

# **Preparation and Characterization of New Carbon Paste Microelectrode Modified with potassium hydrotris{N-(2,6-xylyl)-2-thioimidazolyl}borate and Its Application in the Voltammetric Determination of Hg (II)**

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**Abstract:** A new carbon paste microelectrode modified by potassium hydrotris{N-(2,6-xylyl)-2-thioimidazolyl}borate was developed for the electrochemical determination of Hg(II) by square wave voltammetry. The electrochemical behavior of Hg(II) was studied on the modified electrode in the absence and presence of potassium hydrotris{N-(2,6-xylyl)-2-thioimidazolyl}borate using cyclic voltammetric techniques. At the surface of the prepared electrode, Hg(II) showed a well defined redox couple system, the anodic and cathodic peaks appeared at about 0.14 and 0.01 V (vs. SCE), respectively, with a potential difference ( $\Delta E_p$ ) about 130 mV. Using square wave voltammetry, Hg(II) gave a well defined oxidation peak. The effect of different experimental parameters was optimized to get the highest peak current for the electrochemical determination of Hg(II) using square wave voltammetry. The effect of some interference due to the presence of other heavy metals on the peak height of Hg(II) was studied. Under the optimum conditions, a linear relationship existed between the currents and the concentrations of Hg(II) in the range from  $1 \times 10^{-6}$  M to  $1 \times 10^{-4}$  M with the detection limit of  $1.7 \times 10^{-7}$  M. This method was applied for the determination of Hg(II) different water samples and the resulted data was compared by the data obtained from the standard ICP-AES instrument.

**Keywords:** Carbon paste electrode, Hg(II), Cyclic voltammetry, Water analysis.

## **I. INTRODUCTION**

Environmental pollution is one of the most serious problems in our recent world. The most serious environmental problem is the pollution by heavy metals. Mercury(II) [(Hg)(II)] is one of the most toxic element in the environment, because it is highly reactive, extreme volatile and its relative solubility in water and living tissues. It is highly bioconcentrated through the food chain and cause serious damages to both nervous and immune systems [1]. Hg(II) and its compounds are accumulated in vital organs and tissues such as heart muscles, liver and brain and cause chronic diseases, kidney injury, respiratory failure, central nervous system disorders, brain damages, and can even death [2,3]. Mercury analysis is very important due to its high toxicity and water analysis is also very important because of the continuous contaminations of natural waters by industrial waste. So the development of highly sensitive method for the determination of trace amount of Hg(II) has received considerable attention. Different techniques have been employed for the determination of trace amount of Hg(II) such as potentiometry [4-5], inductivity coupled plasma [6-7] X-ray florescence spectroscopy [8-9], atomic adsorption spectroscopy (AAS) [10-12], spectrophotometry [13] and liquid-liquid extraction [14]. These methods have excellent sensitivity and good selectivity, but have different drawbacks such as time consuming used for analysis and very expensive instruments are required. Electrochemical methods including stripping voltammetric techniques such as anodic stripping voltammetry, cathodic stripping voltammetry, square wave voltammetry, differential pulse voltammetry and chronocoulometry have recognized a powerful tool for measuring

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trace analysis of Hg(II) [15-30]. It have shown different advantages such as speed of analysis, higher selectivity and sensitivity, low cost, easy operation and the ability of analyzing element speciation. Chemically modified electrodes have been develop in the recent years for the electrochemical determination of heavy metals which have different advantages such as easy manufacture, no poison, renewable, fast response, high selectivity, low detection limit, stable in various solvent, longer life time and low cost. A number of chemically modified electrodes were developed for the electrochemical determination of Hg(II) such as Au-nanoparticles sol-gel pt-wire electrode [23] polypyrrole/carbonaceous nanospheres modified screen-printed electrode [31] and chemically carbon paste electrodes [32]. Carbon paste electrode (CPE) is one of the most popular chemically modified electrodes used with voltammetric techniques. This electrode prepared by mixing a graphite powder with a suitable binder and a material known as a modifier that improves the selectivity of the prepared electrode. In the literature survey some modifiers were used with carbon paste electrode for the electrochemical determination of Hg(II) such silica nanoparticles [32], chitosan [33], SBA-15 nanostructured silica organofunctionalised with 2-benzothiazolethiol [34], schiff base [24], and antimony [28]. The aim of this work was the construction a new carbon paste microelectrode modified with potassium hydrotris{N-(2,6-xylyl)-2-thioimidazolyl}borate for the modification of carbon paste microelectrodes to increase the electrode selectivity and sensitivity for the electrochemical determination Hg(II).

## II. METHODOLOGY

### *Chemicals*

All reagents were commercial grade materials and were used without further purification. Graphite powder and paraffin wax were obtained from Sigma-Aldrich.  $H_3BO_3$ ,  $H_3PO_4$ ,  $NaH_2PO_4$ ,  $Na_2HPO_4$ ,  $CH_3COOH$ , and  $NaOH$  were obtained from Merck.  $HgCl_2$  was obtained in a pure form Fluka.

### *Preparation of a bare carbon paste electrode (CPE) and carbon paste modified by potassium hydro tris(N-xylyl-thioimidazolyl)borate*

A bare carbon paste electrode was prepared by mixing 65% graphite powder and 35% paraffin wax. Paraffin wax was heated till melting and then, mixed very well with graphite powder to produce a homogeneous paste. The resulted paste was then packed into the end of an insulin syringe (i.d.: 2mm). External electrical contact was established by forcing a copper wire down the syringe. CPE modified with potassium hydrotris{N-(2,6-xylyl)-2-thioimidazolyl}borate was prepared by mixing 60% graphite powder and 30% paraffin wax with 10% potassium hydrotris{N-(2,6-xylyl)-2-thioimidazolyl}borate. The surface of the electrode was polished with a piece of weighting paper and then rinsed with distilled water thoroughly.

### *Electrochemical Measurements*

Cyclic voltammetry (CV) and square wave voltammetry were performed using an Autolab potentiostat PGSTAT 302 (Eco Chemie, Utrecht, The Netherlands) driven by the General Purpose Electrochemical Systems data processing software (GPES, software version 4.9, Eco Chemie). Electrochemical cell with three electrodes was used; unmodified carbon paste electrode or carbon paste electrode modified with potassium hydrotris{N-(2,6-xylyl)-2-thioimidazolyl}borate was used as a working electrode, SCE was used as a reference electrode while platinum wire was used as a counter electrode. The pH values were measured using a Metrohm pH-meter with a combined glass electrode. pH values were adjusted using 1 M NaOH solution.

## III- RESULTS AND DISCUSSION

### *Cyclic voltammetric studies:*

The electrochemical behavior of CPE modified by potassium hydrotris(N-xylyl-thioimidazolyl)borate in the absence and presence of Hg(II) was examined using different supporting electrolytes such as acetate, phosphate, HCl,  $HNO_3$ , borate and Britton-Robinson buffers. The best peak shape and highest peak current was obtained using acetate buffer. The effect of supporting electrolyte pH on the peak current signals for  $1 \times 10^{-3}$  M Hg(II) was studied using acetate buffer. As shown from Fig. 1 acetate buffer pH 4.5 give the best peak shape and the highest peak current. So for further studies acetate buffer with pH 4.5 was used.

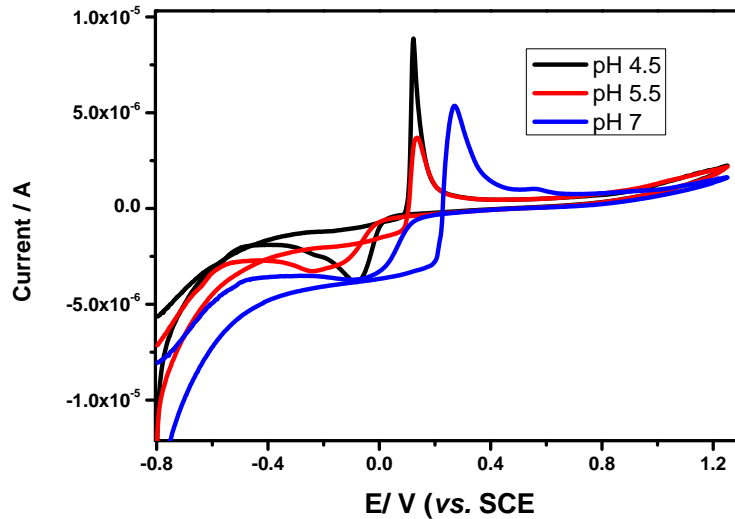


Fig. 1. The effect of acetate buffer pH on the peak current of  $1 \times 10^{-3}$  M Hg(II).

The electrochemical behavior of Hg(II) at carbon paste electrode modified by potassium hydrotris(N-xyllyl-thioimidazolyl)borate was studied using a potential range from -0.3 to +0.8 V (vs. SCE) in 0.1 M acetate buffer (pH 4.5) with a scan rate of 30 mV/s (Fig 2). The resulted voltammograms for  $1 \times 10^{-5}$  M Hg(II) at carbon paste electrode modified by potassium hydrotris(N-xyllyl-thioimidazolyl)borate, showed a well defined redox couple, the anodic peak appeared at about 0.14 V (vs. SCE) and the cathodic peak appeared at about 0.01 V with a 130 mV  $\Delta E_p$  (Fig. 2). The redox peaks due to the oxidation and reduction of Hg(II) ion. As  $\Delta E_p$  is a function of the rate of electron transfer, the lower  $\Delta E_p$ , the higher electron transfer rate. The results obtained greatly improved the voltammetric response of Hg(II) at CPE modified potassium hydrotris(N-xyllyl-thioimidazolyl)borate .

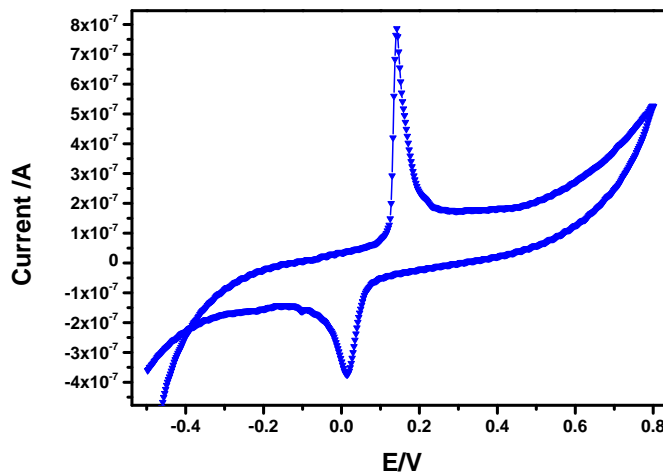


Fig.2. Cyclic voltammetric behavior for  $1 \times 10^{-5}$  M Hg(II) in carbon paste electrode modified by modified by potassium hydrotris(N-xyllyl-thioimidazolyl)borate, scan rate 30mV/s.

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### Effect of scan rate

The effect of scan rate on carbon paste electrode modified by modified by potassium hydrotris(N-xyllylthioimidazolyl)borate was studied using cyclic voltammetry at different scan rate values (from 10 to 30 mV/s) Fig. 3. By increasing the scan rate value the oxidation and reduction peak current increased. By plotting the scan rate value against the peak current for anodic and cathodic peak current for Hg(II), a linear relation was obtained (Fig. 4).

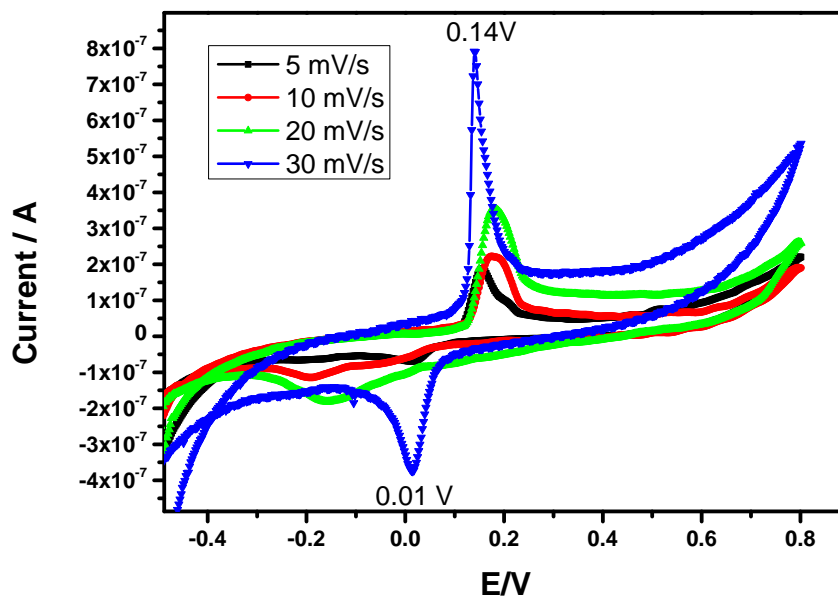


Fig 3. Cyclic voltammograms for  $1 \times 10^{-3}$  M Hg(II) using different scan rate values (from 10 to 30).

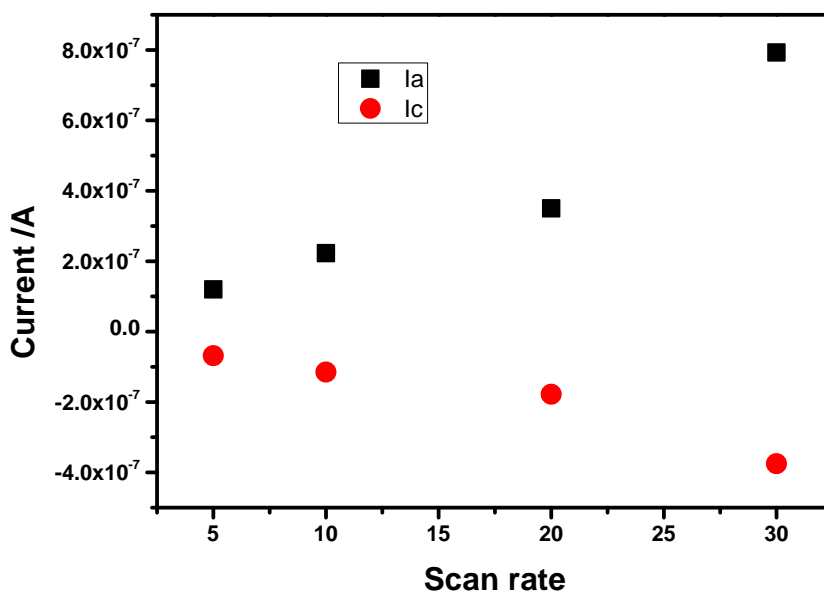


Fig 4. The relation between scan rate and peak current values for anodic (a) and cathodic peak current (b) for  $1 \times 10^{-3}$  M Hg(II).

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The effect of scan rate on the peak current height of Hg(II) ions was also studied using linear sweep voltammetric techniques. By increasing the scan rate values (from 5 to 75 mV/s) the oxidation peak current increased (Fig. 5a). By plotting the relation between the square root of scan rate and the corresponding peak current a linear relation was obtained (Fig. 5b). This result showed the electrode process was diffusion controlled.

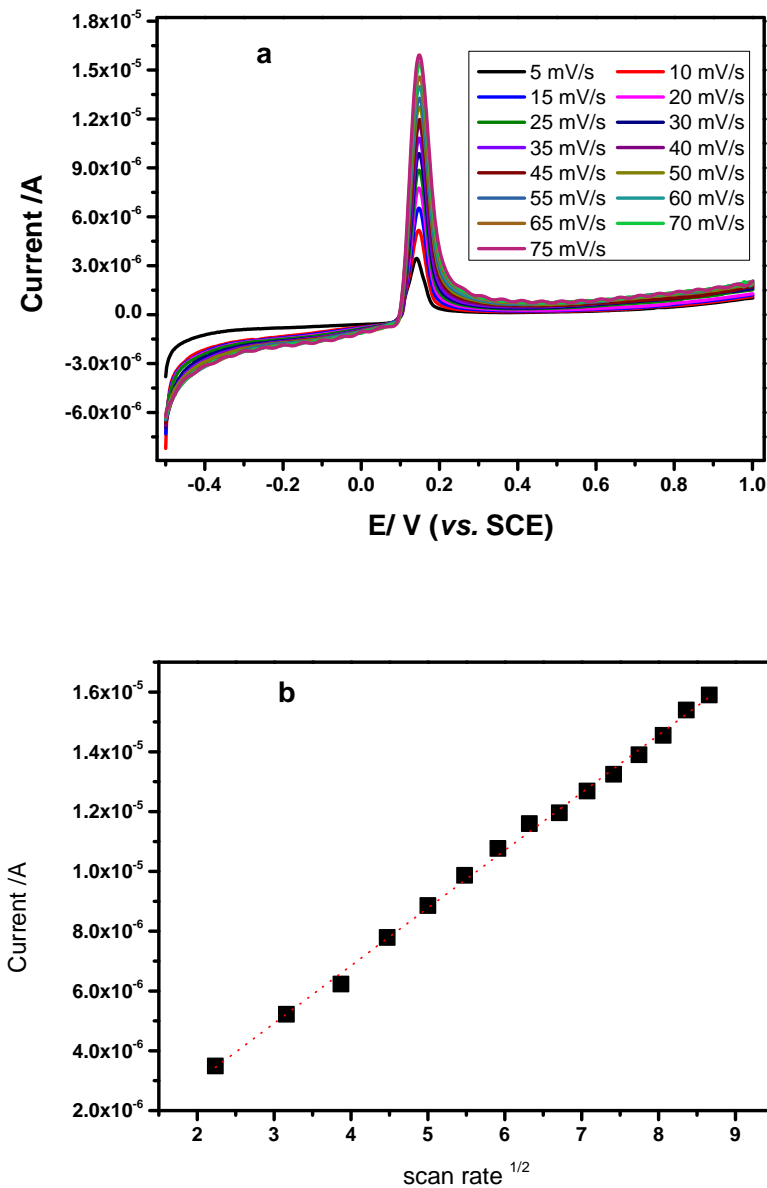


Fig 5. a) Linear Sweep voltammograms for  $1 \times 10^{-3}$  M Hg(II) using different scan rate values (from 5 to 75), b) plotting the relation between the square root of scan rate and peak current for  $1 \times 10^{-3}$  M Hg(II).

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### Square wave voltammetric Studies :

The electrochemical behavior of Hg(II) at a bare CPE electrode and carbon paste electrode modified by potassium hydrotris{N-(2,6-xylyl)-2-thioimidazolyl}borate was studied using acetate buffer at different pH values (4.5) using square wave voltammetry. Fig. 6. showed the resulted voltammograms for  $1 \times 10^{-3}$  M Hg(II) ions. Fig. 6 showed the square wave voltammograms for Hg(II) using at a bare CPE electrode and carbon paste electrode modified by potassiumhydrotris(N-xylyl-thioimidazolyl)borate.

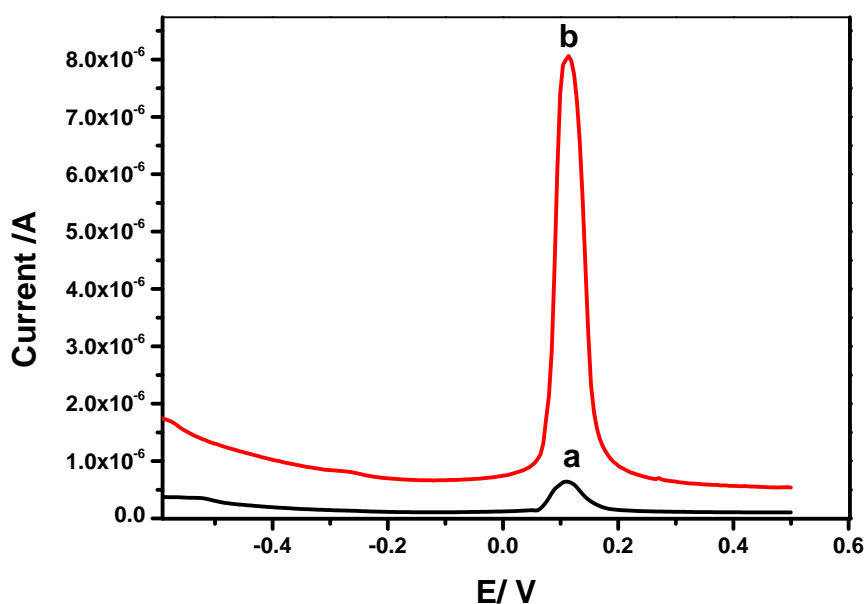
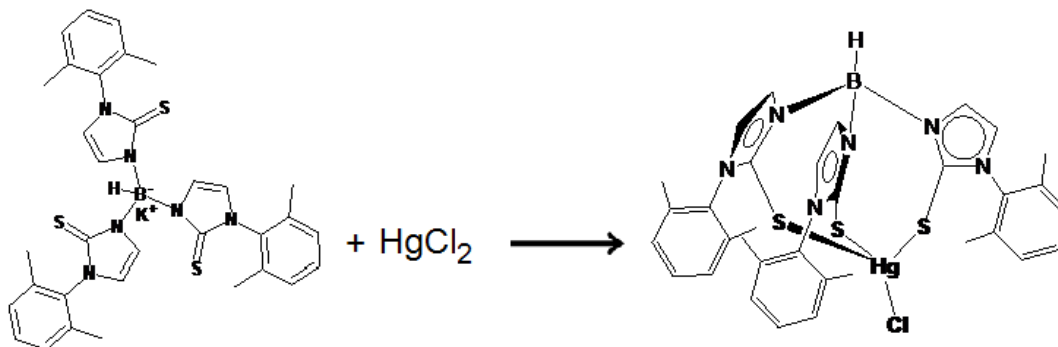


Fig. 6. The square wave voltammograms for Pb(II) using a bare carbon paste electrode (CPE) (a) and carbon paste electrode modified with potassiumhydrotris(N-xylyl-thioimidazolyl)borate (b).

It is clear that the anodic peak current for Hg(II) in the presence of a potassium hydrotris{N-(2,6-xylyl)-2-thioimidazolyl}borate is much higher than the peak current of Hg(II) in case of a bare carbon paste electrode. This enhancement in the oxidation peak current due to the increment of Hg(II) concentration in the electrode surface by the complex formation between Hg(II) and potassium hydrotris{N-(2,6-xylyl)-2-thioimidazolyl}borate ligand according to the following scheme 1.



**Scheme 1.** Complex formation between Hg(II) and potassium hydrotris{N-(2,6-xylyl)-2-thioimidazolyl}borate ligand

**Effect of square wave voltammetric parameters**

The effect of different square wave voltammetric parameters was examined on the peak height of  $1 \times 10^{-3}$  M Hg(II) as shown in Fig. 7. Fig. 7a shows the influence of square wave frequency on the peak current of  $1 \times 10^{-3}$  M Hg(II) using different values from 8 to 20 Hz. By increasing the square wave frequency, the oxidation peak current increased up to 12 and after that by increasing the square wave frequency, the oxidation peak current decreased. A linear part was observed from 8 to 12 Hz for Hg(II). Thus for further study, 12 Hz square wave frequency was selected for further investigations. The effect of square wave pulse amplitude on the peak current of  $1 \times 10^{-3}$  M for Hg(II), using 12 Hz square wave frequency is shown in Fig. 7b. The pulse amplitude ranged from 1.0 to 85 mV. The peak current increased linearly from 1.0 to 55 mV for Hg(II). Therefore, a 55 mV will be the optimum square wave pulse amplitude height and will be used in the next work.

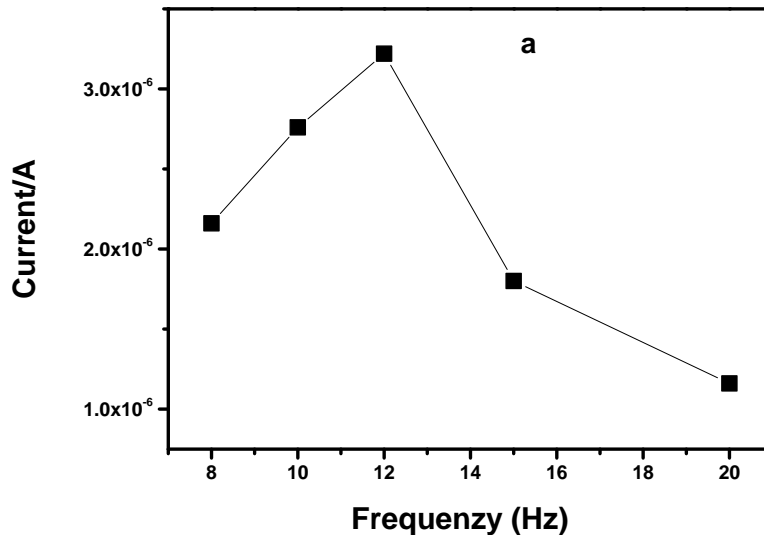


Fig. 7a. The influence of square wave frequency on the peak current of  $1 \times 10^{-3}$  M Hg(II) using different values from 8 to 20 Hz.

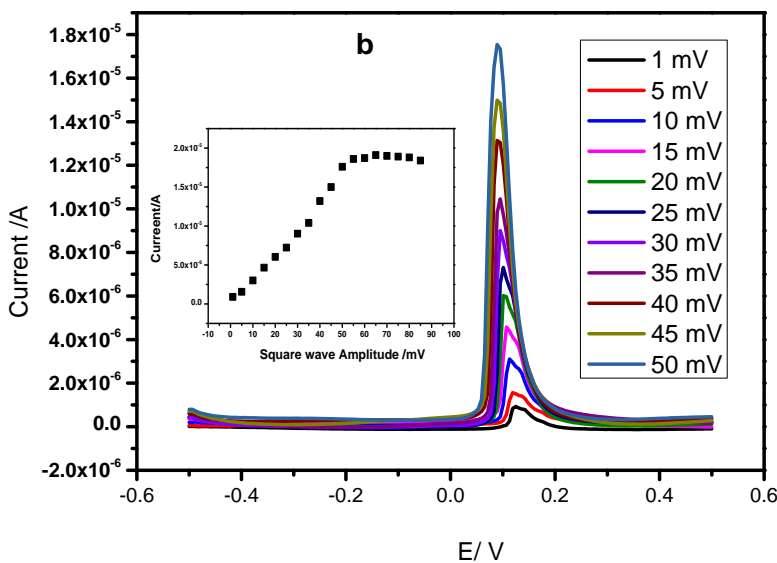


Fig. 7b. The effect of square wave pulse amplitude on the peak current of  $1 \times 10^{-3}$  M Hg(II) using 12 Hz square wave frequency. The pulse amplitude ranged from 1.0 to 85 mV.

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The effect of step potential (1- 10mV) on the peak height of  $1 \times 10^{-3}$  M Hg(II) ions is depicted using 12 Hz square wave frequency and 55 mV square wave pulse amplitude and the other experimental parameters as shown in Fig 7. As the step potential increases, the peak height increases linearly up to 7 mV, after that the increasing in the peak height is not pronounced (Fig. 7c). So step potential with 7 mV was selected for further studies

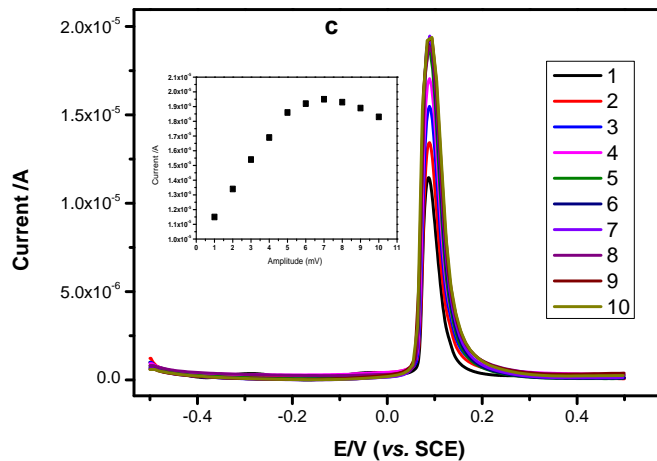


Fig 7c. The effect of step potential (1-10 mV) on the peak height of  $1 \times 10^{-3}$  M Hg(II) ions using 12 Hz square wave frequency and 55 mV square wave pulse amplitude.

### *Effect of Initial potential*

The effect of initial potential on the peak current height of  $1 \times 10^{-3}$  M Hg(II) ions was investigated using a wide range of potential from -2.0 V to 0.0 V using 12 Hz square wave frequency, 55 mV square wave pulse amplitude and 7 mV step potential. As shown from Fig. 8 by increasing the initial potential to more positive values, the peak current for  $1 \times 10^{-3}$  M Hg(II) increased till -1.6 V after this value the peak current decreased. So, -1.6 V was selected as an initial potential for further studies, where a lower base line was observed in this potential.

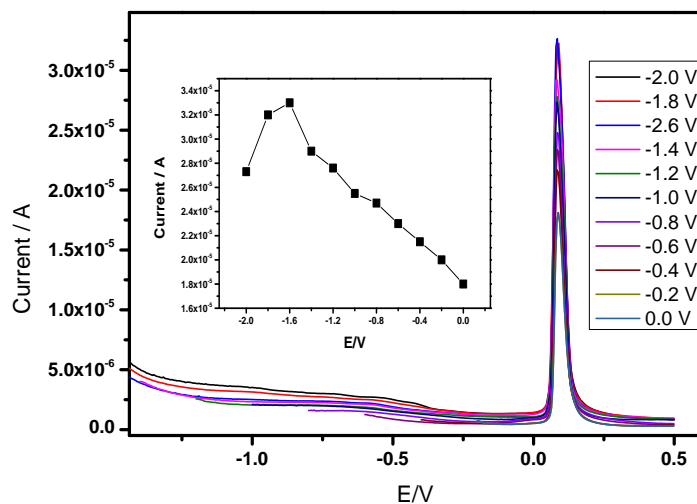


Fig. 8. The effect of initial potential on the peak current height of  $1 \times 10^{-3}$  M Hg(II) ions.



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### Calibration curve and Detection limit

To examine the readability of the prepared electrode under investigation, the following optimum conditions were used for the square wave determination of Hg(II) ions: acetate buffer (pH 4.5), 12 Hz square wave frequency, 55 mV square wave pulse amplitude and 7 mV step potential. The effect of concentration on the peak current of Hg(II) ions was examined from  $1 \times 10^{-6}$  M to  $1 \times 10^{-4}$  M, the resulted calibration curve showed a linear ranges in the concentration range used with a correlation coefficient of 0.994 and a relative standard deviation (RSD) of  $8.73 \times 10^{-8}$  (Fig. 9).

**Limit of detection:** The detection limit is characteristic of limit test. It is lowest amount of analyt present in sample that can be detected but not necessarily quantities, under stated condition. The lower detection limit (LOD) for Hg(II) was calculated based on the standard deviation of the response (SD) and the slope of the calibration curve (m) at levels approximating the LOD according to the formula:  $LOD = 3(SD/m)$  and it was found to be  $1.7 \times 10^{-7}$  M for Hg(II).

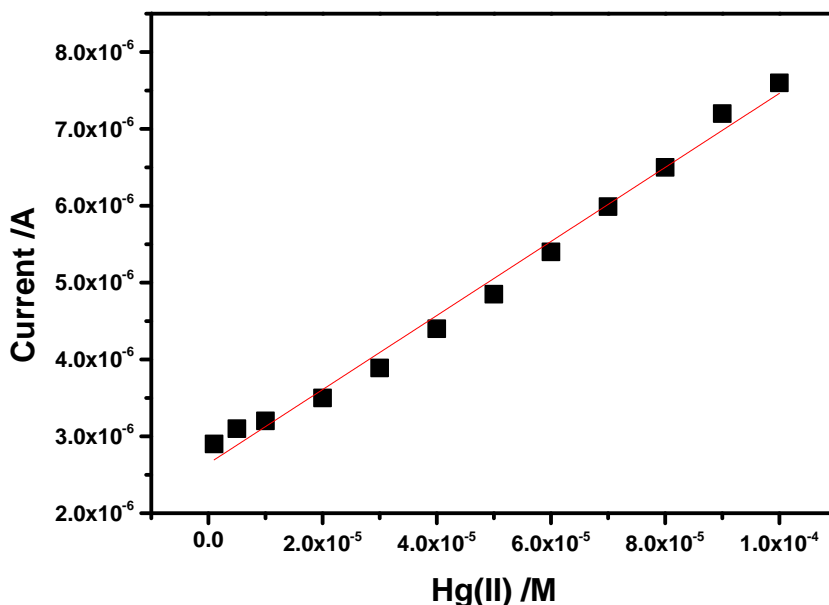


Fig. 9 Calibration plots for different Hg(II) ions concentration from  $1 \times 10^{-6}$  M to  $1 \times 10^{-4}$  M using the optimum conditions mentioned before

### Reproducibility

To examine the readability of the prepared electrode under investigation, the produced peak current of  $1 \times 10^{-5}$  M Hg(II) using the optimum conditions mentioned above was examined by successive ten measurements. The RSD was calculated and it was found to be 5.63 %, and this value indicates that this method gives a good reproducibility for the obtained results.

### Effect of some Interferences

The effect of various metal ions on the simultaneous determination of  $1 \times 10^{-4}$  M Hg(II) was studied. The studied metal ions includes Cd(II), Pb(II) Zn(II), Fe(II), Mg(II), Al(III), Cu(II), Ni(II) and Mn(II). The concentration of added metal ions ranged from  $1 \times 10^{-4}$  to  $1 \times 10^{-4}$  M. Addition of  $1 \times 10^{-4}$  M form Cd(II) and Pb(II) to  $1 \times 10^{-4}$  M Hg(II) showed no effect where Cd(II) and Pb(II) showed a separate signals at about -0.77 V and -0.44 V for Cd(II) and Pb(II), respectively (Fig. 10), and these peaks are far away from the oxidation peak for Hg(II). Addition of other metal ions showed a small error about  $\pm 7\%$  was observed.

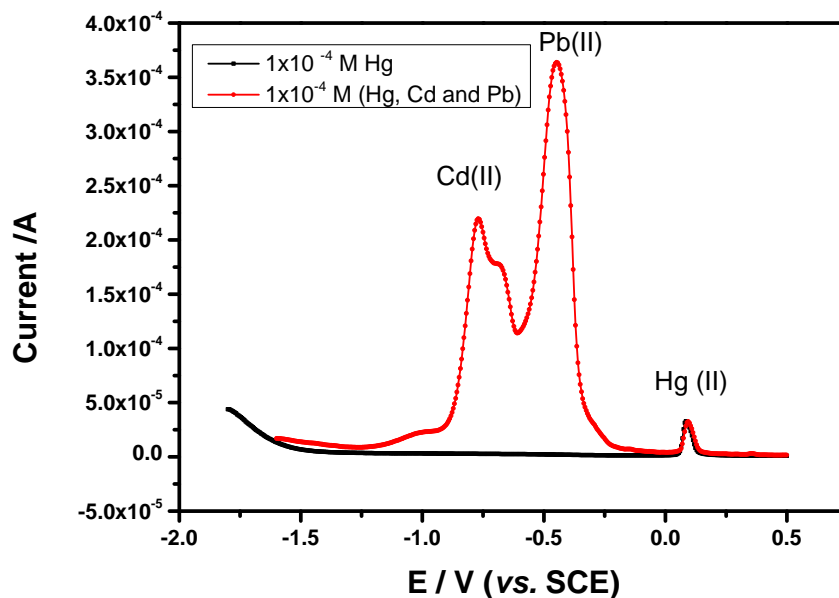


Fig. 10. The effect of  $1 \times 10^{-4}$  M from Cd(II) and Pb(II) ions on the peak current signals for  $1 \times 10^{-4}$  M from Hg(II) ions.

**Analytical Applications**

To study the validity of the prepared carbon paste electrode modified by potassiumhydrotris(N-xyl-ylthioimidazolyl)borate, the proposed method was applied for the simultaneous determination of Hg(II) in water samples. Using the optimum conditions and calibration curve the obtained results are represented in Table 1.

In order to assess the reliability of the new electrode the investigated water samples were also determined using, inductively coupled plasma-atomic emission spectroscopy ICP-AES, independent standard methods. The results obtained were presented in Table 1. The measurements data showed a good agreement with those obtained by standard ICP-AES for the investigated water samples.

No. of sample	Hg(II) [M] Added	Proposed Method	ICP-AES	Relative error (%)
1	$1 \times 10^{-6}$	$1.02 \times 10^{-6}$	$0.96 \times 10^{-6}$	5.88
2	$3 \times 10^{-6}$	$2.89 \times 10^{-6}$	$2.68 \times 10^{-6}$	7.26
3	$5 \times 10^{-6}$	$4.79 \times 10^{-6}$	$5.1 \times 10^{-6}$	-6.47
4	$7 \times 10^{-6}$	$6.67 \times 10^{-6}$	$7.16 \times 10^{-6}$	-7.34
5	$9 \times 10^{-6}$	$9.65 \times 10^{-6}$	$9.34 \times 10^{-6}$	3.21
6	$1 \times 10^{-5}$	$1.10 \times 10^{-5}$	$1.17 \times 10^{-5}$	-6.36
7	$3 \times 10^{-5}$	$2.87 \times 10^{-5}$	$2.94 \times 10^{-5}$	-2.44
8	$5 \times 10^{-5}$	$5.14 \times 10^{-5}$	$5.02 \times 10^{-5}$	2.14
9	$7 \times 10^{-5}$	$7.32 \times 10^{-5}$	$7.21 \times 10^{-5}$	1.50
10	$9 \times 10^{-5}$	$9.19 \times 10^{-5}$	$9.03 \times 10^{-5}$	1.74
11	$1 \times 10^{-4}$	$1.07 \times 10^{-4}$	$1.02 \times 10^{-4}$	4.67

Table 1. Determination of Hg(II) in water samples by the proposed method (n=5) and the reference method, ICP-AES instrument

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## IV. CONCLUSION

A new carbon paste microelectrode modified by potassium hydrotris{N-(2,6-xylyl)-2-thioimidazolyl}borate was developed for the electrochemical determination of Hg(II) by square wave voltammetry. The electrochemical behaviour of the prepared electrode was characterized by cyclic voltammetry. The effect of different experimental parameters was optimized to get the highest peak current for the electrochemical determination of Hg(II) using square wave voltammetry. This method was applied for the determination of Hg(II) in water sample.

## V. ACKNOWLEDGEMENT

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