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# SYNTHESIS AND SURFACE PROPERTIES OF A NOVEL ANIONIC SURFACTANT

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## ABSTRACT

The novel anionic surfactant sodium 3-oxo-2-(3-(4-sulphonatophenyl)triaz-2-enyl) octadecanoate (SSTO) was prepared from renewable raw materials; glycine and palmitic acid. Surface and bulk properties of SSTO were investigated by surface tension technique at 298, 308, 318 and 328 K. A series of important parameters including critical micelle concentration (CMC), maximum surface excess concentration ($\Gamma_{max}$), minimum area per molecule ($A_{min}$), surface tension at CMC ($\gamma_{CMC}$), effectiveness of surface tension reduction ($\Pi_{CMC}$) and efficiency of surface adsorption ($pC_{20}$). The effect of 3 wt% *n*-propanol, *n*-butanol and *n*-pentanol was also considered at 298 K.

**Key Word:** Synthesis, Surface properties, Novel anionic surfactant**,** Surface adsorption.

## INTRODUCTION

Surfactants are organic compounds that have the ability to decrease the surface tension of water and consisting of two moieties: a non-polar hydrophobic group (referred to as the tail) which is usually a straight or branched long hydrocarbon chain which is attached to a polar hydrophilic group (referred to as the head) [1]. Anionic surfactants are the most important class of surfactants since they have low cost of manufacture in comparison with other classes of surfactants, and they are extensively used in industrial applications specially soaps and synthetic detergents [2]. The concentration of surfactant needed to initiate micelle formation is called the critical micelle concentration or CMC [3]. CMC can be determined from the intersection points in the curves of the physical properties as a function of concentration [4]. Because of their surface activity and dual character arise from the presence of both hydrophobic and hydrophilic moieties, surfactants are among the most versatile materials contributing in many chemical applications [5] such as oil recovery, detergency, corrosion inhibition, agrochemicals, and beneficiation of ores as flotation agents [6-8]. The use of renewable raw materials for the synthesis of surfactants has become priority in many fields because they have been found to have better surface and biological properties compared to conventional surfactants and considered as one of the preferred choices for food, pharmaceutical and cosmetic applications due to their low toxicity and quick biodegradation [9]. Several new surfactants have been developed possessing renewable structural parts like fatty acids [10] and amino acids [11]. This study aimed to prepare a new double-headed anionic surfactant and furthermore, to investigate the behavior and stability of micellization process for that surfactant under the effect of either temperature or alcohol.

# MATERIALS AND METHODS

## Materials

Palmitic acid, glycine, thionyl chloride, sulphanilic acid and anhydrous sodium sulphate were purchased from Sigma-Aldrich for chemicals. All the compounds, solvents and reagents were of the highest commercially purity and were used without further purification.

## Synthesis

The synthesis of the anionic surfactant was presented in schemes I, II and III. In scheme I, the sulphanilic acid (0.06 mole) was treated with (3.18 gm) of anhydrous $Na_2CO_3$ dissolved in 10 ml of distilled water with stirring for 15 min at 40 °C.

Where, upon the solution was cooled at 0 °Cand treated with 2.0 gm of $NaNO_2$ dissolved in a minimum volume of water.

The solution was cooled in ice water and a mechanical stirrer was used with a slow addition of (0.2 mole) of HCl.

A solution of glycine ethyl ester hydrochloride in water was added slowly to the diazonium salt solution with stirring for 1 hr at 0°C. The clear solution was treated with a large excess of NaOH (40 ml, 10%) and the triazene precipitated slowly. Precipitation was normally evident after 30 min, and was completed within 3 hr. with continuous warming. The resulting reaction product was filtered off, washed with water and crystallized from ethanol to give 4-(3-(2-ethoxy-2-oxoethyl)triaz-1-enyl) benzenesulphonic acid **(3)**.

In scheme II, palmitoyl chloride was prepared by refluxing a mixture of palmitic acid (26.6 gm, 100 mole) and thionyl chloride (23.7 ml, 200 mole) on water bath maintained at 35-40°C with shaking for 6 hr.The excess of thionyl chloride was removed by washing successively with water and the residue was dried over anhydrous $Na_2SO_4$ to collect the liquid palmitoyl chloride.

Then, a mixture of compound **(3)** (0.01 mole), and palmitoyl chloride **(5)** (0.01 mole) in $CH_2Cl_2$ (30 ml) and pyridine (5 ml) was heated under reflux for 8 hr. The reaction mixture was allowed to cool, poured into crushed ice and acidified with HCl. The solid product was filtered off and crystallized from *n*-hexane to give 4-(3-(1-ethoxy-1,3-dioxooctadecan-2-yl)triaz-1-enyl) benzenesulphonic acid **(6)**.

In scheme III, the anionic surfactant sodium3-oxo-2-(4sulphonatophenyl)triaz-2-enyl) octadecanoate (SSTO) **(7)** was prepared by the reaction of the equivalent volume of 1M aqueous NaOH (0.02 mole) with a stirred solution of compound **(6)** (0.01 mole) in methanol (10 ml) and the reaction was allowed to proceed for 3 hr.

Then, a white solid was extensively precipitated and was collected by suction filtration, after which was rinsed with ice cold water. The IR spectra (KBr) were recorded on a FTIR 5300 spectrometer (υ, $cm^{-1}$), the $^1$H-NMR spectra were recorded in DMSO-$d_6$ and $CDCl_3$ at 200 MHz on a Varian Gemini NMR, the mass spectra were recorded using 1000 EX mass spectrometer at 70 ev and the elemental analysis was obtained using an Elemental Analyzer Model Varioelemenrar. All the measurements were carried out at the Micro Analytical Center, Cairo University, Egypt.

## Surface Tension Measurements

The surface tension was measured using the maximum bubble pressure method [12]. The measurement principle is based on applying pressure equivalent to the pressure inside the capillary to return the liquid level in the capillary to the same level as that of the surfactant solution. The surface tension (□) was determined from the equation [13] where

$$\gamma = \frac{r\,g}{2}(h_v d_v - h\,d) \qquad (1)$$

r is the radius of the capillary tube, g is the gravitational acceleration, $h_v$ is the difference in water levels in manometer created by the pressure, h is the depth of the capillary below the solution level, $d_v$ is the density of water and d is the density of surfactant solution.

## RESULTS AND DISCUSSION

### 1.Characterization of the Prepared Compounds.

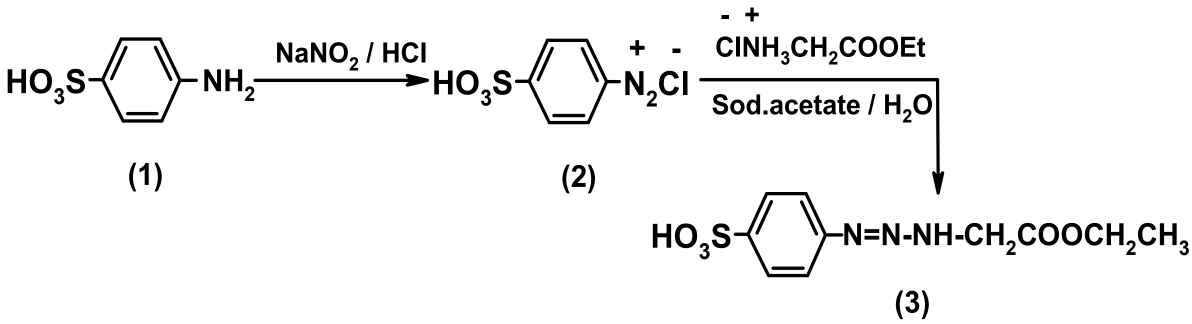
The reaction of diazonium salts with proteins has been extensively used as a structural probe, typically in the elucidation of the topography of the active sites of enzymes [14]. The azo-proteins formed in this way arise largely from diazo-coupling with the activated aromatic rings of tyrosine and histidine residues and the ε-amino group of lysines [15].

Thus, the reaction of aryldiazonium salt **(2)** with glycine ethyl ester in aqueous solution containing sodium acetate afforded stable triazene derivative **(3)** (scheme I) where the diazonium ion attacked at the $NH_2$ moiety and showed no tendency to attack at the activated $CH_2$, which would give rise to hydrozone formation [16, 17], based on its spectral data. Compound **(3)** was formed as brown crystals from ethanol with yield (83%) and m.p 210-212 °C.

The IR and $^1$H-NMR spectra of compound **(3)** revealed the following bands and signals; IR (KBr) ν (cm$^{-1}$) = 3434 (NH), 3100 (CH-arom), 2950-2850 (CH-aliph), 1650 (C=O); $^1$H-NMR (DMSO-$d_6$) δ (ppm) = 1.20 (t, 3H, $CH_3$), 4.18 (q, 2H,$CH_2$-O), 5.23 (br s, 2H, $CH_2$-N), 7.94-8.27 (m, 5H, Ar-H and $SO_3H$), 10.40-10.50 (br s,1H, NH). The mass spectrum showed a very intense molecular ion peak at 287 and a number of fragments agreed with the proposed structure.The foregoing results prompted us to investigate further the synthetic potentiality of stable triazene **(3)** toward palmitoyl chloride. Thus, treatment of triazene derivative **(3)** with palmitoyl chloride **(5)** in refluxing methylene chloride containing a catalytic amount of pyridine (scheme II) afforded 4-(3-(1-ethoxy-1,3-dioxooctadecan-2-yl)triaz-1-enyl)benzenesulphonic acid **(6)** [18].

The structure of compound **(6)** was established as a sole reaction product based on its spectral data. Compound **(6)** was formed as pale yellow crystals from *n*-hexane with yield (69%) and M.P. 50-52 °C. The IR and $^1$H-NMR spectra of compound **(6)** revealed the following bands and signals; IR (KBr) ν (cm$^{-1}$) = 3200 (NH), 2917-2849 (CH-aliph), 1702 (C=O); $^1$H-NMR ($CDCl_3$) δ (ppm) = 0.89 (t, 3H, $CH_3$, J = 8 HZ), 1.27 (s, 24H, 12$CH_2$), 1.33 (t, 3H, $CH_3$, J = 4 HZ), 1.61 (s, 1H, CH), 1.64 (t, 2H, β-$CH_2$, J = 8 HZ), 2.36 (t, 2H, α-$CH_2$, J = 8 HZ), 4.30 (q, 2H, $CH_2$), 7.28-8.39 (m, 6H, Ar-H + $SO_3H$ and NH). The mass spectrum of compound **(6)** revealed the molecular ion peak at m/z = 525 ($M^+$) corresponding to $C_{26}H_{43}N_3O_6S$ Compound **(6)** was then submitted to saponification at room temperature with an aqueous methanolic solution of NaOH, from which the target SSTO **(7)** was easily isolated (scheme III).

The structure of compound **(7)** was established as a sole reaction product based on its $^1$H-NMR spectrum. Compound **(7)** was formed as white crystals from DMSO with yield (62%) and M.P. 280-282 °C. The $^1$H-NMR spectrum of compound **(7)** revealed the following signals; $^1$H-NMR (DMSO-$d_6$) δ (ppm) = 0.85 (t, 3H, $CH_3$, J = 4 HZ), 1.24 (s, 24H, 12$CH_2$), 1.48 ( t, 2H, β-$CH_2$, J = 8 HZ), 1.91 (s, 1H, CH), 2.18 (t, 2H, α-$CH_2$ ,J = 8 HZ), 6.96-7.75 (m, 4H, Ar-H), 8.15 (s, 1H, NH).



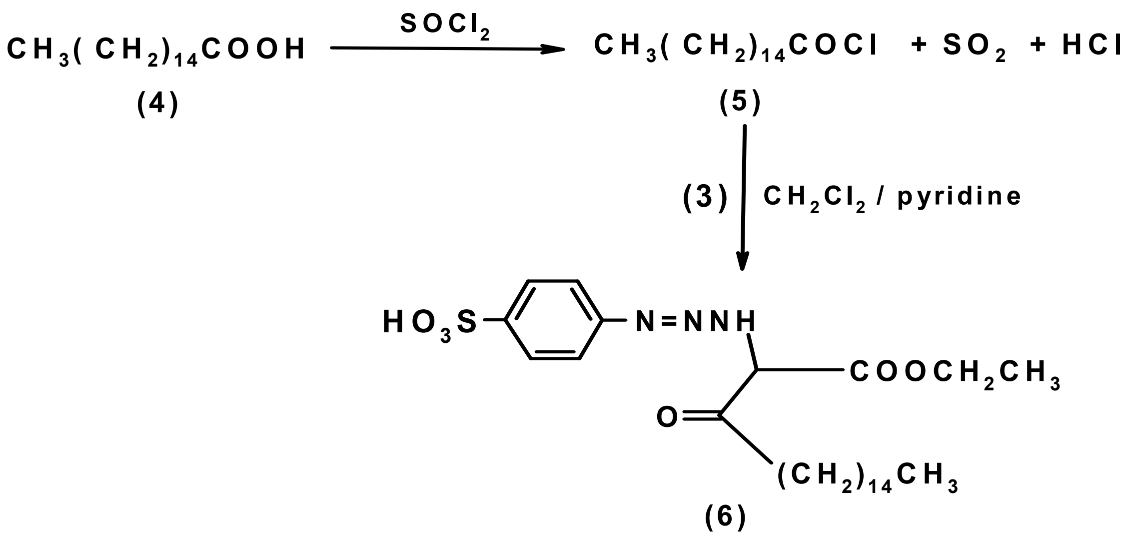
**Scheme I. Reaction of diazonium salt with glycine ethyl ester hydrochloride**

**2. Critical Micelle Concentration (CMC).**

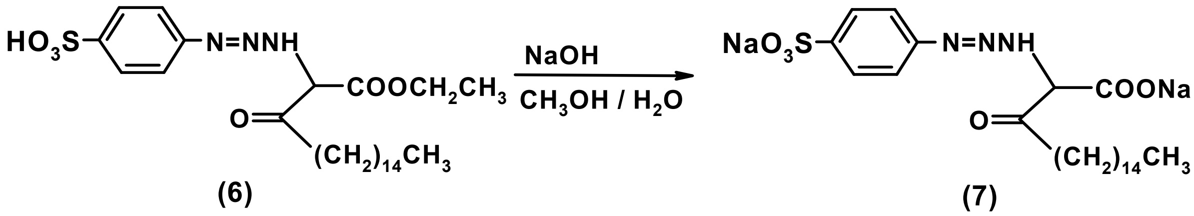
The CMC data obtained from surface tension and electrical conductivity for SSTO at various temperatures were listed in Table (1).

Figure (1) illustrated the results of CMC obtained from surface tension as a function of temperature. SSTO was found to have lower CMC value (136 µM) than SDS (8200 µM) at 298 K [4].This suggesting an excellent ability of micelle formation for SSTO due to increasing the length of hydrophobic tail and modifying the structure of the head group.It was evident from figure 1 that CMC increased with increasing temperature. The effect of temperature on CMC of an ionic surfactant in aqueous solution is usually analyzed in terms of two opposing factors.

The increase in temperature reduces the hydration of the hydrophilic groups that favors micellization. On the other hand, the increase in temperature also causes disruption of the structured water surrounding the hydrophobic group that disfavors micellization [19].



**Scheme II. Reaction of triazine derivative (3) with palmitoyl chloride.**




**Scheme III. Preparation of SSTO (Saponification process).**

**Table (1): CMCs of SSTO from different methods in aqueous solution at various temperatures.**

| T , K | CMC$^a$, µM | CMC$^b$, µM |
|---|---|---|
| 298 | 136 | 139 |
| 308 | 168 | 170 |
| 318 | 204 | 220 |
| 328 | 237 | 239 |

CMC$^a$ determined from surface tension and CMC$^b$ determined from electrical conductivity.

**Table (2): CMCs of SSTO from different methods in 3 wt% *n*-alcohol solution at 298 K.**

| Type of Alcohol | CMC$^a$, µM | CMC$^b$, µM |
|---|---|---|
| -- | 136 | 139 |
| *n*-propanol | 109 | 125 |
| *n*-butanol | 95.50 | 105 |
| *n*-pentanol | 83.20 | 86.20 |

CMC$^a$ determined from surface tension and CMC$^b$ determined from electrical conductivity.

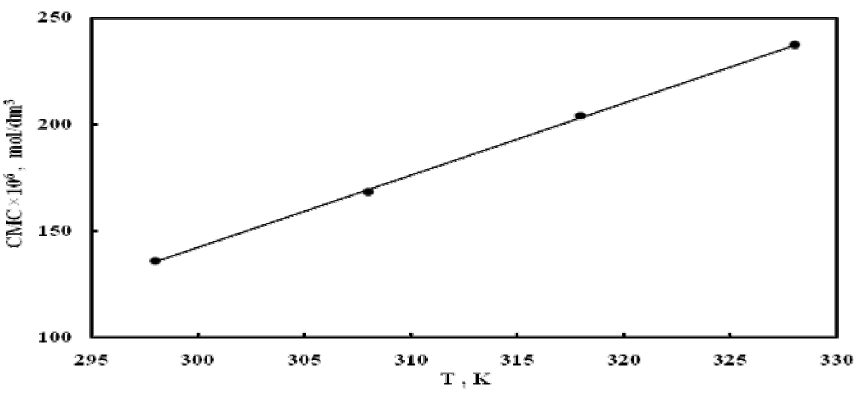



**Figure (1): CMC of SSTO obtained from surface tension in aqueous solution versus absolute temperature.**

Figure 1 revealed that this second effect is predominant in the temperature range studied. Table (2) reported the changes in CMC with the addition of 3 wt% of *n*-propanol, *n*-butanol and *n*-pentanol at 298 K. CMC decreased when various *n*-alcohols were present in solution when compared with aqueous solution. This may be attributed to the incorporation of alcohol molecules within the micelle thus increasing the length between polar heads that causing the repulsive interactions to decrease [20].

Also, the hydrophobic part of alcohols may enhance the hydrophobic character of the surfactant that mainly favors micellization [3]. The decrease in CMC had become more pronounced with the increase in the alcohol alkyl chain in solutions containing an identical alcohol concentration (**Figure 2**).

### 3. Surface Tension

The representative plots of γ versus log concentration of SSTO were shown at various temperatures in figures 3a-d and with the effect of added alcohol in figures 4a-c. A linear decrease in γ was observed with the increase in surfactant concentrations up to CMC because the amphiphilic structure caused the surfactant molecules to get proficiently adsorbed at the surface [21]. Also, increasing the concentration resulted in an increase in the content of hydrophobic chains in water, leading to a rise in free energy of the system.

Therefore, the migration of monomers from the bulk to the air/solution interface increased to minimize that free energy and hence, the surface tension is reduced [22].In all cases, SSTO had the ability to decrease γ until a constant value ($\gamma_{CMC}$) which is defined as the surface tension of the solution at CMC. $\gamma_{CMC}$ was read off directly from Figures 3a-d and figures 4a-c and the data were listed in tables 3 and 4 at various temperatures and for various alcohol solutions respectively.

It was clear that $\gamma_{CMC}$ values decreased with increasing either temperature or carbon number of *n*-alcohol.Based on the results of surface tension, the maximum surface excess concentration ($\Gamma_{max}$) and the minimum area occupied per molecule at the surface ($A_{min}$) of SSTO were calculated using the Gibbs adsorption isotherm equations [6, 23] where dγ/d log C is the slope of surface

$$\Gamma_{max} = -\frac{1}{2.303\, n\, R\, T}\left(\frac{d\gamma}{d\, logC}\right) \quad (2)$$

$$A_{min} = \frac{10^{18}}{\Gamma_{max}\, N_A} \quad (3)$$

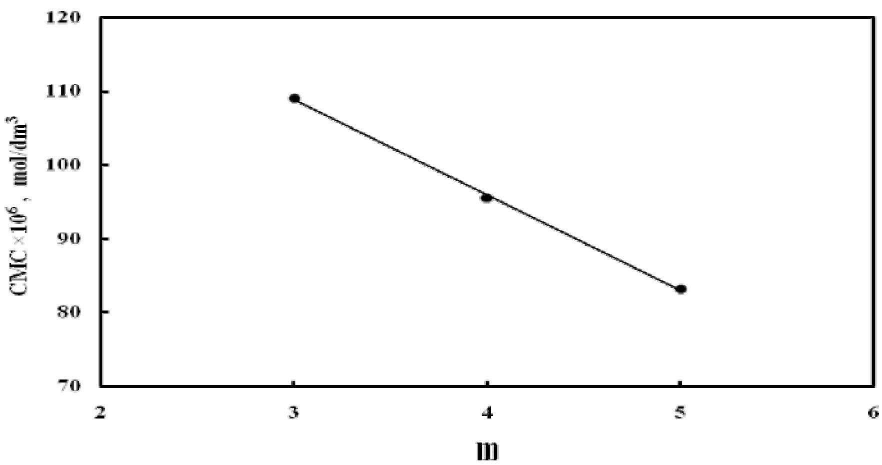



**Figure (2): CMC of SSTO obtained from surface tension in 3 wt% *n*-alcohol solution versus carbon number of alcohol at 298 K.**

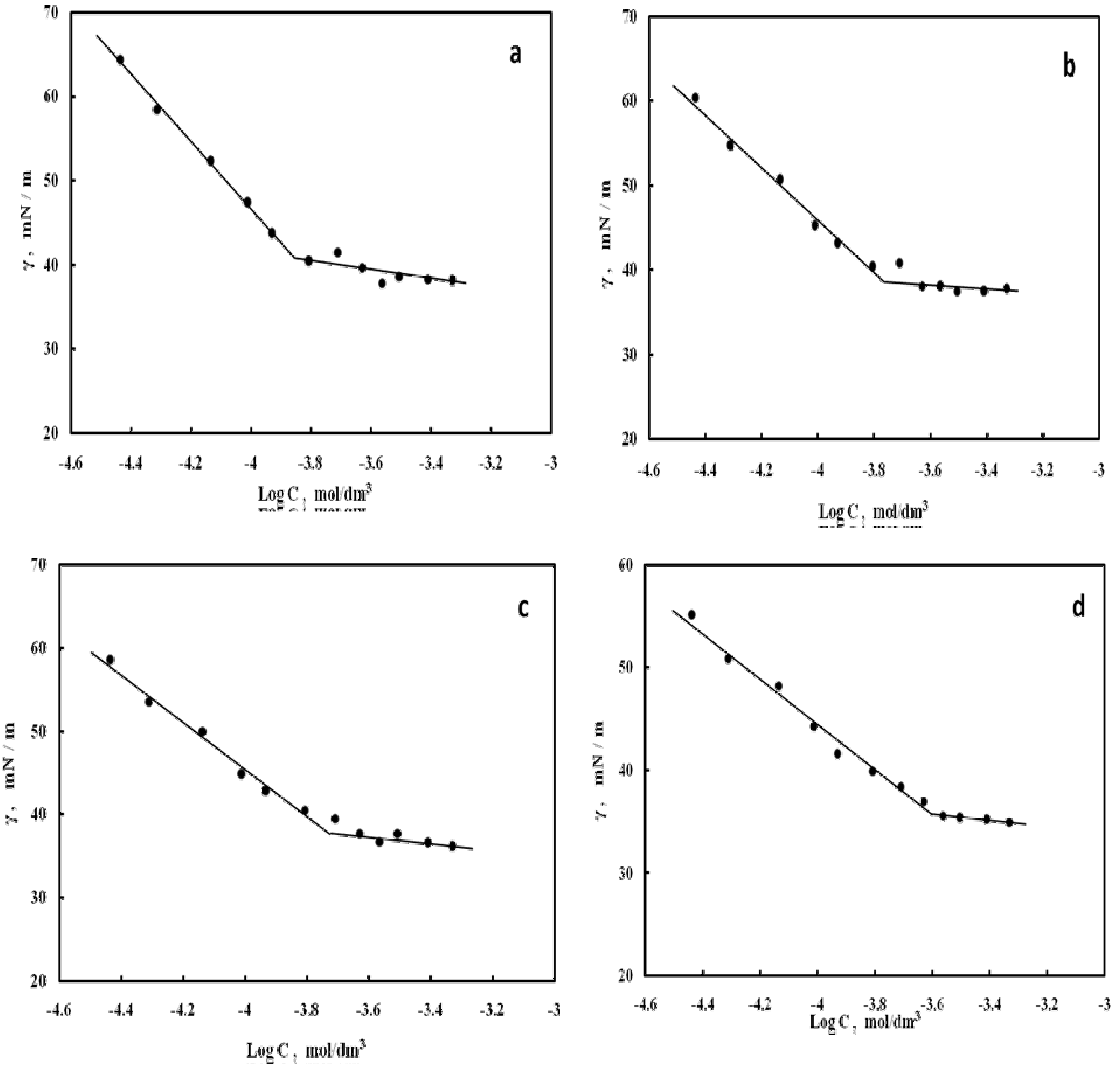



**Figure (3): Surface tension versus log molar concentration of SSTO in aqueous solution at: (a) 298 K, (b) 308 K, (c) 318 K and (d) 328 K.**

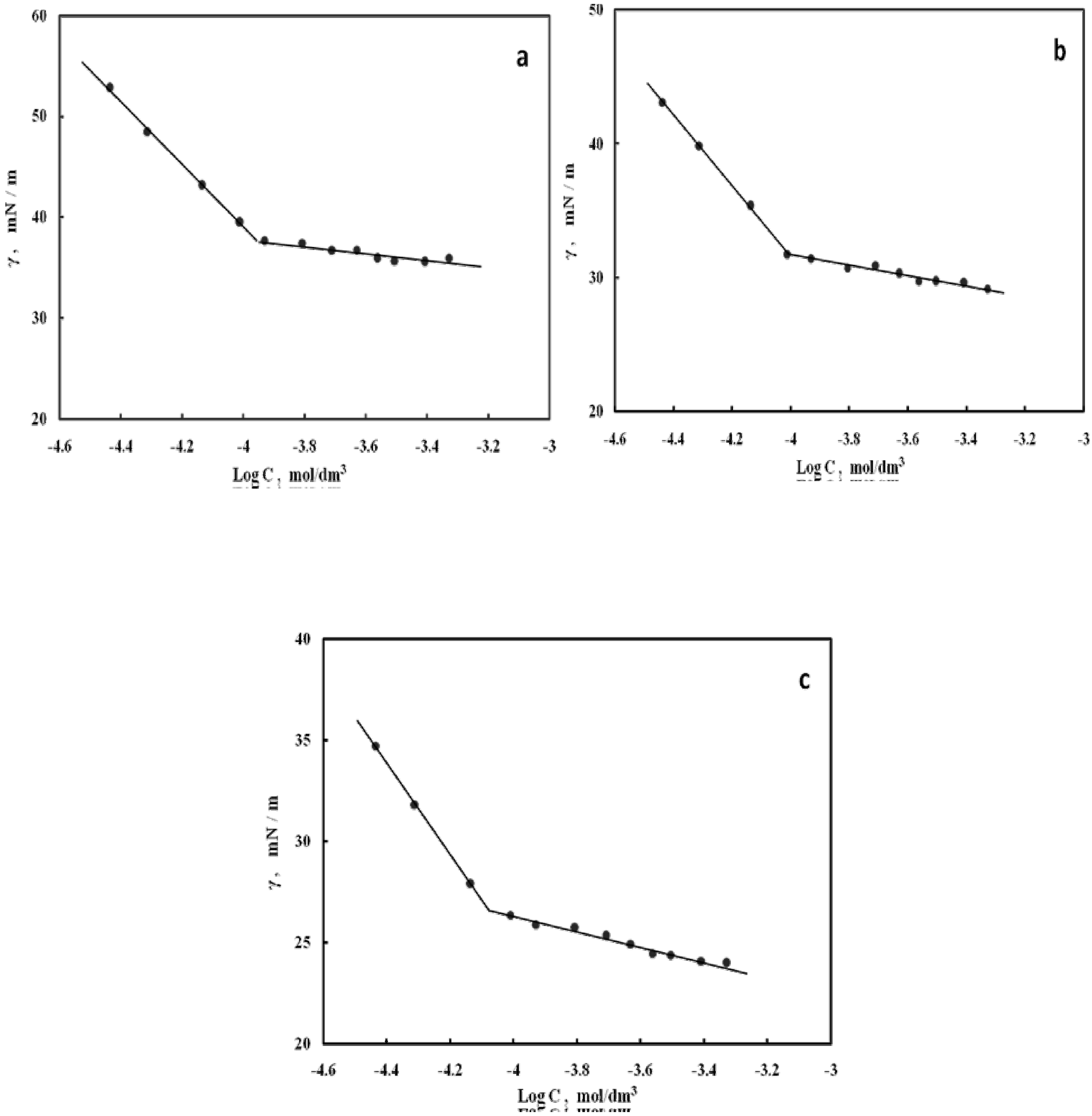



**Figure (4): Surface tension versus log molar concentration of SSTO at 298 K in: (a) 3wt% *n*-propanol, (b) 3 wt% *n*-butanol and (c) 3 wt% *n*-pentanol.**

tension plots below CMC, R is the gas constant, T is the absolute temperature, n is the number of ionic species whose concentration changes with the surfactant concentration at the interface and is taken as three for SSTO since it is a divalent surfactant [24] and $N_A$ is Avogadro's number.

As presented in table (3), $\Gamma_{max}$ decreased with increasing temperature owing to high thermal agitation of molecules [25] whereas the $A_{min}$ values increased upon increasing temperature due to the increase in thermal motion of monomers at the surface [26]. The greatest value of $\Gamma_{max}$ and the smallest value of $A_{min}$ at 298 K meant a denser arrangement of surfactant molecules at the surface at that temperature. Table( 4) also showed a decrease in $\Gamma_{max}$ and an increase in $A_{min}$ as the alkyl chain of the alcohol increased.

**Effectiveness of surface tension reduction (*$\Pi_{CMC}$*) can be calculated from the experimental CMC data through the relation [7].**

$$\Pi_{CMC} = \gamma_{\circ} - \gamma_{CMC} \quad (4)$$

Where $\gamma_{\circ}$ is the surface tension of pure solvent and $\gamma_{CMC}$ is the surface tension of the solution at CMC. According to the results in table 3, $\Pi_{CMC}$ values of SSTO were found to be close and being most effective at 328 K with a reduction of 31.77 mN/m. Also, table 4 revealed a decrease in the values of $\Pi_{CMC}$ in 3 wt% *n*-alcohol solutions in comparison with those of aqueous solution. Also, efficiency of surface adsorption ($pC_{20}$) was determined from the concentration that capable of suppressing the surface tension of the solvent by 20 mN/m, $C_{20}$, [27] where:

$$pC_{20} = -\log C_{20} \quad (5)$$

The values of pC20 of the prepared surfactant were shown in tables 3 and 4. pC20 increased with increasing temperature and this may mean that the hydrophobic tails of SSTO monomers at 328 K can cover the surface more efficiently than the same molecules at lower temperatures. Also, these results showed a decrease in pC20 with the increase in alcohol carbon number. These results illustrate well the complexity of the effect of alcohols on surface and micellar properties.

## CONCLUSIONS

The anionic surfactant sodium 3-oxo-2-(3-(4-sulphonatophenyl)triaz-2-enyl) octadecanoate (SSTO) was prepared by a convenient way from renewable raw materials. The investigated surfactant, based on the results of CMC and other surface properties, had showed high micellar properties and good surface activities that may be ascribed to the use of the long. hydrocarbon chain (16 carbon atoms) and also the presence of the double hydrophilic groups.

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**Table (3): Surface properties of SSTO in aqueous solution at various temperatures.**

| T, K | $\gamma_{CMC}$, mN/m | $\Pi_{CMC}$, mN/m | $\Gamma_{max} \times 10^6$, mol/m$^2$ | $A_{min}$, nm$^2$ | $C_{20}$, μM | $pC_{20}$ |
|---|---|---|---|---|---|---|
| 298 | 40.75 | 31.24 | 2.33 | 0.71 | 75.86 | 4.11 |
| 308 | 38.79 | 31.62 | 1.60 | 1.04 | 72.61 | 4.14 |
| 318 | 37.12 | 31.66 | 1.46 | 1.13 | 73.87 | 4.13 |
| 328 | 35.33 | 31.77 | 1.20 | 1.38 | 69.29 | 4.16 |

**Table (4): Surface properties of SSTO in 3 wt% n-alcohol solution at 298 K.**

| Type of Alcohol | $\gamma_{CMC}$, mN/m | $\Pi_{CMC}$, mN/m | $\Gamma_{max} \times 10^6$, mol/m$^2$ | $A_{min}$, nm$^2$ | $C_{20}$, μM | $pC_{20}$ |
|---|---|---|---|---|---|---|
| -- | 40.75 | 31.24 | 2.33 | 0.71 | 75.86 | 4.11 |
| *n*-propanol | 37.80 | 21.90 | 4.20 | 0.40 | 96.16 | 4.02 |
| *n*-butanol | 31.60 | 17.62 | 3.55 | 0.47 | 402.75 | 3.40 |
| *n*-pentanol | 26.53 | 14.64 | 3.05 | 0.54 | 7498.90 | 2.13 |


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## الملخص العربي

# التحضير والخواص السطحية لمركب أنيوني جديد ذو نشاط سطحي

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٢. قسم الكيمياء، كلية العلوم، جامعة السويس، السويس، مصر.

تم تحضير المادة الأنيونية ذات النشاط السطحي وذلك باستخدام حمض البالمتيك والجليسين كمواد أولية. أيضاً تمت دراسة سلوك وثبات عملية الميسلة إلى جانب العديد من الخواص الفيزيوكيميائية والسطحية للمركب قيد البحث عن طريق خاصية التوتر السطحي في المحاليل المائية عند درجات حرارة ٢٩٨ و ٣٠٨ و ٣١٨ و ٣٢٨ كلفن. أيضاً تم دراسة تأثير بعض الكحولات العادية (البروبانول والبيوتانول والبينتانول) على عملية الميسلة عند درجة حرارة ٢٩٨ كلفن. تم تعيين العديد من العوامل الهامة في عملية الميسلة مثل التركيز الميسيلي الحرج (**CMC**) وأقصى تركيز سطحي ($\Gamma_{max}$) وأقل مساحة يشغلها الجزئ عند السطح ($A_{min}$) وكذلك فعالية هذا المركب في خفض التوتر السطحي للمذيب ($\Pi_{CMC}$) وكفاءته في عملية الامتزاز ($pC_{20}$). بناءاً على هذه النتائج نستطيع القول بأن المركب قيد الدراسة يمتلك نشاط سطحي جيد وذلك بسبب طول السلسلة الهيروكربونية ووجود أكثر من مجموعة قطبية.

**الكلمات الاسترشادية:** الخواص السطحية، الفيزيوكيميائية، التوتر السطحى، المركب الانيونى.

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