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Bisphenol A Analysis in Food Samples Using Modified Nanostructure Carbon Paste Electrode as a Sensor

Vahid Arabali[1] · Mahmoud Ebrahimi[1] · Siamak Gheibi[2] · Fatemeh Khaleghi[3] ·
Majede Bijad[4] · Ali Rudbaraki[4] · Maryam Abbasghorbani[5] · M. R. Ganjali[6]

Abstract The electrochemical oxidation of bisphenol A has been studied by CdO nanoparticle ionic liquid carbon paste electrode. Cyclic voltammetry, square wave voltammetry, and chronoamperometry were used to investigate the suitability of new sensor for the electrooxidation of bisphenol A in aqueous solution. The electrooxidation of bisphenol A occurs at a potential about 50 mV less positive than with the unmodified carbon paste electrode at pH 7.0. The square wave voltammetry (SWV) peak currents of the electrode increased linearly with the corresponding bisphenol A concentration in the range of 0.3–650 μM with a detection limit of 0.1 μM. The influence of pH value and potential interfering substances on the determination of bisphenol A were studied. Finally, the proposed novel sensor was also examined as a sensitive, high selective, simple, and precise electrochemical sensor for the determination of bisphenol A in food samples.

✉ Mahmoud Ebrahimi
ebrachem2007@yahoo.com

✉ Fatemeh Khaleghi
khaleghi.ft@gmail.com

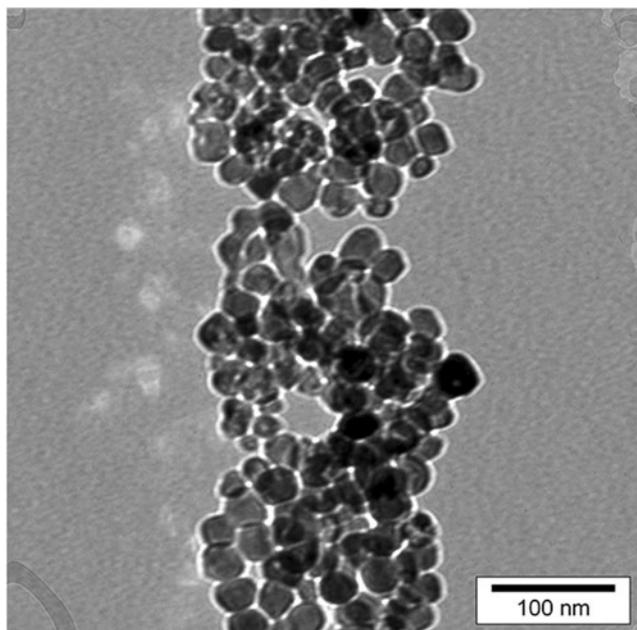1   Department of Chemistry, Mashhad Branch, Islamic Azad University, Mashhad, Iran

2   Department of Food Science & Technology, Faculty of Agricultural Sciences, University of Guilan, Rasht, Iran

3   The Health of Plant and Livestock Products Research Center, Mazandaran University of Medical Sciences, Sari, Iran

4   Department of Chemistry, Sari Branch, Islamic Azad University, Sari, Iran

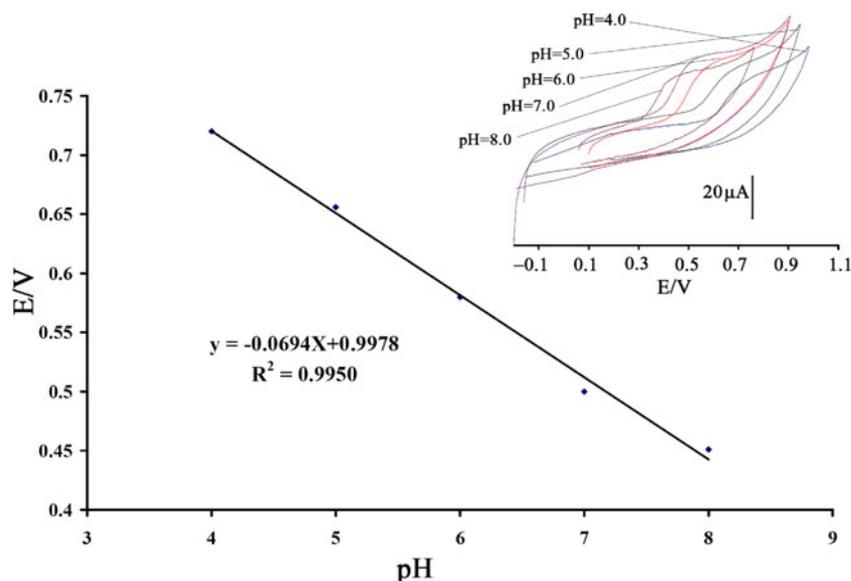5   Gas Division, Research Institute of Petroleum Industry, P.O. Box 14665-137, Tehran, Iran

6   Center of Excellence in Electrochemistry, Faculty of Chemistry University of Tehran, Tehran, Iran

Introduction

Bisphenol A is a chemical compound widely used to make epoxy resins and polycarbonate plastics. Polycarbonate-based plastics have many applications such as food and drink packaging and medical devices (Calafat et al. 2009). On the other hand, epoxy resins are used to metal coating such as bottle tops, food cans, and water supply pipes. Bisphenol A can leach into food produce from the epoxy resin lining of cans and from other products such as food storage containers, polycarbonate tableware, and water and baby bottles. The National Institute of Environmental Health Sciences expects to support more research to determine if bisphenol A in receipts poses a risk to human health. According to the above points, it is very important for preparation of fast and sensitive method for determination of bisphenol A in food samples. In comparison to other analytical methods in analysis, electrochemical based methods have attracted more attention in the recent years due to their high sensitivity, good accuracy, lower cost, high dynamic range, and simplicity (Goyal et al. 2010; Gupta et al. 1997, 2011a, b, c; Jain et al. 1997, 2010; Gupta et al. 2011a, b, c; Srivastava et al. 1995).

In the recent years, several developments in electrochemical investigation have contributed significantly to nanoscience (Sanghavi and Srivastava 2011; Yola et al. 2012, 2013; Karimi-Maleh et al. 2013). Electroanalytical methods are taking advantages from all the possibilities offered by nanomaterials (Sanghavi et al. 2013, 2014; Karimi-Maleh, et al. 2015; Atar et al. 2015; Norouzi et al. 2010).

Ionic liquids are a new class of purely ionic, salt-like materials that are liquid at unusually low temperatures (Anderson

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Fig. 1 TEM image of CdO nanoparticles

et al. 2006). Ionic liquids have some unique properties, such as wide electrochemical window and low vapor pressure (Chen et al. 2007). Transport properties play a key role in electrochemistry application. Ionic liquids have been recently used also in construction of electrochemical sensors (Bijad et al. 2013; Baghizadeh et al. 2015; Faridbod et al. 2009; Ganjali et al. 2009a, b; Sadeghi et al. 2013; Elyasi et al. 2013; Jamali et al. 2014; Pahlavan et al. 2014). Most of the novel sensors which are used in electrochemical analysis and have ability to modify with ionic liquids and nanomaterials are ion-selective electrodes based on polymeric membrane and all solid state electrodes especially carbon paste electrodes (Fouladgar et al. 2015; Zhang and Zheng 2008; Sun et al.

2008; Gupta et al. 2015; Ansari et al. 2013; Fouladgar and Karimi-Maleh 2013; Ganjali et al. 2009a, b; 2010; Musameh and Wang 2008; Haghighi and Hamidi 2009).

Here, we describe a cyclic voltammetry, chronoamperometry, and square wave voltammetric studies of bisphenol A at a CdO nanoparticle ionic liquid carbon paste electrode (CdO/NP/IL/CPE). Compared with the bare carbon paste electrode using nonconductive paraffin as the binder, the CdO/NP/IL/CPE exhibited high conductivity by using conductive n-hexyl-3-methylimidazolium hexafluoro phosphate and CdO/NPs for fabricating the electrode. The direct electrochemical behaviors of bisphenol A on this modified electrode were carefully investigated and further applied to the bisphenol A content detection in real samples with satisfactory results.
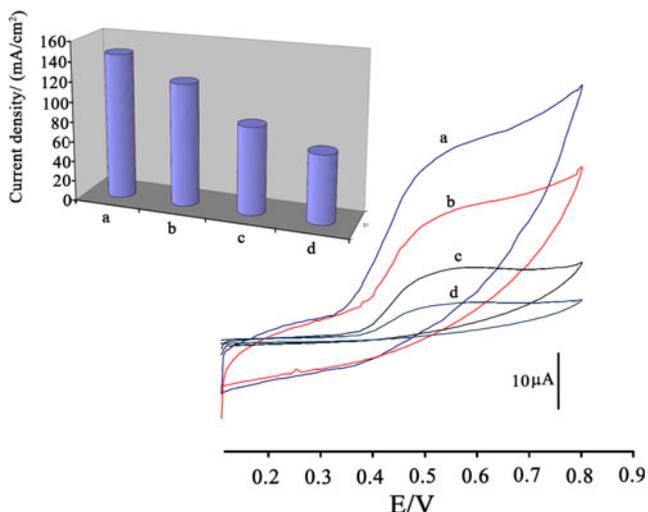
Experimental

Chemicals

All of the chemicals were of A.R. grade and were used as unless otherwise stated. Bisphenol A was procured from Sigma. Mineral oil and graphite powder (<50 μm) were obtained from Merck. All solutions were prepared using double distilled water having a specific conductivity of 0.5–0.8 μS.

Phosphate buffer solutions (sodium dihydrogen phosphate and disodium monohydrogen phosphate plus sodium hydroxide, 0.1 mol L^{-1}) (PBS) with different pH values were used.

Apparatus

All of the voltammetric investigations were performed using μ-Autolab, with potentiostat/galvanostat connected to a three-

Fig. 2 Plot of potential, *E*, versus pH for the electrooxidation of 500 μM bisphenol A at a surface of CdO/NP/IL/CPE. *Inset*: influence of pH on cyclic voltammograms of bisphenol A at a surface of the modified electrode

Fig. 3 Cyclic voltammograms of **a** CdO/NP/IL/CPE, **b** ionic liquid carbon paste electrode, **c** CdO nanoparticle carbon paste electrode, and **d** CPE in presence of 400 μM bisphenol A at a pH 7.0, respectively. *Inset*: the current density derived from cyclic voltammogram responses of 400 μM bisphenol A at pH 7.0 at the surface of different electrodes with a scan rate of 100 mV s^{-1} (surface area is 0.22 cm^2 for the CdO/ NP/IL/CPE, 0.18 cm^2 for ionic liquid carbon paste electrode, 0.14 cm^2 for CdO nanoparticle carbon paste electrode, and 0.09 cm^2 for the CPE)
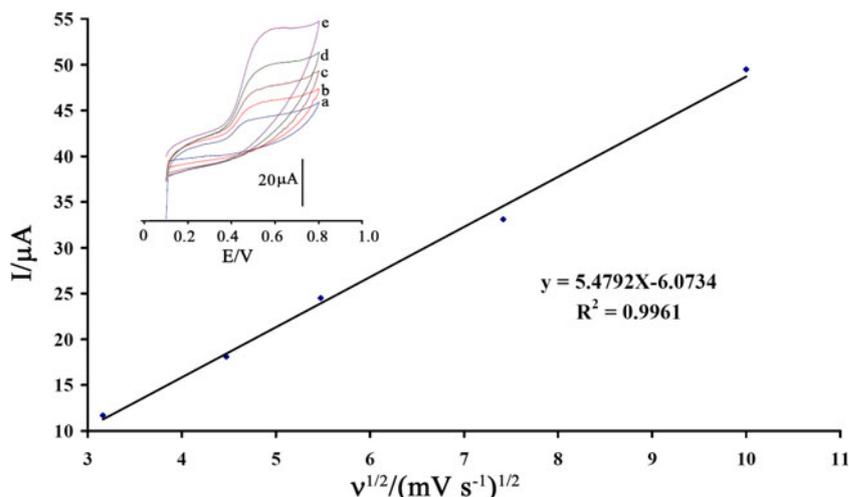
electrode cell, with Metrohm Model 663 VA stand, linked with a computer (Pentium IV), and with μ-Autolab software. The system was run on a PC using GPES and FRA 4.9 software. Three-electrode cell assembly consisting of a platinum wire as an auxiliary electrode and an Ag/AgCl (KCl$_{sat}$) electrode as a reference electrode was used.

Samples for transmission electron microscopy (TEM) analysis were prepared by evaporating a hexane solution of dispersed particles on amorphous carbon–coated copper grids.

Preparation of the Electrode

CdO/NP/IL/CPE was prepared by mixing of 0.10 g of ionic liquids, 0.90 g of the liquid paraffin, 0.05 g of CdO/NPs, and

0.95 g of graphite powder. Then, the mixture was mixed well for 60 min until a uniformly wetted paste was obtained. A portion of the paste was filled firmly into one glass tube as described above to prepare CdO/NP/IL/CPE. Unmodified carbon paste past was prepared by hand mixing of 1.0 g of graphite powder plus paraffin at a ratio of 70:30 (*w/w*) and mixed well for 40 min until a uniformly wetted paste was obtained.

Preparation of Real Samples

Ten cans of each group of different foods (tomato paste, tuna fish corn, stew, and water bottle), totaling 50 samples all bearing the same batch number, were purchased from retail outlets in Sari. Purchasing of the samples was carried out in June 2015. Samples were stored and sealed at room temperature. After opening the samples, the total contents of each sample were homogenized and an aliquot was taken for voltammetric analysis. The remaining contents of each sample were then frozen and stored in a freezer at 4 °C. On the other hand, water sample analysis without any pretreatment for real sample analysis was carried out.

Results and Discussion

CdO Nanoparticle Characterization

The morphology of the as-grown CdO nanostructures was characterized by TEM. Typical TEM micrograph of the synthesized CdO/NPs is shown in Fig. 1. Results confirm synthesis of CdO nanoparticles.

Voltammetric Investigation

According to previous report, the electrooxidation of bisphenol A is closely related to the pH value of solution

Fig. 4 Plot of I_{pa} versus $v^{1/2}$ for the oxidation of bisphenol A at CdO/NP/IL/CPE. Inset shows cyclic voltammograms of bisphenol A at CdO/NP/IL/CPE at different scan rates of **a** 10, **b** 20, **c** 30, **d** 55, and **e** 100 mV s^{-1} in 0.1 M phosphate buffer, pH 7.0

y = 5.4792X-6.0734
R^2 = 0.9961

Fig. 5 **a** Chronoamperograms obtained at CdO/NP/IL/CPE in the presence of (*a*) 400 and (*b*) 500 μM bisphenol A in the buffer solution (pH 7.0). **b** Cottrell's plot for the data from the chronoamperograms
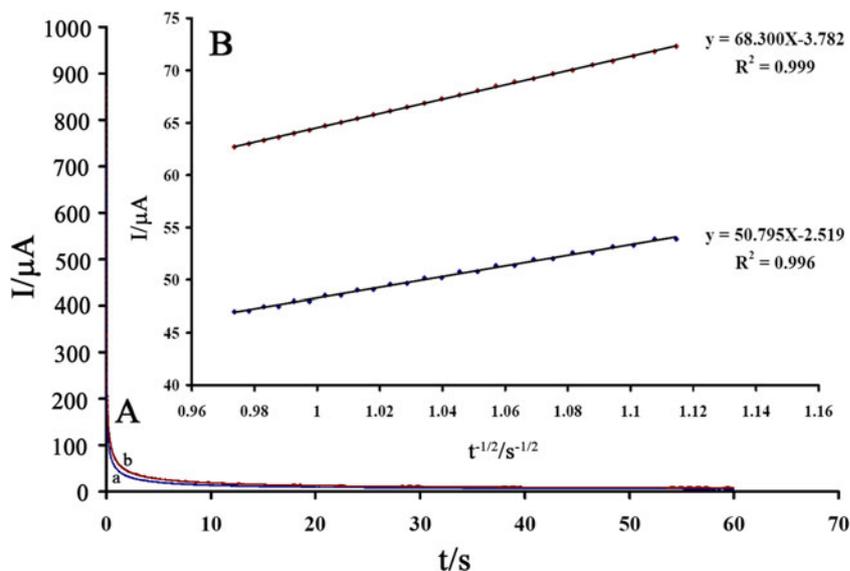

(Najafi et al. 2014). The effect of pH was investigated using cyclic voltammetry technique (Fig. 2 insert). Result shows that the electrooxidation peak current increased slowly from pH 4.0 to 7.0, and then the current conversely decreased when the pH value increased from 7.0 to 8.0. According to obtained results, the pH 7.0 was chosen as the best optimal experimental condition for other experimental. Also, we study the relationship between the oxidation peak potential of bisphenol A and pH. A linear shift of E_{pa} toward negative potential with an increasing pH can be obtained and obeyed the regression equation of E_{pa} (V)$=-0.0694$ pH$+0.9978$ ($R^2=0.9950$), which indicates that protons are directly involved in the oxidation of bisphenol A (Fig. 2). A slope of 69.4 mV/pH suggests that the number of electron transfer is equal to the proton number involved in the sensor reaction.

Figure 3 inset shows the current density derived from the cyclic voltammograms of 400 μM bisphenol A (pH 7.0) at the

surface of modified and unmodified electrodes with a scan rate of 100 mV s^{-1}. The results show that the presence of nanoparticles and ILs together causes the increase of the electrode. Figure 3 shows cyclic voltammograms of 400 μM bisphenol A at pH 7.0 at the surface of different electrodes with a scan rate of 100 mV s^{-1}. Carbon paste electrode modified with CdO/NPs and ILs exhibited significant oxidation peak current around 490 mV with the peak current of 32.1 μA (Fig. 3, curve a). In contrast, low electrooxidation activity peak was observed at carbon paste electrode modified with CdO/NPs (Fig. 3, curve c) and at unmodified carbon paste electrode (Fig. 3 curve d) over the same condition. The bisphenol A oxidation peak potential at carbon paste electrode modified with CdO/NPs and at carbon paste electrode observed around 525 and 540 mV versus the reference electrode with the oxidation peak current of 12.6 and 6.6 μA, respectively. In addition, at the surface of bare ionic liquid–modified

Fig. 6 **a** SWVs of CdO/NP/CPE in 0.1 mol L^{-1} phosphate buffer solution (pH 7.0) containing different concentrations of bisphenol A. From *a–k* correspond to 0.3, 5.0, 15.0, 25.0, 40.0, 110.0, 220.0, 300.0, 400.0, 550.0, and 650.0 μM. **b**, **c** The plots of the electrocatalytic peak current as a function of bisphenol A concentration
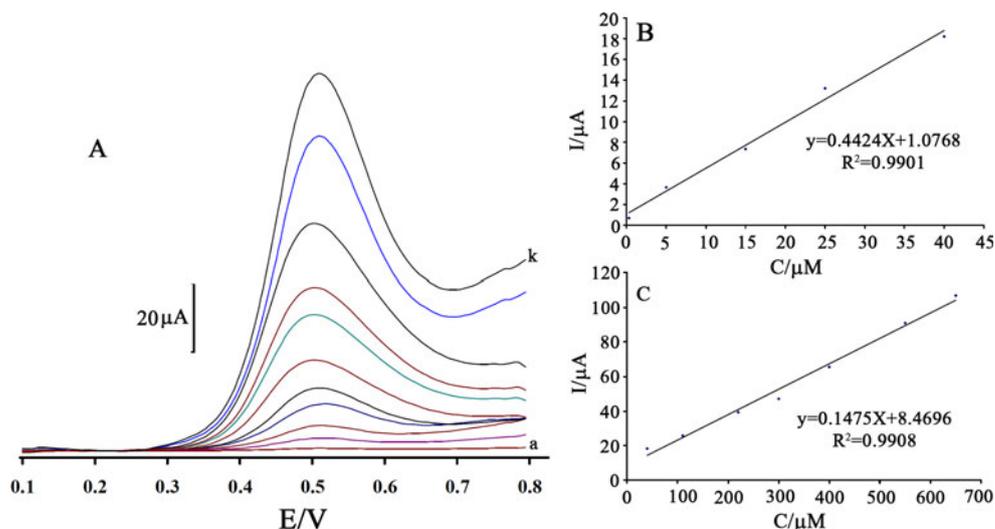
Table 1 Interference study for the determination of 10.0 μM bisphenol A under the optimized conditions

Species	Tolerant limits ($W_{substance}/W_{bisphenol\ A}$)
Glucose, sacarose, lactose, feroctose, ascorbic acid (after addition of 1.0 mM ascorbate oxidase)	1000
K^+, ClO_4^-, Na^+, Li^+, F^-, Cl^-, CO_3^{-2}, Ca^{2+}, Mg^{2+}, SO_4^{2-}	900
Tryptophan, lucine, L-threonine, L-phenylalanine, glycine, alanine, valine, histidine	700
Starch	Saturation

carbon paste electrode, the oxidation peak appeared at 505 mV with the peak current of 21.6 μA (Fig. 3, curve b), which indicated that the presence of ionic liquids in CPE could enhance the peak currents and decrease the oxidation potential. A significant negative shift of the currents starting from oxidation potential for bisphenol A and dramatic increase of current of bisphenol A indicated the catalytic ability of CdO/NP/IL/CPE to bisphenol A oxidation. The results indicated that the presence of nanoparticles on CdO/NP/IL/CPE surface had great improvement with the electrochemical response, which was partly due to excellent characteristics of CdO/NPs such as good electrical conductivity. The suitable electronic properties of metal oxide nanoparticle together with the ILs gave the ability to promote charge transfer reactions, good anti-fouling properties, especially when mixed with a higher conductive compound such as ILs when used as an electrode.

The effect of scan rate (υ) on the oxidation current of bisphenol A was also examined (Fig. 4 inset). The results of this investigation showed that the peak current increased linearly with increasing the square root of scan rate that ranged from 10 to 100 mV s^{-1} (Fig. 4). The result shows that the electrode process for oxidation of bisphenol A is controlled under the diffusion step. Also, the peak potential shifts in negative direction when the scan rate increases, meaning that the electrochemical reaction is irreversible.

The dependence of the anodic peak potential and ln(υ) showed a linear relationship with a regression equation of

$$E_{pa} = 0.0634 \ln(\upsilon) + 0.3091 \ (r^2 = 0.9939,\ E_p \text{in V},\ \upsilon \text{ in V s}^{-1}) \quad (1)$$

According to the following equation [36]:

$$E_{pa} = E^{0/} + m\left[0.78 + \ln\left(D^{1/2}k_s^{-1}\right) - 0.5\ln m\right] + (m/2)\ln(\upsilon) \quad (2)$$

With

$$m = RT/[(1-\alpha)n_\alpha F] \quad (3)$$

where E_{pa} is the oxidation peak potential, $E^{0/}$ is the formal potential, υ is the sweep rate, k_s is electron transfer rate constant. A plot of $E_{pa}=f(\ln (\upsilon/V\ s^{-1})$ yields a straight line with slopes equal to $2(RT/[(1-\alpha)n_\alpha F)$, where $R=8.314$ J mol^{-1} K^{-1}, $F=96485$ C mol^{-1}, and $T=298$ K. The value of $n(1-\alpha)$ is calculated 0.2 from Eq. (3), which indicates that the activation free energy curve is not symmetrical in such an irreversible oxidation process.

Chronoamperometric measurements of bisphenol A at CdO/NP/IL/CPE were carried out by setting the electrode potential at 650 mV for the various concentrations of bisphenol A in buffered aqueous solutions (pH 7.0; Fig. 5a). For bisphenol A, as an electroactive material with a diffusion coefficient of D, the current observed for the electrochemical reaction at the mass transport limited condition is described by the Cottrell equation. Experimental plots of I versus $t^{-1/2}$ were employed, with the best fits for different concentrations of bisphenol A (Fig. 5b). The slopes of the resulting straight lines were then plotted versus bisphenol A concentration. From the resulting slope and Cottrell equation, the mean value of the D was found to be 7.5×10^{-6} cm^2/s.

Table 2 Determination of bisphenol A in food samples

Sample	Added (μM)	Founded (μM)	Published method (μM)	F_{ex}	F_{tab}	t_{ex}	t_{tab}
Tomato paste	–	1.45±0.15	1.50±0.25	8.5	19.0	1.9	3.8
Corn	–	4.34±0.41	4.45±0.52	10.3	19.0	2.5	3.8
Stew	–	3.11±0.20	3.22±0.35	9.9	19.0	2.1	3.8
Water bottle	–	0.32±0.01	0.33±0.03	5.8	19.0	1.3	3.8
Tuna fish	–	2.22±0.11	2.40±0.27	9.5	19.0	1.8	3.8

F_{ex} = calculated F value; reported F value from F test table with 95 % confidence level and 2/2 degrees of freedom. t_{ex} = calculated t; t_{tab} (95 %); reported t value from Student's t test table with 95 % confidence level. ± Shows the standard deviation ($n=3$).

LOD and LDR Determination

Square wave voltammetry (SWV) was used to determine bisphenol A concentrations (Fig. 6a). The results showed two linear segments with different slopes for bisphenol A concentration in the ranges of 0.3 to 40.0, and 40.0 to 650 μM (Fig. 6b, c). The regression equations were I_p (μA)=0.4424 $C_{bisphenol\ A}$+1.0768 (r^2=0.9901, n=5) for 0.3 to 40.0 μM bisphenol A, and I_p (μA)=0.1475 $C_{bisphenol\ A}$ + 8.4696 (r^2=0.9908, n=7) for 40–650 μM bisphenol A, where $C_{bisphenol\ A}$ is the micromolar concentration of bisphenol A. The detection limit of 0.1 μM bisphenol A was obtained by SWV method.

Stability and Reproducibility

The repeatability and stability of modified electrode were investigated by SWV measurements of 20.0 μM bisphenol A. The relative standard deviation (RSD%) for ten successive assays was 1.7 %. When using seven different electrodes, the RSD% for nine measurements was 2.7 %. When the electrode was stored in the laboratory, the modified electrode retains 96 % of its initial response after 10 days and 92 % after 40 days. These results indicate that CdO/NP/IL/CPE has good stability and reproducibility and could be used for bisphenol A.

Interference Study

The influence of various substances as potentially interfering compounds with the determination of bisphenol A was studied under the optimum conditions of 10.0 μM bisphenol A at pH 7.0. The potentially interfering substances were chosen from the group of substances commonly found with bisphenol A in food samples. The tolerance limit was defined as the maximum concentration of the interfering substance that caused an error less than ±5 % for the determination of bisphenol A. The results are shown in Table 1. Those results confirm the suitable selectivity of the proposed method for determination of these compounds.

Real Sample Analysis

In order to evaluate the analytical applicability of the proposed novel sensor, also it was applied to the determination of bisphenol A in food samples. Standard addition method was used for measuring bisphenol A concentration in the samples. The proposed method was compared with a published method too (Najafi et al. 2014). The results are given in Table 2 and confirm that the modified electrode retained its efficiency for the determination of bisphenol A in real samples.

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