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MWCNTs Based Carbon Paste and PVC Membrane Potentiometric Electrodes for Monitoring of Bupropion Hydrochloride

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Bupropion hydrochloride, active component of an antidepressant drug, was determined in the pharmaceutical formulation by two new bupropion potentiometric sensors. One was a PVC membrane sensor and the other one was a modified carbon paste electrode by MWCNTs and ionic liquid. Both sensors were made using bupropion-tetraphenylborate ion-pair complex and they responded based on ion exchange mechanism. The best sensor response, in case of PVC membrane electrode, was obtained by the membrane composition of 7% ion-pair, 30% PVC and 63% DBP and in case of modified carbon paste electrode by 25% ion-pair, 5% MWCNTs, 25% ionic liquid and 45% graphite powder. The PVC membrane sensor showed a Nernstian response in the linear range of 1.0×10^{-5}-1.0×10^{-2} mol L^{-1} and modified carbon paste electrode 5.0×10^{-6}-1.0×10^{-2} mol L^{-1}. The both sensors work well in the laboratory conditions in analysis of bupropion hydrochloride in tablets.

Keywords: Bupropion hydrochloride, Potentiometry, Ion-selective electrode, Carbon paste, Multiwalled carbon nanotubes, Ionic liquid, PVC membrane, Sensor

1. INTRODUCTION

Bupropion (1-(3-chlorophenyl)-2-[(1,1-dimethylethyl)amino]-1-propanone (BUP), Fig. 1, is an antidepressant drug of aminoketone class which chemically unrelated to other known antidepressant agents [1]. Its exact mechanism in smoking is reduction of dopamine reuptake in the mesolimbic system and noradrenalin reuptake in the locus coeruleus [2]. In humans, bupropion is metabolized to hydroxybupropion (HB), the pharmacologically active main metabolite, and to threo-hydrobupropion

(TB) and erythrohydrobupropion (EB) [3]. Studies indicate that the cytochrome P450 enzyme CYP2B6 is involved in the hydroxylation of bupropion.

The reports of the analysis of BUP in biological fluids, such as plasma or serum or urine, concern the use of high performance liquid chromatography (HPLC) with UV detector [3-5] or mass spectrometry [6,7]. The extraction of BUP and its derivatives from the matrices is carried out by liquid–liquid extraction (LLE) [3,4,6] or protein precipitation [5] or solid phase extraction (SPE) [7] procedures. In some reports, effect of BUP on extracellular dopamine and norepinephrine concentrations was studied by in vivo micro-dialysis [8-11].

However, in this work we decide to introduce new electrochemical sensors for determination of BUP in pharmaceutical formulations. These devices can be applied by themselves in pharmaceutical matrix or in combination with other separation methods for determination of this drug in complex matrixes. Like other potentiometric electrodes which have found wide applications in different analyses [13-24], they offer advantages of low cost of analysis, high precision and rapidity, wide concentrations range of measurement, portability, and simplicity.

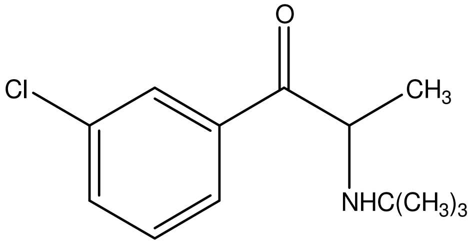

Figure 1. Chemical structure of bupropion (BUP)

Here, BUP hydrochloride and sodium tetraphenyl borate were used in synthesis of the ion-pair complex which acts as a sensing element in the both sensors. Both constructed sensors responded according to the ion-exchange mechanism.

2. EXPERIMENTAL PART

2.1. Reagents

All chemicals used in this study were of highest purity available and used without any further purification. Chemicals (of analytical reagent grade) were high-molecular weight polyvinylchloride (PVC) (Fluka Co., USA), sodium tetraphenyl borate (NaTPB), dibutyl phthalate (DBP), nitrobenzene (NB), benzyl acetate (BA), tetrahydrofuran (THF) and ionic liquid (1-n-butyl-3-methylimidazolium tetrafluoroborate ([bmim]BF4) (Merck Co., Germany). Bupropion hydrochloride and its pharmaceutical formulation were obtained from a local pharmaceutical manufacturer (Tehran, Iran) as gift samples. The multi-walled carbon nanotubes (MWCNTs) with 10-40 nm diameters, 1-25 μm length, core diameter: 5-10 nm, SBET: 40-600 m^2/g, V_{total}: 0.9 cm^3/g, bulk density 0.1 g/cm^3, true density 2.1 g/cm^3 and with 95% purity were purchased from a local company (Research Institute of the Petroleum Industry, Iran).

2.2. Ion-pair complex synthesis

Sensing element used in both sensors was an ion-pair complex made from the interaction of bupropion (BUP) hydrochloride and sodium tetraphenyl borate. It was synthesized by mixing about 20 mL of 0.01 mol L^{-1} BUP-HCl with equivalent value of tetraphenyl borate solution. The resulting precipitate was then filtered, washed with distilled water and dried in room temperature to use in construction of the sensors [23,24].

2.3. Devices

The glass cell in which the measurements were carried out, was contained of an Ag/AgCl electrode (Azar electrode, Iran) as a reference electrode and BUP sensor as an indicator electrode. Both electrodes were connected to a mili-voltmeter (±0.1).

The following cell was assembled for the conduction of EMF (electromotive force) measurements:

In case of PVC membrane electrode:

Ag/AgCl–KCl (satd.) || internal filling solution of BUP-HCl (1.0×10^{-3} mol L^{-1}) |
PVC membrane | BUP-HCl sample solution || Ag/AgCl–KCl (satd.)

In case of nano-composite electrode:

Modified carbon paste surface | BUP-HCl sample solution || Ag/AgCl–KCl (satd.)

These measurements were done using calibration of the electrodes with several standard solutions.

2.4. Preparation of the sensors

2.4.1. PVC membrane sensor

General procedure to prepare PVC membrane was as follow; different amounts of ion-pair along with appropriate amounts of PVC, plasticizer and additive were dissolved in tetrahydrofuran (THF) and the solution was mixed well. Then, THF was evaporated slowly until an oily concentrated solution was obtained. A plastic tube (about 3 mm o.d.) was dipped into the mixture for about 10 s so a transparent membrane of about 0.3 mm in thickness was formed. The tube was then pulled out from the mixture and kept at room temperature for about 5 h. Afterwards, the tube was filled with an internal filling solution (1.0×10^{-3} mol L^{-1} of BUP hydrochloride solution). The electrode was finally conditioned for 15 h by soaking in the same solution [13-16].

2.4.2. Nano-composite based carbon paste sensor

Two kinds of carbon paste sensor were prepared to study the effect of MWCNTs in the paste. The procedure for preparation of carbon paste electrode was as follows; various amounts of ion-pair along with appropriate amount of graphite powder, MWCNTs, ionic liquid, were thoroughly mixed. After homogenization of the mixture, the paste was moved into a plastic tube with 6 mm o.d. and a height of 3 cm. The paste was carefully packed into the tube tip to avoid possible air gaps, which often enhance the electrode resistant. A copper wire was inserted into the opposite end of the tube to make an electrical contact. External surface of the carbon paste was smoothed with soft paper. The electrode was finally conditioned for about 48 h by soaking it in a 1.0×10^{-3} mol L^{-1} of BUP hydrochloride solution [25-30].

2.5. Standard BUP-HCl solutions

A stock solution of 0.01 mol L^{-1} BUP hydrochloride was prepared. The working standard solutions (1×10^{-7} to 1×10^{-3} mol L^{-1}) were prepared by properly dilution of the stock solution with distilled water.

3. RESULTS AND DISCUSSION

Synthesized BUP-TPB ion-pair was applied as a sensing element in the composition of both PVC membrane and carbon paste electrodes. Ion-exchanging is the mechanism of the potential response. In both kinds of sensor the used composition with sensing material has an important effect on the sensor responses. Every constituent plays a particular function in the electrode response. Thus, the type and amount of the component of the membrane and the paste was modified and optimized. The results have been given in Table 1 and 2.

3.1. PVC Membrane Composition Selection

The main component of a membrane sensor is a polymeric matrix. According to the previous studies, PVC is a good choice for being used as a polymeric matrix. Previous studies showed that the membrane having a plasticizer/PVC ratio about 2.2 can show the best performance [31-34]. The optimum amount of PVC was selected 30 mg.

A plasticizer is a water-immiscible liquid with low vapor-pressure, compatible with PVC, no functional groups which can undergo protonation reactions. Selectivity of such electrode based on ion-exchange mechanism, can be drastically influenced by the type of the plasticizer used.

Plasticizer or solvent mediator allows a homogeneous dissolution and diffusional mobility of the ion-pair in the membrane phase [35-43]. Nature of the plasticizer affect on analytical responses such as slope, linear range and selectivity of PVC membrane electrodes. Three plasticizers with different polarity (dielectric constant) were tested, dibutyl phthalate (DBP with DC of 6.4), nitrobenzene (NB with DC of 35.7) and benzylacetate (BA with DC of about 5.7). The sensor responses showed that the membrane with DBP respond better. DBP among the used plasticizers provided an effective linear range and a lower detection limit due to the better extraction of bupropion hydrochloride ions in the organic phase of the membrane. As it can be seen from Table 1, lack of ion-pair in the membrane components causes a very poor response (membrane no. 7), which confirm significance of the ion-pair. The electrodes behavior show that the best Nernstian slope is 57.7±0.4 mV per decade. Finally, membrane no. 3 with the composition of 30% PVC, 7% ion-pair, and 63% DBP was selected as the optimum one for the sensor design.

Table 1. Optimization of PVC membrane components of the sensor

No.	Composition (%)			Slope (mV decade^{-1})	LR (mol L^{-1})	R^2	Response time
	PVC	Plasticizer	BUP-TPB				
1	30	DBP, 67	3	21.7±0.7	5.0×10^{-4}-5.0×10^{-3}	0.911	1.2 min
2	30	DBP, 65	5	43.4±0.5	5.0×10^{-5}-5.0×10^{-3}	0.942	53 s
3	30	DBP, 63	7	57.7±0.4	1.0×10^{-5}-1.0×10^{-2}	0.995	20 s
4	30	DBP, 61	9	56.8±0.5	1.0×10^{-5}-1.0×10^{-2}	0.954	25 s
5	30	NB, 63	7	18.8±0.5	5.0×10^{-5}-5.0×10^{-3}	0.882	1 min
6	30	BA, 63	7	24.7±0.5	1.0×10^{-4}-1.0×10^{-2}	0.900	58 s
7	30	DBP, 70	0	3.1±0.7	5.0×10^{-3}-1.0×10^{-3}	0.723	5 min

3.2. Carbon Paste Composition

Table 2. Optimization of nano-composite carbon paste sensor ingredients

No.	Composition (%)				Slope (mV decade^{-1})	LR (mol L^{-1})	Response time	R^2
	Graphite	Binder	BUP-TPB	MWCNTs				
1	80	20-Paraffin	-	-	-	-	-	-
2	65	20-Paraffin	15	-	22.1±0.8	5.0×10^{-4}-5.0×10^{-3}	1.6 min	0.812
3	60	20-Paraffin	20	-	36.4±0.6	1.0×10^{-5}-5.0×10^{-3}	55 s	0.947
4	55	20-Paraffin	25	-	45.2±0.5	1.0×10^{-5}-5.0×10^{-3}	34 s	0.919
5	50	20-Paraffin	30	-	42.1±0.6	5.0×10^{-5}-5.0×10^{-3}	42 s	0.984
6	55	20-IL	25	-	53.9±0.5	1.0×10^{-5}-1.0×10^{-2}	28 s	0.936
7	50	25-IL	25	-	55.8±0.4	1.0×10^{-5}-1.0×10^{-2}	25 s	0.957
8	45	30-IL	25	-	54.9±0.5	1.0×10^{-5}-1.0×10^{-2}	33 s	0.951
9	47	25-IL	25	3	56.7±0.4	1.0×10^{-5}-1.0×10^{-2}	30 s	0.992
10	45	25-IL	25	5	58.8±0.4	5.0×10^{-6}-1.0×10^{-2}	18 s	0.997
11	43	25-IL	25	7	57.1±0.6	1.0×10^{-5}-1.0×10^{-2}	29 s	0.966
12	70	25-IL	-	5	5.1±0.9	5.0×10^{-4}-1.0×10^{-3}	3.4 min	0.843

Carbon paste electrodes (CPEs) are another category of potentiometric sensors which are mechanically strong. They have attracted more attention than membrane electrodes because of their advantages such as improved renewability, more stable response, low ohmic resistance and no need to internal solutions. Three kinds of carbon paste electrode, unmodified, modified with MWCNTs and modified with ionic liquid carbon paste electrodes with different compositions were prepared and their potential responses were studied.

The carbon paste electrode composed of 20% paraffin oil, 20% BUP-TPB, and 60% graphite powder (no. 4) was found to be optimal for unmodified or normal bupropion hydrochloride electrode. This composition was selected for further examination. Then, the past was modified by ionic liquid instead of paraffin oil. ILs are a good choice as binders in carbon paste electrodes due to their chemical stability, low vapor pressure, low toxicity, low melting temperature, high ionic conductivity and good electrochemical and thermal stability.

The third modification was done by using multi-walled carbon nanotubes (MWCNTs). Because of their particular physicochemical properties, such as ordered structures with high aspect ratios, ultra-light weight, high thermal conductivity, metallic or semi-metallic behavior, high surface area, high electrical conductivity and remarkable mechanical strength. High conductivity of MWCNTs increases the dynamic working range and response time of the carbon paste electrode. Addition of 5% MWCNT to the composition increased the response to a Nernstian slope of about 58.8±0.4 mV per decade (no. 10). From Table 2, it was obvious that in the absence of ion-pair and presence of other components (no. 12), the response of the modified CPE was too low (slope of 5.1±0.9 mV per decade).

Finally, the best sensor response in was obtained by the paste composition of 25% ion-pair, 5% MWCNTs, 25% ionic liquid and 45% graphite powder.

3.3. Characterization of the PVC membrane sensor

The proposed PVC membrane sensor calibration curve has been shown in Fig. 2. According to this figure, the measuring range of the sensor which is the linear part of the calibration curve is in the range of 1.0×10^{-5}-1.0×10^{-2} mol L^{-1}. Detection limit was calculated from the intersection of two extrapolated segments of the calibration graph. Detection limit of the PVC membrane sensor was calculated 8.5×10^{-6} mol L^{-1}. Slope of the calibration curve is 56.8 mV per decade of the bupropion hydrochloride concentration and a standard deviation of ±0.5 mV after five replicate measurements.

Dynamic response time which is the necessary time for the sensor to reach the values within ±1 mV of the final equilibrium potential, after successive immersions in the sample solutions [40-46]. It is obtained by variation and the recording of the bupropion hydrochloride concentration in a series of the solutions from 1.0×10^{-5} to 1.0×10^{-2} mol L^{-1}. The PVC membrane sensors were able to quickly reach its equilibrium response, about 25 s, in the whole concentration range. The sensor lifetime was estimated considering the calibration curve slope and detection limit periodically. Four same sensors were employed 1 hour per day for 10 weeks. After 5 weeks of utilization, a slight gradual decrease in the slope and an increase in the detection limit were observed. After several time of usage, the membrane ingredients leak from the organic layer of the membrane into the solution and affect the membrane response.

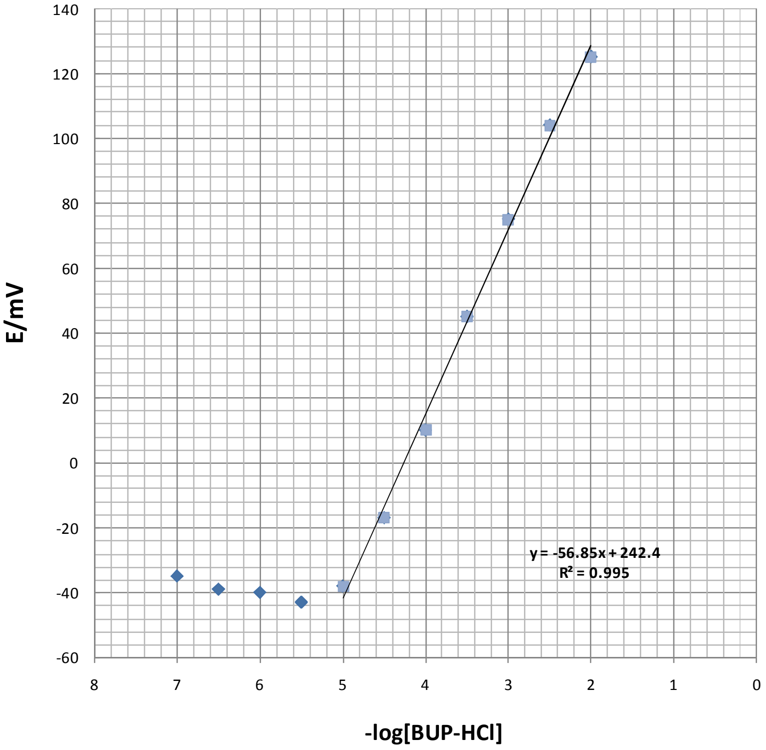

Figure 2. Calibration curves of BUP-HCl PVC membrane electrode (no. 3). The results are based on 5 replicate measurements.

3.4. Characterization of the nano-composite carbon paste sensor

The proposed nano-composite carbon paste sensor calibration curve has been shown in Fig. 3. Linear measuring range of this sensor is in the range of 5.0×10^{-6}-1.0×10^{-2} mol L^{-1}. Detection limit was calculated 3.1×10^{-6} mol L^{-1}. Nernstian slope of the calibration curve is 58.8 mV per decade of the bupropion hydrochloride concentration and a standard deviation of ±0.4 mV after five replicate measurements.

Dynamic response time of nano-composite carbon paste electrode, after successive immersions in the standard solutions from 1.0×10^{-5} to 1.0×10^{-2} mol L^{-1}, is obtained about 18 s. The nano-composite carbon paste sensor lifetime was estimated considering the calibration curve slope and detection limit periodically as mentioned for PVC membrane sensor. After 8 weeks of use, a decrease in the Nenrstian slope and an increase in the detection limit were observed. In comparison with PVC membrane electrode this time is more because in CPEs the surface of the sensor can be renew and thus it can be used for longer time.

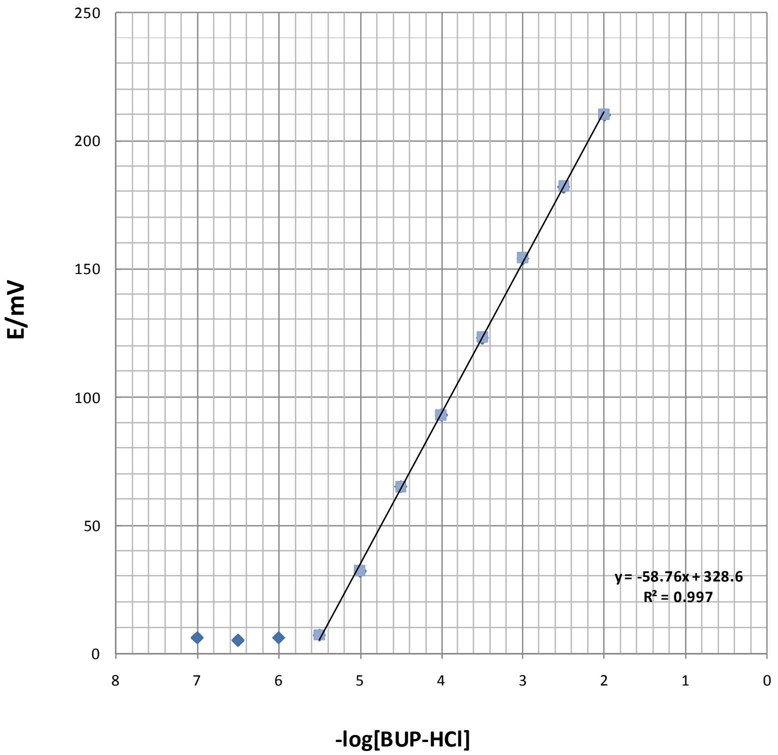

Figure 3. Calibration curves of BUP-HCl nano-composite electrode (no. 10) .The results are based on 5 replicate measurements.

3.5. pH Effect on the sensors response

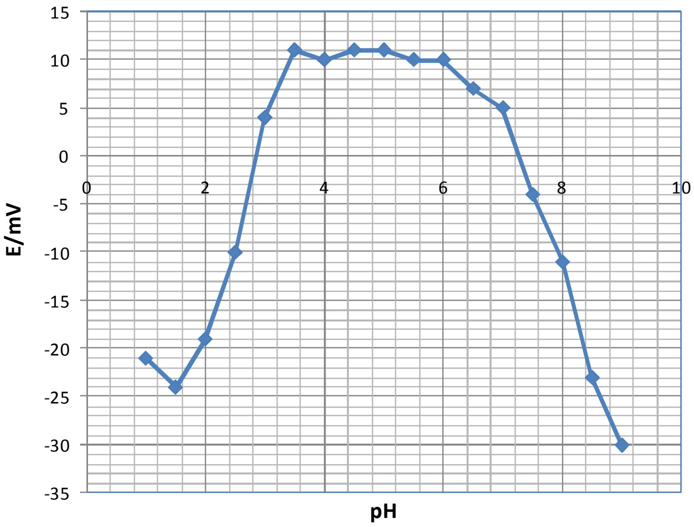

Figure 4. Applicable pH of the electrodes in the test solution of 1.0×10^{-4} mol L^{-1}

Fig. 4 shows the effect of pH on the sensors responses. To examine the effect of pH on the electrode responses, the potential was measured at specific concentration of the bupropion hydrochloride solution (1.0×10^{-4} mol L^{-1}) from the pH value of 1.0 up to 9.0 (concentrated NaOH or HCl solutions were employed for the pH adjustment) by PVC membrane electrode. The results showed that the potential remained constant despite the pH change in the range of 3.0 to 6.0, which indicates the applicability of this electrode in the specified pH range.

Relatively noteworthy fluctuations in the potential *vs.* pH behavior took place below and above the formerly stated pH limits. In detail, the fluctuations above the pH value of 6.0 might be justified by removing the positive charge on the drug molecule. Fluctuations below the pH value of 3.0 were caused by removal of the membrane ingredients or analyte in the solution. In both electrodes the same trend were observed.

3.6. Selectivity

Selectivity, sensor specificity toward the target ion in the presence of interfering ions, is the most important factor for the analytical applications. The potentiometric selectivity coefficients of the bupropion hydrochloride electrode were calculated by the matched potential method (MPM) [47-52]. In this method, a specified concentration of the primary ion (A) is added to a reference solution and the potential is measured. In a separate experiment, interfering ions (B) are successively added to an identical reference solution, until the measured potential matches the one obtained before the primary ions addition. The matched potential method selectivity coefficient is then given by the resulting primary ion to the interfering ion concentration ratio. The resulting values of the selectivity coefficients are shown in Table 3. The selectivity coefficients shows that the interferences form other species is negligible in the performance of the both sensors.

Table 3. Selectivity coefficients of various interfering compounds for bupropion hydrochloride sensors

Interfering ion	Log K_{MPM} (PVC membrane sensor)	Log K_{MPM} (Nano-composite CPE)
Na^+	-3.6	-3.5
K^+	-3.0	-3.1
NH_4^+	-2.7	-3.0
Ca^{2+}	-3.8	-4.0
Mg^{2+}	-3.9	-4.2
Lactose	-4.1	-4.3
Glucose	-4.0	-4.0

3.6. Analysis of bupropion in pharmaceutical formulation

The proposed sensor was evaluated by measuring the bupropion concentration in pharmaceutical formulations (Table 4). The drug concentration of the tables 100 mg was determined using calibration method with direct potentimetry. The results are in satisfactory agreement with the labeled amounts.

Table 4. Potentiometric determination of bupropion hydrochloride in pharmaceutical formulations

Sample	Found by PVC membrane electrode*	Found by Nano-composite CPE*
Sample 1	107.77±1.27 mg/Tab	105.52±1.97 mg/Tab
Sample 2	105.12±2.03 mg/Tab	106.72±2.00 mg/Tab
Sample 3	94.54±2.19 mg/Tab	93.11±1.86 mg/Tab
Sample 4	109.21±1.33 mg/Tab	108.14±1.63 mg/Tab

* The results are based on five replicate measurements.

3.7. Precision, accuracy, and ruggedness/robustness

3 standard samples were analyzed for repeatability study. The RSD values by PVC membrane were 3.5, 3.4, and 3.6% and for nano-composite CPE were 3.2, 3.0, and 3.4%.

For ruggedness of the methods a comparison was performed between the intra- and inter-day assay results for bupropion hydrochloride obtained by two analysts. The RSD values for the intra- and inter-day assays in the cited formulations performed in the same laboratory by the two analysts did not exceed 4.4%. On the other hand, the robustness was examined while the parameter values (pH of the solution and the laboratory temperature) changed slightly. Bupropion hydrochloride recovery percentages were good under most conditions, and not showing any significant change when the critical parameters were modified.

4. CONCLUSIONS

In the present work, two types of potentiometric electrodes were constructed for determination of bupropion hydrochloride. The sensors showed advanced performances with a fast response time, a lower detection limit of 8.5×10^{-6} mol L^{-1} for PVC membrane electrodes and 3.5×10^{-6} mol L^{-1} for carbon paste electrode, the potential responses across the range of 1.0×10^{-5}-1.0×10^{-2} mol L^{-1} and 5.0×10^{-6}-1.0×10^{-2} mol L^{-1}, respectively. The sensors enabled the bupropion hydrochloride determination in pharmaceutical formulations. Both sensors respond based on ion-exchange mechanism. The best PVC membrane electrode performance was achieved by a membrane composition of 30% PVC, 63% DBP and 7% ion-pair complex. Then, a carbon paste electrode was designed to improve the analytical responses. The best electrode was composed of 25% ion-pair, 25% IL, 5% MWCNTs and 45% graphite powder.

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