Journal of Basic and Environmental Sciences, 6 (2019) 112-120

ISSN
Online: 2356-6388
Print: 2536-9202

Research paper Open Access

Synthesis and evaluation of surface and thermodynamic properties of soybean Gums based cationic surfactant

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Abstract:

Cationic surfactant based on the acid profile of soybean gums is synthesized by the reaction with polyethylene glycol than with chloroacetic acid followed by quaternization reaction with pyridine and the final cationic surfactant were isolated. Soy Gums was extracted by water degumming. The Extraction process was carried out in wadigroup / soya oil plant, and the fatty acid characterization and composition were determined using Gas-Liquid chromatography. The chemical structures of the synthesized surfactant were confirmed by FTIR and ^{1}HNMR spectroscopic tool. And the surface and thermodynamic properties were determined and the tendency towards adsorption and micellization was concluded.

Keywords: Soya bean Gums, cationic surfactant, nonionic surfactant, Surface properties, and thermodynamic properties.

Received; 15 Nov. 2018, Revised form; 28 Dec. 2018, Accepted; 28 Dec. 2018, Available online 1 July 2019

1. Introduction:

The crude soybean oil contains from 1.5-2.5 % phospholipids (Gums) [45] wish was extracted using water degumming technique [43-44]. Further utilization of soy gums will be economically beneficial as the soya bean oil is produced in large-scale production. The soybean oil waste is a good renewable resource of fatty acids while the inexpensive nontoxic nature of the extracted soy gums makes it a very economically valid method to obtain a useful surfactant. A wide variety of a neglected uses oil processing by-products can be used in synthesizing a new useful surfactant such as Rice bran [3], Mangifera seeds [4], Guava seeds [5] and Al-cedre seeds [6].

The great Uses of surfactant are because the double functionality arises from the presence of both parts (Hydrophilic and Hydrophobic) in the same molecule [16, 17].

Measuring Critical Micelle concentration for a surfactant will be used as an indication of the minimum required quantity of a surfactant to be used effectively. For concentrations After CMC, an additional amount of surfactant will be self-aggregated forming micelles [7]. There are a wide variety of Surfactant's applications starting with detergents, textile processing, coatings formulation, passing by petroleum industry such as drilling mud and petrochemical recovery, also can be used in paints, demulsification, dispersing agents and corrosion inhibitors [5,8,9].

They are chemical compounds that control, reduce or prevent reactions between metal and its surroundings when added to the medium in small quantities. Corrosion inhibitors are used to minimize the rate of corrosion and prohibit the metal from corrosion [10, 11 12]. Other common application in petroleum industry is that Surfactant are widely used for collecting and dispersing the thin films of petroleum on water surface [15,19,21,23]. Ionic surfactant containing hydroxyl groups have a great potential to collect and disperse the fluffy petroleum films from the water reservoirs surface [13,14]. Moreover, quaternary ammonium salts or complexes based on fatty acids and nitrogen-containing compounds

have a high activity in collecting and dispersing the spilled crude oil [34-40].

In this research, a series of cationic surfactant were synthesized mixed together with ratios based on the fatty acid profile of the soybean Gums. The surface and thermodynamic properties will be exhibited, and the efficiency will be evaluated after interpreting the obtained results.

2. Experimental

2.1. Materials

Soy Gums were brought from a soybean oil extraction plant, out of the water degumming unit, Egypt. 9-octadecenoic acid, Chloroacetic Acid, polyethylene glycol-600, polyethylene glycol-400, and p-Toluene sulfonic acid were purchased from Sigma-

Aldrich Chemicals Co, xylene, Acetone, Methanol and sodium sulfate anhydrous were purchased from AL-Nasr Chemicals Company.

2.2. The composition of Fatty acids of Soybean gums and its specification

Soybean gums were obtained by water degumming method in the soybean oil extraction plant. The fatty acids composition of soybean gums as in **Table 1,** was performed as fatty acid methyl esters **[**20] using GLC (**Fig. 1**).

2.3. Synthesis of fatty acids monoester

A mixture of (0.1 mol.) 9-octadecanoic acid and (0.1 mol) polyethylene glycol-400 in presence of 0.01%p-toluene sulphonic acid as a catalyst and dry xylene as a solvent was refluxed with a Dean-Stark trap, till the calculated amount of water (0.1 mol.) was separated. The reaction mixture was neutralized with sodium carbonate and washed with distilled water twice and dried with anhydrous sodium sulfate. The solvent was distilled off [41]. The chemical structures of the synthesized monoesters were confirmed by FTIR [22].

2.4. Synthesis of fatty acids diester

A mixture of (0.05 mol) the obtained monoesters and (0.05 mol) chloroacetic acid in presence of 0.01%p-toluene sulphonic acid as a catalyst and dry xylene as a solvent was refluxed with a Dean-Stark trap, till the calculated amount of water (0.05 mol.) was separated. The reaction mixture was neutralized with sodium carbonate and washed with distilled water twice and dried with anhydrous sodium sulfate. The solvent was distilled off [41]. The chemical structures of the synthesized monoesters were confirmed by FTIR [22].

2.5. **Synthesis of cationic surfactant**

Cationic surfactant was synthesized by quaternization reaction of (0.05 mol.) the obtained di-esters with (0.05 mol.) pyridine using acetone as solvent for 96 h. The solvent was distilled off to produce cationic surfactant (scheme 1) [42]. The chemical structures of the synthesized cationic surfactant were confirmed by FTIR and ^{1}HNMR.

2.6. Surface characteristics

2.6.1. Surface Tension

The Surface tension (γ) of the freshly prepared cationic solutions with different concentrations were measured by De-Noüy ring Tensiometer (Kruss-K9). Surface tension of the used distilled water was measured before preparing the surfactant's solutions. The platinum ring was immersed in water than rinsed with acetone followed by briefly flaming to remove the remained acetone [18]. For each reading an average value of three concurrent measurements was taken.

2.6.2. Krafft point (T_K)

These temperatures were measured at which the dispersed 0.1% surfactant 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 surfactant [24].

2.6.3. Foaming properties

Powerful shaking for 10 seconds of 100 mL of the surfactant solution (0.1 wt %) in a 100 mL graduated cylinder at (28°C) was performed to produce foaming properties. The foam height was measured in mL and the foam stability was measured by the time at 28 °C [26].

2.6.4. Stability to hydrolysis

10 mL of surfactant solutions (10 mmole) and 10 mL of sodium hydroxide 0.05 N or 10 mL of sulfuric acid 2 N in phenol tube placed in a thermostat system at **40** °C. The surfactant resistance to hydrolysis was determined by the time of sample solution takes to be clouded [27].

2.7. Gas-liquid chromatography (GLC)

Mixed fatty acid methyl ester GLC analysis was performed using Perkin Elmer Auto System XL utilized with a flame ionization detector (FID), a fused silica capillary column ZB-5 (60 m × 0.32 mm I.D) and an oven which temperature was initially maintained at 150 °C with a pre-programmed gradual temperature increase from 150 to 240 °C by an increasing rate of 30 cm/min. The apparatus was operated with helium gas flow rate 1 mL/min and the temperatures of 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).

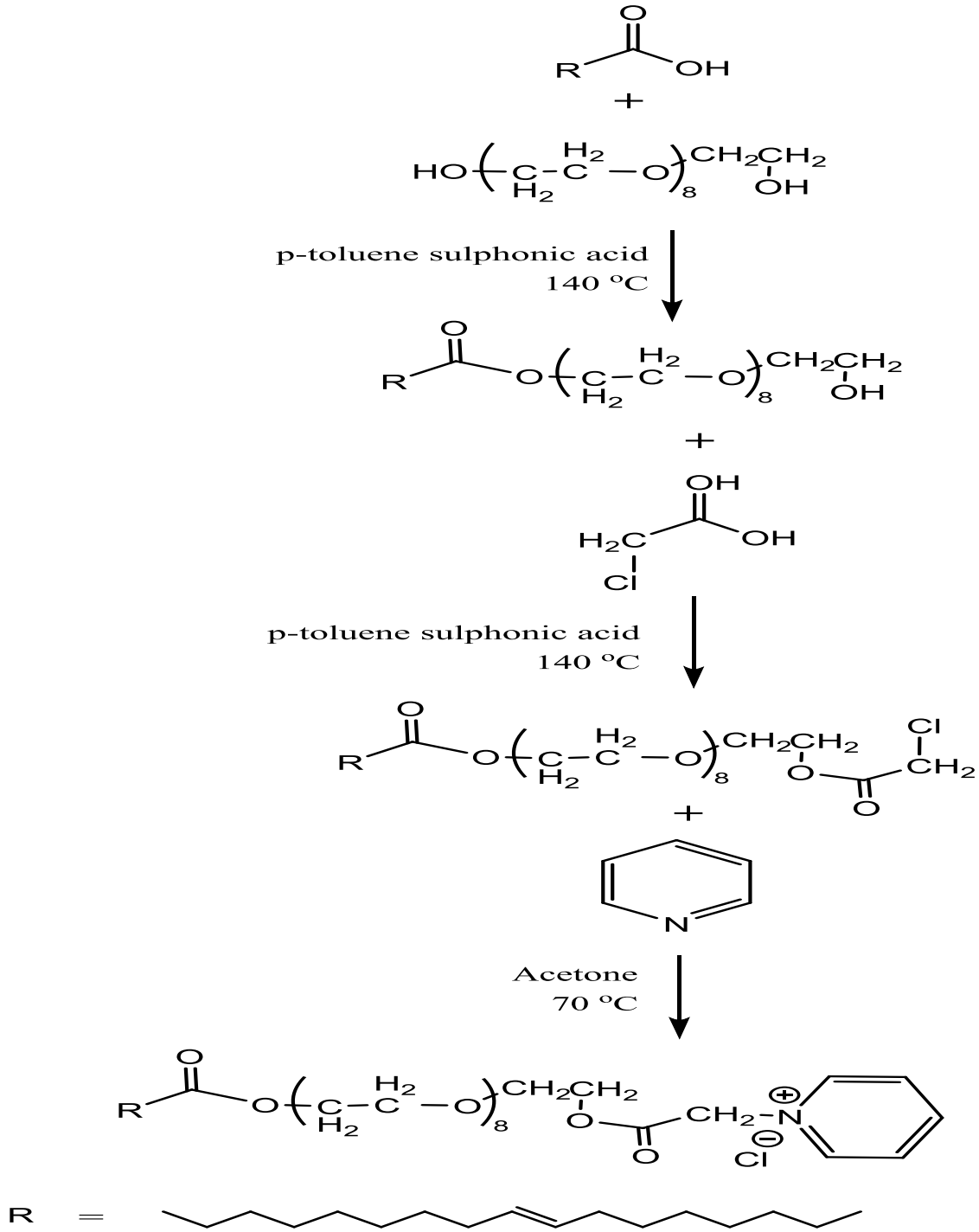
2.8. structural confirmation of the prepared compounds

2.8.1. FTIR Spectra

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).

2.8.2. ^{1}H NMR spectra

^{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.



Scheme-1

3. Results and discussions:

3.1. Determination of acid profile of Soya Gums

Soya Gums was obtained from the water degumming of soybean oil. Fatty acids composition was determined using GLC (Fig. 1) and the data was reported in Table 1. It shows that the majority of fatty acids are unsaturated fatty acids, about 82% and a small amount of saturated fatty acids. This analysis was confirmed by chemical measurement, where the oil shows high iodine value which indicates that the high percentage of unsaturated fatty acids. Four different compounds of cationic surfactant were synthesized from different fatty acids (Palmitic acid, Stearic acid, Oleic acid and Linoleic acid extracted from soya gums).

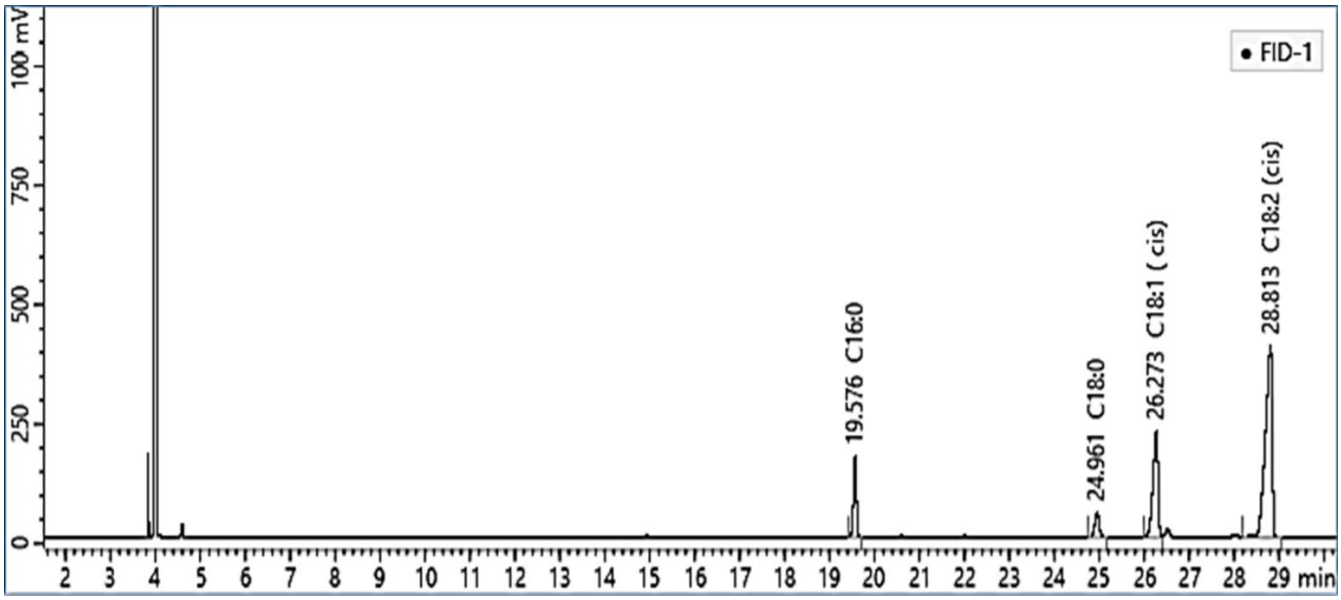


Fig (1): GLC

Table (1): Composition of Soy Gums

Fatty Acids	Percent
C16:0	13.42
C18:2	54.87
C18:1	27.41
C18:0	04.31

3.2. Characterization: -

3.2.1. The FTIR spectra of the mixed methyl ester.

The FT-IR spectra confirm the expected functional groups in the synthesized ester (Fig. 2.) by showing bands at 2926, 2854 cm^{-1} (ν_{C-H} aliphatic fatty chain), 1743 cm^{-1} ($\nu_{C=O}$ of ester), 1645 cm^{-1} ($\nu_{C=C}$ stretch aliphatic fatty chain), and 1171 cm^{-1} (ν_{C-O-C} stretching).

3.2.2. The FTIR spectra of "OS_I"

"1-((52E,54E)-2,43-dioxo-3,6,9,12,15,18,21,24,27, 30,33,36,39,42tetradecaoxahexaconta-52,54-dien-1-yl) pyridin-1-ium chloride", Brownish black Viscous liquid, (yield = 83%). The FT-IR spectra confirm the expected functional groups in the synthesized **(OS_I)** by showing bands at 2925, 2854 cm^{-1} (ν_{C-H} aliphatic fatty chain), 1735 cm^{-1} ($\nu_{C=O}$ stretching of ester), 1637cm^{-1} ($\nu_{C=C}$ olefinic), 1459 cm^{-1} ($\nu_{C=C}$ stretch aromatic ring), and 1118 cm^{-1} (ν_{C-O-C} stretching) as shown in (fig.3).

3.2.3. ^{1}H NMR spectra "OS_I"

^{1}H-NMR spectra of the synthesized cationic surfactant(E)-1-(2,34-dioxo-3,6,9,12,15,18,21,24,27, 30,33-undecaoxahenpentacont-45-en-1-yl)pyridin-1-ium chloride in $CDCl_3$, showed signals at: δ =0.88 ppm (t, 3H, C**H**$_3$-CH_2),1.21 ppm (m, 20H, C**H**$_2$$CH_3$), 2.08 (m, 4H, C**H**$_2$$CH_2$CH=CH), 5.38 ppm (m, 2H, C**H**=C**H**), 1.54 ppm (t, 2H, C**H**$_2$-CH_2-COO), 2.3 ppm (t, 2H, C**H**$_2$ -COO),3.51 ppm (t, 36H, O-C**H**$_2$-CH_2-O), 4.2 ppm (t, 4H, C**H**$_2$ OCO),6.1 ppm (s, 2H, OCO-C**H**$_2$-N^+), 8.29 & 8.67 & 9.17 ppm (Pyridinium CH), as shown in **(fig.4)**

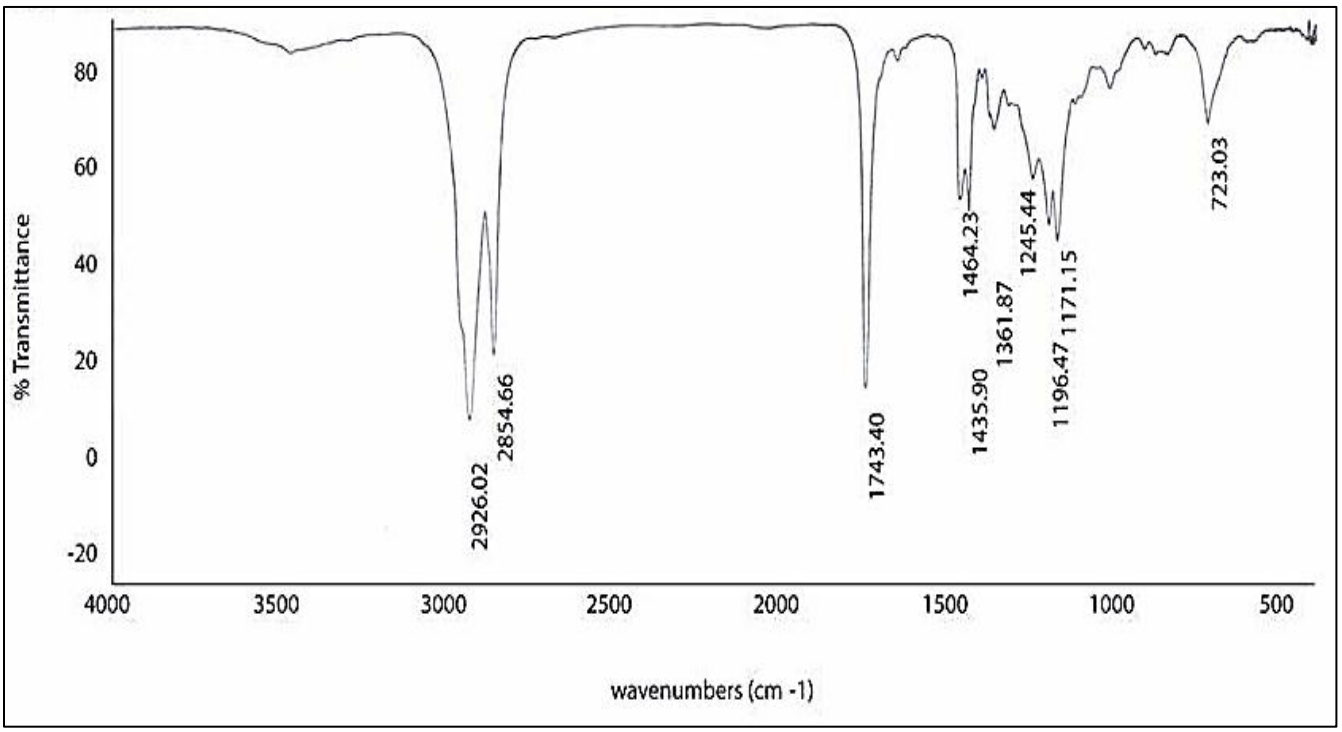


Fig (2): FTIR of Mixed methyl ester

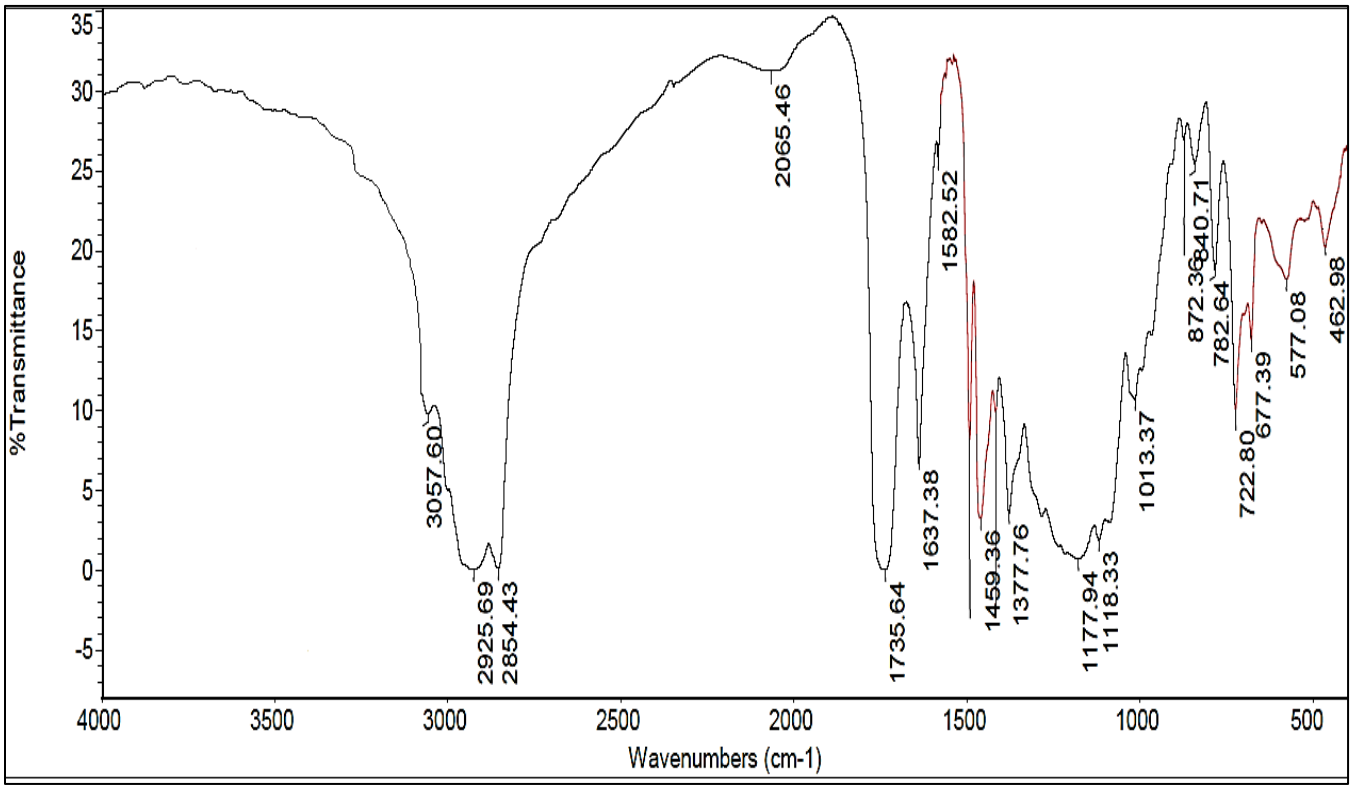


Fig (3): FTIR of "OS_I"

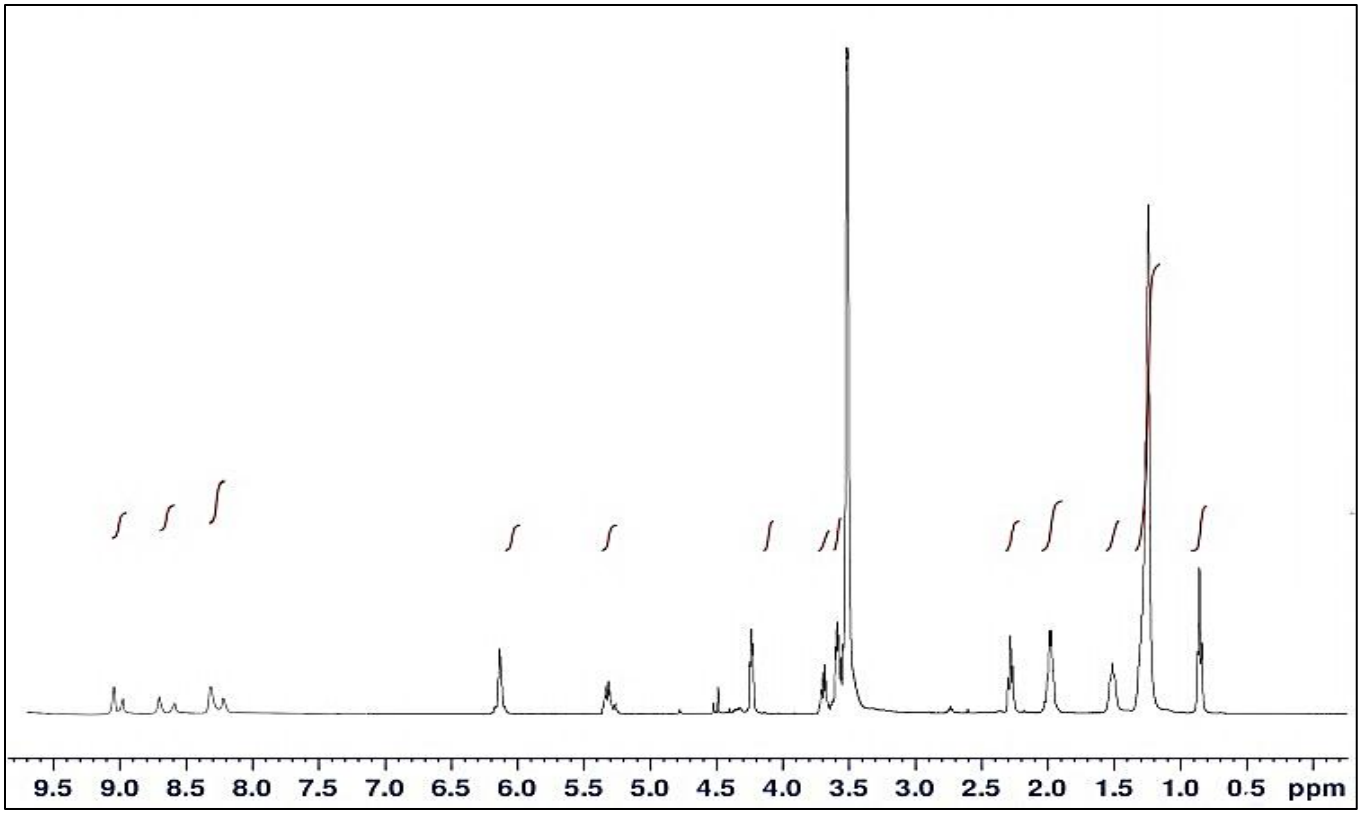


Fig (4): ^{1}HNMR of "OS_I"

3.3. Properties

3.3.1. Surface tension:

Increasing hydrophobicity leads to a decrease in solubility of the surfactant in the aqueous medium subsequently increasing in surface tension of the system [46] Table 2.

3.3.2. Critical Micelle Concentration (CMC):

The CMC Values were estimated for the different cationic surfactant molecules and the results were listed in **Table (2).** A graphical representation of surface tension (γ) of aqueous surfactant solutions and their Ln(conc) in mol/L was in Fig 5.

3.3.3. Effectiveness (π_{CMC}) and Efficiency (PC_{20})

Synthesized cationic surfactant effectiveness can be estimated from the difference between the surface tension of the distilled water (γ_o) and the surface tension of the surfactant solution corresponding to the critical micelle concentration (γ_{CMC}) listed in **Table (3)** based on the following equation.

$$\pi_{CMC} = \gamma_o - \gamma_{CMC}$$

3.3.4. Surface pressure (π_{cmc}):

The surface pressure is adequate measuring criteria to evaluate the effectiveness of the surfactant with decreasing the surface tension of the water, π_{cmc} can be calculated as follows in (mN/m) based on the following equation:

$$\pi_{cmc} = \gamma_o - \gamma_{cmc}$$

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 [33].

3.3.5. Efficiency (PC_{20})

Efficiency or PC_{20} is related to surfactant concentration required to suppress the water surface tension by 20 mN/m [32]. The data of the synthesized cationic surfactant was calculated and listed in Table 2.

3.3.6. Maximum surface excess (Γ_{max}) and minimum surface area (A_{min})

The maximum surface excess of the synthesized cationic surfactant, Γ_{max}, describes the accumulation of surfactant molecules at the air/water interface and can be calculated according to Gibb's equation [25]

$$\Gamma_{max} = 1/\ 2.303\ nRT\ (\partial\gamma/\partial \log C)$$

Where, R= gas constant (8.314 J mol^{-1} k^{-1}) and T= t+273 (°K); the value of **n** is the number of ionic species whose concentrated at the interface varies with the surfactant concentration in the solution. Γ_{max} values of the synthesized cationic surfactant were calculated at different temperatures and listed in Table (3). The average area occupied by each adsorbed molecule of a surfactant is given by the following equation [28].

$$A_{min} = 10^{14}/N_A \Gamma_{max}$$

Where N is Avogadro's number. By inspecting the data in Table (3) the minimum surface per molecule at the aqueous solution/air interface increases with increasing temperature of the measurements this means that the increase of temperature causes a decrease in the number of molecules at the interface the surface area values at the interface of the synthesized cationic surfactant molecules are gradually increased.

3.3.7. Krafft point (T_K):

Krafft point (T_K) of the synthesized cationic surfactant were measured and listed in Table 3 [30].

3.3.8. Foaming properties:

The foaming volume of the prepared cationic surfactant was listed in Table (3). The synthesized cationic surfactant shows relatively good foaming properties

3.3.9. Stability to hydrolysis

The synthesized cationic surfactant has been exposed to acidic and basic mediums to study its hydrolysis. lower stability in an acidic medium was exhibited by the synthesized surfactant, as shown in Table (3). On the other hand, all the surfactant examined under this study exhibited good stability in basic medium [31].

Table (2): Surface-active parameters of the synthesized cationic surfactant.

	CMC **10^-4 M*	*PC_{20} M/L*	*π_{cmc}* *mNm^{-1}*	*τ_{MAX}* *mol.Cm^{-2}*	*A_{min}* *Nm^2*
SO_I	2.00	13.028	35.6197	3.05787E-11	543.039

Table (3): Surface-active parameters of the synthesized cationic surfactant.

	Foam Height	*Krafft Point*	*Stability to Base Hydrolysis*	*Stability to Acid Hydrolysis*
SO_I	250	3	>180 mins	40.5 Min

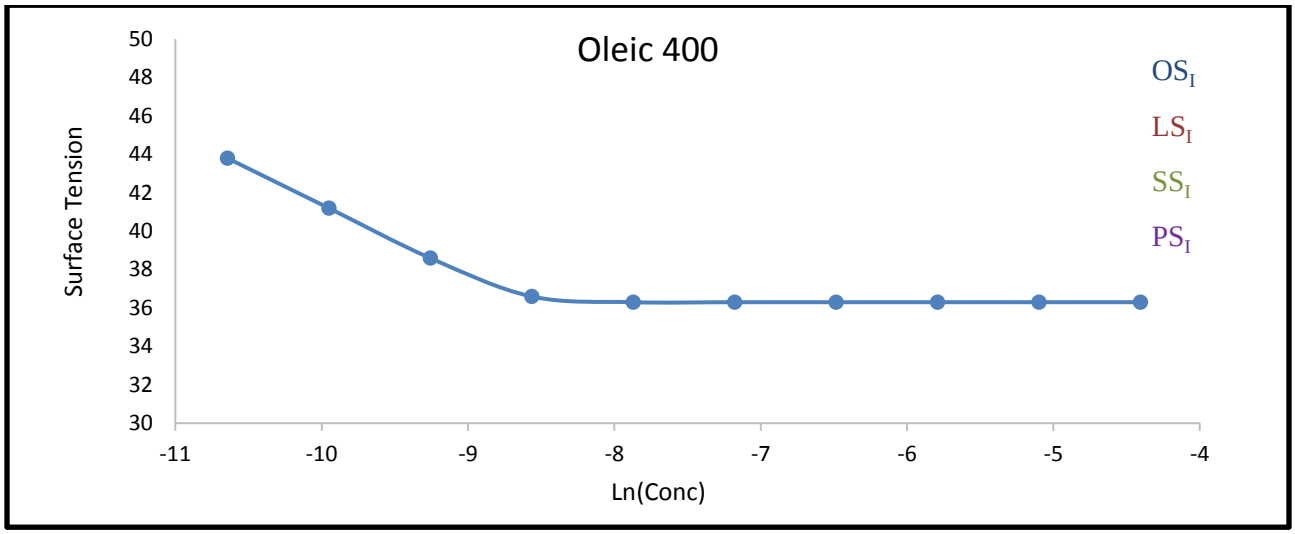


Fig (5): Surface Tension V.S Ln(conc.) of the synthesized surfactant (OS_I)

5. Acknowledgment

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

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