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Influence of Multiwalled Carbon Nanotubes on the Response Performance of Carbon Paste Iodide Ion Selective Electrode Based on Iron (II) Phthalocyanine

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In this research, new iodide-selective electrode based on incorporating of iron (II) phthalocyanine (IPC) into carbon paste matrices has been reported. To achieve nernstian response over a wide linear range, the influence of multiwalled carbon nanotube (MWCNT-CPE), carbon paste composition (lipophilic cationic and anionic additives and amount of carbon nanotubes) was investigated and their values was set as Carbon powder, MWCNT: Nujol, Carrier MTOACl, (0.25g, 60 mg, : 50mg, 8.0 mg : 4.8 mg) respectively. The proposed electrode exhibited near-Nernstian slopes of -58.02± 0.35 of iodide concentration over the range 5.0×10^{-7}-1.0×10^{-1} M with detection limits of 4.0×10^{-7} M for the MWCNT-CPE electrodes based on IPC respectively. The proposed electrode has relatively fast response times (≤ 25 s), satisfactory reproducibility, while it is applicable for at least two months. The potentiometric response of the proposed electrode is independent of pH of the test solution over the range of 3.0–11.5. The selectivity sequence of proposed iodide selective electrode evaluated by different methods such as separate solution method (SSM) and fixed interference method (FIM) show its iodide preference over other ions that due to this advantage, this electrode successfully has been applied for accurate evaluation of iodide ion content in various real samples and as indicator electrode in precipitation titration.

Keywords: Iodide-selective electrode, Potentiometric sensors, Iron (II) phthalocyanine, Carbon paste electrode, Multiwalled carbon nanotubes (MWCNT).

1. INTRODUCTION

Iodine and iodide compounds as essential micronutrient has distinguished role in brain function, cell growth, neurological activities and thyroid function [1]. Because of this wide application and diverse roles different analytical tools have been applied for its quantification at low levels. Some of these previously reported methods [2-12] require expensive instrumentation, sample pretreatments and are labor intensive. Ion selective electrodes (ISEs) as one of alternative with distinguished advantages including simplicity, low cost, wide linear range, reasonable selectivity and non-destructive analysis, is a powerful low cost methods for direct and easy evaluation of analytes [13,14]. Among the various potentiometric methods, carbon paste electrodes (CPEs) is distinct in terms of re-newability, stable response, low ohmic resistance without requiring internal solution is a sufficient instrument for potentiometric measurement [15-18].

It was pointed out that impregnation of carbon nanotubes (CNTs) with high surface area, high mechanical, chemical and thermal stability and unique potentiometric evaluation [19-22]. In addition to metallic or semi-metallic behavior significantly improve the performance of analytical procedure especially [23-25]. Due to important role of carrier on imparting control of the selectivity of electrode there is a strong requirement to search new lipophilic character carrier that able to selectively and reversibly bind to a specific ion [26]. Several I^-- ISE, based on incorporation of the relatively selective transition metal complexes into various matrices have been reported the potentiometric response properties of some of them are presented in Table 1.

Table 1. Characteristics performance of some iodide sensors based on various ionophores.

carrier	Slop (mV/decade)	L.R (µM-M)	D.L (µM)	pH	Response time	Ref
Shiff base of Co(II) (salophen)	-55.8	2.0-0.06	0.7	2.5	< 1 min	[27]
Nickel(II) tetraazaannulene macrocyclic complex	-54.7	8.0-0.1	3	3-4	5 s	[28]
Schiff base complex of Fe(III)	-71.0	1.0-0.5	0.65	3.5-10	<15 s	[29]
Cu(I)-bathocuproine complex	-56.8	5.0-0.2	1	3-9	10 s	[30]
$[Hg(CTP)_2]$	-58.4	1.0-0.1	0.6	3.5-11.5	<10 s	[31]
Thiopyrilium ion derivative	-60.2	0.8-0.1	0.2	5.5-8	15 s	[32]
$[Ni(L^1)]^{2+}$	-58.6	40-0.1	16	7-10	3 s	[33]
$[co(L^1)]^{2+}$	-53.0	40-0.1	4	7-10	3 s	[33]
Iron (II) phthalocyanine	15 (CPE)	1.0-0.1	0.75	3.0-11.5	25	Present work
	22 (MWCNT-CPE)	0.5-0.1	0.4	3.0-11.5	20	

Some of these electrodes suffers from limitation such as the narrow working concentration range, high detection limit, slow response rate and especially poor selectivity over interfering ions and easily leak out from matrix that restrict their widespread application [27-33].

In order to cope with limitation the purpose of this work was to develop a new MWCNT carbon paste iodide selective electrode by application of iron (II) phthalocyanine (IPC). In order to improve the analytical usefulness of the modified electrode following investigation of the influence of effective variables performance of electrode such as slope characteristics, detection limit, response time, selectivity and dependence of the electrode potential on the solution pH.

2. EXPERIMENTAL

2.1. Reagents

All chemicals (such as methyltrioctylammoniun chloride (MTOACl), iron (II) phthalocyanine, dimethysolfoxide (DMSO), etc) with the purity of analytical reagent grade and doubly distilled water were used throughout. Graphite powder, multiwalled carbon nanotube and high purity Nujol oil from Fluka (Buchs, Switzerland) were used for the preparation of the unmodified and modified carbon paste electrode. The compounds used for preparation of complexes including iron (II) phthalocyanine, DMSO, water and all other chemicals were of purity grade from Fluka, and were used without further purification. Stock 0.5 M iodide solutions were prepared daily from potassium iodide. All aqueous solutions were prepared with deionized distilled water. The pH adjustments were made with dilute nitric acid or sodium hydroxide solutions as required.

2.2. Electrode preparation and potential measurements

Unmodified carbon paste (CPE) was prepared by thoroughly mixing of 300 mg of reagent-grade graphite powder and 60 mg of Nujol oil with a mortar and pestle. Modified carbon paste (MWCNT-CPE) was prepared by mixing 250 mg of graphite powder, 60 mg of Nujol with different weight of ionophore and additive to get different compositions. All carbon paste compositions were packed into 5.0 mL polyethylene syringes with diameter of 2.5 mm. Copper wire was glued (imbedded into carbon paste matrices) on the end of carbon paste for electrical contact.

2.3. Apparatus and potential measurement

The electromotive force (emf) measurements were carried out with the following cell assemblies: CPE│Sample solution│Reference electrode that a double junction Ag/AgCl was used as reference electrode with a pH/mV meter. The pH of the sample solutions was monitored simultaneously with a conventional glass pH electrode. The performance of each electrode was investigated by measuring its potential in I^- ion concentration in the range of 1×10^{-7}– 1×10^{-1} M by serial dilution of the 0.5 M stock solution at constant pH. The solutions were stirred and potential readings recorded when they reached steady state values. The data were plotted as observed potential versus the logarithm of I^- concentration. Absorbance measurements were carried out with a Jasco model V-570 spectrophotometer using a 1 cm quartz cells (Jasco Co., Hachioji, Tokiyo, Japan).

3. RESULTS AND DISCUSSION

In a set of primary experiments, the proposed carrier has been used for construction of CPEs for numerous mono, di and trivalent anions (Fig. 1) and it was seen this carrier is able for selective binding of I^- ion to give a Nernstian potentiometric response aver a wide concentration range while it has low linear range and non nernstian response for other anions.

Blank CPE (in the absence of carrier) show low linear range and non Nernstian response which its potentiometric response viz linear range, detection limit, and response time significantly can be improved by incorporating carrier molecules to the carbon paste matrices.

Iron phthalocyanine is a four coordinated complex with a square planner structure; therefore it can accept one or two anions at its axial positions which lead to formation of I(IPC) and/or I_2IPC. On the other hand iodide ion as high polarizable anion can be trapped by two iron(II) phthalocyanine as sandwiched to form a two nuclear iron complex $I(IPC)_2$.

The iron(II) centers are soft and it is capable for efficient and selective binding of soft iodide ions. Oxygen donor ions are hard species and therefore are expected to have weaker interaction with iron(II) centers especially if there is strict effect for their coordination. It is obvious that the more streak hinder of such large anions causes their poor coordination with metallic center of this proposed complex as recognition element that all of these phenomena lead to improvement of selectivity of this electrode. In considering the cited explains and high stability constant, it is confirmed the applicability of proposed carrier for fabrication of iodide selective electrode.

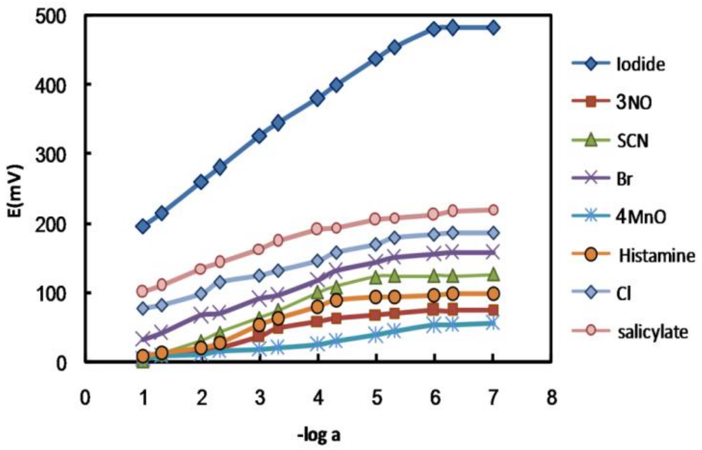

Figure 1. Potential response of carbon paste ion-selective electrode (electrode number 22) at pH=5.0±1.0 over concentration range of 1.0×10^{-7}- 1.0×10^{-1} mol L^{-1}.

3.1. Influence of the membrane composition on the electrodes response

Different membrane compositions by changing the amount of carrier, graphite powder, type and concentration of additive has been prepared according to Table 2, and their influence of the potentiometric response of iodide selective electrode in the range of 1.0×10^{-7} - 0.1 M has been investigated. Due to unique role of carrier and this point that modifier must has enough active sites to bind analyte ions, different electrode composed of various amount of carrier in the range of 2.0-12.0 mg at fixed ratio of carbon/Nujol and NaTPB/carrier was prepared and their responses was traced during experimental working iodide concentration range. It was observed that the working range and sensitivity of the electrode response were improved by increasing the amount of carrier up to 8.0 mg

(Table 2) and at higher value of carrier probably due to saturation or non-uniformity of the membrane the response performance significantly changed.

The influence of the type and concentration of the additives by addition of MTOACl or NaTPB into the carbon paste composition was assessed and its was found that potentiometric response of this proposed electrode greatly improved following addition MTOACl to electrode ingredient. In the presence of NaTPB no anionic response was observed. The effect of MTOACl amount in the carbon paste composition was investigated at several additive/carrier mole ratios according Table 2 and it was seen that MTOACl/carrier mole ratios of 50% exhibited maximum sensitivity over a wide range of iodide concentration. Among the different CPE compositions studied, 250 mg graphite powder, 8 mg carrier, 4.8 mg MTOACl, 60 mg Nujol and 50 mg MWCNT for MWCNT-CPE, show the best potentiometric response which was selected for further studies (Table 2).

3.2. Influence of multiwalled carbon nanotube on performance of iodide ion selective electrode

Table 2. Influence of composition on response of iodide selective electrode.

N	Graphite powder (mg)	IPC (mg)	MTOACl (mg)	Nujol (mg)	MWCNT (mg)	Slope (mV per decade)	L. R. (µM-M)	DL (µM)	Response time (s)
1	300	2	0.9	65	0	41.00	10.0-0.05	7.50	30
2	300	4	1.9	65	0	47.87	5.0-0.1	4.20	30
3	300	6	2.9	65	0	54.25	1.0-0.1	0.90	30
4	300	8	3.8	65	0	56.87	1.0-0.1	0.75	30
5	300	10	4.8	65	0	51.71	1.0-0.1	0.80	30
6	300	12	5.8	65	0	52.10	5.0-0.1	3.50	30
7	300	8	0	65	0	37.01	5.0-0.1	0.80	30
8	300	8	0.9	65	0	38.96	5.0-0.1	4.50	30
9	300	8	1.9	65	0	40.97	1.0-0.1	0.80	30
10	300	8	2.9	65	0	45.96	1.0-0.1	0.80	30
11	300	8	3.8	65	0	47.78	1.0-0.1	0.78	30
12	300	8	4.8	65	0	57.78	1.0-0.1	0.80	30
13	300	8	5.8	65	0	62.91	5.0-0.01	7.00	30
14	300	8	4.8	50	0	64.31	5.0-0.1	7.00	25
15	300	8	4.8	60	0	58.82	1.0-0.1	0.75	25
16	300	8	4.8	70	0	52.96	5.0-0.05	8.50	35
17	300	8	4.8	80	0	38.32	10-0.05	9.00	40
18	290	8	4.8	60	10	58.55	1.0-0.1	0.75	25
19	280	8	4.8	60	20	58.37	1.0-0.1	0.75	22
20	270	8	4.8	60	30	58.12	1.0-0.1	0.75	22
21	260	8	4.8	60	40	57.00	1.0-0.1	0.75	20
22	250	8	4.8	60	50	58.02	0.5-0.1	0.40	20
23	245	8	4.8	60	55	54.65	0.5-0.1	0.40	22
24	240	8	4.8	60	60	56.83	0.5-0.05	0.40	20
25	230	8	4.8	60	70	54.24	0.5-0.01	0.45	18

It seems that addition of MWCNT to the carbon paste improves the conductivity and transduction of the chemical signal to electrical signal. In this regard, response properties of a set of similar carbon paste electrode containing various amounts of MWCNT at fixed optimum value of other variables according to Table 2 has been investigated and their potentiometric response toward I^- ion has been investigated. Result of Table shows that MWCNT till 50 mg improves the conductivity and response property of proposed sensor such as dynamic working range, response time, selectivity and correlation coefficient.

3.3. Effect of pH of the test solution

The behavior of the proposed CPE based on IPC ionophore in the pH range of 3.0-11.5 using 1.0×10^{-3} and 1.0×10^{-2} M iodide solutions was investigated and results are given in Fig. 2. The maximum change ΔE/ΔpH over the pH range of 3.0-11.5 is 1 mV that show OH^- cannot compete with iodide ion even at high pH values. This observation may be due to this fact that the OH^- as hard anion has low tendency for coordination to the soft iron (II) center in the proposed carrier and therefore cannot compete with iodide ions for binding to the complex.

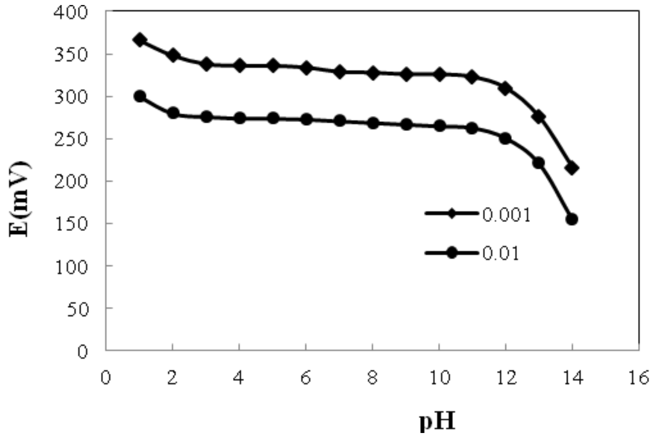

Figure 2. Effect of pH test solution on the potential response of the iodide selective electrode based on MWCNT-IPC in the presence of 1.0×10^{-2} and 1.0×10^{-3} M iodide ion.

3.4. Response characteristics of the electrode

The dynamic response time of the proposed electrode at optimum value of all at various concentrations (1.0×10^{-5} to 1.0×10^{-1} mol L^{-1}) of I^- ion was evaluated and the respective results is shown in Fig. 3. Results illustrate that in the whole concentration range the electrode (No. 22 in Table 2) reaches equilibrium response about 20 s. The life time and stability of the electrodes were tested by repeated monitoring of potentials and plotting calibration curve over a period of 2 months. Due to a very slight gradual decrease in the calibration slopes of the electrode after 2 months seems that it can be efficiently applied for accurate and fast determination of iodide ion concentration. On the other hand, the relative standard deviation (RSD) of the slope of calibration curve for the same electrode (between electrode variation) stored in 0.05 M potassium iodide solution over periods of 15 days and 2 month were 2.0 and 3.0 %, respectively. The standard deviation of slope for several electrodes (constructed by the same composition) was −58.4 ± 1.6 mV/decade of iodide concentration.

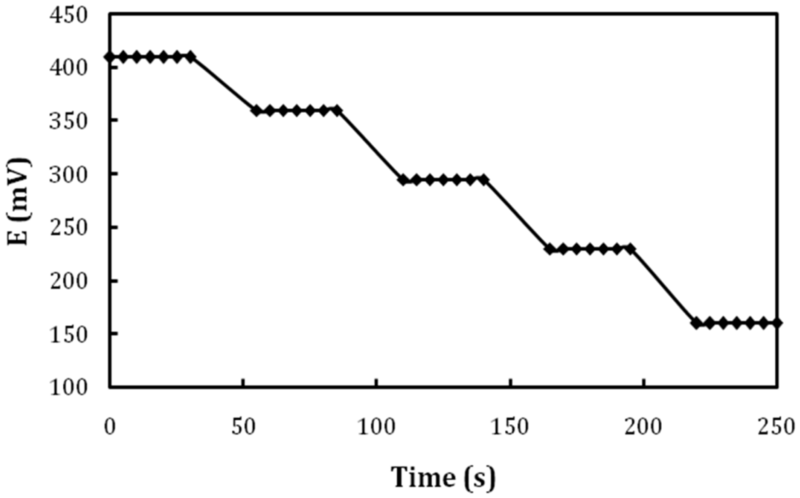

Figure 3. Response time of proposed of the iodide selective electrode based on MWCNT-IPC in the presence of different concentrations (1.0×10^{-5} – 1.0×10^{-1}) of iodide ions**.**

3.5. Selectivity coefficients of iodide ion selective electrode

Potentiometric selectivity coefficient show the accuracy of application of each electrode for evaluation of analytes content strongly depends to analyte content and method of evaluation. Separate solution method (SSM) and mixed solution method (MSM) [34,35] has been used for evaluation of selectivity coefficient of proposed electrode. In SSM, the Nicolskii coefficient determined by comparing potential of two solutions, containing a salt of the primary and interfering ion only, while the selectivity coefficient is determined using following equation (1):

$$ln_{IJ}^{SSM} = \frac{z_I F[E_2 - E_1]}{RT} - ln\alpha_1\left(1 - \frac{z_I}{z_J}\right) \quad (1)$$

In Eq. (1) it is considered that $\alpha_I = \alpha_J$. E_1 and E_2 are the response of the electrode to main and interfering ion, respectively.

In the fixed interference method (FIM) selectivity coefficients of the proposed iodide sensor were evaluated graphically for solution containing a fixed concentration of the interfering ions (1.0×10^{-3} mol L^{-1}) and different amounts of the iodide ion and results are shown in Table 3.

The meaning of selectivity coefficients K_{IJ}^{FIM} determined with this method is intuitively convincing and clearly reflect observation with real-world sensors in relevant samples. The selectivity coefficient, K_{IJ}^{FIM} , is determined by the following equation (2):

$$K_{IJ}^{FIM} = \frac{\alpha_1(DL)}{\alpha_J(BG)^{z_I/z_J}} \quad (2)$$

Table 3. Selectivity coefficients of the proposed carbon paste iodide selective electrodes.

	-Log*K*			
	22 (MWCNT-CPE)		15 (CPE)	
Ion	FIM	SSM	FIM	SSM
Permanganate	3.90	4.10	3.20	3.50
Perchlorate	2.57	3.15	2.18	2.80
Phosphate	3.27	3.50	3.12	3.20
Oxalate	3.85	3.64	3.62	3.44
Salicylate	2.90	2.70	3.00	2.80
Bromide	2.90	2.40	2.70	2.60
Chloride	3.10	3.40	2.90	2.30
Carbonate	3.10	3.30	3.20	3.00
Nitrate	3.80	4.06	3.30	3.80
Nitrite	3.78	3.83	3.60	3.80
Sulfate	3.52	3.68	3.70	3.40
Thiocyanate	2.90	3.30	2.80	2.60
Histamine	3.60	3.50	3.50	3.14

From Table 3, it can be seen that the proposed electrode is highly selective towards iodide with respect to a variety of anions. There is a satisfactory agreement between the two sets of results obtained for several anions by the SSM and FIM methods. It is believed that the order of selectivity the strong and selective interaction of IPC with iodide ion. All performances and properties of proposed electrode are presented in Table 4.

Table 4. Response characteristics of the proposed iodide ion selective electrode.

Properties	Values/range	
Optimized electrode composition	Carbon powder: Nujol, Carrier: MTOACl (0.30 g : 60 mg, 8.0 mg : 4.8 mg)	Carbon powder, MWCNT: Nujol, Carrier MTOACl, (0.25 g, 60 mg, : 50 mg, 8.0 mg : 4.8 mg)
Electrode type	Carbon paste electrode	
pH range	3.0-11.5	
Linear range I^- (M)	1.0×10^{-6} - 1.0×10^{-1}	5.0×10^{-7} - 1.0×10^{-1}
Slope (mV/decade)	58.82	58.02
Detection limit (M)	0.75×10^{-6}	4.0×10^{-7}
S.D of slope (mV/decade)	± 0.56	± 0.35
Response time (s)	≈ 25 s	< 20 s

3.6. Determination of iodide ion in real samples

The proposed electrode was applied for evaluation of iodide content in various water samples (river and well waters were collected from Yasouj, Iran) and method accuracy was examined by standard addition method.

The presented results (Table 5) indicate the obtained amount of I^- ion with the help of the sensor is in good agreement with those obtained by the standard addition method. The detection limits were (obtained from the intersection of the two extrapolated segments of the calibration graph) is 4.0×10^{-7} M that in this regard is superior to other previously reported iodide potentiometric sensors (See Table 1).

The resulting electrodes were applied to the determination of iodide in a drug preparation (Melamine Compound Injection, from Darou Pakhsh Pharmaceutical Co., Iran, is an iodide containing drug used for protection from light and secondary X-rays). The sample was prepared by refluxing 1.0 mL of the drug preparation in concentrated sodium hydroxide solution in the presence of zinc powder for 30 min [36]. The reaction mixture was cooled and filtered. The washed filtrate was acidified with H_2SO_4 and diluted to 1 L with water and its iodide content of the resulting solution was determined potentiometrically by the standard addition method at pH 5.0. The results obtained by the electrode method are in agreement with those obtained by titration with standard $AgNO_3$ solution as given in Table 5.

Table 5. Application of the proposed iodide selective electrode for determination of I^- ions concentration in real samples.

Sample	Add value(M)	Electrode number					
		15 (CPE)			22 (MWCNT-CPE)		
		Found(M)	RSD (%)	Recovery (%)	Found(M)	RSD (%)	Recovery (%)
Meglumine compound	0.0*	0.0056	2.0		0.0058	1.9	
	0.01	0.0153	3.1	98.1	0.0160	2.5	101.2
River water	0.0	0.0002	1.8		0.0002	1.1	
	0.0001	0.0003	2.0	103.5	0.0003	1.9	103.3
Well water	0.0	0.0003	2.6		0.0003	2.5	
	0.0001	0.0004	1.4	105.0	0.0004	1.2	98.6

The proposed electrode successfully has been applied for the potentiometric titration of Ag^+ with iodide and vice versa and typical titration plots are shown in Fig. 4.

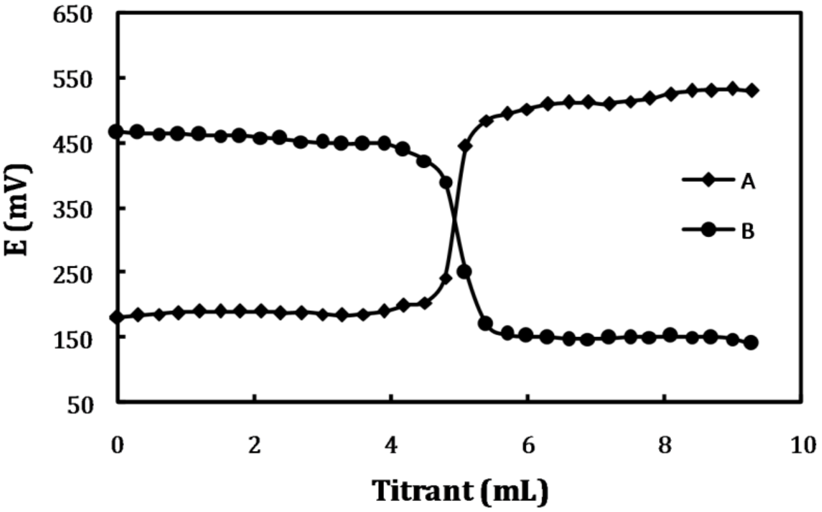

Figure 4. Titration curve of 50 mL of 0.01 M of iodide with 0.1 mol L^{-1} of Ag^+ ion at pH 5.0 ±1.0 using electrode number 22 of Table 2 (A) and reverse (B) at optimum condition of carbon paste according to Table 2.

All of these evaluations show the applicability of proposed sensor for accurate and preciss determination of iodide content in various real samples with complicated matrices.

4. CONCLUSIONS

New iodide-selective CPE by incorporation of IPC as neutral carrier into CPE matrices. It was sent that incorporation of MWCNT lead to improving detection limit (4.0×10^{-7}), increasing linear range (5.0×10^{-7} - 1.0×10^{-1}) in wide pH range (3.0-11.5). Due to strong coordination of iodide ion to and the proposed carriers high selectivity was obtained. As it can be seen incorporation of MWCNT significantly improve the performance of electrode such as linear range, detection limit and especially response time and selectivity probably by improving mass transfer from bulk to the diffusion layer.

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