

# Voltammetric determination of mercury(II) using a modified pencil graphite electrode with 4-(4-methylphenyl aminoisonitrosoacetyl)biphenyl

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**Abstract** A sensitive and selective method for determination of mercury(II) with “4-(4-methylphenyl aminoisonitrosoacetyl)biphenyl (TKO)-modified pencil graphite electrode” was developed. All factors affecting determination process were optimized. Differential pulse voltammetry with 4-(4-methylphenyl aminoisonitrosoacetyl)biphenyl-modified electrode showed a linear response between  $1.0 \times 10^{-5}$  and  $1.0 \times 10^{-3}$  M ( $R^2 = 0.9994$ ). The detection limit of this electrode was found as  $5.85 \times 10^{-7}$  M ( $S/N = 3$ ). The effects of different cations on the determination of mercury(II) were investigated and found that modified electrode is highly selective. The developed method was applied for mercury determination in different water samples.

**Keywords** Mercury(II) · Modified electrode · Oxime · 4-(4-methylphenyl aminoisonitrosoacetyl)biphenyl · Differential pulse voltammetry

## Introduction

The control of mercury is important due to its toxicity in the environment and serious hazardous effects on human

health such as kidney toxicity, birth defects, cerebral and neurological damage. Therefore, determination of mercury at trace levels in a variety of samples especially in aqueous media is in critical importance [1, 2].

The analytical techniques preferred for mercury quantification are cold vapor atomic absorption spectrometry (CV-AAS) [3], cold vapor atomic fluorescence spectrometry (CV-AFS) [4], inductively coupled plasma mass spectrometry (ICP-MS) [5, 6] and, for relatively high concentrations, inductively coupled plasma atomic emission spectrometry (ICP-AES) [7]. Several chromatographic techniques coupled with spectrometric methods have also been used for mercury speciation [8–10]. All these techniques often require treating and enrichment of sample by complex techniques, expensive instrumentation and qualified staff.

Voltammetric methods were extensively employed for the trace analysis of metal ions [2, 11–14] due to their advantages of easy or no sample pretreatment, short analysis time and automated, compact, and low-cost instrumentation. Different types of electrodes were utilized in these techniques, mainly glassy carbon electrode [15, 16], carbon paste electrode [17], gold electrode [18], gold film electrode [19], gold disk electrode [20], rotating gold electrode [21] and chemically modified electrodes [22–26]. In order to enhance the sensitivity and selectivity for voltammetric determination of Hg(II), chemically modified electrodes have been widely used. Many organic and biological modifiers, such as chelating ligand, organic polymers and DNA, are used as receptor materials for voltammetric detection of Hg(II) [27–29]. These modifiers are generally based on two types of interaction: ion exchange and complexation [30].

Organometallic and coordination chemistry of oximes constitute an active area of research, with efforts

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in particular being directed toward unusual reactivity modes of oximes and their complexes [31, 32]. Oximes and their metal complexes are of current interest for their rich physicochemical properties, reactivity patterns and potential applications in many important chemical processes in the areas of medicine [33, 34], bioorganic systems [35, 36], solid-phase extraction [37] membranes [38, 39] catalysis [40], electrochemical [41–43] and electro-optical sensors [44].

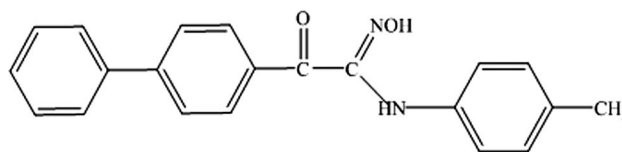
Certainly, the operation mechanism of chemically modified electrodes depends chiefly on the chemical nature and properties of the modifying additives used to boost selectivity and sensitivity of the desired electrode toward the target species. Since, oximes constitute an important class of electroactive compounds as they contain =N–OH group, which form stable complexes with many metal ions, therefore, oximes can be used as electrode modifiers for the analysis of metal ions. So far, many organic synthetic compounds were reported as Hg(II)-carrier with some specific features, which qualifies them as an appropriate ionophore for mercury(II) ion [45–47]. In recent years, oxime-based ionophores have attracted considerable interest because of their easy synthesis and high reactivity toward various compounds under a wide range of distinct physical conditions. But a limited number of studies on the use of oxime-based chemically modified electrodes for voltammetric determination of mercury ion have been reported [48, 49].

In this study, results of the applicability of “TKO modified pencil graphite electrode” for the determination of Hg(II) ions with differential pulse voltammetry were reported. The structure of the TKO was given in Fig. 1. The analytical parameters (e.g., oxime concentration, electropolymerization cycles, scan rate and interference) which effect on electrode reactions and analysis processes were studied. This method was applied for the determination of Hg(II) in synthetic water samples.

## Experimental

### Chemicals and reagents

All the chemicals used for the measurements were of analytical reagent grade. All experimental solutions were prepared in deionized water obtained from a Millipore Milli-Q water purification system. Mercury solutions were prepared from mercury(II) nitrate by dilution with HNO<sub>3</sub>. Metal salts were purchased from various suppliers. The studied oxime (TKO) was synthesized according to a described procedure [50].



**Fig. 1** Structural formula of 4-(4-methylphenyl aminoisonitrosoacetyl)biphenyl

### Instrumentation

Electrochemical measurements were taken with Autolab PGSTAT 302 N Potentiostat/Galvanostat controlled by GPES 4.9 version software (Ecochemic, Netherlands). The three-electrode system was used for all measurements. Modified pencil graphite electrode, a platinum wire and Ag/AgCl electrode were used as working electrode, counter electrode and reference electrode, respectively. Surface morphology of the electrodes was evaluated by scanning electron microscopy (FEI Versa 3D; 2.00 kV).

### Fabrication of TKO-modified PGE

A Noki pencil model 2000 (Japan) was used as a holder for graphite leads (Tombow, HB, 0.7 mm diameter, Japan). The pencil graphite electrode (PGE) was prepared by cutting the leads into 3-cm-long sticks. Electrical contact with the lead was obtained by soldering a metal wire to the metallic part. PGEs were washed with acetonitrile to remove the impurity and dried at room temperature before use. Then, PGE was immersed into the polymerization solution. The TKO-modified PGE was obtained by electrodeposition on the surface of the PGE using cyclic voltammetry in the potential range between  $-0.6$  and  $+1.2$  V during eight cycles (scan rate: 80 mV/s). The polymerization solution includes 0.1 M tetrabutylammonium perchlorate (TBAP), 0.1 M pyrrole and 0.1 M 4-(4-methylphenyl aminoisonitrosoacetyl)biphenyl (TKO) in acetonitrile. A control electrode (polypyrrole electrode without a TKO) was prepared in every experiment under the same experimental conditions but without adding the TKO in order to confirm the reliability of the measurements.

### Voltammetric measurements

The electroanalytical determination of the concentration of mercury in aqueous samples was carried out in three-electrode cell with 0.1 M lithium perchlorate solution. Current measurements were taken using differential

pulse voltammetry in a potential range between 0.00 and +1.00 V at a scan rate of  $15 \text{ mV s}^{-1}$ , modulation amplitude of 50 mV and step potential of 8 mV.

The applicability of the electroanalytical working procedure for mercury determination in natural matrices was tested by preparing different water samples.

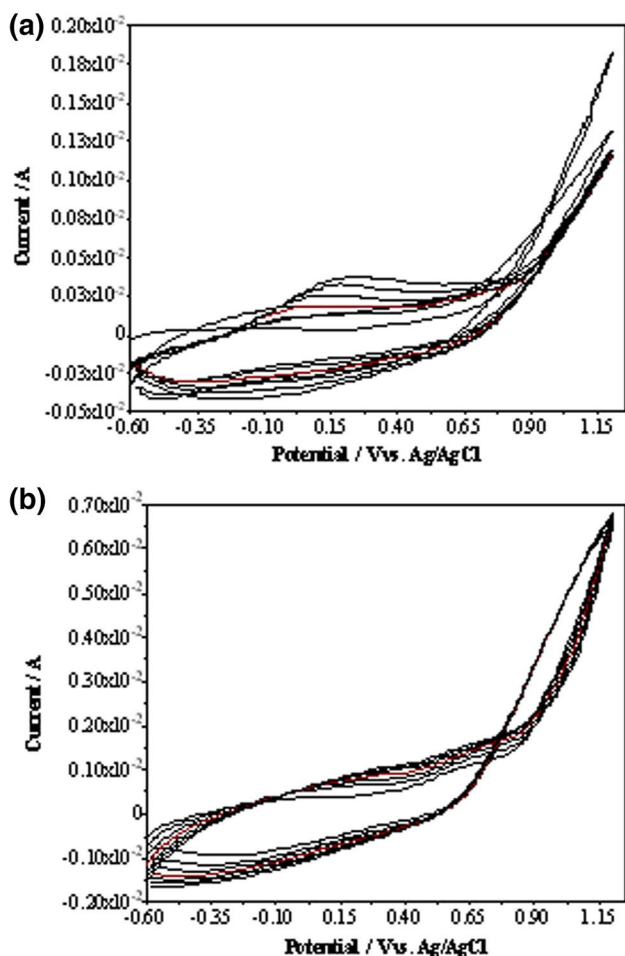
## Results and discussion

### Modification of pencil graphite electrode

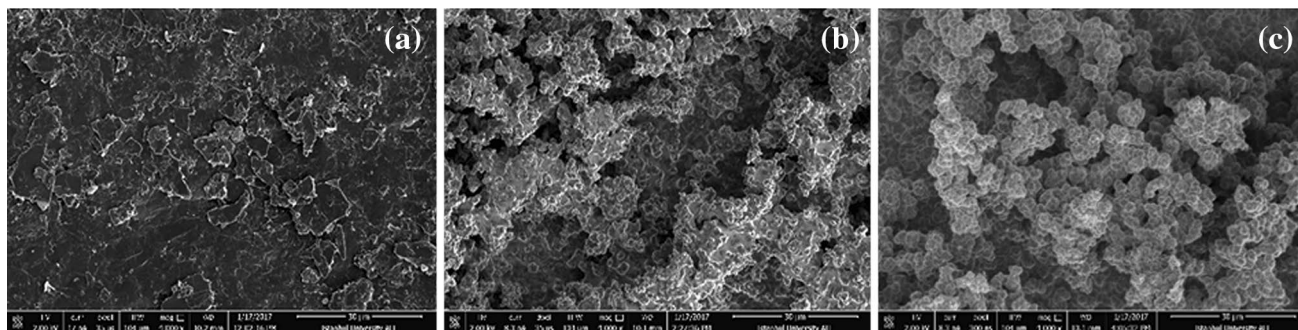
The electrochemical behavior of pyrrole was investigated in acetonitrile solution of 0.1 M TBAP using potential cycling between  $-0.6$  and  $+1.2$  V (versus Ag/AgCl) with PGE. Electrooxidation of pyrrole monomer occurs at the anode, and the resulting polymer deposits on the surface of PGE. An anodic peak of pyrrole was observed at a peak potential of 1.10 V. The oxidation peak corresponds to the formation of pyrrole radical cations. Figure 2a demonstrates eight cycles obtained in the same solution. The formation and growth of the polypyrrole film can be easily seen in this figure. As the film grows, the intensity of the peaks due to the oxidation and reduction of the film was increased.

Modification of the electrode was conducted by adding 1.0 M TKO as a modifier to the electrochemical cell. Figure 2b demonstrates the cyclic scans of electropolymerization of pyrrole in the presence of TKO. The effect of TKO on the electropolymerization of pyrrole can easily be seen in this figure. The oxidation and reduction peak potentials of polypyrrole are shifted toward anodic region, from  $+0.25$  to  $+0.50$  V and from  $-0.25$  to  $+0.03$  V, respectively. These peaks indicate that the oxime compound became a part of the polymeric chain.

SEM measurements were also taken on bare PGE, polypyrrole and TKO-modified electrodes. Scanning electron microscope is used to observe the morphology and roughness of the electrode surfaces before and after modification. Pictured above is the surface morphology under the condition of  $4000\times$  magnification resolution by scanning electron microscopy (SEM) analysis. The surface structures of bare PGE, PPy and PPy-TKO



**Fig. 2** Cyclic voltammograms taken during the electropolymerization of pyrrole (0.1 M) **a** Multisweep cyclic voltammograms without and **b** with TKO (0.1 M) onto a pencil graphite electrode (scan rate:  $80 \text{ mV s}^{-1}$ , supporting electrode: 0.1 M TBAP; number of scan: 8)



**Fig. 3** SEM images for **a** Bare PGE electrode, **b** PPy electrode, **c** TKO-modified electrode under the same magnification

are shown in Fig. 3. It is clear that the surfaces of the electrode were coated (Fig. 3a, b). The porous surface of PPy is shown in Fig. 3a. As can be seen in Fig. 3b, PPy-TKO has a typical “cauliflower” morphology. The difference in SEM images of PPy and TKO-PPy shows that the oxime compound goes into structure during polymerization.

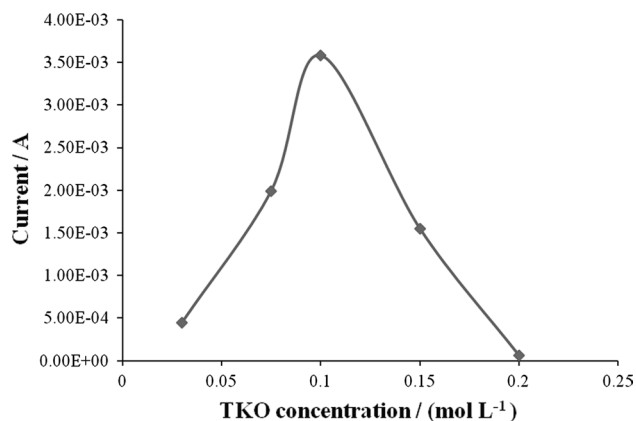
### Effect of the TKO concentration

In order to determine the effect of TKO concentration on the response of TKO-modified PGE, the electrodes were prepared in solutions of varying concentration of TKO in the range of 0.03–0.2 M. The response of the TKO-modified PGE to Hg(II) was found to increase with increasing TKO concentration up to 0.1 M as shown in Fig. 4. There was a considerable decrease in the response of the electrode below and above this TKO concentration. Based on the results, the optimum TKO concentration was chosen as 0.1 M.

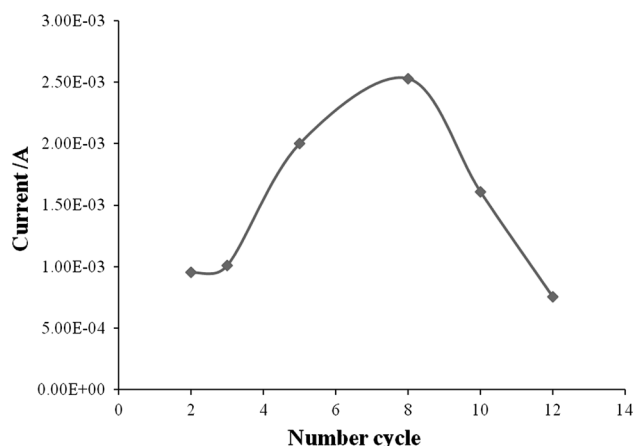
### Effect of the electropolymerization cycles

The optimum number of cyclic voltammetry cycles to form the sensing layer of the electrode was determined from a series of experiments in which electrodes were fabricated with different numbers of cycles (Fig. 5).

It was found that the number of cycles applied to the cell during the electropolymerization affects the sensitivity and linearity of the sensor. There was considerable decrease in the performance of the TKO-modified PGE below and above eight cycles. The optimum polymerization cycles were found to be eight.



**Fig. 4** Effect of the TKO concentration on TKO-modified PGE for 0.01 M mercury(II) ion



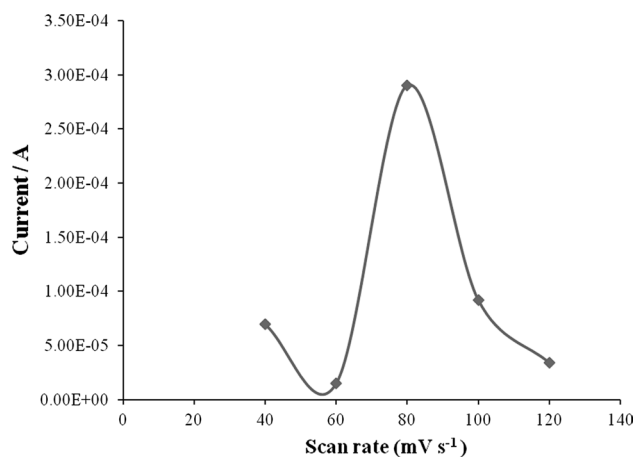
**Fig. 5** Effect of the number cycle on the DPV peak current of mercury(II) ions with TKO-modified PGE

### Effect of scan rate

The scan rate during the electropolymerization process is also a significant factor in the response of the electrode. Figure 6 shows the effect of the scan rate in the range of 20–140  $\text{mV s}^{-1}$ . The highest peak current intensity was observed at a scan rate of 80  $\text{mV s}^{-1}$ .

### Interferences

In the scope of this study, selectivity of the proposed method was evaluated in the presence of different metal ions. Voltammetric response of the modified electrode was examined in the presence of some possible interfering metal ions like Ag(I), Pb(II), Cu(II), Cd(II) and Fe(II). Differential pulse voltammograms were taken for 10 mM Hg(II) after addition of varying concentrations of each



**Fig. 6** Effect of scan rate on TKO-modified PGE for 0.01 M mercury(II) ion

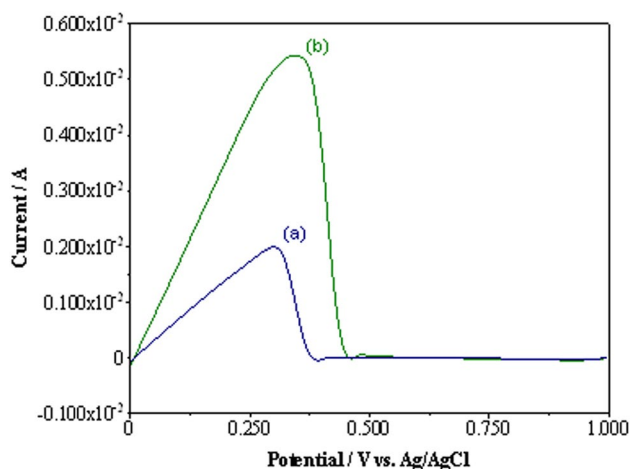
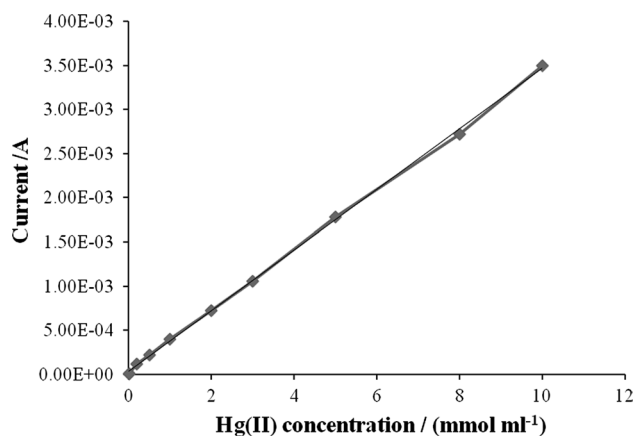
**Table 1** Changes in DPV peak current of 10 mM Hg(II) in the presence of interferents

Concentration (mM)	Change in peak current ( $\mu\text{A}$ ) in the presence of interferents				
	Ag(I)	Pb(II)	Cu(II)	Cd(II)	Fe(II)
5	-1.31	1.01	0.58	1.20	-
10	-1.71	2.74	1.29	1.59	-
20	-2.47	2.86	1.66	2.03	-
50	-4.26	3.05	1.91	3.36	-
100	-8.50	3.11	2.00	6.80	-

interferent (5–100 mM). Obtained current in the absence of any interferent was 3.5 mA with TKO-modified electrode. As can be seen from Table 1, at higher concentrations of these interferents, the variation was within  $\pm 8.5 \mu\text{A}$  relative to their absence. Thus, the response of Hg(II) at the modified electrode is not affected by the studied interferents for at least 10-fold concentrations.

### Voltammetric behavior of mercury(II) ion

The electrochemical behavior of mercury (0.01 M) was investigated for “TKO modified electrode” and “polypyrrole electrode without TKO” by differential pulse voltammetry method. The effect of the TKO electrode is demonstrated in Fig. 7. It was found that the Hg(II) response of polymeric electrode with TKO as modifier was greater than the other electrode. This result proved that the TKO oxime compound was improved the peak current value by acting as a modifier in Hg(II) determination. Thus, Hg(II) selectivity of developed electrode was enhanced by TKO application. As a result, modification process was successfully

**Fig. 7** Differential pulse voltammograms for (a) 0.01 M mercury at polypyrrole electrode (b) 0.01 M mercury at TKO-modified electrode in 0.1 M  $\text{LiClO}_4$ **Fig. 8** Calibration plot of mercury(II) between peak current and concentration of mercury(II) at TKO-modified electrode

completed. Under the optimized conditions, a series of concentration of mercury(II) standard solutions were tested to determine the linearity of mercury(II) ions at the modified electrode. Figure 8 shows a calibration plot obtained from data of differential pulse voltammetry with the TKO-modified electrode. The peak current increased linearly as the concentration of the mercury(II) ion increased in a wide concentration range from  $1.10 \times 10^{-5}$  to  $1.10 \times 10^{-3}$  M with a correlation coefficient of 0.9994. The limit of detection (LOD) and limit of quantification (LOQ) of TKO-modified PGE were calculated according to the 3  $s/m$  and 10  $s/m$  criterious, respectively. The LOD and LOQ were found as  $5.85 \times 10^{-7}$  and  $1.95 \times 10^{-6}$  M, respectively. Table 2 shows a comparison of the proposed electrode with other electrodes described recently in the literature for the quantification of Hg(II).

### Reproducibility of the electrode

The reproducibility of the response of the electrode was investigated for 10 mM Hg(II). The peak current of mercury was determined with six electrodes which are produced under exactly same conditions. The response peak intensity showed a relative standard deviation of 3.6% which confirmed that the results are reproducible.

### Applications

The applicability of the proposed electrode for analysis of real samples was assessed by its application to the determination of Hg(II) ions in tap water samples. Water samples were spiked with different concentrations of Hg(II). Five determinations were performed for each addition. The obtained results are given in Table 3. The recovery values were in the range of 92–103% for Hg(II) in tap water

**Table 2** Comparison of the analytical performance of recently reported modified electrodes for Hg(II) detection

Electrodes	Method	Detection limit	Linear ranges	References
RGO-NH-Ci/GCE <sup>a</sup>	Differential pulse anodic stripping voltammetry (DPASV)	0.2 nM	0.6–1000 nM 10–9000 nM	[51]
AuNP/GCE <sup>b</sup>	Square wave anodic stripping voltammetry (SWASV)	0.01 $\mu\text{g L}^{-1}$	0.2–2 $\mu\text{g L}^{-1}$ , 2–10 $\mu\text{g L}^{-1}$	[52]
AuNPs/GCE <sup>b</sup>	Cyclic voltammetry (CV)	0.8 $\mu\text{g L}^{-1}$	1.2–8.0 $\mu\text{g L}^{-1}$	[53]
PbNPs-SH-PF/GCE <sup>c</sup>	Anodic stripping voltammetry	0.35 nM	0.001–1.0 $\mu\text{M}$	[54]
MCPE <sup>d</sup>	Differential pulse voltammetry (DPV)	$1.0 \times 10^{-8}$ M	$1.0 \times 10^{-6}$ – $8.0 \times 10^{-5}$ M, $1.0 \times 10^{-7}$ – $5.0 \times 10^{-6}$ M	[55]
RGO-IIP/GCE <sup>e</sup>	SWASV	0.02 $\mu\text{g L}^{-1}$	0.07–80 $\mu\text{g L}^{-1}$	[56]
ssDNA/Au electrode <sup>f</sup>	DPV	0.1 $\mu\text{M}$	0.1–2.0 $\mu\text{M}$	[57]
BIM/MCNF/Nafion-GCE <sup>g</sup>	DPASV	0.3 nM	5–500 nM	[58]
KLA-CPE <sup>h</sup>	DPASV	$1.11 \times 10^{-8}$ M	$2.0 \times 10^{-7}$ – $1.0 \times 10^{-4}$ M	[59]
MCPE <sup>i</sup>	DPV	0.0782 $\mu\text{g L}^{-1}$	1–100 $\mu\text{g L}^{-1}$	[60]
NG/GCE <sup>j</sup>	DPSV	0.05 $\mu\text{M}$	0.2–9 $\mu\text{M}$	[61]
TKO/PGE <sup>k</sup>	DPV	$5.85 \times 10^{-7}$ M	$1.0 \times 10^{-5}$ – $1.0 \times 10^{-3}$ M	This work

<sup>a</sup> Reduced graphene oxide-NH-carboimidazole-modified glass carbon electrode

<sup>b</sup> Gold nanoparticle-modified glassy carbon electrode

<sup>c</sup> Lead nanoparticles-modified thiol-functionalized polysiloxane film glassy carbon electrode

<sup>d</sup> Graphitic carbon nitride/chitosan-modified carbon paste electrode

<sup>e</sup> Reduced graphene oxide-ion-imprinted polymer-modified glassy carbon electrode

<sup>f</sup> Single-stranded DNA polycrystalline gold electrode

<sup>g</sup> Bis(indolyl)methane/Mesoporous carbon nanofiber/Nafion/glassy carbon electrode

<sup>h</sup> Lacunar apatite (KCdPb<sub>3</sub>(PO<sub>4</sub>)<sub>3</sub>)-modified carbon paste electrode

<sup>i</sup> 2,2'-((Pyridine-2,6-diylbis(azanylylidene))bis(methanylylidene)) diphenol-modified carbon paste electrode

<sup>j</sup> N-Doped graphene (NG)-modified electrode

<sup>k</sup> 4-(4-Methylphenyl aminoisonitrosoacetyl)biphenyl (TKO)-modified pencil graphite electrode

**Table 3** Results of Hg(II) analysis in tap water samples under the optimized conditions

Added (mM)	Found (mM)	Recovery (%)
0.25	0.23	92 ± 3.32
0.5	0.485	97 ± 1.43
1	1.03	103 ± 3.44

samples. The results proved the successful applicability of the proposed methods for simultaneous determination of Hg(II) in such water samples.

## Conclusions

This study has shown the ability of the 4-(4-methylphenyl aminoisonitrosoacetyl)biphenyl as a very suitable modifier for constructing an efficient and highly selective electrode for determination of Hg(II) ion by differential pulse voltammetry. The experimental conditions were optimized, and the response characteristics were determined. The

electrode generally offers attractive properties such as simplicity of electrode preparation, low-cost fabrication and high stability. A linear relationship between mercury concentration and current response was obtained with excellent reproducibility of the current and a low detection limit of  $5.85 \times 10^{-7}$ . The electrode was successfully applied to determine trace amounts of Hg in a water sample without any interference from other metal ions.

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