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Electrochemical Behavior of Bisphenol-A at Tolbutamide Modified Carbon Paste Electrode: A Voltammetric Study

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Abstract

In present work, a sulfonylurea drug (tolbutamide) modified carbon paste electrode is used as an electrochemical sensor. The newly developed tolbutamide modified carbon paste electrode (TBMCPE) has been employed for the detection of Bisphenol-A (BPA) in presence of 0.2M phosphate buffer solution (pH 7) at the scan rate of 50mVs^{-1} by cyclic voltammetric method. The modified electrode exhibits excellent electrocatalytic behavior towards BPA determination. The parameters such as effect of scan rate, concentration and accumulation time was carried out at modified electrode. Furthermore, simultaneous determination of BPA and Uric acid is carried out by both cyclic voltammetry and differential pulse voltammetric techniques. The limit of detection (LOD) and limit of quantification (LOQ) was calculated and found to be 0.78 nM and 2.622 nM respectively. The proposed modified electrode shows excellent electrochemical activity and found to be effective and economical.

Keywords: Cyclic voltammetry; BPA; Tolbutamide; Modified carbon paste electrode

Introduction

Bisphenol-A (BPA) [2,2-bis (4-hydroxyphenyl) propane ($C_{15}H_{16}O_2$] is a vital chemical raw material for the production used in industries and also in daily life (Figure 1) [1-4]. BPA is used in the preparation of polycarbonates, which are used in huge number of consumer products like plastic water bottles and also found in metal based food and beverage cans where in epoxy resin containing BPA acts as a protective lining inside these cans [5,6]. Endocrine disrupting compounds (EDC's) or endocrine disrupting chemicals are those exogenous agents that with the synthesis, secretion, transport, metabolism, binding action or elimination of natural blood-borne hormones which are responsible for biological processes in human body. BPA comes under EDC's [7-12]. It causes irreversible damage to the organisms, environment and low doses can result in human endocrine disorders [13,14]. BPA also effects on female hormones results in diseases like precocious puberty, reproductive dysfunction, endometrial hyperplasia and recurrent miscarriage [15,16]. Several studies have confirmed the presence of BPA in soil and aquatic environments, also the widespread and continuous human exposure to BPA through drinking water, food, dental sealants, cell phones and inhalation of indoor dusts [17-22].

For these adverse effects the detection of BPA by different methods in scientific way is in progress from several years. Different analytical techniques like high performance liquid chromatography, liquid chromatography–mass spectrometry, gas chromatography, gas chromatography–mass spectrometry, and enzyme-linked immunosorbent assay [23-27]. Even though above methods are employed for the detection of BPA but they were found to be costlier than the simpler and economical way as electrochemically. Since easy, rapid response and high sensitive by electrochemical method many works published based on modified electrodes with surfactant, phthalocyanine, PMAM dendrimer, layered double hydroxide, carbon nanotube (CNT) and grapheme have been reported resulting with good sensitivity for BPA [28-36]. Even though many techniques established in the field of science for the determination of BPA cyclic voltammetric technique gives precise results, ecofriendly and an economical way also.

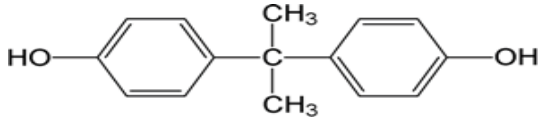

Figure 1: Structure of Bisphenol-A.

Sulphonylureas have been used in the treatment of non-insulin-dependent diabetes mellitus (NIDDM or type-2 diabetes) for more than 30 years [37,38]. Present work proposes a new modifier for detection of Bisphenol-A which comes under this sulphonylurea that is tolbutamide (TB). TB [3-butyl-1-(4-methylphenyl)sulphonylurea) CAS (64-77-7)] is suphonylurea which acts as a hypoglycemic agent in management of type two diabetes mellitus. The immediate action of TB after oral administration is absorption and extensive binding to plasma proteins in humans [39,40].

In this existing work carbon paste is modified using tolbutamide. In order to study drug based sensor capability for determination of BPA, tolbutamide is used. Electrochemical parameters such as scan rate variation, concentration effect and accumulation time were carried out at this modified electrode resulted in promising results and sensitive response.

Experimental Part

Reagents and chemicals

BPA and tolbutamide were obtained from Himedia chemical company and Sigma Aldrich respectively. The compounds were

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of analytical grade used without further purification. 25 x 10^{-4}M Bisphenol-A stock solution was prepared in ethanol. Graphite powder of 50 mm size was purchased from Loba, silicon oil was purchased from Himedia. The chemicals for preparation of buffer solution were purchased from Merck and the phosphate buffer (0.2 M pH 7) was used as supporting electrolyte.

Apparatus

Cyclic voltammetry (CV) was performed in a model CHI-66^{0}C (CH Instrument-660 electrochemical workstation). All experiments were carried out in a conventional electrochemical cell. The electrode system contained a carbon paste working electrode (3.0 mm in diameter), a platinum wire as counter electrode and saturated calomel as reference electrode for the electrochemical measurements.

Preparation of bare and modified carbon paste electrode

The carbon paste electrode was prepared by mixing 70% graphite powder and 30% silicone oil manually using mortar and a dish to get a homogeneous carbon paste. The paste was then packed into the cavity of a carbon paste electrode and smoothed on a weighing paper. Modified carbon paste electrode was prepared by homogeneous mixing 2, 4, 6, 8 and 10 mg of tolbutamide powder to 70% graphite powder and 30% silicone oil. Thorough manual grinding is carried out for twenty five minutes. This tolbutamide modified carbon paste electrode (TBMCPE) was used for further investigations of BPA after rinsing in doubly distilled water.

Results and Discussions

Effect of TBMCPE on Bisphenol-A

Different modified carbon pastes ranging from 2 mg to 10 mg was employed in the investigation of 5 nM BPA at 7 pH in 0.2M phosphate buffer solution (PBS). In Figure 2 anodic peak current for BPA versus different concentrations of tolbutamide were plotted. Anodic peak current for BPA goes in decreasing with increase in the concentration of tolbutamide. This decrease in current may due to monolayer accumulation of tolbutamide on the surface of electrode which might block the determination of any species in the solution of electrochemical cell. By comparing anodic peak current and peak nature for further experiments 2 mg of tolbutamide modified carbon paste was used for the determination of BPA.

Electrocatalytic behavior of $K_4Fe(CN)_6$ at TBMCPE

To know the significance of the proposed modified electrode 1 mM $K_4Fe(CN)_6$ in the presence of 1MKCl as a supporting electrolyte potential is applied at the scan rate of 50mVs^{-1} by using cyclic voltammetric technique. Figure 3 shows cyclic voltammograms of 1 mM $K_4Fe(CN)_6$at both bare carbon paste electrode (solid line) and TBMCPE (dashed line). The low redox peak currents response was obtained at BCPE (Ipa=1.616 μA and Ipc=1.247 μA) but in the same condition TBMCPE exhibited good enhancement of redox peak currents (Ipa=3.204 μA and Ipc=2.6 μA) and showed the fast rate of electron transfer kinetics. The voltammetric response is apparently improved which suggests that the surface property of modified electrode significantly changed and also shows elecrtrocatalytic activity of TBMCPE towards potassium ferrocyanide.

Electrochemical behavior of Bisphenol-A at TBMCPE

Electrochemical response for 5 nM BPA at 7 pH in 0.2 M PBS by applying potential at the scan rate of 50mVs^{-1} at TBMCPE. Figure 4 shows cyclic voltammograms of Bisphenol-A at bare carbon paste electrode (solid line) and at TBMCPE (dashed line). Enhancement in the oxidation peak current and irreversible peak shows clear evidence that our TBMCPE of 2 mg has better elecrtrocatalytic activity by exposing to larger surface area. A slight shift in the oxidation peak potential from 0.566V (bare) to 0.546V (modified) can be observed. This shift in potential may be due to basic character (less basic) of tolbutamide molecules present in carbon paste. The improved current signals for BPA at TBMPE show electrode efficiency and excellent elecrtrocatalytic activity.

Effect of scan rate

To know the electrode process involved in electrochemical oxidation of BPA effect of scan rate is carried out. The scan rate varied from 20 to 90mVs^{-1} for 5 nM BPA in 0.2 PBS at 7 pH at TBMCPE. As the scan rate increased the anodic peak current increases linearly. In Figure 5 Cyclic voltammograms for bipshenol-A at different scan rates is shown. The graph of anodic peak current versus square root of scan rate and anodic peak current versus scan rate is plotted Figures 6 and 7 respectively. The correlation coefficient for Ipa *versus* scan rate

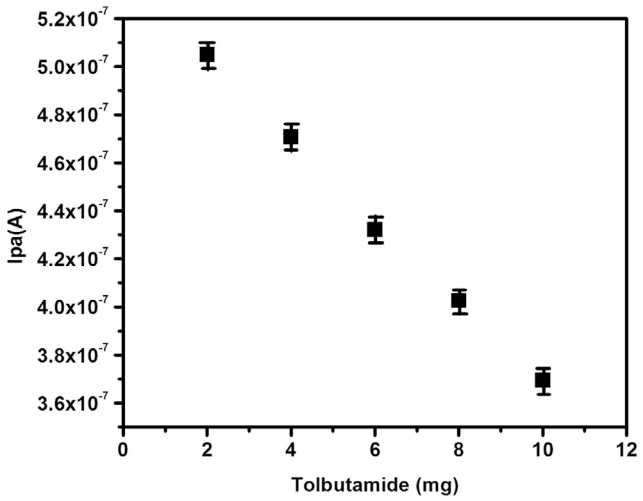

Figure 2: Plot of Ipa versus concentration of tolbutamide (mg).

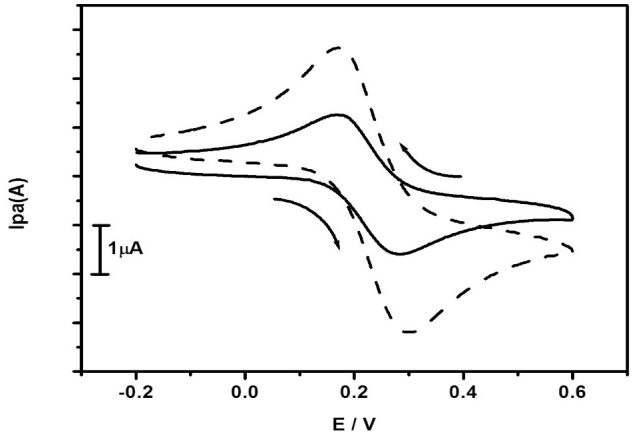

Figure 3: Cyclic voltammograms of 1 mM $K_4Fe(CN)_6$ at bare carbon paste electrode (solid line) and modified carbon paste electrode TBMCPE (dashed line).

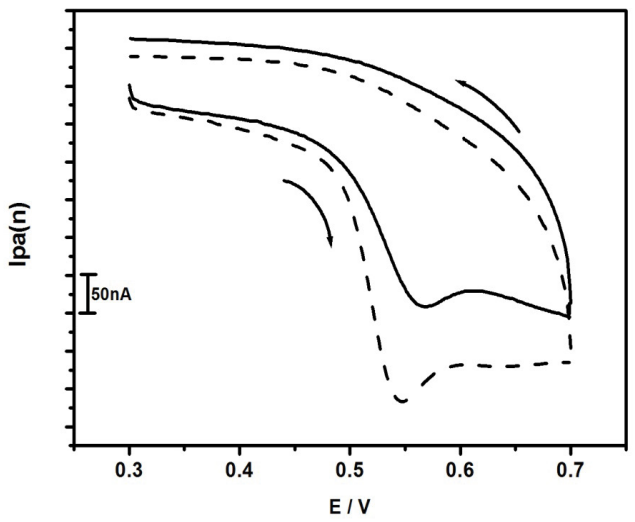

Figure 4: Cyclic voltammograms of 5 nM Bisphenol-A at bare carbon paste electrode (solid line) and modified carbon paste electrode TBMCPE (dashed line) with scan rate of 50mVs^{-1}.

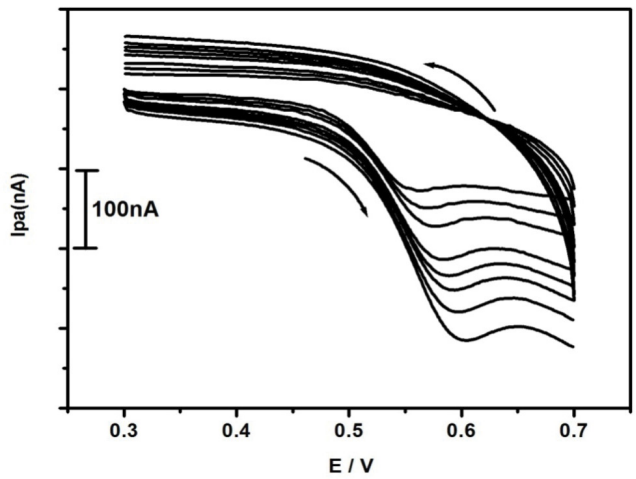

Figure 5: Cyclic voltammograms for 5 nM Bipshenol-A at different scan.

was 0.9957 and Ipa versus square root of scan rate was 0.9736. The results indicated that the electron transfer reaction was controlled by adsorption of BPA to the electrode surface [41,42].

Effect of concentration

The relationship between oxidation peak current of BPA and its concentration was investigated by using cyclic voltammetric method. Electrochemical oxidation of BPA was carried out by varying its concentration from 7 nM to 11 nM in 0.2 PBS at 7 pH at TBMCPE at the scan rate of 50mVs^{-1}. The Figure 8 shows cyclic voltammograms of different concentrations of BPA at TBMCPE. A plot of anodic peak current versus concentration of BPA as shown in the Figure 9 which shows concentration proportional to Ipa with a very good linearity. Linear regression coefficient was found to be R^2= 0.9991. The limit of detection (LOD) and limit of quantification (LOQ) was calculated using equation 1 and 2 [43-45]. Comparison of detection limits presented in Table 1.

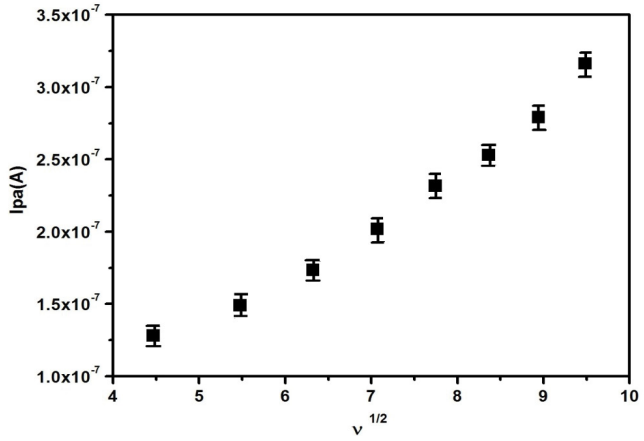

Figure 6: Graph of anodic peak current versus square root of scan rate.

Electrode	Detection limit	Methods	References
(MIP/ABPE)	60 nM	LSV	47
MCM-41 sensor	38 nM	DPV	48
Graphene-modified GCE	46.8 nM	DPV	49
Tyr-SF-MWNTs- CoPc/GCE	30 nM	Amperometry	50
HDS/SDS/CPE	18.9	DPV	43
TBMCPE	0.78 nM	CV	This work

Table 1: Comparison of detection limit of this work and literature reported ones.

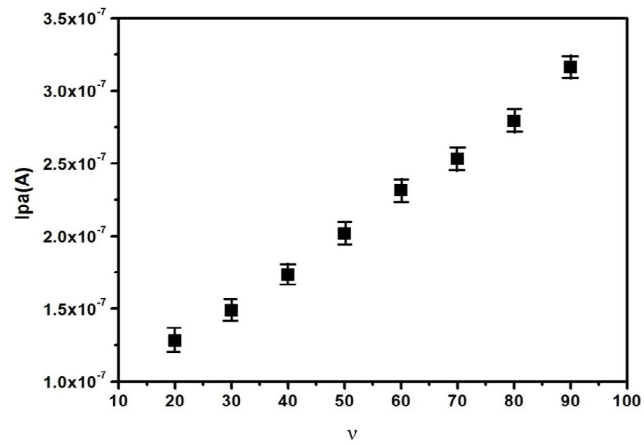

Figure 7: Graph of anodic peak current versus scan rate.

LOD=3S/M (1)

LOQ=10S/M (2)

LOD and LOQ were found to be 0.78 nM and 2.622 nM respectively.

IP/ABPE: Molecularlyimpringnated polymer modified acetylene block paste electrode

MCM-41 sensor: Mesoporous silica molecular sieves

Graphene-modified GCE: Graphene-modified glassy carbon electrode

Tyr-SF-MWNTs-CoPc/GCE: Immobilizing tyrosinase (Tyr) on multiwalled carbon nanotubes (MWNTs)-cobalt phthalocyanine

(CoPc)-silk fibroin (SF) composite modified glassy carbon electrode (GCE)

LSV: Linear Sweep Voltammetry

CV: Cyclic Voltammetry

Effect of accumulation time

Influence of accumulation time is significant on oxidation of BPA at TBMCPE. This reaction is carried out for 5 nM BPA in 0.2 PBS at 7 pH by cyclic voltammetric method. When potential is applied at the scan rate of 50mVs^{-1} with the time gap increased values ranged from 0-10 minutes the anodic peak current for BPA found to be decreasing. Figure 10 shows a plot of anodic peak current versus time in minutes. This decrease in the current may be due to BPA molecules layer formation on the electrode which might blocks for the next subsequent scan. Effect of accumulation time reveals as the time increases decrease in the anodic peak current.

Simultaneous detection of Bisphenol-A and Uric acid at TBMCPE by both CV and DPV method: One of the literature reported that human urine sample contains BPA by leaching out in food can linings and having said that human urine contains certain milligrams of uric acid (UA) in human urine [46]. This reaction is carried out to know the electrocatalytic activity and simultaneous determination of UA and BPA at TBMCPE. To establish the same uric acid of 2.5 mM and 7 nM of BPA at 7 pH in 0.2M PBS at the scan rate of 50mVs^{-1} potential is applied. The Figure 11 shows cyclic voltammograms for BPA and UA at bare carbon paste electrode (solid line) and at TBMCPE (dashed line), the anodic peak current found to be more enhanced for BPA and UA. This represents the sensor proposed found to be more electrocatalytic activity and higher active surface area.

At the same pH and scan rate potential is applied to 1 mM UA and 7 nM BPA by using DPV technique. Figure 12 shows DPV for BPA at TBMCPE [47-49]. The newly proposed sensor TBMCPE for BPA found to be effective, selective and sensitive in determination. This sensor can also be used to detect simultaneously UA and BPA in real samples.

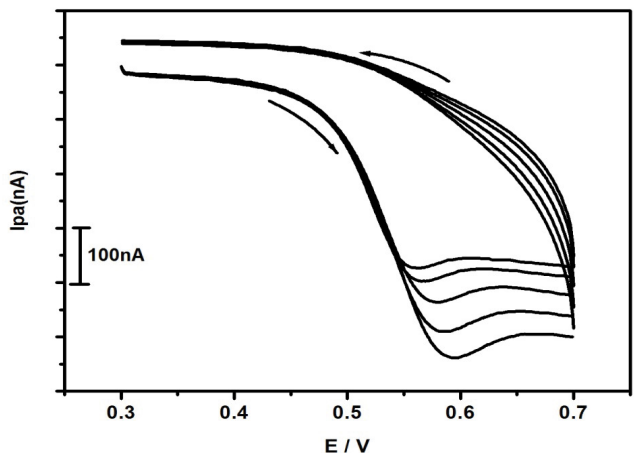

Figure 8: Cyclic voltammograms of different concentrations for BPA at TBMCPE.

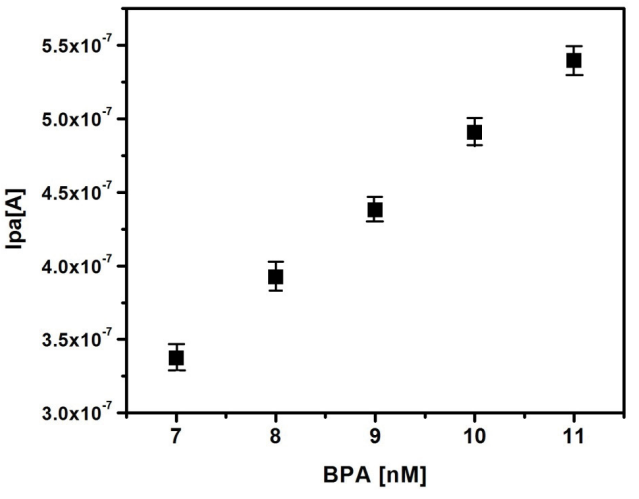

Figure 9: Plot of anodic peak current *versus* concentration of BPA.

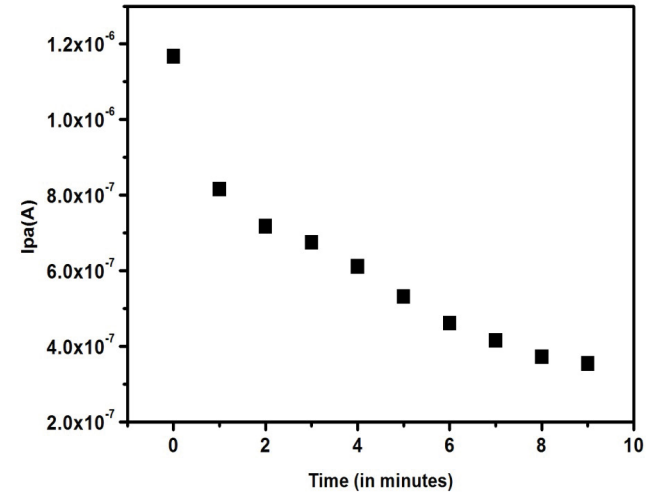

Figure 10: Plot of anodic peak current versus concentration of BPA.

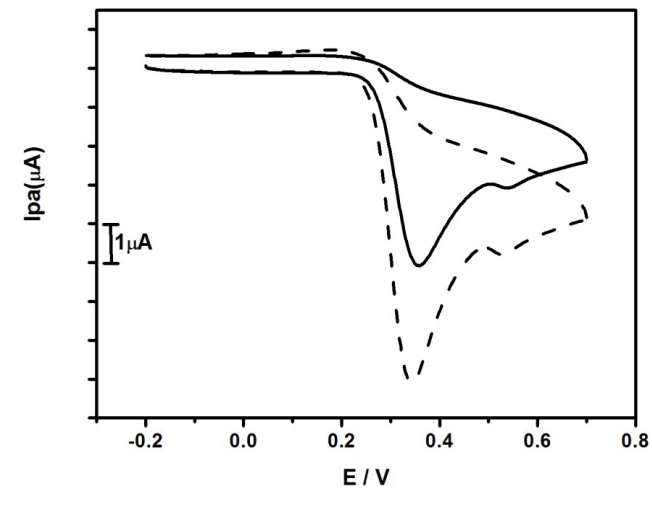

Figure 11: Cyclic voltammograms of 7nM BPA and 1mMUA at TBMCPE.

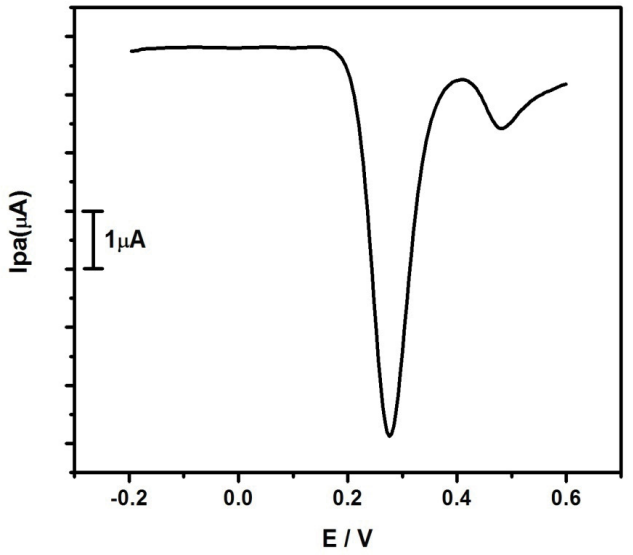

Figure 12: DPV of 7 nM BPA and 1 mM UA at TBMCPE.

Conclusion

Inexpensive, easy and sensitive modified electrode was developed to detect the electrochemical behavior of Bisphenol-A. This modified electrode showed excellent sensitivity, selectivity and strong electrocatalytic properties towards the oxidation of BPA using both cyclic voltammetric and differential pulse voltammetric methods. The influence of accumulation time resulted in the decrease in the anodic peak current. The proposed sensor shows lower detection limit out of the reported literatures.

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