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Amperometric Detection of Hydroquinone at Modified Ionic Liquid Graphene Composite Paste Electrode

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The ionic liquid 1-ethyl-3-methylimidazolium bromide (EMIMBr) based graphene composite paste electrode (GrPE) was fabricated and applied for the determination of hydroquinone. By using square wave voltammetry, the electrochemical response of hydroquinone on EMIMBr-GrPE was greatly enhanced in 0.7 M phosphate buffer solution (pH 7.0). Under optimized conditions, the proposed electrode showed a linear relationship with concentration in the range of 1.0×10^{-6} to 1.0×10^{-2} M hydroquinone with limit of detection of 2.77×10^{-7} M. The interferences of Ba^{2+}, Cu^{2+}, Na^{+}, K^{+}, NO^{3-}, Cl^{-}, SO_4^{2-}, glycine, glucose, fructose and sucrose were negligible. Electrochemical impedance spectroscopy indicated that the charge transfer at the electrode-solution interface was excellent. The proposed electrode was successfully applied to determine of hydroquinone in wastewater and cosmetic creams with range of recovery 97 – 104%.

Keywords: Graphene paste electrode, Hydroquinone, Ionic liquid, Square wave voltammetry

1. INTRODUCTION

Hydroquinone is the polyphenolic organic compound which is widely used in pharmaceutical, photography, rubber antioxidant, dyes and cosmetic industries [1,2]. Hydroquinone is also the compound with high toxicity and low degradability in the ecological environment. Hence, the European Union (EU) and Environmental Protection Agency (EPA) were banned hydroquinone from the European cosmetic market [3,4]. Many instrument can be used to determine hydroquinone and its isomer such as high performance liquid chromatography [5], spectroscopy [6], chemiluminescence [7], pH based-flow injection analysis [8] and electrochemical methods [9,10] have been used for the detection of hydroquinone and its isomers. Electrochemical methods have attracted enormous attentions by virtue of high accuracy, fast response and bulk modification.

About 50 years ago, the carbon paste electrode (CPE) was firstly discovered by Ralph Norman Adams. It became one of the most commonly used electrodes due to its simple preparation, low cost, easy renewability and good sensitivity [11-13]. However, the absence of conducting binder in the CPE will lower the sensitivity of the detection system. Thus, numerous carbon nanomaterials were utilized with a good surface area and high conductivity such as graphene. Graphene is the great materials with great chemical properties such as good thermal conductivity, electrical conductivity, mechanical strength and high surface area.

In recent years, many types of graphene based sensors have been fabricated to identify its inherent properties. Liu et. al [14] reported graphene nanocomposite modified electrode for the determination of hydroquinone and catechol in the presence of ionic liquid. Hadi [15] was fabricated graphene paste electrode (GrPE) for the detection of chlorpromazine. While Li et. Al [16] developed CPE based on graphite and then introduced the various amount of graphene into the CPE for sensitive determination of ascorbic acid.

In last decade, ionic liquids (ILs) have been used to be an efficient binders/modifiers in the electrode fabrication. ILs is entirely composed of organic cation and anion which have greatly unique properties such as good solubility and viscosity, wide electrochemical window, good ionic conductivity and high chemical stability [17-22]. In order to enhance the sensitivity of electrochemical sensors, the applications of ionic liquids for electrode modification has become a trend. Lately the collaboration between ionic liquids and graphene has brought a novel dimension to the electrochemical sensors field.

In this work, GrPE based on an ionic liquid, 1-ethyl-3-methylimidazolium bromide, EMIMBr (EMIMBr-GrPE) was fabricated for determination of hydroquinone was investigated.

2. EXPERIMENTAL

2.1. Reagents and chemicals.

Graphene with the 99.9% purification was synthesized according to previous method. The ionic liquid, 98% EMIMBr was purchased from Acros Organics (New Jersey, USA). All analytical grade chemicals used in this experiment; barium chloride, copper sulphate, sodium hydroxide, potassium iodide, glycine, glucose, fructose and sucrose were obtained from Merck (Germany). Phosphate buffer solution (PBS) and another supporting electrolyte (potassium nitrate, potassium chloride, sodium acetate, sodium sulphate, sodium chloride and sodium nitrate) were also supplied by Merck. The pH of solutions was adjusted to the desired value by drop wise addition of sodium hydroxide and hydrochloric acid. All solutions were prepared using distilled deionised water from Barnstead EasyPure LF (USA). Stock solutions of hydroquinone were freshly prepared by adding distilled deionized water in appropriate amount of hydroquinone by dissolving it. The graphene was synthesized and purified as previously reported [23].

2.2. Apparatus

Square wave voltammetry (SWV) and electrochemical impedance spectroscopy (EIS) were performed with a Gamry Potentiostat Series-G750, USA. All electrochemical data were obtained by using three-electrode system consisting of an Ag/AgCl electrode MF-2052 (Bioanalytical system, USA) as the reference electrode, a platinum foil as the auxiliary electrode and carbon paste electrode (unmodified and modified) was used as the working electrode. All pH values were measured using Orion 720A plus meter of Thermo Electron Corp. (USA) with glass electrode 915600 (Orion, USA). Prior to any measurement, all the solutions were deoxygenated by bubbling oxygen free nitrogen (Nissan Oxygen, Malaysia) for about 15 minutes. The surface morphological of modifier was characterized using Hitachi S-3000 H feild emission scanning electron microscope (FESEM, Japan).

2.3. Preparation of electrode

The modified graphene composite paste electrode was prepared by hand mixing 1.5 g graphene and 0.5 g EMIMBr in a mortar and pestle until form a homogeneous paste. Then, 2 drops of paraffin oil were added to the mixture and mixed until a uniform paste. The unmodified graphene paste electrode was fabricated using the same way but without the presence of ionic liquid, EMIMBr for comparison purposes. The modified graphene (EMIMBr-GrPE) and unmodified graphene paste (GrPE) were finally packed into Teflon tubes (i.d. 2.0 mm). One end of the tubing inserted with copper wires to establish electrical contact while the other end behaved as a disc electrode which once smoothened with filter paper.

2.4. Measurement procedure

Voltammetry measurements were performed on 0.7 M PBS as supporting electrolyte containing hydroquinone at a desired concentration and pH 7.0. The solutions were deoxygenated for 15 minutes before every measurement by bubbling with nitrogen gas. The solutions were adjusted to desire pH 7.0 by using hydrochloric acid and sodium hydroxide. The voltammogram was recorded between -0.4 V to 0.4 V by applying SWV with frequency of 80 Hz, step increment of 3 mV and pulse height 30 mV. Prior to the measurement, the electrode surface must be polished by smoothened on a filter paper. All the experiments were handled at room temperature (25 ± 2˚C) and in air atmosphere.

3. RESULTS AND DISCUSSION

3.1. FESEM characterization of graphene and EMIMBr-Gr

From the previous report, the surface morphology of graphene paste was examined which has typical thin wrinking, crumpled paper-like structure closely associated with each other and form a disordered solid [23]. Figure 1 shows the imaging of EMIMBr-GrPE surface which has a smooth surface, uniform topography and no separated layers was found which attributed the good adherence of EMIMBr to graphene due to the high viscosity of ionic liquid. The ionic liquid was filled into the void space of graphene paste.

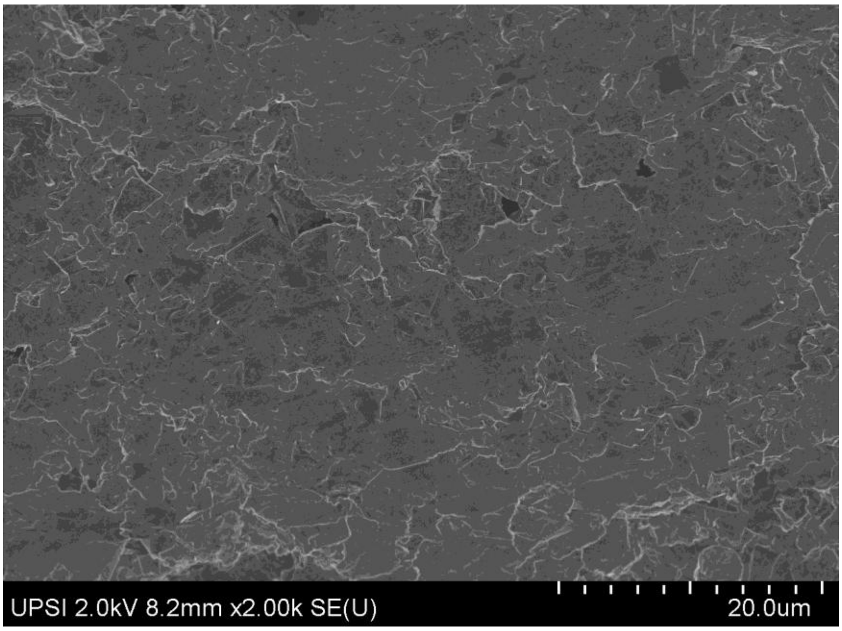


Figure 1. The FESEM micrograph of EMIMBr-GrPE with 2000X magnification.

3.2. Voltammetric response of the unmodified and modified EMIMBr-GrPE

Figure 2 shows the voltammograms of the unmodified and modified EMIMBr-GrPE were in 0.7 M PBS and pH 7.0 towards hydroquinone. The high reduction peaks was observed at unmodified GrPE, which might be ascribed to the absence of ionic liquid. It is indicated that the GrPE was stimulated the electron transfer rate. It is because graphene, a 2-dimensional sp^2-hybridized bonds owing extraordinary features such as high electrical and thermal conductivities. The collaboration between graphene and EMIMBr might contribute to the synergistic effect. The intrinsic conductivity of EMIMBr and the high conductivity and specific surface area of graphene on the electrode surfaces enhance the electrocatalytic activity toward the oxidation of hydroquinone. The whole possible reaction mechanism is illustrated in Scheme 1.

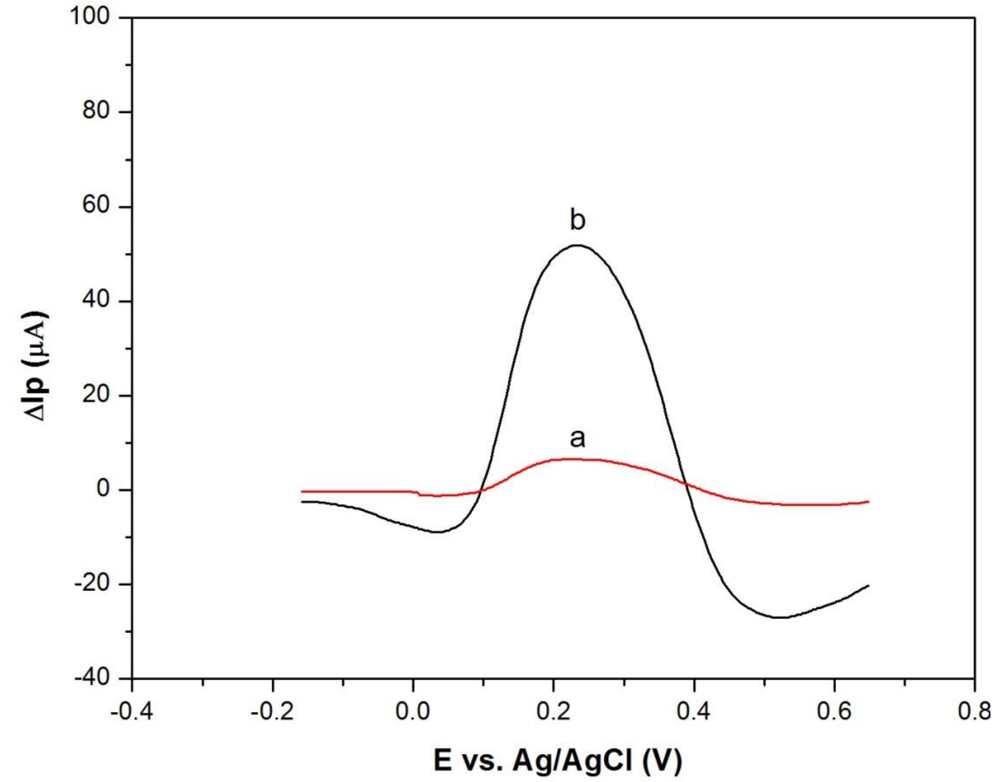
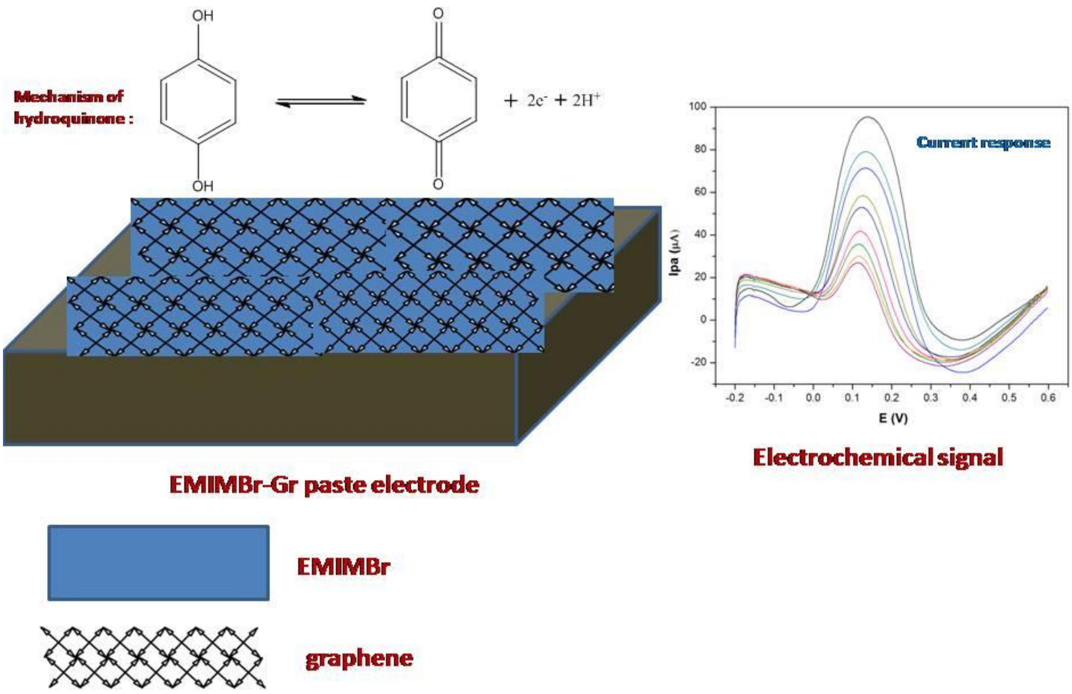


Figure 2. SWV of (a) unmodified GrPE and (b) modified EMIMBr-GrPE towards 1.0 x 10^{-3} M hydroquinone in 0.7 M PBS at pH 7.0.



Scheme 1. Proposed mechanism of hydroquinone detection at the EMIMBr-GrPE electrode.

3.3. Optimization of the experimental conditions

3.3.1. The effect of solution pH

Figure 3 show the effect of pH over the range 4.0-8.0 and it was found that the peak potential shifted negatively when pH of PBS solution increased indicating that proton is involved in the redox reaction of hydroquinone. It can be see that the peak current increased gradually until it reached the maximum I_{pa} at pH 7.0 then it decreased slightly. Therefore, pH 7.0 was chosen as the optimum pH value in the electrochemical detection of hydroquinone.

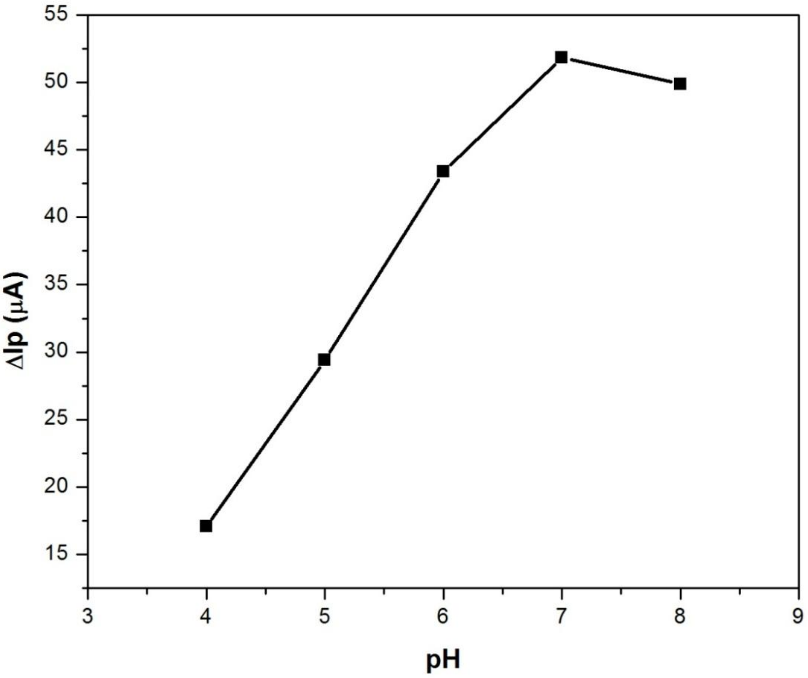


Figure 3. Plot of I_{pa} vs. pH of 1 x 10^{-3} M of hydroquinone, 0.7 M PBS.

3.3.2 Types of supporting electrolyte

The voltammetric measurement results showed that different properties owned by different supporting electrolyte will affect the square-wave voltammetric peak height at the modified electrode. The several types of supporting electrolyte such as KNO_3, KCl, PBS, CH_3COONa, Na_2SO_4 and NaCl were investigated in order to examine their effect on the voltammetric behavior of the modified EMIMBr-GrPE. Table 1 showed value of I_{pa} of different electrolytes on the determination of hydroquinone and the well defined reduction peak with higher I_{pa} were obtained in 0.7 M PBS. Hence, 0.7 M PBS was chosen as suitable supporting electrolyte.

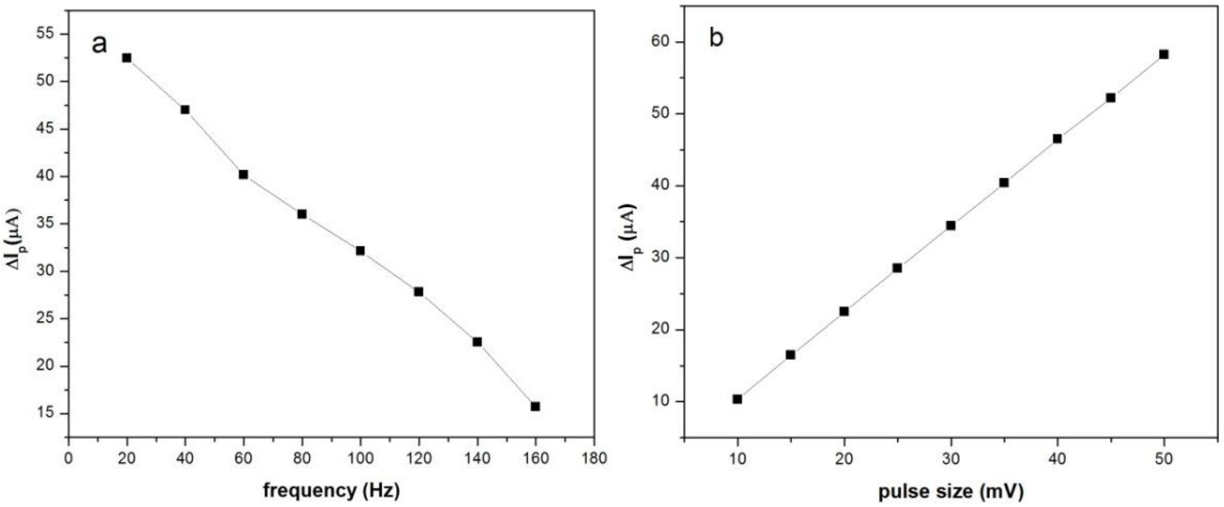
Table 1. Value of I_{pa} of different electrolytes on the determination of hydroquinone.

Types of electrolytes	Peak current, I_{pa} (μA)
PBS	28.76
KCl	10.80
Na_2SO_4	6.03
CH_3COONa	4.25
NaCl	3.42
KNO_3	0.00

3.3.3. Square wave parameter

The square wave parameter were investigated to find the suitable frequency, the pulse height and the step size in a solution containing hydroquinone in order to get a peak current at high scan speed with better sensitivity and reduced background noise when using square wave voltammetry which is strongly affected the outcome signal [24].

Based on theoretical calculations made by Osteryoung, the voltammogram characteristics is linearly dependent on some parameter used. The frequency is linearly decreased from 20 to 180 Hz. As the frequency increase, the peak current of the hydroquinone also gradually decreased as shown in Figure 4a. Thus, 80 Hz was chosen as an optimum frequency as it has improved the sensitivity without distortion of the peak or the baseline. The effect of pulse size parameter was measured in the range of 10 to 50 mV at a fixed frequency, 80 Hz. The I_{pa} of hydroquinone increased with increasing pulse size and at the same time, the peak current was shifted to the positive direction (Figure 4b). The pulse size of 30 mV was chosen as the best pulse size as it generated a very good resolution. The step size was varied from 1 mV to 10 mV. The peak current showed the linear decrease in relationship (Figure 4c). As the step size increase, the current decrease, hence, 3 mV was chosen as the optimum step size with a sharper peak. Therefore, the optimum SWV conditions for the determination of hydroquinone were at frequency 80 Hz, pulse size 30 mV and step size of 3 mV.



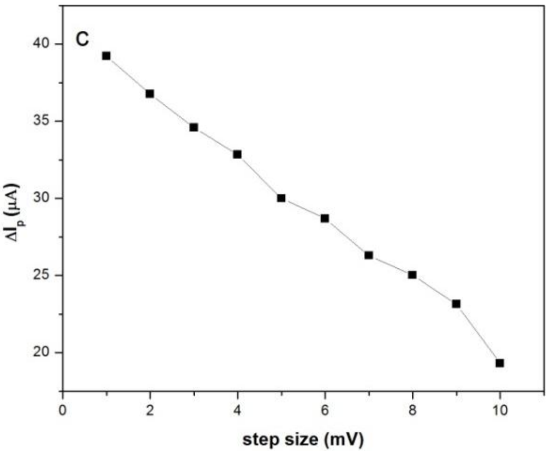


Figure 4. Influence of the square wave parameter (a) frequency (b) pulse size and (c) step size on SWV signal of 1 x 10^{-3} M hydroquinone, pH 7.0.

3.4. Calibration data

Figure 5 shows the peak currents of modified EMIMBr-GrPE towards the different concentrations of hydroquinone by using SWV. The peak current versus hydroquinone concentration is linear for 1×10^{-6} mol L^{-1} to 1×10^{-2} mol L^{-1} with correlation coefficients, R^2, 0.99616. The limit of detection (LOD) of hydroquinone is 2.77×10^{-7} mol L^{-1} calculated as 3σ/S, where σ is a standard deviation of the response and S is the slope of calibration curve which is lower than that at most of modified electrodes reported below. From the previous reports of some of relevant electrodes, it can be compared that the LOD of the proposed method is comparable than others. Table 2 shows the results so obtained compared favorably if not better than many other reported hydroquinone detector.

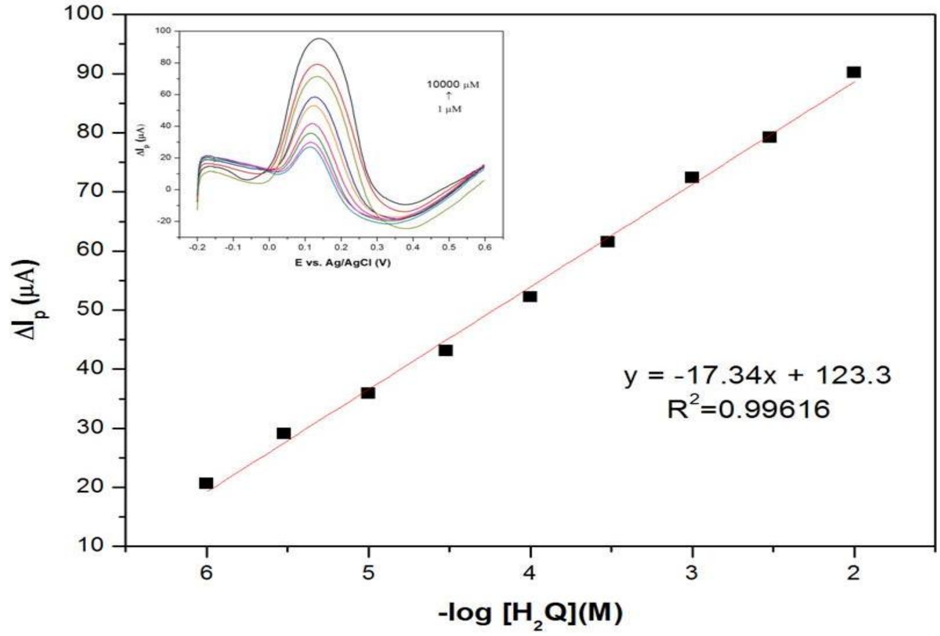


Figure 5. The SWV of different concentrations of hydroquinone.

Table 2. Parameters of modified electrodes for determination of hydroquinone.

Modifier	Electrode	Method	Calibration curve	Limit of detection	Ref.
N-butylpyridinium hexafluorophosphate ($BPPF_6$)	MCPE[a]	CV[b]	5.0×10^{-6} – 5.0×10^{-3}	2.5×10^{-6}	[26]
1-butyl-3-methylimidazolium hexafluorophosphate ($BMIMPF_6$)	MWNTs[c]	DPV[d]	2.0×10^{-7} – 3.5×10^{-5}	6.7×10^{-8}	[27]
1-octyl-3-methylimidazolium hexafluorophosphate	MCPE	CV	1.0×10^{-5} – 1.0×10^{-2}	8.1×10^{-7}	[28]
Poly-amidosulsulfonic acid and Multi-wall carbon nanotubes	MGCE[e]	DPV	6.0×10^{-6} – 4.0×10^{-4}	1.0×10^{-6}	[29]
Graphene-chitosan composite film	MGCE	DPV	1.0×10^{-6} – 3.0×10^{-4}	7.5×10^{-7}	[30]
Poly (3,4-ethylenedioxythiophene)	MCNTPE[f]	CV	1.1×10^{-6} – 1.25×10^{-4}	3.0×10^{-7}	[31]
Cobalt hydroxide film	MGCE	CV	5.0×10^{-6} – 1.25×10^{-4}	5.0×10^{-7}	[32]
Multi-wall carbon nanotubes	MGCE	DPV	1×10^{-6} – 1×10^{-4}	7.5×10^{-7}	[33]
Zucchini crude extract-palladium	MCPE	CV	6.2×10^{-5} – 8.9×10^{-3}	8.3×10^{-6}	[34]
Zn/Al layered double hydroxide film	MGCE	DPV	1.2×10^{-5} – 8.0×10^{-4}	9.0×10^{-6}	[35]
Nano-gold	MGCE	CV	4.0×10^{-6} – 3.2×10^{-3}	1.02×10^{-6}	[36]
L-cysteine	AuPE[g]	DPV	2.0×10^{-6} – 2.0×10^{-4}	4.0×10^{-7}	[37]
Aspartic acid	MGCE	DPV	5.0×10^{-6} – 6.0×10^{-3}	9.0×10^{-7}	[38]
1-ethyl-3-methylimidazolium bromide (EMIMBr)	MGrPE[h]	SWV	1.0×10^{-6} – 1.0×10^{-2}	2.77×10^{-7}	This work

[a] Modified carbon paste electrode
[b] Cyclic voltammetry
[c] Multi-wall nanotubes
[d] Differential pulse voltammetry
[e] Modified glassy carbon electrode
[f] Modified carbon nanotube paste electrode
[g] Gold electrode
[h] Modified graphene paste electrode

3.5. Reproducibility and stability

The reproducibility and stability of EMIMBr-GrPE were carried out in 0.7 M PBS (pH 7.0). The relative standard deviation (RSD) for ten measurements with the same electrode was 4.82% and the RSD for ten different modified electrodes with the same test solution was 4.42%. The proposed sensor had only a slightly decrease in its oxidation peak current after 14 days storage. This indicated that the proposed electrode had good reproducibility and storage stability.

3.6. Electrochemical impedance spectroscopy

EIS is a popular technique to investigate the capability of electron transfer of the electrode [39]. It is vital in EIS study to compare the electron-transfer resistance among the electrodes for comparison purpose. The size of the semicircles represent the electron-transfer resistance, R_{ct} while the straight-line behaviors indicated the diffusion process [40,41].

Figure 6 showed the Nyquist plots of the modified EMIMBr-GrPE and unmodified GrPE in the 0.7 M PBS (pH = 7.0) containing 1×10^{-3} M hydroquinone which is the diagrams of the imaginary part of the impedance (Z_{im}) and the real part of impedance (Z_{re}) of EIS. As its shown in Figure 6, both electrodes showed almost a straight lines, revealing that graphene attributed the ability good electron transfer. The EIS measurements were operated at open circuit with the frequencies swept applied is 10^5 Hz to 1 Hz and an amplitude of 5 mV. The semicircle diameter of Nyquist plot reflects the electron transfer resistance (Rct). Data comparison shown that charge transfer resistance decreased when GrPE mixed with ionic liquid, EMIMBr. The same results were reported by other researchers [42].

By fitting the Randles equivalent electrical circuits (Figure 6 inset) the R_{ct} values for unmodified GrPE was 93.95 ohms (A). After the addition of ionic liquid, EMIMBr into graphene paste to get EMIMBr-GrPE, the value of Rct was decreased to 0.142 ohms (B) that was much more smaller than the unmodified GrPE. It was due to the presence of high conductive ionic liquid in the GrPE. The electron transfer apparent rate constant, $k_{app,}$ for 1.0×10^{-3} M hydroquinone on the unmodified GrPE and modified EMIMBr-GrPE was calculated by using this equation:

$$k_{app} = RT/F^2 R_{ct} C \qquad (1)$$

Where R, T and F represent the gas constant, temperature, Faraday constant concentration of the hydroquinone. The apparent rate constant k_{app} for unmodified GrPE and modified EMIMBr-GrPE are 2.84×10^{-6} cm s^{-1} and 1.87×10^{-3} cm s^{-1} respectively. The high value of k_{app} indicated fast electron transfer process and the low value of R_{ct} showed an improvement in the conductivity [43].

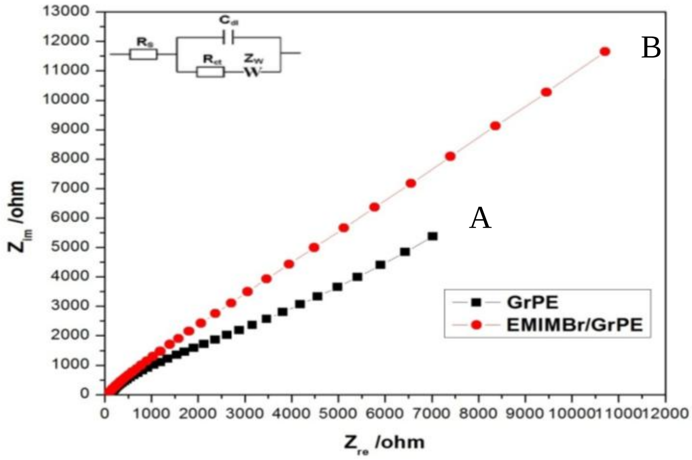


Figure 6. EIS of unmodified (A) and modified EMIMBr-GrPE (B) in 1.0×10^{-3} M hydroquinone dissolved in 0.7 M PBS (pH 7.0).

3.7. Chronocoulometry

In this work a double step chronocoulometry was carried out on 1.0×10^{-3} M hydroquinone and 0.7 M PBS at pH 7.0 with the initial potential range from -0.4V to 0.4V. The Anson plot of charge (Q) *vs.* square root of time ($t^{1/2}$) was linear (Figure 7) after several point-by-point background substraction. According to Cottrell equation, the diffusion coefficient can be estimated from the intercept while adsorption charge (Q_{ads}) of modified EMIMBr-GrPE is determined from the slope [44,45]. By using the equation,

$$Q_{ads} = nFA\Gamma \qquad (2)$$

where;

n = number of electron transferred

F = Faraday constant (96485 C/equiv)

A = area of the electrode

Γ = surface coverage of electrode (mol cm-2)

As n = 2, A = 0.0314 cm^2, D = 1.22×10^{-1} cm^2s^{-1} and the value of Q_{ads} is 0.002932 C, the surface coverage for modified EMIMBr-GrPE was 4.8388×10^{-7} $molcm^{-2}$ which is larger than the previous studied value of 2.09×10^{-11} $molcm^{-2}$ [46]. This indicated that modified electrode has a high surface coverage, hence, good sensitivity towards hydroquinone. The result of this experiment showed that there was an initial rapid increase in the charge during the oxidation of hydroquinone as shown in Figure 7.

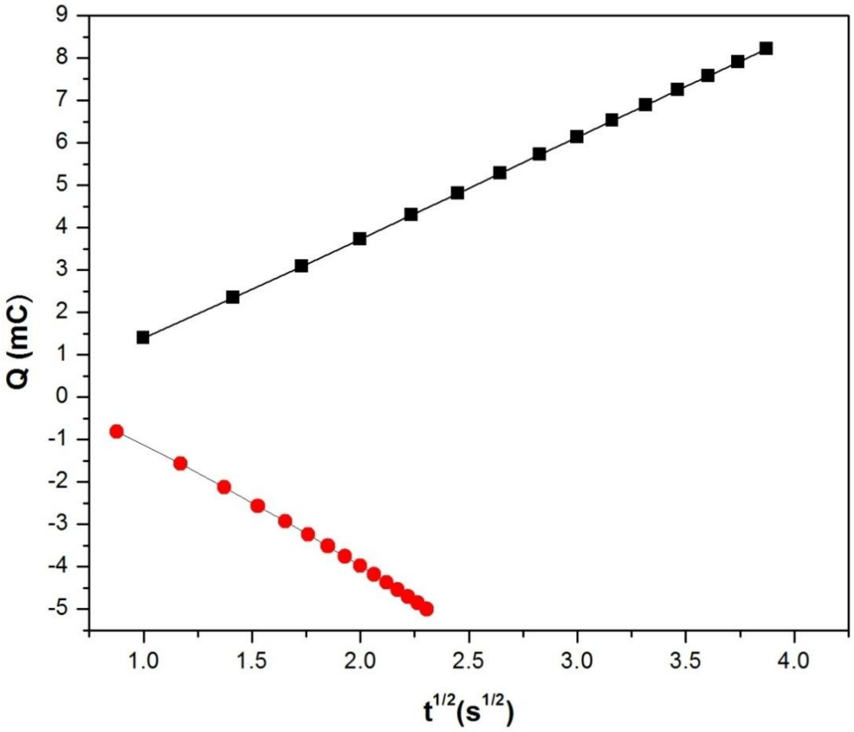


Figure 7. Double step chronocoulograms of 1 x 10^{-3} M hydroquinone at modified EMIMBr-GrPE in 0.7 M PBS (pH 7.0) at scanning potential of -0.4V – 0.4V.

3.8. Interferences studies

Under the selected conditions, the interferences of some common ions and other organic compounds were examined with 1.0×10^{-3} hydroquinone by using SWV. Results indicates that at 50-folds and 100-folds of Ba^{2+}, Cu^{2+}, Na^+, K^+, NO^{3-}, Cl^-, SO_4^{2-}, glycine, glucose, fructose and sucrose do not interfere with the determination of hydroquinone. Hence, a satisfactory selectivity of the proposed method was achieved.

3.9. Application of real sample

To assess the applications of the developed method for the detection of hydroquinone, several water samples and cosmetic creams were tested by standard addition method. The recoveries in the range of 97 – 104% (Table 3) indicate that the proposed sensor is efficient for the determination of trace amount of hydroquinone in water samples and cosmetic creams. The RSD value of below 5% (n = 3) also indicates that the proposed method is viable for determination of hydroquinone in practical samples.

Table 3. Determination of hydroquinone in different water samples and cosmetic creams.

Sample	Hydroquinone added (M)	Hydroquinone found (M)	Recovery (%)	RSD (%)
Tap water	-	0.0	-	-
	$3.00x10^{-4}$	$3.12x10^{-4}$	104	2.50
	$1.00x10^{-3}$	$1.02x10^{-3}$	101	4.54
Mineral Water	-	0.0	-	-
	$3.00x10^{-4}$	$3.06x10^{-4}$	102	2.27
	$1.00x10^{-3}$	$1.02x10^{-3}$	101	5.52
Lake Water	-	0.0	-	-
	$3.00x10^{-4}$	$3.11x10^{-4}$	103	2.92
	$1.00x10^{-3}$	$9.75x10^{-4}$	98	2.51
Cream A	-	0.0	-	-
	$3.00x10^{-4}$	$2.96x10^{-4}$	99	5.70
	$1.00x10^{-3}$	$1.02x10^{-3}$	102	2.03
Cream B	-	0.0	-	-
	$3.00x10^{-4}$	$2.93x10^{-4}$	98	2.06
	$1.00x10^{-3}$	$9.65x10^{-4}$	97	0.89

4. CONCLUSIONS

This study has demonstrated the modified graphene paste electrode using ionic liquid, 1-ethyl-3-methylimidazolium bromide, as a mediator was successful to be used in the determination of hydroquinone by square wave voltammetry. The proposed electrode was sensitive, selective, simple, low cost and applicable in the analysis of hydroquinone in different water samples and cosmetic creams.

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