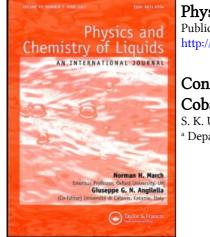
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CONDUCTOMETRIC AND ACOUSTICAL STUDIES AND COMPRESSIBILITY BEHAVIOUR OF COBALT CAPRATE IN DIMETHYLFORMAMIDE

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The conductivity results showed that the cobalt caprate behaves as weak electrolytes in dimethylformamide and the micellization process is dominant over the dissociation process. The values of CMC increase with increasing temperature. The ultrasonic results indicated that there was a significant interaction between the soap and solvent molecules. The ultrasonic velocity, specific acoustic impedance, apparent molar compressibility and apparent molar volume increase while adiabatic compressibility, intermolecular free length, available volume, solvation number and relative association decrease with increasing the concentration of cobalt caprate in DMF.

Keywords: Critical micellar concentration; phase separation model; specific acoustic impedance

INTRODUCTION

Metal soaps, divalent or trivalent metal salt of saturated and unsaturated higher fatty acids, have a wide variety of technical applications. The utility of metallic soaps as dispersants [1], lubricants [2, 4], catalysts [5, 6], stabilizers [7] and corrosion inhibiting agents [8, 9] has motivated research workers. The density measurements of cobalts [10] and copper [11] soaps of lower fatty acids in non-aqueous media have been used to determine the critical micelle concentration. The colloidal behaviour and time effect in the supramolecular

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formation of cobalt monooleate in a polar solvent have been investigated by Benjamin Chu *et al.* [12, 13]. Studies with ultrasonic waves have gained in importance in recent years. Recent developments have made it possible to use ultrasonic energy in medicine, engineering, agriculture and industry. Particularly in Chemistry, ultrasonic energy is found useful in studying the chemical processes playing a major role in synthesis of chemical substances. The ultrasonic waves with low amplitude have been used by many research workers to investigate the nature of interactions, which occur between the molecules of the medium.

The present work deals with the conductivity and ultrasonic measurements of cobalt caprate in dimethylformamide to determine various thermodynamic and acoustical parameters. The ultrasonic results are also discussed in terms of different theories of propagation of ultrasonic waves.

EXPERIMENTAL

Analar grade (Glaxo, EM & BDH) cobalt acetate, capric acid, alcohol, acetone and dimethyl formamide were used for the preparation of soap and solutions.

The cobalt caprate was prepared by direct metathessis of potassium caprate with the required amount of cobalt acetate in water alcohol (1:1) medium. The bluish precipitate was filtered by suction and washed by distilled water and acetone to remove excess of cobalt ions and unreacted capric acid. The infrared absorption measurements showed no free capric acid. The purity of the soap was checked by elemental analysis and the results were found in agreement with the theoretically calculated values. The solutions of cobalt caprate were prepared by dissolving a known amount of soap in dimethylformamide and were kept for 2h in a thermostat at the desired constant temperature. The conductance of the solutions was measured with a Systronics conductivity Bridge 305 (SR. No. 993) and a dipping type conductivity cell (cell constant 1.6) at different temperatures. The reproducibility ($\pm 0.1\%$) of the measurements were examined by repeating them several times.

The ultrasonic measurements were recorded on a multi-frequency ultrasonic interferometer (MX-3, Mittal Enterprises, New Delhi) at 40 \pm 0.5°C using a crystal of frequency 1 MHz. The uncertainty of velocity measurements is 0.2%. The densities of solvent and solutions were determined with a pyknometer calibrated with pure benzene. The volume of pyknometer was 10 ml.

RESULTS AND DISCUSSION

Conductance

The specific conductance, k of the soap solutions increases with an increase in soap concentration, C (Fig. 1). This may be due to the fact that cobalt caprate behaves as a simple electrolyte in dilute solutions and is ionized to a considerable extent into metal cation and capric acid anions. The increase in specific conductance with concentration is due to increase in the number of ions per unit volume of solutions. In case of cobalt caprate the increase is not so large because of the changing partial ionization of the soap below the CMC and consequently the conductance does not go up so rapidly as in strong electrolytes. The plots of specific conductance vs. soap concentration (Fig. 1) exhibit a break at the CMC at which the anions begin to aggregate together to form ionic micelles. The results show that the increase in temperature results in the increase of the CMC (Tab. I), because the micellization process is assumed to occur when the energy released as a results of aggregation of the hydrocarbon chains of the monomer is sufficient to overcome to the electrical repulsion between the ionic head groups and to balance the decrease in entropy accompanying aggregation. Therefore, increase in temperature would have been expected to increase the CMC values since the kinetic energy of the monomers would have been raised.

Unlike the specific conductance the molar conductance, μ of the dilute solutions of cobalt caprate increases with dilution. The reason for this is that the decrease in specific conductance is more than compensated by the increase in the value of I/C on dilution, and hence molar conductance, μ goes up. The critical micellar concentration, CMC, cannot be however, obtained from the plots of molar

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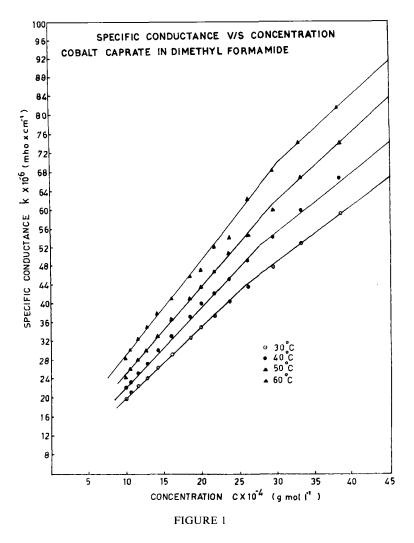


TABLE I Critical micellar concentration (CMC), limiting molar conductance (μ_0) and dissociation constant (K_D) of cobalt caprate in dimethylformamide at different temperatures

Temperature °C	30	40	50	60
$\frac{\text{CMC (g-mol } 1^{-1} \times 10^{-4})}{\mu^0}$ $K_D \times 10^{-9}$	26.0 29.1 1.79	27.5 30.1 2.05	29.4 31.7 2.51	31.0 32.8 2.97

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conductance vs. square root of soap concentration, which are concave upwards with increasing slopes indicating that the Debye-Hückel Onsager's equation is not applicable to these soap solutions. Since the cobalt caprate behaves as a weak electrolyte in dilute solutions, the following expression for the dissociation of soaps can be derived.

$$\frac{\operatorname{Co}(\operatorname{C}_{9}\operatorname{H}_{19}\operatorname{Coo})_{2}}{C(1-\alpha)} \stackrel{\operatorname{Co}^{++}}{\longrightarrow} \frac{2\operatorname{C}_{9}\operatorname{H}_{19}\operatorname{Coo}^{-}}{2C\alpha}$$
(1)

where C and \propto are the concentration and degree of dissociation of cobalt caprate, respectively. The dissociation constant K for the above mentioned Eq. (1) can be expressed as:

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

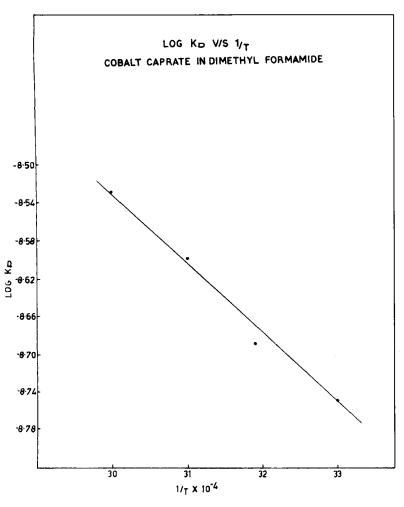
Since the values of degree of dissociation of cobalt caprate in dimethyl formamide are small, the ionic concentrations are low and the interionic effects are almost negligible. Therefore, the very dilute solutions of soaps do not deviate appreciably from ideal behaviour and the activities of ions can be taken as almost equal to concentration. The degree of dissociation ∞ has been determined by the conductance ratio, μ/μ_0 where μ is the molar conductance at finite concentration and μ_0 is the limiting molar conductance at infinite dilution. On substituting the value of ∞ and rearranging Eq. (2) the following expression for the dissociation of soap can be derived.

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

The values of the dissociation constant, K_D and μ_0 (Tab. I) have been obtained from the slope, $[K_D\mu_0^3/4]$ and the intercept, $[-K_D\mu_0^2/4]$ of the linear plots of $\mu^2 C^2$ vs. $1/\mu$ for dilute soap solutions. It follows from Table I that an increase in temperature brings about an increase in the limiting molar conductance and dissociation constant. The degree of dissociation, \propto may be evaluated by assuming it to be equal to the conductance ratio, μ/μ_0 , and using the value of μ_0 (Tab. I) as observed from the plots of $\mu^2 C^2$ vs. $1/\mu$.

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The value of heat of dissociation ΔH_D^0 for cobalt caprate in dimethylformamide has been obtained from the slope of the linear plot of log K_D vs. 1/T (Fig. 2). The negative value (-14.57 KJ mol⁻¹) of heat dissociation ΔH_D indicates that the dissociation of cobalt caprate in dimethylformamide is exothermic. The standard free energy change ($\Delta G_D^0 = -RT \ln K_D$, where K_D is the equilibrium constant),



and standard entropy change $\Delta S_D^0 = (\Delta H_D^0 - \Delta G_D^0)/T$, are evaluated for the dissociation process. For micellization, i.e., aggregation process, when counter-ions are bound to a micelle, the standard free energy change of micellization per mole of monomer, ΔG_A for the phase separation model [15–17] is expressed by the relationship

$$\Delta G_A^0 = 2RT \ln X_{\rm CMC} \tag{4}$$

where X_{CMC} is the CMC expressed as a mole fraction and is defined,

$$X_{\rm CMC} = \frac{n_s}{n_s + n_0} = \frac{n_s}{n_0} \tag{5}$$

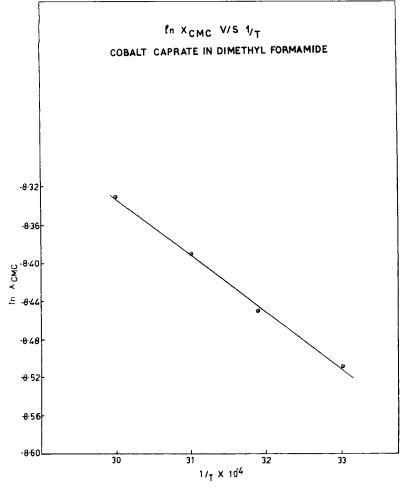
Since the number of mole of free surfactants, n_s is small as compared to the number of moles of solvent, n_0 . The standard enthalpy change of micellization, ΔH_A^0 , per mole of monomer for the phase separation model [15–17] is evaluated as follows:

$$\frac{\partial (\ln X_{\rm CMC})}{\partial T} = -\frac{\Delta H_A^0}{2RT^2}$$
(6)
$$\ln X_{\rm CMC} = \Delta H_A^0 / 2RT + C$$

The value of ΔH_A^0 has been obtained from the slope of linear plots of ln $X_{\rm CMC}$ vs. 1/T (Fig. 3). The positive enthalpy (11.88 KJ mol⁻¹) for micellization indicates that the association of cobalt caprate in DMF is endothermic. The standard entropy change per mole of monomer is determined as follows :

$$\Delta S^0_A = (\Delta H^0_A - \Delta G^0_A)/T \tag{7}$$

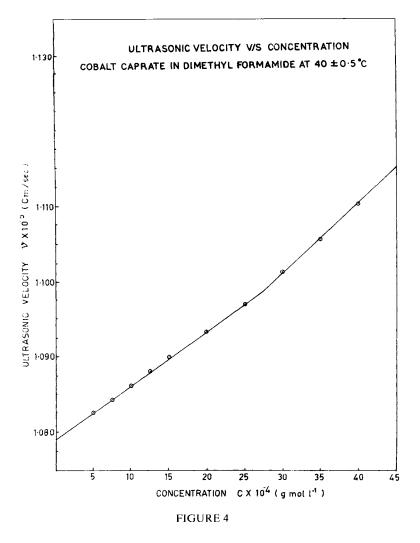
The micellization of the soaps (Tab. II) in DMF is consistent with $\Delta H_A^0 > 0$, $\Delta G_A^0 < 0$, $\Delta S_A^0 > 0$. On the other hand, dissociation of cobalt caprate in DMF (Tab. II) in consistent with $\Delta H_D^0 < 0$, $\Delta G_D^0 > 0$, $\Delta S_D^0 < 0$. The negative enthalpy change of dissociation more than make up for the unfavourable change in free energy and the entropy of the dissociation (Tab. II). For the process of dissociation, the unfavourable entropy change (i.e., $\Delta S_D^0 < 0$) may in fact be due to ion-solvent interaction, i.e., solvation of the soap molecules.



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TABLE II Thermodynamic parameters of the dissociation and association of cobalt caprate in dimethylformamide

Temp. °C	Dissociation Process		s Association Process			
°C	$\Delta G_D \ KJ. \ mole^{-1}$	$\Delta S_D imes 10^2$ KJ mole ⁻¹	ΔG_A KJ mole ¹	$\Delta S_A imes 10^2$ KJ mole ⁻¹		
30	25.4	-13.2	-21.50	11.0		
40	26.1	-12.9	-22.1	10.7		
50	26.7	-12.8	-22.6	10.3		
60	29.1	-13.1	-23.1	9.9		



The higher values of negative enthalpy as well as lower values of unfavourable free energy change $(\Delta G_D^0 > 0)$ for the dissociation of cobalt caprate suggest an increased dissociation in DMF. After a careful scrutiny of the thermodynamic parameters it may, therefore, be inferred that in DMF the association of cobalt caprate is predominant over dissociation.

Ultrasonic Studies

The ultrasonic velocity $v(\pm 0.2 \%)$ and other acoustic and soapsolvent interaction parameters for cobalt caprate in dimethylformamide are recorded in Tables III and IV. The variation in ultrasonic velocity v with soap concentration C follows the relationship.

$$v = v_0 + GC \tag{8}$$

where v_0 is the ultrasonic velocity in pure solvent and G is Garnsey's constant [18]. The value of Garnsey's constant G for cobalt caprate is 7.0×10^{-5} . Both ultrasonic velocity $v ~(\pm 0.2 \%)$ and adiabatic compressibility $\beta ~(\pm 3.5 \%)$ (Tab. III), when plotted as a function of soap concentration C, show an intersection of two straight lines at a definite soap concentration, the CMC of cobalt caprate. The values of the CMC is consistent with the value obtained from conductivity and density measurements. The plots of v vs. C and β vs. C when extrapolated to zero soap concentration give pure solvent values ($v_0 = 1.078 \times 10^{-5}$ cm/sec and $\beta_0 = 1.062 \times 10^{-10}$ cm²/dyne) in accordance with the experimental values ($v_0 = 1.075 \times 10^5$ cm/sec and $\beta_0 = 1.061 \times 10^{-10}$ cm²/dyne), indicating that the soap molecule do not aggregate to an appreciable extent below the CMC.

S.No.	Concentration (g. mol^{-1}) $C \times 10^4$ g. mol 1^{-1}	Density (g. mol ⁻¹) p	Ultrasonic Velocity (cm/sec.) $v \times 10^{-5}$ (cm/sec.)	Adiabatic Compressibility $\beta \times 10^{10}$ (cm ² dyne)	Intermolecular Free Length L _f (Å)	Specific Acoustic Impedance $Z \times 10^{-5}$ (C. G. S. Unit)
1.	5.0	0.8108	1.0825	1.053	0.6588	8.777
2.	7.5	0.8115	1.0843	1.048	0.6572	8.799
3.	10.0	0.8124	1.0860	1.044	0.6560	8.823
4.	12.5	0.8133	1.0876	1.039	0.6544	8.847
5.	15.0	0.8140	1.0898	1.034	0.6528	8.871
6.	20.0	0.8158	1.0933	1.026	0.6503	8.919
7.	25.0	0.8174	1.0968	1.017	0.6474	8.965
8.	30.0	0.8189	1.1010	1.007	0.6442	9.016
9.	35.0	0.3200	1.1053	0.998	0.6414	9.063
10.	40.0	0.8213	1.1100	0.988	0.6381	9.116
11.	45.0	0.8225	1.1148	0.978	0.6349	9.169

TABLE III Ultrasonic velocity of cobalt caprate in dimethylformamide at $40 \pm 0.5^{\circ}$ C

S .No.	Concentration (g. mol 1 $^{-1}$)	Apparent Molar Volume	Apparent Molar Compressibility	Solvation Number	Relative Association	Available Volume
	$C \times 10^4$ (g·mol 1 ⁻¹)	$cm^5 dyne^{-1}$ (g·mol ⁻¹)	$\phi_k imes 10^6 \ cm^5 dyne^{-1} \ (g\cdot mol)^{-1}$	$S_n \times 10^8$	R _A	V _a
1.	5.0	-7141.37	-3.23	24.21	0.999	92.23
2.	7.5	-5820.91	-3.09	24.21	0.998	91.83
3.	10.0	-5408.27	-2.93	23.00	0.998	91.42
4.	12.5	-5160.68	-2.91	23.24	0.987	90.92
5.	15.0	-5116.78	-2.92	23.41	0.996	90.57
6.	20.0	-4665.51	-2.77	21.11	0.995	89.75
7.	25.0	-4467.44	-2.73	21.99	0.994	88.96
8.	30.0	-4294.13	-2.74	22.60	0.992	88.05
9.	35.0	-4028.86	-2.70	22.48	0.990	87.18
10.	40.0	-3891.85	-2.70	22.70	0.987	86.21
11.	45.0	-3757.70	-2.70	22.87	0.984	85.24

TABLE IV Acoustic and other ellied parameters of cobalt caprate in dimethylforma-mide at 40 \pm 0.5°C

The adiabatic compressibility has been calculated by the following relationship:

$$\beta = p^{-1} v^{-2} \tag{9}$$

The adiabatic compressibility of cobalt caprate in dimethylformamide decreases with increasing soap concentration (Tab. III). The decrease in adiabatic compressibility is attributed to the fact that the cobalt caprate in dilute solution is considerably ionized into cobalt cation and capric acid anions. These ions are surrounded by a layer of solvent molecules firmly bound and oriented towards the ions. The orientation of solvent molecules around the ions is attributed to the influence of electrostatic field of the ions and thus the internal pressure increases, which lowers the compressibility of soap solutions i.e., solutions become harder to compress [19].

The decrease in adiabatic compressibility in post micellization region may be explained on the basis of closed packing of ionic head groups in the micelles, resulting in an increase in ionic repulsion and finally internal pressure.

The values of apparent molar compressibility ϕ_k (Tab. IV) at different soap concentrations have been calculated by the following relationship:

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$$\phi_k = \frac{10^3 (\beta - \beta_0)}{C p_0} + \beta \phi_v \tag{10}$$

where β , β_0 , p_0 , C and ϕ_v are the adiabatic compressibility of the solutions, adiabatic compressibility of the solvent, density of the solvent, soap concentration and apparent molar volume, respectively.

From the Debye-Huckel theory it follows that the apparent molar compressibility ϕ_k is related to the molar concentration of soap C by the relationship:

$$\phi_k = \phi_k^0 + S_k C^{\frac{1}{2}} \tag{11}$$

The value of limiting apparent molar compressibility ϕ_k^0 (-3.71 × 10⁻⁶) and constant S_k (2.24 × 10⁻⁵) have been evaluated from the intercept and slope of the plot of ϕ_k vs . $C^{1/2}$ below the CMC and compared with the results of different electrolytes [20–22]. This plot is also characterised by a break at the CMC. The positive value of S_k signifies a considerable soap solvent interaction below the CMC. It is apparent that the negative values of the apparent molar compressibility decrease linearly with concentration in pre-micellization region, but this decrease is very sharp in pre-micellization region as compared to post micellization region, indicating the poor compressibility at higher concentrations. However, the elevation of temperature increases the negative value of the apparent molar compressibility probably due to the decreasing internal pressure.

The intermolecular free length [23] (L_f) and specific acoustic impedance [24] (Z) have been evaluated using the following relationships:

$$L_f = K\sqrt{\beta} \tag{12}$$

$$Z = pv \tag{13}$$

where K is the temperature dependent Jacobson's constant (K at $40^{\circ}C = 6.42 \times 10^{-4}$ dyne cm⁻¹). The decrease in the values of intermolecular free length L_f (\pm 0.2 %) (Tab. III) and increase in the values of specific acoustic impedance Z (\pm 2.2 %) with increasing the concentration of cobalt caprate can be explained on the basis of

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hydrophobic interaction between soap and solvent molecules, which increases the intramolecular distance, leaving relatively wider gap between the molecules and thus becoming the main reason of impediment to the propagation of ultrasound waves. The plots of L_f vs. C and Z vs. C show breaks indicating the CMC, and extrapolation gives pure solvent values ($L_f = 0.6576$ Å and $Z = 8.802 \times 10^{-5}$ CGS unit) in accordance with the experimental values.

The solvation number [25] (Sn) and relative association [26] (R_A) of cobalt caprate (Tab. IV) have been determined by the relationships:

$$S_n = \frac{n_1}{n_2} \left(1 - \frac{\beta}{\beta_0} \right) \tag{14}$$

$$R_{A} = \frac{p}{p_{0}} \left(\frac{v_{0}}{v}\right) \tag{15}$$

where n_1 , n_2 ; p_0 , p, β_0 , β , v_0 , v are the number of moles, density, adiabatic compressibility and ultrasonic velocity of the solvent and solution respectively. The values of solvation number and relative association (Tab. IV) decrease with increasing soap concentration. The values of Sn correspond to the number of solvent molecules in the primary solvation sheaths of the ions. On account of electrostriction, molecules in the primary solvation sheath will be highly compressed so that these molecules will be less compressible than those in the bulk of the solution when an external pressure is applied. The compressibility of solvent molecules near but not in the primary solvation sheaths is the same as that of pure solvent. The decrease in the values of relative association R_A has been attributed either to the decreased association between soap and organic solvent molecules at higher concentration or decreasing solvation of ions. The values of apparent molar volume increase (Tab. IV) while the values of available volume decrease with increasing soap concentration. The plots of apparent molar volume vs. $C^{1/2}$ and available volume vs. C are characterised by the break at the CMC.

The results of conductivity and ultrasonic studies show that the cobalt caprate behaves as weak electrolyte in DMF. These results also confirm that there is a significant interaction between soap and solvent molecules and the values of various acoustic parameters are in good agreement with the results of other workers [27, 19].

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