.62-time oxidation potential of antioxidants compared to the unmodified electrode. The SWCNTs/PE showed a good limit of detection (10 nM) for monitoring TBHQ in an aqueous solution that is sufficient for sensing this antioxidant in real samples. The SWCNTs/PE showed a recovery range of 97.2 to 104.3 % to sensing of TBHQ in food products.

Acknowledgment: *This is thesis of Niloofar Dehdashtian, who was inducted in Ayatollah Amoli Branch, Islamic Azad University, Amol, Iran.*

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