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Batch and Flow-Injection Analysis of Lauryl Sulfate in Industrial Products and Wastes Using Membrane Sensors Based on Methyltrioctylammonium Chloride

Hisham S. M. Abd-Rabboh[1,2,*]

[1] Chemistry Department, Faculty of Science, King Khalid University, PO Box 9004, Abha 61413, Saudi Arabia
[2] Department of Chemistry, Faculty of Science, Ain Shams University, Abbassia, Cairo 11566, Egypt
[*]E-mail: hasalah@hotmail.com

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Liquid polymeric membrane sensors based on the hydrophobic cationic surfactant methyltrioctayammonium chloride (MTOAC) are described herein for batch and flow injection analysis of sodium lauryl sulfate (SLS) surfactant. PVC-based membrane sensors were prepared using different plasticizers in two different electrode designs: conventional polymeric membrane discs for batch measurements and flow-through half cell module for flow-injection analysis. o-nitrophenyloctyl ether plasticized sensors exhibited the most distinguished response characteristics among other sensors in 10^{-2} M phosphate buffer solution at pH =7, with a lower detection limit of $3.97x10^{-7}$ M, a linear dynamic range of $9.33x10^{-7}$ – $5.31x10^{-3}$ M, a slope of -59.4 mV/concentration decade, a correlation coefficient of 0.999 and a response time of 60 s. The sensors were selective towards SLS over other common anions and exhibited a long life time (more than 2.5 months) with a constant detection limit, an enhanced linear range, and a slight increase in its slope. The fabricated sensors were efficiently used in the determination of SLS concentrations in a wide range of samples including powder detergents, tooth paste, liquid hand wash, and shampoo, and wastewater.

Keywords: Potentiometry; flow-injection analysis; ion-selective electrode; sodium lauryl sulfate; surfactant analysis

1. INTRODUCTION

Sodium lauryl sulfate (SLS) is one of the widely used surfactants in a variety of industrial applications that include cosmetics, body care products, toothpastes, lotions, and formulated detergents [1]. Such a family of compounds is known to be safe and non-toxic for human daily use. However, through discharge to the aquatic systems, most surfactant species accumulate in the environment and some transforms into a series of toxic analogues [2-4]. A vast number of analytical methods and procedures were supplied for the accurate and low-level determination of a large number of anionic, cationic, as well as non-ionic surfactants. A series of organic dye reagents were used for the spectrophotometric analysis of sodium dodecyl sulfate in water and wastewater samples [5-12]. Spectrophotometry introduces the privilege of very good detection limits, but needs tedious sample preparation and treatment steps. Other techniques used for surfactant analysis include chromatography [13, 14], mass spectroscopy [15], fluorimetry [16], as well as solid phase extraction [17]. Most of these methods require advanced and expensive equipment, with the operation by high-quality, well-trained personnel.

Electrochemical analyses introduce simple, low-cost, and fast analytical tools for the direct determination of surfactants in complex sample matrices, as complicated as industrial wastewaters [18, 19]. Within the last few decades, a number of electrochemical sensors were introduced for the selective detection of anionic and cationic surfactants [20-26]. Potentiometric ion-selective electrodes in different formats were used for surfactant analyses using standard calibration, standard addition, titration end point detection, as well as flow-injection analysis systems [27-32].

In this article, a new polymeric membrane-based lauryl sulfate chemical sensor is introduced. The sensor is formulated with methyltrioctylammoniumchoride as the sensing element in PVC-plasticized membranes. The sensor detects SLS selectively, sensitively and directly in a variety of samples, without any need of sample pre-treatment steps.

2. EXPERIMENTAL

2.1. Materials and equipment:

Poly(vinyl chloride) (PVC, selsctophore grade), Bis-(ethyl hexyl) sebacate (DOS, selectophore grade), dioctyl phthalate (DOP, ≥ 99.5%), tridodecylmethyl-ammonium chloride (TDMAC, selectophore grade), sodium loryl sulfate (SLS) were all obtained from Sigma-Aldrich (www.sigmaaldrich.com), and methyltrioctylammonium chloride (MTOAC, ≥ 97%) was a product of Fluka (www.sigmaaldrich.com). Other chemicals used within this article were purchased with analytical grade, and de-ionized water was used for all solution preparations.

Potential measurements were recorded at room temperature using a custom-made high-impedance multichannel data-acquisition system equipped with an ADC-16 computer interface operated with Pico-log data logging software version 5.22.6 (Pico Tech, UK), and a sure-flow double-junction $Ag/AgCl/Cl^-$ reference electrode model 900200 (Thermo-Orion, USA). pH adjustment and buffer preparation were carried out at 25^oC using a 720/SA pH/mV (Thermo-Orion, USA) and a Ross Sure-flow junction combination pH electrode model 8172BNWP (Thermo-Orion, USA). Solution delivery in flow-injection analysis (FIA) was supplied by a four-channel peristaltic pump, model ISM843C (Ismatec, USA) with PVC Tygon tubes of different diameters. Sample injection in FIA experiments was performed using a 6-way Rheodyne injection valve, model 9725 (Rheodyne, USA), supplied with sample injection loops of different volumes.

2.2. Polymeric membrane preparation and sensor assembly:

Liquid polymeric membranes were prepared by mixing various amounts of membrane cocktail ingredients, including polymer, plasticizer, sensing element, and solvent. The exact quantities of membrane film solutions are tabulated in table 1. Membrane cocktail solutions were vortex-shaken for 5 minutes in order to dissolve all the components in THF. After dissolution, membrane solutions were powered into flat-bottom Teflon cups, 2.2 cm in diameter. The cups were covered with filter paper circles for 12 hours to allow slow solvent evaporation. After that, the formed membrane films were pealed-off the cup and stored in a dry container.

In order to fabricate the sensors, 6 mm membrane disks were cut-off the mother membrane films and glued to 1 cm long, flat-cut, cylindrical PVC Masterflex tubing (3.1 mm ID and 6 mm OD). A 3.5 mm diameter, 5 cm long plastic tube was then inserted into the open end of the Tygon tube, and the tube was filled with the sensor filling solution, 10^{-2} M MTOAC/10^{-2} M KCl. A silver wire coated with silver chloride (Ag/AgCl) was then inserted into the sensor's filling solution to act as the inner reference electrode.

Table 1. Liquid membrane film Compositions

Membrane No.	**Polymer, mg**	**Plasticizer, mg**			**Sensing element, mg**		**Solvent, mL**
	PVC	**o-NPOE**	**DOP**	**DOS**	**TDMAC**	**MTOAC**	**THF**
1	66	132	0	0	2	0	2
2	66	0	132	0	2	0	2
3	66	0	0	132	2	0	2
4	66	132	0	0	0	2	2
5	66	0	132	0	0	2	2
6	66	0	0	132	0	2	2

The assembled working electrode, together with the double-junction reference electrode, was connected to the potentiometric measuring system, and when inserted into the sample solution, the following potentiometric assembly is established:

Ag/AgCl/Cl$^-$ internal reference│fillingsolution│polymericmembrane‖testsolution‖liquid junctions│ Ag/AgCl/Cl$^-$ external reference

2.3. Sensor calibration and pH effect:

10 mL aqueous solution (water or buffer) were transferred into a 50 mL glass beaker, stirred at 500 rpm at room temperature, then the working and reference electrodes were immersed into the solution. Immediately, potential signals were recorded every 30 seconds all over the experimental time. A steady state potential equilibrium was obtained after 3 minutes of continuous stirring and

signal recording, then 1 mL of 10^{-7} M standard SLS solution was delivered into the beaker. Stirring was continued for another 3 minutes, with signal recording every 30 s, then the next SLS standard solution (1 mL of 10^{-6} M solution) was added to the working solution. The process was repeated every 3 minutes for the SLS standard solutions covering the concentration range10^{-7} - 10^{-2} M. Finally, the potential readings were plotted against the logarithm of the SLS concentrations in order to obtain calibration graphs.

To study the effect of pH of the solution on the electrode potential response towards SLS, 20 mL of one SLS standard solution, belonging to the linear part of the calibration relation, were transferred into a 50 mL glass beaker. The working and reference electrodes, together with a pH combined glass electrode, were immersed in the stirred solution, and potential and pH readings were recorded immediately. After reaching the steady state potential equilibrium in 3 minutes, few drops of concentrated sodium hydroxide solution were added to raise the solution pH up to 1 pH unit, and potential reading was recorded. The process was repeated several times until the pH reaches a value of about 12. The same experiment was repeated with a fresh aliquot of SLS standard solution but with replacing NaOH with hydrochloric acid to lower the solution pH. A graph was then plotted between the solution pH and the potential readings.

2.4. Sensor selectivity coefficients:

A study on the selectivity coefficients of the assembled sensors for SLS against other common anionic species that may be present in the assayed sample types (industrial products and industrial wastes) was performed using the well-known matched potential method [33]. A 10 mL portion of a selected SLS standard solution, belonging to the linear part of the calibration relation, were transferred into a 50 mL glass beaker, and the potential reading of the stirred solution was recorded in 3 minutes. In a separate experiment, the SLS standard solution of the previous experiment was substituted with 10 mL water and potential readings were recorded. Increased concentrations of the interfering ion were then added until the cell potential matched the recorded potential for the SLS standard solution. Values of selectivity coefficient were then calculated from the following equation:

$$k_{\mathrm{ij}}^{\mathrm{MPM}} = \frac{\mathrm{a_i}}{\mathrm{a_j}}$$

where:$k_{\mathrm{ij}}^{\mathrm{MPM}}$ represents the matched potential selectivity coefficient,

$\mathrm{a_i}$ is the effective concentration of the analyte in M,

$\mathrm{a_j}$ is the effective concentration of the interfering ion that generated the same potential signal of $\mathrm{a_i}$.

3. RESULTS AND DISCUSSION

3.1. Performance characteristics of SLS sensors in batch and flow-injection setups:

Membrane sensors were fabricated using two different membrane formats: circular disc (conventional) and tubular flow-through half cells. Conventional membrane sensors, formulated according to table 1 (sensors 1-6), were calibrated initially in water. The performance (response) characteristics of sensors 1-6 are represented in table 2. It is clear from the reported data that sensor 4 showed the lowest detection limit ($3.97x10^{-7}$ M), the longest linear range ($9.33x10^{-7}$ – $5.31x10^{-3}$ M), the most acceptable Nernstian slope (-58.79 mV/concentration decade), together with sensor 2, and comparable correlation coefficient and response time with the other sensors. As a result, sensor 4 was selected for performing all subsequent experiments.

Table 2. Performance characteristics of SLS membrane sensors from batch experiments

Performance characteristics	**Sensor 1**	**Sensor 2**	**Sensor 3**	**Sensor 4**	**Sensor 5**	**Sensor 6**
Detection limit, M	$8.12x10^{-7}$	$4.78x10^{-7}$	$9.80x10^{-6}$	$3.97x10^{-7}$	$1.17x10^{-6}$	$1.17x10^{-6}$
Linear range, M	$1.32x10^{-6}$ – $7.59x10^{-4}$	$9.55x10^{-7}$ – $5.31x10^{-3}$	$9.80x10^{-6}$ – $5.31x10^{-3}$	$9.33x10^{-7}$ – $5.31x10^{-3}$	$3.24x10^{-6}$ – $5.31x10^{-3}$	$3.02x10^{-6}$ – $5.30x10^{-3}$
Slope, mV/decade	-60.81	-58.79	-33.18	-58.79	-57.25	-57.16
Correlation coefficient, r^2	0.999	0.999	0.999	0.999	0.999	0.999
Response time, s	60	60	60	60	60	60

Performance characteristics of some previously reported methods for the detection of SLS based on different detection systems, compared with the current assay method using sensor 4, are reported in table 3. It can be seen from the presented values that spectroscopic methods suffer from tedious preparation/pretreatment or extraction steps. On the other hand, chromatographic methods require preconcentration and purification steps, expensive instrumentation, and show narrow application ranges. Most of the reported potentiometric methods acquire relatively high detection limits, some have abnormal high slopes, and others suffer from anionic interferences.

Table 3. Comparison between the performance characteristics of previously reported SLS analysis methods

Method, Regent, and Reference	**Detection limit, M**	**Linear range, M**	**Slope**	**Correlation coefficient, r^2**	**Response time, s**	**Notes**
Sorption-photometry, Crystal violet [5]	$1x10^{-8}$	$3.5x10^{-8}$ – $4.2x10^{-6}$	N/A	0.997	N/A	Tedious, 2-step
Spectrophotometry, methylene blue, and spectrofluorimetry, Rhodamine B [8]	$2x10^{-6}$	$2x10^{-6}$ – $1x10^{-5}$	N/A	N/A	N/A	Extraction then analysis
FIA Spectrophotometry, Malachite green [9]	$6.3x10^{-8}$	$3.5x10^{-7}$ – $1.4x10^{-6}$	N/A	N/A	180	Phase separation, extraction
FIA Spectrophotometry, Various dyes [10]	$3.5x10^{-7}$	$3.5x10^{-6}$ – $6.9x10^{-5}$	N/A	0.988	72	Precipitation then dissolution
Photometric titration, o-Toluidine blue [12]	$1x10^{-4}$	$1x10^{-4}$ – $1.8x10^{-1}$	N/A	N/A	N/A	For high concentrated samples

Extraction then LC-MS, [14]	3.5x10^{-10} – 1.7x10^{-9}	8.7x10^{-8} – 2.2x10^{-7}	N/A	N/A	N/A	Preconcentration, purification, very narrow range
Microextraction – spectrophotometry [17]	2.8x10^{-8}	1x10^{-7} – 1.4x10^{-6}	N/A	0.999	N/A	Limited linear range
Amperometric sensor, Polyethyleneimine [18]	3.1x10^{-6}	3.5x10^{-6} – 1.4x10^{-4}	3.05 mA/decade	0.970	N/A	High detection limit
Potentiometric sensor, Mercurated polystyrene [20]	1x10^{-7}	1x10^{-7} – 1x10^{-3}	-59.0 mV/decade	N/A	30-180	High interference of OH^{-} ions
Clay membrane electrode, Laponite [22]	3x10^{-7}	5x10^{-7} – 155x10^{-2}	-55.0 mV/decade	0.990	1	Heterogeneous membranes
Potentiometric sensor, Imidazolium salt [23]	9x10^{-7}	2x10^{-6} – 5x10^{-3}	-58.9 mV/decade	0.999	120	Relatively high DL
Potentiometric sensor, CTAB [25]	1x10^{-6}	3x10^{-6} – 1x10^{-3}	-52.5 mV/decade	N/A	25	Relatively high DL
Potentiometric sensor, CTA-TPB [26]	5x10^{-6}	5x10^{-6} – 5x10^{-3}	-59.6 mV/decade	---	45	Relatively high DL
Potentiometric sensor, Polyaniline [27]	1x10^{-6}	5x10^{-6} – 4.1x10^{-3}	-59.1 mV/decade	0.990	20	Relatively high DL
Potentiometric sensor, Cetylpyridinium salt [29]	6.3x10^{-7}	1x10^{-6} – 3.8x10^{-3}	-108.0 mV/decade	0.994	15	Abnormal slope at low concentrations
Potentiometris sensor, Teflonized graphite [32]	3.4x10^{-6}	5x10^{-6} – 2x10^{-3}	-64.2 mV/decade	0.999	30	Relatively high DL
Potentiometris sensor, MTOAC	3.97x10-7	9.3x10^{-7} – 5.3x10^{-3}	-58.8 mV/decade	0.999	> 60	Current sensor

3.2. Effect of pH and buffer solution selection:

To study the effect of pH on the response behavior of sensor 4 towards SLS, two standard SLS solutions belonging to the linear response range of the sensor (1x10^{-4} and 1x10^{-5} M) were selected. As can be seen from figure 1, sensor 4 established almost constant responses to the two SLS standard solutions within the pH range of 4.50-7.00. Accordingly, a buffer solution within that range should be selected and used as a working solution for the following experiments.

Figure 2 represents a calibration graph of sensor 4 for SLS using 10^{-2} M phosphate buffer solution (PBS), pH =7. The sensor acquired the following performance characteristics: 7.4x10^{-7} M detection limit, 9.33x10^{-7} – 5.31x10^{-3} M linear range, -59.43 mV/concentration decade slope, 0.999 correlation coefficient, and 60 s response time. Compared with the sensor’s performance in water, it showed the same linear range, correlation coefficient, and response time. On the other hand, slight acceptable increases in both detection limit and slope are noticed. As a result, 10^{-2} M phosphate buffer solution (PBS), pH =7, is selected as a working solution for SLS determination.

The second membrane format, tubular flow-through half cell, was fabricated as described earlier [31]. Figure 3 illustrates a calibration graph and a flow-injection analysis (FIA) chart for sensor 4 towards SLS standard solutions in PBS buffer solution. The detection limit of the sensor appeared to increase a little, compared with the conventional sensors, up to 8.3x10^{-7} M, and the linear range was slightly shifted to higher concentrations (1x10^{-6} – 5x10^{-3} M), which is expected for FIA analysis, where the membrane doesn’t have enough time to reach the steady state potential equilibrium. Still both of the slope (-54.79 mV/concentration decade) and correlation coefficient (r^2 = 0.9959) were not affected much. Apparently, the calculated performance characteristics of the FIA calibration experiment allow the determination of SLS in the types of samples under investigation.

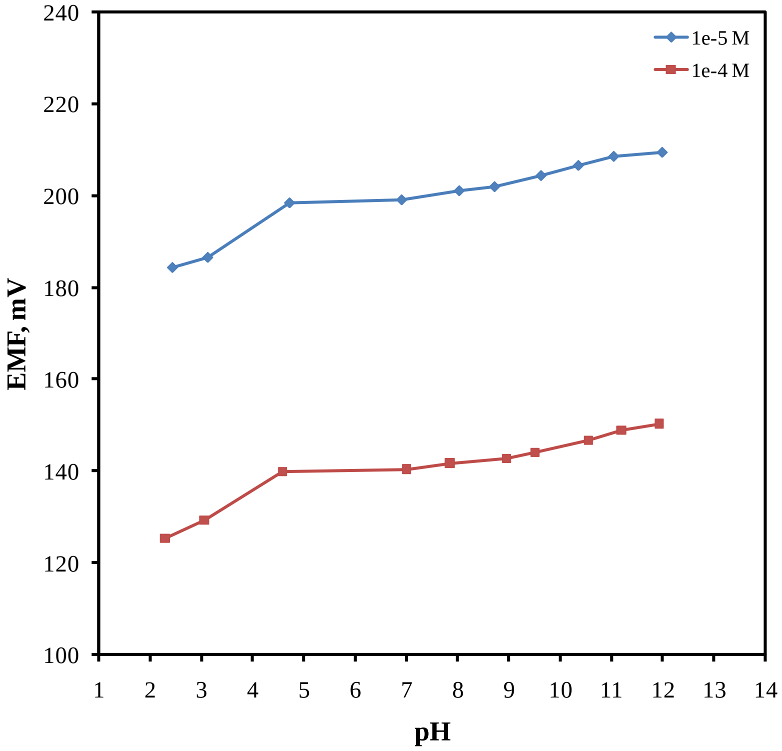

Figure 1. Effect of changing the solution pH on the potentiometric response of sensor 4

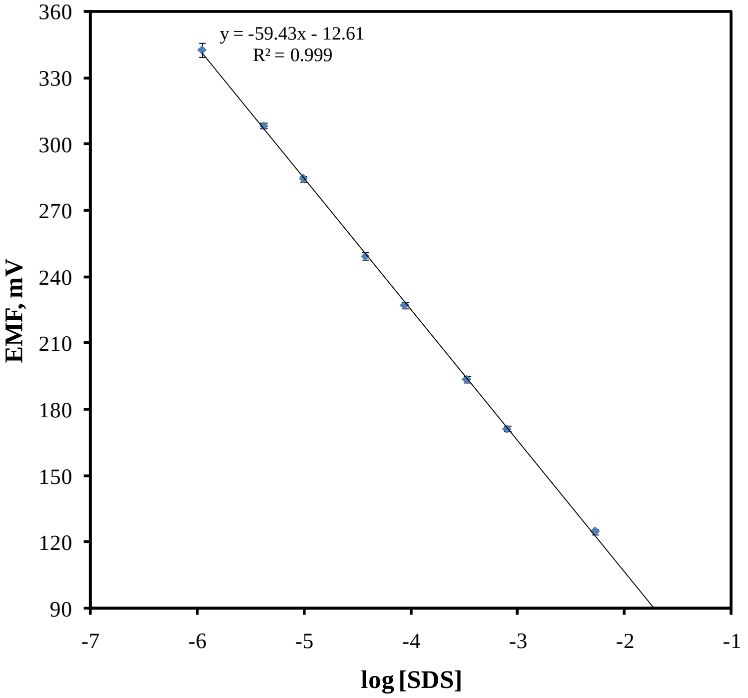

Figure 2. Calibration curve of sensor 4 for SLS in 10^{-2} M PBS buffer, pH 7 (n = 3)

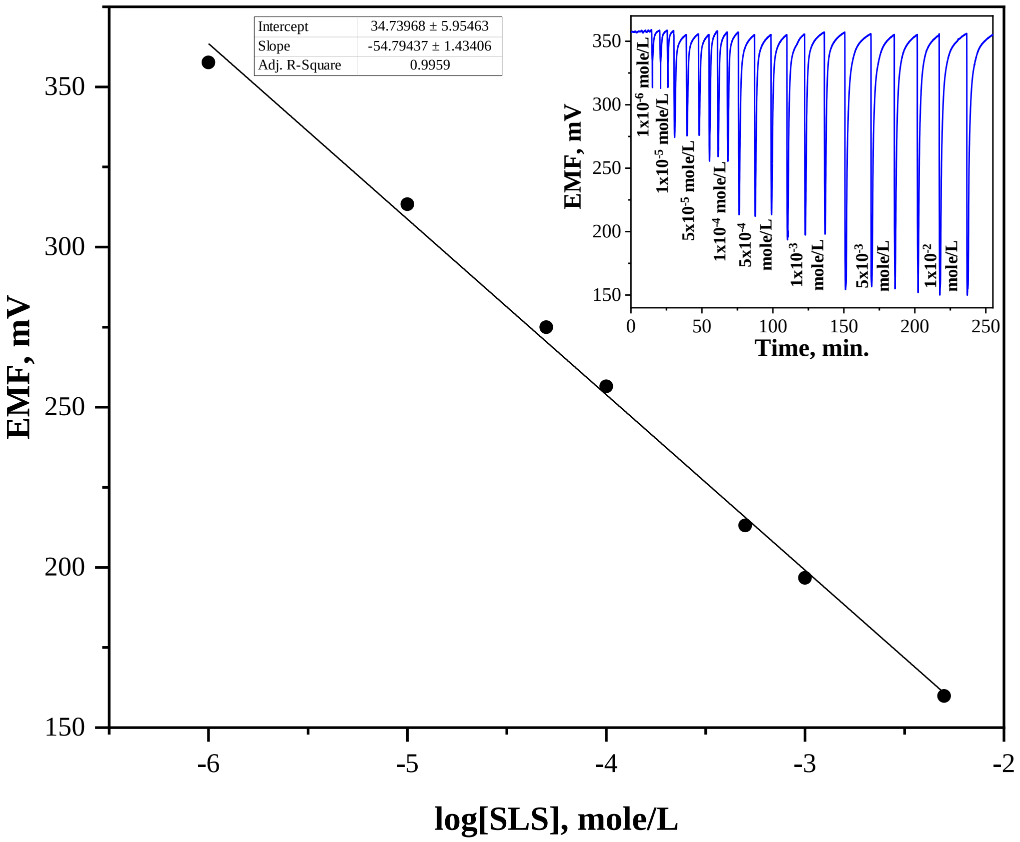

Figure 3. FIA calibration curve of sensor 4 for SLS standard solutions in the range of 10^{-6} – 10^{-2} M in 10^{-2} M PBS buffer, pH 7, and FIA chart in the inset.

3.3. Effects of interfering anions:

To test the sensor's selectivity towards SLS, a series of standard solutions of interfering anions was prepared. Table 4 summarizes both the selected anions for the study and their corresponding selectivity coefficient values of sensor 4 towards SLS against each interferent. It is clear from the table that k_{ij}^{MPM} values for Cl^-, NO_2^-, CO_3^{2-}, SO_4^{2-}, CN^-, and N_3^- anions are < 10^{-4}, indicating that the sensor prefers to respond to SLS over the mentioned anions by a factor of 10000. The sensor had favorable selectivity towards SLS over I^- anion (1808 times), SCN^- anion (237 times), and ClO_4^- anion (47.8 times). The order of SLS sensor 4 selectivity is as follows:

$Cl^- \equiv NO_2^- \equiv CO_3^{2-} \equiv SO_4^{2-} \equiv CN^- \equiv N_3^- < I^- < SCN^- < ClO_4^-$

As a result, both conventional and FIA sensor modules can be used for the analysis of SLS in formulated detergents, cosmetics, toothpaste, as well as wastewater samples without the presence of interference effects from most of the common anions.

Table 4. Selectivity coefficients of sensor 4 for SLS against a series of common interfering anions

No.	**Interfering anion**	k_{ij}^{MPM}
1	Cl^-	$< 10^{-4}$
2	NO_2^-	$< 10^{-4}$
3	CO_3^{2-}	$< 10^{-4}$
4	SO_4^{2-}	$< 10^{-4}$
5	CN^-	$< 10^{-4}$
6	N_3^-	$< 10^{-4}$
7	I^-	$5.53x10^{-4}$
8	SCN^-	$4.22x10^{-3}$
9	ClO_4^-	$2.09x10^{-2}$

3.4. Sensor lifetime:

One of the most important features of a chemical sensor is its lifetime. In this experiment, sensor number 4 was freshly prepared, calibrated in PBS buffer solution and its performance characteristics were calculated (table 5). The sensor was kept soaked in PBS buffer solution for 2.5 months, then it was recalibrated. It appears from table 4 that the detection limit, the correlation coefficient, and the response time were not affected by the long storing time. On the other hand, minor increases in the linear range (from $9.33x10^{-7} – 5.31x10^{-3}$ to $8.13x10^{-7} – 5.31x10^{-3}$ M) and the slope (+7.8%) were observed. These results indicate that the sensing element (MTOAC) did not leach out of the membrane phase even with prolonged soaking/storing times and also the membrane did not lose its exchange properties. As a result, sensor 4 can be stored and reused within a time period of at least 2.5 months without deterioration and with minor and acceptable changes in its performance characteristics.

Table 5. Effect of lifetime on the performance characteristics of sensor 4

Performance characteristics	**New sensor**	**2.5 months-old sensor**
Detection limit, M	$7.4x10^{-7}$	$7.4x10^{-7}$
Linear range, M	$9.33x10^{-7} – 5.31x10^{-3}$	$8.13x10^{-7} – 5.31x10^{-3}$
Slope, mV/decade concentration	-59.43	-64.08
Correlation coefficient, r^2	0.999	0.999
Response time, s	60	60

3.5. Analysis of industrial products and wastes:

A series of samples with different origins containing SLS was collected. The samples were analyzed, without any pretreatment or extraction steps, using the conventional type sensors with the standard calibration curve and the standard addition methods, and using the FIA system with direct calibration. Samples were also analyzed using an independent reference spsectrophotometric method for the determination of anionic surfactants using methylene blue dye [34]. Statistical analysis of the obtained results in table 6 revealed a between batch variability (CV_b) of 0.65 – 2.18%, and that all the results lie within 90% confidence intervals. Moreover, for all the analyzed samples, the results of standard calibration curve method for both conventional and FIA systems appear to be higher than the results obtained using the standard addition method. This could be explained by the effect of interfering materials in the samples that cause positive errors, ranging between 0.55 – 2.15%. Accordingly, both the standard calibration and the standard addition methods can be used for the analysis of SLS in real samples with 95% confidence level. Results for the standard addition potentiometric method are in good agreement with those results obtained using the standard methylene blue spectrophotometric method, an all data are located within 90% confidence intervals.

Table 6. Analysis of SLS in formulated detergents, toothpaste, shampoo, and wastewater samples using both static and FIA modules

No.	Sample	Standard calibration curve method*	Standard addition method*	Direct FIA method*	Reference methylene blue method*
1	Toothpaste, w/w%	1.18	1.13	1.15	1.15
2	Hand-wash gel, w/w%	3.22	3.18	3.19	3.20
3	Hair shampoo, w/w%	6.60	6.57	6.72	6.60
4	Fabric powdered detergent, w/w%	11.32	11.29	11.44	11.28
5	Washing machine wastewater, mg/L	195.8	193.5	197.2	194.1
6	Industrial untreated wastewater, mg/L	89.3	88.7	91.5	89.1

*Samples were triplicate measured (n=3)

4. CONCLUSIONS

A new ion selective electrode for lauryl sulfate anion is introduced. The sensor is based on methyltrioctylammonium chloride as the sensing element in a plasticized PVC-based polymeric matrix. Sensor response characteristics were examined in two different sensor formats: conventional static format and flow-injection format. Both modules showed reliable response characteristics in terms of the analysis of detergents, toothpaste, hand-wash gel, shampoo, and wastewater samples. The sensors exhibited a detection limit of $7.4x10^{-7}$ M, a linear range of $9.33x10^{-7}$ – $5.31x10^{-3}$ M, a slope of -59.43 mV/decade, a correlation coefficient of 0.999, and a fast response time of 60 s. Sensors were successfully applied for the analysis of lauryl sulfate levels in a variety of samples with both direct calibration and standard addition protocols.

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