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ORIGINAL ARTICLE

Synthesis, Characterization and Evaluation of the Surface Active Properties of Novel Cationic Imidazolium Gemini Surfactants

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Abstract New imidazolium gemini surfactants were synthesized by reaction of epichlorohydrin with long chain fatty alcohols furnishing products 2-(alkoxymethyl)oxirane followed by their subsequent treatment with imidazole resulting in the formation of 1-(1*H*-imidazol-1-yl-3 alkoxy)propane-2-ol which on subsequent treatment with 1,2-dibromoethane and 1,3-dibromopropane resulted in the formation of title gemini surfactants:1,2-bis(1(3-alkoxy-2-hydroxypropyl)-1*H*-imidazol-3-ium)ethane bromide (**7**), 1,3-bis(1(3-alkoxy-2-hydroxypropyl)-1*H*-imidazol-3-ium) propane bromide (**8**), 1,2-bis(1(3-alkoxy-2-hydroxypropyl)-1*H*-imidazol-3-ium)ethane bromide (**9**), 1,3-bis(1(3-alkoxy-2-hydroxypropyl)-1*H*-imidazol-3-ium)propane bromide (**10**), 1,2-bis (1(3-alkoxy-2-hydroxypropyl)-1*H*-imidazol-3-ium)ethane bromide (**11**) and 1,3-bis (1(3-alkoxy-2-hydroxypropyl)-1*H*-imidazol-3-ium)propane bromide (**12**). Their identification was based on IR, ^{1}H-, ^{13}C-NMR, DEPT, COSY and mass spectral studies. Their surface active properties were also evaluated on the basis of surface tension and conductivity measurements.

Keywords Gemini surfactants · Synthesis · Conductivity and surface tension

Introduction

The development of modern surfactants has been in rapid progress over the last two decades. Much research has dealt with how the properties of gemini surfactants change with the structure of molecules [1–3]. Gemini surfactants are a new generation of surfactants composed of two monomeric surfactants molecules chemically bonded together by a spacer at or near their head groups. The birth of gemini surfactants was due to the search for novel surfactants with higher efficiency and effectiveness. They are more surface active and have much lower CMC value than their monomeric counterpart [4, 5]. Because of the vast number of their applications, gemini surfactants continue to gain widespread interest in modern research and for various applications [6, 7].

Consumption of cationic surfactants is around 700,000 tons per year [8]. They have many applications such as fabric softeners, asphalt additives, corrosion inhibitors, biocides, textile auxiliaries and so forth. On adsorbing onto various surfaces they change the surface properties [9–11]. On the other hand, environmental problems may be an important concern during the development of new kinds of surfactants. Owing to increasing legislative pressure and requirements with regard to environmental protection, the design of environmentally benign products to replace conventional surfactant may be the main trend. Different approaches have been taken to overcome this problem. One approach is to introduce an easily cleavable bond into the surfactant structure. The search for novel surfactants with higher efficiency and effectiveness gave birth to the

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concept of gemini surfactants. The gemini surfactants are made up of two monomeric surfactant molecules with their head groups chemically bonded together by a spacer [12–14]. It was found that the surfactant properties of gemini-type surfactants, such as a low critical micelle concentration (CMC) values and lower surface tension, were superior to those of the corresponding single-type surfactants [15]. They are highly efficient in lowering interfacial tension and form micelles at very low critical micellar concentrations [16, 17]. Because of these unique properties, they have great potential to be used as effective emulsifiers, bactericidal agents, dispersants, anti-foaming agents, detergents etc. They are also applicable in the solubilization of dyes and pigments in the textile industry and in gene therapy [18].

Keeping in view the past work and interest centered on cationic gemini surfactants, we have attempted to synthesize a series of gemini surfactants from renewable raw materials such as fatty alcohols and epichlorohydrin. Here, we have chosen the greener approach to make the process environmentally friendly and cost effective, too. The purpose of this work was to prepare and characterize the imidazole-based cationic gemini surfactants from renewable raw materials and to evaluate their surface active properties.

Materials and Measurements

Chemicals: epichlorohydrin and tetrabutyl ammonium iodide were purchased from Sigma-Aldrich Chemical Co. USA. Lauryl alcohol (dodecyl alcohol), myristyl alcohol (tetradecyl alcohol), cetyl alcohol (hexadecyl alcohol) and silica gel for TLC were purchased from S. D. Fine Chemicals Ltd; Mumbai India. 1,2-Dibromo ethane, 1,3-dibromo propane, imidazole and sodium hydroxide were purchased from Merck, Germany.

Instrumentation

IR spectra were recorded as a thin film on a KBr Pellet on a Shimadzu 8400 s FT-IR (Kyoto, Japan) instrument. Mass spectra were recorded on a Waters Q-T of micro mass using ESI as an ion source at the sophisticated analytical instrumentation facility (SAIF), Panjab University, Chandigarh. ^{1}H-, DEPT, COSY and ^{13}C-NMR spectra were recorded on a JEOL AL-300(JEOL, Japan) system as a solution in $CDCl_3$, using tetramethylsilane (TMS) as an internal standard.

Synthesis

Synthesis of 2-(Alkoxymethyl)oxirane (**1–3**)

The preparation of compounds (**1–3**) were prepared by reacting fatty alcohols (hexadecyl, tetradecyl and dodecyl) with epichlorohydrin in 1:1.5 using tetrabutylammonium iodide as a catalyst and sodium hydroxide (1:2) ratio. The resulting products were characterized as 2-alkoxy methyloxirane (**1–3**).The synthesis of the compounds (**1–3**) was reported in parenthesis [19].

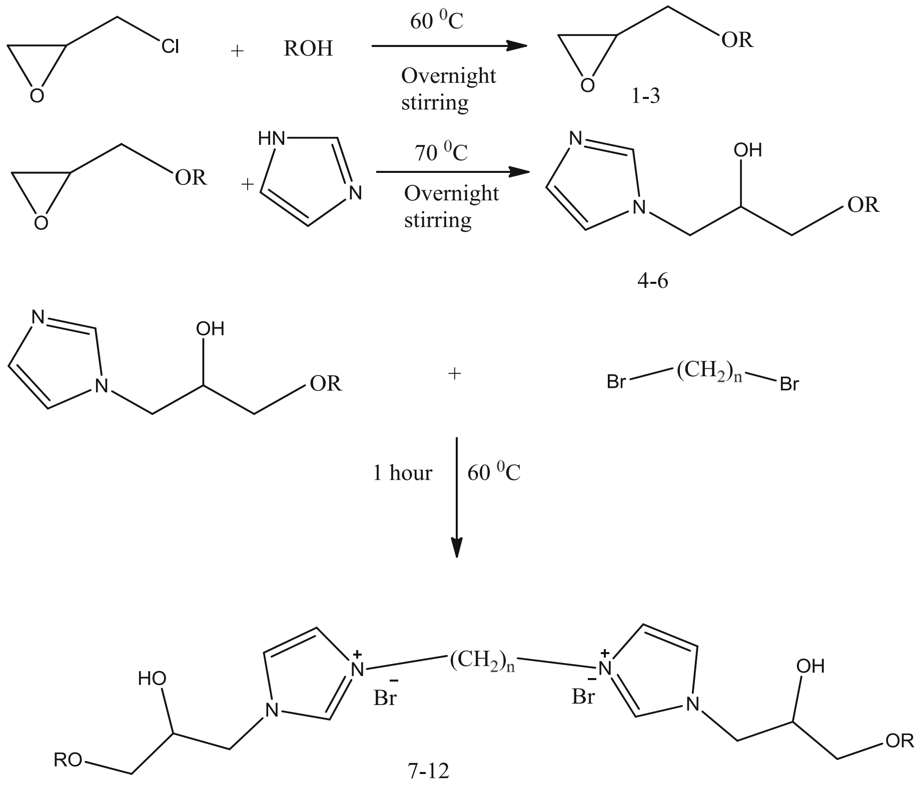
Synthesis of 1-(1*H*-Imidazol-1-yl-3-alkoxy)propane-2-ol (**4–6**)

The resulting products (**1–3**) on further overnight stirring with imidazole in 1:1.2 ratio at 60 °C. The resulting compounds (**4–6**) were obtained as oily liquids [20]. The purification of these resulting compounds was done by washing with hot water. TLC-homogenous products (**4–6**) were characterized by IR, ^{1}H-, ^{13}C-NMR, DEPT, COSY and mass spectral studies as 1-(1*H*-imidazol-1-yl 3-alkoxy)propane-2-ol (**4–6**).

Synthesis of Gemini Surfactants (**7–12**)

The above compounds (**4–6**) were subsequently treated with 1,2-dibromo ethane and 1,3-dibromo propane (2:1) separately at 60 °C for 1 h. In each case the resulting crude product was crystallized with diethylether and then recrystallized with cold acetone to give the pure compounds (**7–12**) which were characterized on the basis of IR, ^{1}H-, ^{13}C-NMR, DEPT, COSY and mass spectral analysis as 1,2-bis (1(3-alkoxy 2-hydroxypropyl)-1*H*-imidazol-3-ium) ethane bromide (**7**),1,3-bis(1(3-alkoxy-2-hydroxypropyl)-1*H*-imidazol-3-ium)propane bromide (**8**), 1,2-bis (1(3-alkoxy 2-hydroxypropyl)-1*H*-imidazol-3-ium)ethane bromide (**9**),1,3-bis (1(3-alkoxy-2-hydroxypropyl)-1*H*-imidazol-3-ium)propane bromide (**10**), 1,2-bis(1(3-alkoxy-2-hydroxypropyl)-1*H*-imidazol-3-ium)ethane bromide (**11**) and 1,3-bis (1(3-alkoxy 2-hydroxypropyl)-1*H*-imidazol-3-ium)propane bromide (**12**).

The schemes of the reactions are shown below.



Where R = CH$_3$(CH$_2$)$_{11}$-; CH$_3$(CH$_2$)$_{13}$-and CH$_3$(CH$_2$)15-.
Where n = 2 and 3.

Measurements

Conductivity Measurements [21]

The critical micelle concentrations (CMC) of these surfactants (**7–12**) were determined by the conductivity method. The conductance as a function of surfactant concentration was measured at 25 °C. Measurements were performed with an Equiptronic Conductometer (Auto temperature conductivity meter model E.Q.661) with stirring to control the temperature. The solutions were thermostated in the cell at 25 °C. For each series of measurements, an exact volume of 25 ml Millipore water (resistivity 18 MΩ) was introduced into the vessel, and the specific conductivity of the water was measured. For the determination of CMC, adequate quantities of concentrated stock surfactant solutions were added in order to change the surfactant concentration from concentrations well below the CMC and repeated to verify our results. The break in the curve of specific conductivity versus surfactant concentration was taken as the CMC (Fig. 1a). The degree of counterion binding (β) was calculated as (1–α), where $\alpha =$ Smicellar/Spremicellar, i.e., Dispersion of micelles in solvent/the existence of surfactant molecules below CMC.

Surface Tension Measurements [21]

Surface tension values were used to calculate CMC using a CSC Du Nouy tensiometer (Central scientific Co., Inc.) equipped with platinum-iridium ring (circumference 5.992 cm) at 25 °C. The tensiometer was calibrated using triple distilled water. For the determination of CMC and surface tension, adequate quantities of a concentrated stock solution were used. The data of this determination is presented in Table 1.

Thermal stability measurements. The thermal stability of the present gemini surfactants was measured with SDT Q600 Thermal Gravimetric Analyzer (TGA), using a nitrogen atmosphere. All samples were in aluminum pans under a nitrogen atmosphere at a heating rate of 10 °C per minute.

Results and Discussion

Spectral Results

The structures of ether based gemini imidazolium surfactants (**7–12**) were established by IR, ^{1}H-, ^{13}C-NMR, DEPT, COSY and mass spectral data. The IR spectra of the

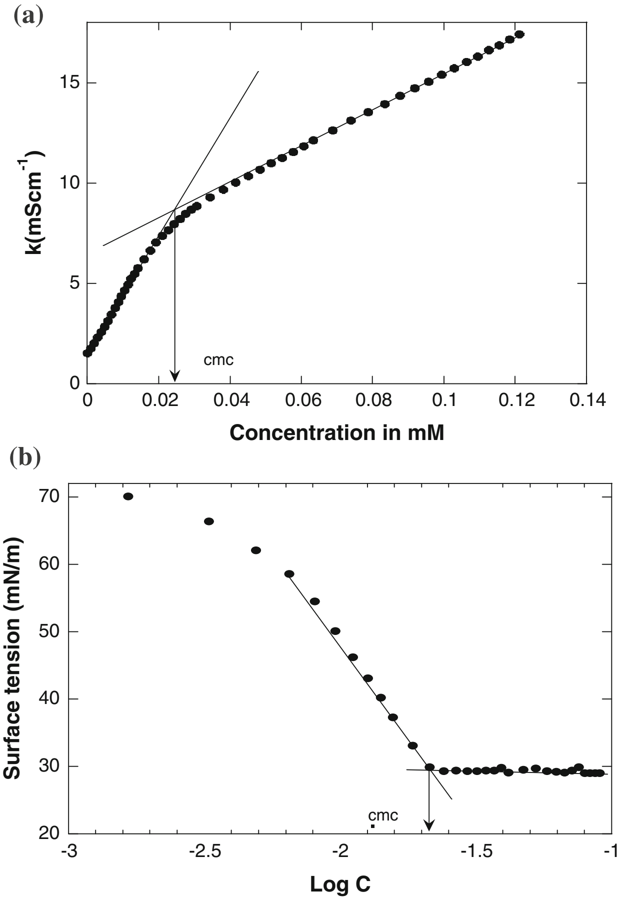


Fig. 1 **a** Specific conductivity versus concentration plot of gemini surfactant **7**, **b** Surface tension versus concentration plot of gemini surfactant **7**

imidazolium gemini surfactants (**7**–**12**) showed the absorption bands in the region at 3,340–3,345 cm^{-1} indicating the presence of hydroxyl groups. The absorptions at 2,950–2,930 cm^{-1} indicate the presence of C–H stretching whereas other absorptions at 3,047–3,030 cm^{-1} indicate the presence of C–H in an aromatic ring. The band at 1,570–1,540 cm^{-1} was very well established, the presence of aromatic C = C of all products (7–12) and the band at 1,126–1,130 cm^{-1} were well established, the presence of aromatic C–N group of all products. The two terminal methyl protons of these gemini surfactants (**7**–**12**) are observed as a distorted triplet at δ 0.80–0.88 in their ^{1}H-NMR spectra. Broad singlets in (**7**–**12**) are observed at δ 1.18–1.87 accountable for methylene protons of chain. Multiplet signals are observed at δ 1.47–1.57 due to protons next to terminal methyl groups. The two middle protons of (**8**, **10** and **12**) ImCH2*CH2*CH2Im are observed as triplets at δ 2.70–2.72. A doublet is observed at δ 3.29–3.45 due to hydroxyl groups. A triplet is observed at δ 3.36–3.47 due to methylene protons attached to ether linkage. Other doublets are observed at δ 3.51–3.58 due to methylene protons next to secondary carbon. Other doublets are observed at δ 3.60–4.22 due to methylene protons attached to nitrogen of imidazole. Other multiplets are observed at δ 4.18–4.35 due to two protons of secondary carbons. Another type of multiplet is observed at δ 4.30–5.10 due to Im$\mathit{CH}_2\mathit{CH}_2$Im or Im$\mathit{CH}_2CH_2$$\mathit{CH}_2$Im protons. The three sets of ring protons of imidazole ring are observed as a singlet at δ 7.27–7.33, δ 7.92–8.17 and δ 9.56–9.67. ^{13}C/DEPT NMR spectra displayed sp^3 carbon of terminal methyl group at δ 13.49–14.06. The carbons next to terminal methyl groups are observed in the range of δ 22.26–22.75. The carbons (OCH$_2$CH$_2$$\mathit{CH}_2$) are observed at δ 25.87–26.28. The chain carbons are observed at δ 29.05–29.32. The methylene carbons i.e. (CH$_2$CH$_2$CH$_3$) are observed at δ 31.35–31.95. The middle carbon of spacer in (**8**, **10** and **12**) ImCH$_2$$\mathit{CH}_2CH_2$Im are observed at δ 31.70–31.86. The methylene carbons of spacer i.e. (ImCH$_2$$\mathit{C}H_2$Im) are observed at δ 46.77–48.92. The α methylene carbons attached to imidazole nitrogen are observed at δ 50.02–53.42. Other signals are observed at δ 67.38–67.67 due to secondary carbons and at δ 71.0–71.90 due to methylene carbon attached to ether linkage. A signal is observed at δ 71.84–72.17 due to (CH$_2$O). Other structure revealing signals are observed at δ 103.90–123.85 due to (NCHCHN) and at δ 123.05–137.21 due to NCHN. On all these accounts the structures of (**7**–**12**) are deduced to be 1,2-bis(1(3-alkoxy-2-hydroxypropyl)-1*H*-imidazol-3-ium)

Table 1 Surface, thermodynamic and thermal properties of the synthesized surfactants (7–12)

S. no	CMC (mM)	α (%)	β (%)	CMC (mM)	γ (mN/m)	Π_{CMC} (mN/m)	C_{20} (M)	CMC/ c_{20}	$10^6 T_{max}$ (Mol/m^2)	A_{min} (nm^2)	ΔG_{mic} KJ/mol	ΔG_{ads} (KJ/mol)	T_{ONSET} (°C)	T_{START} (°C)
7	0.021	48	52	0.022	30.04	42.36	0.010	2.21	4.53	0.036	−36.49	−45.84	279.5	235.8
8	0.020	49	51	0.020	36.08	36.32	0.008	2.54	3.48	0.047	−36.75	−47.15	285.3	248.4
9	0.232	32	68	0.234	43.51	28.89	0.035	2.01	3.22	0.051	−20.87	−29.84	270.6	230.0
10	0.071	34	66	0.073	44.51	27.81	0.117	2.08	2.99	0.055	−23.81	−33.13	281.9	240.7
11	0.421	33	67	0.429	48.07	24.33	0.299	1.43	2.71	0.061	−20.99	−29.96	253.3	221.6
12	0.411	38	62	0.415	50.06	21.84	0.350	1.48	2.01	0.082	−22.90	−33.76	270.4	232.1

ethane bromide (**7**), 1,3-bis(1(3-alkoxy-2-hydroxy propyl)-1*H*-imidazol-3-ium)propane bromide (**8**), 1,2-bis (1(3-alkoxy-2-hydroxypropyl)-1*H*-imidazol-3-ium)ethane bromide (**9**), 1,3-bis(1(3-alkoxy-2-hydroxypropyl)-1*H*-imidazol-3-ium) propane bromide (**10**), 1,2-bis(1(3-alkoxy-2-hydroxypropyl)-1*H*-imidazol-3-ium)ethane bromide (**11**) and 1,3-bis (1(3-alkoxy-2-hydroxypropyl)-1*H*-imidazol-3-ium)propane bromide (**12**). The structures of these gemini surfactants (7–12) are further consolidated by ESI–MS (positive ion) mass spectral data. Important peaks in these spectra are found at *m*/*z* 761.2, 762.2, 773.8, 774.7, 704.5, 705.7, 717.7, 718.7, 648.5, 649.5, 662.7, 663.7. These ion peaks account for the loss of proton and two bromide ions from the molecule leading to the formation of positively charged parent ion $\{M\text{- }2Br)\text{-}1)\}^+$ and the direct loss of two bromide ions from the molecule leading to formation of $(M\text{-}2Br)^+$ positively charged ions. All the spectral results of the synthesized compounds are provided in the supplementary material file.

Surface Active Properties of Gemini Surfactants (**7–12**)

Critical Micelle Concentration

Geminis have lower CMC values than the corresponding single tail surfactants. Only a few reports are available regarding the syntheses and CMC values of gemini imidazolium surfactants [21]. The CMC and degree of counter ion binding of these new imidazolium amphiphiles were determined by the conductivity method. These new gemini imidazolium amphiphiles have low CMC values. The values of CMC and degrees of counter ion binding are given in Table 1. The graph of the concentration versus conductivity has been plotted Fig. 1a. It was found that the imidazolium gemini surfactants having short spacer lengths and short chain lengths have lower CMC values than gemini surfactants having long spacer lengths and long chain lengths [21].

The Degree of Counterion Binding (β)

The ratio of the slopes of the conductivity versus concentration curve above and below the CMC gives the degree of counterion dissociation α (i.e., α = Smicellar/Spremicellar) and (1- α) gives the degree of counterion binding, β. It is an important parameter because it shows the counterions that are contained in the Stern layer counterbalance the electrostatic force that opposes micelle formation. Quagliotto et al. [21] determined the β value for a series of gemini bis-pyridinium bromides having different spacers where they showed that a different spacer is responsible for different β value. The β value signifies the ability of counter ion to bind micelles. It was found that the β value decreases with increasing chain length and decrease with increasing spacer length (Table 1) [22].

Surface Tension Measurements

The CMC of new imidazolium gemini surfactants were calculated by using surface tension measurements Fig. 1b. The increase in the surface tension for the series of imidazolium gemini surfactants with increasing hydrophobic alkyl chain lengths can be explained on the basis of the CMC/*C*20 ratio observed for these amphiphiles. The affinity of a particular surfactant to reduce surface tension of solvent depends upon CMC/*C*20 ratio, greater the observed value greater is the tendency of the amphiphile to reduce surface tension of the system [23]. Thus, imidazolium surfactants **7** and **8** have maximum ability while amphiphile **11** and **12** have minimum ability to reduce surface tension of aqueous system in the series of amphiphile being reported. Two important parameters of gemini surfactants, i.e. the effectiveness of surface tension reduction (ΠCMC) and the adsorption efficiency (p*C*20) were obtained from the surface tension plots. The former parameter, ΠCMC is the surface pressure at the CMC and is defined as:

$$\Pi\text{CMC} = \gamma_0 - \gamma_{\text{CMC}} \tag{1}$$

Where γ_0 is the surface tension of pure solvent and γ_{CMC} is the measured surface tension at CMC. The maximum reduction in surface tension caused by the dissolution of amphiphilic molecules has been indicated by ΠCMC and as a result ΠCMC becomes a measure for the effectiveness of the amphiphile to lower the surface tension of the water [23]. Imidazolium gemini surfactants synthesized in present work (**7**, **8**, **9**,**10**, **11** and **12**) have greater ability to reduce surface tension of the aqueous system. The adsorption efficiency (p*C*20) is determined by using the following equation [23]:

$$\text{pC20} = -\log\text{C20} \tag{2}$$

In this equation *C* is the molar concentration of surfactant and *C*20 stands for the concentration required to reduce the surface tension of the pure solvent by 20 mN m^{-1} [24]. Thus *C*20 becomes a measure of adsorption efficiency of amphiphilic molecules at the air–water interface. The results from Table 1 indicate that the compounds **11** and **12** have higher adsorption efficiency among six long chain gemini surfactants [24]. The values of these two parameters obtained for the six gemini surfactants are listed in Table 1 along with their CMC and γ values. The maximum surface excess concentration (Γ_{max}) was estimated by applying the Gibbs adsorption isotherm [24] to the surface tension data:

$$\Gamma_{max} = -1/2nRT\ (\partial\gamma/\partial \ln C)T \qquad (3)$$

where, R is the gas constant (8.314 J $mol^{-}K^{-}$), *T* is the absolute temperature, and *C* is the surfactant concentration. The value of *n* is taken as 2 as there is one counter ion associated with each cationic head group. The minimum area occupied by a single amphiphilic molecule at the air–water interface (A_{min}) was also obtained by applying the Gibbs adsorption isotherm to the surface tension data:

$$A_{min} = 1/\ N_A.\Gamma_{max}\left(\times 10^{23}\right) \qquad (4)$$

where N_A is the Avogadro constant. All imidazolium gemini surfactants have lower A_{min} values (Table 1). The lowest A_{min} values of imidazolium gemini surfactants (**7** and **8**) can be attributed to tighter packing of the longer chains at the interface [24]. A part from positively charged imidazolium cation in all these imidazolium gemini surfactants contains free hydroxyl group. The presence of hydroxyl group in addition to positively charged center plays important role in their aggregation behavior. The energy required for the closer packing of hydrocarbon alkyl chain length at interface may come from the energy released upon hydrogen bond formation between the hydroxyl group present close to positively charged imidazolium center and water molecule [24]. The Gibbs free energy of the micellization (ΔG_{0mic}) was calculated by use of following equation.

$$\Delta G_{0mic} = (2 - \alpha)RT\ln X_{CMC} \qquad (5)$$

where X_{CMC} is the mole fraction at the CMC and α is the extent of counter ion dissociation. The micellization free energy has a negative sign because thermodynamically stable micelles are formed spontaneously. The results from Table 1 indicate that the driving force for micellization becomes large as ΔG_{0mic} becomes more negative. The standard Gibbs free energy of adsorption (ΔG_{0ads}) was obtained from the following relationship [25].

$$\Delta G_{0ads} = \Delta G_{0mic} - \Pi CMC/\Gamma_{max} \qquad (6)$$

Here, πCMC denotes the surface pressure at the CMC ($\pi CMC = \gamma_0\text{-}\gamma_{CMC}$, where γ_0 and γ_{CMC} are the surface tensions of water and the surfactant solution at the CMC, respectively). The free energy of adsorption (ΔG_{0ads}) represents the free energy of transfer of 1 mol of surfactant in solution to the surface, and the free energy of micellization (ΔG_{0mic}) represents the work done to transfer the surfactant molecules from the monomeric form at the surface to the micellar phase. The standard free energy of micellization (ΔG_{0mic}) and adsorption (ΔG_{0ads}) is always negative, indicating tendencies to form micelles in solution and to adsorb at the air/water interface [25]. If the value of (ΔG_{0ads}) is more negative and greater than the difference between (ΔG_{0ads}) and (ΔG_{0mic}), then the adsorption of surfactant molecules at the interface becomes more favorable because of the greater freedom of motion of hydrocarbon chains at the planar air/aqueous solution interface than in the interior of the micelle. However, if the energy difference is small, then less work has to be done to transfer surfactant molecules from the monomeric form at the surface to the micellar phase. When the difference in the free energies is small, the surfactant undergoes aggregation more readily than when the difference in the free energies is large. This is evident from the results obtained by Yeshimua et al. [26]. The (ΔG_{0mic}) and (ΔG_{0ads}) values of gemini imidazolium surfactants are summarized in Table 1. The difference in the free energy gap is small for gemini imidazolium surfactants, therefore these surfactants have a greater tendency to aggregate in solution.

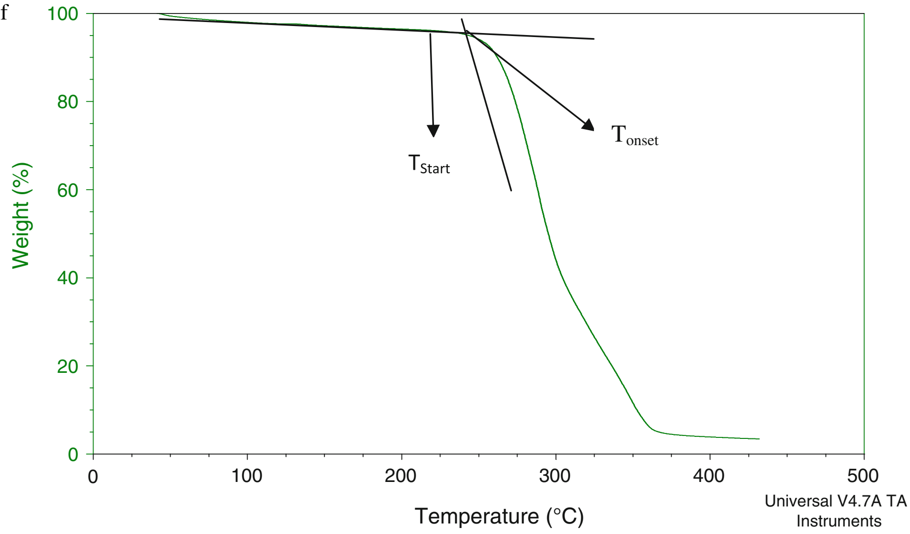


Fig. 2 The thermal decomposition curve of surfactant (**7**) determined by TGA, indicating the Start (T_{START}) and onset (T_{ONSET}) temperature

The graph of the surface tension versus concentration is shown for gemini surfactants (**7–12**). A clear break is observed Fig. 1b. It is observed from the graphs that imidazolium gemini surfactants having short spacer length and short chain length have low CMC values as compared to the imidazolium gemini surfactants having long spacer length and long chain length. The CMC values are reported in Table 1 for all the gemini surfactants. The values for both the conductivity methods and surface tension method correspond well with each other.

Thermal Stability Measurements

Thermal stability measurement shows that these long chain gemini surfactants are stable up to 320 °C. Figure 2 shows a characteristic curve for the decomposition of the gemini surfactants as measured by thermal gravimetric analyzer. The onset temperature (T_{ONSET}) is the intersection of the baseline weight, either from the beginning of the experiment and the tangent of the weight versus temperature curve as decomposition occurs [27]. The start temperature (T_{START}) is the temperature at which the decomposition of the sample begins. The example of the onset and start temperatures is shown in figure. The onset and start temperatures for present imidazolium gemini surfactants are listed in Table 1. Thermal stability measurements designated that these surfactants have better thermal stability. Thermal stability of these gemini surfactants increases as chain length and spacer length increases.

Conclusion

In the present study we have described a new protocol for the synthesis of novel imidazolium gemini surfactants through an environmental friendly process. All the gemini cationic surfactants (**7–12**) were produced in excellent yields and these surfactants were examined and found to have good surface active properties. These imidazolium amphiphiles were investigated for their self-aggregation properties by surface tension and conductivity methods. These imidazolium amphiphiles have lower CMC values. The results show that gemini imidazolium surfactants with longer hydrophobic chains as well as longer chain lengths have a lower CMC value. Further results showed that these gemini surfactants have good thermal properties. The thermal stability of these gemini surfactants was investigated by TGA analysis. The thermal stability of these amphiphiles increases with increasing size of the hydrophobic alkyl chain length and increasing spacer length. Owing to their ease of synthesis, superior self-aggregation and thermal properties, these gemini surfactants may find use in several industrial applications. In addition, these imidazolium cationic gemini surfactants may show good antimicrobial properties, DNA binding capability if tested properly.

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