Article

Fabrication of a Food Nano-Platform Sensor for Determination of Vanillin in Food Samples

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Abstract: Herein, we describe the fabrication of NiO decorated single wall carbon nanotubes (NiO-SWCNTs) nanocomposites using the precipitation method. The synthesized NiO-SWCNTs nanocomposites were characterized by X-ray diffraction (XRD) and Transmission electron microscopy (TEM). Remarkably, NiO-SWCNTs and 1-butylpyridinium hexafluorophosphate modified carbon paste electrode (CPE/NiO-SWCNTs/BPrPF6) were employed for the electrochemical detection of vanillin. The vanillin sensor showed an ultra-high sensitivity of 0.3594 µA/µM and a low detection limit of 0.007 µM. In the final step, the NiO-SWCNTs/BPrPF6 was used as the suitable tool for food analysis.

Keywords: vanillin; NiO-SWCNTs nanocomposites; 1-butylpyridinium hexafluorophosphate

1. Introduction

The food analysis is an important strategy for the investigation of food quality [1]. The forbidden additives must be checked by an analytical sensor before consuming by customer [2]. Ensuring the safety of food can be checked by the analysis of food compounds. Although numerous analytical methods are available to analyze foods—including gas chromatography [3], capillary electrophoresis [4], spectrophotometry [5], resonance Raman spectroscopy [6], high-performance liquid chromatography [7], and electrochemical sensors [8–13]. However, electrochemical sensors are better suited for this goal due to portable ability, fast response, easy operation, and low cost [14–20]. Recently, chemically modified sensors improved on the ability of electrochemical methods for analysis of trace amounts of food or other electro-active materials [21–31]. With the growth of new nanomaterials and their unique properties [32–34], the electrochemical sensors showed better ability for determination of electroactive compounds, and especially, food products [35–40]. In addition, the coupling of nanomaterials with other conductive mediators showed a powerful ability for trace level analysis of electroactive materials [41–45].

Vanillin is a natural phenolic product with a great smell that is extensively used in food and pharmaceutical products. This phenolic product can be synthesized by chemical methods. The high level of vanillin in food or pharmaceutical products can cause an increased risk of allergic reactions and so the control of its level is very important in food and pharmaceutical samples [46].

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In this research, a CPE/NiO-SWCNTs and 1-butylpyridinium hexafluorophosphate modified carbon paste electrode (CPE/NiO-SWCNTs/BPrPF\(_6\)) is employed for the electrochemical detection of vanillin in food samples. The analytical ability of CPE/NiO-SWCNTs/BPrPF\(_6\) to determine the quantity of vanillin is compared to that of recently developed technologies which use electrochemical sensors (see Table 1). In addition, the proposed sensor showed other advantages compared to previous suggested sensors such as easy preparation, low cost, and high sensitivity.

**Table 1.** The analytical data obtained by some previous voltammetric sensors for vanillin determination.

| Electrode                  | Mediator                                      | pH   | LDR (µM)  | LOD (µM) | Ref.  |
|----------------------------|-----------------------------------------------|------|-----------|----------|-------|
| carbon paste               | CdO/SWCNTs and ionic liquid                   | 6.0  | 0.03–1200 | 0.009    | [47]  |
| carbon paste               | CuFe\(_2\)O\(_4\) nanoparticles and ionic liquid | 7.0  | 0.1–700   | 0.07     | [48]  |
| glassy carbon              | AuPd nanoparticles–graphene                  | 0.1 M H\(_2\)SO\(_4\) | 0.1–40   | 0.02     | [49]  |
| boron-doped diamond        | anodically pre-treated                        | 2.5  | 3.3–9.8   | 0.167    | [50]  |
| acetylene black paste      | graphite–polyvinylpyrrolidone                 | 0.1 M H\(_3\)PO\(_4\) | 0.02–400 | 0.01     | [51]  |
| carbon paste               | NiO-SWCNTs and ionic liquid                  | 6.0  | 0.01–350  | 0.007    | This work |

2. Materials and Methods

Vanillin, mineral oil, nickel nitrate hexahydrate, graphite powder, sodium hydroxide, single wall carbon nanotubes-COOH, phosphoric acid, diethyl ether, and sulfuric acid were obtained from Sigma-Aldrich. For experimental investigation, a stock standard solution of vanillin (10 mM) was prepared daily by dissolving 0.038 g vanillin in 25 mL water solution.

The electrochemical study was performed using the PGSTAT 302 N system. TEM (Philips CM30, 300 kV) and X-ray powder diffraction instruments were used for the investigation of NiO-SWCNTs structure and morphology.

The NiO-SWCNTs were synthesized according to our previous recommended procedure—the chemical precipitation method with SWCNTs-COOH, nickel nitrate hexahydrate, and sodium hydroxide as precursors [52].

2.1. Preparation of CPE/NiO-SWCNTs/BPrPF\(_6\)

CPE/NiO-SWCNTs/BPrPF\(_6\) were prepared by mixing 0.95 g of graphite powder and 0.05 g of NiO-SWCNTs in the presence of an appropriate amount of mineral oil and 1-butylpyridinium hexafluorophosphate until a uniformly wetted paste was obtained. The paste was input into the end of a glass tube in the presence of copper wire as a conductive binder.

2.2. Preparation of Real Sample

Coffee, milk, biscuit, and chocolate, were purchased and used for checking the ability of NiO-SWCNTs/BPrPF\(_6\) to perform vanillin analysis in real samples. Ten real samples were obtained from the local market and were ground using a mortar and pestle. Half a gram of powder or 0.5 mL coffee was transferred in 5 mL ethanol solution and then sonicated for 1.0 h. The obtained samples, including the vanillin extract, were centrifuged (3000× g rpm) for 50 min and directly used for determination of vanillin by standard addition method.

3. Results

3.1. NiO-SWCNTs Morphological and Structure Investigation

The XRD pattern of NiO-SWCNTs are presented in Figure 1 and the results confirmed the FCC structure for the NiO nanoparticle with a spherical shape and also the presence of a layer with miller index (002) at 2\(^{\circ}\)~26\(^{\circ}\) confirmed the presence of single wall carbon nanotubes. The TEM image of NiO-SWCNTs matches the XRD results. The NiO nanoparticle decorated the surface of single wall of carbon nanotubes (Figure 1 insert).
3.2. Electrochemical Behavior of Vanillin at the Surface of the Proposed Sensor

The electrochemical behavior of vanillin at different pH values was investigated by the linear sweep voltammetric method (Figure 2 insert). The oxidation potential shifted to a negative value with increasing pH and the plot of E vs. pH showed a linear relation with the equation of $E = -0.0639 \times \text{pH} + 1.1064$. As can be seen, the slope of E vs. pH is near to the Nernst equation for equal value of electron and proton (see the Scheme 1).

Figure 2. The Ep. vs. pH curve for electro-oxidation of 350 µM vanillin. Insert the linear sweep voltammograms of 700 µM vanillin at a surface of CPE/NiO-SWCNTs/BPrPF$_6$ at 4.0 < pH < 8.0.
The maximum value of current for electro-oxidation of vanillin occurred at pH = 6.0 and this condition was selected for the next steps.

The linear sweep voltammograms of vanillin at the surface of the CPE/NiO-SWCNTs/BPrPF$_6$ (curve a), CPE/BPrPF$_6$ (curve b), NiO-SWCNTs (curve c), and CPE (curve d) was recorded (Figure 3). With moving of CPE to NiO-SWCNTs/BPrPF$_6$, the oxidation signal of vanillin increased and the oxidation potential of vanillin decreased. This phenomenon can be attributed to the presence of NiO-SWCNTs and CPE/BPrPF$_6$ at a surface of the carbon paste electrode. The NiO-SWCNTs and CPE/BPrPF$_6$ improved the oxidation current of vanillin ~11.9 times and decreased the oxidation overpotential of vanillin by approximately 50 mV.

**Figure 3.** Linear sweep voltammograms of 800 μM vanillin at a surface of (a) CPE/NiO-SWCNTs/BPrPF$_6$, (b) CPE/BPrPF$_6$, (c) CPE/NiO-SWCNTs, and (d) CPE.

The linear relation between oxidation current of vanillin and $v^{1/2}$ (Figure 4) confirm the diffusion process for electro-oxidation of vanillin at a surface of CPE/NiO-SWCNTs/BPrPF$_6$. The oxidation potential of vanillin shifted to a positive value with increasing in-scan rates that confirm an irreversible process for electro-oxidation of vanillin (Figure 4 inert).
The plot of current vs. $\nu^{1/2}$ for electro-oxidation of vanillin at a surface of CPE/NiO-SWCNTs/BPrPF. Insert the linear sweep voltammograms of vanillin at a surface of CPE/NiO-SWCNTs/BPrPF at scan rates of (a) 10.0; (b) 20.0; (c) 30.0; (d) 60.0; and (e) 100 mV/s.

The value of diffusion coefficient (D) was determined by obtained data from chronoamperometric investigation (Figure 5A).

Using the slopes from Figure 5B and Cottrell equation (Equation (1)), we determined the value of $D \approx 3.57 \times 10^{-6}$ cm$^2$ s$^{-1}$.

$$I = nFAD^{1/2} \pi^{1/2} t^{1/2}$$

(1)

The square wave voltammetric method was used for investigation of the linear dynamic range and limit of detection of vanillin at a surface of CPE/NiO-SWCNTs/BPrPF (Figure 6 inset). We detected a linear dynamic range 0.01–350 μM with a detection limit of 0.007 μM (LOD = 3S$_B$/m) for vanillin at a surface of CPE/NiO-SWCNTs/BPrPF (Figure 6).
The selectivity of CPE/NiO-SWCNTs/BPrPF$_6$ for determination of vanillin was checked by an acceptable error of 5% in current (the obtained currents were compared before and after the addition of interference). The 1000-fold of K$^+$, Na$^+$, Cl$^-$, glucose, and 300-fold of folic acid, vitamin B$_6$, vitamin B$_1$, and tartrazine had no influence on the determination of vanillin.

The ability of CPE/NiO-SWCNTs/BPrPF$_6$ was checked for determination of vanillin in coffee milk, biscuit, and chocolate samples. The results are presented in Table 2. According to the results in Table 1, the CPE/NiO-SWCNTs/BPrPF$_6$ was suggested as a powerful sensor for vanillin analysis in food samples.

### Table 2. Determination of vanillin in real samples (n = 4).

| Sample      | Added (µM) | Expected (µM) | Founded (µM) | Recovery % |
|-------------|------------|---------------|--------------|------------|
| Coffee milk | —          | 10.00         | 4.12 ± 0.44  | —          |
|             | 10.00      | 14.12         | 14.43 ± 0.65 | 102.19     |
| Chocolate   | 10.00      | 11.95         | 11.75 ± 0.59 | 98.32      |
| Biscuit     | 10.00      | 14.56         | 14.98 ± 0.87 | 102.88     |

### 4. Conclusions

This work described fabrication of a highly sensitive and new sensor for determination of vanillin in food samples. The presence of NiO-SWCNTs and BPrPF$_6$ at a surface of a carbon paste electrode improved the ability of the sensor for analysis of vanillin at the nanomolar level. The NiO-SWCNTs and CPE/BPrPF$_6$ improved the oxidation current of vanillin ~11.9 times and decreased the oxidation overpotential of vanillin by ~50 mV. The CPE/NiO-SWCNTs/BPrPF$_6$ showed a powerful ability for determination of vanillin in food samples such as coffee milk, biscuit, and chocolate.

**Author Contributions:** This work is part of the Ph.D. thesis of M.B., F.K. (synthesis part) and M.B. (electrochemical part) conducted the experimental portion together. H.K.-M. and M.F. are supervised the thesis, and analyzed and obtained the data. S.-A.S. was the advisor of the thesis and helped us with the preparation of the real samples. V.K.G. wrote the paper and helped us with analysis of the data. S.A. helped characterize of the synthesized nanomaterials and helped for one part of the electrochemical investigation.

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