# Synthesis and Surface Active Properties of Gawafa Fats Based Amphoteric Surfactants

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ÖSSZEFOGLALÁS

Amfoter felületaktív anyagok sorozatát készítették tiszta (oktán $C_{8:0}$, dodekán $C_{12:0}$, tetradekán $C_{14:0}$, hexadekán $C_{16:0}$, oktadekán $C_{18:0}$, 9-oktadekanoil $C_{18:1}$) savak, valamint Gawafa magvakból extrahált kevert zsírsavak N-acil-N, N-dimetil-etilén-diaminjának 3-klór-2-hidroxi-propán-szulfonáttal, klór-ecetsavval, ill. hidrogén-peroxiddal végrehajtott reakciójával, amelynek során amino-szulfo-betaint, amido-betaint és amino-oxidot állítottak elõ. Az így kapott felületaktív anyagok szerkezetét mikro-analízissel, IR- és $H^1$NMR spektroszkópiával igazolták. Vizsgálták és értékelték a termékek felületaktív sajátságait és biológiai lebonthatóságukat is.

**Kulcsszavak:** Gawafa-zsír, amfoter felületaktív anyagok és felületaktív sajátságaik

ABSTRACT

Series of amphoteric surface active agents were prepared by the reaction of N-acyl –N, N-dimethylethylene diamine of pure [octanoic $C_{8:0}$, dodecanoic $C_{12:0}$, tetradecanoic $C_{14:0}$, hexadecanoic $C_{16:0}$, octadecanoic $C_{18:0}$, 9-octadecenoic $\mathbf{C}_{18:1}$ acids and mixed fatty acids extracted from Gawafa seeds] with 3-chloro 2-hydroxypropane sulfonate, chloroacetic acid and hydrogen peroxide to produce amino sulfobetaine, amidobetaine and amino oxide respectively. The structures of the prepared surface active agents were confirmed by micro-analysis, IR and $H^1$NMR spectra. Also, the surface active and biodegradability properties of the prepared compounds were evaluated and studied.

**Key words:** Gawafa fat, amphoteric surfactants and surface active properties.

ZUSAMMENFASSUNG

Serie von amfoteren, oberflächenaktiven Materien wurden aus N-acyl-N, N-dimethyl-Äthylen-Diamine von reinen (Octan $C_{8:0}$, Dodekan $C_{12:0}$, Tetradekan $C_{14:0}$, Hexadekan $C_{16:0}$, Octadekan $C_{18:0}$, 9-Octadekanoil $C_{18:1}$) Säuren, bzw. von aus Gawafa-fett extrahierten, gemischten Fettsären durch Reaktion mit 3-Chlor-2-Hydroxypropan-Sulfonat, Chloressigsäure oder mit Wasserstoffperoxid hergestellt. So hatte man Amino-Sulfo-Betain, Amido-Betain und Aminooxid produziert. Die Struktur der so erhaltenen oberflächenaktiven Materien wurde mit Mikroanalyse, IR- und $H^1$NMR Spektroskopie bestätigt. Die oberflächenaktiven Eigenschaften und die biologische Abbaubarkeit der Produkte wurden auch geprüft und ausgewertet.

**Schlüsselwörter:** Gawafa-fett, amfotere oberflächenaktive Materien und ihre oberflächenaktive Eigenschaften

## 1-Introduction

The surface active agents derived from natural source were acquired more valuable and interest from two points of view economic (has low price) and environmental pollution (reduced pollution). Our interest were extended to prepare the surface active agents from rubbish sources like fatty acids were extracted from both Mangifera indica [1] and rice bran oil [2]. Since Gawafa seeds have good percentage of saturated fatty acids especially octanoic, dodecanoic, hexadecanoic and octadecanoic acids and small percentage of decanoic and tetradecanoic acids c. f. Table 1. [3], it is interesting to be used as precursors for preparing different types of surface active agents.

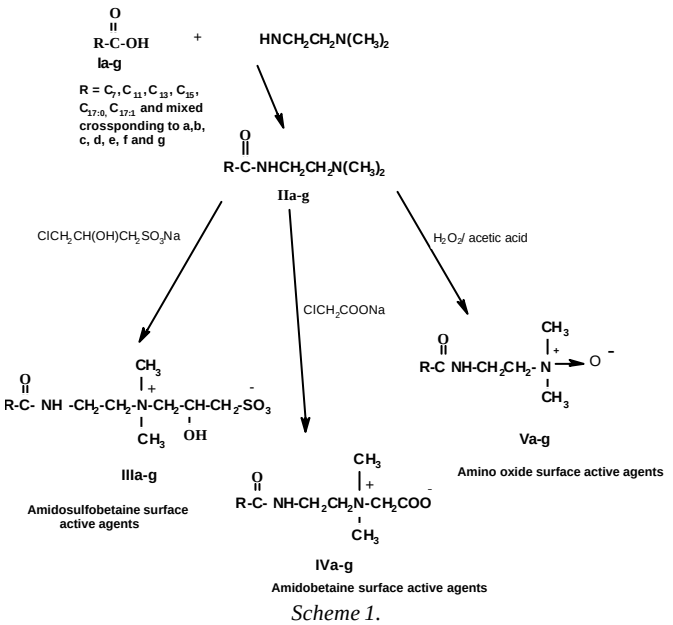
The cationic surface active agents containing in their molecule amido group [4] are used in the toilet personal care type applications rather than the amphoteric surface active agents prepared by using 2-hydroxypropane sulphate (sulfobetaines) offer the most promise as dispersant in the soap based detergents [5]. Also amine oxide may be viewed as amphoteric surface active agents; they differ from sulfobetaines in so far as the distances between the oppositely charged atoms are considered. The importance of amino oxide is acquired from using in shampoos, hair dressings and as foam stabilizers for liquid detergents [6] rather than used as synergistic fabric softeners [7].

In the extension of our interest in the preparation, study of the surface properties of surface active agents derived from natural source, we tried to prepare the amidosulfobetain, amidobetaine and amino oxide amphoteric surface active agents c. f. Scheme 1. and study their surface activity and biodegradability properties.

*Table 1:*

**Fatty Acids Composition and Chemical Characteristic of Gawafa Fat [3].**

| Chemical Characteristics | | | | Fatty Acid Composition | Peak Area % |
|---|---|---|---|---|---|
| | 1st | 2nd | 3rd | Octanoic ($C_{8:0}$) | 12.51 |
| Acid Value | 25.50 | 36.50 | 64.50 | Decanoic ($C_{10:0}$) | 01.94 |
| | | | | Dodecanoic ($C_{12:0}$) | 26.61 |
| | | | | Tetradecanoic ($C_{14:0}$) | 03.25 |
| Iodine Value | | | 11.80 | Hexadecanoic($C_{16:0}$) | 20.25 |
| Saponification Value | | | 197.80 | Octadecanoic($C_{18:0}$) | 31.00 |
| Unsaponified | | | 02.03 | 9-Octadecenoic($C_{18:1}$) | 04.44 |




*Scheme 1.*

## 2- Experimental

All the melting points are uncorrected. The IR spectrum was measured by Pye-Unicam SR-1000 infra red Spectrophotometer as KBr disk or nujl mul and H¹NMR was done in DMSO as solvent and tetramethylsilane (TMS) as internal standard [Varian EM-390] Spectrophotometer operating at 90 MHz.

**2.1. Industrial wastes of Gawafa seeds.** Were kindly supplied by El-Nasr Company of canned products, Kaha, Egypt. The oil was extracted from the seeds. The specifications are given in Table 1.

**2.2. Hydrolysis of crude Gawafa seeds fat.** The procedure described by El-Sawy et al. [3] was followed. The fatty acids mixture was analyzed by G. L. C. and their compositions are given in Table 1.

**2.3. Preparation of amido derivatives of N, N-dimethyl-etheylene diamine IIa-g [2].** Typical procedure: they were prepared by condensation of equamolar amounts of the corresponding pure fatty acids (octanoic $C_{8:0}$, dodecanoic $C_{12:0}$, tetradecanoic $C_{14:0}$, hexadecanoic $C_{16:0}$, octadecanoic $C_{18:0}$, 9-ctadecenoic $C_{18:1}$ acids) and or mixed fatty acids extracted from Gawafa fat with dimethylethylene diamine. The reaction mixture was heated at 140 °C for 10 hrs. The librated theoretical amount of water was collected to ensure the reaction completion by using Dean and Stark apparatus and dry benzene as solvent. The organic solvent was removed under reduced pressure, and residue was recrystallized from isopropanol. The yield percentage was 70–65 %, the structures of compounds IIa-g was confirmed by H¹NMR c.f. Table 5.

**2.4. Preparation of sodium 1-chloro-2-hydroxy-3-propanesulfonate [5]:** Typical procedure: Epichlorohydrine (50 g., 0.45 mol) was gradually added to sodium bisulfate (64.7 g., 0.621 mol) and sodium sulfate (25.0 g., 0.199 mol) dissolved in 130 ml water. The reaction temperature was maintained between 18–30 °C with aid of a cooling ice bath due to the exothermal reaction. The reaction product precipitated gradually and the pH of the solution remained at 6. After agitation for 2 hrs. at room temperature, the reaction product was removed by filtration and dried, yield percentage was 96% and was used as crude products in the next steps.

**2.5. Preparation of amido sulfobetaines IIIa-g [8].** They are prepared by placing 0.02 mol of sodium 1-chloro-2-hydroxy-3-propylene sulphate and 0.02 mol of IIa-g (N, N-dimethyl derivatives) were dissolved in 45 ml of water and 100 ml of ethanol separately. The mixture of the above solution was placed in reaction flask and refluxed for 3 hrs., the reaction mixture were cooled to 50 °C, then 0.03 mol of sodium carbonate was added with refluxing for 6 hrs. After cooling the reactant was filtered to remove the residual solid and extracted with 200 ml of petroleum-ether (40–60) triple to remove the unreacted amine. The remainder (water and alcohol phase) was evaporated to dryness, then washed with mixed solvent dichloroethane and acetone and dissolved in 75% ethanol. Finally, the pure product was obtained in 44–48% yield. The structure and purity were identified by TLC, elemental analysis and H¹NMR. c.f. Table 2.

*Table 2*

**Physical Properties of The Prepared amido sulfobetaines IIIa-f**

$CH_3(CH_2)_nCONHCH_2CH_2N^+(CH_3)_2CH_2CH(OH)CH_2SO_3^-$

| No. | R | M. F. | Mol. Wt. | m.p. °C | Yield % | Analysis data Calc./Found % C | H | N | S |
|---|---|---|---|---|---|---|---|---|---|
| IVa | 8 | $C_{15}H_{32}N_2O_5S$ | 352 | 80–82 | 45 | 51.14<br>51.00 | 9.09<br>9.10 | 7.95<br>7.90 | 9.09<br>8.90 |
| IVb | 12 | $C_{19}H_{40}N_2O_5S$ | 408 | 95–97 | 48 | 55.88<br>55.85 | 9.80<br>9.80 | 6.86<br>6.72 | 7.84<br>7.75 |
| IVc | 14 | $C_{21}H_{44}N_2O_5S$ | 436 | 120–122 | 46 | 57.80<br>57.72 | 10.09<br>10.00 | 6.42<br>6.31 | 7.34<br>7.20 |
| IVd | 16 | $C_{23}H_{48}N_2O_5S$ | 464 | 114–116 | 48 | 59.48<br>59.32 | 10.34<br>10.21 | 6.03<br>6.00 | 6.90<br>6.81 |
| IVe | 18:0 | $C_{25}H_{52}N_2O_5S$ | 492 | 130–132 | 45 | 60.98<br>60.94 | 10.57<br>10.64 | 5.69<br>5.60 | 6.50<br>6.51 |
| IVf | 18:1 | $C_{25}H_{50}N_2O_5S$ | 490 | Waxy | 44 | 61.12<br>61.11 | 10.20<br>10.00 | 5.71<br>5.80 | 6.53<br>6.48 |

$R = CH_3(CH_2)_nCO^-$

**2.6. Preparation of amidobetaines (IVa-g) [9]** They were prepared by dissolving 0.016 mol of of IIa-g (N,N-dimethyl derivatives) in 100 ml of absolute alcohol, 140 ml of aqueous solution containing 40 grams of sodium chloroacetate and 5 grams of sodium bicarbonate were added in three necked flask by stirring, then heated to reflux at 80 °C for 9 hrs. After cooling and removing the solvent to dryness, the reminder was dissolved in distilled water and filtered to remove the insoluble material. The filtrate was evaporated to remove water. The reminder was dissolved in absolute alcohol and then filtered to remove inorganic salts (three times). The final products were identified by elemental analysis, IR, and ¹HNMR. The yield percentage was (80–90%). c. f. Table 3.

*Table 3*

**Physical Properties of The Prepared amidobetaines IVa-f**

$CH_3(CH_2)_nCONHCH_2CH_2N^+(CH_3)_2CH_2COO^-$

| No. | R | M. F. | Mol. Wt. | m.p. °C | Yield % | Analysis data Calc./Found % C | H | N |
|---|---|---|---|---|---|---|---|---|
| Va | 8 | $C_{14}H_{28}N_2O_3$ | 272 | 60–62 | 90 | 61.76<br>61.32 | 10.29<br>10.01 | 10.29<br>10.11 |
| Vb | 12 | $C_{18}H_{36}N_2O_3$ | 328 | 70–72 | 86 | 65.81<br>65.70 | 10.98<br>11.00 | 08.54<br>08.40 |
| Vc | 14 | $C_{20}H_{40}N_2O_3$ | 356 | 84–86 | 85 | 67.42<br>67.30 | 11.24<br>11.30 | 07.86<br>07.50 |
| Vd | 16 | $C_{22}H_{44}N_2O_3$ | 384 | 116–118 | 84 | 68.75<br>68.60 | 11.46<br>11.40 | 07.29<br>07.30 |
| Ve | 18:0 | $C_{24}H_{48}N_2O_3$ | 412 | 124–126 | 84 | 69.90<br>69.80 | 11.65<br>11.60 | 06.80<br>06.70 |
| Vf | 18:1 | $C_{24}H_{46}N_2O_3$ | 410 | Waxy | 82 | 70.24<br>70.10 | 11.22<br>11.10 | 06.83<br>06.80 |

**2.7. Preparation of N-oxide derivatives (Va-g) [10]:** They were prepared by stirring a cold solution of IIa-g (0.1 mol) in 100 ml of acetone in an ice bath and 17 g., 0.15 mol of a 30%

hydrogen peroxide aqueous solution was added drop by drop over 30 min., the reaction mixture was allowed to come to room temperature and stand overnight. The excess peroxide was decomposed by stirring the reaction mixture with 0.1 g. of platinum black for 6 hrs., the products were obtained by separation of platinum black by filtration, a solvent of a filtrate was evaporated under vacuum. The products Va-g were crystallized from acetone c. f. Table 4.

*Table 4:*

**Physical Properties of the Prepared Amino Oxide Derivatives Va-f**

$CH_3(CH_2)_nCONHCH_2CH_2N^+(CH_3)_2®O^-$

| No. | R | M. F. | Mol. Wt. | m.p. °C | Yield % | Analysis data Calc./Found % C | H | N |
|---|---|---|---|---|---|---|---|---|
| VIa | 8 | $C_{12}H_{26}N_2O_2$ | 230 | 90–92 | 85 | 62.61<br>62.55 | 11.30<br>11.21 | 12.17<br>12.10 |
| VIb | 12 | $C_{16}H_{34}N_2O_2$ | 286 | 110–112 | 85 | 67.13<br>67.09 | 11.89<br>11.69 | 09.79<br>09.78 |
| VIc | 14 | $C_{18}H_{38}N_2O_2$ | 314 | 80–82 | 80 | 68.79<br>68.71 | 12.10<br>12.00 | 08.92<br>08.90 |
| VId | 16 | $C_{20}H_{42}N_2O_2$ | 342 | 100–102 | 80 | 70.18<br>70.03 | 12.28<br>12.12 | 08.19<br>08.00 |
| VIe | 18:0 | $C_{22}H_{46}N_2O_2$ | 370 | 75–76 | 78 | 71.35<br>71.51 | 12.43<br>12.32 | 07.57<br>07.43 |
| VIf | 18:1 | $C_{22}H_{44}N_2O_2$ | 368 | Waxy | 75 | 71.74<br>71.63 | 11.96<br>11.90 | 07.61<br>07.52 |

## 3. Surface properties of the prepared amphoteric surface active agents

The surface properties were measured under neutral conditions, in aqueous solution of prepared compounds.

**3.1. The surface and interfacial tensions** were measured by a Du-Nouy Tensiometer (Kruss, type 8451) at surfactant concentration 0.1 wt % and at room temperature 25 °C, using [11]

**3.2. Kraft point** the temperature at which 1% solution becomes clear on gradual heating is a convenient measure of aqueous solubility [12].

**3.3. Cloud point** it was determined by gradually heating of the prepared surface active compound solution (1.0 wt % concentration) in controlled temperature bath and recording the time at which the clear, or nearly clear solutions become definitely turbid. The reproducibility of this temperature was checked by cooling the solutions until they become clear again [13].

**3.4. Wetting time** was determined by immersing a sample of cotton fabric (3 g. on a stainless steel hook) in 0.1 wt % aqueous solutions of the surfactants at 25 °C. The recorded time from the moment the cotton was put into the solution until the moment it stated going down is wetting time [14].

**3.5. Foaming height** was measured according vigorous shaking of 25 ml surfactant solution (0.1 wt %) in 100 ml glass stopper graduated cylinder at 25 °C for 10 seconds. The solution was allowed to stand for 30 seconds and the foam height was measured [15].

**3.6. Emulsion stability determination** was prepared from 10 ml of 20 mmol aqueous solution of surfactants and 5 ml of toluene at 40 °C. The emulsion stability was determined as the time of separation of water (9 ml) from the emulsion layer from the moment of the ceasing of shaking [16].

**3.7. Calcium stability** it was determined by a modified Hart method [17]; (10 mmol) the prepared surfactant solution was titrated against (0.1N) calcium chloride solution. The end point was determined by visual observation of cloudiness of the surface active agents solution.

**3.8. Stability to hydrolysis** a mixture of 10 mmol of the prepared surfactant compounds and 10 ml of 2.0 N sulfuric acid was placed in a thermostat at 40 °C. The time it takes for a sample solution to be clouded as the result of hydrolysis shows the stability of the surfactant to hydrolysis [16].

**3.9. Biodegradability in** percentage was determined according to Eter et al. [18].

*Table 5.*

**Spectral data of some examples from the prepared compounds**

| Compd. No. | $^1$HNMR (d = ppm) | IR ($cm^{-1}$) |
|---|---|---|
| **IIa:** | d 0.80 (t, 3H, term. $CH_3$); d 1.2–1.3 (m, 10H, –5 $CH_2$-chain): d 1.7 (t, 2H, –$CH_2$CO);<br>d 5.4 (s, 1H, NH):d 2.3 (t, 2H, $NHCH_2$–); d 3.4 (t, 2H, –$CH_2N(CH3)_2$); d 3.5–3.6 (s, 6H, –$N(CH_3)_2$); | 2995–2750 $cm^{-1}$n CH ali.<br>1665–1645 $cm^{-1}$n CO of amid 3300–3250 $cm^{-1}$n of NH |
| **IIe:** | d 0.85 (t, 3H, term. $CH_3$); d 1.1–1.3 (m, 30H, –15 $CH_2$-chain): d 1.6 (t, 2H, –$CH_2$CO);<br>d 5.3 (s, 1H, NH):d 2.3 (t, 2H, $NHCH_2$–); d 3.4 (t, 2H, –$CH_2N(CH3)_2$); d 3.5–3.6 (s, 6H, –$N(CH_3)_2$); | 2990–2850 $cm^{-1}$n CH ali.<br>1650–1645 $cm^{-1}$n CO of amid 3300–3250 $cm^{-1}$n of NH |
| **IIIb:** | d 0.9 (t, 3H, term. $CH_3$); d 1.1–1.3 (m, 18H, –9 $CH_2$-chain): d 2.1 (t, 2H, –$CH_2$CO);<br>d 5.8 (s, 1H, NH):d 2.3 (t, 2H, $NHCH_2$–); d 3.2 (t, 2H, –$CH_2N(CH3)_2$); d 3.5–3.6 (s, 6H, –$N(CH_3)_2$);<br>d 3.3 (d, 2H, $N(CH_3)_2$–$CH_2$–); d 3.9 (m, 1H, $CH_2$–CH(OH)–);<br>d 4.2 (s, 1H, –CH(OH));d 2.9 (t, 2H, –CH(OH)–$CH_2$). | 2980–2950 $cm^{-1}$n CH ali.<br>1650–1645 $cm^{-1}$n CO of amid<br>3350–3200 $cm^{-1}$n of NH<br>1315–1220 and 1140–1075 $cm^{-1}$n of $SO_3$ |
| **IIIe**: | d 0.85 (t, 3H, term. $CH_3$); d 1.1–1.3 (m, 30H, –15 $CH_2$-chain): d 1.6 (t, 2H, –$CH_2$CO);<br>d 5.3 (s, 1H, NH):d 2.3 (t, 2H, $NHCH_2$–); d 3.4 (t, 2H, –$CH_2N(CH3)_2$);<br>d 3.5–3.6 (s, 6H, –$N(CH_3)_2$); d 3.2 (d, 2H, $N(CH_3)_2$–$CH_2$–); d 3.85 (m, 1H, $CH_2$–CH(OH)–);<br>d 4.0 (s, 1H, –CH(OH));d 2.9 (t, 2H, –CH(OH)–$CH_2$). | 2985–2960 $cm^{-1}$n CH ali.<br>1650–1645 $cm^{-1}$n CO of amid<br>3300–3250 $cm^{-1}$n of NH<br>1315–1220 and 1140–1075 $cm^{-1}$n of $SO_3$ |
| **IVc**: | d 0.88 (t, 3H, term. $CH_3$); d 1.1–1.25 (m, 22H, –11 $CH_2$-chain): d 2.1 (t, 2H, –$CH_2$CO);<br>d 5.8 (s, 1H, NH):d 2.3 (t, 2H, $NHCH_2$–); d 3.4 (t, 2H, –$CH_2N(CH3)_2$);<br>d 3.5–3.6 (s, 6H, –$N(CH_3)_2$); d 3.8–3.9 (s, 2H, N–$CH_2$CO); | 2988–2850 $cm^{-1}$n CH ali.<br>1650–1645 $cm^{-1}$n CO of amid 3300–3250 $cm^{-1}$n of NH<br>1600–1590 $cm^{-1}$n of $COO^-$ |
| **IVc**: | d 0.85 (t, 3H, term. $CH_3$); d 1.1–1.25 (m, 30H, –15 $CH_2$-chain): d 2.2 (t, 2H, –$CH_2$CO);<br>d 5.6 (s, 1H, NH):d 2.4 (t, 2H, $NHCH_2$–); d 3.4 (t, 2H, –$CH_2N(CH3)_2$);<br>d 3.5–3.7 (s, 6H, –$N(CH_3)_2$); d 3.8–3.6 (s, 2H, N–$CH_2$CO); | 2988–2820 $cm^{-1}$n CH ali.<br>1660–1650 $cm^{-1}$n CO of amid 3300–3250 $cm^{-1}$n of NH<br>1600–1590 $cm^{-1}$n of $COO^-$ |
| **Vd**: | d 0.85 (t, 3H, term. $CH_3$); d 1.1–1.3 (m, 26H, –13 $CH_2$-chain): d 1.6 (t, 2H, –$CH_2$CO);<br>d 5.3 (s, 1H, NH):d 2.3 (t, 2H, $NHCH_2$–);<br>d 3.8 (t, 2H, –$CH_2N(CH3)_2$); d 4.2–3.9 (s, 6H, –$N(CH_3)_2$); | 2890–28850 $cm^{-1}$nCH ali.<br>1650–1645 $cm^{-1}$n CO of amid 3300–3250 $cm^{-1}$n of NH<br>1398–1370 $cm^{-1}$n of N–O |

## 4. Results and Discussion

In the present work, three types of amphoteric surface active agents were synthesized from pure and mixed fatty acids extracted from Gawafa seeds. Since the most constituents of Gawafa fats are saturated octanoic $C_{8:0}$, dodecanoic $C_{12:0}$, tetradecanoic $C_{14:0}$, hexadecanoic $C_{16:0}$, octadecanoic $C_{18:0}$, acids and small percentage of 9-octadecenoic $C_{18:1}$ acid c. f. Table 1. it would be interesting to use pure named maintained above fatty acids and mixed fatty one extracted from Gawafa fat in the preparation of amphoteric surface active agents and study their surface active properties. The preparations of the amphoteric surface active agents IIIa-g, IVa-g and Va-g were easy and could be isolated in suitable yield c. f. Table 2, 3 and 4. Infra red IR and proton nuclear magnetic resonance $H^1$NMR spectroscopies were conducted to confirm their structures c. f. Table 5.

*Table 6.*

**Surface Properties of Amidobetaine surface active agents:**

| Surf. Prop. / Acids. | Surface tension (dyne/cm) 0.1 wt% | Interfacial tension (dyne/cm) 0.1 wt% | Kraft Point °C 1.0 wt% | Wett. Time Sec, 0.1 wt% | Foam height mm 0.1 wt% | Emul. Stab. Sec. 20 mmol | $Ca^{++}$ Stab. (ppm) 10mmol | Stab. to hydr. Base Sec. 10 mmol | Acidmin: |
|---|---|---|---|---|---|---|---|---|---|
| **$C_8$** | 35.5 | 16.5 | 5 | 30 | 210 | 221 | 1600 | 50:13 | More than one day |
| **$C_{12}$** | 34.0 | 15.0 | <0 | 20 | 230 | 272 | 1800 | 52:17 | |
| **$C_{14}$** | 35.0 | 11.5 | <0 | 35 | 235 | 293 | 1800 | 55:36 | |
| **$C_{16}$** | 36.5 | 13.0 | 4.0 | 50 | 255 | 306 | 2000 | 57:07 | |
| **$C_{18}$** | 38.0 | 13.5 | 6.0 | 80 | 260 | 320 | 2200 | 60:23 | |
| **$C_{18}$** | 37.5 | 13.0 | 7.0 | 90 | 290 | 365 | >2200 | 62:36 | |
| **Mix** | 39.0 | 14.5 | 8.0 | 105 | 320 | 377 | >2200 | 60:38 | |

### 4.1. Surface properties of the prepared compounds:

The surface properties of the prepared amphoteric surface active agents have been measured in neutral medium by traditional procedures. The resulted data are given in Tables 6, 8 and 10.

*Table 7.*

**Biodegradability of Amidobetaine surface active agents**

| Days / Acids | 1st day | 2nd day | 3rd day | 4th day | 5th day | 6th day | 7th day |
|---|---|---|---|---|---|---|---|
| $C_8$ | 50.5 | 59.0 | 66.5 | 79.5 | 89.0 | 93.0 | – |
| $C_{12}$ | 49.0 | 58.0 | 65.5 | 78.0 | 88.0 | 92.0 | – |
| $C_{14}$ | 48.5 | 57.5 | 64.5 | 77.5 | 88.0 | 91.5 | – |
| $C_{16}$ | 48.0 | 57.5 | 64.0 | 77.0 | 87.5 | 91.0 | 99.0 |
| $C_{18}$ | 48.0 | 57.0 | 64.0 | 77.0 | 87.0 | 91.0 | 98.0 |
| $C_{18}$ | 47.5 | 56.5 | 64.0 | 76.5 | 86.5 | 90.0 | 96.0 |
| Mix | 47.0 | 56.0 | 63.5 | 76.0 | 86.0 | 90.0 | 95.0 |

#### *4.1.1. The surface and interfacial tensions*

They were determined according to Findly [11]. The measured surface and interfacial tensions data are given in Table 6, 8 and 10. The values follow the general term, that the surface and interfacial tensions were increased with increasing in the number of carbon atoms in the hydrophobic part at same amphoteric molecules [19]. Also it appeared that, the measured surface and interfacial tensions of amido sulfobetaines IIIa-g) were 35.5–44), amidobetaines IVa-g) (35.5–39) and amino oxide Va-g (34.5–41 dyne / cm). Overall, the amphoteric surface active agents prepared from mixed fatty acids of Gawafa oil recorded almost the same sequences surface and interfacial tensions as those prepared from individual commercial fatty acids.

*Table 8.*

**Surface Properties of Amino oxide surface active agents**

| Surf. Prop. / Acids. | Surface tension (dyne/cm) 0.1 wt% | Interfacial tension (dyne/cm) 0.1 wt% | Kraft Point °C 1.0 wt% | Wett. Time Sec, 0.1 wt% | Foam height mm 0.1 wt% | Emul. Stab. Sec. 20 mmol | $Ca^{++}$ Stab. (ppm) 10mmol | Stab. to hydr. Base Sec. 10 mmol | Acidmin: |
|---|---|---|---|---|---|---|---|---|---|
| **$C_8$** | 34.5 | 7.5 | 12.0 | 39.0 | 150 | 3:57 | 95 | 48:01 | More than one day |
| **$C_{12}$** | 37.0 | 8.0 | 13.0 | 55.0 | 160 | 4:02 | >100 | 48:03 | |
| **$C_{14}$** | 38.0 | 9.5 | 14.5 | 60.0 | 180 | 4:16 | 80 | 49:03 | |
| **$C_{16}$** | 39.5 | 11.0 | 15.0 | 63.0 | 180 | 4:37 | 76 | 50:39 | |
| **$C_{18}$** | 40.5 | 12.5 | 16.0 | 72.0 | 190 | 5:02 | 70 | 52:53 | |
| **$C_{18}$** | 40.0 | 13.0 | 17.0 | 99.0 | 205 | 6:05 | 68 | 55:05 | |
| **Mix** | 41.0 | 13.5 | 20.0 | 125.0 | 230 | 6:33 | 65 | 56:32 | |

#### *4.1.2. Kraft and Cloud points*

The data are given in Table 6, 8 and 10 show, that the Kraft point increases with increasing hydrophobic part [5]. Also amidobetaines and sulfobetaines have good Kraft points in a comparison with amino oxide. The remarkable decrease in the Kraft point especially for amido sulfobetaine and amidobetaine is attributed to co-operative effect of quaternary ammonium moiety with amido linkage. Also cloud point was measured for amino oxide and it has good cloud point c. f. Table 8.

*Table 9.*

**Biodegradability of Amino oxide surface active agents**

| Days / Acids | 1st day | 2nd day | 3rd day | 4th day | 5th day | 6th day | 7th day | 8th day |
|---|---|---|---|---|---|---|---|---|
| $C_8$ | 45.5 | 56.5 | 62.0 | 73.0 | 83.0 | 91.0 | 97.0 | – |
| $C_{12}$ | 43.0 | 53.0 | 59.0 | 72.5 | 82.0 | 90.0 | 95.0 | 99.0 |
| $C_{14}$ | 42.5 | 52.0 | 58.5 | 72.0 | 81.0 | 89.0 | 94.0 | 98.0 |
| $C_{16}$ | 41.0 | 51.0 | 58.5 | 71.0 | 81.0 | 88.0 | 92.0 | 97.5 |
| $C_{18}$ | 40.5 | 50.5 | 58.0 | 70.0 | 80.5 | 87.5 | 91.5 | 97.0 |
| $C_{18}$ | 40.5 | 50.0 | 57.0 | 69.5 | 79.0 | 86.0 | 91.0 | 96.5 |
| Mix | 40.0 | 50.0 | 56.5 | 68.0 | 78.0 | 85.0 | 90.0 | 95.0 |

#### *4.1.3. Wetting time*

All prepared amphoteric surface active agents have good wetting time. From the date in Tables 6, 8 and 10, it can be seen that the prepared amphoteric surface active agents afforded good wetting time, also amidobetaine and amido sulfobtaine have good wetting time to compare with amino oxide. The wetting properties is attributed to hydrophobic parts and not improved by head polar group [20].

#### *4.1.4. Foam height*

The foaming height of the prepared amphoteric compounds was investigated [15]. The measured data are given in Tables

6, 8 and 10. It can be seen that the prepared amphoteric surface active agents have high foam height. The foam height increases with increasing the length of hydrophobic part. Also the amidobetaine and amido sulfobetaine have superior foam height.

*4.1.5. Emulsion stability*

The emulsion stability of the prepared amphoteric surfactants was determined by standard methods c.f. Tables 6, 8 and 10 [16]. The prepared amphoteric surface active agents posses good emulsion stability especially amino oxide derivatives. As general, the emulsion stability increases with increasing hydrophobic chain length. Also it appears that the amphoteric surface active agents prepared from mixed fatty acid extracted from Gawafa oils have highest emulsification stability.

*Table 10.*

**Surface Properties of Amido sulfobtaine surface active agents:**

| Surf. Prop. / Acids. | Surface tension (dyne/cm) 0.1 wt % | Interfacial tension (dyne/cm) 0.1 wt % | Kraft Point ℃ 1.0 wt % | Wett. Time Sec, 0.1 wt % | Foam height mm 0.1 wt % | Emul. Stab. Sec. 20 mmol | $Ca^{++}$ Stab. (ppm) 10mmol | Stab. to hydr. Base min. Sec. | Acidmin: min Sec. |
|---|---|---|---|---|---|---|---|---|---|
| $C_8$ | 35.5 | 5.0 | 3.0 | 35.0 | 210 | 345 | 2100 | 29:12 | 63:00 |
| $C_{12}$ | 37.0 | 7.5 | 5.0 | 46.0 | 220 | 363 | 2200 | 38:08 | 62:22 |
| $C_{14}$ | 39.5 | 9.0 | 7.0 | 55.0 | 245 | 379 | 1900 | 42:09 | 71:05 |
| $C_{16}$ | 40.5 | 10.5 | 9.0 | 73.0 | 260 | 420 | 1900 | 52:13 | 73:06 |
| $C_{18}$ | 41.0 | 11.5 | 11.0 | 85.0 | 270 | 451 | 1800 | 53:42 | 75:36 |
| $C_{18}$ | 43.5 | 12.5 | 12.0 | 89.0 | 265 | 443 | 1800 | 62:31 | 79:13 |
| **Mix** | 44.0 | 15.0 | 13.0 | 115.0 | 280 | 510 | 1950 | 71:15 | 81:35 |

*4.1.6. Calcium stability*

The calcium stability values of the prepared amido sulfobetaine and amidobetaine surface active agents were determined using [17]. From the data are given in the Tables 6 and 10, it can be seen that all prepared amphoteric compounds have good calcium ion stability.

*4.1.7. Stability to hydrolysis*

The stability to acid and base hydrolysis was investigated; the data are in Tables 6, 8 and 10 demonstrate stability of hydrolysis of the prepared amphoteric surface active agents. It can be seen that all the prepared compounds have higher stability in acid than in basic medium, this may be due to the presence of nitrogen atoms which coordinated with the acid forme more cationic center [21].

**4.2 Biodegradability**

In the course of degradation the river die-away tests was followed by surface tension measurements [18]. The biodegradability data are given in Tables 7, 9 and 11. Within the experimental accuracy all prepared amphoteric surface active agents seem to degrade easy. Also it can be seen that amidobetaine and amidosulfobetaine have higher degradability than amino oxide type.

*Table 11.*

**Biodegradability of amido sulfobtaine surface active agents**

| Days / Acids | 1st day | 2nd day | 3rd day | 4th day | 5th day | 6th day | 7th day |
|---|---|---|---|---|---|---|---|
| $C_8$ | 55.5 | 62.5 | 71.5 | 89.0 | 90.0 | 95.0 | – |
| $C_{12}$ | 52.0 | 60.0 | 69.5 | 80.0 | 89.5 | 93.0 | – |
| $C_{14}$ | 50.0 | 59.5 | 68.5 | 78.5 | 87.5 | 91.5 | – |
| $C_{16}$ | 49.0 | 57.0 | 66.0 | 78.0 | 86.5 | 90.5 | 98.5 |
| $C_{18}$ | 48.0 | 57.0 | 66.0 | 78.5 | 85.5 | 98.0 | 97.0 |
| $C_{18}$ | 49.0 | 56.0 | 65.5 | 79.0 | 83.5 | 90.0 | 96.0 |
| Mix | 48.0 | 55.5 | 64.0 | 77.5 | 85.5 | 89.5 | 96.0 |

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## BÖLCSESSÉGEK*

A pénz olyan tiszta, amilyen tisztán jutnak hozzá és használják fel.

Esendõek vagyunk! Bárkivel megtörténhet, hogy jogosnak vélt önvédelemböl az objektív tényeket kozmetikázza.

A pénz az ember számára csak eszköz lehet. Ha céllá válik, az emberbõl lesz eszköz.

* A szerzõ saját gondolatai, de korábban már másnak is eszébe juthattak (a szerzõ megjegyzése).