



Tannic acid functionalized N-doped graphene modified glassy carbon electrode for the determination of bisphenol A in food package



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ABSTRACT

A rapid, environmental friendly, and sensitive sensor for the detection of bisphenol A (BPA) was developed at glassy carbon electrode (GCE) modified with Tannic acid functionalized N-doped graphene (TA/N-G) immobilized by Nafion. Compared with other sensors, the proposed sensor greatly enhanced the response signal of BPA due to the active surface area of N-G and high absorption efficiency of TA. Under the optimal conditions, the oxidation current increased linearly with increasing the concentration of BPA in the range of 0.05–13 μM with the detection limit of 4.0 nM. The fabricated electrode showed good reproducibility, stability and anti-interference. The developed electrochemical sensor was successfully applied to determine BPA in food package.

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1. Introduction

Bisphenol A is an indirect food additive which is used as a material for the production of phenol resins, polyacrylates and polyesters but mainly for the production of epoxy resins and polycarbonate plastics. Epoxy resins are used in engineering applications, in paints and adhesives, and in a variety of protective coatings in metal cans for foods, bottle tops, and water supply pipes. The polycarbonate plastics are used to make a variety of common products including baby bottles and water bottles, medical and dental devices, eyeglass lenses, and household electronics, because they have high impact strength, hardness, toughness, transparency and resistance to many acids and oils [1]. Unfortunately, BPA has been known to be leached from these plastics, especially those that are cleaned with harsh detergents or those that contain acidic or high-temperature liquids. The excessive level of bisphenol A leads to coronary heart disease and diabetes [2,3]. The United States Environmental Protection Agency has set a maximum acceptable level of bisphenol A as 0.05 mg kg^{-1} body weight per day [4]. Therefore, the development of new sensors for the detection of BPA at trace concentrations has become one of the most attractive subjects of investigation in analytical chemistry. There have been various detection techniques, such as fluorescence, liquid chromatography

(HPLC), liquid chromatography coupled with mass spectrometry (LC-MS), gas chromatography (GC), gas chromatography coupled with mass spectrometry (GC-MS), enzyme linked immunosorbent assays (ELISA) and electrochemical sensors, are investigated to detect and determine BPA [5–10]. BPA is electrochemically active, but direct determination of BPA using traditional electrochemical sensors is very poor because the electrochemical oxidation of BPA may cause the inactivation of carbon or noble metal electrodes due to the deposition of electro-polymerized films [11]. In addition, BPA oxidation also involves a relatively higher over potential, which increases the interference of determination, leading to a low selectivity and sensitivity [12]. Therefore, there is still a challenge to develop novel electrode modification materials with excellent conductivity, good catalytic activity and high stability.

N-doped graphene (N-G) has a better electron transfer efficiency and a larger amount of edge sites than pristine graphene [13,14], so it has been applied in many fields including supercapacitor electrodes, electrocatalysis, and electrochemical biosensors [15–17]. Tannic acid (TA) is a high molecular weight polyphenolic compound containing a central carbohydrate (glucose) core, which is esterified into ester by phenol acids (gallic acid). It may adsorb onto individual graphene, and thus alter their surface physicochemical properties and enhance their stabilization in water. Recently, the *p-p* interactions between the aromatic rings of TA and graphene oxide (GO) sheets have been used to disperse GO sheets in aqueous medium successfully [18].

In this work, a facile and green method was employed to synthesize the TA/N-G nanocomposite for an electrochemical

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sensor. The working electrode used was a glass carbon electrode (GCE) modified with the TA/N-G nanocomposite immobilized by Nafion. The oxidation response of BPA was enhanced clearly, thought to be due to the large active surface area and high absorption efficiency of the TA/N-G nanocomposite. Moreover, the fabricated sensor had preferable stability, the modification process was simple, and the preparation of the sensor had a lower cost. Therefore, a novel electrochemical method was proposed for the determination of BPA, which was successfully demonstrated with analysis of food package samples.

2. Experimental

2.1. Chemicals and reagents

N-doped graphene (N content of 3.0–5.0 at%) was purchased from Nanjing XFNANO Materials Tech Co., Ltd. Tannic acid (TA) and Nafion (5 wt% solution in a mixture of distilled water) were obtained from Sangon Biotech Co., Ltd. (Shanghai). Other reagents were of analytical grade or better quality, which were purchased from J&K Chemical Ltd. (Shanghai, China). Milli-Q ultrapure water (Millipore, $\geq 18.2 \text{ M}\Omega \text{ cm}$) was used throughout.

The electrochemical experiments were performed on a CHI660C Electrochemical Analyzer (Chenhua Instruments, Shanghai, China). A three-electrode system was employed with a GC electrode (3 mm inner diameter, Tianjing Incole Union Technology Co., Ltd.) or modified electrodes as the working electrode. A saturated calomel electrode (SCE) and a platinum wire served as the reference electrode and counter electrode, respectively. The size and morphology of N-G and TA/N-G were measured using an S-4800 field-emission scanning electron microscope (Hitachi, Japan) for obtaining the scanning electron microscopy (SEM) images.

2.2. Preparation of TA/N-G and TA/N-G/Nafion/GCE

In a typical experiment, 0.5 mg of N-G was dispersed in 2 mL of 5 mg L^{-1} TA aqueous solution under ultrasonication for 30 min. Then the excess TA was discarded by centrifugation and the as-prepared TA/N-G was dissolved into 500 μL of water.

Before modification, the bare GCE was polished with 0.3 and 0.05 μm alumina particles on chamois leather in sequence, then washed sequentially with doubly distilled water in an ultrasonic bath and dried by N_2 before use. For preparation of TA/N-G/Nafion modified electrode, 0.5% Nafion solution was added into the 4.5 mg mL^{-1} TA/N-G solution, followed by ultrasonication for 5 min. With a microinjector, 5 μL of TA/N-G/Nafion solution was deposited on the freshly prepared GCE surface, and kept in the air

at room temperature. The obtained modified electrode was noted as TA/N-G/Nafion/GCE.

2.3. Real samples preparation

The polycarbonate (PC) food package was purchased from a local supermarket. These samples were cut into small pieces and washed five times with 50 mL Millipore water. Then, 1.0 g of plastic sample and 30 mL of ethanol were kept in a flask and stirred at 55°C for 4 h in order to extract BPA. After cooling to room temperature, the mixture was filtered with 0.45 μm filter membrane. Then the solvent was evaporated under reduced pressure. The resulting product was finally dissolved with 1 mL ethanol. After the sample solutions were prepared, the liquid phase was collected in a 50 mL volumetric flask and diluted to volume with 0.1 M phosphate buffer (PBS, pH 6.0), and was determined with TA/N-G/Nafion/GCE. Otherwise, the spiked sample solution was prepared as same method after adding known-amount of BPA standard solution.

3. Results and discussion

3.1. Morphological and spectra characterization of the N-G and TA/N-G nanocomposites

Fig. 1 shows the SEM of N-G and TA functionalized N-G. Although N-G contains some surface functional groups, such as hydroxyl and carbonyl groups, it can be partly dispersed in water by ultrasonication. After several hours, many graphene sheets may still be close-packed. When TA was added, the adsorption of TA made the surface of N-G positively charged, leading to a good dispersion *via* electrostatic repulsion among individual N-G sheets. Compared with Fig. 1A and B, it is evident that TA is highly dispersed and uniformly distributed on N-G. Fig. 1 inset shows photographs of the corresponding samples and a distinct N-G precipitates at the bottom of the vial in the absence of TA after 3 h.

The successful preparation of TA/N-G nanocomposites was confirmed by UV-vis absorption spectra, as shown in Fig. 2. The UV-vis spectrum of TA shows two peaks at 214 and 274 nm (curve a), which are assigned to $p-p^*$ and $n-p^*$ transition, respectively [18]. Taken together, the UV-vis spectrum of N-G shows two peaks at 230 and 300 nm (curve b). In the suspension of TA/N-G, the $n-p^*$ absorbance peak of TA was blue shifted to 268 nm, as shown in curve c, indicating the presence of $p-p$ interaction between the aromatic rings of TA and N-G. The good stability of the TA/N-G in water could be mainly ascribed to the good biocompatibility and the strong interactions of the composite system.

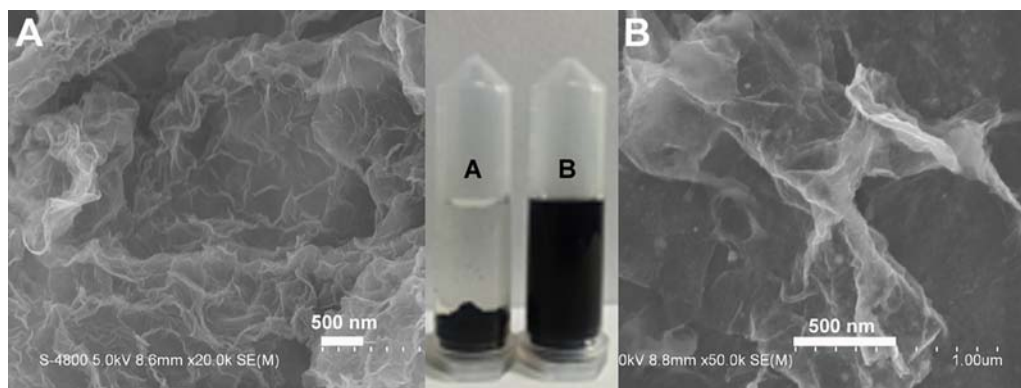


Fig. 1. SEM images of N-G and TA/N-G. (Inset shows photographs of corresponding samples).

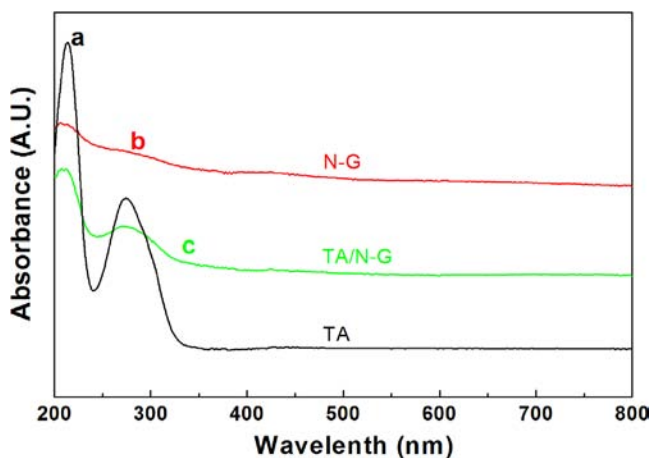


Fig. 2. UV-vis absorption spectra of aqueous dispersions of TA (a), N-G (b), TA/N-G (c).

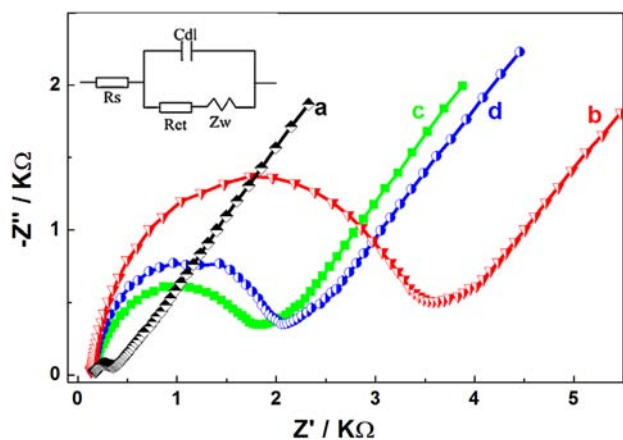


Fig. 3. Nyquist plots of different electrodes in 5 mM $\text{Fe}(\text{CN})_6^{3-/4-}$ solution containing 0.1 M KCl. (a) GCE, (b) Nafion/GCE, (c) N-G/Nafion/GCE, and (d) TA/N-G/Nafion/GCE. The inset is the equivalent circuit.

3.2. Characterization of the sensor fabrication

For further characterization of the modified electrode, the electrochemical impedance spectroscopy (EIS) was used. Fig. 3 presents the Nyquist diagrams of bare GCE (a), Nafion/GCE (b), N-G/Nafion/GCE (c) and TA/N-G/Nafion/GCE (d) in 5 mM $[\text{Fe}(\text{CN})_6]^{3-/4-}$ containing 0.1 M KCl. The Nyquist plots were fitted using the Randles equivalent circuit (Fig. 3, inset) which takes into consideration the diffusion and kinetic control parameters. The Randles equivalent circuit used for fitting impedance data consisted of the solution or electrolyte resistance (R_s) connected in series to the parallel combination of the capacitance and electron transfer resistance (R_{et}) in series with Warburg impedance (Z_w). The simulated values of R_{et} for $[\text{Fe}(\text{CN})_6]^{3-/4-}$ at the bare GCE (a), Nafion/GCE (b), N-G/Nafion/GCE (c) and TA/N-G/Nafion/GCE (d) are 225, 3265, 1615 and 1875 Ω , respectively. It can be seen that a small well defined semi-circle at higher frequencies was obtained at the bare GCE, indicated small interface impedance. When Nafion was deposited on the surface of GCE, the impedance values was much larger than that at GCE, which could be attributed to the Nafion film itself, introducing a resistance into the electrode/solution system, leading to a lower rate of the electron transfer of $[\text{Fe}(\text{CN})_6]^{3-/4-}$. After N-G was added, N-G could accelerate the charge migration. When TA/N-G/Nafion was deposited on the surface of GCE, the impedance values changed

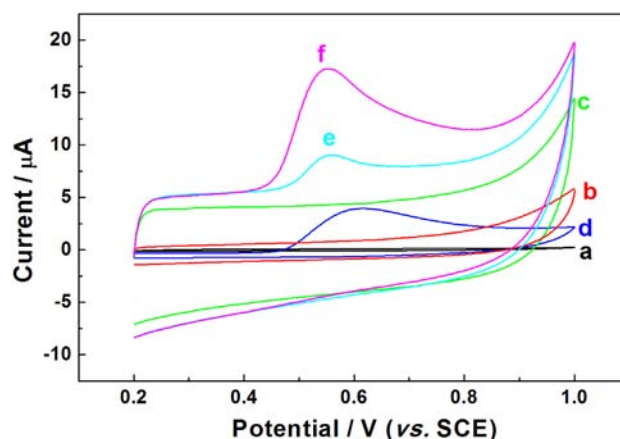


Fig. 4. Cyclic voltammograms of GCE ((a) and (d)), N-G/Nafion/GCE ((b) and (e)) and TA/N-G/Nafion/GCE ((c) and (f)) in the absence and presence of 0.05 mM BPA in 0.1 M PBS (pH 6.0) at scan rate 50 mV s^{-1} .

little larger. This also demonstrated that TA/N-G/Nafion was successfully immobilized on the GCE surface.

3.3. Cyclic voltammetric behaviours of BPA

Fig. 4 displays the cyclic voltammograms (CVs) obtained at GCE (a and d), N-G/Nafion/GCE (b and e), and TA/N-G/Nafion/GCE (c and f) in the absence (a–c) and present (d–f) of 0.05 mM BPA in 0.1 M phosphate buffer (PBS, pH 6.0). It is not difficult to believe that the oxidation of BPA at those electrodes is totally irreversible under the selected experimental conditions, which was in agreement with previous reports [19,20]. The oxidation current of BPA on N-G/Nafion/GCE (curve e) was higher than that at GCE and the oxidation potential (E_{pa}) was found to shift toward negative value, indicating that N-G had obviously catalytic activity towards the BPA oxidation. When TA/N-G/Nafion was immobilized on GCE surface, a higher current response for the oxidation of BPA was observed (curve f) compared with the bare GCE (curve d) and N-G/Nafion/GCE, which could be attributed to electrostatic adsorption between the positively charged TA and the negatively charged BPA and the synergistic effect of TA and N-G, leading to increase the determination sensitivity and decrease the limit of detection.

3.4. Effects of scan rate

Fig. 5 shows the CVs of 3.0 μM BPA at the TA/N-G/Nafion/GCE with different scan rate. It can be seen that the oxidation peak currents increased gradually with increase of the scan rate. As shown in the inset, the peak current increased linearly with the scan rate in the range of 20–100 mV s^{-1} , and can be expressed as follows: $I_{pa} (\mu\text{A}) = 0.1757v (\text{mV s}^{-1}) + 3.1612$ ($R = 0.9981$). It indicates that the oxidation of BPA on the TA/N-G/Nafion/GCE is an adsorption-controlled process. In this work, the scan rate was chosen as 50 mV s^{-1} .

3.5. Selectivity of pH and the effect of accumulation time

The effect of solution pH on the electrochemical response of the sensor toward the determination of the BPA was studied by cyclic voltammetry. The variations of peak current with respect to the change of the electrolyte in the pH range from 4.0 to 9.0 are shown in Fig. 6. It can be seen that the peak current increases with increasing of the solution pH from 4.0 to 6.0, and then decreases with a further increasing of pH, indicating that protons have taken part in the electrode reaction processes [21].

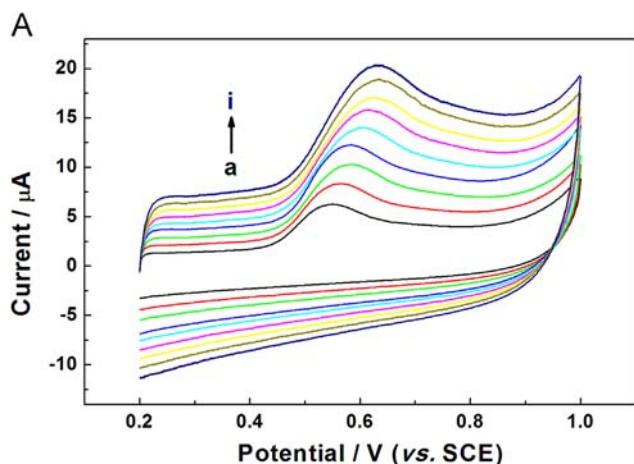


Fig. 5. Cyclic voltammograms of 0.05 mM BPA at TA/N-G/Nafion/GCE with different scan rates. Curve a–i is obtained at 20, 30, 40, 50, 60, 70, 80, 90, and 100 mV s^{-1} , respectively. Inset: the plot for the dependence of peak current on the scan rate.

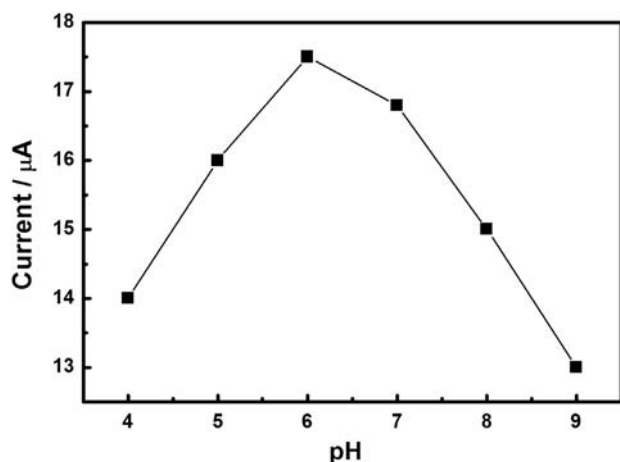


Fig. 6. Effect of pH value on the peak current of 0.05 mM BPA at TA/N-G/Nafion/GCE.

At pH 6.0, BPA ($pK_a=9.60\text{--}10.2$) would be present predominantly in molecular (non-dissociated form) while TA in anionic form (the dissociation degree of tannic acid is 0.93) [22,23]. TA possesses both aromatic carbon and hydroxyl functionalities, which have been shown to be crucial to the sorption of BPA [24]. So BPA could be adsorbed onto the surface of TA possibly through hydrogen bonding and van der Waals interactions involving the carbonyl groups of TA and the phenolic groups of BPA.

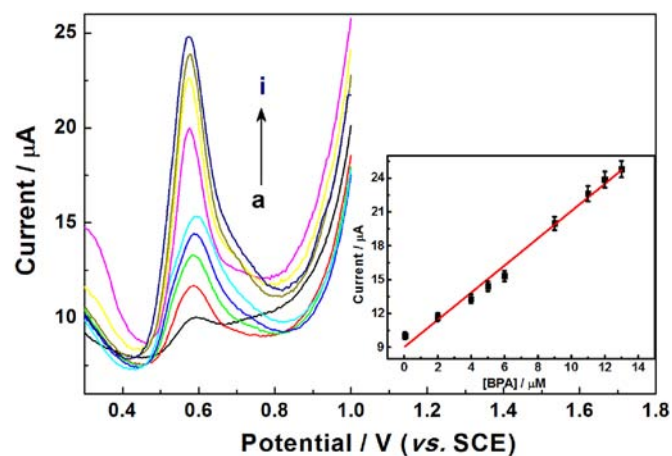


Fig. 7. Differential pulse voltammograms of BPA at TA/N-G/Nafion/GCE in 0.1 M PBS (pH 6.0) containing different concentrations of BPA (from a to i: 0.05, 2, 4, 5, 6, 9, 11, 12, 13 μM). Inset: Linear calibration curve in the range of 0.05–13 μM BPA.

Accumulation improves the surface amount of BPA and then enhances the oxidation signal of BPA. As further extending the accumulation time from 1 to 5 s, the oxidation peak current of BPA rapidly increases, suggesting that the surface amount of BPA tends to a limiting value. So, the sensitivity of the sensor does not improve when the accumulation time is beyond 5 s. Therefore, the accumulation time reduced largely and the oxidation peak current reached the maximum value within 5 s under stirring.

3.6. Calibration curve, linear range, and detection limit

Fig. 7 shows the typical differential pulse voltammograms (DPV) of TA/N-G/Nafion/GCE under optimum experimental conditions after successive addition of BPA with concentrations ranging from 0.05 to 1.3 μM into 0.1 M PBS (pH 6.0) under magnetic stirring. The modified electrode can achieve the steady-state current within 5 s, which is a very rapid response to the change of BPA concentration. As shown in the inset of Fig. 7, the calibration curve between the peak current (I) and the PBA concentration (c) can be described by the equation:

$$I(\mu\text{A}) = 1.4845c(\text{nM}) + 0.9327 (R = 0.9873)$$

The linear range is 0.05–1.3 μM PBA. The limit of detection (LOD) is estimated to be 4.0 nM at a ratio of signal to noise of 3. The enhanced performance of TA/N-G/Nafion/GCE can be attributed to the combination of the excellent electrocatalytic properties of N-G and the electrostatic adsorption of TA to BPA. A comparison of the TA/N-G/Nafion/GCE with other reported modified electrodes for BPA detection is listed in Table 1. As can be seen, the proposed method had comparative, even better sensitivity and linear range. Moreover, the electrode fabrication in present paper is much simpler.

3.7. Reproducibility, stability and interference

The fabrication reproducibility for ten TA/N-G/Nafion/GCE was carried out by comparing the oxidation peak current of 0.1 μM BPA. The relative standard deviation (RSD) was 4.6%, revealing that this method had good reproducibility. The stability of the electrode was also investigated by measuring the electrode response of 0.1 μM BPA every 5 days. Between measurements the electrode was stored at 4 $^{\circ}\text{C}$ in a refrigerator. The current response decreased to 97% after 10 days, while 94% of the original response retained after 15 days. On the other hand, in order to evaluate the selectivity of the sensor, the influence of some possible interfering

Table 1
Comparison of the proposed method with other sensors based on differential matrices.

Electrode	Method	Linear range (μM)	LOD (nM)	References
MWCNT–GNPs/GCE ^a	DPV ^f	0.02–20	7.5	[25]
NGS and CS/GCE ^b	Amperometry	0.01–1.3	5.0	[26]
Mesoporous silica ^c	DPV	0.2–8.8	38.0	[27]
PAMAM–Fe ₃ O ₄ /GCE ^d	Amperometry	0.01–3.07	5.0	[28]
MWCNTs–GNPs/GE ^e	Amperometry	0.113–8200	3.6	[29]
TA/N-G/Nafion/GCE	DPV	0.05–13.0	4.0	Present work

^a Multiwalled carbon nanotubes (MWCNTs)–gold nanoparticles (GNPs) hybrid film modified glassy carbon electrode (GCE).

^b Nitrogen-doped graphene sheets with chitosan modified glassy carbon electrode (GCE).

^c Mesoporous silica-based electrochemical sensor.

^d Poly(amidoamine) (PAMAM) and Fe₃O₄ magnetic nanoparticles modified glassy carbon electrode (GCE).

^e Multi-walled carbon nanotubes (MWCNTs) and gold nanoparticles (GNPs) modified gold electrode (GE).

^f Differential pulse voltammetry.

Table 2
Determination results of BPA in food package samples.

Samples	Measured (μM)		Added (μM)	Found (μM)	Recovery (%)
	By this method	By HPLC			
1	0.175	0.181	0.20	0.371	98.0
2	0.186	0.194	0.20	0.392	103
3	0.243	0.251	0.20	0.433	95.0
4	0.312	0.326	0.20	0.507	97.5
5	0.348	0.353	0.20	0.549	100.5

substances was examined in PBS solution (pH 6.0) containing 1 μM BPA. The results suggested that 100-fold concentration of phenol, hydroquinone, hydroxyphenol, pyrocatechol and 4-nitrophenol had no influence on the signals of BPA with deviations below 10%. Otherwise, some ions such as 500-fold concentration of Na⁺, Ca²⁺, Mg²⁺, Fe³⁺, Al³⁺, Zn²⁺, Cu²⁺, Cl⁻, SO₄²⁻, PO₄³⁻ and NO₃⁻ had no influence on BPA determination.

3.8. Real sample analysis

To demonstrate the suitability and potential application for sample analysis, this method was used to detect BPA in food package samples which were collected from local supermarkets. Table 2 shows the concentrations of BPA in different food package samples, which measured by the standard addition method. To further demonstrate the accuracy, high performance liquid chromatography (HPLC) was also employed. From the comparative results of two methods, we can see that the two methods are in good agreement. The recoveries of BPA standard added into the samples were in the range of 98.9–100.5%, suggesting that the recoveries of this method are satisfactory and the determination of BPA using the TA/N-G/Nafion/GCE is feasible.

4. Conclusions

In this investigation, a rapid, environmental friendly and sensitive electrochemical sensor for the detection of bisphenol A was constructed based on TA/N-G/Nafion nanocomposite modified GC electrode. The oxidation peak current of BPA was significantly enhanced at the as-prepared electrode. The reasons can be attributed to the catalytic activity and conductivity of N-G, and the high adsorption capacity to TA of BPA. The fabricated electrode not only displayed good electrocatalytic activity towards the oxidation of BPA, but also had an excellent long-term stability and quantitatively reproducible analytical performance. The experimental results demonstrated that the TA/N-G/Nafion/GCE is promising for the determination of BPA in real samples due to the high sensitivity, wide linear range and low detection limit.

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