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Research Article

Carbon Paste Electrodes for Oxomemazine Hydrochloride: Fabrication and Evaluation

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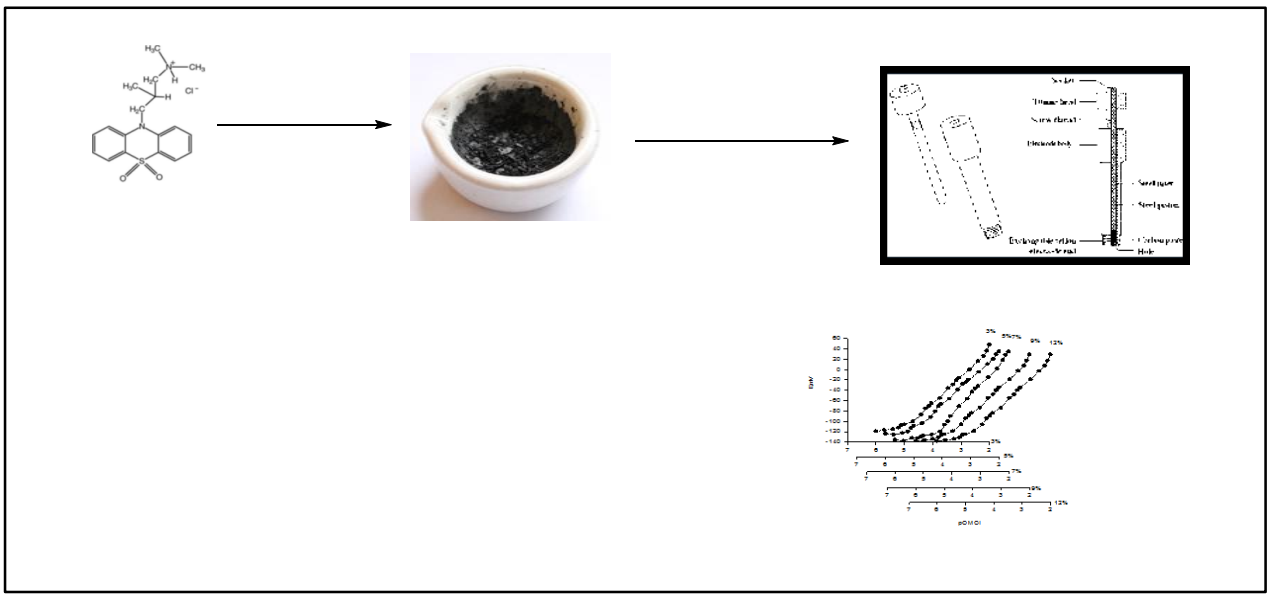
ABSTRACT

The evaluation of oxomemazine hydrochloride (OXCl) prepared in carbon paste electrodes is reported in detail. There are many practical techniques and experiments for electrode preparation one of them is mixing of the ion-pair with ion pairing agent. Prepared electrodes gave satisfactory data in ranging from (0.018 to 18.35) mg based on Nernstian cationic slopes, the used electrodes refer to the standard data obtained at (slope 58.7±2.1 mV decade^{-1}) by using sodium tetraphenylborate (NaTPB) and rapid response time equal to 15 s and lifetime approximately equal to (4 weeks). The prepared electrodes can be counted as end point indicator electrode for potentiometric titration of oxomemazine Hydrochloride OXCl resulting in a high percentage of accuracy.

GRAPHICAL ABSTRACT



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Introduction

Oxomemazine, a phenothiazine group, is regarded as a drug which is very significant, owing to its connection to hypersensitivity is deemed an important drug helping patients which suffer from cough, also common cold. Drug can be taken by several ways for the patients orally, rectally by using suppositories on a daily basis and precautions. Oxomemazine hydrochloride (OXCl) chemically has the formula [3-(5, 5-dioxodo-10H-phenothiazin-10-yl)-N, N, 2-trimethylpropan-1-amine] (Figure 1). Several techniques have been cited due to its general important characteristic studying, involving spectrophotometry [2,3] and HPLC [4]. With regard to the quality control, drug is utilized in necessary pharmaceutical industry with the aim of analyzing the starting materials, and finalized products.

ISEs ion selective electrodes counting up on a film, which is helpful in potentiometric measurements of activity of some ions by the means of other ions in the sample solution. Currently, ISEs ion selective electrodes exert a necessary role in measurements of solubility products of many economically soluble salts. Despite the progress of highly selective electrodes for various ions, no reference to progress of sensitive sensors for oxomemazine was reported [5,6].

The present study seeks to design and evaluate simple and easy sensors for rapid measurements of Oxomemazine OX. The obtained sensors were utilized as indicator electrode for potentiometric titration of Oxomemazine OX [7]**.** We used natural plasticizers in our study and this is due to low cost, more sensitive and safer for treatment of patients by drugs including them especially which suffers from liver and kidney diseases

Experimental

Material and methods

Oxomemazine hydrochloride Mwt, 366.91) and its pharmaceutical preparation were purchased by Egyptian Pharmaceutical Industries Company (Alexandria, Egypt), (NaTPB) $Na[C_{24}H_{20}B]$ (Fluka), some natural plasticizers, polyvinylchloride (PVC), Tetrahydrofuran, Graphite powder 1-2 micron, some organic and inorganic cations were purchased from Aldrich chemical company. Other chemicals have been taken from Riedel de Haen Chemical Company.

Apparatus

Measurements were carried out by system consists of double junction electrode silver/silver chloride and connected by very sensitive amount of drug [8,9].

The electrochemical system was as follows:

Ag/AgCl/filling solution/membrane/test solution//KCl salt bridge//SCE.

Results and discussion

Composition selection

Preparation of matrices of compositions was done by mixing amounts of graphite, natural plasticizers, and ion-pair which represent sensing material. The components of the membrane represent a very important role in electrode response. Data concluded that the optimum compositions showing the best performance, for OM-TPB (oxomemazine-sodium tetraphenylborate) attained up to 9% ion associate is 45.5% of graphite and 45.5% of plasticizer) in 18.34 mg OMCl as indicated in (Table 1).

Plasticizer selection

In the practical study, a group of natural plasticizers such as olive oil, corn oil and sun

flower oil, which are more preferred due to their low cost, and very safety in comparison to other types were used variation of natural plasticizers provides us the best sensitive of them. The data shows that sun flower oil is the more favorable and higher sensitivity, owing to low solubility of OM-TPB (oxomemazine-sodium tetraphenylborate) ion exchange in those solvents (Table 2).

Table 1. Composition of oxomemazine carbon paste membrane electrodes and slopes of their corresponding calibration graphs at 25±1 °C and 30 min of soaking

OM-TPB oxomemazine sodium tetraphenylborate electrodes parameters	Composition % (w/w) [Ion associate]				
Ion associate	3	5	7	9	12
Graphite	48.5	47.5	46.5	45.5	44.0
Sun flower oil	48.5	47.5	46.5	45.5	44.0
Slope mV/decade	51.2	52.3	58.5	58.7	47.0
RSD relative standard deviation %	1.56	1.79	0.89	2.86	3.50
Linearity range (mg)	27.52-183.45	56.87-183.45	73.19-183.45	5.687-183.45	5.687-183.45
LOD limit of detection (mg)	22.02	27.52	56.87	4.586	4.586
LOQ limit of Quantification (mg)	73.19	91.54	1.16	1.50	0.150
Response time (s)	8	8	8	8	8

Table 2. Effect of plasticizers on oxomemazine carbon paste membrane electrodes and slopes of their corresponding calibration graphs at 25±1 °C and 30 min soaking in 18.34 mg of OMCl oxomemazine hydrochloride

OM-TPB oxomemazine sodium tetraphenylborate	Plasticizer (45.5%)			
Electrodes Parameters	Without plasticizer	Corn oil	Sun flower oil	Olive oil
Slope mV/decade	51.2	52.3	58.7	57.5
RSD relative standard deviation%	1.79	1.22	1.96	2.46
Linearity range (mg)	27.52-183.45	0.22 - 183.45	5.687-183.45	5.687-183.45
LOD limit of detection (mg)	0.22	27.52	0.0459	0.0459
LOQ limit of Quantification (mg)	73.19	0.61	0.15	0.15
Response time (s)	8	8	8	8

Role of buffer

Matrix of buffers such as phthalate, phosphate and acetate buffers which very important for standardization of the accuracy of electrode response and showed that phthalate, gives the highest slope compared to phosphate and acetate (Table 3). This showed the electrode enjoys a high degree of stability In contrast with the others.

Following of soaking time

Freshly electrodes produced were immersed consequently in different soaking solutions. These preconditioning different soaking solutions cause formation of a thin gel layer at membrane surface where ion-exchange process can take place, also this conditions requires variations of soaking periods which related to wider spread and equilibrium beside interface. As reaching to that equilibrium definitely an ample indicator for excellent response. Significant characteristics of obtained electrodes were investigated in regarding to soaking times. For this purpose, obtained electrodes were studied and we concluded that soaking had effect on calibration curve by variation of times in independent way. For (OM-TPB) (oxomemazine-sodium tetraphenylborate) electrode, data obtained was revealed in (Table 4).

The life span of electrode is counting upon the adjectives of ion-exchanger; soaking electrodes for extended periods of time can decrease their response regarding drug action. This relates to filtering of mixture which formed ion-exchanger and plasticizer in solution. This is due to spread rates [10-15].

Response time consumed

The response time can be defined as the time which passes through the instant at the moment at which an ion-selective electrode and a reference electrode became in connection with a sample solution, In accordance with this data indicating very short response time approximately 8 s referred to in (Figure 3).

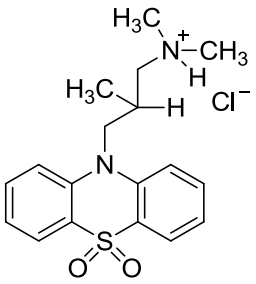
Table 3. Effect of buffer on oxomemazine carbon paste membrane electrodes and slopes of their corresponding calibration graphs at 25±1 °C and 30 min soaking in 18.34 mg of oxomemazine hydrochloride OMCl

OM-TPB oxomemazine sodium tetraphenylborate electrodes	Buffer		
electrodes Parameters	Phosphate	Acetate	Phthalate
Slope mV/decade	57.8	56.4	59.5
RSD relative standard deviation%	0.89	1.79	1.68
Linearity range (mg)	34.86-183.45	56.87-183.45	73.19-183.45
LOD limit of detection (mg)	27.52	45.86	56.87
LOQ limit of Quantification (mg)	91.54	15.26	0.189
Response time (s)	8	8	8

Table 4. Effect of soaking on sun flower oil carbon paste membrane electrodes at 25.0±1.0 °C

OM-TPB oxomemazine sodium tetraphenylborate electrodes	Soaking time				
Electrodes Parameters	0.5-24 h	2 days	8 days	21 days	30 days
Slope mV/decade	59.7	59.7	58.5	56.4	50.9
RSD relative standard deviation%	1.22	1.79	0.86	1.45	1.43
Linearity range (mg)	73.19-183.45	73.19-183.45-	0.459-183.45	27.52-183.45	56.87-183.45
LOD limit of detection (mg)	56.87	34.86	34.86	0.22	0.459
LOQ limit of Quantification (mg)	0.24	1.16	1.16	73.19	15.26
Response time (s)	8	8	8	8	8

Figure 1. The chemical structure of OMCl oxomemazine hydrochloride



PH measurements

Following the necessary influence of pH test solution the potential readings of prepared electrodes was investigated. The difference in potential with variation in pH measurements was resultant from pH 2.1 to pH 12.2 by adding small amounts of HCl and NaOH (each 0.1-1.0 M) to 183.45, 18.34 for 1.83 mg solution of OMCl. The data showed that the prepared electrodes did not give any response to variation in pH range 2.2-9.9 for OM-TPB (Figure 4). The descending in potential happening at very high pH values is a result of foundation of free Oxomemazine base in solution, leading to a descending in concentration of Oxomemazine cation: a pK_a= 10.6 [16,17].

Effect of temperature

Evaluation of the electrodes thermal stability

Contieously thermal stability of the obtained Electrode was investigated from calibration graphs ($E_{elect.}$ vs pdrug) at various test solution temperature which varies from 30-70 °C.

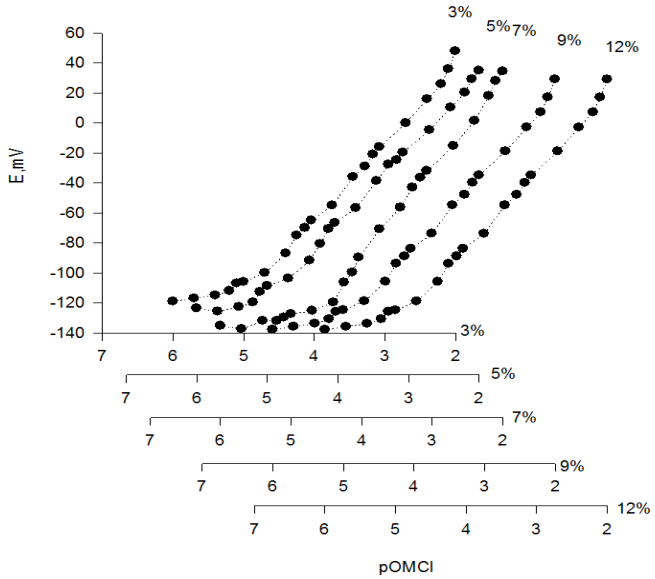


Figure 2. Calibration graphs using OM-TPB oxomemazine-sodium tetraphenylborate) carbon paste electrode sun flower oil at different ion associate percentage

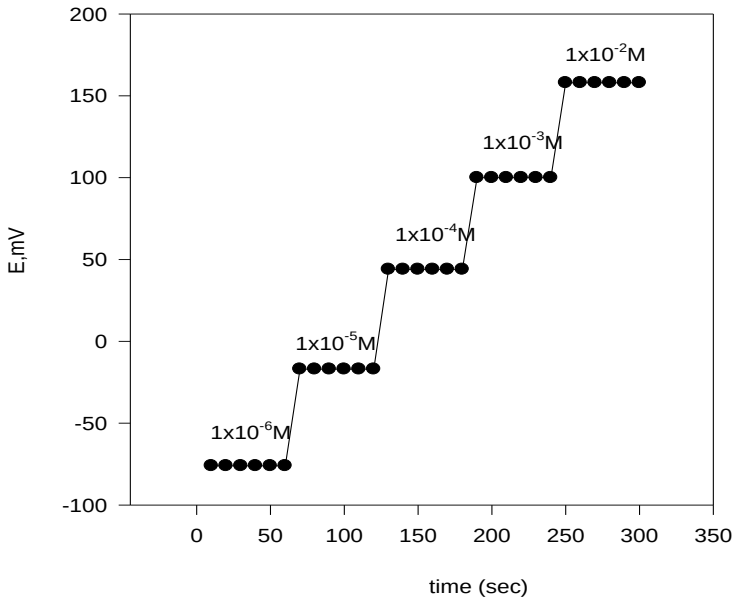


Figure 3. Potential-time plot for OM-TPB oxomemazine-sodium tetraphenylborate sun flower oil carbon paste electrode

The data obtained are shown in (Table 5) which explain although the slopes are slightly increasing, still follows the Nernstian range despite the variation of temperature of test solutions until reaching to 70 °C.

Calculation of the isothermal coefficient of the prepared electrodes

In fact we note that the temperature of the test solution has a great relation with the potential of ion-selective electrodes. The electrode which enjoys by low thermal coefficient is considered achieving a high degree of thermal stability. This shows applicability of the electrode can be succeeded through a wide range of temperature. Based on calculating (dE°/dt) the cells, we must determine the standard cell potentials, $E°_{cell}$, at various temperatures from the respective calibration plots as the intercept of these plots at p drug = 0, knowing that $E°_{cell}$ is related to (dE°/dt) according to [18,19].

$E°_{cell} = E°_{25°C} + (dE°/dt)\ (t-25)$ (1)

Plot of ($E°_{cell}$) versus (t-25) produces straight line; the thermal coefficient of the cell can be obtained from the slope of this line. The standard potentials of electrodes ($E°_{elec.}$) values can be measured after the subtraction of the standard electrodes potential of the calomel electrode at various temperatures severally; and this in turn reveals no difference from the theoretical Nernstian behavior.

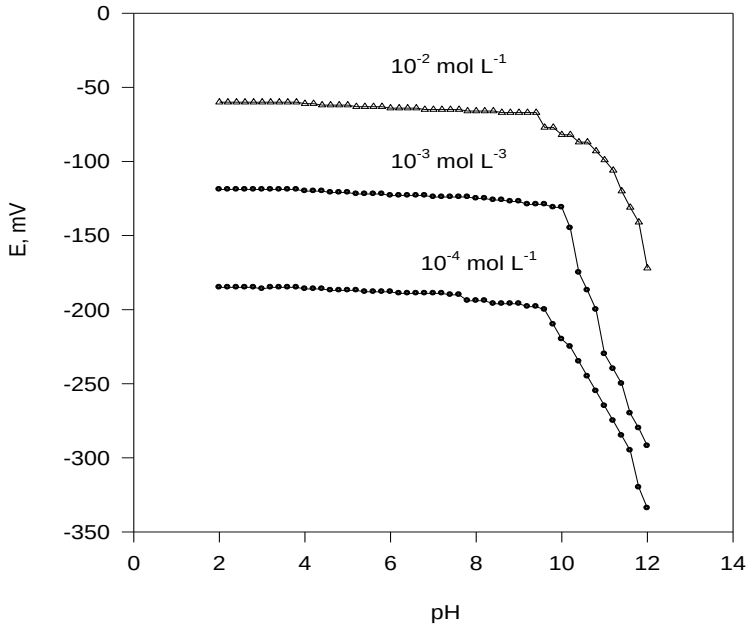


Figure 4. Effect of pH of the test solution on the potential response using OM-TPB oxomemazine-sodium tetraphenylborate sun flower oil carbon paste electrode

Table 5. Performance characteristics of oxomemazine carbon paste membrane sun flower oil electrodes at different temperatures

OM-TPB oxomemazine sodium tetraphenylborate	Temperature °C				
Electrodes Parameters	30	40	50	60	70
Slope mV/decade	54.2	55.5	56.6	59.0	59.4
RSD relative standard deviation%	3.45	2.95	3.67	1.78	3.50
Linearity range (mg)	22.02-183.45	27.52-183.45	34.86-183.45	73.19-183.45	73.191.183.45
LOD limit of detection (mg)	0.18	22.02	27.52	56.87	56.87
LOQ limit of Quantification (mg)	0.61	73.19	91.54	1.89	18.93
E^o cell the standard cell potentials, mV	180	190	200	210	220
Response time (s)	8	8	8	8	8

Selectivity measurements

The selectivity of the examined electrodes under occurance of some cations can be investigated using matched potential method. The careful studying of selectivity is fundamental resource of information with regard to interference on the examined electrode response.

There was an investigation for the studying of the influence of variations of inorganic cations, amino acids and sugars. The results were shown in (Table 6).

For many years, the method of determination of degree of interference by means of selectivity coefficients of electrodes in potentiometric measurements was a subject of discussion in the literature [20]. Two specialized IUPAC committees were convened with regard to the measurements of potentiometric selectivity coefficients. In the first IUPAC committee held [21], separate solution method (SSM)was recommended only if the electrode exhibits Nernstian response, but it was favorable in comparison to fixed interference method (FIM); owing to the fact that it does not give the real conditions under which the electrodes are utilized. In 1995, the second IUPAC committee recommended matched potential method (MPM) for reporting selectivity coefficient which is independent of Nicolsky Eisenman equation [22].

The examined electrodes indicated on excellent behavior towards inorganic cations, amino acids and some sugars. This behavior can be explained due to inorganic cations don't interfere is that there are differences in ionic size, mobility and permeability. In cases of sugars, amino acids and vitamins, the high selectivity is fundamentally related to the difference in polarity and to the lipophilic nature of their molecules relative to oxomemazine hydrochloride OMCl.

Analytical applications

Several methods were used in use for quantitative analysis using ion-selective electrodes. These include: (i) Direct calculation of the concentration applying Nernst equation. This method is subjected to several types of errors, e.g.1mV shift in electrode potential reading results in 4 n% errors in which n is defined as the change of the ion. (ii) Potentiometric titration regarding the use of counter ion as titrant ion which is rather accurate depending fundamentally on using of ion selective electrode (ISE) for detecting end point detector. (iii) Standard addition method (SAM), which is frequently applied in using ISE.

Potentiometric determinations applying the standard addition method SAM

The Standard addition method (SAM, shown in the experimental part, was evidenced to be successful for Oxomemazine determination in pure solutions and in pharmaceutical preparations, *via* the prepared electrodes as sensors.

Measurements of the drug in bulk and pharmaceutical analysis

The standard addition method, described in the experimental part, was evidenced to be sufficient for measurements of (oxomemazine-sodium tetraphenylborate) OM-TPB in pure solutions via using the respective electrode as sensor. This is evident from the small relative standard deviation RSD values in ranges (0.69-2.22%, 2.71-2.98% and 0.98 -0.49) for (Oxomemazine-sodium tetraphenylborate) OM-TPB membrane (Table 7), which shows the high percentage of accuracy and precision of the electrodes [5,6].

Potentiometric titrations

Although the fact that the measurements of concentration using potentiometric titration is time consuming, enjoys from the

advantages as high percentage of accuracy and precision. Moreover, the calculation of the end point can be easily indicated by a sharp potential break; not to mention the utilization of partially exhausted electrode is possible and the exact potential value at the end point is of secondary interest [23].

The basic idea of this type of potentiometric titration is related to ion-associates formation. The titration process is influenced by several factors such as degree of completeness of the reaction, also the relation between the equilibrium constant of precipitation titration and the product solubility, *i.e.,* the smaller solubility of product of the formed ion-exchanger, the sharper is the end point. As a result, the calculation of the solubility of product of the precipitate is of grand significance if a titration, which causes the formation of this precipitate, is under investigation. The reciprocity of the solubility product is nearly equivalent to the equilibrium constant of the precipitation reaction added to in the titration.

Table 6. Selectivity coefficients $-\log K^{pot}_{OM,J^{Z+}}$ for the OM sun flower oil electrodes

Interferent	OM-TPB oxomemazine sodium tetraphenylborate	
	SSM	MPM
Na^{+}	2.99	3.47
K^{+}	2.21	2.68
NH_4^{+}	1.65	1.91
Ca^{2+}	3.59	4.76
Mg^{2+}	2.78	3.55
Co^{2+}	3.57	4.30
Zn^{2+}	3.48	4.78
Ni^{2+}	3.44	3.98
Sr^{2+}	3.68	2.88
Ba^{2+}	4.26	4.38
Fe^{3+}	3.55	4.40
Cu^{+2}	3.95	2.76
Cd^{+2}	4.25	4.55
Glucose	-	2.40
Fructose	-	2.18
Maltose	-	3.25
Lactose	-	2.36
Alanine	-	4.37
Glycine	-	2.28
Urea	-	3.68
Ascorbic	-	1.19

Table 7. Determination of OMCl oxomemazine hydrochloride in pure solution and pharmaceutical preparations applying the standard addition method on 9% sun flower oil

Sample	Carbon paste membrane electrodes			
	Taken mg	Found mg	Mean Recovery %	RSD %
OM-TPB oxomemazine	10.80	10.70	99.1	0.69
sodium tetraphenylborate	21.60	21.27	98.5	1.96
pure solution	32.40	32.56	100.5	2.50
	43.20	44.17	97.2	2.22
Toplexile syrup	32.72	32.55	99.5	2.71
	65.44	66.42	99.0	2.65
	98.16	97.00	98.9	2.98
Rectoplexile susppositotry	3.30	3.26	99.0	0.98
	6.60	6.43	97.5	0.76
	9.90	9.94	100.5	0.49

Table 8. Determination of OMCl oxomemazine hydrochloride in pure solution applying potentiometric titrations method on 9% sun flower oil

Sample	Carbon paste membrane electrodes			
	Taken mg	Found mg	Mean Recovery %	RSD %
OM-TPB oxomemazine sodium	10.80	10.70	99.1	0.69
tetraphenylborate pure solution	21.60	21.27	98.5	1.96
	32.40	32.56	100.5	2.50
	43.20	44.17	97.2	2.22
Toplexile syrup	32.72	32.55	99.5	2.71
	65.44	66.42	99.0	2.65
	98.16	97.00	98.9	2.98
Rectoplexile susppositotry	3.30	3.26	99.0	0.98
	6.60	6.43	97.5	0.76
	9.90	9.94	100.5	0.49

Table 9. Critical response characteristics of carbon paste electrodes electroanalytical pramaters

Parameters	Value
Slope mV/decade	58.7
RSD relative standard deviation %	2.86
Linearity range (mg)	5.687-183.4
LOD limit of detection (mg)	4.586
LOQ limit of Quantification (mg)	1.50
P^H range	2-9.8
(dE°/dt) thermal coefficient electrode	$1.666x10^{-3}$
Response time (s)	8
Life time day	30min to 30 day
Recovery %	99.1

Furthermore, it has been indicated [24] that in a precipitation titration curve, the point with maximum slope may somewhat come before the equivalence point when the solubility product of the precipitate obtained is proportionally high. The (oxomemazine-sodium tetraphenylborate) OM-PB electrodes were evidenced to be of much use for the measurements in pure solutions by potentiometric titration against standard solution of sodium tetraphenyl borate. Representative titration curves are indicated when, it is observed that when concentration of the drug is on increasing and, the inflection of the break point becomes sharper in comparison with the low drug concentration. Relative standard deviation RSD; besides the recovery values are referred to in Table 8. Electroanalytical parameters of new carbon paste electrode showed in Table 9.

Conclusion

The present work has successfully indicated that the fabrication of oxomemazine carbon paste electrode OX-CPE electrode utilizes different preparation methods. The prepared electrodes showed Nernstian slopes in the concentration range 0.018 to 18.35 mg with rapid response time (15 s), and long life span (4 weeks). The fabricated electrodes exert a significant role in its application for determination of end point indicator electrode for potentiometric titration of oxomemazine OX with NaTPB in the concentration ranging from 0.018 to 18.35 mg with very good accuracy and sensitivity. Fabricated electrodes had shorter response time (10 s) compared to drug electrode.

Aknowledgement

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