

# Simultaneous Determination of HQ and CC Using GE and Different Modified Electrodes (GE /PFA, GE/MWCNTs,GE/MWCNTs /PFA)

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Article Info	Abstract:
<ul> <li>Volume 83</li> <li>Page Number: 2898 - 2908</li> <li>Publication Issue:</li> <li>March - April 2020</li> <li>Differential Pulse Voltammetric techn electrochemical beaviour of Hydroquin (GE), GE /Poly Furfurylamine (PFA), Tubes(MWCNTs ), GE/MWCNTs /PI Catechol(CC) .A three – electrodes detect of bare or modified graphite electrode.</li> <li>/silver chloride saturated potassium reference electrode and 1.0 mm dia auxiliary electrode. Calibration curves we electrode under the optimum conditions, been examined the standard deviation</li> </ul>	Differential Pulse Voltammetric technique has been used to study the electrochemical beaviour of Hydroquinone (HQ) on Graphite electrode (GE), GE /Poly Furfurylamine (PFA), GE / Multiwalled Carbon Nano Tubes(MWCNTs), GE/MWCNTs /PFA in the present and absent of Catechol(CC) .A three – electrodes detection system was employed consist of bare or modified graphite electrodes as the working electrode, silver /silver chloride saturated potassium chloride (Ag/AgCl,sat.KCL) as reference electrode and 1.0 mm dimeter platinum wire(Pt-wire) as auxiliary electrode.Calibration curves were constructed for each working electrode under the optimum conditions, stability and the effect of pH have been examined ,the standard deviation (S.D) and R <sup>2</sup> values have been
Article History Article Received: 24 July 2019	calculated .The best working electrode was the GE modified by MWCNTs with PFA polymer ,a modification electrode was successfully applied for simultaneous determination of HQ in mixture containing CC.
Revised: 12 September 2019 Accepted: 15 February 2020 Publication: 20 March 2020	<i>Keywords:</i> Modified Electrodes , Conducting Polymer, Hydroquinone and Catechol , Differential Pulse Polarography ,MWCNTs.

## I. Introduction

Hydroquinone(1,4Dihydroxy benzene) is the important compound due to its wider biological and industrial applications, such as: reducing agent(1) , antioxidant(2), cosmetics(3), pesticides, black white film developer(4), dye–generating materials , pharmaceutical intermediate .The US Enviromental Protection Agency has classified the HQ compound as highly toxic enviromental pollutant due to its effect on health such as sever liver injuries, abnormal pigentation and hemolytic jaundice(5).Compared with many analytical methods, including capillary electrphoresis (6) , synchronousfluresense(7), chromatography(8),

spectroscopy(9),colorometry(10),chemiluminesce nce with flow injection (11) ,electrochemical

techniques (12,13) electrochemical techniques are good candidates for detecting various substanses like HQ because their cheap instrument ,simple operation ,high sensitivity ,high accuracy and time -saving (14). The simultaneous determination of HQ,CC suffer from the peak overlaps specially at low concentration, because they have redox potential very closed to each other so it is difficult to distingwish between the oxidation peaks to solve this problem many sensing materials with high stability, high catalytic activity, such as polymer (15,16), quantum duts(17,18), mesoporous silica (19), were employed to modify electrodes surface . Carbon nano maerials become a superior modifier to improve the electroconductivity and stability of electrochemical biosensor.Carbon nano tubes



(CNTs) have got much attention (20)due to their properties such as excellent electroconductivity ,huge surface area and high electron transport rates make them base materials in the fabrication of electrochemical sensors.Many interests were concentrated on the detection of HQ and CC simultaneouly by CNTs – based electrode(21-24). Modification electrodes by polymer film have been chosen by researbers because of their good stability, reprducibility and more active sites. To immobilize polymers the electropolymerization, has an important rule because it is a one step polymerization , it can control film thickness ,permeation,charge transport characteristic by adjusting electrochemical parametrs(25,26).

In this work different modified electrodes were tested for simultaneous determination of HQ in the precense of CC, the best working electrode was GE/MWCNT/PFA modified electrode because it, s large surfase area and gives good peaks separation.

## **II. EXPERIMETAL**

## Apparatus

Differential pulse voltammetry (DPV) and Cyclic voltammetry(CV) measurments were performed using797VA Computrace voltammetric analyzer supplied by Metrohm company, Switzerland, coupled with a three electrode detection system consists of solid electrodes( bare Graphite,GE/ PFA. GE /MWCNTs,GE/MWCNTs/PFA as working electrodes, Ag/AgCl, sat. KCl as reference electrode and 1mm pt-wire was used as an auxliary electrode. measurments pН were performed using a digital pH meter supplied by HANNA Company, Portugal, model pH211 ,microprocessor pH meter with accurate to  $\pm 0.05$ .

## **Reagents and Chemicals**

Hydroquinone was purchased from British Drug House LTD, Catechol was purchased from Hopking and Williams British LTD . Freshly standard solution of HQ was prepared in absolute ethanol. Phosphate buffer solution(PBs) was prepared by mixing appropriate ammount of K2HPO4 and KH2PO4which is used as supporting electrolyte.

#### Procedure

Phosphate buffer solution was added to voltammetric cell and deoxygenated via passing N2 gas for 5 minutes prior the measurments, and the voltammogram was recorded. Then test solution was added to the voltammetric cell and the voltammograms were also recorded under the optimum conditions for a sequence additions of stock solutions of CC, after which the calibration curves were constructed.

## Fabrication of PFA Modified GE

Before modification ,the bare GE was polished with 0.05 µm of Al2O3 powder, rinsed and ultrasonicated in water (27).Then the Furfurylamine eletropolymerization of was performed in acetonitrile supporting electrolyte containing Furfurylamine  $(0.01 \text{ mol } L^{-1})$  with lithium perchlorate (0.06 mol L<sup>-1</sup>) and sodium hydroxide(0.06 mol  $L^{-1}$ ) (28)The electropolymerization performed using cyclic voltammetric technique by repetitive scanning the poential between (0.5-1.8) V versus Ag/AgCl, sat.KCl for 5cycles with scan rate equal to 0.14 V/s ,then the electrode was ready to use after the final wash in water to remove unwanted any physically absorbed materials.

## Construction of MWCNTs / PFA Electrode

Before the modification,the GE was polished with 0.05  $\mu$ m of Al2O3 powder ,rinsed and ultrasonicated in water for 3 minutes.The freshly cleaned GE was electrochemically activated in a 0.1  $\mu$ mol L<sup>-1</sup> H2SO4 solution by repetitive scanning the potential ,20 cycles between (-0.5 – 2.0)V. A 20  $\mu$ L of the freshly prepared (5mg/5ml)dispersion of MWCNTs in DMF was dropped onto the GE surface and the solvent was evaporated with IR lamp(29) .Then MWCNTs – modified electrode was further modified with a



thin film by placing  $10\mu L$  of PFA solution over the surface of the electrode and evaporating the solvent in air .

## **III. RESULTS AND DISCUSSION**

## 1- Electrochemical Behaviour of HQ at GE Using Differential Pulse Voltammetric Technique

Differential pulse voltammogram on GE was recorded for 14.778×10-6 M of HQ in phosphate buffer (pH= 8)under the default conditions of instrument. HQ gives a well defined oxidation peak current at -0.15 V versus Ag/AgCl.sat.KCl ( Fig.1a).Optimum Conditions

In order to optimize the conditions of various instrumental and measurments , experimental variables were examined and optimized using 14.778×10<sup>-6</sup>M of HO in phosphate buffer a (pH=8), the results obtained are shown in (Table 1) and the voltammogram after optimum conditions was recorded (Fig. 1b).

Table(1):The optimum conditions for HQ in phosphate buffer solution (pH=8) at GE

Parameters	Optium Coditions Values
Deposition potential	-1.7V
Deposition time	20 s
Equilibration time	30 s
Pulse Amplitude	0.11V
Pulse time	0.03 s
Voltage step	0.009V
Voltage step time	0.1 s









## Stability

To study the stability of HQ oxidation peak, a voltammogam of  $14.7 \times 10^{-6}$  M was recorded versus time under the mentioned optimum conditions(Tabe 1) ,the results obtained are shown in (Table 2) ,its clear that the oxidation peak current is stable within the studied time (Fig.2).

Table 2:Stability of HQ oxidation peak

Time(min)	Ip(µA)
0	5.77s
5	5.98
10	6.09
15	6.11
20	6.17
25	6.17
30	6.2
35	6.24
40	6.23
45	6.19
50	6.21
55	6.21
60	6.21
S.D=±0.131173	



Fig:2 D.P.Voltammogram for HQ oxidation peak stability at GE



## **Calibraion Curve:**

The calibration curve was constructed by adding a sequence additions of standard HQ solution  $(10^{-3}M)$  and the voltammogram was recorded for each addition under the previous optimum conditions , the plot of peak current versus concentrations give a two straight line, first at low concentration range(2.991×10<sup>-6</sup>- 17.681×10<sup>-6</sup>)M,(Fig.3), and the second at higher concentration range (19.607×10<sup>-6</sup> - 65.42×10<sup>-6</sup>) M (Fig.4), with R<sup>2</sup> values equal 0.9909 and 0.9719 respectively .



Fig:3 Calibraion curve of HQ at GE/PFA at low concentration



Fig:4 Calibration curve of HQ at GE/PFA at high concentration

**Hydroquinone and Catechol in mixture** The voltammogram in Fig.5shows that we could obtained weak separation between HQ and CC in mixture on GE at cocentration value equal to  $7.936 \times 10^{-6}$  M,whereas at lower concentration we got one composite peak represent the overall concentrations of HQ and CC .So we tried to develope the GE surface with PFA .



Fig: 5 Show the separation between HQ and CC in mixture at GE

## 2- Electrochemical Behaviour of Hydroquinone at GE /PFA Modified Electrode:

Differential pulse voltammogram of HQ on GE /PFA modified electrode was recorded for  $14.778 \times 10^{-6}$  M of HQ in phosphate buffer (pH=7) under the default conditions of instrument , HQ exhibit well-defined oxidation peak at (-0.084)V versus Ag/AgCl sat.KCl with oxidation current equal to 2.96 Aµ (Fig.6-a).

#### **Optimum Conditions**

order to optimize the conditions In of various. instrumental measurments and experimental variables were tested and optimized using  $14.778 \times 10^{-6}$  M of HQ in phosphat buffer (pH7), the results obtained are shown in (Table 3) and the voltammogram after optimization can be seen in(Fig. 6-b).

Table (3):The optimum conditions for HQ in phosphate buffer solution (pH=7) at GE /PFA modified electrode

Parameters	Optimum ConditionValues
Deposition potential	-1.7V
Deposition time	20 s
Equilibration time	20 s
Pulse Amplitude	0.09V
Pulse time	0.02 s
Voltage step	0.007V
Voltage step time	0.1 s







b

Fig:6 D.P.voltammogram of HQ on GE/PFA, abefore and b-after optimum conditions

## Stability

To study the stability of HQ oxidation peak voltammogam of  $14.7 \times 10^{-6}$  M HQ was recorded in different intervals time under the mentioned optimum conditions (Table 3) ,the results obtained are shown in (Table 4) ,its clear that the oxidation peak current is stable within the studied time.

Table 4:Stability of HQ	oxidation	peak
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Time(min)	Ip(µA)
0	11.6
5	12.2
10	12.5
15	12.7
20	12.8
25	12.6
30	12.6
35	12.8
40	13.0
45	12.8
50	12.7
55	12.7
60	12.4
S.D= 0.354459	

## **Calibraion Curve**

The calibration curve was constructed by adding a sequence additions of standard HQ solution(10<sup>-3</sup> M) and the voltammogram was recorded for each addition under the previous optimum conditions ,the plot of peak current versus concentrations (Fig.7) give a two straight lines ,one at lowe concentrations (1.497×  $10^{-6}$  - 9.9×  $10^{-6}$ ) M with R<sup>2</sup>= 0.9792(Fig.7a) and second at high concentrations (10.88×  $10^{-6}$  -56.603×  $10^{-6}$ ) M with R<sup>2</sup>= 0.9895 (Fig.7b).



Fig: 7a Calibraion curve of HQ at GE/PFA at low concentrations



Fig: 7b Calibraion curve of HQ at GE/PFA at high concentrations

# Calibration Curve of HQ in the Present of the CC at GE /PFA

A calibation curve of HQ in the present of CC was constructed by adding a sequence additions of HQ to the voltammetric cell containing  $(99.0\times10^{-7})$ M of CC using phosphate buffer (PH=7) .A plot of peak current versus concentrations give a two straight lines ,first at low concentration range( $4.479\times10^{-6}$ - $10.39\times10^{-6}$ ) M ,(Fig. 8a),and second at higher concentration range( $10.881\times10^{-6}$ - $19.607\times10^{-6}$ ) M(Fig.8b) with R<sup>2</sup> values equal to 0.9697 and 0.9799 respectively. The voltammgram (Fig.9) shows a small separation between two peaks at( $4.479\times10^{-6}$  M )of HQ.





Fig:8a Calibation curve of HQ in HQ and CC mixiture at GE/PFA at low concentrations



Fig:8b Calibation curve of HQ in HQ and CC mixiture at GE/PFA at high concentrations



Fig:9 The voltammogram of HQ in HQ and CC mixture at GE/PFA modified electrode

#### 5- Electrochemical Behaviour of Hydroquinone on GE /MWCNTs Modified Electrode

Differential pulse voltammogram of HQ was recorded for  $14.778 \times 10^{-6}$ M of HQ in phosphate buffer pH= 8 under the default conditions of instrument HQ exhibit oxidation peak at -0.215 V versus with oxidation peak current equal to 26.2 Aµ(Fig .10-a).

## **IV.** Optimum conditions

In oreder to optimize the conditions of various. measurments instrumental and experimental variables were eximined and 14.778 ×10<sup>-6</sup> M of HO optimized using in phosphate buffer a (pH= 8), the results obtained are shown in Table (5) and the voltammogram after the optimum condition can be seen in (Fig. 10-b).

Table (5): Optimum conditions for HQ in phosphate buffer solution (pH=8) at GE /MWCNTs modified electrode.

Parameters	<b>Optimum Condition</b>
	Values
Deposition potential	-0.2V
Deposition time	5 s
Equilibration time	4 s
Pulse Amplitude	0.06V
Pulse time	0.05 s
Voltage step	0.001V
Voltage step time	0.2 s



Fig:10 D.P.voltammogram of HQ on GE /MWCNTs modified electrode : a-before and b-after optimum conditions

## Stability

To study the stability of HQ oxidation peak , a voltammogam of  $14.7 \times 10^{-6}$  M of HQ was recorded at different intervals time under the mentioned optimum conditions (Table 5) ,its clear that oxidation peak current is stable within the studied time. the results obtained are shown in(Table 6).



Table 6: Stability of HQ oxidation peak :

Time(min)	Ιp(μΑ)
0	30.1
5	34.9
15	33.6
20	33.3
25	32.9
30	33.9
35	33.8
40	33.8
45	33.1
50	33.0
55	34.0
60	33.8
S.D=± 1.157976	

#### **Calibraion Curve**

The calibration curve was constructed by adding a sequence additions of standard HQ solution ( $10^{-4}$  M) and the voltammogram was recorded for each addition under the previous optimum conditions ,the plot of peak current versus concentrations give a two straight lines, one at lowe concentrations ( $9.99 \times 10^{-8} - 44.798 \times 10^{-8}$ ) M with R<sup>2</sup>= 0.9861(Fig.11a) and second at high concentrations ( $54.699 \times 10^{-8} - 99.009 \times 10^{-8}$ ) M with R<sup>2</sup>= 0.9873 (Fig.11b).



Fig:11-a Calibraion curve of HQ at GE/ MWCNTs at low concentrations



Fig:11b Calibraion curve of HQ at GE/ MWCNTs at high concentrations

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## Hydroquinone and Catechol in Mixture

HQ determined in the present of CC in phosphate buffer (pH=8) the voltammogram was recorded (Fig.12) and the calibraion curve was constructed (Fig.13) within the concentrations range  $(9.99 \times 10^{-1})$  $^{8} - 9.41 \times 10^{-7}$  )M ,the calibration shows two straight line ,first at low concentrations (4.997  $\times 10^{-8}$  -29.91 $\times 10^{-8}$  )M (Fig.13-a)with R<sup>2</sup> = 0.992 higher the second at concentrations and  $\mathbf{R}^2$  $(34.84 \times 10^{-8} - 94.1 \times 10^{-8})$ M with =0.9542(Fig.13-b) .



Fig.:12 The voltammogram of HQ in the pesent of CC at GE/MWCNTs modified electrode



Fig.13-a Calibation curve of HQ in CC and HQ mixiture at GE/MWCNTs modified electrode at low concentrations



Fig:13-b Calibation curve of HQ in CC and HQ mixiture at GE/MWCNTs modified electrode at high concentrations

## 6- Electrochemical Behaviour of Hydroquinone at GE /MWCNTs/PFA Modified Electrode

Differential pulse voltammogram of HQ at GE/MCNTs /PFA modified electrode was



recorded for  $14.779 \times 10^{-6}$  M of HQ in phosphate buffer (pH= 7) under the default instrument conditions, HQ exhibit oxidation peak at (-0.104) V and peak current equal to 151  $\mu$ A.

## **Optimum Conditions**

In oreder to optimize the conditions of measurments, various instrumental and variables eximined experimental were and 14.778 ×10<sup>-6</sup> M of HQ in optimized using phosphate buffer a (pH=7), the results obtained are shown in Table (7) and the voltammogram can be seen in Fig. (14).

Table (7):The optimum conditions for HQ at GE /MWCNTs/PFA modified electrode

Parameters	Optimum Condition Values
Deposition potential	-1.1V
Deposition time	40 s
Equilibration time	30 s
Pulse Amplitude	0.07V
Pulse time	0.02 s
Voltage step	0.002V



Fig.14 D.P.voltammogram of HQ on GE /MWCNTs/PFA ,a-before and b-after optimum conditions

## Stability

To study the stability of the oxidation peak of HQ, a voltammogram of HQ was recorded under the measured optimum conditions (Table 7) versus time ,the results obtained (Table 8) indicates that the oxidation peak is stable within the studied time .

Table 8: Stability of oxidation peak current of HQ at GE/MWCNTs /PFA modified electrode

Time(min)	Ip(µA)
0	84.6
5	81.2
15	84.6
20	82.4
25	83.7
30	79.7
35	80.1
40	81.6
45	75.4
50	79.7
55	80.0
60	82.0
S.D=±2.564	

#### Calibraion curve

The calibration curve was constructed by adding a sequence additions of standard HQ solution ( $10^{-5}$  M) and the voltammogram was recorded for each addition under the previous optimum conditions, the plot of peak current versus concentrations give a two straight lines ,one at lowe concentrations ( $14.977 \times 10^{-9} - 74.441 \times 10^{-9}$ ) M with R<sup>2</sup>= 0.9846(Fig.14a) and second at high concentrations ( $79.365 \times 10^{-9} - 128.33 \times 10^{-9}$ ) M with R<sup>2</sup>= 0.9747 (Fig.14b).



Fig.15a Calibraion curve of HQ at GE/ MWCNTs /PFA at low concentrations





Fig.15b Calibration curve of HQ at GE/ MWCNTs /PFA at high concentrations

## 4 Hydroquinone and Catechol in Mixture

HQ was determined in the present of CC in phosphate buffer (PH=8) on GE/MWCNTs/PFA modified electrode in the concentration range  $(14.977 \times 10^{-9} - 69.513 \times 10^{-9})$ M as shown in Fig.16, the voltammogram was recorded (Fig.17). The results obtained shows an excellent separation of HQ and CC oxidation peaks at low conentration which can not acheived with bare GE.

Table 9: HQ determination in the present of (99.0  $\times 10^{-7}$ ) M CC at GE/MWCNTs/PFA modifid electrode

Conc.× 10 <sup>-9</sup>	Ip correct (µA)
14.977	3.41
19.96	4.41
29.911	8.41
34.877	9.41
39.84	12.41
49.751	14.41
59.642	15.41
64.58	16.41
69.513	18.41



Fig:16 Calibation curve of HQ in the present of  $(99.0 \times 10^{-7})$  M CC at GE/MWCNTs/PFA modified electrode



Fig:17 The voltammogram of HQ in the present of  $(99.0 \times 10^{-7})$  M CC at GE/MWCNTs/PFA modified electrode

#### V. CONCLISION

The modification of electrode by MWCNTs and PFA conducting polymer improve the response of electrode towards the enhancement of peaks separation of materials has very close oxidationreduction potentials.

The resuls obtained shows an excellent separation of HQ and CC oxidation peaks at lower concentration can acheived with GE/MWCNTs/PFA modified electrode .

The proposed electrode exhibits good stability ,sensitivity and reproducibility towrds the determination of HQ by differntial pulse voltammetry.

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