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ORIGINAL ARTICLE

Undecanoic and 10-Undecenoic Acid-Based Amidobetaines and Amidoamine Oxides

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Abstract Three types of undecanoic and 10-undecenoic acid-based surfactants were synthesized in the present work: amphoteric amidobetaines, cationic amidobetaine chlorides and nonionic amidoamine oxides. Structural characterizations of synthesized compounds were based on nuclear magnetic resonance (NMR) (^{1}H and ^{13}C) and mass spectrometry. Surface properties, such as critical micelle concentration (CMC), surface tension at cmc (γ_{cmc}), efficiency of surface adsorption (pC20), surface excess ($\Gamma_{\max}$) and minimum area per molecule ($A_{\min}$) at the air–water interface, were determined by surface tension methods. Fluorescence probing techniques were also employed for the measurement of CMC, as well as steady state anisotropy (r) at the micellar core. The CMC of the studied surfactants follow the order: amidobetaine > amidobetaine chloride > amidoamine oxide. The influence of the terminal double bond in the hydrophobic alkyl chain on CMC was also assessed, and a significant increase in CMC was found due to the introduction of the double bond in the cases of amidobetaine chlorides and amidoamine oxides. These two types of surfactants showed higher rigidity at the micellar core compared to their corresponding unsaturated counterparts. However, such influence of unsaturation on the hydrophobic moiety was not observed in the case of amidobetaines. In all three types of surfactants, the saturated surfactant exhibited a lower γ_{cmc} and $A_{\min}$, but higher $\Gamma_{\max}$, r and pC20 compared to its unsaturated counterpart.

Keywords 10-Undecenoic acid · Undecanoic acid · Amidobetaine chloride · Amidobetaine · Amidoamine oxide · Surface tension · Fluorescence

Introduction

Undecylenic acid or 10-undecenoic acid (10-UDA) is a first- generation derivative of castor oil, produced by high temperature cracking of ricinoleic acid, the major fatty acid present in castor oil [1]. 10-UDA is an interesting renewable raw material readily used in insecticidal, fungicidal and perfumery formulations [1, 2]. It is also used as precursor for the synthesis of antitumor compounds, antibiotics and a host of insect pheromones [3, 4]. Despite its potential, 10-UDA has not been exploited appropriately. In the present research work, an attempt has been made to prepare 10-UDA-based surfactants, namely amidobetaine, amidobetaine chloride and amidoamine oxide.

Amidobetaines contain both cation and anion functions in the head group region and hence are amphoteric in nature. Though the potential market for amphoteric surfactants is little compared to that of cationic or anionic surfactants, it has a significance because of their mildness and nonirritating nature, and consequently they are largely used in cosmetic and personal care products [5]. Theoretical studies on alkyl amidobetaines are quite rare in the literature. However, there are several patents on cleaning formulations wherein alkyl amidobetaines are used. An aqueous composition containing hydrogenated coconut fatty acid mixture-based amidobetaines has been used in the application of oral care [6]. There are some patents involving 10-UDA-based amidobetaines in hair and skin care formulations [7–12]. On the other hand, amine oxide possesses dual charge in the head group, but can’t be claimed as truly amphoteric.

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It is mostly classified as a nonionic surfactant as it remains nonionic in neutral and alkaline medium, although it undergoes protonation at low pH, making the molecule cationic. Amine oxides, more specifically amidoamine oxides, are versatile surfactants, being used in diverse industries, e.g. from personal care to textile industry and from home care to polymer industry. Alkyl amidoamine oxide possesses interesting properties in a mixed surfactant formulation, such as thickening of surfactant solutions, boosting and stabilizing of foam, and also compatibility to skin [13–16]. The study also includes synthesis and evaluation of surface properties of cationic amidobetaines.

The present research work reports the synthesis of three types of 10-UDA-based surfactants, such as amphoteric amidobetaine, cationic amidobetaine chloride and nonionic amidoamine oxide, and evaluates their surface properties. All these surfactants are designed to have an amide moiety in the hydrophobic part in order to increase their biodegradability [17, 18]. Undecanoic acid (UDA), the saturated analog of 10-UDA, has also been taken up for the synthesis of these three types of surfactants. Different surface properties, such as critical micelle concentration (CMC), surface tension at cmc (γ_{cmc}), efficiency of surface adsorption (pC20), surface excess, i.e., the amount of surfactant adsorbed per unit area at the air–water interface ($\Gamma_{\max}$), minimum area per molecule at the air–water interface ($A_{\min}$) and rigidity at the micellar core (r), of these synthesized surfactants were evaluated and compared. The objective was to synthesize not only castor-based surfactants, but also to study the influence of head group and the presence of a terminal double bond on the surface properties of the synthesized surfactants.

Experimental Procedures

Chemicals

10-Undecenoic acid (10-UDA), undecanoic acid (UDA), *N*,*N*-dimethylpropane-1,3-diamine and Na-chloroacetate were purchased from Aldrich (Milwaukee, WI). Chloroacetic acid was purchased from Rankem (Ranbaxy, New Delhi, India). The fluorescence probes *N*-phenyl-1-naphthylamine (NPN) and 1,6-diphenyl-1,3,5-hexatriene (DPH) were purchased from Sigma Aldrich. All other solvents and 30 % H_2O_2 were purchased from SD Fine (Mumbai, India). Double-distilled water was used for making all solutions prior to measurements.

Synthesis

The synthesis of 10-UDA- and UDA-based amidobetaines, amidobetaine chlorides and amidoamine oxides was carried out as per Fig. 1 [19]. The synthesis protocol for only 10-UDA-based surfactants is given below

Synthesis of *N*-[3-(dimethylamino)propyl]undec-10-enamide (**1a**)

10-UDA (**1**, 20 g, 0.108 m) and *N*,*N*-dimethylpropane-1,3-diamine (**3**, 13.6 ml, 0.108 m) were placed in a 250-ml round-bottomed flask. The reaction mixture was stirred at 110 °C for 24 h [19]. The progress of reaction was monitored by thin layer chromatography (TLC). After completion of reaction, the reaction mixture was extracted with ethyl acetate, washed with 5 % NaCl and also with water, dried over anhydrous sodium sulfate, and finally concentrated using rotary evaporator to get the product (22.3 g). The isolated yield of *N*-[3-(dimethylamino) propyl] undec-10-enamide was 76.75 %. The structural confirmation of the compound was confirmed by nuclear magnetic resonance (NMR) (^{1}H and ^{13}C) and electrospray ionization mass spectrometry (ESI-MS).

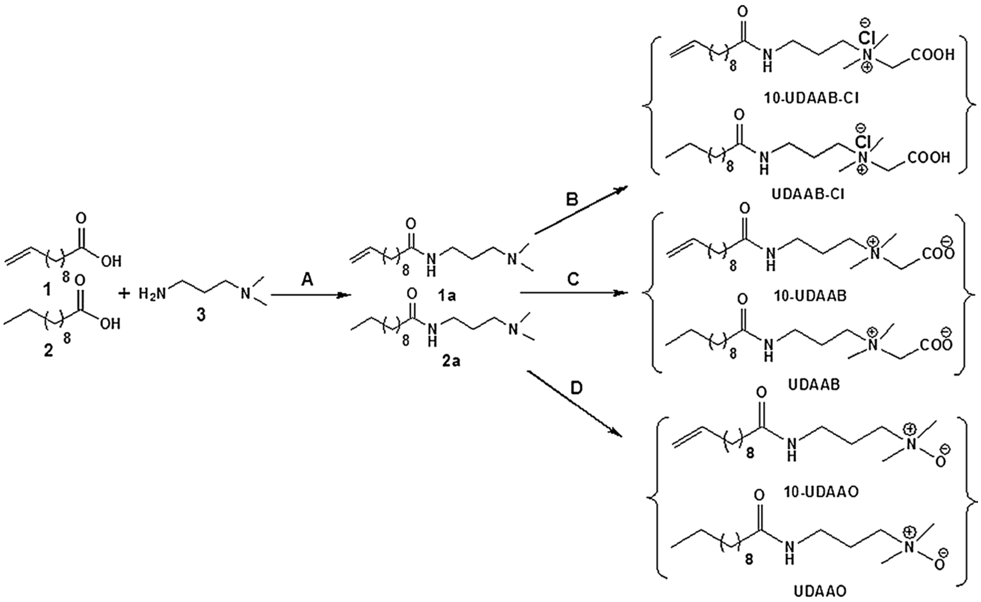
Synthesis of 10-UDA Amidopropyl Betaine Chloride (10-UDAAB-Cl)

Synthesis was carried out by quaternization of *N*-[3-(dimethylamino)propyl]undec-10-enamide (**1a**, 10 g, 0.037 m) with chloroacetic acid (3.5 g, 0.037 m) in toluene. The reaction mixture was refluxed for 12 h at 110 °C. Progress of reaction was monitored by TLC. After completion of reaction, the organic phase was removed using a rotary evaporator. The reaction mixture was placed in dry diethyl ether and kept in an ice bath. A semi solid product separated out and ether was decanted from the top. The semi solid was further solubilized in dry ether, cooled in an ice-bath and the organic solvent was decanted from the top. This operation was repeated thrice to get pure quaternized product (isolated yield, 89.64 %). The structure of the final quaternized product was confirmed using TLC and high-resolution mass spectrometry (HRMS).

Synthesis of 10-UDA Amidopropyl Betaine (10-UDAAB)

Synthesis was carried out by quaternization of *N*-[3-(dimethylamino)propyl]undec-10-enamide (**1a**) with sodium chloroacetate [19]. Initially, **1a** (10 g, 0.037 m) was dissolved in 100 ml of ethanol in a 250-ml round-bottomed flask, followed by the addition of 25 ml of aqueous solution containing sodium chloroacetate (5.18 g, 0.044 m) and sodium bicarbonate (3.73 g, 0.044 m). The contents were refluxed for 8 h and the progress of the reaction was monitored by TLC. After completion of the reaction, ethanol was evaporated and water was removed

Fig. 1 Schematic representation of synthesis of amidobetaine, amidobetaine chloride and amidoamine oxide: *A* 110 °C, 1:1, 24 h; *B* HOOC-CH_2Cl, Toluene, reflux, 12 h; *C* Na^+-OOC-CH_2Cl, $NaHCO_3$, Ethanol, Water, reflux, 8 h; *D* H_2O_2, acetone, 24 h



from the reaction mixture by azeotropic mixture with acetone (100 ml × 4), and the acetone was removed using a rotary evaporator. Finally, the reaction mixture was solubilized in dry diethyl ether and kept in an ice bath. A semi-solid product separated out and ether was decanted from the top. The semi solid was further solubilized in dry ether, cooled in an ice bath, and the organic solvent was decanted from the top. This operation was repeated thrice to get pure quaternized product (isolated yield, 92.76 %). The structure of the final quaternized product was confirmed using TLC and HRMS.

Synthesis of 10-UDA Amidopropyl Amine Oxide (10-UDAAO)

N-oxide derivative was prepared by stirring a cold solution of *N*-[3-(dimethylamino) propyl]undec-10-enamide (**1a**, 5 g, 0.0186 m) in 200 ml of acetone in a 250-ml round-bottomed flask kept in an ice bath. In this stirred solution, 30 % aqueous H_2O_2 (0.061 m) solution was added drop by drop over a period of 30 min [18]. After completion of the addition, the reaction mixture was allowed to come to room temperature and stirred for 24 h. Progress of the reaction was monitored by TLC. After completion of the reaction, the organic phase was removed using a rotary evaporator. The reaction mixture was placed in dry diethyl ether and kept in an ice bath. A semi solid product separated out and ether was decanted from the top. The semi solid was further solubilized in dry ether, cooled in an ice-bath and the organic solvent was decanted from the top. This operation was repeated three times to get pure oxidized product (isolated yield, 72.2 %). The structure of the final product was confirmed using TLC and HRMS.

Analytical Methods

All ^1H and ^{13}C NMR spectra were recorded on 300 and 75 MHz (Varian, Palo Alto, USA) spectrometers, respectively. HRMS data were recorded on a Thermo Scientific Exactive Orbitrap Mass spectrometer (Germany) and are given in mass units (*m*/*z*). ESI–MS spectra were also recorded on a Waters LC–MS mass spectrometer (Palo Alto, USA) in the EI mode and are given in mass units (*m*/*z*). Synthesized compounds were crystallized from ethanol before evaluating their surface properties using surface tension and fluorescence probing techniques. The surface tension was measured using a Krüss K100 tensiometer equipped with a platinum ring having a mean circumference of 6 cm. Steady state fluorescence spectra were recorded in a Varian (model Cary Eclipse) fluorescence spectrometer. Surfactant solutions were prepared in a saturated solution of NPN in double distilled deionized water. The probe solution was excited at 340 nm, and the emission spectra were recorded in the wavelength range 360–550 nm with excitation and emission slits having band-pass equal to 2 nm. Steady state fluorescence anisotropy (*r*) of DPH was measured on a fluorolog-3 spectrofluorometer (Horiba Jobin–Yvon, UK) equipped with a polarization accessory, which uses the L-format instrumental configuration. For anisotropy measurements, surfactant concentrations were kept at five times the CMC value. DPH solution in methanol (5 µl of 10^{-3} M) was added to 10 ml of surfactant solution to make the final concentration of the probe 0.5 µmol. Prior to measurements, the surfactant samples were kept in the dark overnight. The sample was excited at 350 nm and the emission intensity was recorded at 450 nm, using excitation and emission slit widths of 2 and 3 nm, respectively.

Statistical Analysis

All results reported in the present work are the means of three independent measurements (presented as mean ± standard deviation SD) and were analyzed by a paired Student's t test to evaluate the level of statistical significance. Differences were assessed by one-way analysis of variance. A p value <0.05 were considered significant.

Results and Discussion

Synthesis and Characterization

Three types of surfactants, i.e. amidobetaine, amidobetaine chloride and amidoamine oxide, were synthesized starting from the same substrate, *N*-[3-(dimethylamino)propyl] fatty amide as per Fig. 1. Synthesis involved initial amidation of fatty acid with *N*,*N*-dimethyl-1, 3-diaminopropane, and the resulting amide was converted to amidobetaine, amidobetaine chloride and amidoamine oxide, respectively, by quaternization with sodium chloroacetate and chloroacetic acid, and also by oxidation using H_2O_2. The structures of UDA- and 10-UDA-based amides were established by NMR (^{1}H and ^{13}C) and mass spectral data. The structures of quaternized/oxidized final surfactants were confirmed by HRMS.

N-[3-(Dimethylamino)propyl]undec-10-enamide Purity: 98.8 % as per GC; ^{1}H-NMR (300 MHz, $CDCl_3$, δ): 6.85 (s, 1H, NH), 5.7–5.8 (m, 1H, CH=C), 4.9–5.0(dd, 2H, $C{=}CH_2$), 3.35 (t, 2H, $(CH_3)_2N{-}C{-}C{-}CH_2$), 2.38 (t, 2H, $(CH_3)_2N{-}CH_2{-}C{-}C$), 2.25 (s, 6H, $(CH_3)_2N{-}$), 2.1 (t, 2H, $COCH_2$), 2.0 (q, 2H, $CH_2{-}C{=}C$), 1.6 (m, 4H, $CO{-}C{-}CH_2$, $(CH_3)_2N{-}C{-}CH_2{-}C$), 1.3–1.4 (m, 10H, $CO{-}C{-}C{-}(CH_2)_5{-}C{-}C{=}C$). ^{13}C-NMR (75 MHz, $CDCl_3$, δ): 173.25 (C=O), 138.5 (CH=C), 114.3 ($C{=}CH_2$), 58.5 ($(CH_3)_2N{-}CH_2{-}C{-}C$), 46.5 ($(CH_3)_2N{-}$), 26.0–40.0 (CH_2). ESI-Mass: *m/z* 291 $[M^+ + Na]$, 292 $[M^+ + 1 + Na]$, 269 $[M^+ + 1]$, 270 $[M^+ + 2]$.

N-[3-(Dimethylamino)propyl]undecanamide Purity: 99.5 % as per GC; ^{1}H-NMR (300 MHz, $CDCl_3$, δ): 6.9 (s, 1H, NH), 3.39 (t, 2H, $(CH_3)_2N{-}C{-}C{-}CH_2$), 2.4 (t, 2H, $(CH_3)_2N{-}CH_2{-}C{-}C$), 2.26 (s, 6H, $(CH_3)_2N{-}$), 2.15 (t, 2H, $COCH_2$), 1.6–1.7 (m, 4H, $CO{-}C{-}CH_2$, $(CH_3)_2N{-}C{-}CH_2{-}C$), 1.3–1.4 (m, 14H, $CO{-}C{-}C{-}(CH_2)_5{-}C{-}C{=}C$),0.96 (t, 3H,$(CH_2)_7{-}CH_3$). ^{13}C-NMR (75 MHz, $CDCl_3$, δ): 173.25 (C=O), 58.5 ($(CH_3)_2N{-}CH_2{-}C{-}C$), 46.5 ($(CH_3)_2N{-}$), 26.0–40.0 (CH_2), 14.5 (CH_3). ESI-Mass: *m/z* 293 $[M^+ + Na]$, 294 $[M^+ + 1 + Na]$, 271 $[M^+ + 1]$, 272 $[M^+ + 2]$.

The structures of quaternized final surfactants were confirmed by HRMS data: *10-UDAAB-Cl:* HRMS (*m/z*) calculated for $C_{18}H_{35}O_3N_2$, $[M]^+$ 327.2634, found 327.26422; *UDAAB-Cl:* HRMS (*m/z*) calculated for $C_{18}H_{37}O_3N_2$, $[M]^+$ 329.2789, found 329.27987; *10-UDAAB*: HRMS (*m/z*) calculated for $C_{18}H_{34}O_3N_2Na$, $[M + Na]^+$ 349.24487, found 349.24616; *UDAAB*: HRMS (*m/z*) calculated for $C_{18}H_{36}O_3N_2Na$, $[M + Na]^+$ 351.26041, found 351.26181; 10-UDAAO: HRMS (*m/z*) calculated for $C_{16}H_{33}O_2N_2$, $[M]^+$ 285.25281, found 285.25365; UDAAO: HRMS (*m/z*) calculated for $C_{16}H_{35}O_2N_2$, $[M]^+$ 287.26837, found 287.2693.

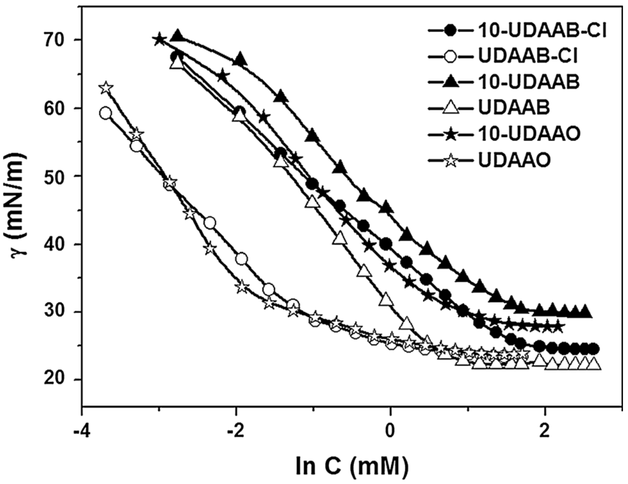


Fig. 2 Surface tension versus concentration of surfactants

The hydrophobic part of these three types of surfactants consists of either 10-UDA or its saturated analog, UDA. 10-UDA is produced by high temperature cracking of ricinoleic acid, the major fatty acid present in castor oil [1]. Thus, the objective of the present work was not only to prepare castor oil-based surfactant, but also to study the influence of the head group and the terminal double bond in the alkyl chain on the surface properties of these three groups of surfactants.

Surface Activity

Aqueous solutions of these surfactants were prepared by dissolving appropriate amounts in Milli-Q water, and surface tension and CMC was measured using a Krüss K100 tensiometer. Variation of surface tension as a function of logarithm of surfactant concentration is shown in Fig. 2. The CMC was determined from this plot of surface tension vs. ln C, by the intersection of two lines representing the linear matchings before and after the break in the trends. Three measurements were taken in total for three different solutions of surfactants, and were averaged.

The surface properties of the synthesized surfactants, obtained by the surface tension method, are given in Table 1. In the surface tension study, ~tenfold increase in CMC was observed due to the introduction of a terminal double bond in the cases of cationic amidobetaine chloride

Table 1 Surface property of UDA- and 10-UDA-based amidobetaine chloride, amidobetaine and amidoamine oxide

Surfactant	CMC_{ST} (mM)	CMC_{FL} (mM)	γ_{cmc} (mN/m)	pC20	Γ_{max}, $\times 10^{12}$ (mol/mm^2)	A_{min} (nm^2/mol)	r
10-UDAAB-Cl	$3.4 \pm 0.21^*$	$2.4 \pm 0.54^*$	25.8 ± 1.76	3.74 ± 0.41	4.12 ± 0.62	0.39 ± 0.04	0.025
UDAAB-Cl	$0.39 \pm 0.03^*$	$0.36 \pm 0.05^*$	24.85 ± 0.12	4.23 ± 0.05	5.52 ± 0.60	0.30 ± 0.03	0.082
10-UDAAB	$4.2 \pm 1.45^{**}$	$4.5 \pm 0.2^*$	29.94 ± 0.62	3.22 ± 0.2	4.37 ± 0.20	0.38 ± 0.02	0.031
UDAAB	$1.9 \pm 0.3^{**}$	$1.75 \pm 0.1^*$	22.44 ± 0.09	3.67 ± 0.04	5.24 ± 0.31	0.32 ± 0.02	0.033
10-UDAAO	$2.2 \pm 0.56^*$	$2.3 \pm 0.8^*$	28.15 ± 0.37	3.49 ± 0.11	4.81 ± 0.17	0.34 ± 0.01	0.016
UDAAO	$0.22 \pm 0.003^*$	$0.46 \pm 0.03^*$	25.68 ± 0.54	4.30 ± 0.05	6.57 ± 0.54	0.25 ± 0.02	0.038

CMC_{ST} critical micelle concentration measured by surface tensiometer, CMC_{FL} critical micelle concentration measured by fluorescence, γ_{cmc} surface tension at CMC, *pC20* efficiency of surface adsorption, Γ_{max} surface excess at the air–water interface, A_{min} minimum area per molecule at the air–water interface, r anisotropy

Differences are drawn between saturated and unsaturated analog, and the significance levels are: * $P < 0.001$, ** $P < 0.01$

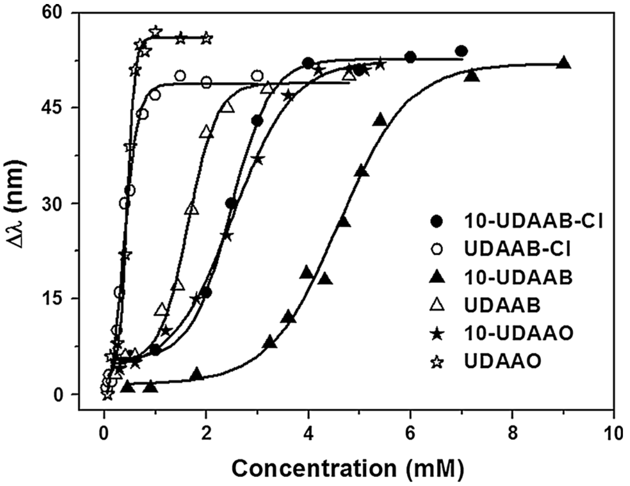


Fig. 3 Shift in emission maxima of NPN versus concentration of surfactants

and nonionic amidoamine oxide. However, the amphoteric amidobetaine showed a two to threefold increase in CMC due to the introduction of a terminal double bond in the hydrophobic chain. Such an effect of unsaturation in the hydrophobic chain of an ionic surfactant on its CMC has been reported earlier in the literature [20]. Authors reported a two to threefold increase in CMC by tensiometric method, due to the introduction of unsaturation, a double or triple bond at the terminal or near the centre of the hydrophobic chain of an ionic surfactant. This is due to the looping back of the moderately polar unsaturated moiety away from the core of the micelle. Results obtained in the present work indicated that the effect of insertion of unsaturation in the hydrophobic moiety of amphoteric amidobetaine type surfactant matched well with the literature report [20]. However, such an effect is much more pronounced in the cases of cationic amidobetaine chloride and nonionic amidoamine oxide. Thus, the effect of unsaturation at the hydrophobic alkyl chain on CMC is dependent on the type of surfactant, especially its head group. The CMC of the studied surfactants with the same tail group but a different head group follow the order: amidobetaine > amidobetaine chloride > amidoamine oxide. In all the studied surfactants, the saturated counterpart showed lower surface tension at CMC (γ_{cmc}) compared to its unsaturated counterpart.

The efficiency and effectiveness of a surfactant can also be assessed by the value of pC_{20}, estimated as the negative logarithm of concentration of surfactant required to reduce the surface tension of water by 20. A higher pC_{20} value indicates higher hydrophobic character of the surfactant, resulting in higher efficiency of reduction of surface tension. As expected, saturated analogs of the three types of surfactants showed significantly larger pC_{20} values, indicating their tendency to be adsorbed at the air–water interface and also to form micelle much greater than their unsaturated counterparts. The surface excess, Γ_{max} (mol/mm^2), i.e., the amounts of surfactant adsorbed per unit area at the air/water interface after complete monolayer formation and minimum surface area occupied by the each surfactant molecule (A_{min}), were calculated using Gibbs adsorption isotherm [21]. In all the studied surfactants, a decrease in Γ_{max} and increase in A_{min} value were observed due to the introduction of unsaturation in the hydrophobic chain.

To further check the hydrophobic domain formation by these surfactants in aqueous medium, fluorescence probe solubilization technique was employed. NPN, the polarity sensitive fluorescence probe, shows an increase in the quantum yield along with a blue shift in the emission maximum in the presence of surfactant micelles, as compared with its emission properties in aqueous solutions. The shift in fluorescence emission maximum ($\Delta\lambda = \lambda_{water} - \lambda_{solution}$) was plotted against the surfactant concentration in Fig. 3. The concentration corresponding to the inflection point could be referred to as the CMC of the surfactants [22, 23]. The CMC values obtained here for all the surfactants are comparable to the values obtained from the surface tension method. Insertion of a terminal double bond significantly increased the CMC value for all the three kind of surfactants.

In order to understand the effect of a terminal double bond on the self assembly property, more precisely the nature of hydrophobic micellar core, steady state fluorescence anisotropy (r) measurements were carried out using DPH as the fluorescence probe. The anisotropy values were measured using the following equation,

$$r = (I_{\mathrm{VV}} - GI_{\mathrm{VH}})/(I_{\mathrm{VV}} + 2GI_{\mathrm{VH}}) \quad (1)$$

where I_{VV} and I_{VH} are the fluorescence intensities polarized parallel and perpendicular to the excitation light, and $G = I_{\mathrm{HV}}/I_{\mathrm{HH}}$ is the instrumental correction factor. The anisotropy values of all six of the surfactants correspond to typical micellar hydrophobic core, and are presented in Table 1 [24]. Introduction of a double bond in the terminal position of cationic amidobetaine chloride and nonionic amidoamine oxides caused a two to threefold decrease in the anisotropy value. In the case of saturated compounds, the molecules are more tightly packed in the micelle compared to their unsaturated counterparts. Surprisingly, no such effect was observed in the case of amphoteric amidobetaines compounds, in which both compounds exhibited a similar packing.

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