Results and Discussion

RESULTS AND DISCUSSION

Synthesis of surfactants:

The synthesis of nonionic and anionic surfactants were reported previously[1, 31, 32]. Preparation of nonionic surfactants refers chiefly to polyoxyethylene derivatives, they are usually prepared by the addition of different moles of ethylene oxides to organic hydrophobic compounds containing active hydrogen in the presence of catalyst[1]. It was reported[1, 31] that most of nonionic surfactants can be constructed by addition of hydrophilic part (ethylene oxide) to hydrophobic part specially fatty alkyl chain containing active hydrogen. Also preparation of nonionic surfactants from succinate and maleate was reported[96, 97] which used half ester as nonionic[98] and used as cationic also used as zwitterionic surfactants. Nonionic surfactants had diverse applications, both in industry and in the house field due to its moderate foaming and good detergency. It was also employed in a variety of ways in leather industry. It was used to accelerate soaking and liming which was improved by the addition of wetting agents[99]. Also nonionic surfactants are used extensively because of their good detergency, easy rinsing and low foaming in cleaning of milk and other purposed.

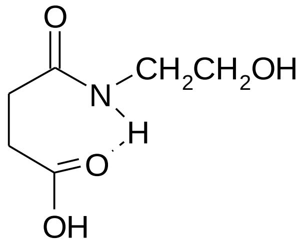
In the present work the set of surfactants have been enlarged using two substrates succinic and maleic anhydride with hydrophobic fatty alcohol alkyl chain having different moles of ethylene glycols, also constructing nonionic and anionic surfactants have hydrophilic part in the middle of the molecule which have good surface character.

1.1. Preparation of nonionic surfactants derived from succinic anhydride:

1.1.1 Reaction of succinic anhydride with ethanol amine to afford N-hydroxyethyl succinic acid amide (Succinic acid amide surfactants):

It was completed in many steps reactions according to Scheme 1. First step is the preparation of N-hydroxyethyl succinic acid amide by fusion of ethanolamine with succinic anhydride at 80 ^{o} C, the structure of the product was confirmed by IR. (cf. Fig. 1.); the IR spectra of N-hydroxy ethyl succinic acid amide showed that $\nu_{C\text{-}H}$ aliphatic at 2884.5-2947 cm^{-1} due to symmetrical and asymmetrical stretching of C-H of methylene bond. $\nu_{C=O}$ of acid and amide in the region of 1697.5 cm^{-1}. $\nu_{C\text{-}N}$ at 1558.1 cm^{-1} of secondary amide, broad band at 3200-3400 cm^{-1} corresponding to ν_{OH} of acid, ν_{NH} of secondary amide and ν_{OH} of glycol.

Stretching frequency of $\nu_{C=O}$ of acid at 1697 cm^{-1} to be at lower value, is due to intra molecular hydrogen bond was formed between carbonyl group of acid and NH as:

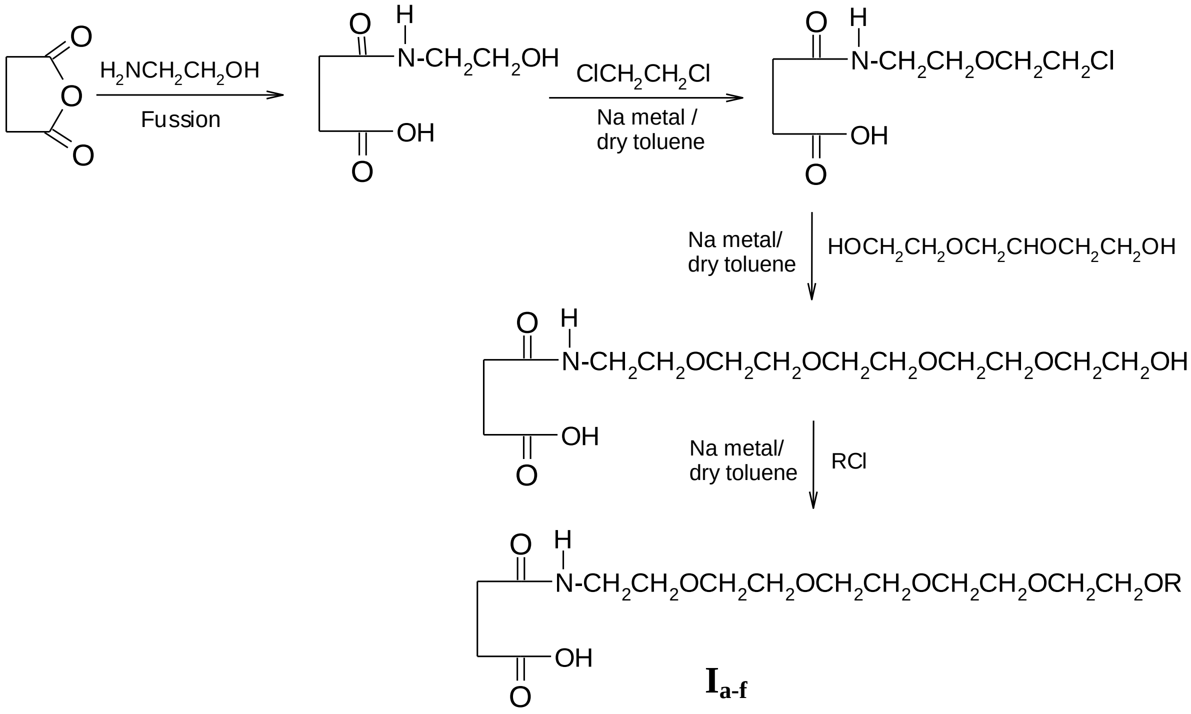


Second step is the construction of hydrophilic part by reaction of N-hydroxy-ethyl succinic acid amide with 1,2-dichloroethane to give N-chloroethyloxyethyl succinic acid amide, where its structure was confirmed by IR. (cf. Fig 2.) showing that; $\nu_{C\text{-}H}$ aliphatic at 2889.5-2946.6 cm^{-1} due to symmetrical and asymmetrical stretching of C-H of methylene bond. $\nu_{C=O}$ of acid, secondary amide in region of 1693 cm^{-1}.

ν_{C-N} at 1556.4 cm^{-1} of secondary amide ν_{OH} and ν_{N-H} at 3200-3400 cm^{-1}. Combination band of secondary amide at 2105 cm^{-1}. The structure of product was terminated by chlorine atom which can be easily reacted through Williamson reaction to elongate the hydrophilic chain with triethylene glycol to give N-hydroxy tetra-oxy penta-ethylene succinic acid amide, the structure of the later was confirmed by IR (see Fig.3). The IR spectra showed that ν_{C-H} aliphatic broad stretching frequency at 2900-2929.4 cm^{-1} due to symmetrical and asymmetrical of CH of methylene band, of $\nu_{C=O}$ acid and $\nu_{C=O}$ of amide ranging in between 1585-1700 cm^{-1} broad stretching frequency band at 3100-3500 cm^{-1} of ν_{OH} of acid, ν_{NH} of secondary amide and ν_{OH} of glycol and ν_{C-O-C} of glycol at 1179.6 cm^{-1}.

The last step in the preparation of these compounds is the reaction of N-hydroxy tetra-oxy penta-ethylene succinic acid amide with fatty alkyl chloride (C8 – C18) to introduce hydrophobic part, producing corresponding series of nonionic surfactants (Ia –f). All these prepared compounds have the same hydrophilic moiety with different hydrophobic part. The structure of the prepared nonionic surfactants was confirmed by IR and ^{1}HNMR. (cf. Fig. 4 and 5; Ia).

IR. Show that; ν_{C-H} aliphatic at 2854.3-2925.0 cm^{-1} due to symmetrical and asymmetrical stretching of methylene and methyl groups, broad stretching frequency of $\nu_{C=O}$ of acid and $\nu_{C=O}$ of amide in between 1600-1700 cm^{-1}. Broad band at 3200-3400 cm^{-1} due to ν_{OH} of acid and ν_{NH} of amide. ν_{C-O-C} of glycol at 1121.6 cm^{-1}; ^{1}HNMR of compound Ia showed that the presence of the following signals: δ ppm = 0.8 (t, 3H of CH_3); δ ppm =1.2-1.4 (m, 12H of 6CH_2 groups of alkyl chain δppm = 1.5-1.8 (t, 6H of CH_2-O and CO-CH_2CH_2CO of succinate part) and δ ppm = 3.6 –4.2 (m, 20H of 5 O-CH_2CH_2O- of ethylene glycol chain; and NH-CH_2CH_2O-unit) *c.f.* Fig. (5).



I_{a-f}

where R = C_8H_{17}; $C_{10}H_{21}$; $C_{12}H_{24}$; $C_{14}H_{29}$; $C_{16}H_{33}$; $C_{18}H_{37}$ corresponding to I_{a-f}

Ia Ib Ic Id Ie If

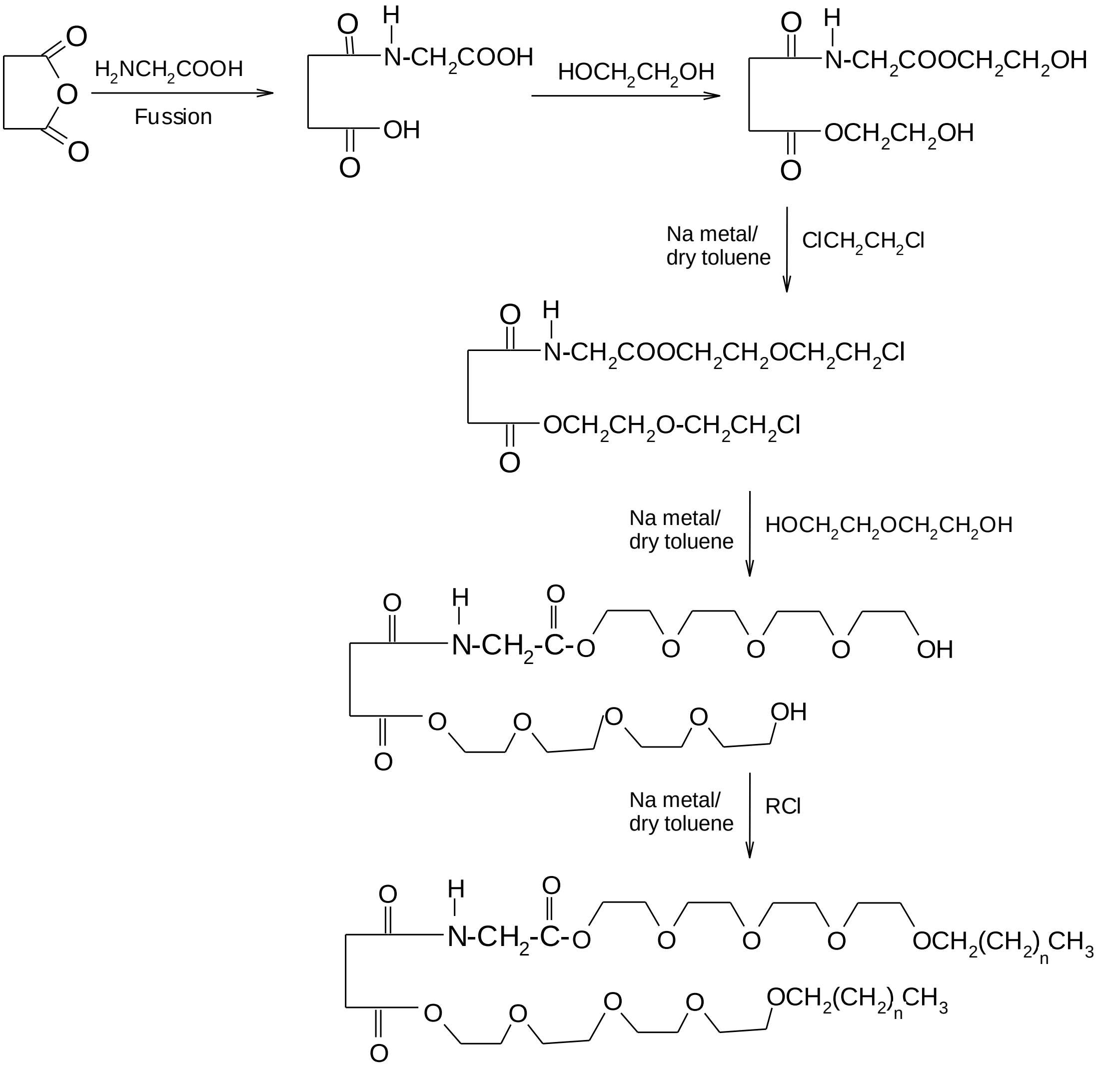
Respectively

Scheme 1

1.1.2 Reaction of succinic anhydride with glycine to afford N- succinic amide acetic acid (Succinic amide ester surfactants):

It was carried out in many steps reactions according to Scheme 2. First step is the preparation of N- succinyl amide acetic acid by fusion of glycine with succinic anhydride at 80 ^{o} C to give the product. The structure of the product was confirmed by IR. (cf. Fig 6.) the IR. Spectra show that broad stretching frequency peaks at ν = 1640-1710 cm^{-1} are characteristic for $\nu_{C=O}$ of acid and amide and also a broad band ranging at ν = 2500 –3465 cm^{-1} of OH of acid, and NH of secondary amide; ν_{C-H} aliphatic at 2946.8 cm^{-1} and combination band of secondary amide at ν 1974.8 cm^{-1}.

Second step is the construction of hydrophilic part by esterification with excess ethylene glycol to give dihydroxy-diethyl-N-succinyl amide acetate and its structure was confirmed by IR. (cf. Fig 7.), which show that broad band at 3200-3500 cm^{-1} corresponding to ν_{N-H} of amide and ν_{OH} of glycol, $\nu_{C=O}$ of ester at 1778.3 cm^{-1} broad absorption frequency of amide and $\nu_{C=O}$ of amide at 1566 cm^{-1}. Since the ethylene glycol was taken by excess mole; it can be reacted with both of COOH groups the disappearance of $\nu_{C=O}$ of acid indicates that glycols reacted with both COOH groups. The product is followed by the reaction with 1,2-dichloroethane to elongate products with ethylene units; the structure was terminated by chlorine atom which can be easy to react through Williamson reaction to further elongated the hydrophilic chain with excess diethylene glycol to give 1-hydroxy-3,6,9-trioxytetraethylene, N-succinic amide hydroxytetraoxy tetraethyl acetate. The structure of the later was confirmed by IR. Fig. (8) showing that $\nu_{C=O}$ of ester at 1731.6 cm^{-1} and $\nu_{C=O}$ of amide at 1682.7 cm^{-1}; broad band at 3200-3500 cm^{-1} corresponding to ν_{OH} of glycol and ν_{NH} of amide. The last step in the preparation of these compounds is the reaction of 1-hydroxy-3,6,9-trioxytetraethylene-N-succinic amide hydroxytetraoxy tetraethyl acetate with fatty alkyl chloride (C8 – C18) to produce the corresponding series of nonionic surfactants (IIa –f). All these prepared compounds have the same hydrophilic moiety with different hydrophobic part. The structure of the prepared nonionic surfactants was confirmed by IR. (cf. Fig. 9 as example corresponding to IIf). In this figure (9) shows that $\nu_{C=O}$ of ester at 1733 and $\nu_{C=O}$ of amide at 1680 cm^{-1}. Also νCH aliphatic at 2851-2920 cm^{-1} due to symmetrical and asymmetrical stretching of methylene and methyl groups. Also, ν_{NH} of amide at 3362.1 cm^{-1}. And $\nu_{C\text{-}O\text{-}C}$ of glycol at 1129.1 cm^{-1} and ^{1}HNMR. (cf. Fig. 10 and 11; corresponding to IIa, IIe respectively).



II$_{a\text{-}f}$

where R = C_8H_{17}; $C_{10}H_{21}$; $C_{12}H_{24}$; $C_{14}H_{29}$; $C_{16}H_{33}$; $C_{18}H_{37}$ corresponding to $II_{a\text{-}f}$
IIa IIb IIc IId IIe IIf
Respectively

Scheme 2

^{1}HNMR of compound IIa (Fig. 10), shows the presence of the following signals: δ ppm = 0.8 (t, 6H of $2CH_3$); δ ppm =1.1-1.8 (m, 24 H of $12CH_2$ alkyl chain; δ ppm = 2.0 –2.4 (t, 4H of $2CH_2O$ alkyl chain). δ ppm = 2.2 –2.4 (t, 4H of $OC.CH_2CH_2.CO$; δ ppm = 4.3 –4.8 (t, 34H of 8 CH_2CH_2O and CH_2 of NH-CH_2-C=O), δ ppm = 7.6-7.8 (1H of $NHCH_2CO$) *c.f.* (Fig. 10).

^{1}HNMR of compound IIe (Fig. 11) shows the presence of the following signals: δ ppm = 0.8 (t, 6H of $2CH_3$); δ ppm =1.2-1.8 (t, 56H of $28CH_2$ alkyl chain); δ ppm = 2.3-2.6 (t, 8H of $COCH_2CH_2CO$; $2CH_2$-O of alkyl chain) δ ppm = 4.4-4.8 (t, 34H of OCH_2CH_2O, and NH-CH_2-CO) and at 7.6-7.8 (1H of $NHCH_2CO$), cf. (Fig.11).

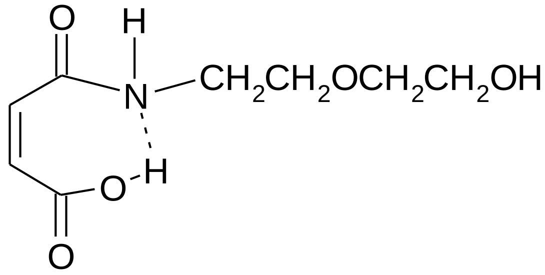
Part II

1.2. Preparation of Nonionic and Anionic Surfactants Derived from Maleic Anhydride

1.2.1. Preparation of nonionic surfactants:

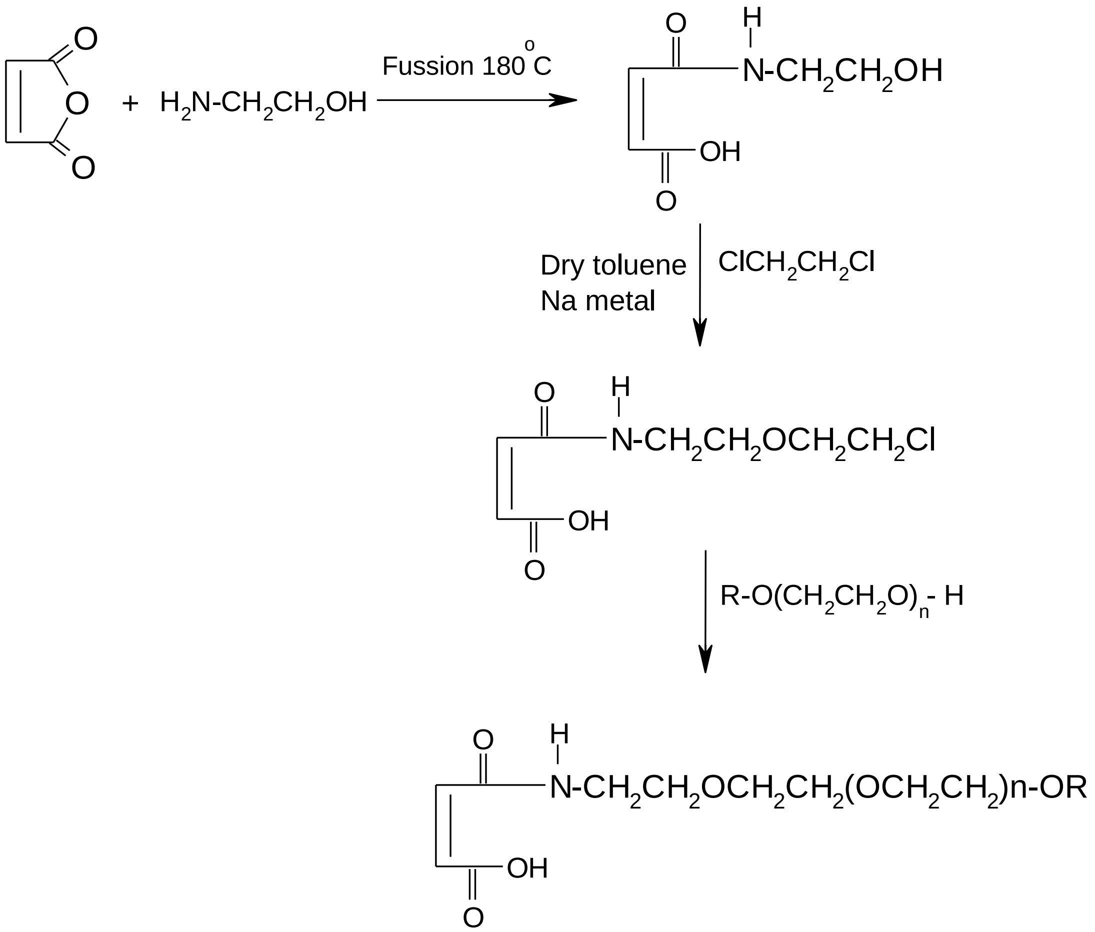
It was completed in many steps reactions according to Scheme 3. First step is the preparation of N-hydroxyethyl maleamide by fussion of ethanolamine with maleic anhydride at 80 °C. The structure of the product was confirmed by IR. (cf. Fig 12.) which showed the presence of stretching frequency I, peak at $\nu = 1633$ cm^{-1} due to $N_{C=O}$ of amide and at $\nu = 1714.5$ cm^{-1} due to carbonyl of acid. At $\nu = 2500\text{-}3287.6$ cm^{-1} (broad band due to hydroxyl group and NH of amide) at $\nu = 2886.6\text{-}2958.5$ cm^{-1} is characteristic of aliphatic CH due to symmetrical and asymmetrical stretching.

Second step is the reaction of N-hydroxyethylmalamide with 1, 2-dichloroethane to give N-chloroethyloxyethyl maleamide and its structure was confirmed by IR. (cf. Fig 13), $\nu = 1654.6$ cm^{-1} due to $\nu_{C=O}$ of amide, $\nu_{C=O} = 1699.5$ cm^{-1} due to carbonyl of acid, a weak stretching frequency of ν_{OH} of acid may be due to intra molecular hydrogen bond between OH of acid and N as in the structure.



ν_{OH} of acid and NH of secondary amide at 3465.7 cm^{-1}, $\nu = 1126$ cm^{-1} is characteristic for C-O-C bond; the structure was terminated by chlorine atom which can be easily reacted through Williamson reaction to produce nonionic surfactants in the last step.

Also preparation of ethoxylated alcohols from C8 to C18 with different moles of ethylene oxide (n = 5, 8 and 12 moles) was carried out, the average degree of ethylene oxide units added was confirmed by IR and ^{1}H NMR aiming to produce nonionic surfactants having their hydrophilic chain in the middle (flanked between maleamide and alkyl fatty chain) means that a capped nonionic surfactant type has produced. This is completed by the reaction of ethoxylated fatty alcohols with N-chloroethyloxyethyl maleamide to give nonionic surfactants IIIa-c to VIIIa-c (cf. Scheme 3).



$III_{a-c} – VIII_{a-c}$

Where R = C_8H_{17}; $C_{10}H_{21}$; $C_{12}H_{25}$; $C_{14}H_{29}$; $C_{16}H_{33}$; $C_{18}H_{37}$ and n = 5, 8 and 12 corresponding t
IIIa-c to VIIIa-c reapectively

Scheme (3)

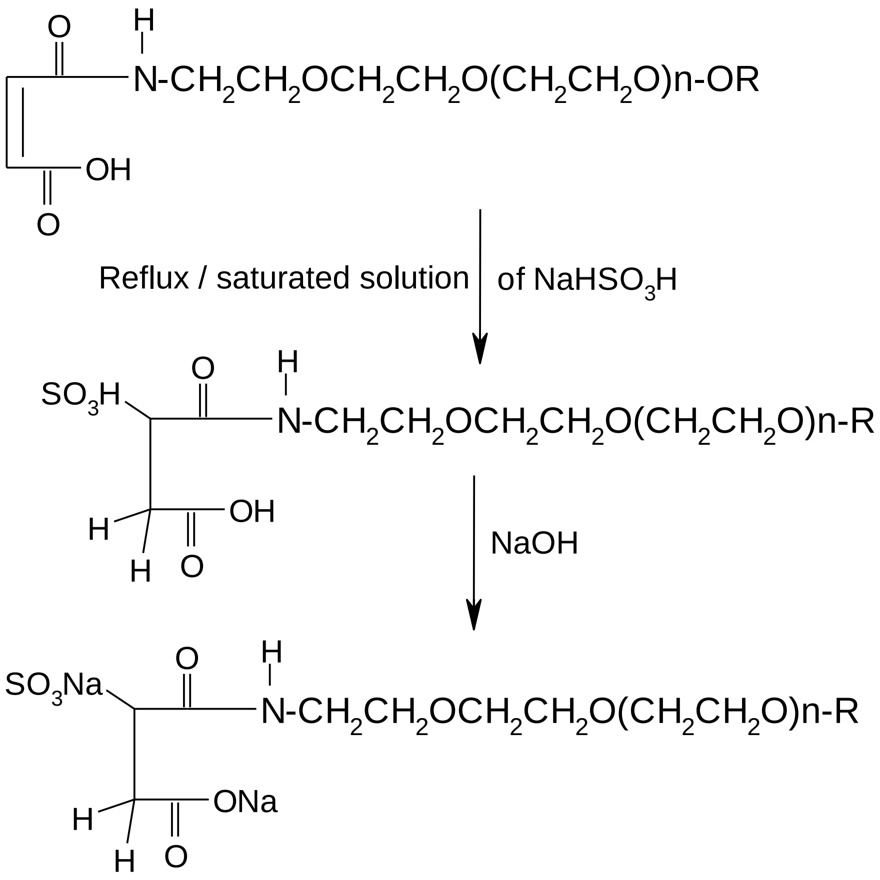
The structures of the synthesized nonionic surfactants were confirmed by: IR spectroscopy: the IR spectra of compounds IIIa (C8, 5EO) (Fig. 14); Vb (C12, 8EO) (Fig. 15); VIc (C14, 12EO) (Fig. 16); VIIIa (Fig. 17) show at ν = 1113 – 1122 cm^{-1} is characteristic for CH_2-O-CH_2 ethylene oxide band; $N_{C=O}$ = 1647 – 1652 cm^{-1} of amide, ν = 1697-1703 cm^{-1} of acid, ν at 2891-2930 cm^{-1} is characteristic for CH_2 aliphatic; ν = 3280-3469 cm^{-1} due to OH of acid and NH of amide.

^{1}HNMR of compound IIIb (Fig. 18) show the presence of the following signals: δ ppm = 0.8 (t, 3H of CH_3); δ ppm =1.1-1.3 (m, 12 H of 6CH_2 alkyl fatty chain); δ ppm = 2.4 –2.6 (t, 2H of CH_2O); δ ppm = 3.2 –3.6 (m, 40 H, of 10CH_2CH_2O- ethylene oxide); δ ppm = 5.3-5.4 (d, 2 H of CH =CH).

^{1}HNMR of compound VIa (Fig. 19) show the presence of the following signals: δ ppm = 0.8 (t, 3H of CH_3); δ ppm =1.2-1.4 (m, 24 H of 12CH_2 alkyl fatty chain); δ ppm 2.4-2.6 (t, 2H of CH_2O); δ ppm = 3.2 –3.6 (m, 28 H, of CH_2CH_2O- ethylene oxide); δ ppm = 5.3-5.4 (d, 2 H of CH =CH).

1.2.2. Preparation of anionic surfactants:

It was completed in two steps reaction according to Scheme 4. First one as mentioned above is the preparation of nonionic surfactants (compounds from IIIa-c to VIIIa-c); which are considered as starting materials for preparation of anionic surfactants. This is completed by sulphation of compounds (IIIa-c to VIIIa-c) by using saturated solution of sodium bisulphate and sodium sulphate, followed by neutralization with NaOH to give anionic surfactants IXa-c-XIVa-c (Scheme 4).



IX_{a-c}-XIV_{a-c}

Where R = C_8H_{17}; $C_{10}H_{21}$; $C_{12}H_{25}$; $C_{14}H_{29}$; $C_{16}H_{33}$; $C_{18}H_{37}$ and n = 5, 8 and 12 corresponding to IX_{a-c}-XIV_{a-c} respectively.

Scheme (4)

The structures of the synthesized anionic surfactants were confirmed by:

IR spectroscopy: the IR spectrum of compounds IX_b (Fig. 20); Xa (Fig. 21); XII_c (Fig. 22); XIV_b (Fig. 23) show at $\nu = 1404\ cm^{-1}$ is characteristic for SO_3; beside the basic bands mentioned in the elucidation of the structure.

^{1}HNMR of compounds XIIIa (Fig. 24) show the presence of the following signals: δ ppm = 0.8 (t, 3H of CH_3); δ ppm = 2.2-2.4 (m, 30H of CH_2 alkyl fatty chain); δppm = 3.2-3.6 (m, 28H of CH_2CH_2O.[EO]).

Surface Active Properties of The Synthesized Nonionic Surfactants

The surface active and related properties, including surface and interfacial tensions, cloud point, wetting, emulsification and foaming properties were investigated systematically under neutral conditions, in aqueous solution of the prepared compounds in order to evaluate their possible applications in different industrial fields.

1.1 Surface and interfacial tensions:

In the evaluation of the prepared surfactants by surface and interfacial tensions measurements, two factors, efficiency and effectiveness need to be considered. Ross[100] suggested that a good measurement of efficiency is the amount of a surfactant required to reduce the tension by 20 dyne/cm and the minimum tension obtainable with the surfactant solution measured its effectiveness. The surface and interfacial tensions of the prepared surfactants were determined according to Findly[101]. The measured surface and interfacial data given in Tables 1, 2, 3 and 4. The values follow the general term, that the surface and interfacial tensions were increased with increasing the number of ethylene oxide moles added to the molecule and also with increasing in number of carbon atoms in the hydrophobic part at the same ethylene oxide moles[102]. It was seen that the surfactants IIa-f, showed good surface tension than Ia-f this may be due to presence of ester group in their molecule beside amide group [103]. Also anionic surfactants $IX_{a\text{-}c}$ to $XIV_{a\text{-}c}$ showed good surface activity than nonionic $III_{a\text{-}c}$ to $VIII_{a\text{-}c}$.

1.2. Foaming height:

It was reported that nonionic surfactants have low foam, on the other hand, the foam height of the prepared surfactants increase with increasing ethylene oxide moles per molecules[104], the foam height was investigated for the prepared nonionic and anionic surfactants. The measured data of nonionic surfactants were tabulated in Tables 1, 2 and 3, while for anionic surfactants were in Table 4. It was observed that at constant number of ethylene glycol n = 5 units) the foaming height was increased with increasing alkyl chain length, also it was noted that compounds IIa-f have low foaming than Ia-f. For foaming data at Table 3. the foaming height was gradually increasing with both number of ethylene oxide moles in the molecule at the same number of carbon atom and with increasing number of carbon atoms in their molecule i.e., the maximum foam height was recorded for compound with high moles of ethylene oxides (n = 12) and molecules with high hydrophobic part (C18). Also the anionic surfactants showed good foam comparing to that produced by nonionic one.

1.3. Cloud Point:

Cloud point is an important property of nonionic surfactants. Below this temperature a single phase of molecular or micellar solution exists; above it, the surfactant loses sufficient water solubility and a cloudy dispersion results. Several trends in cloud point with surfactant molecular structure are commonly known: cloud point increases with relative polyoxyethylene chain content and decreases with increasing alkyl carbon chain length[105].

As the temperature of the solution is gradually raised the solution becomes cloudy (due to dehydration of the ethylene glycol chain) and the temperature at which this occurs is referred to as the cloud point of the surfactant. Cloud point changes with changing concentration of the surfactant solution and the trade literature usually quotes the cloud point of a 1% solution.

The cloud point of the prepared nonionic surfactants was measured according to Durham[106], the cloud points of the synthesized surfactants were shown in Tables 1, 2 and 3. Generally, the cloud point increases with increasing the number of ethylene oxide moles per starting hydrophobic molecule[106, 96]. All the synthesized nonionic surfactants showed high cloud points, that gave the good performance in hot water. Also it was found that the compounds with high moles of ethylene oxide (12 moles per molecule) showed high cloud point >100, and also that with high hydrophobic chain, this may be attributed to formation of many hydrogen bond with water.

1.4 Kraft point:

Kraft point of the prepared anionic surfactants was measured as the temperature where 1% dispersion becomes clear on gradual heating. All the prepared surfactants are freely soluble in water at 1 wt % concentration and at any temperature. The prepared anionic surfactants showed lower values of Kraft point (ranged from 7-16 °C) Table 4, the Kraft point's measurements proved that the higher molecular weight, the higher the kraft points [107].

1.5. Emulsification Properties:

The Emulsion properties were measured using standard procedure[82] the measured data was given in Table 1, 2, 3 and 4. It was found that greater emulsifying properties values were obtained for compounds containing less ethylene oxide moles, where the emulsifying properties increase with decreasing number of ethylene oxide moles. It was declared that the surfactants of low emulsification properties can be used in many field of application, like in pesticide and cosmetics formulation. The prepared anionic surfactants showed higher emulsification properties than nonionic one (c.f. Table 4).

1.6. Wetting Time:

It is the concentration of the surfactant at which a special purpose cotton fabric circle sinks after a time of 100 second, nonionic surfactants are among the most powerful wetting; wetting properties of 0.1 wt % solutions in distilled water were measured by Draves test. All the synthesized surfactants are efficient wetting agents Tables 1, 2, 3 and 4. The wetting time of the surfactants get worse with increasing number of ethylene oxide in their molecules (less wetting time with compounds containing more ethylene oxide contents), it was reported that a nonionic surfactants with low ethylene oxide content has the most efficient wetting time[107]. This is sufficient to define the wetting time that depends on the ethylene oxides moles in molecules. The anionic surfactants (Table 4) showed higher wetting time (less wetting agent) than the nonionic one.

Biodegradability:

In the further alcohol biodegradation, the length of the alkyl chain is a crucial factor, it has significant effect in the rate of biodegradation of nonionic surfactants containing alcohol in their molecule. Ethylene oxide chain length also has some effect on the surfactant biodegradation [108].

The free fatty alcohol fraction contains free alcohol as such and short-chained ethoxylates (having 1–3 oxyethylene subunits). This fraction's compounds are relatively reluctantly biodegraded in the case of C16–18 fatty alcohols and is moderately biodegraded in the case of C12 fatty alcohol moiety[109, 110].

In the course of degradation the river die-away tests was followed by surface tension measurements[111] [Eter et al.]. The biodegradability data were given in Tables 5-8. within the experimental accuracy all the prepared nonionic (Ia-f); (IIa-f); (IIIa-c to IVa-c) and anionic (IXa-c to XIVa-c) types seem to degrade easy. It can be seen that compounds (Ia-f) and (IIa-f) have good degradability, while compounds with long chain carbon atoms and high ethylene oxide moles seem low degradability. This may be attributed to the higher hydrophobic content of their molecules. Also the biodegradability rate decreased with increasing the number of the carbon atoms in the hydrophobic part. This led to conclusion that a longer hydrophobic part makes the diffusion of the molecule through the cell membrane and their degradation more difficult.

Table 1. Surface properties of the synthesized nonionic surfactants (Ia-f):

Compd.	**Surface tension dyne/cm 0.1 %**	**Interfacial tension dyne/cm 0.1 %**	**Foaming height 0.1 %**	**Cloud point °C 1.0 %**	**Emulsion Time sec. 20 mml**	**Wetting Time sec. 0.1 %**
Ia; C8	40	12	50	68	62	90
Ib; C10	41	11	70	74	60	95
Ic; C12	42	12.5	85	75	58	100
Id; C14	43	13	95	76	55	105
Ie; C16	45	13	98	80	52	110
If; C18	46	14	105	90	50	115

Table 2. Surface properties of the synthesized nonionic surfactants (IIa-f):

Comps no.	**Surface tension dyne/cm 0.1 %**	**Interfacial tension dyne/cm 0.1 %**	**Foaming height 0.1 %**	**Cloud point °C 1.0 %**	**Emulsion Time sec. 20 mml**	**Wetting Time sec. 0.1 %**
IIa; C8	34	11	45	73	58	90
IIb; C10	35	11	70	76	57	93
IIc; C12	35.5	11.5	80	78	53	95
IId; C14	37	12	90	80	50	98
IIe; C16	42	12.5	95	85	48	100
IIf; C18	43	13	100	>100	47	110

Table 3. Surface properties of the synthesized nonionic surfactants (IIIa-c to VIIIa-c)

Compds. No.	No. of carbon atom	No. of EO moles	Surface tension dyne/cm 0.1 %	Interfacial tension dyne/cm 0.1 %	Foaming height mm 0.1 %	Cloud point °C 1.0 %	Emulsion Time sec. 20 mml	Wetting Time sec. 0.1 %
IXa-c	a	5	35	9	55	70	60	83
	b	8	36	9	60	75	58	80
	c	12	36	10	65	80	55	79
Xa-c	a	5	37	10	70	75	58	85
	b	8	38	10	75	85	56	82
	c	12	38	11	80	>100	56	80
XIa-c	a	5	38	11	85	75	55	88
	b	8	38	12	90	90	53	85
	c	12	39	13	95	>100	50	84
XIIa-c	a	5	40	12	100	78	53	90
	b	8	41	13	105	>100	52	88
	c	12	41	14	110	>100	48	86
XIIIa-c	a	5	43	12	100	80	48	95
	b	8	43	13	115	>100	47	93
	c	12	44	13	120	>100	45	90
XIVa-c	a	5	42	13	110	85	46	100
	b	8	44	14	120	>100	44	98
	c	12	46	15	130	>100	42	95

Table 4. Surface properties of the synthesized anionic surfactants (IXa-c to XIVa-c).

Compds No.	No. of carbon atom	No. of EO moles	Surface tension dyne/cm 0.1 %	Interfacial tension dyne/cm 0.1 %	Foaming height mm 0.1 %	Kraft point ^{o}C 1.0 %	Emulsion Time sec. 20 mml	Wetting Time sec. 0.1 %
IXa-c	C8 a	5	30	7	100	7	210	150
	b	8	31	7.5	105	8	206	145
	c	12	32	8	108	8	205	143
Xa-c	C10 a	5	32	8	110	9	200	160
	b	8	33	8.5	115	10	195	158
	c	12	33	9	115	12	195	155
XIa-c	C12 a	5	34	9	118	9	190	165
	b	8	35	10	123	11	190	163
	c	12	36	10.5	125	12	185	158
XIIa-c	C14 a	5	35	9	120	10	185	166
	b	8	36	10	125	12	183	164
	c	12	37	11	128	13	180	160
XIIIa-c	C16 a	5	38	10	130	12	175	175
	b	8	38	10.5	135	12	172	173
	c	12	40	12	136	14	170	170
XIVa-c	C18 a	5	37	12	130	12	165	185
	b	8	39	12.5	135	14	160	180
	c	12	42	14	140	16	156	172

Table 5. Biodegradability of the prepared nonionic surfactants (Ia-f).

Compds.	1st day	2nd day	3rd day	4th day	5th day	6th day	7th day	8th day
Ia	60	65	73	79	85	90	96	-
Ib	55	59	65	73	80	85	91	98
Ic	52	58	65	72	78	84	89	95
Id	51	56	62	69	77	84	88	95
Ie	49	55	60	66	72	79	84	92
If	45	50	57	63	70	76	82	90

Table 6. Biodegradability of the prepared nonionic surfactants (IIa-f).

Compds.	1st day	2nd day	3rd day	4th day	5th day	6th day	7th day	8th day
Ia	56	65	73	81	89	96	-	-
Ib	55	63	71	79	87	95	-	-
Ic	53	61	69	75	82	89	95	-
Id	52	59	65	72	80	86	92	-
Ie	52	58	63	69	75	82	89	96
If	50	55	60	66	72	80	86	93

Table 7. Biodegradability of the synthesized nonionic surfactants (IIIa-c to VIIIa-c).

Comps	No. of Carbon atoms	No. of EO. moles	1st day	2nd day	3rd day	4th day	5th day	6th day	7th day	8th day	9th day
IIIa-c	C8 a	5	50	55	61	69	78	86	92	-	-
	b	8	50	56	62	70	79	85	93	-	-
	c	12	48	55	60	68	75	82	89	95	-
IVa-c	C10 a	5	48	54	60	67	75	82	90	96	-
	b	8	46	52	58	65	72	80	88	95	-
	c	12	45	50	55	62	69	75	82	91	-
Va-c	C12 a	5	46	52	58	64	70	78	86	94	-
	b	8	45	52	59	63	70	78	85	94	-
	c	12	44	50	56	62	69	76	84	92	-
VIa-c	C14 a	5	45	51	58	63	69	77	85	93	-
	b	8	44	50	57	62	68	75	83	90	96
	c	12	42	49	55	61	69	76	83	90	97
VIIa-c	C16 a	5	45	50	57	62	70	76	84	92	-
	b	8	44	49	56	62	69	75	81	89	96
	c	12	42	48	55	60	68	74	80	88	95
VIIIa-c	C18 a	5	44	50	56	62	68	75	82	89	96
	b	8	42	48	55	61	67	74	80	88	95
	c	12	40	46	53	60	67	72	79	86	94

Table 8. Biodegradability of the synthesized anionic surfactants (IXa-c to XIV a-c)

Comps	No. of Carbon atoms		No. of EO. moles	1st day	2nd day	3rd day	4th day	5th day	6th day	7th day	8th day	9th day
IXa-c	C8	a	5	54	60	68	75	82	89	97	-	-
		b	8	52	58	66	74	80	88	96	-	-
		c	12	51	56	65	73	80	87	94	-	-
Xa-c	C10	a	5	52	59	66	74	81	88	96	-	-
		b	8	52	58	65	73	80	87	95	-	-
		c	12	51	56	63	71	79	86	94	-	-
XIa-c	C12	a	5	51	57	64	72	79	86	93	-	-
		b	8	50	56	62	69	76	84	90	97	-
		c	12	50	56	51	69	75	83	90	96	-
XIIa-c	C14	a	5	50	56	63	70	77	85	93	-	-
		b	8	48	55	62	70	75	83	89	95	
		c	12	47	53	60	68	74	81	88	94	-
XIIIa-c	C16	a	5	48	54	61	68	73	80	86	93	-
		b	8	48	53	60	66	72	78	85	92	-
		c	12	47	53	59	65	71	78	84	90	96
XIVa-c	C18	a	5	47	53	59	67	72	79	85	92	-
		b	8	46	52	58	65	71	78	83	89	95
		c	12	45	51	57	62	69	75	81	87	93