Synthesis and Evaluation of Surface Properties of Anionic Surfactants Based on Fatty Matter of Egyptian Peach Kernel

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Abstract

Sulfonated anionic surfactants were synthesized by the esterification of acrylic acid (AA) with different types of fatty alcohols ($C_{16:0}$, $C_{18:0}$, $C_{18:1}$ and mixed Peach oil fatty alcohols) followed by addition of sodium bisulfite. The Peach oil was extracted from Peach kernel waste, its chemical characterization and fatty acids compositions were determined. The chemical structure of the prepared anionic surfactants was confirmed by FTIR and ^1H NMR spectroscopic tools. The surface properties and thermodynamic parameters, which illustrate the spontaneity of the adsorption and micellization processes of the synthesized surfactants, were determined. Furthermore, the biological activity of these surfactants was examined against sulfate reducing bacteria (SRB) and showed the aggressive influence on their growth.

Keywords — *Peach kernel oil, Anionic surfactants, Surface, thermodynamic properties, Biological activity.*

I. INTRODUCTION

Fruit remains are one of the major waste resources that pose a dangerous problem in world countries. So inexpensive and available methods were applied to dispose these residues and extract the oils and bioactive compounds that can be used in different industrial fields [1]. The fruit seed waste is good renewable resources for oil extraction while they are nontoxic, environmentally friendly and inexpensive. For example, tomato oil was extracted from its seeds obtained from tomato processing industry that used for nutritive or industrial purposes [2]. On the other hand, different types of surfactants were prepared by using rubbish resources such as Rice bran [3], Mangifera seeds [4], Guava seeds [5], Al-cedre seeds [6], Malia azedarach seeds [7], Soya bean gums [8] and Frying oil [9]. Surfactants are amphiphilic compounds that composed of a hydrophilic portion (polar group) connected with a long hydrophobic portion. In the polar solvents, at concentrations above the critical micellar concentration (CMC), they self-associate to form micelles [10]. They have wide range applications such as detergents, textile processing, coatings formulation, drilling mud and petrochemical recovery in the petroleum industry, paints, emulsification, petroleum-collecting and dispersing agents and corrosion inhibitors [5, 11 and 12]. The fatty sulfonated surfactants have scores of uses because they are less skin irritating and greater biodegradable [13]. In recent years, researchers are interested in the preparation and evaluation of anionic surfactants from natural, renewable resources due to its importance and usage in different industrial fields [14-19]. SRB is sulfate reducers, anaerobic bacteria in souring systems. The presence of these bacteria in pipes of an oil well causes intense corrosion problems which lead to environmental health and safety hazards and economic losses that made by the stabilized mixed cultural activity containing SRB [20,21]. The existence of sodium sulfonate group in the prepared surfactants may be the reason for decreasing of the SRB aquatic toxicity and increasing of its biodegradability. Complementing our continuing research, in this research program, we synthesized a new series of anionic surfactants based on non-edible natural resource, which is environmentally friendly and non-toxic oil extracted from *Peach* kernel. The surface, thermodynamic properties and biological activity against sulfate reducing bacteria (SRB) were studied for these surfactants to evaluate its efficiency.

II. EXPERIMENTAL

1) Materials

Peach kernels were obtained from Kaha Company for food products and juices, Egypt. Octadec-9-en-1-ol and *p*-Toluene sulfonic acid were purchased from Sigma-Aldrich Chemicals Co, hexadecanol, toluene, sodium bisulfite and sodium sulfate anhydrous were purchased from AL-Nasr Chemicals Co. Solvents such as petroleum ether (40-60 °C), *n*-hexane and methanol are higher grades, octadecanol and acrylic acid were purchased from Algomhoria Chemicals Co.

2) *Instrumentation*

Gas-liquid chromatography (GLC) of a *Peach* methyl ester was carried out by Perkin Elmer Auto System XL, equipped with flame ionization detector (FID), fused silica capillary column ZB-5 (60 m × 0.32 mm I.D) and oven whose temperature was maintained initially at 150 °C and programmed from 150 to 240 °C at rate 3°C/min. The apparatus was operated with helium gas flow rate 1 ml/min and the temperatures of the injector and detector were adjusted at 230 °C and 250 °C respectively. The split must be 1:10 and the size of the sample become 2 µL. Identification of GLC peaks was carried out using chromatograms of standard fatty acid methyl esters (Sigma, USA), flavor and odor lab (National Research Centre, Dokki, Cairo, Egypt). FTIR Spectra of the synthesized compounds was measured as liquid or solid in KBr disk on a thermo Nicolet iS10 FTIR spectrophotometer (Faculty of Science, Benha university, Benha, Egypt). ^{1}H NMR spectra was carried out by Bruker Avance (III) 400 MHz signal (Switzerland) with (128) scans at 298 °k in deuterated (DMSO-d6) and/or in ($CDCl_3$-d) as a solvent and tetramethyl silane (TMS) as an internal reference. De-Noüy ring Tensiometer (Kruss-K6) is utilized to determine surface tension of water (γ) with different molar concentration of prepared surfactant solutions.

3) *Synthesis of sulfonated anionic surfactants*

The anionic surfactants were synthesized from *Peach* kernel oil by the following steps:

a) *Extraction, chemical and physical specification of Peach kernel oil*

Peach kernels were cleaned, cut and dried in oven at 60 °C, then ground and soaked in *n*-hexane for 16 hrs. After filtration, the solution was dried using sodium sulfate anhydrous, and the solvent was vacuum evaporated. The obtained *Peach* oil conducted to determine the chemical and physical specification and estimated according to AOCS Methods [22] **Table 1**.

b) *Fatty acid composition of Peach oil*

The fatty acid composition of *Peach* oil, which represented in **Table 1,** was performed as fatty acid methyl esters **[**23] using GLC (**Fig. 1**).

c) *Reduction of fatty acids*

The fatty alcohols of *Peach* oil were obtained by reduction of the corresponding *Peach* oil fatty acids methyl esters using lithium aluminum hydride **(LAH)** [24].

d) *Synthesis of fatty alkyl acrylate*

Acrylic acid (7.204 gm, 0.1 mole) was esterified with various fatty alcohols, namely, hexadecanol, octadecanol, octadec-9-en-1-ol and mixed fatty alcohols of *Peach* oil (0.1 mole) using Dean-stark adapter in the presence of catalytic amount of *p*-Toluene sulfonic acid and hydroquinone in dry toluene as solvent [25]. The purified hexadecyl, octadecyl, octadec-9-enyl and mixed *Peach* fatty alkyl acrylate (**I_{a-d}**) were obtained by the extraction with petroleum ether (40-60 °C), dried over anhydrous sodium sulfate and the solvent was evaporated.

Table 1: Fatty acid composition, chemical and physical specification of Peach kernel oil

Fatty acid composition of *Peach* kernel oil		**Chemical and physical specification of *Peach* kernel oil**	
Fatty acids	**Peak area %**		
Saturated fatty acids		Appearance	Light, yellow oil
Palmitic ($C_{16:0}$)	6.57	Density	0.889
Stearic ($C_{18:0}$)	1.43	Refractive index	1.4687
Unsaturated fatty acids		Acid value	0.036
Palmitoleic ($C_{16:1}$)	0.46	Saponification value	201.96
Oleic ($C_{18:1}$)	53.72	Iodine value	111.58
Linoleic ($C_{18:2}$)	34.47	Peroxide value	4.90
Linolenic ($C_{18:3}$)	3.35	Unspecifiable matter	5.27

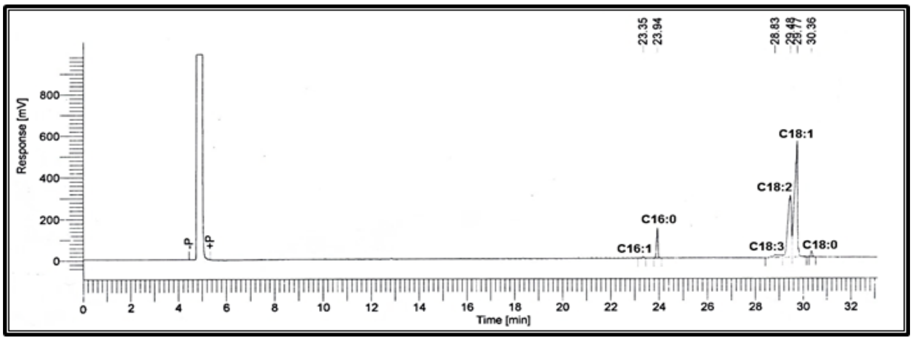


Fig. 1: GLC of the methyl ester of Peach kernel oil

e) Synthesis of anionic surfactants (SCAS, SSAS, SOAS and $SMAS_{Peach}$)

A series of anionic surfactants were prepared as shown in **Scheme 1.** A solution of $NaHSO_3$ (1.64 gm, 0.0158 mole) in 5 mL of distilled water is added drop wisely to a solution of acrylate esters (0.01 mole) in 50 mL of methanol. This mixture was refluxed for 24 hours at 80 °C [26]. The resulting anionic surfactants were obtained after evaporation of the solvent, washed with hot petroleum ether and dried.

4) Surface characteristics

a) Surface and interfacial tension

Surface tension (γ) of freshly prepared aqueous solutions of synthesized anionic surfactants with various concentrations ranged from (1×10^{-2}) to (3×10^{-7}) mole/L was measured by Kruss-K6 Tensiometer at different temperatures. The surface tension of distilled water was determined before the preparation of different surfactant solutions. During the performance of this measurement; the platinum ring was rinsed with distilled water and acetone, then flamed briefly to remove the remaining acetone [21]. The surface tension values were measured at least three times and the average value was taken.

Interfacial tension values were measured in the 0.1% surfactant solution - toluene system at 28 °C. When the ring was separated at the interface, this value was determined as the interfacial tension point [6].

b) Krafft point (T_K)

These temperatures were measured at which the dispersed 0.1% surfactants solutions become completely soluble on gradually heating and give clear solutions. This point was considered as a standard for completely aqueous solubility for these anionic surfactants [27].

c) Emulsion stability

The emulsion was produced by vigorous shaking of 10 mmole surfactant solution (10 mL) with light paraffin oil (5 mL) at room temperature into a 25 mL graduated cylinder. The emulsion stability was determined by the consumed time counting from the moment of the ending of the shaking until the separation of 9 mL of the clear aqueous volume of the emulsion layer [28].

d) Foaming properties

Powerful shaking for 10 seconds of 40 mL of the surfactant solution (0.1 %) in a 100 mL graduated cylinder at different temperatures (28, 40, 50, 60 °C) was performed to produce excellent foaming properties. The foam volume was measured in ml and the foam stability was measured by the time at 28 °C [29].

e) Stability to hydrolysis

10 mL of surfactant solutions (10 mmole) and 10 mL of sodium hydroxide 0.05 N or 10 mL of sulphuric acid 2 N in phenol tube placed in a thermostat system at 40°C. The surfactant resistance to hydrolysis was determined by the consumed time counting from the moment of the placing of the samples in a thermostat system until the turbidity occurs as a result of the hydrolysis process [30].

5) The biocidal activity of the synthesized anionic surfactants against sulfate reducing bacteria (SRB)

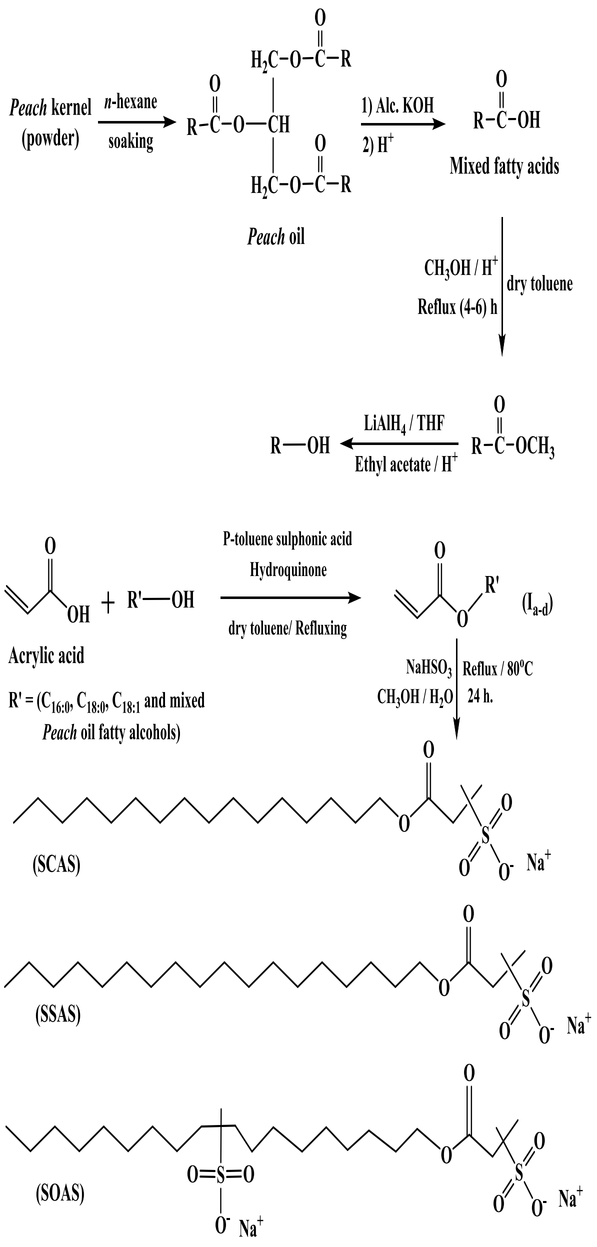
The inhibitory activity of the tested samples on the SRB growth was determined using a modified Kirby-Bauer disc diffusion method [31]. This method summarized in that 100 µL of the test bacteria was grown in 10 mL of fresh media until they reached a count of approximately 10^8 cells/mL. Plates inoculated with the Gram - negative bacteria as ***Desulfomonas pigra* ATCC 29098T** and a filter paper disc that was impregnated with a tested chemical solution (20 mg/mL of DMSO), were incubated at 35-37 °C for 24-48 hours then the diameters of the inhibition zones were measured in millimetres. Blank paper disks (Schleicher & Schuell, Spain) with a diameter of 8.0 mm were impregnated in 10 µL of testing concentration of the stock solution. Standard discs of Ampicillin (Antibacterial agent), served as positive controls for antimicrobial activity.

III. RESULTS AND DISCUSSION

1) *Synthesis*

Peach kernel was obtained from the waste resources. The crude oil was extracted from these kernels using *n*-hexane. *Peach* kernel oil was directed to some experiments to determine oil characteristics and fatty acid composition using GLC (**Fig. 1**), and the data were reported in **Table 1**. It shows that the most of fatty acids are unsaturated fatty acids, about 92% and small amount of saturated fatty acids. This analysis was confirmed by chemical measurement, where the oil shows high iodine value which indicate that the high percent of unsaturated fatty acids.

Four different compounds of anionic surfactants were synthesized from different fatty alcohols obtained by reduction of the corresponding fatty acids ($C_{16:0}$, $C_{18:0}$, $C_{18:1}$ and mixed fatty acids extracted from *Peach* oil respectively), esterified with acrylic acid followed by addition of sodium hydrogen sulfite as shown in **Scheme 1**.



Scheme 1: Synthetic procedures of novel anionic surfactants

2) Characterization

A) Spectroscopic analysis of synthesized esters

The structures of synthesized esters **($I_{a\text{-}d}$)** were confirmed using FTIR and ^{1}H NMR spectroscopy as shown below:

a) Hexadecyl acrylate (I_a) $CH_2=CH\text{-}COO\text{-}CH_2\text{-}(CH_2)_{14}\text{-}CH_3$, colourless viscous liquid, (yield = 92%). Its IR spectrum revealed that characteristic bands at 2921, 2852 cm^{-1} ($\nu_{C\text{-}H}$ aliphatic fatty chain), 1725 cm^{-1} ($\nu_{C=O}$ of ester), 1636 cm^{-1} ($\nu_{C=C}$ olefinic) and 1185.83 cm^{-1} ($\nu_{C\text{-}O\text{-}C}$ stretching). Furthermore, its ^{1}H NMR gives δ (ppm): 0.81 (t, 3H, $[\underline{CH_3}\text{-}CH_2\text{-}]$), 1.2 (s, 24H, $[(CH_2)_{12}\text{-}]$), 1.46 (m, 2H, $[CH_2\underline{CH_2}CH_2CH_2\text{-}O\text{-}]$), 1.57 (m, 2H, $[CH_2\underline{CH_2}CH_2\text{-}O\text{-}]$), 4.04 (t, 2H, $[CH_2\underline{CH_2}\text{-}O\text{-}]$), 5.7 (d.d, H, $[H_a\underline{H_b}C=CH_c\text{-}COO\text{-}]$), 6.02 (m, H, $[H_aH_bC=C\underline{H_c}\text{-}COO\text{-}]$), 6.28 (d.d, [H, $\underline{H_a}H_bC=CH_c\text{-}COO\text{-}]$).

b) Octadecyl acrylate (I_b) $CH_2=CH\text{-}COO\text{-}CH_2\text{-}(CH_2)_{16}\text{-}CH_3$, white solid (m.p. 41-42°C), (yield = 90%). The IR spectrum showed the following characteristic bands: 2915, 2848 cm^{-1} ($\nu_{C\text{-}H}$ aliphatic fatty chain), 1727cm^{-1} ($\nu_{C=O}$ stretching of ester), 1635cm^{-1} ($\nu_{C=C}$ olefinic), and 1185 cm^{-1} ($\nu_{C\text{-}O\text{-}C}$ stretching) **Fig. 2a**. On the other hand, ^{1}H NMR gives δ (ppm): 0.9 (t, 3H, $[\underline{CH_3}\text{-}CH_2\text{-}]$), 1.28 (s, 28H, $[(CH_2)_{14}\text{-}]$), 1.59 (m, 2H, $[CH_2\underline{CH_2}CH_2CH_2\text{-}O\text{-}]$), 1.68 (m, 2H, $[CH_2\underline{CH_2}CH_2\text{-}O\text{-}]$), 4.17 (t, 2H, $[CH_2\underline{CH_2}\text{-}O\text{-}]$), 5.84 (d.d, H, $[H_a\underline{H_b}C=CH_c\text{-}COO\text{-}]$), 6.15 (m, H, $[H_aH_bC=C\underline{H_c}\text{-}COO\text{-}]$), 6.42 (d.d, H, $[\underline{H_a}H_bC=CH_c\text{-}COO\text{-}]$) **Fig. 2b**.

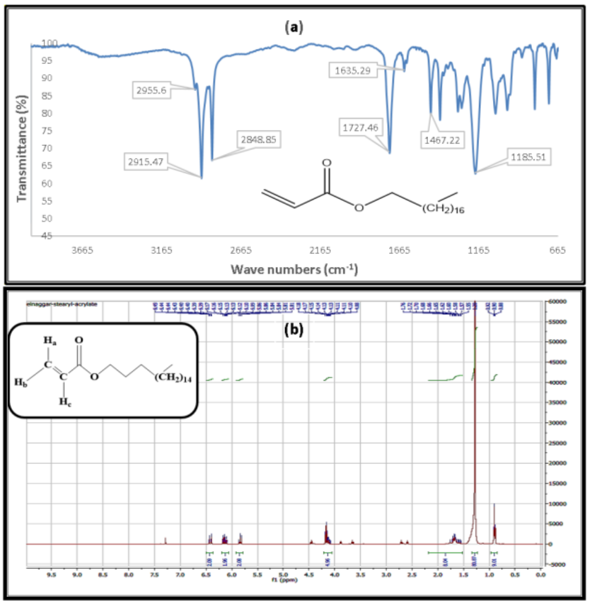


Fig. 2 a & b: IR and ^{1}H NMR spectra of octadecyl acrylate

c) Octadec-9-enyl acrylate (I_c) $CH_2=CH\text{-}COO\text{-}CH_2\text{-}(CH_2)_7\text{-}CH=CH\text{-}(CH_2)_7\text{-}CH_3$, yellowish brown liquid, (yield = 90%). It shows characteristic IR bands at 3004 cm^{-1} ($\nu_{=C\text{-}H}$, olefinic), 2925, 2854 cm^{-1} ($\nu_{C\text{-}H}$, aliphatic fatty chain), 1729 cm^{-1} ($\nu_{C=O}$ of ester), 1636 cm^{-1} ($\nu_{C=C}$, olefinic), and 1189.7 cm^{-1} ($\nu_{C\text{-}O\text{-}C}$). On the other hand, ^{1}H NMR gives δ (ppm): 0.77 (t, 3H, [CH_3 terminal]), 1.16 (s, 20H, $[\underline{(CH_2)_6}\text{-}CH_2\text{-}CH=CH\text{-}CH_2\text{-}\underline{(CH_2)_4}]$), 1.52 (m, 2H, $[CH_2\underline{CH_2}CH_2CH_2\text{-}O\text{-}]$), 1.9 (m, 2H, $[CH_2\underline{CH_2}CH_2\text{-}O\text{-}]$), 2.53 (m, 4H, $[CH_2\underline{CH_2}CH=CH\text{-}\underline{CH_2}CH_2]$), 3.99 (t, 2H, $[CH_2\underline{CH_2}\text{-}O\text{-}]$), 5.2 (m, 2H, $[CH_2\underline{CH}=\underline{CH}CH_2]$), 5.64 (d.d, H, $[H_a\underline{H_b}C=CH_c\text{-}COO\text{-}]$), 5.96 (m, H, $[H_aH_bC=C\underline{H_c}\text{-}COO\text{-}]$), 6.22 (d.d, H, $[\underline{H_a}H_bC=CH_c\text{-}COO\text{-}]$).

d) Mixed alkyl acrylate (I_d) yellowish brown liquid, (yield = 91%) shows characteristic IR bands at 3003cm^{-1} ($\nu_{=C\text{-}H}$, olefinic), 2924, 2854 cm^{-1} ($\nu_{C\text{-}H}$, aliphatic fatty chain), 1729 cm^{-1} ($\nu_{C=O}$ of ester), 1636 cm^{-1} ($\nu_{C=C}$, olefinic), and 1189.68 cm^{-1} ($\nu_{C\text{-}O\text{-}C}$).

B) Spectroscopic analysis of anionic surfactants

The structures of the synthesized surfactants were characterized by FTIR and ^{1}H NMR analysis as mentioned below:

a) Sodium 1-(hexadecyl oxy)-1-oxopropane-3-sulfonate, SCAS, white powder, (m.p. 190-191 °C), (yield = 80%). FT-IR (KBr pellet) shows characteristic bands at 2956, 2918 and 2850 cm^{-1} ($\nu_{C\text{-}H}$, aliphatic fatty chain), 1724.6 cm^{-1} ($\nu_{C=O}$ of ester), 1471.34 cm^{-1} ($\nu_{C\text{-}H}$ bending of CH_2 group), 1192.61 cm^{-1} ($\nu_{C\text{-}O\text{-}C}$), and 1270 cm^{-1} ($\nu_{SO3}$ stretching). Furthermore, ^{1}H NMR gives δ (ppm): 0.87 (t, 3H, [CH_3 terminal]), 1.25 (s, 24H, [$(CH_2)_{12}$- chain]), 1.56 (m, 2H, [$CH_2\underline{CH_2}CH_2$-O-]), 2.65 (t, 2H, [-O-CO-$\underline{CH_2}CH_2SO_3Na$]), 4.00 (t, 2H, [-O-CO-$CH_2\underline{CH_2}$ SO_3Na]), 4.20 (t, 2H, [$CH_2\underline{CH_2}$-O-]).

b) Sodium 1-(octadecyl oxy)-1-oxopropane-3-sulfonate, SSAS, white powder, (m.p. 205-207°C), (yield = 78%). FT-IR (KBr pellet) shows 2955, 2916 and 2849 cm^{-1} ($\nu_{C\text{-}H}$, aliphatic fatty chain), 1723 cm^{-1} ($\nu_{C=O}$ of ester), 1469 cm^{-1} ($\nu_{C\text{-}H}$ bending of CH_2 group), 1188 cm^{-1} ($\nu_{C\text{-}O\text{-}C}$) and 1266 cm^{-1} ($\nu_{SO3}$ stretching). On the other hand, ^{1}H NMR gives δ (ppm): 0.86 (t, 3H, [CH_3 terminal]), 1.24 (s, 28H, [$(CH_2)_{14}$- chain]), 1.56 (m, 2H, [$CH_2\underline{CH_2}CH_2$-O-]), 2.63 (t, 2H, [-O-CO-$\underline{CH_2}CH_2SO_3Na$]), 4.03 (t, 2H, [-O-CO-$CH_2\underline{CH_2}SO_3Na$]), 4.21 (t, 2H, [$CH_2\underline{CH_2}$-O-]).

c) Sodium 1-((3-sulfonato propanoyl) oxy) octadecane-10-sulfonate, SOAS, white powder, (m.p. 220-222 °C), (yield = 82%). FT-IR (KBr pellet) shows 2955, 2920 and 2851 cm^{-1} ($\nu_{C\text{-}H}$, aliphatic fatty chain), 1725 cm^{-1} ($\nu_{C=O}$ of ester), 1467 cm^{-1} ($\nu_{C\text{-}H}$ bending of CH_2 group), 1182 cm^{-1} ($\nu_{C\text{-}O\text{-}C}$) and 1266 cm^{-1} ($\nu_{SO3}$ stretching) **Fig. 3a**. Furthermore, ^{1}H NMR gives δ (ppm): 0.86 (t, 3H, [CH_3 terminal]), 1.24 (s, 22H, $\underline{[(CH_2)_6}$-$CH_2CH(SO_3Na)CH_2\underline{(CH_2)_5}$]), 1.55 (m, 2H, [$CH_2\underline{CH_2}CH_2$-O-]), 1.98 (m, 4H, [$CH_2\underline{CH_2}CH$- $(SO_3Na)\underline{CH_2}CH_2$]), 2.54 (t, 2H, [-O-CO-$\underline{CH_2}CH_2$ SO_3Na]), 2.66 (m, H, [$CH_2\underline{CH}(SO_3Na)CH_2$]), 3.98 (t, 2H, [-O-CO-$CH_2\underline{CH_2}SO_3Na$]), 4.21 (t, 2H, [$CH_2\underline{CH_2}$-O-]) and 5.3 (m, 2H, [$CH_2\underline{CH}$=$\underline{CH}CH_2$] of olefinic bond in fatty chain) where the addition of $NaHSO_3$ on fatty olefinic double bond is partial **Fig. 3b**.

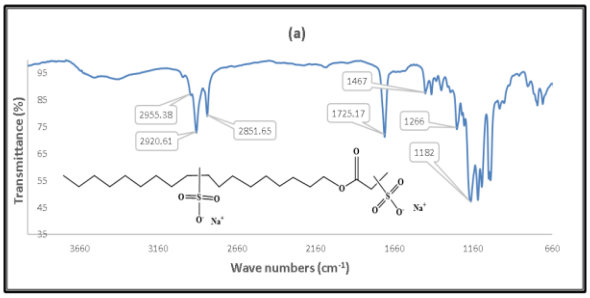
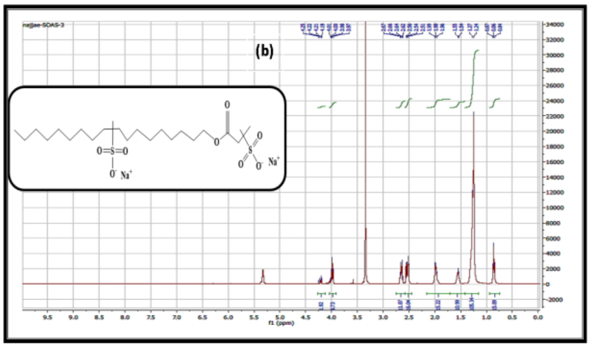


Fig. 3 a & b: IR and ^{1}H-NMR spectra of sodium 1-((3-sulfonatopropanoyl)oxy)octadecane-10-sulfonate (SOAS)



d) Mixed anionic surfactant, SMAS$_{Peach}$, white powder, (m.p. 205-213 °C), (yield = 81%). FT-IR (KBr pellet) shows 2954, 2917 and 2850 cm^{-1} ($\nu_{C\text{-}H}$, aliphatic fatty chain), 1723 cm^{-1} ($\nu_{C=O}$ of ester), 1469cm^{-1} ($\nu_{C\text{-}H}$ bending of CH_2 group), 1178.86 cm^{-1} ($\nu_{C\text{-}O\text{-}C}$ stretching), and 1266.83 cm^{-1} (ν_{SO3} stretching).

To evaluate the performance of these anionic surfactants and the possibility of their utilization in industrial fields, we studied their surface, thermodynamic properties and biological activities.

3) Surface active properties

Surface characteristics of the synthesized anionic surfactants were investigated such as surface and interfacial tension, emulsion stability, foam stability, krafft point and stability to hydrolysis in acidic or basic media. The data were represented in **Table 2** and **3**. Furthermore thermodynamic parameters of the prepared surfactants were calculated and listed in **Table 4** and **5**.

a) Surface and interfacial tension

From **Table 2**, we found that by increasing the hydrophobicity of the prepared compounds, the solubility of the surfactant decreases, leading to increasing of the surface tension of water [6].

Also the interfacial tension values were recorded in **Table 2**, that discuss the decrease of the interfacial tension by increasing the chain length of the surfactant molecule [32], due to increasing the hydrophobicity of molecule which increases its migration to the interface between aqueous media and oily layers to reduce the system free energy. However, the SOAS molecule showed the highest interfacial tension due to its owning of two polar groups that increase its solubility in water, and the molecules prefer the presence in the bulk of water than adsorption at the interface.

b) Krafft point (T_K)

The (T_K) data were measured and showed in **Table 2**. It indicates that the SCAS & SSAS surfactants have a relatively low Krafft point. On the other hand, the SOAS & SMAS$_{Peach}$ surfactants have the very lowest values (less than 0 °C). By comparing, we can say that the SOAS & SMAS$_{Peach}$ surfactants can give good applications and surface properties below 0°C.

c) Emulsion stability

Table 2: Surface active properties of anionic surfactants

Compounds	**Surface tension 0.1% (mN/m)**	**Interfacial tension 0.1% (mN/m)**	**Krafft point 0.1% (°C)**	**Emulsion stability 10 m moles (sec.)**	**Stability to acid hydrolysis (min. : sec.)**	**Stability to base hydrolysis (min. : sec.)**
SCAS	37	12	22	54	5 : 00	31 : 12
SSAS	38	14	22	63	2 : 30	22 : 42
SOAS	37.5	16	<0	50	32 : 27	> 6 h
SMAS$_{Peach}$	40	8	<0	59	26 : 15	68 : 00

The results of this property were represented in **Table 2**, it can be seen that the prepared surfactants have low emulsifying stability and can be used as demulsifying agents. Also the SOAS surfactant has the lowest value due to containing two hydrophilic groups that increase the solubility of the molecule in aqueous media and facilitate the separation of this surfactant from emulsion layer to the aqueous phase. Likewise, the SMAS$_{Peach}$ molecule has the disparate value as a result of this surfactant is considered as a mixture of these surfactants.

d) Foaming properties

The foam stability and foam volume at different temperatures of the prepared anionic surfactants were studied and given in **Table 3** and **Fig. 4**.

Table 3: Foamability at different time of prepared surfactants

Surfactant type	**Foam volume (0.1%) (ml) (28 °C)**		
	(0) min.	**(3) min.**	**(5) min.**
SCAS	280	180	175
SSAS	270	220	160
SOAS	610	560	540
SMAS$_{Peach}$	700	680	680

As known that the anionic surfactants have high foaming properties. Also, our prepared surfactants give excellent foaming properties. From **Table 3**, we can notice that compounds SOAS and SMAS$_{Peach}$ give the highest foam volume comparing with surfactants derived from saturated fatty acids SCAS & SSAS, this may be attributed to their ownership of the higher hydrophilicity which increase the surfactant solubility, subsequently; the foamability increases. Moreover, the foam stability of the prepared surfactants was studied by measuring of the alteration in foam volume with time. The collected data showed that the synthesized surfactants give good foam volume and stability due to the foam volume slightly decrease with time.

Over and above, we studied the relation between foam volume and temperature. From **Fig. 4** we can notice that all prepared compounds show higher foam volume at elevated temperatures as compared with room temperature.

As long as the temperature increases, the foam volume increases due to increasing the surfactant solubility as studied before [33].

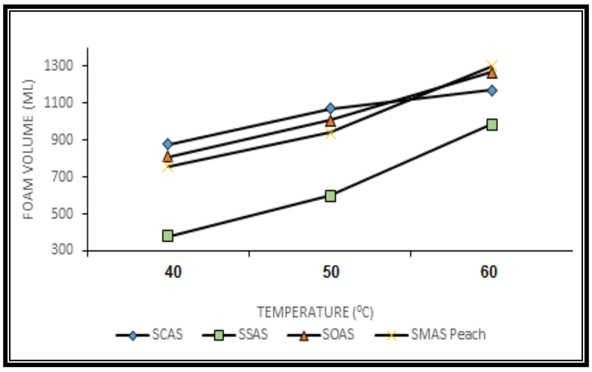


Fig. 4: Foam volume at different temperatures of prepared surfactants

By evaluation of the data represented in **Table 3** and **Fig. 4**, we can generally say that all prepared anionic surfactants give excellent foaming properties and can be applied as detergents in industrial fields.

e) Stability to hydrolysis

All the synthesized anionic surfactants were tested towards acidic and basic media. The data were listed in **Table 2**, and showed weak resistance in acidic and basic medium, except the surfactant derived from octadec-9-en-1-ol, which shows high resistance in a basic medium. This weak resistance attributed to the hydrolysis of ester linkage of the surfactant. On the other hand, they have more stability in basic media than acidic media, due to the sulphuric acid solution can hydrolyse the ester group in addition to the acidification process of the sodium sulphonato group of the molecule and precipitated from the aqueous phase.

f) Critical micelle concentration (CMC)

The values of critical micelle concentration of the prepared anionic surfactants have been appointed from the slopes of the graphs between surface tension (γ) of aqueous surfactant solutions and their Ln conc. in mol/L **Fig. 5**.

By examining the data in **Table 4**, we found that the SCAS and SSAS surfactants derived from saturated fatty alcohols have higher CMC values than SOAS and SMAS$_{Peach}$ surfactants derived from unsaturated fatty alcohols and mixed fatty alcohols prepared from *Peach* oil. As well, the SOAS has the lowest CMC value between all the examined surfactants and this may be attributed to its possession two hydrophilic groups that increase the solubility and decrease the migration to the interface [6].

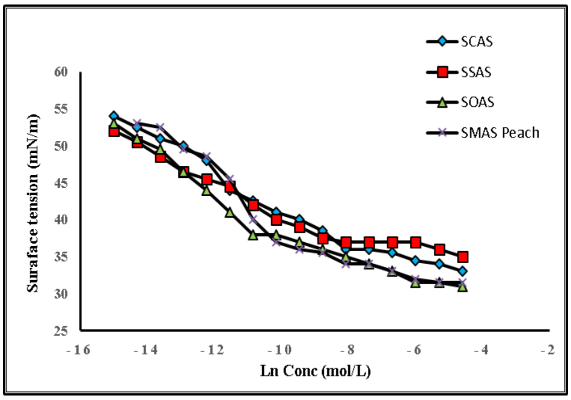


Fig. 5: Variation of surface tension versus Ln concentrations of anionic surfactants in aqueous solution at 301 K

Another effort to study the effect of temperature on the CMC values, we measure the CMC values of the SCAS compound as an example of these surfactants at 40, 50 and 60 °C.

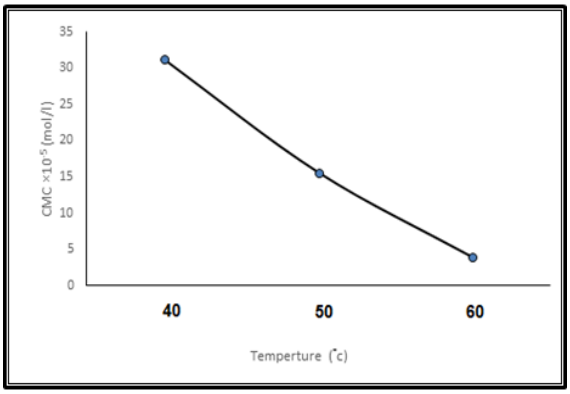


Fig. 6: Effect of temperature on CMC values of (SCAS) anionic surfactant

From **Fig. 6** we can notice that, by rising the temperatures of the surfactant solutions, the CMC values decrease, and this may be attributed to the decreasing of the hydration of hydrophilic group, which prefer micellization [34].

g) Efficiency (PC$_{20}$)

PC$_{20}$ is the required concentration to decrease the water surface tension by 20 mN/m [35]. The data were computed and represented in **Table 4**. From these results, it was found that the efficiency values are convergent from each other and there is no big difference was noticed.

h) Surface pressure (π_{cmc})

The surface pressure is an appropriate measure of the surfactant effectiveness in decreasing of the water surface tension, the values of the surface pressure were calculated in (mN/m) by (Eq. (1)):

$$\pi_{cmc} = \gamma_o - \gamma_{cmc} \qquad \textbf{(1)}$$

Where, γ_o is the distilled water surface tension and γ_{cmc} is the surfactant solution surface tension at CMC. The most efficient synthesized surfactant is one which gives the maximum decrease of the surface tension at the CMC [36]. By screening the results in **Table 4**, it was obvious that the compound SMAS$_{Peach}$ has a greater effectiveness in the reduction of the surface tension at CMC.

i) Maximum surface excess (Γ_{max})

It is defined as the maximum concentration of surfactant molecules that existed at the air/water interface per unit area, which depends on the surfactant solution temperature and hydrophobicity moiety. The maximum surface excess (Γ_{max}) values have been obtained in mol/cm^2 by using Gibb's equation [38].

$$\Gamma_{max} = - [1/2.303nRT]\ [\delta\gamma/\delta logC]_T \qquad \textbf{(2)}$$

Where, n is the number of solute species of surfactant molecules, R is the universal gas constant equal to 8.314 Jmol^{-1}K^{-1}, T is the absolute temperature in Kelvin and ($\delta\gamma/\delta$logC) is the slope of the graph below the CMC value in the surface tension versus Ln (c) plot.

By detection the results in **Table 4**, we found that the Γ_{max} data shifted to lower values when the carbon chain length of prepared anionic surfactant increases, as a result of increasing of the hydrophobicity of the surfactant molecules and directed towards the interface quickly to reduce the surface energy of the system. On the contrary, by rising the temperatures, the Γ_{max} values increase as represented in **Table 5** due to increasing the repulsion forces with the aqueous media lead to increasing of the thermal motion and the surfactant molecules migrated to the interface faster followed by more packing of adsorbed molecules at the interface.

j) Minimum surface area (A_{min})

It is the average area exploited by each surfactant molecule adsorbed at the air/water interface in square angstrom [38]. The A$_{min}$ data of prepared anionic surfactants shown in **Table 4 & 5**, have been computed by Gibb's equation (3) [37].

$$A_{min} = 10^{16}/\Gamma_{max}\ N_A \qquad \textbf{(3)}$$

Table 4: Surface and thermodynamic parameters of the prepared anionic surfactants

Compounds	CMC × 10^{-5} (mol/L)	γ_{cmc} (mN/m)	PC_{20} × 10^{-7} (mol/L)	π_{cmc} (mN/m)	Γ_{max} × 10^{-11} (mol cm^{-2})	A_{min} (A^{o2}/molecule)	ΔG°_{mic} (KJ mol^{-1})	ΔG°_{ads} (KJ mol^{-1})
SCAS	31.25	36	7.68	33.5	5.11	325.20	-20.993	-21.649
SSAS	15.63	37.5	3.05	32	3.93	422.14	-22.796	-23.609
SOAS	1.95	38	4.30	34	4.84	342.98	-27.123	-27.825
$SMAS_{Peach}$	3.91	37	14.13	35	5.29	314.03	-25.389	-26.051

Where, N_A is Avogadro's number equal to (6.023 × 10^{23}molecule mol^{-1}). The values listed in **Table 4** which reflects that the A_{min} values increase with increasing the hydrophobic chain length, due to the presence of the balance between repulsion forces between polar head groups and Van der Waals forces among hydrophobic chains [39], and can also be explained by the increasing of hydrophobicity of the surfactant molecule lead to increasing of its volume that occupy a large area at the interface. On the other hand, when we studied the effect of temperature on the A_{min} value, we found from the data listed in **Table 5** that the A_{min} value decreases with increasing the temperature that decreases the hydration (repulsion forces) between hydrophilic groups [34] and increases the thermal motion, also it can be vertical to the interface and occupy less area, where it is difficult to be tucked.

k) Micellization and adsorption thermodynamic parameters

The micellization and adsorption thermodynamic parameters of the prepared anionic surfactants were studied at 40, 50 and 60 °C and computed using Gibb's adsorption equations [36]:

$$\Delta G^{\circ}_{mic} = RT \ln CMC \qquad \textbf{(4)}$$

$$\Delta G^{\circ}_{ads} = \Delta G^{\circ}_{mic} - [0.06023 \ x \ \pi_{cmc} \ x \ A_{min}] \qquad \textbf{(5)}$$

$$\Delta S_{mic} = -d\ (\Delta G^{\circ}_{mic}/\Delta T) \qquad \textbf{(6)}$$

$$\Delta S_{ads} = -d\ (\Delta G^{\circ}_{ads}/\Delta T) \qquad \textbf{(7)}$$

$$\Delta H_{mic} = \Delta G^{\circ}_{mic} + T\Delta S_{mic} \qquad \textbf{(8)}$$

$$\Delta H_{ads} = \Delta G^{\circ}_{ads} + T\Delta S_{ads} \qquad \textbf{(9)}$$

Where, (ΔG°_{ads}) is adsorption free energy and (ΔG°_{mic}) is micellization free energy. The adsorption and micellization parameters were represented in **Table 4 and 5**.

All the micellization and adsorption free energy (ΔG_{ads} & ΔG_{mic}) values are negative, reflect that the micellization and adsorption are spontaneous processes. The ΔG_{ads} values are more negative than the values of ΔG_{mic} at the same hydrocarbon chain length and temperature, which indicate that the surfactant molecule prefers the adsorption process at the interface.

To study the thermodynamic parameters of the prepared sulfonated anionic surfactants we choose the SCAS compound as an example. From the thermodynamic data recorded in **Table 5**, we can conclude that the formation of micelles becomes more easily at 60 °C due to the greater positive value of ΔS_{mic} for the tested surfactant and the action is reversed at 50 °C, where the value of ΔS_{ads} become more positive [40]. On the other hand, the ΔS_{mic} and ΔS_{ads} values increase with increasing the temperature. Furthermore, the micellization and adsorption enthalpies data of the prepared surfactants were calculated and listed in **Table 5**, the results of this parameter become higher on the raising of the solution temperature at the same chain length due to increasing the total heat content of the system according to [Eqs. 8, 9].

4) Antimicrobial activity against sulfate reducing bacteria of the synthesized anionic surfactants

The cytoplasmic membrane of sulfate-reducing bacteria (**SRB**) is composed of phospholipids and proteins that considered as the essential ingredients where, the phosphatidylethanolamine is the main component of cytoplasmic membrane (neutral at physiological pH), also cardiolipin and phosphatidy-l-glycerol are the main acidic components (negatively charged at physiological pH) [41].

Table 5: The micellization and adsorption thermodynamic parameters of the SCAS anionic surfactant

Comp.	Temp. °C	CMC × 10^{-5} mol/L	γ_{cmc} mN/m	π_{cmc} mN/m	Γ_{max} × 10^{-11} mol/cm^2	A_{min} A^{o2}/molecule	ΔG°_{mic} KJ/mol	ΔG°_{ads} KJ/mol	ΔS_{mic} kJ/mol.k	ΔS_{ads} kJ/mol.k	ΔH_{mic} KJ/mol	ΔH_{ads} KJ/mol
SCAS	40	31.25	36	33.5	5.11	325.20	-20.993	-21.649	-	-	-	-
	50	15.625	35.5	32.5	4.166	398.575	-23.524	-24.304	0.253	0.266	58.227	61.452
	60	3.906	36	30	10.427	159.231	-28.088	-28.376	0.456	0.407	123.893	107.222

By reading the data given in **Table 6**, we found that SOAS surfactant is the most effective antibacterial agent against SRB (IZD = 24 mm/mg sample) followed by $SMAS_{Peach}$ surfactant that gives considerable and promising antibacterial activity (IZD = 18 mm/mg sample) and SCAS surfactant that shows (IZD = 14 mm/mg sample) then SSAS surfactant that gives (IZD = 12 mm/mg sample) as compared to the standard ampicillin.

Table 6: The biocidal effect of prepared anionic surfactants against sulfate reducing bacteria (SRB)

Sample	**Inhibition zone diameter (IZD) (mm/mg sample) Desulfomonas pigra (G -)**	**Inhibition efficiency (%)**
<u>Control</u>: DMSO	0	-
<u>Standard</u>: Ampicillin	20	-
SCAS	14	70
SSAS	12	60
SOAS	24	120
$SMAS_{Peach}$	18	90

The excellent inhibition efficiency of SOAS and $SMAS_{Peach}$ surfactants may be due to their containing of two sodium ions of the hydrophilic head that have ability to replace the proton of sulfhydryl or thiol (SH) groups on the protein surface of the cell membrane [42,43] inactivating it which lead to disruption of the permeability of the membrane and finally causing cellular death. On the other hand, the SCAS and SSAS surfactants exhibit good inhibition efficiency due to their containing of only one sodium cation. The destructive action of the SRB may be attributed to the surfactant adsorption process at the cytoplasmic membrane, at which the lipophilic tail of the surfactant can dissolve and penetrate the lipid bi-layer of SRB cytoplasmic membrane which creates large gaps in the structure of the membrane leading to uncontrolled permeability of essential components through the membrane, and finally cell death [21]. These bacteria are considered as a production source of H_2S that works as a corrosive agent for the petroleum pipes. So, the suppression of this microbial corrosion growth of SRB can be performed by the utilization of these prepared surfactants that may be evaluated as new categories of biocides in the petroleum field.

IV. CONCLUSION

New families of anionic surfactants were synthesized from oily matter derived from *Peach* kernel, and their structures were confirmed by spectroscopic tools such as IR and ^1H NMR spectra. The synthesized anionic surfactants elucidated good surface activities and show adsorption tendency at the interface more than micellization process according to the free energy of micellization and adsorption. Finally, all the surfactants under study show high antimicrobial activity against SRB especially SOAS indicates the best inhibition efficiency reached to (120%). From this point, we can say that these surfactants can be used as antimicrobial compounds against sulfate reducing bacteria in petroleum field to prevent corrosion problems that cause enormous economic losses.

ACKNOWLEDGMENT

The authors are grateful too much for the Faculty of Science, Benha University, Benha, Egypt for the financial support.

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