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Determination of Bisphenol in Food Samples Using an Electrochemical Method Based on Modification of a Carbon Paste Electrode with CdO Nanoparticle/Ionic Liquid

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An electroanalytical sensor for bisphenol A has been constructed through modification of a carbon paste electrode with a cadmium oxide nanoparticle (CdO/NPs) and 1-methyl-3-octylimidazolium tetrafluoroborate as an ionic liquid (MOITFB). The CdO/NPs were prepared and characterized using FESEM and XRD techniques, and were also indicated to have particle diameters less than 45 nm. The resulting sensor (CdO/NPs-MOITFB-CPE) was used for analyzing the trace level of bisphenol A in the concentration ranged from 0.01 µM to 280.0 µM, and a detection limit of 0.001 µM was determined for that. The signal recorded for bisphenol A using the modified CPE, was found to be improved 3.64 times as compared to unmodified CPEs, and the oxidation potential of bisphenol A was also decreased to 170 mV. Furthermore, the selectivity of the CdO/NPs-MOITFB-CPE to common interfering substances was found to be at a good level. The CdO/NPs-MOITFB-CPE showed a good performance in the determination of bisphenol A in food samples such as tuna fish, tomato paste, and stew with acceptable recovery data.

Keywords: Bisphenol A, CdO nanoparticle, 1-methyl-3-octylimidazolium tetrafluoroborate, Electrochemical food sensor

1. INTRODUCTION

The analysis of food additives, especially the restricted and forbidden ones, is an important task in quality control analyses [1-5]. The presence of banned compounds or their release from the food packages into food products, is another major issue in certain cases [6]. Therefore, applying analytical methods to investigate the food quality has been suggested as common practice for many years [7-15]. High performance liquid chromatography (HPLC), UV-Vis spectroscopy, and electrochemical sensors are the main methods used for the determination of food additives in quality control laboratories [16-24].

On the other hand, electrochemical sensors have been associated with a wide range of advantages such as low cost and ease and speed of operation, in comparison with other analytical techniques [25-35]. Hence, this category has been used to determine various drug and food additives in the recent years [36-40]. One of the areas of investigation in electrochemistry involves the modification of electroanalytical sensors using conductive materials such as nanoparticles (due to their high surface area and unique properties showed more application in different fields [41-50]), to enhance their response and detection limits [51-60].

Bisphenol A is an important phenol derivative used in producing resins and certain plastics since the 1960s [61]. Many researchers have confirmed that the bisphenol A can leak into beverages or food samples from food packaging, and given the fact that high levels of bisphenol A is harmful to human body, its determination in food samples is of great importance [62-65]. Consequently, fabrication of fast and portable analytical tools to determine the bisphenol A is very important in food safety analyses [66-68].

Accordingly, this research focused on the synthesis, XRD, and FESM analysis of CdO nanoparticles as a conductive mediator, and also on its usage in the modification of a carbon paste electrode further involving 1-methyl-3-octylimidazolium tetrafluoroborate as a conductive binder. The resulting sensor, i.e. CdO/NPs-MOITFB-CPE, was successfully used in the determination of nanomolar levels of bisphenol A in real samples.

2. MATERIALS AND METHODS

2.1. Reagents

Bisphenol A, 1-methyl-3-octylimidazolium tetrafluoroborate, cadmium acetate dihydrate, and paraffin oil were procured from Sigma-Aldrich. Graphite powder, acetic acid, sodium hydroxide and phosphoric acid were purchased from Merck Co. CdO nanoparticles were synthesized through a chemical precipitation technique disclosed by cheraghi and Taher [69].

2.2. Apparatus

All electrochemical experiments and determination of bisphenol A were performed using a Vertex-Ivium potentiostat/galvanostat. An electrochemical cell composed of an Ag/AgCl/KCl_{sat} (Azar

Electrode Co.), a Pt wire (counter electrode) and CdO/NPs-MOITFB-CPE (working electrode) was used in electrochemical studies. A Mira 3-XMU FESEM was used for morphological investigations.

2.3. Fabrication of CdO/NPs-MOITFB-CPE

The optimal CdO/NPs-MOITFB-CPE was prepared by mixing 970 mg of graphite powder, 30.0 mg of CdO nanoparticles, and a quantity of methanol as a solvent, followed by methanol evaporating. Next, suitable amounts of paraffin oil and MOITFB were added into a pestle, and were then mixed together using a mixture and also by hand mixing for 50 min.

2.4. Real sample preparation

Canned tuna fish, tomato paste, and stew were purchased from a local market. The samples were homogenized and selected for analysis, and were then filtered and dissolved in an ethanol/water mixture.

3. RESULTS AND DISCUSSION

3.1. Characterization of CuO nanoparticles

The synthesized nanoparticles were characterized by FESEM and XRD methods and results are presented in figure 1.

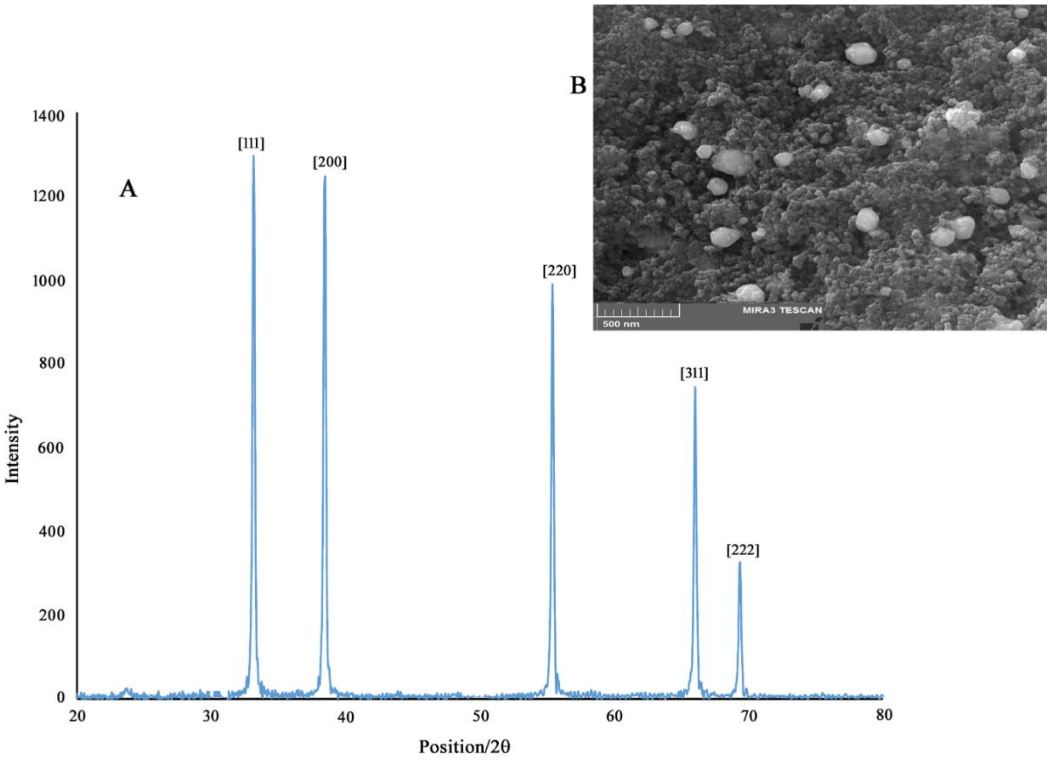
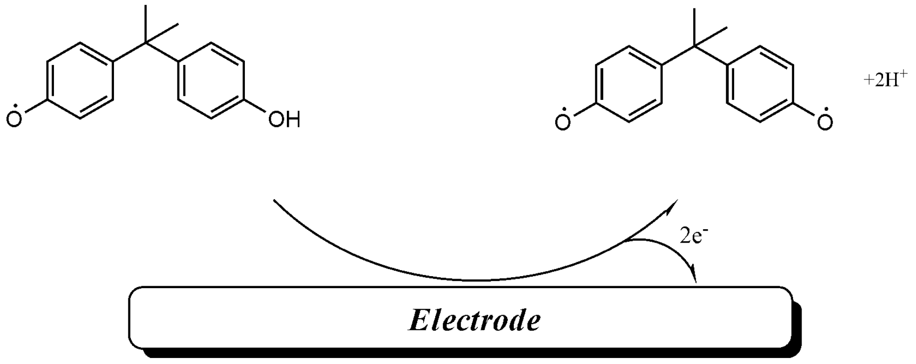


Figure 1. A) The XRD pattern and B) FESEM image of CdO/NPs.

The structure of CdO/NPs was detected using XRD method, and results showed that the XRD pattern of the nanoparticles contained five planes in terms of the Miller indexes [111]; [200]; [220]; [311] ;and [222] matching the reference data in JCPDS card No. 65-2908 for CdO nanoparticle (Figure 1 A). This result confirms a crystalline structure for synthesized nanoparticle. The CdO particles were found to have dimeters of less than 45 nm with spherical shape in FESEM analyses (Figure 1 B), which confirm the ability of recommend procedure in synthesizing the CdO nanoparticle [69].

3.2. Electrochemical behavior of bisphenol A at CdO/NPs-MOITFB-CPE

According to the mechanism reported in scheme 1 by other researchers, the redox behavior of bisphenol A is directly dependent on the pH of the solution.



Scheme 1. The electro-oxidation mechanism of bisphneol A.

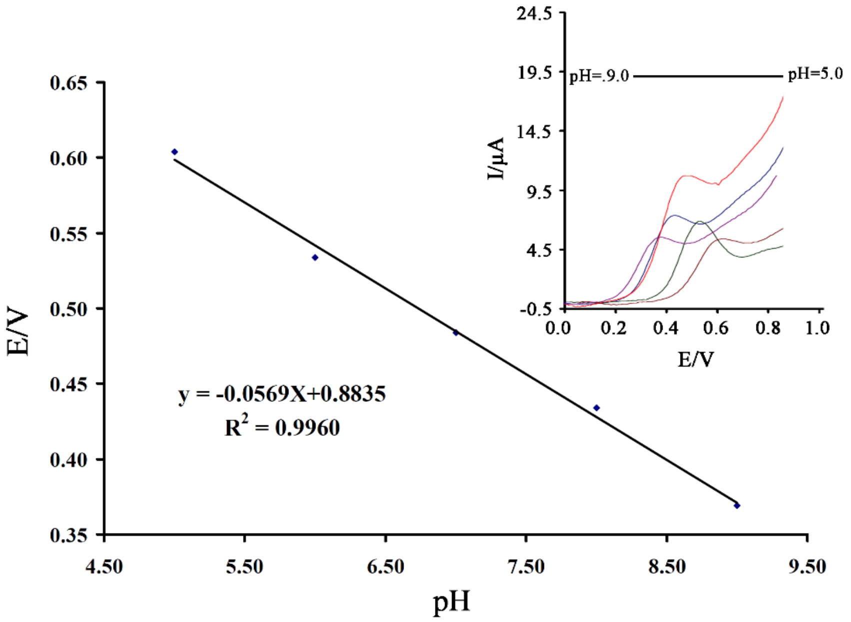


Figure 2. The E-pH plot relative to electro-oxidation of 400 µM bisphenol A at CdO/NPs-MOITFB-CPE. Inset) DPVs of 50.0 µM bisphenol A at CdO/NPs-MOITFB-CPE at pH range 4.0-8.0.

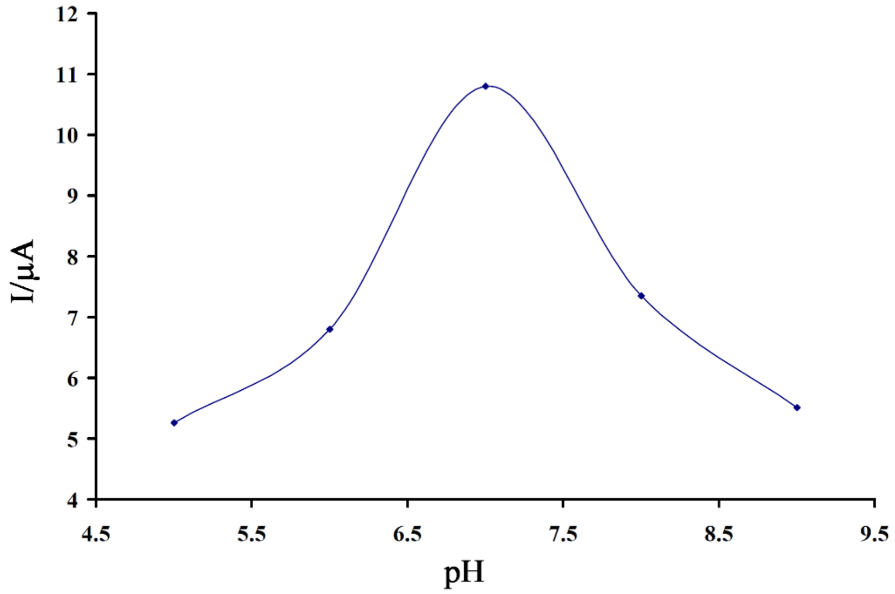


Figure 3. The I-pH plot relative to electro-oxidation of 50.0 µM bisphenol A at CdO/NPs-MOITFB-CPE.

Therefore, the differential pulse voltammogram of a 50.0 µM bisphenol A solution was recorded using CdO/NPs-MOITFB-CPE in the pH range between 4.0 and 8.0. As can be observed in the inset of figure 2, by changing the pH from 4.0 to 8.0, the oxidation potential of bisphenol A decreased and the plot of oxidation potential vs. pH had a slope of 56.9 mV/pH (Figure 2), which confirm the equal effects of electron and proton exchange phenomena in a redox reaction according to scheme 1. On the other hand, as illustrated in figure 3, the maximum oxidation current for bisphenol A occurred at pH=7.0, which was hence selected as the optimal pH.

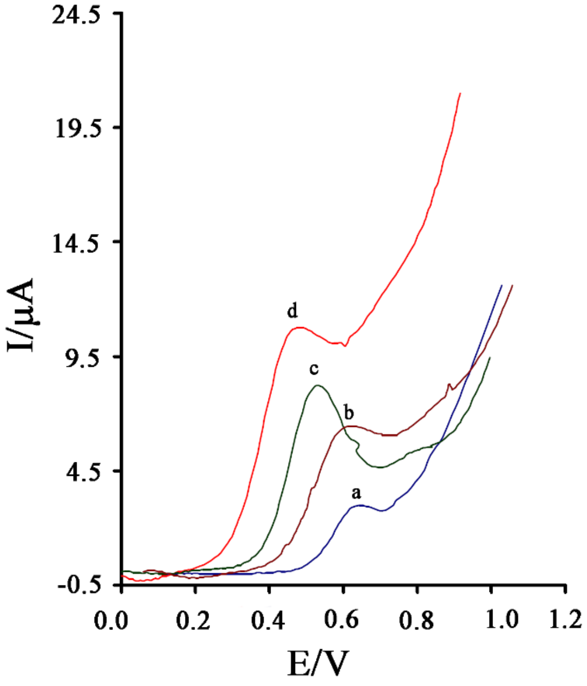


Figure 4. DPVs of 50.0 µM bisphenol A at surface of a) CPE, b) CdO/NPs-CPE, c) MOITFB-CPE and d) CdO/NPs-MOITFB-CPE recorded at pH=7.0

The differential pulse voltammogrs of a 50.0 µM bisphenol A solution was recorded using a CPE (figure 4 curve a), a CdO/NPs-CPE (figure 4 curve b), an MOITFB-CPE (Figure 4 curve c), and a CdO/NPs-MOITFB-CPE (figure 4 curve d), respectively. also as can be observed, by moving from curve a to d, the current oxidation of bisphenol A increased from 2.99 µA up to 10.8 µA, and the corresponding oxidation potential decreased from 650 mV to 480 mV, further confirming the enhanced properties of the modified electrodes, due to the good electrical conductivity of CdO/NPs and MOITFB. Moreover, Due to good electrical conductivity of MOITFB and CdO/NPs, the oxidation signal of bisphenol A improved, and relative over-potential decreased [69].

The linear sweep voltammograms of a 500 µM bisphenol A solution was recorded in scan rate range 10.0-200.0 mV/s (Figure 5). The linear relationship between the oxidation curve of bisphenol A and $v^{1/2}$ confirmed the diffusion controlled nature [70] of the electro-oxidation of the analyte at CdO/NPs-MOITFB-CPE (Figure 5). On the other hand, along with increasing in scan rate, the oxidation potential of bisphenol A was shifted to positive value confirming an irreversible behavior for electro-oxidation of electroactive compound [71].

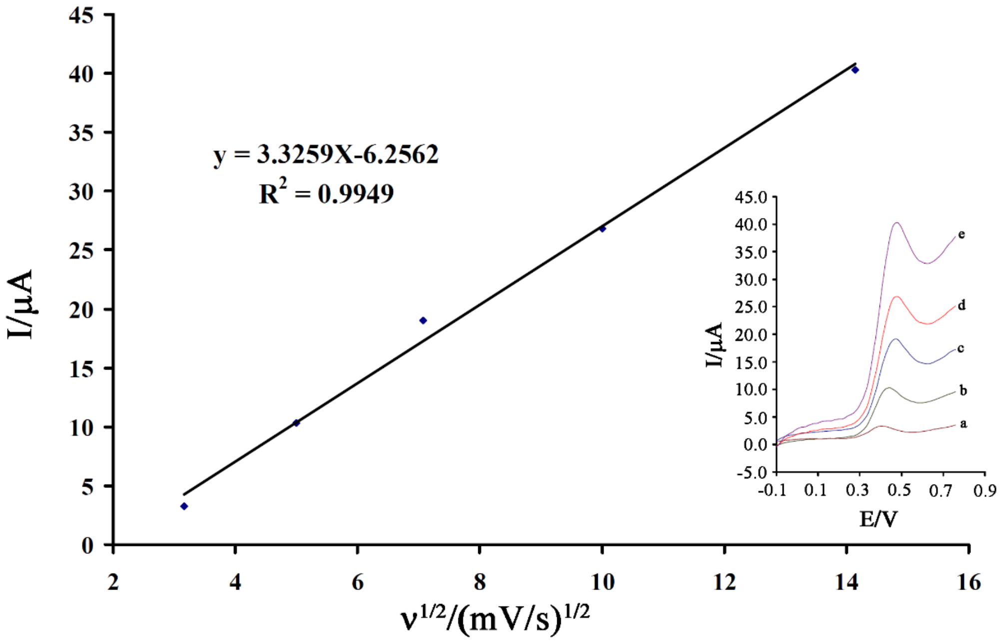


Figure 5. The linear sweep voltammograms of CdO/NPs-MOITFB-CPE in the solution containing bisphenol A at scan rates a) 10, b) 15, c) 40, d) 70, e) 100 and f) 200 mV/s.

The chronoamperograms of 50.0 µM, 100.0 and 150 µM bisphenol A were recorded at surface of CdO/NPs-MOITFB-CPE at 700 mV (Figure 6 A). Cottrell's plot for the electro-oxidation of 300 µM and 500 µM bisphenol A solutions at of CdO/NPs-MOITFB-CPE are presented in figure 6B, and the value of diffusion coefficient was determined to be ~1.91×10^{-5} cm^2/s.

The selectivity of CdO/NPs-MOITFB-CPE for bisphenol A was evaluated in the presence of some interfering species such as vitamin B_6, tryptophan, glycine, vitamin B_9, K^+, Cl^-, and methionine, and also acceptable error of 5% was recorded for the current readings. The results confirm that even 700-fold excess of the tested interfering compounds had no interfering effects on the determination of 50 µM bisphenol A.

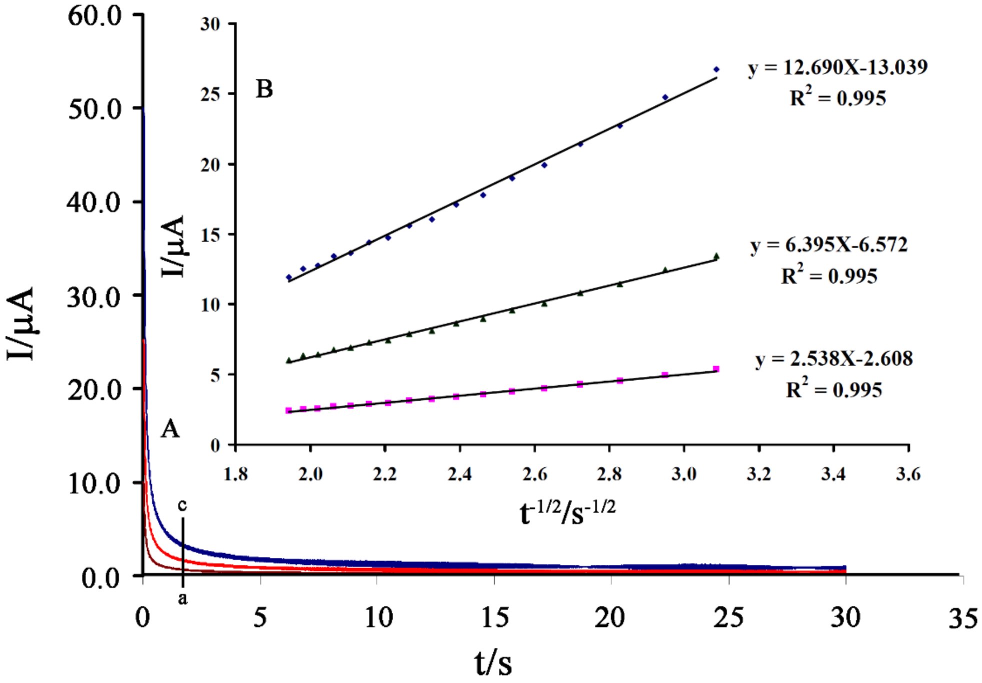


Figure 6. A) Chronoamperograms obtained at the CdO/NPs-MOITFB-CPE in the solution containing (a) 300 and (b) 500 µM bisphenol A. B) Cottrell plots obtained from chronoamperometry.

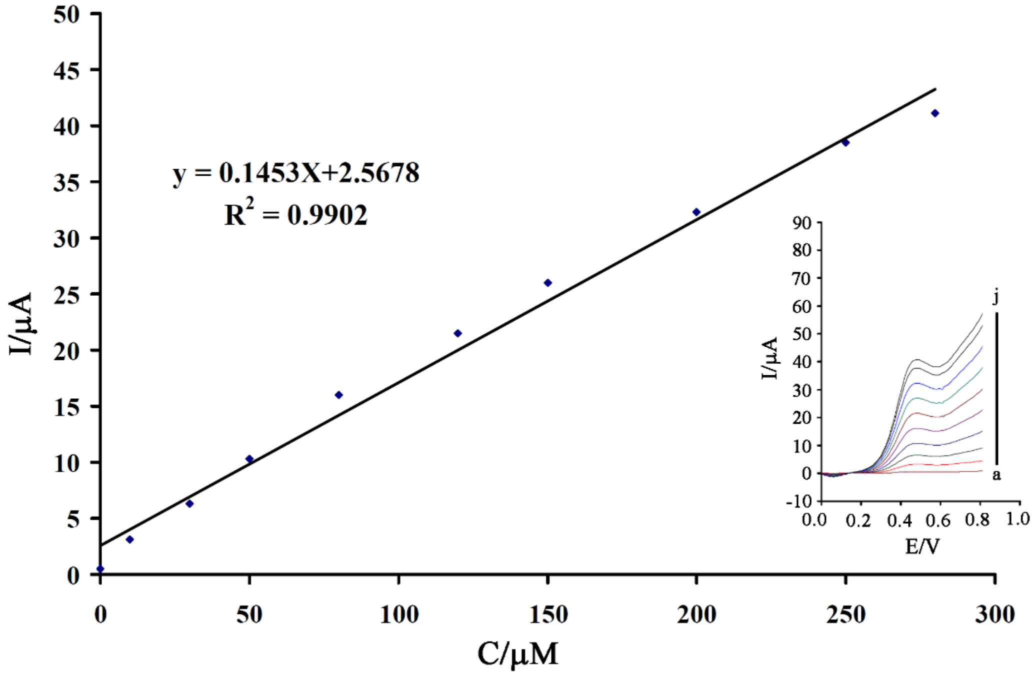


Figure 7. A) The plots of the I_{pa} as a function of bisphenol A concentration. The DP voltammograms of CdO/NPs-MOITFB-CPE in the presence solution containing a) 50.0; b) 90.0; c) 120.0; d) 200.0; e) 240.0 and f) 320.0 µM bisphenol A.

The stability of CdO/NPs-MOITFB-CPE responses for the determination of bisphenol A was checked over a 30-day period. After 30 days, detected signal equal to 92% of the initial signal of bisphenol A was recorded, which confirms the good stability of CdO/NPs-MOITFB-CPE as an analytical tool for the determination of bisphenol A.

Inset of Figure 7 illustrates the DPV voltammograms recorded using CdO/NPs-MOITFB-CPE for different concentrations of bisphenol A. Evidently, a stepwise increase was observed in the oxidation current of bisphenol A, along with the stepwise increase of the concentration of bisphenol A, which indicate the applicability of CdO/NPs-MOITFB-CPE to the electrochemical determination of bisphenol A. Figure 7 shows the changes in the oxidation signal recorded using CdO/NPs-MOITFB-CPE, as a function of bisphenol A concentration in the range of 0.01-280.0 µM, with detection limit of 1.0 nM. These values of linear dynamic range and limit of detection, are better or comparable with previous reported electrochemical sensors (as shown in table 1).

Table 1. Comparison of the efficiency of previous suggested sensors in the determination of bisphenol A

Electrode	Modifier	LDR (µM)	LOD ((µM)	Ref.
Carbon paste electrode	ZnO/CNTs + ionic liquid	0.02-700	0.009	[66]
Glassy carbon electrode	poly(chromotropic acid)	0.1-20.0	0.06	[72]
Glassy carbon electrode	gold nanoparticles loading on the reduced graphene oxide	0.005-20.0	0.001	[73]
Carbon paste electrode	multi-walled carbon nanotubes-titanium dioxide	1.0-600.0	3.0	[74]
Carbon paste electrode	CdO/NPs-MOITFB	0.01-280	0.001	This work

The ability of CdO/NPs-MOITFB-CPE in determining the bisphenol A in tuna fish, tomato paste, and stew was evaluated through standard addition (table 2). The recovery data in the range of 98.09-103.35% confirmed the considerable performance of the CdO/NPs-MOITFB-CPE as an analytical tool for the determination of bisphenol A in real samples.

4. CONCLUSIION

A high performance and sensitive electroanalytical sensor was fabricated for the determination of bisphenol A in food samples. CdO nanoparticles and MOITFB were used as conductive mediators to improve the electrical activity of the fabricated sensor. The presence of CdO nanoparticles and MOITFB in the sensor led to a linear dynamic ranged from 0.01 to 280 µM with a detection limit set as 1.0 nM for bisphenol A. The recovery data confirmed the high performance of CdO/NPs-MOITFB-CPE for the determination of bisphenol A in real samples such as tuna fish, tomato paste, and stew.

Table 2. The real sample analysis of bisphenol A

Sample	Added (µM)	Expected (µM)	Founded (µM)	Recovery %
Stew	---	---	3.11±0.22	---
	10.00	13.11	13.55±0.68	103.35
Tuna fish,	---	5.22±0.65	---	---
	10.00	15.22	14.93±0.75	98.09
Tomato paste	---	---	2.01±0.12	---
	10.00	12.01	12.34±0.44	102.74

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