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# THERMODYNAMICS AND ACOUSTICAL STUDIES ON CALCIUM SOAP SOLUTIONS

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The critical micelle concentration, degree of dissociation, dissociation constant and thermodynamic parameters for dissociation and micellization processes for calcium myristate in a mixture of chloroform and propylene glycol of varying composition and different temperatures were evaluated from the conductivity measurements. The results showed that these soaps behave as weak electrolytes in dilute solutions and the values of the CMC increase with the increasing temperature. The ultrasonic measurements for calcium soaps have been carried out to determine the CMC, soap-solvent interaction and various acoustic parameters. The results showed that the ultrasonic velocity, specific acoustic impedance, apparent molal compressibility and molar sound velocity increase while the adiabatic compressibility, intermolecular free length and solvation number decrease with increasing soap concentration.

**KEY WORDS:** Sound velocity, acoustic impedance.

## INTRODUCTION

The alkaline earth metal soaps are being widely used in industry, technology and allied sciences. Their selection for specific applications is governed by various fundamental properties. The physico-chemical characteristics and the structure of calcium soaps in the solid state have been investigated using infrared absorption spectra, x-ray diffraction patterns and thermal analysis.<sup>1,2</sup> Several workers<sup>3-10</sup> have determined the colloidal and physico-chemical properties of alkali and other metal soaps in aqueous and non-aqueous medium but the alkaline earth metal soaps have not yet been thoroughly studied. The present work deals with the conductivity and ultrasonic measurements of the solutions of calcium myristate in chloroform-propylene glycol mixture.

## EXPERIMENTAL

Calcium myristate was prepared by direct metathesis of potassium myristate with the required amount of aqueous solution of calcium acetate at 50-60°C under vigorous stirring. The precipitated calcium soap was washed with distilled water and alcohol. The soap was recrystallised, dried under reduced pressure and stored over calcium

**Table 1** Percentage of elements in calcium myristate.

<i>Elements</i>	<i>Found</i>	<i>Calculated</i>
Carbon	67.4	67.9
Hydrogen	10.6	10.9
Calcium	7.8	8.1

chloride. The absence of hydroxyl group in the soap was confirmed by studying the infrared absorption spectrum. The soap was analysed for carbon, hydrogen and metal contents and the results were found in agreement with the theoretical values (Table 1). The reproducibility of the results was checked by preparing two samples under similar conditions.

A digital conductivity meter (Toshniwal model CL 01.10A) and a dipping type conductivity cell with platinized electrode (cell constant 0.90) were used for measuring the conductance of the solutions at different temperatures.

The measurements of ultrasonic velocity of the soap solutions were carried out by an ultrasonic interferometer (F 81, Mittal Enterprises, Delhi) operating at a frequency of 4 MHz at a constant temperature  $40 \pm 0.5^\circ\text{C}$ .

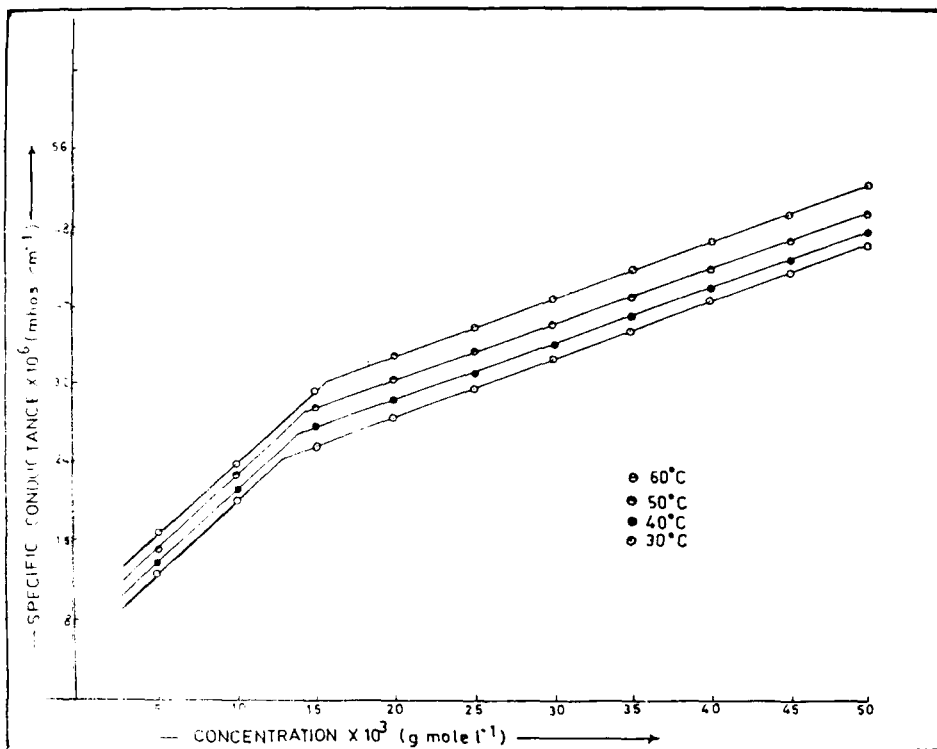
## RESULTS AND DISCUSSION

### A Conductivity Measurements

The specific conductance,  $k$  of the solutions of calcium myristate in mixtures of 70% chloroform + 30% propylene glycol and 50% chloroform + 50% propylene glycol (V/V) increases with the increase in soap concentration and temperature (Table 2). The plots of specific conductance vs soap concentration (Figure 1) are characterised

**Table 2** Values of specific conductance ( $\text{mhos cm}^{-1}$ ) of calcium myristate in chloroform propylene glycol mixtures of varying composition and temperatures.

<i>S. No.</i>	<i>Concentration</i> $C \times 10^3$ ( $\text{g mol l}^{-1}$ )	<i>50 % chloroform + 50 %</i> <i>propylene glycol</i>				<i>70 % chloroform + 30 %</i> <i>propylene glycol</i>			
		$30^\circ$	$40^\circ$	$50^\circ$	$60^\circ$	$30^\circ$	$40^\circ$	$50^\circ$	$60^\circ$
1	5.0	12.5	13.9	15.0	15.8	4.6	4.8	5.0	5.3
2	7.5	16.5	17.5	19.2	20.0	5.2	5.4	5.6	5.9
3	10.0	20.1	21.6	22.8	23.6	5.8	6.1	6.3	6.5
4	12.5	23.2	24.7	26.3	28.1	6.4	6.7	6.9	7.2
5	15.0	25.6	27.6	29.2	31.2	6.9	7.2	7.5	7.7
6	20.0	28.8	30.4	32.4	34.8	7.6	7.9	8.2	8.5
7	25.0	31.2	33.2	35.2	37.6	8.3	8.6	8.8	9.2
8	30.0	34.2	36.1	38.0	40.8	9.1	9.3	9.6	9.9
9	35.0	37.2	38.8	41.2	43.2	9.6	9.9	10.3	10.6
10	40.0	40.4	41.6	43.6	46.4	10.4	10.6	10.9	11.3
11	45.0	43.2	44.4	46.4	49.2	11.1	11.3	11.6	12.0
12	50.0	46.4	47.2	49.6	52.4	11.8	12.0	12.4	12.7



**Figure 1** Specific conductance v/s concentration plots for calcium myristate in 50% chloroform and 50% propylene glycol.

by an intersection of two straight lines at a concentration which corresponds to the CMC. The results show that the increase in temperature results in the increase of the CMC (Table 3).

The molar conductance,  $\mu$  of the dilute solutions of soaps decreases with increasing soap concentration. However, the CMC values cannot be obtained from the plots of molar conductance vs square root of soap concentration because the plots are concave upwards with increasing slopes indicating that these soaps behave as weak

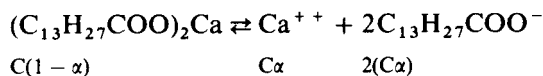
**Table 3** CMC values.

S. No.	Temperature	$CMC \times 10^3 (g. mol. l^{-1})$	
		50% chloroform and 50% propylene glycol	70% chloroform and 30% propylene glycol
1	30°C	12.6	12.6
2	40°C	13.8	13.8
3	50°C	14.6	14.6
4	60°C	15.7	15.7

**Table 4** Values of limiting molar conductance,  $\mu_0$ .

S. No.	Temperature	Limiting molar conductance, $\mu_0$	
		50 % chloroform and 50 % propylene glycol	70 % chloroform and 30 % propylene glycol
1	30°C	3.13	1.10
2	40°C	3.79	1.84
3	50°C	4.06	2.67
4	60°C	5.83	5.33

electrolytes in dilute solutions and Debye-Huckel Onsager's equation is not applicable to these soap solutions. Since the calcium myristate behaves as weak electrolyte in dilute solutions, an expression for the dissociation may be developed in Ostwald's manner.



where  $C$  and  $\alpha$  are the concentration and degree of dissociation of soap, respectively. The dissociation constant  $K$  can be expressed as:

$$K = \frac{[Ca^{++}][C_{13}H_{27}COO^-]^2}{[(C_{13}H_{27}COO)_2Ca]} = \frac{(C\alpha)(2C\alpha)^2}{C(1 - \alpha)}$$

$$= \frac{4C^2\alpha^3}{(1 - \alpha)} \quad (1)$$

Since the degree of dissociation of calcium soap in dilute solutions is small, ionic concentrations will be low and the interionic effects are almost negligible. Therefore, the dilute soap solutions do not deviate appreciably from the ideal behaviour and the activities of ions can be taken as almost equal to the concentrations. The degree of dissociation,  $\alpha$  may be replaced by the conductance ratio,  $\mu/\mu_0$ , where  $\mu$  is the molar conductance at finite concentration and  $\mu_0$  is the limiting molar conductance at infinite dilution. On substituting the value of  $\alpha$  and rearranging, Eq. (1) can be written as:

$$\mu^2 C^2 = \frac{K\mu_0^3}{4\mu} - \frac{K\mu_0^2}{4} \quad (2)$$

**Table 5** Values of dissociation constant from the plots of  $\mu^2 C^2$  vs  $1/\mu$ 

S. No.	Temperature	50 % chloroform and 50 % propylene glycol -log K	70 % chloroform and 30 % propylene glycol -log K
1	30°C	3.53	4.02
2	40°C	3.74	4.89
3	50°C	3.81	5.17
4	60°C	4.24	6.10

**Table 6** The values of  $-\log K$  at different temperatures.

S. No.	Concentration $C \times 10^3$ ( $\text{g mol}^{-1}$ )	50% chloroform + 50% propylene glycol				70% chloroform + 30% propylene glycol			
		30°	40°	50°	60°	30°	40°	50°	60°
1	5.0	3.284	3.477	3.459	3.752	4.474	4.533	5.095	6.000
2	7.5	3.590	3.883	3.688	4.392	3.818	4.660	5.164	5.883
3	10.0	3.019	3.113	3.125	3.309	4.896	4.668	5.138	6.105
4	12.5	3.442	3.705	3.636	4.257	3.948	4.668	5.135	6.029
5	15.0	2.786	2.857	2.868	3.000	4.939	4.670	5.118	6.000
6	20.0	2.641	2.707	2.707	2.815	4.021	4.662	5.197	6.050
7	25.0	2.513	2.567	2.567	2.666	4.016	4.750	5.200	6.035
8	30.0	2.387	2.441	2.452	2.543	4.013	4.672	5.151	6.082
9	35.0	2.286	2.340	2.340	2.433	4.050	4.710	5.135	5.948
10	40.0	2.191	2.247	2.247	2.330	3.934	4.595	5.148	6.075
11	45.0	2.100	2.156	2.167	2.240	3.893	4.588	5.046	5.972
12	50.0	2.019	2.076	2.087	2.162	3.865	4.597	5.096	5.881

The values of the limiting molar conductance,  $\mu_0$  and dissociation constant,  $K$  have been obtained from the slope,  $[K\mu_0^3/4]$  and intercept  $[-K\mu_0^2/4]$  of the linear plots of  $\mu^2C^2$  vs  $1/\mu$  below the CMC and are recorded in Tables 4 and 5. The results show that the values of limiting molar conductance increase while of dissociations constant decrease with increasing temperature.

The values of the degree of dissociation,  $\alpha$  at different soap concentrations and temperatures have been calculated by assuming it as equal to the conductance ratio,  $\mu/\mu_0$ . The results show that the calcium soap behaves as a weak electrolyte in these solutions. The values of the degree of dissociation and dissociation constant decrease rapidly in dilute solutions below the CMC but slowly above the CMC with increasing soap concentrations. The decrease in the values of the dissociation constant (Table 6), with increasing temperature indicates the exothermic nature of the dissociation of calcium soaps in chloroform propylene glycol mixtures.

The values of the heat of dissociation,  $\Delta H_D$  for calcium soap solutions have been obtained from the slope of linear plots of  $\log K$  vs  $1/T$  and are found to be  $-52.73$  and  $-134.25 \text{ K.J. mol}^{-1}$  for 50% and 70% chloroform, respectively. The heat of dissociation of calcium soap is negative indicating that the dissociation of calcium soap is exothermic.

**Table 7** Thermodynamic parameters for dissociation process.

S. No.	Temperature	50% chloroform + 50% propylene glycol		70% chloroform + 30% propylene glycol	
		$\Delta F_D$ ( $\text{KJ mol}^{-1}$ )	$\Delta S_D \times 10^2$ ( $\text{KJ mol}^{-1}$ )	$\Delta F_D$ ( $\text{KJ mol}^{-1}$ )	$\Delta S_D \times 10^2$ ( $\text{KJ mol}^{-1}$ )
1	30°	10.25	-20.79	11.68	-48.16
2	40°	11.22	-20.43	14.69	-47.58
3	50°	11.80	-19.98	16.03	-46.53
4	60°	13.52	-19.89	19.46	-46.16

The values of the changes in free energy,  $\Delta F_D$  and entropy,  $\Delta S_D$  per mole for the dissociation process have been calculated by using the relationships:

$$\Delta F_D = -RT \ln K_D \quad (3)$$

$$\Delta S_D = \frac{\Delta H_D - \Delta F_D}{T} \quad (4)$$

The calculated values of  $\Delta F_D$  and  $\Delta S_D$  are recorded in Table 7.

For the aggregation process, when counter ions are bound to micelle, the standard free energy of micellization (per mole of monomer),  $\Delta F_A$ , for the phase separation model<sup>11,12</sup> is given by the relationship:

$$\Delta F_A = 2RT \ln X_{\text{CMC}} \quad (5)$$

where  $X_{\text{CMC}}$  is the CMC expressed in terms of mole fraction and is given by the relationship:

$$X_{\text{CMC}} = \frac{n_s}{n_s + n_0}$$

where  $n_s$  and  $n_0$  are the number of moles of surfactant and solvent, respectively. Since the number of moles of free surfactants,  $n_s$ , is small as compared to the number of moles of solvent,  $n_0$  it follows that:

$$X_{\text{CMC}} = \frac{n_s}{n_0}$$

The standard enthalpy change of micellization per mole of monomer for the phase separation model<sup>11,12</sup>  $\Delta H_A$  is given by the relationship:

$$\frac{\partial(\ln X_{\text{CMC}})}{T} = -\frac{\Delta H_A}{2RT^2}$$

$$\ln X_{\text{CMC}} = \frac{\Delta H_A}{2RT} + C \quad (6)$$

The values of  $\Delta H_A$  have been obtained from the slope of the linear plots of  $\ln X_{\text{CMC}}$  vs  $1/T$  and are found to be  $-26.87$  and  $-28.92$  K.J. mol<sup>-1</sup> for 50% and 70% chloroform, respectively.

The negative values of  $\Delta F$  and positive values of  $\Delta S$  for micellization process (Table 8) and positive values of  $\Delta F$  and negative values of  $\Delta S$  for dissociation process (Table 8) indicate that the micellization is favoured over dissociation process. The values of  $\Delta H$  show that the micellization is less exothermic than the dissociation process but the entropy factor predominates over the enthalpy and so the micellization is predominant over dissociation.

It is, therefore, concluded that the thermodynamics of dissociation and association can satisfactorily be explained in the light of phase separation model and conductivity measurements. The results showed that the micellization process is predominant over dissociation process and calcium soaps behaved as weak electrolytes in chloroform-propylene glycol mixtures.

**Table 8** Thermodynamic parameters for association process.

S. No.	Temperature	50 % chloroform + 50 % propylene glycol		70 % chloroform + 30 % propylene glycol	
		$\Delta F_A$ (KJ mol <sup>-1</sup> )	$\Delta S_A \times 10^2$ (KJ mol <sup>-1</sup> )	$\Delta F_A$ (KJ mol <sup>-1</sup> )	$\Delta S_A \times 10^2$ (KJ mol <sup>-1</sup> )
1	30°	-39.55	4.18	-40.43	3.80
2	40°	-40.30	4.29	-41.22	3.93
3	50°	-41.26	4.46	-42.18	4.11
4	60°	-42.06	4.56	-43.02	4.23

### B Ultrasonic Measurements

**Calculations** The specific acoustic impedance,<sup>13</sup>  $Z$ , intermolecular free length,<sup>14</sup>  $L_f$ , adiabatic compressibility,  $\beta$ , apparent molal compressibility,  $\phi_k$  and molar sound velocity,  $R$  have been calculated by using the relationship:

$$Z = \rho \cdot v \quad (7)$$

$$\beta = v^{-2} \rho^{-1} \quad (8)$$

$$L_f = K\sqrt{\beta} \quad (9)$$

$$\phi_k = \frac{1000}{C\rho_0} (\rho_0\beta - \beta_0\rho) + \frac{\beta_0 M}{\rho_0} \quad (10)$$

and

$$R = \frac{\bar{M}}{\rho} v^{1/3} \quad (11)$$

$$\bar{M} = \frac{n_0 M_0 + nM}{n_0 + n} \quad (11)$$

where,  $\rho_0$ ,  $\rho$ ;  $\beta_0$ ,  $\beta$ ;  $n_0$ ,  $n$ ; and  $M_0$ ,  $M$  are the density, adiabatic compressibility, number of mole, and molecular weight of the solvent and solute, respectively and  $K$ ,  $C$  and  $v$  are the temperature dependent Jacobson's constant, concentration in  $\text{g} \cdot \text{mol}^{-1}$  and ultrasonic velocity, respectively.

The results of ultrasonic velocity,  $v$  and various acoustical parameters for the solutions of calcium myristate in a mixture of 50% chloroform and 50% propylene glycol are recorded in Table 9. The variation of ultrasonic velocity with concentration,  $(dv/dc)$  depends on the concentration derivatives of density and adiabatic compressibility.  $(dv/dc) = -v/2[1/\rho(d\rho/dc) + 1/\beta(d\beta/dc)]$

The results show that the quantity  $(d\rho/dc)$  is positive while  $(d\beta/dc)$  is negative. Since the values of  $1/\beta(d\beta/dc)$  are higher than  $1/\rho(d\rho/dc)$  for the solutions of calcium myristate, the concentration derivative of velocity,  $(dv/dc)$  is positive which is in agreement with the results of other workers<sup>15-17</sup> reported for electrolytic solutions.



**Table 9** Ultrasonic velocity and other acoustic parameters of calcium myristate in a mixture of 50% chloroform and 50% propylene glycol (V/V) at  $40 \pm 0.5^\circ\text{C}$ .

<i>S. No.</i>	<i>Concentration</i> $C \times 10^3$ (g mol l <sup>-1</sup> )	<i>Density</i> (g ml <sup>-1</sup> ) $\rho$	<i>Velocity</i> $v \times 10^{-5}$ (cm/sec)	<i>Adiabatic com- pressibility</i> $\beta \times 10^{11}$ (cm <sup>2</sup> /dyne)	<i>Inter- molecular free length</i> $L_f \times 10^3$ cm.	<i>Specific acoustic impedance</i> $Z \times 10^{-5}$ (CGS)	<i>Apparent molal com- pressibility</i> $\phi_k \times 10^7$	<i>Molar sound velocity</i> $R \times 10^{-1}$
1	5.0	1.0695	1.423	4.618	4.362	1.532	-3.324	481.2
2	5.7	1.0710	1.431	4.560	4.335	1.533	-3.042	485.8
3	10.0	1.0726	1.438	4.509	4.311	1.542	-2.780	490.2
4	12.5	1.0742	1.446	4.452	4.284	1.443	-2.637	494.7
5	15.0	1.0757	1.452	4.409	4.263	1.562	-2.545	499.0
6	20.0	1.0770	1.457	4.374	4.246	1.569	-2.054	507.5
7	25.0	1.0792	1.462	4.335	4.227	1.578	-1.794	515.5
8	30.0	1.0811	1.466	4.304	4.212	1.585	-1.587	523.4
9	35.0	1.0830	1.473	4.261	4.191	1.594	-1.476	531.6
10	40.0	1.0849	1.475	4.237	4.179	1.600	-1.348	539.2
11	45.0	1.0868	1.482	4.189	4.155	1.611	-1.298	547.3
12	50.0	1.0886	1.485	4.166	4.144	1.617	-1.208	554.8

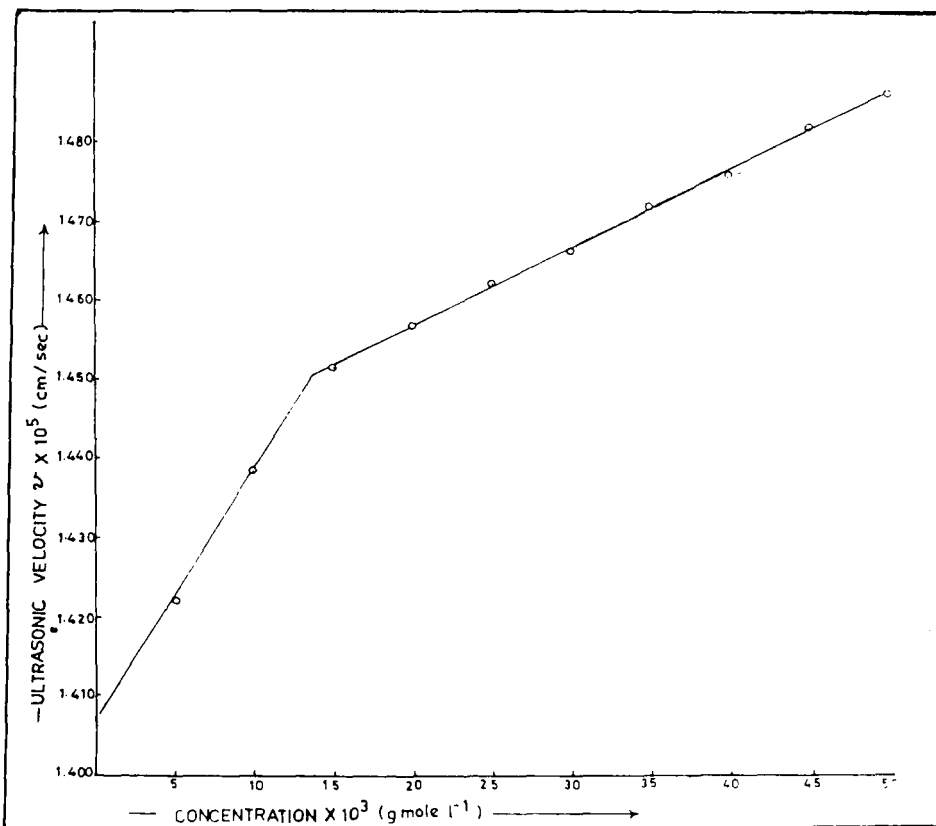


Figure 2 Ultrasonic velocity  $v$  vs concentration plot for calcium myristate in 50% chloroform and 50% propylene glycol.

The plots of ultrasonic velocity,  $v$ , adiabatic compressibility,  $\beta$ , intermolecular free length,  $L_f$ , and specific acoustic impedance,  $Z$ , vs soap concentration,  $C$  are characterized by an intersection of two straight lines at a concentration which corresponds to the critical micelle concentration, (CMC) of calcium myristate ( $13.8 \times 10^{-3}$  M). The value of the CMC is in agreement with the results of other micellar properties. The variation in the ultrasonic velocity,  $v$  with the soap concentration,  $C$  follow the relationship:

$$v = v_0 + GC \quad (12)$$

where  $v_0$  is the ultrasonic velocity of solvent and  $G$  is the Garnsey's constant. The calculated value of Garnsey's constant,<sup>18</sup>  $G$  for calcium myristate is  $3.15 \times 10^5$ .

The plot of ultrasonic velocity,  $v$  vs soap concentration,  $C$  (Figure 2) is extrapolated to zero soap concentration and the extrapolated value of the ultrasonic velocity ( $1.408 \times 10^5$  cm/sec) is in close agreement with the ultrasonic velocity of solvent mixture ( $1.402 \times 10^5$  cm/sec).

The adiabatic compressibility,  $\beta$  and intermolecular free length,  $L_f$  of the solutions of calcium myristate decrease with the increase in ultrasonic velocity and soap concentration. The adiabatic compressibility,  $\beta$  of the solutions of calcium myristate is found to obey Bachem's relationship.<sup>19</sup>

$$\beta = \beta_0 + AC + BC^{3/2} \quad (13)$$

where  $\beta_0$  is the compressibility of solvent,  $C$  is the molar concentration and  $A$  and  $B$  are constants. The values of constants  $A$  and  $B$  have been obtained from the intercept and slope of the plots of  $(\beta - \beta_0)/C$  vs  $\sqrt{C}$ . The values of constants  $A$  and  $B$  for calcium myristate are  $-4.4 \times 10^{-10}$  and  $16.7 \times 10^{-10}$ , respectively.

The decrease in the values of adiabatic compressibility and intermolecular free length with increase in ultrasonic velocity indicates that there is a significant interaction between soap and solvent molecules due to which the structural arrangement is considerably affected.<sup>20</sup> The soap-solvent interaction in calcium soap solutions has also been confirmed from the conductivity,<sup>21</sup> density and viscosity measurements.<sup>22</sup>

The plot of  $\phi_k$  vs  $\sqrt{C}$  shows a break at a concentration which corresponds to the CMC of the soap. The value of  $\phi_k^0$  has been obtained by extrapolation of the plot of  $\phi_k$  vs  $\sqrt{C}$  below the CMC to zero soap concentration. The limiting value of apparent molal compressibility,  $\phi_k^0$  is found to be  $-4.680 \times 10^{-7}$  for calcium myristate. The results are in agreement with the results reported by Masson<sup>23</sup> for electrolytic solutions.

The increase in the value of specific acoustic impedance,  $Z$ , with soap concentration,  $C$ , can be explained on the basis of lipophobic interaction between soap and solvent molecules which increases the intermolecular distance making relatively wider gaps between the molecules and becoming the main cause of impediment in the propagation of ultrasound waves. The values of molar sound velocity,  $R$  increase linearly with increasing soap concentration (Table 9).

The ultrasonic measurements show that the adiabatic compressibility and intermolecular free length for calcium soaps decrease while the specific acoustic impedance, apparent molal compressibility and molar sound velocity increase with increasing soap concentration.

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