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AMMONIUM SALTS OF PALM OIL FATTY ACIDS

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ABSTRACT

Palm oil acid fraction-based monoethanolamine (MEA), diethanolamine (DEA), triethanolamine (TEA), methylaminoethanol (MAE), piperidine (PP), morpholine (Mr), diethylamine (DEtA), triethylamine (TEtA), ethylenediamine (EDA), diethylenetriamine (DETA) and polyethylenepolyamine (PEPA) salts were synthesised. By stalagmometric method it was established that the synthesised salts are highly surface-active. DEA and TEA-based salts reduce the interface tension at the air-water surface from 72.0 mN m^{-1} down to 25.7 and 24.2 mN m^{-1} (25°C). Using surface tension isotherms of the salts their colloidal-chemical parameters were calculated. An increase in the number of ethylol groups in the surfactant composition decreases critical micelle concentration (CMC) and area per molecule at the interface (A_{min}) and increases effectiveness of surface tension reduction (π_{CMC}) and surface excess concentration (Γ_{max}). It was revealed that DETA-based salt has a higher petroleum-collecting ability than the other salts.

Keywords: surfactant, ammonium salt, surface tension, misellisation, adsorption.

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INTRODUCTION

Fatty carboxylic acids of palm oil origin are valuable initial products for obtaining surfactants. Such surfactants are expected to have high biodegradability as well as non-toxicity (Goh *et al*., 2011). There are reports on surfactants based on fatty acid and ethanolamines (Koganei, 1923). They find wide usage in personal care and household detergents (Musial and Kubis, 2003). It has been reported (Novales *et al*., 2008) that ammonium-type salts based on palmitic acid, 8-hydroxy palmitic acid and 12-hydroxy stearic acid (counterion is tetrabutyl ammonium) generate isotropic micellar solutions but the salts of these acids with ethanolamines form turbid and birefringent solutions of lamellar character. The solubility of the salts in well-water is higher than in environmentally relevant water. According to Prats *et al*. (1999), such salts are even less toxic in environmentally relevant waters than in clean water. The biodegradation of these salts positively effects soils. Adsorption as well as corrosion-inhibiting properties of triethanolammonium salt of unsaturated C_{18} fatty acids were studied (Szauer and Brandt, 1981). Corrosion of iron in 0.5 M deaerated H_2SO_4 solution was considered. It was established that the preferential adsorption of fatty acids over ammonium cations took place.

Fatty acids of palm oil origin may be used in rising amounts for production of cheap laundry and toilet soaps as well as various surface-active compositions (Bhattacharyya and Chatterjee, 1984). According to Kuntom *et al*. (1996), the production of soaps based on distilled fatty acids of palm oil origin in Malaysia is well organised.

Japanese authors (Ogoshi and Miyawaki, 1985) proposed soap making on the basis of saponification of methyl esters of fatty acids derived from palm oil, tallow and coconut oils. The toilet soaps produced are of a high quality.

Many interesting surfactants based on palm oil are reported (Lim *et al*., 2000; 2009; Lim and Ahmad, 2001; Kifli *et al*., 1991; Al-Mulla *et al*., 2009). Thus, surfactants on the basis of palm oil acid fraction and amine group-containing compounds are of great interest. This article is devoted to the synthesis

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of surfactants based on palm oil acid fraction and nitrogenous compounds as well as the determination of surface activity parameters and their possible application in petroleum-collecting and dispersing.

EXPERIMENTAL METHOD

Materials

Reagents. IR spectra were recorded on a model FT-IR, Spectrum BX spectrometer using KBr disks. The pH of the solutions was measured using an pH-150M potentiometer with an ESKL-08M combined laboratory electrode adjusted by standard buffer solutions.

Palm oil was obtained from Ngo Chew Hong Oils and Fats (M) Sdn Bhd, Malaysia. Methylamine (MEA) (purity>99% Merck®, Germany), diethanolamine (DEA) (purity>98.5% Merck®, Germany), triethanolamine (TEA) (purity>99% Merck®, Germany), MAE (>98 % purity, Merck®, Germany), diethylamine (TEtA) (purity>98% Merck®, Germany), DEtA (purity>98%, Russian Federation), diethylenetriamine (DETA) (purity>96%, Alfa Aesar GmbH & Co. KG, Germany), polyethylenepolyamine (PEPA) (purity>96%, 'INKHIMTEK Ltd', Russia) were of analytical grade. Ethylenediamine (EDA), morpholine (Mr) and piperidine (PP) were from the 'Kazanorgsintez' Joint Stock Company (Russia), all the reactants were used without further purification.

Palm oil was hydrolysed with NaOH solution and corresponding fatty acids and glycerine were obtained. The palm oil fatty acid is white solid wax, acid number 218.6 mg KOH g^{-1}.

Waters. In the experiments, three kinds of water were used: 1. distilled water, 2. fresh water, and 3. the Caspian sea water having the following physico-chemical characteristics and composition: ρ^{20}=1.0098 g ml^{-1}, pH=7.7. Contents of ions and other species (mg $litre^{-1}$): Na^+-2650; K^+-90; Ca^{2+}-250; Mg^{2+}-900; NH_4^+-0.15; Cl^--500; SO_4^{2-}-2800; NO_3^--0.1; PO_4^{3-}-0.35; NO_2^--0.007; SiO_2-0.5; petroleum products -0.005%; dissolved oxygen -8 mg $litre^{-1}$, total hardness -69.0 mg-eq $litre^{-1}$.

Methods

Surface-tension measurements. All the surface tension measurements were carried out using deionised water to make the solutions. The solutions maintained at the desired temperature were measured 45 s after being transferred to the thermostated measuring dish. The actual temperature within the dish was controlled prior to and after the measurement by means of a thermocouple. Deviations from the desired temperature were ±0.2°C. The surface tension as a function of concentration was measured at 25°C using a drop volume stalagmometer. For mixed solutions, particularly at low surfactant concentrations, the approach to equilibrium can take several hours, thus it was assumed arbitrarily that equilibrium was less than 0.2 mN m^{-1} over 20 min. The reproducibility (including long equilibration times and/or contamination effects) was ±1%.

Synthesis of palm oil fatty acid ammonium salts. Palm oil fatty acid (0.1 mol) was added to 0.1 mol of (alkanol) amine and stirred for 12-15 hr at 60°C temperature. Palm oil acid fraction was neutralised with (alkanol) amine (Scheme I-V) as follows. Reactions were carried out in the absence of a solvent. During the reaction, heat liberation was observed.

I: (salt based on DEtA), yellow paste, soluble in water, ethanol, isopropanol, acetone, benzene, hexane and carbon tetrachloride. The pH of 0.1 M aqueous solution equals 8.8.

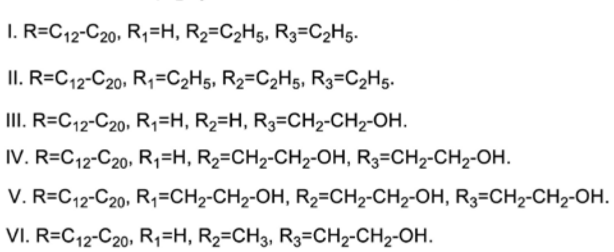
II: (salt based on TEtA), yellow paste, soluble in water, ethanol, isopropanol, acetone, benzene, hexane and carbon tetrachloride. The pH of 0.1 M aqueous solution is 9.2.

III: (salt based on MEA), yellow paste, soluble in ethanol, isopropanol, acetone, benzene, carbon tetrachloride and partially soluble in water. The pH of 0.1 M aqueous solution is equal to 9.9.

IV: (salt based on DEA), yellow-transparent waxy substance, soluble in water, ethanol, isopropanol, acetone, benzene, hexane and carbon tetrachloride. The pH of 0.1 M aqueous solution is 9.8.

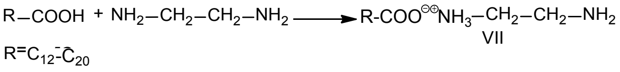
V: (salt based on TEA), yellow-transparent waxy substance, soluble in water, ethanol, isopropanol, acetone, benzene, hexane and carbon tetrachloride. The pH of 0.1 M aqueous solution equals 9.6.

VI: (salt based on MAE), yellow-transparent waxy substance, soluble in water, ethanol, isopropanol, acetone, benzene, hexane and carbon tetrachloride. The pH of 0.1 M aqueous solution is 10.1.



Scheme 1: Reaction scheme of the synthesis of palm oil acid (alkanol) amine salts.

VII: (salt based on EDA), white-yellow solid, soluble in water, ethanol, isopropanol, acetone, benzene, hexane and carbon tetrachloride. The pH of 0.1 M aqueous solution equals 10.8.



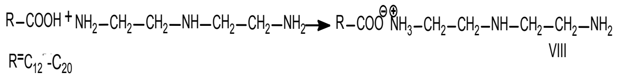
$$R{-}COOH + NH_2{-}CH_2{-}CH_2{-}NH_2 \longrightarrow R{-}COO^{\ominus}\overset{\oplus}{N}H_3{-}CH_2{-}CH_2{-}NH_2$$

VII

$R = C_{12} - C_{20}$

Scheme 2: Reaction scheme of the synthesis of palm oil acid EDA salt.

VIII: (salt based on DETA), white-yellow paste, soluble in water, ethanol, isopropanol, acetone, benzene, hexane and carbon tetrachloride. The pH of 0.1 M aqueous solution is equal to 11.0.



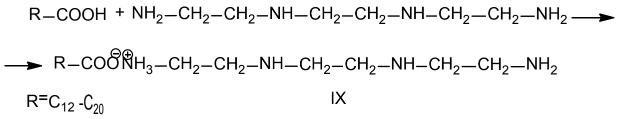
$$R{-}COOH + NH_2{-}CH_2{-}CH_2{-}NH{-}CH_2{-}CH_2{-}NH_2 \longrightarrow R{-}COO^{\ominus}\overset{\oplus}{N}H_3{-}CH_2{-}CH_2{-}NH{-}CH_2{-}CH_2{-}NH_2$$

VIII

$R = C_{12} - C_{20}$

Scheme 3: Reaction scheme of the synthesis of palm oil acid DETA salt.

IX: (salts based on TETA), white-yellow paste, soluble in ethanol, isopropanol, acetone, benzene, carbon tetrachloride and partially soluble in water and hexane. The pH of 0.1 M aqueous solution is 11.3.



$$R{-}COOH + NH_2{-}CH_2{-}CH_2{-}NH{-}CH_2{-}CH_2{-}NH{-}CH_2{-}CH_2{-}NH_2 \longrightarrow R{-}COO^{\ominus}\overset{\oplus}{N}H_3{-}CH_2{-}CH_2{-}NH{-}CH_2{-}CH_2{-}NH{-}CH_2{-}CH_2{-}NH_2$$

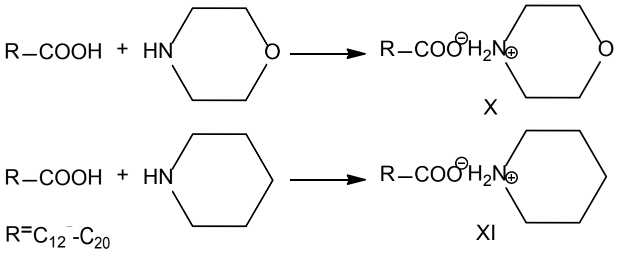
IX

$R = C_{12} - C_{20}$

Scheme 4: Reaction scheme of the synthesis of palm oil acid TETA salt.

X: (salt based on Mr), yellow paste, soluble in water, ethanol, isopropanol, acetone, benzene, hexane and carbon tetrachloride. The pH of 0.1 M aqueous solution is 9.4.

XI: (salt based on PP), white solid, soluble in ethanol, isopropanol, acetone, benzene, carbon tetrachloride and no soluble in water. The pH of 0.1 M aqueous solution is equal to 8.7.



Scheme 5: Reaction scheme of the synthesis of palm oil acid PP and Mr salts.

Determination of petroleum-collecting and petroleum-dispersing capacities. Petroleum-collecting properties of the synthesised fatty acid ammonium salts (in pure state and in the form of 5% wt. water or alcoholic solution) were studied using the crude oil (density and kinematic viscosity at 20°C are 0.86 g cm^{-3} and 0.16 $cm^2\ s^{-1}$) respectively from the Ramana oil field in the Absheron peninsula (Azerbaijan). The surfactant (0.02 g) or its solution (0.02 g) was added to a thin film (thickness 0.15–0.16 mm) of petroleum on the surface of distilled water and the Caspian sea water (separately) in Petri dishes. The maximum values of petroleum collecting coefficient (*K*) are found which is calculated by the formula $K=S_o/S$, where S_o is an area of the surface of initial petroleum film and *S* is an area of the surface of accumulated petroleum (as a thickened spot). Since the moment of the surfactant applying the spot surface area was measured and the *K* values were determined at fixed time intervals (τ).

RESULTS AND DISSCUSSION

Synthesis of Palm Oil Fatty Acid Ammonium Salts

A comparison of IR-spectra of the synthesised salts with that of the acid fraction shows that the absorption band at 1705-1710 cm^{-1} observed in the spectrum of this acid fraction disappears in the IR-spectra of the salts. The FT-IR absorption spectra for both salts showed an absorption band at the 1565-1595 cm^{-1} region characteristic for δ COO^- stretches. This proves the formation of the target salts.

Effect of the Number of the Hydrophilic Groups in the Surfactants on their Surface Activity Properties

Figures 1, 2 and *3* depict the effect of changing the concentration of different surfactants on the surface tension at constant temperature (25°C). These results show that, for each type of surfactant, there is a gradual decrease in the surface tension with increase in concentration for solutions up to a certain level above which a nearly constant value is obtained. Such constant values of surface tension were found to be 41.0, 28.8, and 27.9 mN m^{-1} for the surfactants containing respectively one, two and three ethylol groups attached to their N atom. For the surfactant containing one ethylol and one methyl group bonded to the nitrogen atom, γ_{CMC} is equal to 26.6 mN m^{-1}. Constant values of surface tension were found to be 25.7, and 24.2 mN m^{-1} for the surfactants having correspondingly two and three ethyl groups attached to their N atom. The constant values of surface tension were found to be 28.0, 29.1, and 29.6 mN m^{-1} for the surfactants containing respectively one, two, and four ethylenediamine groups attached to their COO^- group. The concentration of surfactant corresponding to a sudden increase of the surface tension from its constant value is known as the critical micelle concentration (CMC). The CMC values were determined from the abrupt change in the slope in the γ-lnC plot. The values of the CMC are 3.00×10^{-2}, 2.70×10^{-2}, and 1.50×10^{-2}, mol dm^{-3} for

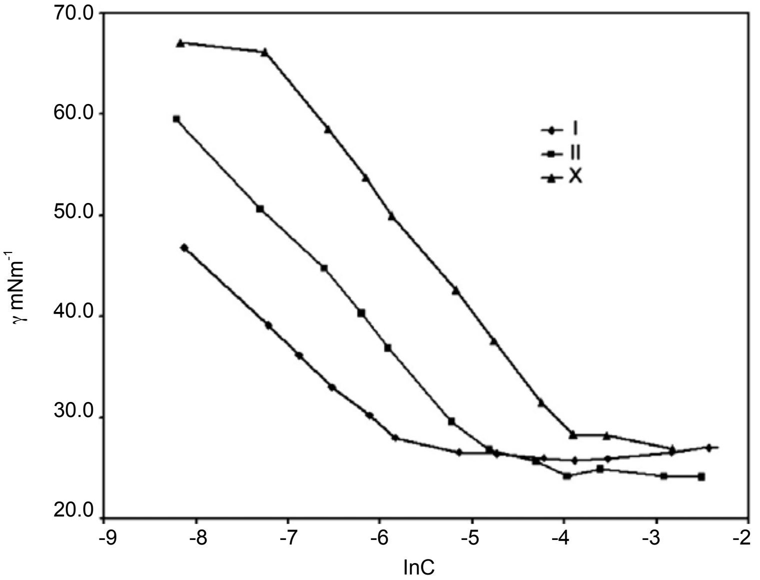


Figure 1. Variation of the surface tension with logarithm of the prepared I, II, X surfactants concentrations in water at 25°C.

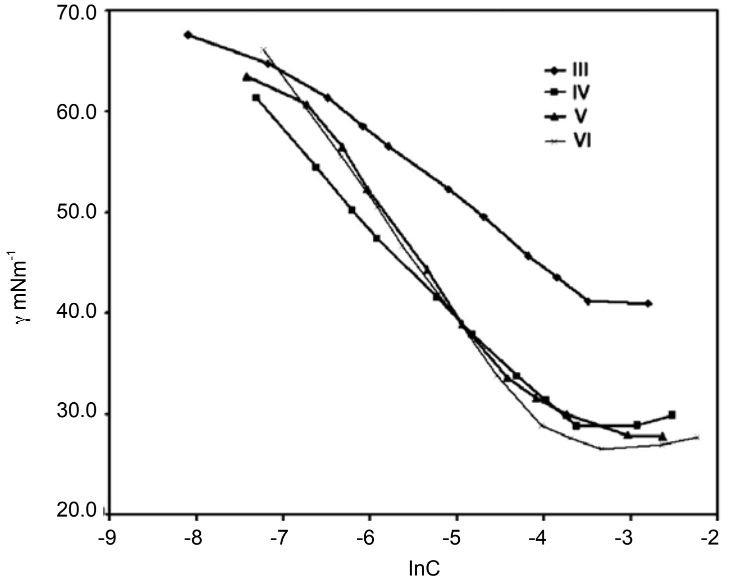


Figure 2. Variation of the surface tension with logarithm of the prepared III-VI surfactants concentrations in water at 25°C.

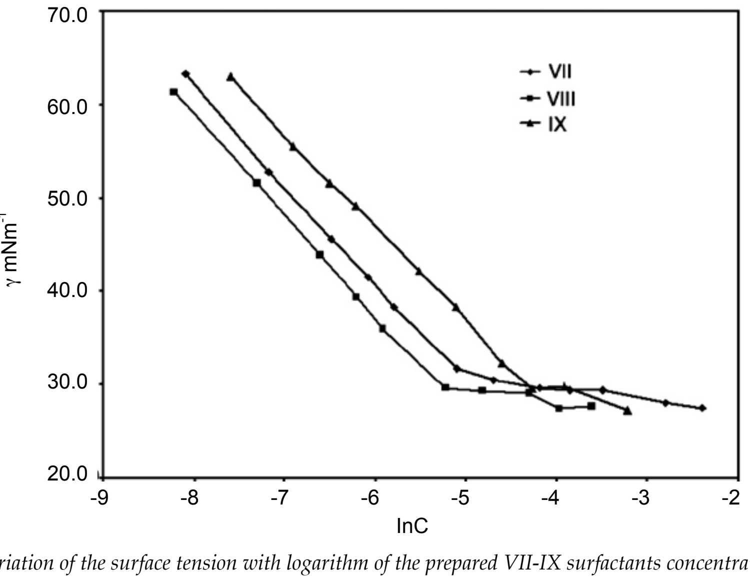


Figure 3. Variation of the surface tension with logarithm of the prepared VII-IX surfactants concentrations in water at 25°C.

the surfactants containing correspondingly one, two, and three ethylol groups attached to their N-atom. This decrease in the values of CMC shows the effect of increasing the number of ethylol groups of the surfactants on their surface tension-concentration relationship. Increasing the number of OH groups at the N-atom of fatty acid MEA salt from one to three causes a gradual reduction in the respective CMC and, in turn, the surface tension. But the CMC rises when the number of ethyl groups at the nitrogen atom is increased from 2 to 3, which may be related to the steric factor. Rosen (2004) has mentioned that the number of ionic groups in the surfactant molecule affects the CMC. Furthermore, the CMC in an aqueous solution depends not only on the structure of the surfactant but also on the number and kind of counterions in the system (Rosen, 2004). Thus, the greater the number of the hydrophilic (ethylol) groups, the higher the solubility in water and, in turn, the lower the surface tension in solution. Meanwhile, the steric hindrance between the three ethyl groups is expected to be larger in comparison with the surfactant containing two ethyl groups. This steric hindrance increases the CMC.

As can be seen from *Table 1*, in fatty acid MEA salt, substitution of the H atom attached to the nitrogen by a methyl group sharply increases the surface activity of the substance. It is also noticeable that at 25°C the CMC of III is 3.00×10^{-2} mol dm^{-3}, whereas CMC of the VI is 2.00×10^{-2} mol dm^{-3}. Hence, it may be said that the introduction of methyl group enhances hydrophobic interaction and decreases the CMC.

A plot of surface tension as a function of equilibrium concentration of surfactant in one of the liquid phases, rather than an adsorption isotherm, is generally used to describe adsorption at this interface. The concentration of surfactants at the surface may therefore be calculated from surface tension data by using Ephatimol (Rosen, 2004):

$$\Gamma = \frac{1}{RT}\left(\frac{-\partial \gamma}{\partial \ln C}\right)_T \quad (1)$$

where $\left(\frac{-\partial \gamma}{\partial \ln C}\right)_T$ is the slope of the plot of γ *vs*. lnC at a constant temperature (T), and *R is* the gas constant in J×mol^{-1}×K^{-1}. The surface excess concentration at surface saturation (Γ_{max}) can be readily found and it is a useful measure of the effectiveness of adsorption of surfactant at the liquid-gas or liquid-liquid interface, since it is the maximum value which adsorption can attain.

From the surface excess concentration, the area per molecule at surface is calculated using Equation (2).

$$A_{min} = \frac{10^{16}}{N_A \Gamma_{max}} \quad (2)$$

where N_A is Avogadro's number.

The effectiveness of surface tension reduction, $\pi_{CMC} = \gamma_0 - \gamma_{CMC}$, where γ_0 is the surface tension at the air/water border and γ_{CMC} is the surface tension at the border aqueous solution of surfactant/air at CMC, was determined at 298 K temperature. The Γ_{max}, A_{min} and γ_{CMC} values are calculated and listed in *Table 1*. The data for (I-V) listed in *Table 1* show that the minimum area per molecule at the aqueous solution/air interface is enlarged with an increase of the number of ethylol and ethyl units in the molecule. The values of A_{min} increase with the decrease in the

TABLE 1. SURFACE PROPERTIES OF THE SALTS BASED ON PALM OIL FATTY ACID AND NITROGENOUS COMPOUNDS AT 298 K TEMPERATURE[a]

Surfactants	CMCx10^2, mol·dm^{-3}	Γ_{max}x10^{10}, mol·cm^{-2}	A_{min}x10^2, nm^2	γ_{CMC}, mNxm^{-1}	π_{CMC}, mNxm^{-1}	pC_{20}	ΔG_{mic}, kJxmol^{-1}	ΔG_{ad}, kJxmol^{-1}
I	0.37	1.62	102.4	25.7	46.3	3.84	-13.87±0.02	-16.73±0.02
II	0.87	1.98	83.9	24.2	47.8	3.23	-11.75±0.02	-14.17±0.02
III	3.00	1.36	122.4	41.0	31.0	2.21	-8.69±0.01	-10.97±0.02
IV	2.70	1.77	93.7	28.8	43.2	2.77	-8.95±0.01	-11.39±0.02
V	1.50	2.42	68.7	27.9	44.1	2.60	-10.41±0.02	-12.23±0.02
VI	2.00	2.38	69.8	26.6	45.4	2.64	-9.69±0.02	-11.60±0.02
VII	0.68	2.13	78.0	28.0	44.0	3.11	-12.37±0.02	-14.43±0.02
VIII	0.49	2.17	76.5	29.1	42.9	3.19	-13.18±0.02	-15.15±0.02
IX	1.40	2.02	82.1	29.6	42.4	2.85	-10.58±0.02	-12.67±0.02
X	1.95	2.31	72.0	27.0	45.0	2.62	-9.76±0.02	-11.71±0.02

Note: [a]CMC - critical micelle concentration. γCMC - equilibrium surface tension at the CMC. Γ_{max}- the maximum surface excess concentration at the aqueous solution/air interface. A_{min} - the minimum area per surfactant molecule at the aqueous solution/air interface. πCMC - effectiveness of surface tension reduction. pC_{20} - the efficiency of surface adsorption, measured as the negative logarithm of the bulk surfactant concentration C_{20} required to reduce the surface tension of water by 20 mN m^{-1}. ΔG_{mic} - change of standard free energy of micellisation. ΔG_{ad} - change of standard free energy of adsorption.

number of ethylol groups in the molecule, in accord with the observation of others (Omar and Abdel-Khalek, 1998; Asadov *et al.*, 2010). The A_{min} in the case of MAE salt is larger in comparison with MEA salt due to an additional methyl group.

The data on A_{min} and Γ_{max} indicate the dependence of the effectiveness of adsorption at the aqueous solution/air interface from the structure of surfactants.

The effectiveness of surface tension reduction in these compounds shows a steady rise with an increase in the number of ethylol and ethyl groups. The extension of the ethyleneamine chain of fatty acids counterion decreases the values of π_{CMC}.

Further, the values of efficiency of adsorption pC_{20}, are useful in comparing the efficiency of adsorption of surfactant at air/water interface (Rosen, 2004). The larger the pC_{20} value, the more efficiently the surfactant is adsorbed at the interface and the more efficiently it reduces surface tension.

Standard Free Energies of Micellisation and Adsorption at the Aqueous Solution/air Interface

Table 1 lists standard free energies of micellisation, ΔG_{mic}, and adsorption, ΔG_{ad}, for the compounds investigated. The ΔG_{mic} values were calculated by use of the relationship:

$$\Delta G_{mic} = RT \ln CMC \quad (3)$$

The ΔG_{ad} values were calculated by use of the relationship:

$$\Delta G_{ad} = RT \ln CMC - \pi_{CMC} A_{CMC} \quad (4)$$

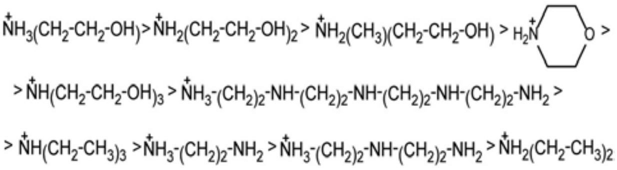
The standard state for the adsorbed surfactant here is a hypothetical monolayer at its minimum surface area/molecule, but at zero surface pressure. When π_{CMC} is in mN/m, $A_{CMC}(= A_{min})$ is in nm^2, and R is in $J \times mol^{-1} \times K^{-1}$, the value of ΔG_{ad} in $kJ \times mol^{-1}$ is obtained by the expression:

$$\Delta G_{ad} = RT \ln CMC - 6.023 \times 10^{-1} \pi_{CMC} A_{CMC} \quad (5)$$

The found values of ΔG_{mic} and ΔG_{ad} are listed in *Table 1*. From these data, it may be concluded that the micellisation prosess has a spontaneous character ($\Delta G_{mic}<0$). As the number of ethylol groups rises, the values of ΔG_{mic} decrease. When passing from ethyl groups to ethylol groups, the values of ΔG_{mic} increase.

All values found for ΔG_{ad} are negative. Moreover, they are more negative than those of ΔG_{mic}, *i.e.*, adsorption of the mentioned surfactants at the water/air interface is associated with a larger decrease in free energy of the system.

A comparison of ΔG_{mic} and ΔG_{ad} values for various types of ionic surfactants, each containing a straight alkyl chain of fatty acid fraction, indicates that the resistance to micellisation and adsorption of the head groups decreases in the order:



This indicates that the decrease in the number of ethylol groups at *N* atom and shortening of the chain containing ethylenediamine groups in surfactants inhibit both micellisation and adsorption, while steric effects in surfactants hydrophilic part inhibit micellisation more than adsorption at the aqueous solution/air interface.

The Prepared Surfactants as Oil Slicks Collection Agent

In this work, petroleum-collecting and petroleum-dispersing properties of the surfactants have been studied on the example of thin films of Ramana crude oil on the surface of distilled, fresh and sea waters, the surfactant being taken in undiluted form and the as 5% wt alcoholic and water solutions.

Salt VIII in an unthinned state and as 5% aqueous solution exhibits a high petroleum-collecting capacity in distilled water (K=30.4, τ=72 hr). In distilled and sea waters, salt VIII is more effective when used in unthinned form. Thus, in fresh water *K* is 10.1 (τ=72 hr) whereas in the sea water *K* has the value 5.8 (τ=72 hr). When salt VI is applied in distilled water, during the initial hours petroleum-collecting effect is relatively weak but then *K* reaches the magnitude of 30.4. Salt **X** has a higher effect than other salts in fresh water (K=13.4, τ=72 hr). Salt IV has a higher petroleum-collecting coefficient (K=10.1, τ=168 hr) than the other salts and their 5% aqueous solutions. It has a high petroleum-collecting capability in distilled and fresh waters (τ=168 hr). Salts I, II, III, IV and VII also possess noticeable petroleum-collecting and petroleum-dispersing capacities in soft waters.

CONCLUSION

Various ammonium salts have been obtained by the interaction of palm oil fatty acid with compounds containing amine group (DEtA, TEtA, MEA, DEA, TEA, MAE, EDA, DETA, PEPA, Mr, PP). The synthesised ammonium salts were characterised by different physico-chemical parameters and their surface-active properties were measured. Increasing the number of ethylol groups in the surfactant composition decreases the CMC and A_{min} values and increases the π_{CMC} and Γ_{max} values. The larger decrease of ΔG_{ad} compared with ΔG_{mic} indicates that for molecules of the investigated surfactants adsorption is more preferable than micellisation.

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