

Research Paper :

A comparative study of dissociation/association facets of alkali and alkaline earth metal myristates

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ABSTRACT

The present investigation deals with the relative facets of alkali (Li, Na, K) and alkaline earth metal (Mg, Ca, Sr, Br) Myristates at different temperatures (30^o–50^oC). Thermodynamic parameters (change in enthalpy/ free energy /entropy) for both dissociation and association processes have been used to look into the solution behaviour of these surfactants in 50% methanol +50% chloroform (V/V). Besides thermodynamic consideration, other parameters such as degree of dissociation, α and dissociation constant, K_D etc are used to delve into the dissociation aspects of these systems. The data for critical micelle concentration, cmc are also reported and explained.

Key words : Specific conductivity, Alkali and alkaline earth metal myristates, Dissociation/association behaviour, Thermodynamic approach, Critical micelle concentration (cmc).

The determination of critical Micelle concentration, cmc serves to be an important consideration while deciding the quality of a product. Researchers and academicians alike (Aicart *et al.*, 2006; Atwood and Flovenie, 1983; Avakawa and Brain, 1980; Barry and Russel, 1972 and Bufe and wolff, 2006) have already shown a keen interest for various surface active agents, also termed as Surfactants. They have been enthusiastic about their various facets *viz.* the physicochemical characterization, shape/size determination of micellar aggregates, W.J. Leigh and Co-workers (Bunajdad and Eastore, 2004; cook *et al.*, 2001 and hartl *et al.*, 2007) have, of late, shown how significant organometallics are to the wide domin of surfactants. Several national/international publications (Jacobs *et al.*, 2006; Jaliceour and Philip, 1975 ; Kim *et al.*; Kumar, 1994 and Leigh and Li, 2002) have appeared in literature just to prove the merit of various physical properties of surfactants. Techniques such as viscometry and electrical conductivity have proved handy to study neutral polymer micelle interactions (Lelong *et al.*, 1951). Bumajdad and Eastoe (Malik *et al.*, 1984) employed conductivity to study water in oil microemulsions stabilized by mixed surfactants. Tania *et al.* (Mc. Brain, 1939) have resorted to spectroscopy and conductometry to probe interaction between water soluble poly{1,4-Pheny- lene-[9,9-bis(4-Phenaxy butyl-sulfonate)]flurenl-2, 7-diy1} Copolymer and ionic surfactants. Aicart and Co-workrs (Mehta *et al.*, 1979) examined electro- chemical, microscopic and spectroscopic characterization of vesicles and prevesicle

nanostructures of mixed cationic surfactant systems.

Researchers (Mehrota *et al.*, 1970) of late have undertaken a study on electrically conductive beacterial cellulose by incorporation of carbon nanotubes. Kim and co-workers (Modaressi *et al.*, 2007) have, however, carried out a similar looking study using dielectrophoresis of surface conductance modulated single-walled carbon nanotubes with cationic surfactants. Hartl *et al.* (Niisson *et al.*, 2006) have investigated into ion sensitivity of surface conductive single crystalline diamond. Jacobs *et al.* (Niisson *et al.*, 2006) have dealt with aspects on dynamics of alkyl ammonium intercalants within organically modified montmorillohite: Dielectrical relaxation and ionic conductivity. Rajamani *et al.* (Robins *et al.*, 2003) have performed a study on carbon nanotube based transparent conductive thin films. NMR diffusometry and electric conductometric techniques have been employed to study interactions between gemine surfactants, 12-s-12, and beta cyclodextrin (Sarah *et al.*, 2006). Bufe and wolf (Sibel and Aoman, 2007) have recently undertaken a study on switching electrical conductivity in an AOT-isoctane- water microemulsion through photodimerization of solubilized N-methyl-2-quinoline. Conductometric measurements have been found extremely handy to look into CTAB aggregation in aqueous solutions of ammonium based ionic liquids (Sharma *et al.*, 1986). Conductometric method (Shun-Cheng *et al.*, 2004) has also been a worthy tool to investigate interaction between some anionic dyes and cationic surfactants. Sarah *et al.* (Tania *et al.*, 2005) have carried out work on

conductometry and fluorometry on using mixed micellar systems of cationic surfactants in aqueous media.

The present study however deals with a comparative thorough probe into the dissociation/ association behaviour of alkali (Li, Na, K) and alkaline earth metal (Ba, Sr, Ca, Mg) Myristates in 50% methanol +50% chloroform (v/v) at different temperatures (30^o–50^oC).

MATERIALS AND METHODS

GR grade magnesium, calcium, strontium and barium carbonates were used. Myristic acid obtained from Sigma Chemical Co. U.S.A., stated high purity was used as received. The conductivity water used for experimental work was prepared by redistilling good quality distilled water over alkaline KMnO₄.

Myristates of lithium, sodium and potassium were first prepared by reaction warm aqueous solutions of alkali metal carbonates containing phenolphthalein as indicator with fused myristic acid. Stoichiometric amounts of both alkali metal carbonates and myristic acid were employed. The completion of the reaction is thus indicated by the decolourisation of the pink coloured solutions. The dried and recrystallised pure compounds were found to decompose between 190^o–205^oC. Now, alkaline earth metal myristates were prepared by metathesis method. Warm aqueous solutions of alkaline earth metal carbonates, containing phenolphthalein as indicator, were individually treated with the warm aqueous solutions of potassium myristate employing their stoichiometric amounts. The completion of the reaction is indicated as the pink colour gradually vanishes. The compounds formed were separated from their mother liquor by filtration, washed with benzene and dried. The white coloured compounds were recrystallised from 50% methanol +50% chloroform solvent-mixture and oven dried to obtain high quality products. The compounds are found to decompose

between 190^o–205^oC.

A digital conductivity meter (Elico Pvt. Ltd., Hyderabad, India, Type 032, No. 1455) and a dipping type conductivity cell with platinised electrodes were used for measuring the conductance of the solutions of alkali and alkaline earth metal myristates in 50% methanol+50% chloroform (v/v). All the measurements were made at a constant temperature in a thermostat (+0.01^oC).

RESULTS AND DISCUSSION

A representative plot (Fig. 1) indicates an increase in specific conductance, $K(\text{m scm}^{-1})$ with increasing concentration, $c(\text{mol dm}^{-3})$ of alkali and alkaline earth metal myristates in 50% methanol +50% chloroform at 40^oC. The measurements are however made at different temperatures 30^o–50^oC (Table 1, 2 and 3) and the trend is found to be the same for all temperatures. The specific conductance (Tables 1, 2 and 3) are also found to increase with increasing temperatures. It is interesting to note that the κ - C plot (Fig.1) exhibit an intersection of two straight lines indicating onset of micellisation *i.e.*, the compounds form charged micellar aggregates. It is also significant to note that cmc_s (Table 4) as deduced from κ - C plots decrease with increasing temperatures, and vary with different alkali metals as Lithium myristate > Sodium myristate > Potassium myristate. The variation of cmc_s with alkaline earth metal soaps is observed as magnesium myristate > calcium myristate > strontium myristate > barium myristate.

Equivalent conductivity, λ ($\text{mho cm}^2/\text{g-eq}$) as computed from specific conductance ($\lambda=10^3k/c$) is found to decrease with increasing surfactant concentration. The concave nature of λ vs. $c^{1/2}$ plots for alkaline earth metal myristates indicates weak electrolytic nature of these compounds, where λ vs. $c^{1/2}$ plots for alkali metal myristates are found to be linear indicating their high

Table 1: Specific conductance of Li, Na and K myristates at different temperatures (30^o–50^oC)

Conc. C×10 ⁴ (mol dm ⁻³)	Specific conductance, K×10 ³ (mscm ⁻¹) of Li-myristate					Specific conductance, K×10 ³ (mscm ⁻¹) of Na-myristate					Specific conductance, K×10 ³ (mscm ⁻¹) of K-myristate				
	30 ^o C	35 ^o C	40 ^o C	45 ^o C	50 ^o C	30 ^o C	35 ^o C	40 ^o C	45 ^o C	50 ^o C	30 ^o C	35 ^o C	40 ^o C	45 ^o C	50 ^o C
2.0	12.0	15.0	17.0	20.0	24.0	16.0	18.0	20.0	23.0	26.0	20.0	23.0	25.0	28.0	31.0
4.0	17.0	20.0	23.0	27.0	31.0	23.0	26.0	28.0	31.0	34.0	30.0	33.0	36.0	40.0	44.0
6.0	23.0	26.0	29.0	34.0	38.0	30.0	34.0	37.0	40.0	44.0	40.0	43.0	46.0	56.0	54.0
8.0	27.0	32.0	35.0	41.0	46.0	36.0	41.0	45.0	47.0	51.0	49.0	52.0	56.0	61.0	63.0
10.0	32.0	37.0	41.0	48.0	52.0	43.0	48.0	52.0	55.0	59.0	58.0	62.0	65.0	70.0	74.0
12.0	37.0	43.0	46.0	53.0	58.0	46.0	53.0	57.0	62.0	66.0	66.0	71.0	74.0	80.0	84.0
14.0	41.0	48.0	52.0	59.0	64.0	51.0	58.0	63.0	69.0	74.0	73.0	79.0	83.0	90.0	94.0
16.0	46.0	53.0	57.0	65.0	71.0	55.0	63.0	69.0	76.0	81.0	80.0	86.0	92.0	98.0	106.0
18.0	49.0	58.0	63.0	71.0	77.0	60.0	68.0	75.0	83.0	89.0	88.0	96.0	100.0	109.0	114.0
20.0	54.0	63.0	68.0	76.0	83.0	65.0	72.0	80.0	90.0	96.0	96.0	105.0	110.0	118.0	124.0

Table 2 : Specific conductance of Mg-myristate and Ca-myristate at different temperatures (30⁰-50⁰C)

Conc. C×10 ⁴ (mol dm ⁻³)	Specific conductance, K×10 ³ (mscm ⁻¹) of Mg- myristate					Specific conductance, K×10 ³ (mscm ⁻¹) of Ca -myristate				
	30 ⁰ C	35 ⁰ C	40 ⁰ C	45 ⁰ C	50 ⁰ C	30 ⁰ C	35 ⁰ C	40 ⁰ C	45 ⁰ C	50 ⁰ C
	2.0	19.0	22.0	25.0	29.0	32.0	12.0	14.0	16.0	18.0
4.0	25.0	30.0	33.0	36.0	41.0	17.0	20.0	22.0	24.0	26.0
6.0	31.0	36.0	40.0	44.0	49.0	22.0	25.0	27.0	30.0	33.0
8.0	37.0	43.0	47.0	50.0	58.0	27.0	30.0	33.0	36.0	40.0
10.0	43.0	49.0	55.0	58.0	65.0	32.0	35.0	40.0	42.0	46.0
12.0	49.0	55.0	59.0	64.0	70.0	36.0	39.0	43.0	47.0	50.0
14.0	53.0	60.0	65.0	69.0	77.0	39.0	44.0	49.0	51.0	55.0
16.0	57.0	64.0	70.0	75.0	83.0	42.0	47.0	53.0	57.0	60.0
18.0	60.0	67.0	75.0	80.0	90.0	44.0	50.0	56.0	60.0	64.0
20.0	63.0	72.0	79.0	86.0	96.0	48.0	54.0	60.0	64.0	68.0

Table 3: Specific conductance of Sr-myristate and Ba-myristate at different temperatures (30⁰-50⁰C)

Conc. C×10 ⁴ (mol dm ⁻³)	Specific conductance, K×10 ³ (mscm ⁻¹) of Sr- Myristate					Specific conductance, K×10 ³ (mscm ⁻¹) of Ba- Myristate				
	30 ⁰ C	35 ⁰ C	40 ⁰ C	45 ⁰ C	50 ⁰ C	30 ⁰ C	35 ⁰ C	40 ⁰ C	45 ⁰ C	50 ⁰ C
	2.0	14.0	16.0	17.0	19.0	21.0	14.0	15.0	17.0	19.0
4.0	20.0	22.0	25.0	27.0	29.0	20.0	24.0	26.0	28.0	32.0
6.0	26.0	29.0	32.0	35.0	39.0	28.0	31.0	35.0	38.0	40.0
8.0	32.0	36.0	40.0	42.0	45.0	34.0	38.0	41.0	44.0	48.0
10.0	38.0	42.0	56.0	47.0	52.0	39.0	44.0	48.0	52.0	55.0
12.0	42.0	47.0	50.0	54.0	58.0	44.0	50.0	55.0	60.0	63.0
14.0	45.0	50.0	55.0	61.0	65.0	50.0	55.0	62.0	68.0	70.0
16.0	49.0	53.0	60.0	65.0	72.0	56.0	61.0	69.0	75.0	79.0
18.0	52.0	58.0	66.0	72.0	79.0	62.0	67.0	75.0	82.0	85.0
20.0	56.0	62.0	70.0	77.0	84.0	68.0	73.0	82.0	89.0	93.0

Table 4 : C.M.C data (md cm⁻³) for alkali and alkaline earth metal myristates in 50% methanol + 50% Chlorform at different temperatures (30⁰-50⁰C)

Compounds	C.M.C×10 ⁴				
	30 ⁰ C	35 ⁰ C	40 ⁰ C	45 ⁰ C	50 ⁰ C
Lithium Myristate	12.0	11.0	10.2	9.0	8.0
Sodium Myristate	10.0	9.4	8.4	7.1	6.3
Potassium Myristate	8.9	8.0	7.2	5.8	5.0
Magnesium Myristate	12.0	11.0	10.0	8.9	8.0
Calcium Myristate	11.0	10.0	8.0	7.0	6.0
Strontium Myristate	10.0	9.2	7.9	7.1	6.1
Barium Myristate	8.0	7.0	6.0	5.2	4.0

ionization, a fact made explicit by the perusal of a value recorded in Table 5. The applicability of log λ -log c plots (also intersecting at c.m.c.) enables us to use the expression, $\log \lambda = A+B \log C$. The significance of the constants A and B lies in the fact that they furnish information on micellization. It is found that the values

for constants, A, (Pre micellar region) and A₂ (post micellar region) increase with increasing temperatures, and A₂ > A₁. However, constant B₁ (pre-micellar region) and B₂ (Post micellar region) are not found to vary appreciably with increasing temperatures, but B₂ > B₁. The data for A₁, B₁, (pre-micellar region) and A₂, B₂ (post

Compound	30°C				35°C				50°C			
	λ_{∞}	α	K_D	λ_{∞}	α	K_D	λ_{∞}	α	K_D	λ_{∞}	α	K_D
Calcium Myristate	65	0.78 0.92	1.39×10^{-3}	82	0.75 0.91	9.57×10^{-4}	93	0.77 0.91	8.68×10^{-4}	110	0.73 0.91	5.65×10^{-4}
Sodium Myristate	87	0.76 0.91	9.20×10^{-4}	99	0.77 0.90	7.83×10^{-4}	101	0.77 0.90	7.09×10^{-4}	121	0.73 0.90	6.77×10^{-4}
Potassium Myristate	109	0.76 0.92	8.30×10^{-4}	121	0.73 0.91	6.75×10^{-4}	139	0.73 0.90	6.07×10^{-4}	155	0.72 0.90	5.07×10^{-4}
Magnesium Myristate	120	0.71 0.61	3.2×10^{-3}	125	0.32 0.62	2.8×10^{-3}	185	0.30 0.62	2.2×10^{-3}	259.26	0.22 0.66	1.20×10^{-3}
Calcium Myristate	90	0.33 0.67	3.8×10^{-3}	117	0.31 0.61	2.6×10^{-3}	130	0.29 0.61	1.6×10^{-3}	151	0.22 0.56	1.59×10^{-3}
Sodium Myristate	167.06	0.75 0.30	7.7×10^{-3}	227.73	0.70 0.77	3.60×10^{-3}	665.35	0.56 0.73	1.75×10^{-3}	700	0.71 0.32	9.50×10^{-4}
Potassium Myristate	362.90	0.71 0.20	1.7×10^{-3}	192.0	0.08 0.16	1.76×10^{-3}	660.70	0.07 0.10	9.33×10^{-4}	2687	0.02 0.07	1.30×10^{-4}

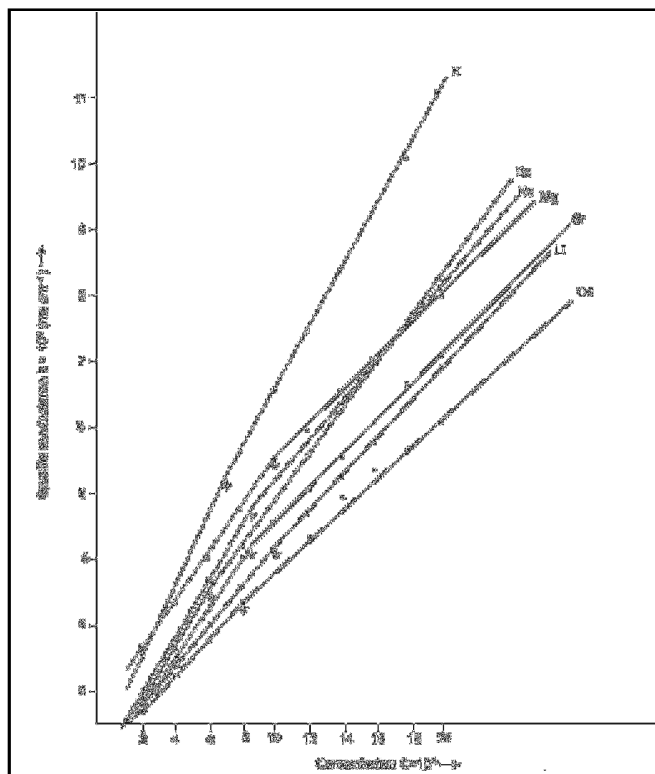


Fig. 1 : The plots of specific conductance, k (ms cm^{-1}) vs. conc. (mol dm^{-3}) for alkali and alkaline earth metal myristates in 50% methanol + 50% chloroform solvent-mixture at 40°C

micellar region) suggest that these compounds do not show any micellar aggregates below the c.m.c, while micellar size increases with increasing surfactant concentration in post-micellar region.

To evaluate dissociation constant, K_D (Pre micellar region) for alkaline earth metal (Mg, Ca, Sr, Ba) myristates in 50% methanol+50% chloroform solutions, the following expression may be employed.

$$K_D = \frac{4C^2 a^3}{1-a}$$

$$\text{or } K_D = \frac{Ca\alpha(2C\alpha^2)}{C(1-\alpha)} \quad (i)$$

For weak electrolytes, the ionic concentrations are too low, and therefore interionic effects would be negligible *i.e.* suggesting that these solutions do not deviate appreciably from their ideal behavior. Therefore, substituting $\alpha = \lambda/\lambda_{\infty}$ in expression (i), one gets,

$$C^2 \lambda^2 = \frac{K\lambda_{\infty}^3}{4\lambda} - \frac{K\lambda_{\infty}^2}{4} \quad (ii)$$

The plots of $C^2 \lambda^2 v_s 1/\lambda$ are found to be linear in pre-micellar region indicating the validity of the expression (ii) for these solutions. Equivalent conductance at infinite

dilution (λ_∞) and dissociation constant (K_D) have been evaluated from the slope and intercept ($\lambda_\infty = -\text{slope}/\text{intercept}$), (Table 5) of the linear plots. The equivalent conductance at infinite dilution, λ_∞ thus evaluated is found to increase with increasing temperatures as ionic mobility increases even as addition of surfactant increases the viscosity of the solvent. The data for degree of dissociation, α (Table 5) for Mg, Ca, Sr, Ba myristates have been computed for premicellar region as $\alpha = \lambda/\lambda_\infty$. The variation of α (0.02-0.67) and K_D (3.3×10^{-12} to 3.8×10^{-7}) for alkaline earth metal soaps under study suggests (over the full temperature range) weak electrolytic behaviour of these systems. The dissociation constant, K_D (Table 5) however decreases with increasing temperature. K_D deduced from expression (i) is however found to be consistent with K_D obtained from $C^2\lambda^2$ vs. $1/\lambda$ plots.

Since alkali metal (Li, Na, K) myristates belong to 1:1 type electrolytes, the value for λ_∞ may be computed from the linear equation (Premicellar region) as proposed by Debye-Huckel-Onsager, [$\lambda = \lambda_\infty - (A+B\lambda_\infty)C^{1/2}$]. The intercepts of $\lambda-C^{1/2}$ plots (below the c.m.c.) give λ_∞ (Table 5), which is found to increase with increasing temperature. Degree of dissociation ($\alpha = \lambda/\lambda_\infty$) and dissociation constant ($K_D = C\alpha^2/(1-\alpha)$) for alkali metal myristates in 50% methanol +50 % chloroform have also been

computed (Table 5). The keen perusal of Table 5 (α and K_D data) conclusively prove that alkali metal myristates are much stronger electrolytes as compared to alkaline earth metal myristates.

Standard enthalpy change for dissociation process, ΔH_D^0 is computed from the slope of $\log K_D$ vs. $1/T$ plots (slope = $-\Delta H_D^0/2.303R$). The standard free energy change for dissociation process, ΔG_D^0 was then deduced from, $\Delta G_D^0 = -RT \ln K_D$. Here, K_D is the equilibrium constant for dissociation process. Eventually, Helmholtz expression, $T\Delta S_D^0 = \Delta H_D^0 - \Delta G_D^0$ was used to compute standard entropy change, ΔS_D^0 for dissociation. The thermodynamic parameters for dissociation (ΔH_D^0 , ΔG_D^0 and ΔS_D^0) are recorded in Table 6.

For the association or aggregation process, wherein counterions are bound to micelle, the standard free energy of micellization (per mole of monomer), ΔG_A^0 (Table 7) for the phase separation model (Barry *et al.*, 1972; Rabins and Thomas, 1968 and Atwood and Florence, 1983) is given by the relationship, $\Delta G_A^0 = 2RT \ln X_{cmc_s}$ where X_{cmc_s} is expressed as mole fraction and defined as: $X_{cmc_s} = n_s/n_s + n_o$. Since the number of moles of free surfactant (n_s) is small as compared to the number of moles of solvent (n_o), the expression: $X_{cmc_s} = n_s/n_o$ may be used. The standard enthalpy change of micellization per mole of monomer, ΔH_A^0 for the phase separation

Table 6 : Thermodynamic parameters for dissociation of alkali and alkaline earth metal myristates in 50% Chloroform + 50% methanol solution at different temperatures (30⁰-50⁰C)

Compounds	$-\Delta H_D^0$ (kJ mol ⁻¹)	ΔG_D^0 (kJ mol ⁻¹)					$-T\Delta S_D^0$ (kJ mol ⁻¹)				
		30 ⁰ C	35 ⁰ C	40 ⁰ C	45 ⁰ C	50 ⁰ C	30 ⁰ C	35 ⁰ C	40 ⁰ C	45 ⁰ C	50 ⁰ C
Lithium Myristate	172.32	17.099	17.813	18.356	19.785	20.208	155.221	154.507	153.964	152.535	152.112
Sodium Myristate	201.04	17.626	18.327	18.881	19.560	20.347	183.414	182.713	182.159	181.48	180.693
Potassium Myristate	153.17	17.883	18.707	19.288	20.072	20.827	135.287	134.463	133.882	133.098	132.343
Magnesium Myristate	191.47	37.694	38.590	39.824	42.166	46.742	153.776	152.880	151.646	149.304	144.728
Calcium Myristate	248.91	37.225	38.838	40.591	43.396	47.993	211.685	210.072	208.319	205.514	200.917
Strontium Myristate	172.324	41.303	43.920	53.014	66.314	52.320	131.021	28.404	199.310	106.010	120.004
Barium Myristate	382.942	48.517	51.677	54.144	66.314	71.033	334.425	331.265	328.798	316.628	311.909

Table 7 : Thermodynamic parameters for association of alkali and alkaline earth metal myristates at different temperatures (30⁰-50⁰C)

Compounds	$+\Delta H_D^0$ (kJ mol ⁻¹)	$-\Delta G_D^0$ (kJ mol ⁻¹)					$T\Delta S_D^0$ (kJ mol ⁻¹)				
		30 ⁰ C	35 ⁰ C	40 ⁰ C	45 ⁰ C	50 ⁰ C	30 ⁰ C	35 ⁰ C	40 ⁰ C	45 ⁰ C	50 ⁰ C
Lithium Myristate	41.57	30.95	31.82	32.82	34.00	35.17	72.52	73.9	74.9	75.57	76.74
Sodium Myristate	49.88	31.87	32.71	33.87	35.26	36.46	81.75	82.59	83.75	85.14	86.34
Potassium Myristate	49.88	32.46	33.54	34.63	36.33	37.70	82.34	83.59	84.51	86.21	87.58
Magnesium Myristate	89.79	30.95	31.91	32.92	34.06	35.17	120.74	121.70	122.71	123.85	124.96
Calcium Myristate	54.87	31.39	32.40	34.08	35.33	36.72	86.26	87.27	88.95	90.20	91.59
Strontium Myristate	46.56	31.87	32.80	34.08	35.26	36.63	78.43	79.38	80.64	81.82	83.19
Barium Myristate	33.26	33.00	34.22	35.58	36.91	38.90	66.26	67.48	68.84	70.17	72.16

model may thus be given by the relationship:

The values of ΔH_A^0 have been obtained from the slope of the linear plots of $\ln X_{cmc_s}$ vs. $1/T$ and are recorded in Table 7. The standard entropy change per mole of monomer (Table 7) is then calculated: $T\Delta S_A^0 = \Delta H_A^0 - \Delta G_A^0$.

From the knowledge of evaluated thermodynamic parameters (ΔH_A^0 , ΔG_A^0 , $T\Delta S_A^0$), as recorded in Table 7, one can say that the unfavorable enthalpy change for micellisation ($\Delta H_A^0 > 0$) is more than compensated by $\Delta G_A^0 < 0$ and $T\Delta S_A^0 > 0$. The data suggest good chance of aggregation or micellisation for both alkali and alkaline earth metal myristates (50% methanol + 50% chloroform) solution. A comparative study of thermodynamic parameters for dissociation and association processes in the present case (Table 6, 7) suggests that the latter process (micellisation) is dominant over the former.

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