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Viscometric and Ultrasonic Studies on Chromium Soap Solutions

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The molar volume, critical micelle concentration, and various acoustic parameters (adiabatic compressibility, intermolecular free length, specific acoustic impedance, molar sound velocity, molar sound compressibility, apparent molar compressibility, relative association, available volume, and solvation number) were evaluated from the density, viscosity, and ultrasonic measurements of the solutions of chromium soaps (myristate, palmitate, and stearate) in a benzene−methanol mixture (4:1 v/v). The molar volumes obtained by using Einstein's and Vand's equations showed a decrease with increasing chain length of the soap molecules. The values of the critical micelle concentration obtained from ultrasonic velocity measurements were found to be in close agreement with those evaluated from viscosity measurements and showed a decrease with increasing chain length of the fatty acid constituent of soap molecules.

Introduction

Recently, the transition-metal soaps have found wide applications in many industries. The physicochemical properties and the structure of these soaps depend largely on the method and conditions of their preparation. The exact information on the nature and structure of these soaps is of great significance for their use in different industries and for explaining their characteristics under different conditions. The IR spectra of the cobalt soaps, cobalt complexes, and copper soaps were investigated by Chuev and Shchennikova (*1*), Kambe (*2*), and Kuroda (*3*), respectively. Zul'Fugarly et al. (*4*) and Mehrotra et al. (*5, 6*) studied the IR spectra of nickel, copper, and manganese soaps. The absorption spectra of nickel and chromium soaps in different solvents were studied by Malik and Ahmad (*7*). The critical micelle concentration (CMC) of copper, nickel, and chromium soaps and their chemical behavior as colloids were investigated by Mehrotra et al. (*8, 9*) and by Malik and Ahmad (*10*). Varma et al. (*11−13*) determined the CMC of nickel, cobalt, iron, and zinc soaps in alcohols and mixed solvents at different temperatures using conductivity and viscosity measurements.

The present investigation deals with the studies on the viscosity and ultrasonic velocity of the solutions of chromium soaps (myristate, palmitate, and stearate) in a benzene−methanol (4:1 v/v) mixture with a view to study the soap−solvent and soap−soap interactions and to evaluate the CMC and various other parameters.

Experimental Section

All chemicals were of Anal R grade, Chromium soaps (myristate, palmitate, and stearate) were prepared by direct metathesis of the corresponding potassium soap with the required amount of aqueous solution of chrome alum. The soaps were washed with distilled water, methanol, and acetone to remove the excess of metal ion, potassium soap, and unreacted fatty acid. The soaps were purified by recrystallization with a benzene−methanol mixture, and the purity of the soap was checked by its melting point and elemental analysis: Myristate, (mp 61 °C). Anal. Calcd C, 60.2; H, 9.6. Found: C, 60.3; H, 9.8. Palmitate (mp 66 °C). Anal. Calcd C, 62.5; H, 10.1. Found: C, 62.7; H, 10.2. Stearate (mp 69 °C). Anal. Calcd C, 64.5; H, 10.4. Found: C, 64.6; H, 10.3. The absence of a hydroxyl group in the soaps was confirmed by the absence of absorption maxima in the region of 3500−3300 cm^{-1} in their IR spectra.

The solutions of chromium soaps were prepared by dissolving a known weight of soap in a benzene−methanol (4:1 v/v) mixture and were kept in a thermostat to maintain the desired temperature (50 ± 0.05 °C).

The density and viscosity of the solvent and soap solutions were measured by using a dilatometer and an Ostwald-type viscometer. The volume of the dilatometer was 15 cm^3, and the accuracy of the density results was ±0.0001. The probable error of the viscosity results was about ±0.3%.

The ultrasonic velocity measurements of the chromium soap solutions were carried out in an Ultrasonic Interferometer (model F-81, Mittal Enterprises, New Delhi) using a crystal of frequency 1 MHz. The uncertainty of the velocity measurements was ±0.2%. The details of the experimental procedures have been described in the previous papers (*26, 27, 30, 31*).

Calculations

The apparent molar volume (ϕ_v), viscosity (η), and various acoustic parameters [viz., adiabatic compressibility (β), intermolecular free length (*14*) (L_f), specific acoustic impedance (*15*) (Z), apparent molar compressibility (ϕ_K), molar sound velocity (R), molar sound compressibility (W), relative association (*16*) (R_A), and solvation number (*17*) (S_n)] were evaluated using the relationships

$$\phi_v = \frac{1000}{c\varrho_o}(\varrho - \varrho_o) + \frac{M}{\varrho_o} \qquad (1)$$

$$\frac{\eta_o}{\eta} = \frac{\varrho_o t_o}{\varrho t} \qquad (2)$$

$$\beta = v^{-2}\varrho^{-1} \qquad (3)$$

$$L_f = \{\beta/K\}^{1/2} \qquad (4)$$

$$Z = v\varrho \qquad (5)$$

$$\phi_K = \frac{1000}{c\varrho_o}(\beta\varrho_o - \varrho\beta_o) + \frac{M\beta_o}{\varrho_o} \qquad (6)$$

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Table 1. Density, Apparent Molar Volume, and Viscosity of Chromium Soaps in a Benzene–Methanol (4:1 v/v) Mixture at 50 ± 0.05 °C

$c \times 10^3$/ (mol·dm^{-3})	$\varrho/(\text{kg·m}^{-3})$			$\phi_v \times 10^3$ $(\text{dm}^3\text{·mol}^{-1})$			$\eta \times 10^3/(\text{Pa·s})$		
	myristate	palmitate	stearate	myristate	palmitate	stearate	myristate	palmitate	stearate
1.0	0.843 72	0.843 81	0.843 89	954	1162	1358	0.4281	0.4290	0.4296
2.0	0.843 81	0.843 92	0.844 03	966	1132	1298	0.4289	0.4307	0.4320
3.0	0.843 89	0.844 08	0.844 14	966	1142	1266	0.4298	0.4327	0.4347
4.0	0.843 91	0.844 13	0.844 31	948	1114	1268	0.4307	0.4348	0.4366
5.0	0.844 02	0.844 32	0.844 50	959	1131	1274	0.4323	0.4370	0.4405
6.0	0.844 10	0.844 49	0.944 58	960	1138	1276	0.4335	0.4399	0.4423
7.0	0.844 22	0.844 65	0.844 89	968	1141	1283	0.4374	0.4315	0.4456
8.0	0.844 35	0.844 76	0.845 15	975	1136	1295	0.4398	0.4440	0.4497
9.0	0.844 43	0.845 03	0.845 29	974	1154	1289	0.4431	0.4473	0.4538
10.0	0.844 65	0.845 17	0.845 42	990	1152	1282	0.4472	0.4500	0.4579

$$R = (\bar{M}/\varrho)v^{1/3} \tag{7}$$

$$\bar{M} = \frac{n_o M_o + nM}{n_o + n}$$

$$W = (\bar{M}/\varrho)\beta^{-1/7} \tag{8}$$

$$R_A = \left(\frac{\varrho}{\varrho_o}\right)\left(\frac{v_o}{v}\right)^{1/3} \tag{9}$$

$$S_n = \frac{n_o}{n}\left(1 - \frac{\bar{V}\beta}{n_o \bar{V}_o \beta_o}\right) \tag{10}$$

where K is Jacobson's constant.

Results and Discussion

Molar Volume and Density. The apparent molar volume, ϕ_v, of the solutions of chromium soaps in a mixture of benzene and methanol (4:1 v/v) decreases with increasing soap concentration in dilute solutions but increases at higher soap concentrations (Table 1). The calculated (eq 1) values of the apparent molar volume, ϕ_v, for dilute solutions below the critical micelle concentration were found to vary linearly with the square root of the soap concentration in conformity with Masson's equation (*18*):

$$\phi_v = \phi_v{}^\circ + S_v c^{1/2} \tag{11}$$

where $\phi_v{}^\circ$ is limiting apparent molar volume and S_v is a constant. The values of $\phi_v{}^\circ$ (0.976, 1.210, and 1.472 $\text{dm}^3\text{·mol}^{-1}$ for myristate, palmitate, and stearate, respectively) and of S_v (−0.0022, −0.0151, and −0.0374 $\text{mol}^{-3/2}\text{·dm}^{9/2}$ for myristate, palmitate, and stearate, respectively) were evaluated from the intercept and slope of the plots of ϕ_v vs $c^{1/2}$ for dilute solutions below the critical micelle concentration. The difference in the values of $\phi_v{}^\circ$ for chromium soaps of homologous saturated fatty acids (myristate, palmitate, and stearate) is found to be almost constant (0.23–0.26 $\text{dm}^3\text{·mol}^{-1}$). Similar results were also obtained for the chromium soap solutions in a benzene–DMF mixture.

The density results have been explained in terms of Root's equation:

$$\varrho = \varrho_o + Ac - Bc^{3/2} \tag{12}$$

where ϱ and ϱ_o are the densities of the soap solution and solvent, respectively. The constants A and B refer to the soap–solvent and soap–soap interactions, respectively. The values of constants A (0.093, 0.198, and 0.337 for myristate, palmitate, and stearate, respectively) and B (−0.73, −1.23, and −3.23 for myristate, palmitate, and stearate, respectively) were obtained from the intercept and slope of plots of $[(\varrho - \varrho_o)/c]$ vs $c^{1/2}$ for dilute solutions below the critical micelle concentration.

The results confirm that the soap–solvent interactions are stronger than soap–soap interactions in dilute solutions of the soap. The results are in agreement with those obtained from Masson's equation (*18*).

Viscosity. The viscosity, η, and specific viscosity, η_{SP}, of the solutions of chromium soaps in a benzene–methanol (4:1 v/v) mixture increase with increasing soap concentration (Table 1) which may be due to an increasing tendency of the soap to form aggregates with an increase in the soap concentration. The plots of viscosity and specific viscosity vs soap concentration are characterized by an intersection of two straight lines at a concentration which corresponds to the critical micelle concentration of the soap (myristate, 0.0059; palmitate, 0.0054; stearate, 0.0049 mol·dm^{-3}), indicating a marked change in the aggregation of soap molecules at the critical micelle concentration. The results show that the critical micelle concentration increases with decreasing chain length of the soap molecules. The plots of viscosity vs soap concentration below the critical micelle concentration have been extrapolated to zero soap concentration, and the extrapolated values of η_o [(0.4270–0.4271) × 10^{-3} Pa·s] are in agreement with the viscosity of the solvent mixture (0.4271 × 10^{-3} Pa·s).

The results of viscosity have been explained on the basis of the following equations:

Einstein (*19*): $$\eta_{SP} = 2.5\bar{V}c \tag{13}$$

Vand (*20*): $$1/c = \left[\frac{0.921}{\bar{V}}\right]^{-1} \frac{1}{\log(\eta/\eta_o)} + \phi\bar{V} \tag{14}$$

Moulik (*21*): $$(\eta/\eta_o)^2 = M + Kc^2 \tag{15}$$

Jones–Dole (*22*): $$(\eta_{SP}/c^{1/2}) = A + Bc^{1/2} \tag{16}$$

where $\bar{V}$, c, ϕ, η, η_o, and η_{SP} are the molar volume ($\text{dm}^3\text{·mol}^{-1}$), concentration ($\text{mol·dm}^{-3}$), interaction coefficient, viscosity of solution, viscosity of solvent, and specific viscosity, respectively. M and K are Moulik's constants, and the constants A and B of Jones–Dole's equation refer to soap–soap and soap–solvent interactions, respectively.

The plots of specific viscosity vs soap concentration (Figure 1) are linear below the critical micelle concentration with the intercept almost equal to zero which shows that Einstein's equation is applicable to dilute solutions of soaps. The values of the molar volume of the soap, $\bar{V}$ (Table 2), have been obtained from the slope of plots of η_{SP} vs c below the critical micelle concentration (Figure 1). The values of the molar volume, $\bar{V}$ (Table 2), were also obtained from the slope of Vand's plots [$1/c$ vs $1/\log(\eta/\eta_o)$] and were found to be in close agreement with those obtained from Einstein's plots. The interaction coefficient, ϕ, obtained from

Table 2. Viscosity Parameters of Chromium Soaps

name of soap	$\bar{V}$ (dm^3·mol^{-1}) Einstein's equation	$\bar{V}$ (dm^3·mol^{-1}) Vands' equation	ϕ	Jones–Dole's equation constants A	Jones–Dole's equation constants B	Moulik's equation constants M	Moulik's equation constants K
myristate	1.27	0.84	+65.40	+0.020	+1.83	1.004	886
palmitate	1.70	1.73	+5.80	−0.015	+4.72	1.007	1650
stearate	2.19	2.21	−2.26	−0.030	+6.33	1.009	2667

Table 3. Ultrasonic Velocity (v), Adiabatic Compressibility (β), and Solvation Number (S_n) of Chromium Soaps in a Benzene–Methanol (4:1 v/v) Mixture

	myristate			palmitate			stearate		
$c \times 10^3$/(mol·dm^{-3})	v (m·s^{-1})	$\beta \times 10^{10}$/(m^2·N^{-1})	S_n	v (m·s^{-1})	$\beta \times 10^{10}$/(m^2·N^{-1})	S_n	v (m·s^{-1})	$\beta \times 10^{10}$/(m^2·N^{-1})	S_n
1.0	1022.1	11.345	44	1022.4	11.335	56	1023.7	11.307	90
2.0	1023.8	11.306	45	1024.4	11.292	54	1027.1	11.231	90
3.0	1024.7	11.285	39	1026.7	11.239	57	1029.5	11.177	82
4.0	1026.6	11.243	42	1029.6	11.175	62	1032.5	11.110	81
5.0	1028.3	11.205	42	1031.3	11.136	59	1035.6	11.041	82
6.0	1029.6	11.175	41	1034.7	11.060	64	1040.4	10.937	89
7.0	1033.1	11.098	49	1038.9	10.969	71	1044.8	10.843	92
8.0	1035.8	11.039	51	1043.0	10.882	75	1050.3	10.726	98
9.0	1038.9	10.972	55	1048.1	10.773	81	1053.2	10.665	96
10.0	1041.5	10.914	56	1050.8	10.715	80	1058.5	10.557	99

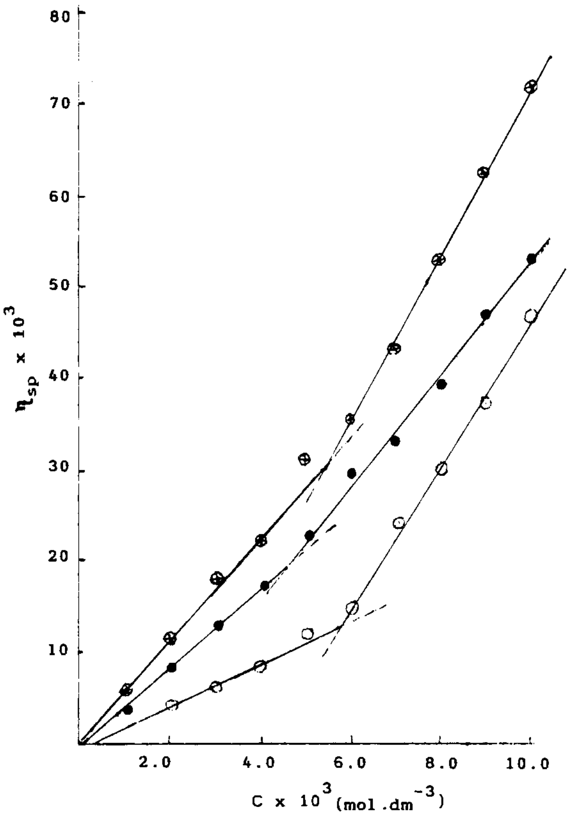

Figure 1. Einstein's plots for myristate (⊙), palmitate (●), and stearate (⊗).

the intercept of Vand's plots were found to decrease with increasing chain length of the soap (Table 2).

The values of constants A and B of Jones–Dole's equation have been obtained from the linear plot of η_{SP}/c vs c below the critical micelle concentration (Table 2). The negative values of constant A confirm that there is no soap–soap interaction in dilute solutions.

The plots of $(\eta/\eta_o)^2$ vs c^2 are linear which shows that Moulik's equation is applicable for these soap solutions. The values of constants M and K increase with increasing chain length of the fatty acid constituent of the soap molecules.

Ultrasonic Measurements. The ultrasonic velocity of the solutions of chromium soaps increase with increasing concentration and chain length of the soaps (Table 3). The variation of velocity, v, with concentration, c, in solutions depends on the concentration derivative of density and compressibility:

$$\frac{dv}{dc} = -\frac{v}{2}\left[\frac{1}{\varrho}\left(\frac{\partial\varrho}{\partial c}\right) + \frac{1}{\beta}\left(\frac{\partial\beta}{\partial c}\right)\right] \qquad (17)$$

The results (Tables 1 and 3) indicate that the density increases while adiabatic compressibility decreases with increasing soap concentration, and so the quantity $\partial\varrho/\partial c$ is positive while $\partial\beta/\partial c$ is negative. Since the values of $1/\beta(\partial\beta/\partial c)$ are larger than $1/\varrho(\partial\varrho/\partial c)$ for soap solutions, the concentration derivative of velocity is positive which is in agreement with the results of other researchers (*23*, *24*) reported for electrolytic solutions.

The variation of ultrasonic velocity, v, with concentration, c, follows the relationship

$$v = v_o + Gc \qquad (18)$$

where v_o is the utlrasonic velocity in the solvent and G is Garnsey's constant (*25*).

The plots of ultrasonic velocity, v, vs soap concentration, c (Figure 2), for solutions of chromium soaps are characterized by an intersection of two straight lines at the concentration which corresponds to the critical micelle concentration of the soaps (myristate, 0.0060; palmitate, 0.0054; stearate, 0.0050 mol·dm^{-3}). The values of the intercept of the plots give the ultrasonic velocity of the solvent, v_o, which is found to be in close agreement with the experimental values (Table 4). Garnsey's constant, G (Table 4), increases with the chain length of the fatty acid constituent of the soap molecule (*26*, *27*).

The adiabatic compressibility, β, of chromium soap solutions decreases with an increase in concentration (Table 3) and the chain length of the soap. This may be due to the fact that these soaps are considerably ionized into metal cations, Cr^{3+}, and fatty acid anions ($C_{13}H_{27}COO^-$, $C_{15}H_{31}COO^-$, and $C_{17}H_{35}COO^-$ for myristate, palmitate, and stearate, respectively). The ions in solutions are surrounded by a layer of solvent molecules firmly bound and oriented toward the ions. The orientation of solvent molecules around the ions is attributed to the influence of the electrostatic field of ions, and the resultant increase

Table 4. Ultrasonic Parameters of Chromium Soaps

				Bachem's constants					
name of soap	v_0 (m·s^{-1})	$\beta_0 \times 10^{10}$/ (m^2·N^{-1})	G/(mol^{-1}·s^{-1}·dm^4)	$A \times 10^{10}$/ (m^2·N^{-1}·dm^3·mol^{-1})	$B \times 10^{10}$/ (m^2·N^{-1}·dm$^{9/2}$·mol$^{-3/2}$)	$\phi_K{}^\circ \times 10^7$/ (kg^{-1}·m^5·N^{-1}·mol^{-1})	$S_K \times 10^6$/ (mol$^{-3/2}$·dm$^{13/2}$·N^{-1}·kg^{-1})	$Z_0 \times 10^{-5}$/ (kg·m^{-2}·s^{-1})	$L_f{}^\circ$/Å
myristate	1020.7	11.380	1500	−39.4	65	−39.4	5.3	8.611	27.40
palmitate	1020.2	11.385	2174	−51.2	132	−54.6	17.0	8.608	27.40
stearate	1020.7	11.378	3000	−81.9	218	−86.1	25.0	8.611	27.39
experimental	1020.5	11.382						8.609	27.42

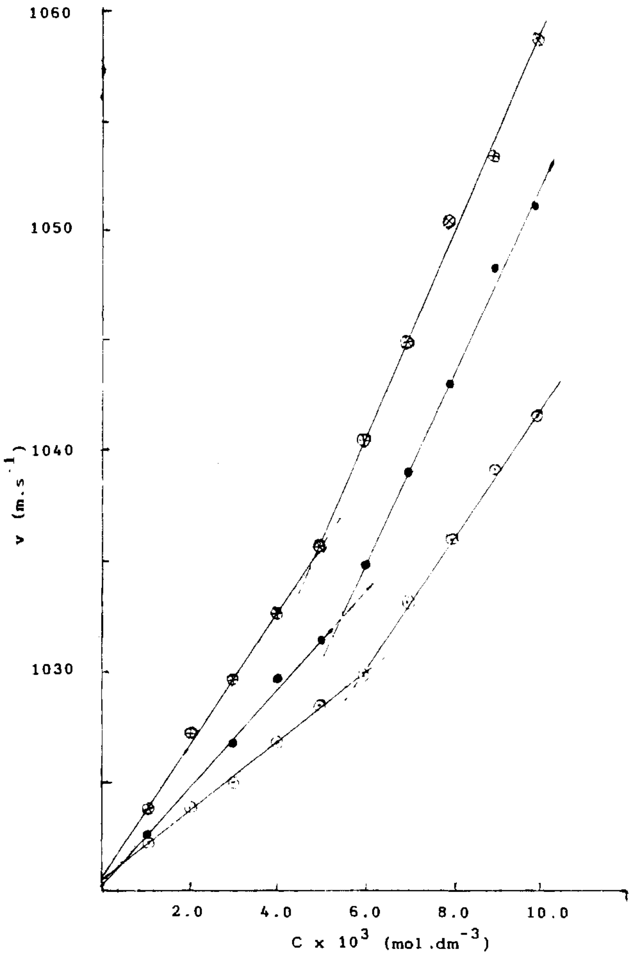

Figure 2. Ultrasonic velocity vs soap concentration for myristate (⊙), palmitate (●), and stearate (⊗).

in internal pressure lowers the compressibility of the solutions; i.e., the ions become harder to compress (*28*).

The adiabatic compressibility, β, of the solutions of chromium soaps is found to obey Bachem's (*29*) relationship:

$$\beta = \beta_0 + Ac + Bc^{3/2} \tag{19}$$

where A and B are constants and c is the concentration of the soap solutions. The constants A and B have been determined from the intercept and slope of plots of $(\beta - \beta_0)/c$ vs $c^{1/2}$ (Table 4). The values of constant A decrease while those of constant B increase with increasing chain length of the fatty acid constituent of the soap molecules (*30*).

It follows from Debye–Huckel's theory that the apparent molar compressibility, ϕ_K, is related to concentration, c, by the relationship

$$\phi_K = \phi_K{}^\circ + S_K c^{1/2} \tag{20}$$

where $\phi_K{}^\circ$ is the limiting apparent molar compressibility and S_K is a constant.

The values of ϕ_K increases in dilute solutions but show a decrease at higher soap concentrations. The decrease in the values of ϕ_K at higher soap concentration may be explained on the basis of close packing of ionic head groups in the micelles, resulting in an increase in ionic repulsion and finally internal pressure. The values of the constant S_K and limiting apparent molar compressibility, $\phi_K{}^\circ$, have been obtained from the slope and intercept of a plot of ϕ_K vs $c^{1/2}$ (Table 4) for dilute solutions. The values of $\phi_K{}^\circ$ are negative and decrease with an increase in the chain length of soap molecules (*31*).

The decrease of the intermolecular free length, L_f, and increase of the specific acoustic impedance, Z, with an increase in the concentration and chain length of the soap can be explained on the basis of lyophobic interactions between the soap and solvent molecules which increase with the intermolecular distance, leaving relatively wider gaps between the molecules and thus becoming the main cause of impediment to the propagation of ultrasound waves. Both the plots L_f vs c and Z vs c show breaks at a definite soap concentration which corresponds to the critical micelle concentration of the soap.

The values of the relative association, R_A, decrease with the concentration and chain length of soap. The plots of R_A vs soap concentration show a break at the critical micelle concentration of the soap. The decrease in the values of the relative association, R_A, is attributed to the fact that the solvation of ions decreases with increasing soap concentration. The values of the molar sound velocity and molar sound compressibility increase with increasing concentration and chain length of the soap.

The values of the solvation number for chromium soaps increase with the chain length of the soap molecule. The higher values of the solvation number have also been reported in the literature (*27, 31*).

The ultrasonic measurements confirm that there is a significant interaction between the soap and solvent molecules in dilute solutions and the values of the critical micelle concentration decrease with increasing chain length of the soap molecule and are found to be in close agreement with those obtained from viscosity measurements.

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