

## Highly Selective Potentiometric Determination of 1-dodecyl-5-methyl-1H-benzo[d][1,2,3]triazol-1-ium Bromide Surfactant in Polluted Water Samples Using 1,4-bis-(8-Mercaptooctyloxy)-Benzene Ionophore

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Sensitive potentiometric sensors for determination of 1-dodecyl-5-methyl-1H-benzo[d][1,2,3]triazol-1-ium bromide (DMTB) have been constructed. The fabricated modified carbon paste electrodes (MCPE) are based on 1,4-bis-(8-mercaptooctyloxy)-benzene as sensing ionophore. Electrode matrices compositions were optimized referring to the effect of nature and content of the sensing ionophore and plasticizer. The electrodes displayed a linear potential response over wide concentration range from  $2.6 \times 10^{-7}$  to  $1 \times 10^{-2}$  and  $1.4 \times 10^{-7}$  to  $1 \times 10^{-2}$  mol L<sup>-1</sup> with Nernstian slopes of  $59.33 \pm 0.27$  and  $58.59 \pm 1.31$  mV decade<sup>-1</sup> of DMTB cation concentration with a detection limit of  $2.6 \times 10^{-7}$  and  $1.4 \times 10^{-7}$  mol L<sup>-1</sup> for MCPEs with dibutylphthalate (DBP) (sensor I) and *o*-nitrophenyloctylether (*o*-NPOE) (sensor II) as plasticizers, respectively. DMTB in the analytical grad solutions has been successfully determined via potentiometric titration using the MCPEs as indicator electrodes, with a potential jump amount to 498 mV. The optimized sensors (I and II) were used successfully for direct and indirect determination of DMTB species in different spiked real water samples with satisfactory results. The obtained results with MCPEs are compared with those obtained with two phase titration method with satisfactory agreement.

**Keywords:** 1-dodecyl-5-methyl-1H-benzo[d][1,2,3]triazol-1-ium bromide surfactant; Modified carbon paste sensor; Different water samples.

### 1. INTRODUCTION

Surfactants represent one of the most common pollutants for both natural and technogenic waters [1,2]. Surfactants contain two parts a hydrophilic polar head (that determines its application) and a lipophilic part (consisting of hydrocarbonated chains usually with a (CH<sub>2</sub>)<sub>n</sub> motif). It is quite

usual to find those anions in environments such as lakes and rivers as the result of their anthropogenic origin and extensive use. They can cause serious environmental problems that can mainly attributed to foam formation and because they act as transfer system of other pollutants (petroleum products, oils, pesticides and organochlorine compounds) [3-8].

Therefore, it was important to determine the concentration of these surfactants accurately in various samples. The two-phase titration method has usually been utilized to determined cationic and anionic surfactants [5,9-11]. Unfortunately, this technique suffer from numerous number of drawbacks, e.g. the formation of an emulsion during titration (risks of errors in visual end-point detection), numerous matrix interferences and the use of a chlorinated solvent [12]. Also, the procedure was not efficient for surfactants having a short carbon chain length and needs long time (time consuming). Several methods were utilized for determination of surfactants in environmental samples using ion-sensitive field effect transistor (ISFET) devices, flow injection analysis [13,14], spectrophotometry [15,16], calorimetry [17], spectrofluorimetry [18,19] and optical sensors [20]. Most of the limitations and disadvantages of these procedures can be overcome by using ion-selective electrodes in direct potentiometry or as indicator electrodes in potentiometric surfactant titration [4-7,9,10,21,22].

Ion selective electrodes (ISEs) were simple analytical devices used for measuring monomer concentration of surfactant in solution and they described as useful tool for monitoring specific species in environmental samples such as soil, waste water, etc [1,5,23-25].

Ion-selective electrodes were initially based on a membrane containing an active ionic species named (ionophore) and a suitable plasticizer [21,22,26-30]. The ionophore (active species) would consider as the form of a large charged hydrophobic molecule of opposite charge to that of the surfactant under investigation. Under such circumstances, the plasticizers add a liquid-like state to the membrane, facilitating the entrance of the surfactant to the membrane and interact ionically with the ionophore in an attempt to form a complex. The titration of all surfactant were based on antagonist reaction, where an ionic surfactant reacted with an oppositely charged ion (mainly surfactant, too) forming a water insoluble ion-pair [25,31,32].

This article describes the preparation and potentiometric characterization of a novel cationic surfactant electrodes based on using 1,4-bis-(8-mercaptooctyloxy)-benzene (MPB) as the electroactive material and dibutylphthalate (DBP) (sensor I) and *o*-nitrophenyloctylether (*o*-NPOE) (sensor II) as plasticizers. The characteristics and analytical performance of MCPEs such as plasticizers, effect of the interfering cations, pH range, temperature and influence of ionophore content have been investigated.

## 2. EXPERIMENTAL

### 2.1. Reagents

The chemicals used in this study were of analytical reagent grade and doubly distilled water was used to prepare the required solutions. 1-dodecyl-5-methyl-1H-benzo[d][1,2,3]triazol-1-ium bromide (DMTB) was prepared according to the previously published method [33,34]. Sodium tetraphenylborate (NaTPB) was supplied from Sigma-Aldrich. Dibutylphthalate (DBP) supplied from

Merck was used for the preparation of the sensors. Tricresylphosphate (TCP), dioctylphthalate (DOP), dioctylsebacate (DOS) and *o*-nitrophenyloctylether (*o*-NPOE) plasticizers used in this study were supplied from Alfa Aesar, Sigma, Merck and Fluka, respectively. Graphite powder (synthetic 1–2  $\mu\text{m}$ ) supplied from Aldrich was used for the preparation of different electrodes used in this study. Chloride salts of aluminum, barium, calcium, cadmium, cobalt, ferrous, ferric, lead, mercury, magnesium, nickel, potassium, sodium and strontium are tested as interfering ions.

### 2.1.1. Samples

They included formation water (Amry deep (10); sample 1 and Amry deep (12); sample 2 from Western Desert, Agiba Petroleum Company, Egypt), tap water (sample 3) and river water sample (sample 4), Mit Ghamr, Dakahlia, Egypt).

### 2.2. Apparatus

Jenway 3505 pH-meter was used for laboratory potential measurements. The reference electrode used is silver-silver chloride double-junction (supplied from Metrohm 6.0726.100) in conjugation with different ion selective electrodes used in this study. Thermo-Orion, model Orion 3 stars (USA) was used for pH measurements. All glasswares used were washed carefully with distilled water Prior to analysis, and dried in the oven before use.

### 2.3. Procedures

#### 2.3.1. Preparation of modified carbon paste electrodes:

5-15 mg of 1,4-bis-(8-mercaptooctyloxy)-benzene (MPB) ionophore, 250 mg carbon powder and 100 $\mu\text{l}$  of DOP, TCP, DBP, DOS or *o*-NPOE plasticizer are the matrices compositions for preparation of modified carbon paste electrodes used to determine the analyte under study. The matrix was thoroughly mixed in the mortar and the electrode body was filled with the resulted paste [22,26-29,35-39]. The fabricated electrodes were used directly for potentiometric measurements without preconditioning and a fresh surface of the paste was obtained by squeezing more out. The surplus paste was wiped out and the freshly exposed surface has shiny appearance when polished on a paper.

#### 2.3.2. Sensor calibration

The calibration of the newly prepared MCPEs was done by immersion in conjunction with a reference electrode in a 25-mL beaker containing a 2.0 mL of acetate buffer solution of pH 4. Then 10 ml aliquot of 1-dodecyl-5-methyl-1H-benzo[d][1,2,3]triazol-1-ium bromide (DMTB) solution of concentration ranging from  $1 \times 10^{-7}$  to  $1 \times 10^{-2}$  mol L<sup>-1</sup> was added with continuous stirring. The potential was recorded after stabilization to  $\pm 0.1$  mV. By plotting the recorded potentials as a function of  $-\log [\text{DMT}^+]$ , the calibration graphs were constructed and the resulting graph was used for subsequent determination of unknown DMTB surfactant concentration [22,35,36,40,41].

### 2.3.3. Solutions

#### 2.3.3.1. Surfactant solution

The adsorption of DMTB surfactant on the inner surface of vessels during its preparation was eliminated according to the previously reported methods [4-7,9,10].

#### 2.3.3.2. Sodium tetraphenylborate solution

Sodium tetraphenylborate (NaTPB) solution (ca.  $10^{-2}$  mol L<sup>-1</sup>) was prepared by dissolving the accurately weighed amount in bidistilled water and completed to the desired volume with water. The resulting solution was standardized potentiometrically using standard thallium (I) nitrate solution ( $10^{-2}$  mol L<sup>-1</sup>) [6,9].

#### 2.3.4. Determination of cationic surfactant in water sample

Transfer 2 mL of water sample to a 10 mL beaker containing 1 mL acetate buffer of pH 4.0 and definite concentration of the DMTB surfactant was added. The concentration of DMTB surfactant was estimated via potentiometric titration with NaTPB solution using MCPE electrode in addition to the two-phase titration method [4,6,9,10].

#### 2.3.5. Two phase titration method

This has been described in detail elsewhere [42,43]. It consists in the titration of an aqueous sample mixed with chloroform phase containing a mixed indicator of dimidium bromide and Acid Blue 1 (Patent Blue VF CI 42045). The titrant is standard aqueous solution of the appropriate counter-ion surfactant, either SDS. This displaces the appropriate indicator ion, shifting the organic phase color between pink and blue with a grey end-point.

## 3. RESULTS AND DISCUSSION

### 3.1. Electrodes linearity

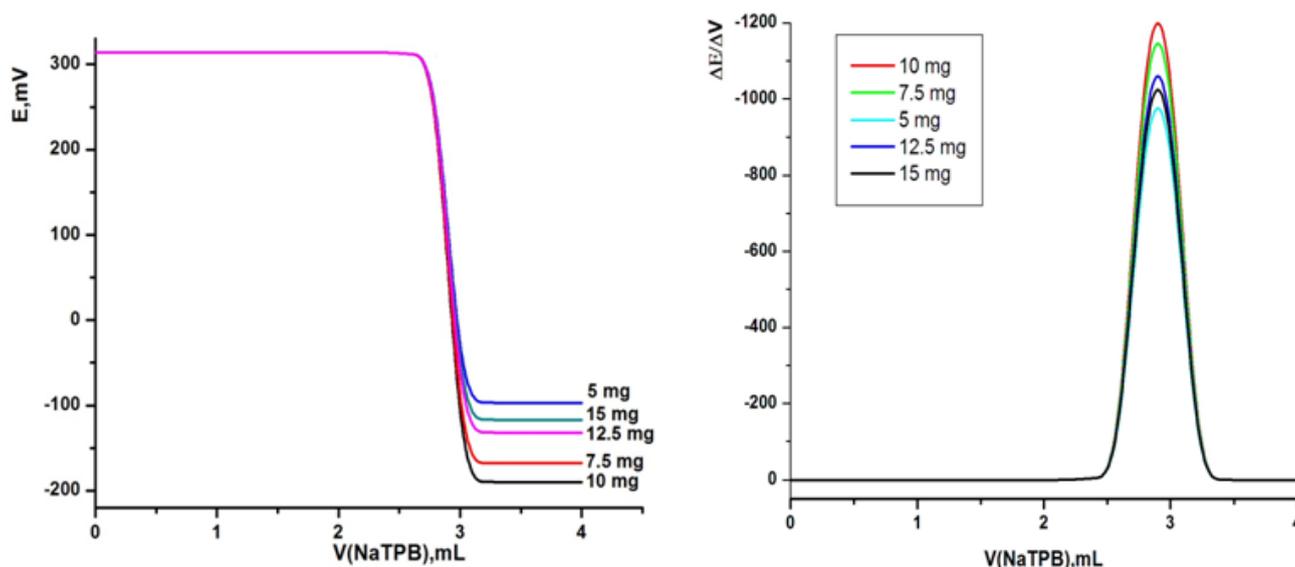
Two MCPEs with dibutylphthalate (DBP) (electrode I) and *o*-nitrophenyloctylether (*o*-NPOE) (electrode II) as plasticizers were prepared. The potential response of modified carbon paste electrodes (I and II) for determination of 1-dodecyl-5-methyl-1H-benzo[d][1,2,3]triazol-1-ium bromide (DMTB) solutions was studied. The electrodes were found to have a linear response in the concentration range of  $2.6 \times 10^{-7}$  -  $1 \times 10^{-2}$  and  $1.9 \times 10^{-7}$  -  $1 \times 10^{-2}$  mol L<sup>-1</sup> with a Nernstian slope of  $59.33 \pm 0.27$  and  $58.59 \pm 1.31$  mV decade<sup>-1</sup> for electrodes (I) and (II), respectively. The limit of detection (LOD) is found to be  $2.6 \times 10^{-7}$  and  $1.4 \times 10^{-7}$  mol L<sup>-1</sup> for electrodes (I) and (II), respectively. The analytical parameters of the modified carbon paste electrodes (I and II) such as working concentration range, limit of detection, the response time, the slope of the linear section of the response curve were determined from the results of these measurements and are summarized in Table 1.

**Table 1.** Response characteristics of modified CPE sensors (I and II).

Parameter	CPE	
	Sensor (I)	Sensor (II)
Slope (mV decade <sup>-1</sup> )	59.33 ± 0.27	58.59 ± 1.31
Concentration range (mol L <sup>-1</sup> )	2.6 × 10 <sup>-7</sup> – 1.0 × 10 <sup>-2</sup>	1.4 × 10 <sup>-7</sup> – 1.0 × 10 <sup>-2</sup>
Correlation coefficient, r	0.998	0.997
Limit of detection (mol L <sup>-1</sup> )	2.6 × 10 <sup>-7</sup>	1.4 × 10 <sup>-7</sup>
Working pH range	2.5 - 9.0	2.0 - 9.0
Life time (days)	58	63
Response time (sec)	7	6
Standard deviation (SD)*	0.087	0.045
Relative standard deviation (RSD%)*	1.263	1.003
Working temperature range (°C)	10-60	10-60
Accuracy (%)	98.88	99.01

\* Average of four determination

### 3.2. Effect of ionophore content

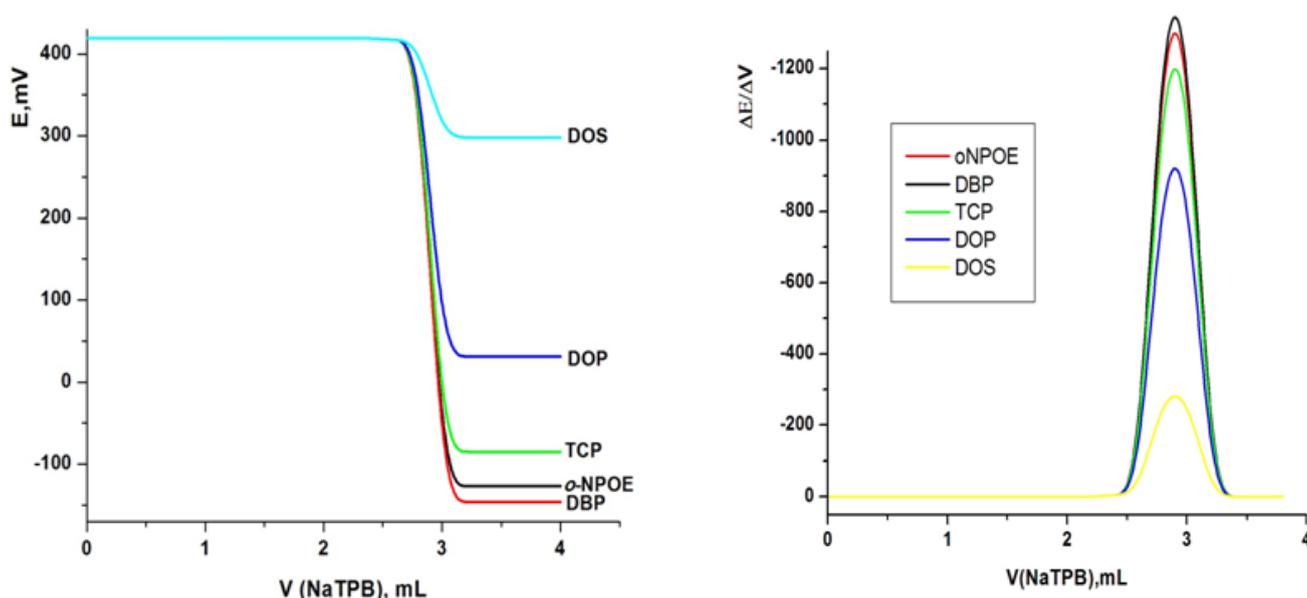
**Figure 1.** Effect of ionophore contents on MCPE sensors using TCP plasticizer.

The main ingredient of carbon paste electrodes is the incorporated carrier i.e. the ionophore that defines the selectivity for a certain ion selective sensor by forming a complex with the ion of interest. In preliminary experiments, modified CPE electrodes containing plasticizer were prepared incorporating 1,4-bis-(8-mercaptooctyloxy)-benzene (MPB) ionophore as a neutral carrier. Thus, to

achieve this goal, five modified carbon paste electrodes were prepared in order to select the best electrode content that have the best response characteristics. Different proportions of MPB ionophore were varied as 5, 7.5, 10, 12.5 and 15 mg (w/w %). The potentiometric titration curves for each electrode are represented graphically as given in Figure 1. It is obvious from this figure that the resulting potential breaks at the end point were found to be 405, 476, 498, 440 and 425 mV mL<sup>-1</sup>. These electrodes give sharp and reproducible inflection at the end point of 498 mV mL<sup>-1</sup>. It is clear from these results that the highest potential break at the end point was obtained using 10 mg of MPB ionophore. The increase or decrease in the amount of ionophore content over or below 10 mg, leads to a decrease in the total potential change as shown in Figure 1. The presence of a lipophilic anion in cation-selective paste electrodes not only diminishes the ohmic resistance but also enhances the response behavior, selectivity but also increases the sensitivity of the paste electrodes even where the extraction capability is poor.

### 3.3. Effect of the plasticizer type

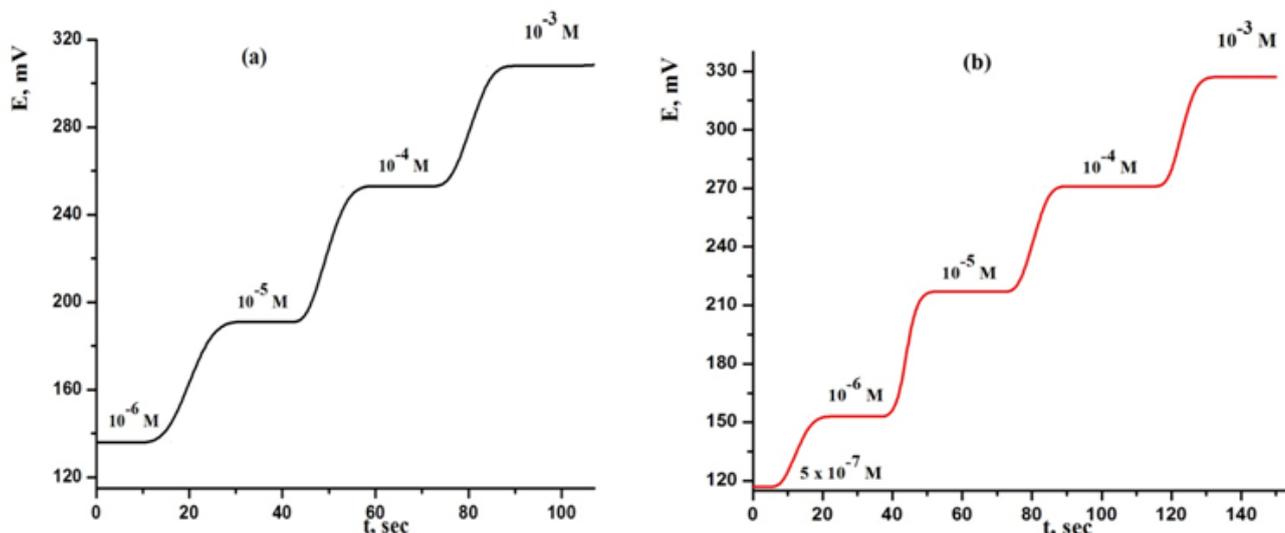
It is well-known that the nature of ionophore used and the properties of plasticizers affect significantly the sensitivity and the linearity of the ion-selective electrodes. Thus, the effect of the plasticizer type on the potential response of the carbon paste electrodes was investigated by using five different type of plasticizer and the data obtained are illustrated in Figure 2. The effect of the plasticizer on the performance of MCPEs had been studied. It is found that the electrode plasticized with DBP was compared with that with *o*-NPOE, DOP, DOS or TCP. The highest total potential change ( mV) and the highest potential break at the end point (559 mV) are obtained using DBP plasticizer which may be attributed to the high extractability of the MPB ionophore compared with other tested plasticizers [22,35-37] (Figure 2).



**Figure 2.** Effect of plasticizer type on the performance of MCPEs.

### 3.4. Response time

For any ion selective electrode, the evaluation of the dynamic response time is one of the most important factors. Therefore, the dynamic response time of the proposed potentiometric sensors (I and II) was measured by successive changing the concentration of the test solution from  $1.0 \times 10^{-7}$  to  $1.0 \times 10^{-2}$  mol L<sup>-1</sup>. The resulting data depicted in Figure 3, showed the time needed to reach a potential within  $\pm 1.0$  mV of the final equilibrium value after successive immersion of the electrodes (I and II) in a series of surfactant solutions, each having a 10-fold difference. According to IUPAC definition, the response time of the modified carbon paste electrode (I and II) were evaluated by measuring the time required to achieve a 90% value of steady potential for a DMTB solution. The modified carbon paste electrodes (I and II) were found to have a response time of 7 and 6s, respectively. These fast response times can be explained taking into account that these modified carbon paste electrodes (I and II) contain carbon particles surrounded by a very thin film of DBP and *o*-NPOE plasticizers and acting as a conductor in addition to the absence of the internal reference solution. The fast and stable potential reading is reflected on the short time needed to achieve complete titration process which is found to be only about 2-3 min [21,27-30].

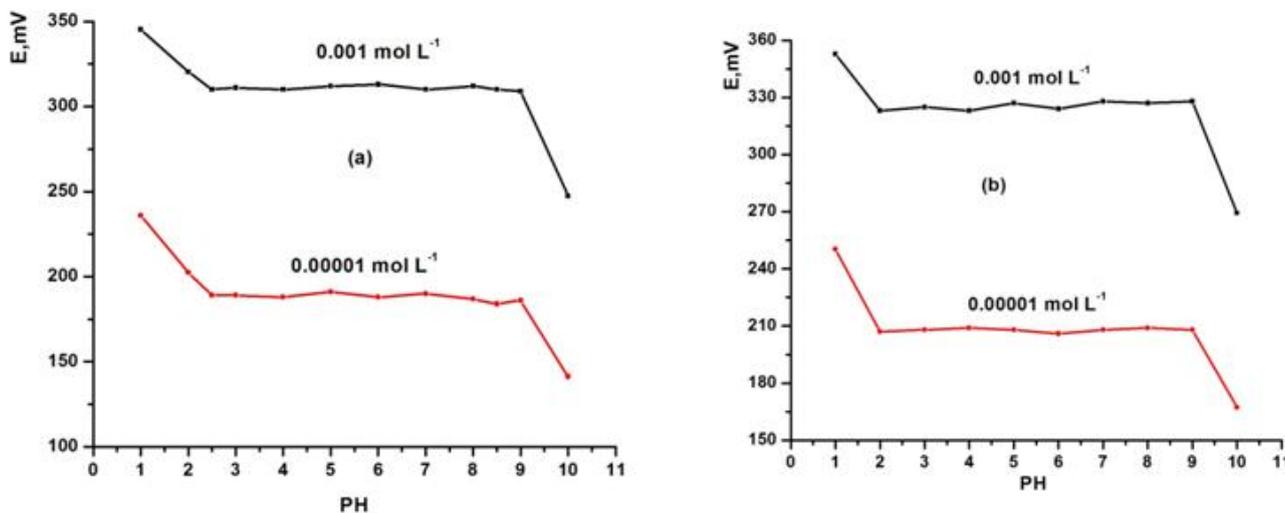


**Figure 3.** Dynamic response time of MCPEs (a) Sensor (I) and (b) Sensor (II).

### 3.5. Effect of pH

The effect of pH of the test solution on the response of the MCPE electrodes (I and II) was studied using two DMTB concentrations ( $1.0 \times 10^{-3}$  and  $1.0 \times 10^{-5}$  mol L<sup>-1</sup>). The pH was adjusted with 0.1M hydrochloric acid and 0.1M sodium hydroxide solutions. The potential readings of the modified carbon paste electrodes (I and II) were recorded as a function of pH. The results are represented graphically as shown in Figure 4. It is obvious from Figure 4 that, the potential readings remain almost constant over the pH range of 2.5-9.0 and 2.0-9.0 for modified carbon paste electrodes (I and II), respectively. This pH range can be taken as the working pH range of the sensors (I and II) assembly. At pH lower than 2.0, the interference of H<sup>+</sup> ions is more obvious which can be attributed to the high

rate of diffusion of H<sup>+</sup> ions from sample solution to membrane matrix (extract H<sup>+</sup> ion) where they interact with carrier and its protonation takes place resulting in decreased selectivity of the modified carbon paste electrodes (I and II) to DMT<sup>+</sup> cation. In this case, the the modified carbon paste electrodes (I and II) then respond to hydrogen ions more than DMTB surfactant. At pH higher than 9.0, the decrease in the potential readings and hence deviation in the electrodes response may be attributed to the formation of some hydroxyl complexes of DMT<sup>+</sup> in solution [6,38,39].



**Figure 4.** Effect of pH of the test solution on MCPEs (a) Sensor (I) and (b) Sensor (II)

3.6. Potentiometric selectivity

**Table 2.** Potentiometric selectivity coefficients of some interfering ions using MCPEs (Sensors I and II)

Interfering ions	$-logK_{A,B}^{SSM}$		Interfering ions	$-logK_{A,B}^{SSM}$	
	Sensor (I)	Sensor (II)		Sensor (I)	Sensor (II)
Ca <sup>2+</sup>	5.90	5.88	SO <sub>4</sub> <sup>-2</sup>	5.22	5.39
Co <sup>2+</sup>	5.01	5.37	Cl <sup>-</sup>	5.10	5.26
Fe <sup>2+</sup>	4.51	4.34	Na <sup>+</sup>	5.77	5.92
Cd <sup>2+</sup>	5.23	5.63	K <sup>+</sup>	5.56	5.68
Sr <sup>2+</sup>	4.97	5.11	CTAB	1.28	1.33
Ni <sup>2+</sup>	5.74	5.84	CPC	0.99	1.02
Hg <sup>2+</sup>	4.27	4.40	Al <sup>3+</sup>	4.69	4.97
Mg <sup>2+</sup>	5.82	5.91	Fe <sup>3+</sup>	3.21	3.54
Pb <sup>2+</sup>	3.25	3.64	Ba <sup>2+</sup>	4.05	4.33

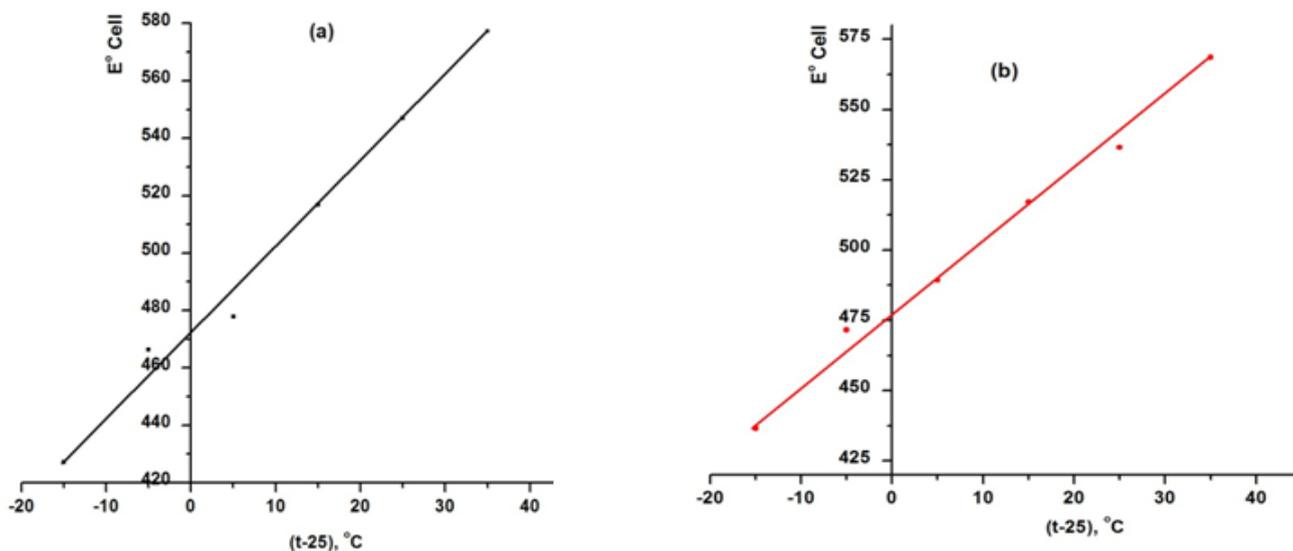
The selectivity coefficient is one of the foremost important characteristics of ISEs. It gives informations about the ability of the sensing paste in discriminating the primary ion (DMTB surfactant) against other interfering ions of the same charge sign [5,7]. The selectivity coefficients of the modified carbon paste electrodes (I and II) to cetyltrimethylammonium bromide (CTAB), cetylpyridinium chloride (CPC) and a number of inorganic cations were determined. A lot of methods for selectivity coefficient determination were known and the separate solution method (SSM) (one of the methods recommended by IUPAC) was used in this study. The potential of the cell (comprising the modified carbon paste electrodes (I and II) and a reference electrode) was measured for solutions containing  $\text{DMT}^+$  cation ( $1.0 \times 10^{-3} \text{ mol L}^{-1}$ ) and interfering ion of the same concentration ( $1.0 \times 10^{-3} \text{ mol L}^{-1}$ ). The obtained values of selectivity coefficients were presented in Table 2. For determining the selectivity coefficients of the modified carbon paste electrode (I and II) by this method, the following relationship was applied:

$$\text{Log}K^{\text{pot}}_{A,B} = ((E_B - E_A)/S) + (1 - (Z_A/Z_B)) \log a_A$$

where  $K^{\text{pot}}_{A,B}$  is the potentiometric selectivity coefficient,  $E_A$  is the potential measured in  $1.0 \times 10^{-3} \text{ mol L}^{-1}$  DMTB surfactant,  $E_B$  the potential measured in  $1.0 \times 10^{-3} \text{ mol L}^{-1}$  of the interfering compound (B),  $S$  the slope of the calibration plot,  $a_A$  the activity of DMTB surfactant, and  $Z_A$  and  $Z_B$  are the charges on DMTB surfactant and the interfering ion, respectively [28,30]. It is clear from the data given in Table 2 that the inorganic cations have no interference effect on the response of the modified carbon paste electrodes (I and II) for DMTB surfactant. In addition, CTAB and CPC surfactants seriously interfere with the DMTB surfactant under study.

### 3.7. Effect of temperature

According to the Nernst equation, the electrode potential is a temperature dependence factor. To study the thermal stability of the modified carbon paste electrodes (I and II), calibration curves were constructed at different temperatures ranged from 10 to 60 °C and the potential of  $1.0 \times 10^{-7}$ - $1.0 \times 10^{-2} \text{ mol L}^{-1}$  DMTB surfactant solutions was determined.



**Figure 5.** Effect of temperature on the performance of MCPEs (a) sensor (I) and (b) sensor (II).

The isothermal coefficients ( $dE^{\circ}/dt$ ) of the modified carbon paste electrodes (I and II) were determined by measuring the standard electrode potentials ( $E^{\circ}$ ) against normal hydrogen electrode, at different temperatures (10 to 60 °C) which obtained from the calibration graphs as the intercepts at  $p[\text{DMTB}] = 0$  (after subtracting the values of the standard electrode potential of the silver-silver chloride double-junction reference electrode at these temperatures). They plotted versus  $(t-25)$ , where  $t$  was the temperature of the test solution in °C (Figure 5). A straight-line plot was obtained according to Antropov's equation:

$$E^{\circ} = E^{\circ}(25) + (dE^{\circ}/dt)(t-25)$$

where  $E^{\circ}(25)$  was the standard electrode potential at 25 °C. The slope of the straight-line obtained represented the isothermal coefficient of the modified carbon paste electrodes (I and II). The isothermal coefficients were found to be 0.00295 and 0.00241 V/°C for sensors I and II, respectively. These values indicated that the electrodes had fairly high thermal stability within the investigated temperature range and can be used up to 60 °C without noticeable deviation from the Nernstian behavior [44].

### 3.8. Analytical applications

The applicability of the DMTB-MCPE (Sensors I and II) for determining the concentration of DMTB surfactant in four real spiked water samples was evaluated. 2.0 mL from each water sample was taken and diluted with distilled water in a 10.0 mL volumetric flask. The sample was prepared according to the experimental part and the potential of the solutions was measured by the proposed potentiometric sensors (I and II). The proposed electrodes were successfully employed for the assay of DMTB surfactant in spiked real water samples. The results of the modified CPE (Sensors I and II) were compared with the two phase titration method. The data obtained were shown in Table (3) [4-7,9,10]. The data given clearly indicated satisfactory agreement between the surfactant contents in different real spiked water samples determined by the proposed potentiometric sensors (I and II) and the two phase titration method. The low values of standard deviation and relative standard deviation in comparison with the two phase titration method reflected the high accuracy and precision of the proposed potentiometric method. Also using of the portable system proposed in the present work allowed analysis of DMTB surfactant in sample field rather than transferring to the laboratory.

**Table 3.** Potentiometric determination of DMTB in spiked real water samples using MCPEs (sensors I and II).

Samples	MCPE Found (mg mL <sup>-1</sup> )									
	Sensor (I)				Sensor (II)			Two Phase Titration Method		
	Taken	Found	SD <sup>a</sup>	RSD%	Found	SD	RSD%	Found	SD	RSD%
1	0.20	0.193	0.971	1.107	0.197	0.384	1.001	0.190	0.996	2.038
2	0.25	0.246	0.927	1.130	0.248	0.273	1.009	0.245	1.005	2.001

3	0.15	0.148	0.105	0.994	0.150	0.096	0.389	0.149	0.099	0.977
4	0.20	0.196	0.327	1.038	0.199	0.039	0.957	0.195	0.973	1.093

<sup>a</sup> Average of five determination.

#### 4. CONCLUSION

Based on the electrode performance studies, 1,4-bis-(8-mercaptooctyloxy)-benzene (MPB) ionophore was selected as a suitable sensing material in fabrication of modified carbon paste sensors for potentiometric determination of DMTB in pure and in real spiked water samples. A wide linear range of concentration ranged from  $2.6 \times 10^{-7}$  to  $1.0 \times 10^{-2}$  and  $1.4 \times 10^{-7}$  to  $1.0 \times 10^{-2}$  mol L<sup>-1</sup>, low detection limit of  $2.6 \times 10^{-7}$  and  $1.4 \times 10^{-7}$  mol L<sup>-1</sup> and fast response time of 7 and 6s for modified carbon paste sensors (I) and (II), respectively, were reported in this study. The electrodes were successfully applied as end point indicator electrodes in potentiometric titration of DMTB in pure and in real spiked water samples.

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