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Ultrasonic Studies of Cu (II) Soap Derived from Seed Oil of Karanj (*Pongamia pinnata*) in Non-aqueous Binary and Ternary Systems at 298.15 K

Shema Khan[1], Rashmi Sharma[2] and Arun Kumar Sharma[2]
[1]Department of Chemistry, Government P.G. College, Dausa 303303, Rajastan, India
[2]Department of Chemistry, S.P.C. Government College, Ajmer 30500, Rajastan, India
*Corresponding author: (e-mail: sharmaarun423@gmail.com)

Ultrasonic speed was measured in non-aqueous binary and ternary systems containing copper soap derived from karanj oil (newly synthesized natural fungicide) in 100% benzene, 80%, and 60% methanol-benzene mixture. The acoustic behaviour was studied to understand and analyze the micellar features of the solute in a ternary system which might provide valuable information for its applications in different fields. From ultrasonic velocity values, the specific acoustic impedance Z, adiabatic compressibility β, intermolecular free length L_f, apparent molar compressibility ϕ_k, molar sound velocity R, primary solvation number S_n were calculated. The results were fitted to Bachem's and Masson equation. The results were explained by intermolecular interactions and indicated that there was sufficient solute–solvent interaction due to which micellar alignment was considerably affected. The decrease in adiabatic compressibility, β, and intermolecular free length L_f of the Cu (II) karanj soap solution with increasing concentration of soap in 100% benzene 80% benzene–methanol, and 60% benzene–methanol was observed which suggested that the non-polar long chains or lypophilic segment of the molecules in the solution were enveloped by a layer of solvent molecules bound and oriented towards lypophilic end.

Key words: Copper soap; methanol; benzene; karanj oil; ultrasonic; intermolecular interactions

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Properties of surface active solutions are essential to access the thermodynamic, acoustic and transport aspects. The compositional dependence of acoustic properties has proved to be very useful in understanding the nature and extent of the pattern of molecular aggregation resulting from the intermolecular interaction between the components [1–2]. Ultrasonic technique is a powerful means for characterizing the various aspects of physicochemical behaviour of the system and also for studying the interaction between the molecules [3–4]. Some workers [5–6] have discussed the physicochemical aspects of ultrasonic velocity and related parameters on different types of soaps and detergents. The Gruneisen parameter and internal pressure obtained from ultrasonic velocity and density data play a significant role in understanding internal structure, clustering phenomenon and quasi-crystalline nature of binary mixture [7–8]. Ultrasonic measurements have also been used to determine solvation number in aqueous media [9–10]. The present work deals with the determination of ultrasonic velocity measurements which have been used to obtain information regarding various acoustic parameters and solute-solute interactions in pure solvents, solvent mixtures and solutions of Cu (II) soap of non-edible oil of Pongamia pinnata (Karanj) in non-aqueous solvent (benzene and its varying compositions with methanol) to know the effect of polarity on the micellar features of the surfactant. It is anticipated that it will generate new hopes in various industrial and analytical applications. This information is of fundamental importance for understanding solute-solute, solvent-solvent interactions in solutions below and above critical micelle concentration.

EXPERIMENTAL

All the chemicals used were of LR/AR grade. Solutions of the soaps were prepared in hot benzene (Qualigens) due to maximum solubility as compared to other

solvents. The other chemicals used were methanol, KOH, HCl, $CuSO_4$ etc. (Merck) procured from the reputed Indian market. Copper soap was prepared by refluxing the non-edible oil, i.e., *Pongamia pinnata* (Karanj) oil (extracted from kernels and purified) with ethyl alcohol and 2 N KOH solutions for 3–4 hours (Direct metathesis). The neutralization of excess of KOH present was done by slow addition of 0.5N HCl. A saturated solution of copper sulphate was then added to it, for conversion of neutralized potassium soap into their corresponding copper soap. Copper soap so obtained was then washed with warm water and 10% alcohol at 50°C and recrystallized using hot benzene. Molecular weights of copper soaps were determined from saponification value [11]. The copper soap is abbreviated as copper-karanj soap (CK).The underlying principle of the ultrasonic experimental technique used in the measurement of velocity (U) is based on the accurate determination of the wavelength (λ) of the medium. The Ultrasonic waves of known frequency (f) are produced by a quartz plate fixed at the bottom of the cell. Ultrasonic interferometer from Mittal Enterprises, Model M-82 with the accuracy of ± 0.03% was used for the measurements of ultrasonic velocities in various solutions at a fixed frequency 2 MHz. Water maintained at the 298.15 K temperature in a thermostat was passed through the jacket of the cell before the measurement was made. Further, the operative part of the measurement involved the slow movement of micrometer till the anode current meter showed a maximum. Some maxima readings of anode current are passed on, and their number 'n' are counted. The total distance d (1 cm) thus moved by the micrometer gives the value of wavelength λ with the help of the following relation:

$$d = n \times \lambda \tag{1}$$

Once the wavelength is known, the ultrasonic velocity U (m/s) in the solution could be calculated with the help of the following relation:

$$U = \lambda \times f \tag{2}$$

RESULTS AND DISCUSSIONS

Acoustic Parameter and Micellar features of Cu (II) Karanj Soap (CK)

Of late, ultrasonic investigations have established their versatility to furnish information as to the understanding of solute–solvent interactions [12–13] Some researchers have discussed the physicochemical aspects of ultrasonic velocity and related parameters. Ultrasonic measurements of the referred molecule in the ternary system at constant temperature have been used to explore its micellar behaviour regarding its applications in industry.

The specific acoustic impedance Z, adiabatic compressibility β, intermolecular free length L_f apparent molar compressibility ϕ_k molar sound velocity R and primary solvation number S_n have been calculated by using following relationships [14].

$$Z = \cup\rho \tag{3}$$

$$\beta = \frac{1}{\cup^2 \rho} \tag{4}$$

$$L_f = K\sqrt{\beta} \tag{5}$$

$$\emptyset_k = \left[1000\ \frac{\beta\rho_0 - \beta_0\rho}{c\rho_0}\right] + \frac{\beta M}{\rho_0} \tag{6}$$

$$R = M.(\cup)^{1/3}\ \rho_0 \tag{7}$$

$$M = X_1M_1 + X_2M_2 + X_3M_3 \tag{8}$$

$$S_n = \left[\frac{n^0}{n}\right].\left[1 - \frac{V\beta}{n^0V_0\ \beta_0}\right] \tag{9}$$

Where β_0, β, β_0, ρ, n^0, n, V_0 and V are adiabatic compressibility, density, number of moles and molar volume of solvent and soap (solute) solution respectively. K, c and U respectively, are the temperature dependent Jacobson's constant, concentration of the solution and ultrasonic velocity. The expression of molar volume of the solvent $\bar{V}_0$ and soap solution $\bar{V}$ could be written as:

$$V = X_1M_1 + X_2M_2 + X_3M_3/\rho \quad (10)$$

$$V_0 = X_1M_1 + X_2M_2 + X_3M_3/\rho_0 \quad (11)$$

In Equations 6, 8 and 9 M represents the molecular weight and X the mole fraction of the component. Here the 1 refers to solute and 2, 3 refer to benzene and methanol solvent respectively.

The values of U and allied parameters for CK soap in pure benzene (CK_{100}), 80% benzene–methanol (CK_{80}) and 60% benzene–methanol (CK_{60}) system are recorded in Tables 1–3. The measured values of density compared with literature values. The possible error in the density was estimated to be less than ± 1. The density ρ was reproducible to within ± 0.020 kg m^{-3}. Smilarly the mistake in ultrasonic velocity was ± 0.020 m/s.

The perusal of the data suggests that, in general the values of L_f, R, β and ϕ_k decrease then increase with increasing concentration of the soap till a particular concentration corresponding to critical micelle concentration (CMC) whereas Z value increases than decreases till a particular concentration and then increases again with the increase in soap concentration. The values of solvation number S_n increases consistently with increase in CK soap concentration [15].

Table 1. Acoustic parameters of Cu (II) karanj soap solutions in 100% benzene (at 298.15 K).

C (mol l^{-1})	ρ (g.cm^{-3})	U (m/s)	$Z\times10^{-5}$ (g/cm^2.s)	$\beta\times10^{11}$ (cm^2/dyn)	L_f (Aº)	$\phi_\kappa\times10^{10}$	R	$S_n\times10^{-3}$
0.0002	0.8754	1379	1.2074	6.0047	0.4890	−5716.16	4611.108	−143.641
0.0003	0.8760	1379	1.2083	6.0002	0.4888	−6675.33	4607.951	−95.569
0.0004	0.8766	1379	1.2095	5.9959	0.4886	−7050.36	4604.956	−71.541
0.0005	0.8763	1379	1.2087	5.9983	0.4887	−4586.70	4607.112	−57.302
0.0006	0.8750	1379	1.2068	6.0074	0.4891	−701.02	4614.433	−47.958
0.0007	0.8755	1379	1.2076	6.0038	0.4889	−1574.12	4611.956	−41.042
0.0008	0.8762	1379	1.2085	5.9993	0.4887	−2451.38	4608.799	−35.840
0.0009	0.8764	1379	1.2089	5.9974	0.4887	−2538.67	4607.696	−31.833
0.0010	0.8771	1379	1.2099	5.9928	0.4885	−3170.48	4604.440	−28.591
0.0012	0.8780	1379	1.2111	5.9864	0.4882	−3625.88	4600.195	−23.7600
0.0014	0.8782	1379	1.2113	5.9851	0.4882	−3226.95	4599.832	−20.357
0.0016	0.8783	1379	1.2114	5.9849	0.4882	−2799.41	4600.255	−17.814

Table 2. Acoustic parameters of Cu (II) karanj soap solutions in 80% benzene-methanol (at 298.15 K).

C (mol l^{-1})	ρ ($g.cm^{-3}$)	U (m/s)	$Z\times10^{-5}$ ($g/cm^2.s$)	$\beta\times10^{11}$ (cm^2/dyn)	L_f (Aº)	$\phi_\kappa\times10^{10}$	R	$S_n\times10^{-3}$
0.0002	0.8578	1290	1.1069	7.0016	0.5280	−4166.13	3636.651	−130.234
0.0003	0.8584	1290	1.1075	6.9974	0.5278	−5368.34	3634.752	−86.674
0.0004	0.8589	1290	1.1083	6.9929	0.5277	−6172.15	3632.644	−64.883
0.0005	0.8586	1290	1.1079	6.9953	0.5278	−3847.58	3634.175	−51.968
0.0006	0.8575	1290	1.1064	7.0048	0.5281	43.63	3639.353	−43.491
0.0007	0.8581	1290	1.1072	6.9995	0.5279	−1400.17	3636.858	−37.196
0.0008	0.8584	1290	1.1076	6.9969	0.5278	−1788.38	3635.806	−32.514
0.0009	0.8587	1290	1.1080	6.9946	0.5277	−2053.85	3634.840	−28.874
0.0010	0.8589	1290	1.1083	6.9929	0.5277	−2135.56	3634.212	−25.970
0.0012	0.8590	1290	1.1083	6.9925	0.5277	−1741.43	3634.566	−21.644
0.0014	0.8590	1290	1.1083	6.9924	0.5276	−1436.60	3635.004	−18.554
0.0016	0.8586	1290	1.1079	6.9953	0.5278	−820.47	3637.051	−16.260

Table 3. Acoustic parameters of Cu (II) karanj soap solutions in 60% benzene-methanol (at 298.15 K).

C (mol l^{-1})	ρ ($g.cm^{-3}$)	U (m/s)	$Z\times10^{-5}$ ($g/cm^2.s$)	$\beta\times10^{11}$ (cm^2/dyn)	L_f (Aº)	$\phi_\kappa\times10^{10}$	R	$S_n\times10^{-3}$
0.0002	0.8441	1176	0.9931	8.5592	0.5838	−4907.12	2942.854	−116.69
0.0003	0.8447	1176	0.9938	8.5529	0.5836	−7166.75	2940.914	−77.608
0.0004	0.8450	1176	0.9941	8.5503	0.5835	−6499.35	2940.229	−58.152
0.0005	0.8445	1176	0.9935	8.5558	0.5837	−2906.39	2942.328	−46.629
0.0006	0.8438	1176	0.9927	8.5629	0.5839	22.74	2944.989	−38.973
0.0007	0.8443	1176	0.9933	8.5575	0.5837	−1394.01	2943.360	−33.338
0.0008	0.8444	1176	0.9934	8.5562	0.5837	−1457.9	2943.127	−29.159
0.0009	0.8445	1176	0.9936	8.5550	0.5836	−1485.37	2942.928	−25.910
0.0010	0.8447	1176	0.9938	8.5529	0.5836	−1666.95	2942.451	−23.303
0.0012	0.8448	1176	0.9939	8.5522	0.5835	−1390.51	2942.646	−19.419
0.0014	0.8448	1176	0.9938	8.5527	0.5836	−1022.02	2943.260	−16.652
0.0016	0.8456	1176	0.9949	8.5438	0.5833	−1904.88	2940.635	−14.523

The increase in the values of specific acoustic impedance Z with the concentration can be explained by lyophobic interactions between the solute and solvent molecules, which increases the intermolecular distance leaving relatively wider gaps between the molecules and becoming the main cause of impedance in the propagation of ultrasound waves. Also, it is suggested that the decrease in the intermolecular free length L_f with the increase in the concentration of the soap indicated that there is significant interaction between solute and solvent molecules and that the structural arrangement is considerably affected [16].

The plots U versus c shows a parallel line concerning X-axis suggesting that the ultrasonic velocity (U) of very dilute solutions of Cu (II) karanj soap in 100% benzene, 80% benzene–methanol and 60% benzene–methanol system remains the same on increasing the concentration of CK soap. The difference in the ultrasonic velocity (U) values of soap solutions in varying composition of benzene, i.e. CK_{100},CK_{80} and CK_{60} is seen which is mainly due to the difference in the 'U' of the solvents only.

The plots of Z versus c, β versus c and L_f versus c are all characterized by an intersection of two curvatures at a definite concentration (Figures 1–3) corresponding to the CMC of CK_{100} CK_{80} and CK_{60}. At CMC, the hydrocarbon chain structure of the soap molecules derived from karanj oil, allow extensive contact between adjacent chain, possibly accompanied by the change in the vibrational and rotational degree of freedom of methylene groups of fatty acids of varying composition present in the karanj oil [17]. The values of CMC of these plots are recorded in Table 4.

The decrease in adiabatic compressibility β and intermolecular free length L_f of the Cu (II) karanj soap solution with increasing concentration of soap in pure benzene 80% benzene–methanol and 60% benzene–methanol might be interpreted on the basis of the fact that the non-polar long chains or lypophilic segment of the molecule in the solution were enveloped by a layer of solvent molecules bound and oriented towards lypophilic end. The orientation of the solvent molecules around the solutes might be due to the influence of electrostatic fields of solutes and resulted in the increase in the internal pressure and in lowering the compressibility of the solution, i.e. the solution becomes harder to compress. This indicates that there is sufficient solute–solvent interaction due to which micellar alignment is considerably affected [18].

Literature survey [19–20] also revealed that the decrease in the values of β and L_f indicates that there is a significant interaction between solute–solvent molecules suggesting a consistent change in the lucid micellar orientation in such systems.

Various scientists [21–22] have suggested that the correlation between solvation number S_n and molar concentration of the solute in the solvent can be understood assuming that solvent molecules in the immediate vicinity of solute molecules have modified properties, similar to those of pure solvent under high pressure. Thus solvation about a solute is conceived as the sphere of bound solvent at high pressure and virtually incompressible at the boundary of which compressibility falls to zero. A close perusal of Table 1 leads us to see a persistent rise in solvation property of the solute with an increase in concentration. This might be attributed to a greater number of solvent molecules forming solvation shells around the solute in the non-polar segment of the molecule in CK_{100} and it was around the whole molecule (i.e. polar and non-polar segment) in CK_{80} and CK_{60}. Including therefore it was observed from the data that, in general, the numerical values of S_n increased in all the three systems and follows the order: (Figure 5)

S_n values: $CK_{100}<CK_{80}<CK_{60}$.

The adiabatic compressibility β of CK soap solutions, i.e. CK_{100}, CK_{80} and CK_{60} system used is found to obey Bachem's relationships [23].

$$\beta = \beta_0 + Ac + Bc^{3/2} \qquad (12)$$

$\beta =$ On re-arranging, we get :

$$\frac{(\beta - \beta_0)}{c} = A + B\sqrt{c} \qquad (13)$$

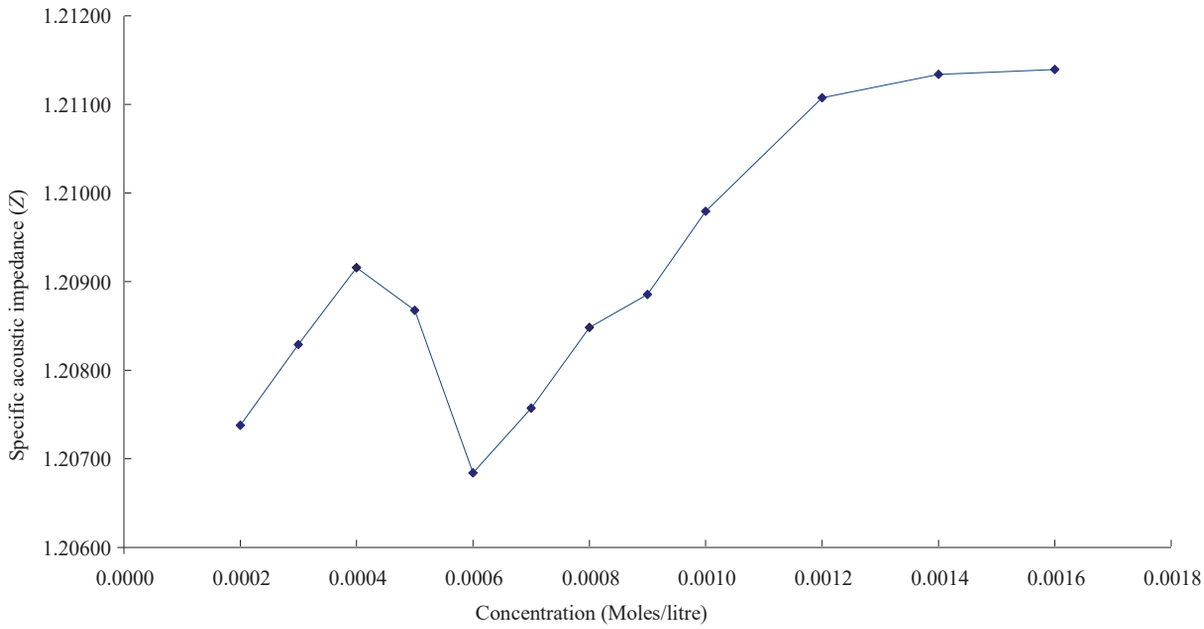

Figure 1. Plot of specific acoustic impedance *versus* concentration for Cu (II) karanj soap solutions in pure benzene.

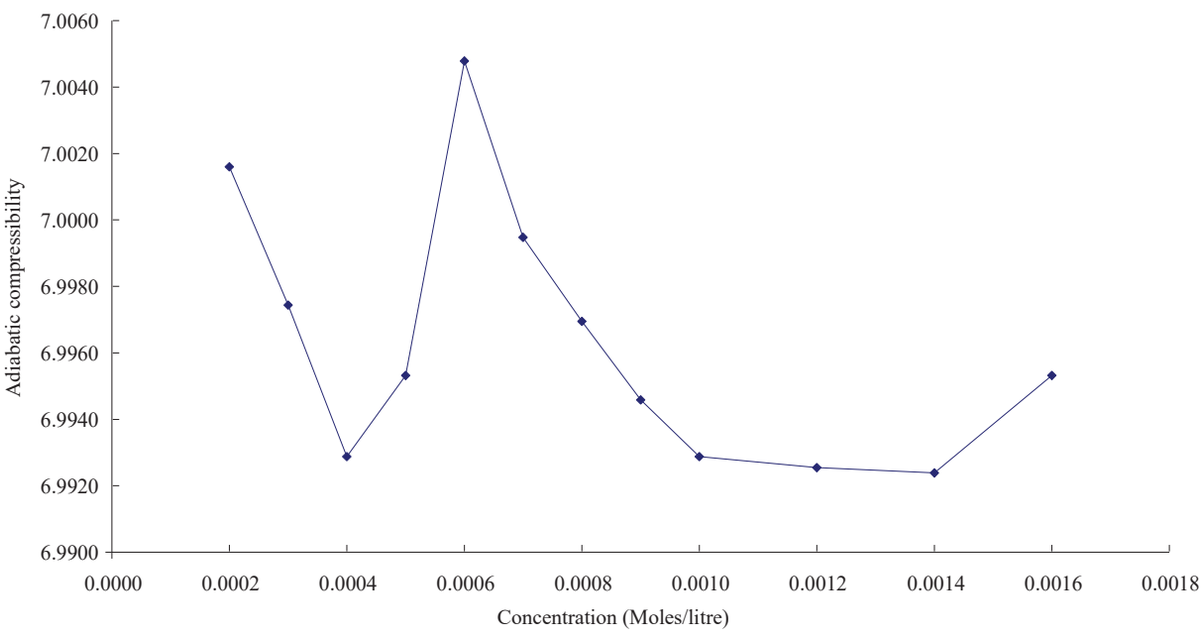

Figure 2. Plot of adiabatic compressibility *versus* concentration for Cu (II) karanj soap solutions in 80% benzene-methanol.

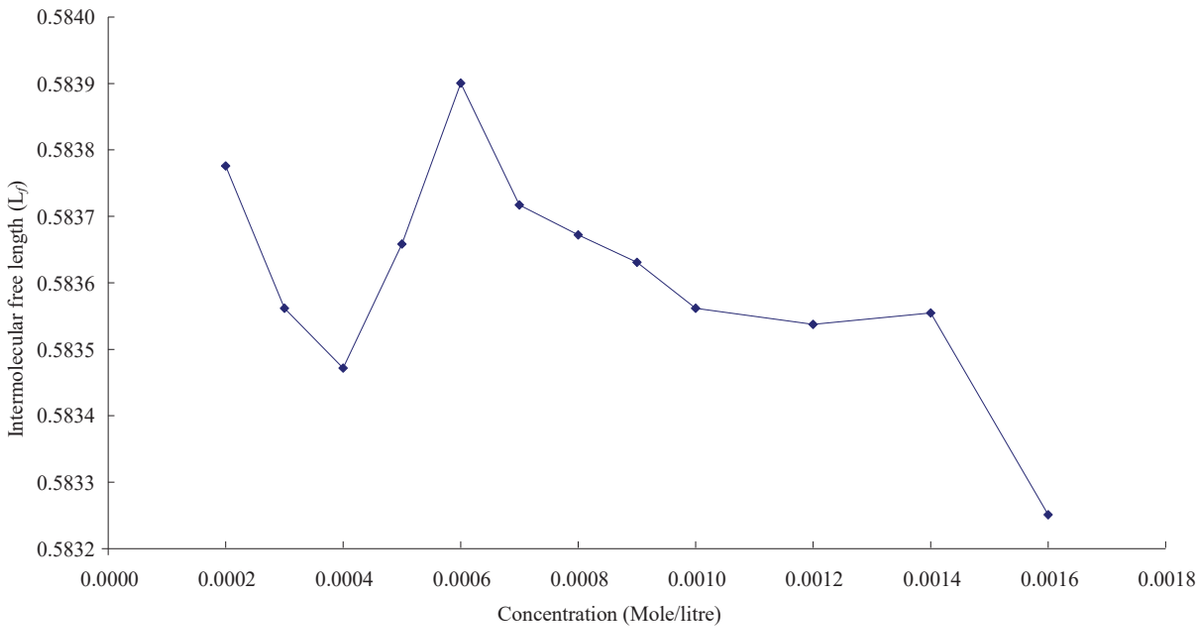

Figure 3. Plot of intermolecular free length *versus* concentration for Cu(II) karanj soap solutions in 60% benzene-methanol.

Table 4. Value of CMC obtained from various acoustic parameters for Cu (II) karanj soap solutions in 100% benzene, 80% benzene-methanol and 60% benzene-methanol system at 298.15 K.

Graph plotted	CK_{100}	CK_{80}	CK_{60}
Z versus c	0.00061	0.00057	0.00054
B versus c	0.00061	0.00057	0.00054
L_f *versus c*	0.00061	0.00057	0.00054
ϕ_k *versus* $\sqrt{c}$	0.00061	0.00057	0.00054
$(\beta-\beta_0)/c$ *versus* $\sqrt{c}$	0.00061	0.00057	0.00054

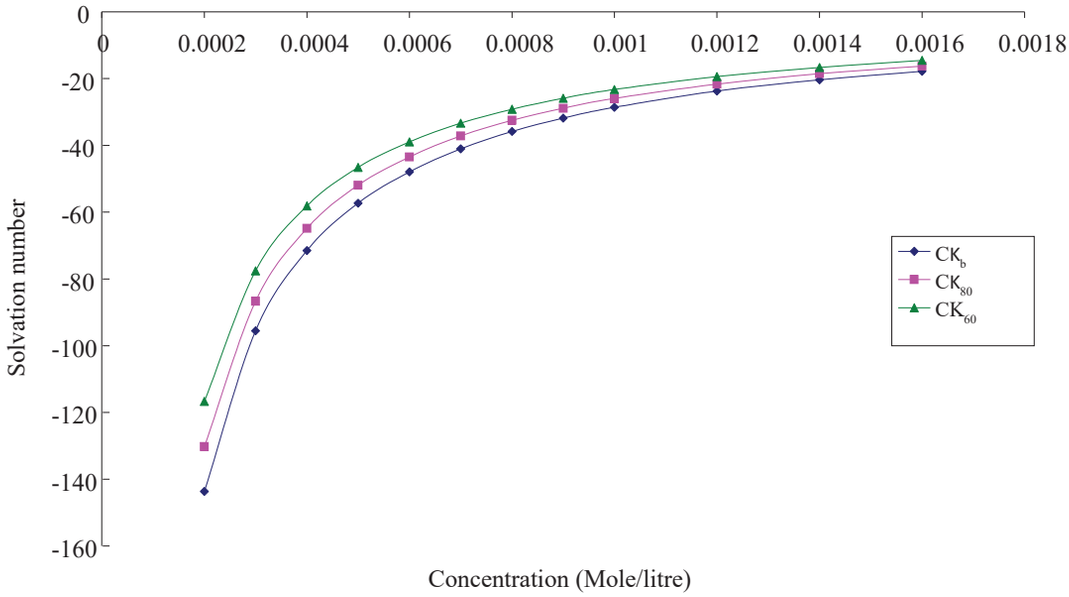

Figure 5. Plot of solvation number for Cu (II) karanj soap in pure benzene, 80% benzene-methanol and 60% benzene-methanol system.

The values of constants 'A' and 'B' were evaluated from the intercept and slope of the plots $(\beta-\beta_0)$/ c *versus* √c.

Unlike other plots of ϕ_k, R, β_0 and L_f *versus* c the plot of $(\beta-\beta_0)$/c *versus* √c was characterized by the intersection of possible two straight lines of two curves at CMC of the soap solution (Figure 4). Thus the values 'A' and 'B' were evaluated both below and above CMC and were designated A_1, A_2 and B_1, B_2, respectively. The values of A1 A2 and B1, B2 follow the order:

$$A_1 < A_2 \text{ and } B_1 > B_2.$$

The value of apparent molar compressibility ϕ_k has also been evaluated in terms of Masson's equation [24].

$$\phi_k = \phi_k^0 + S_k \sqrt{c} \qquad (14)$$

Here, ϕ_k^0 is the limiting apparent molar compressibility and is a measure of solute–solvent interactions. S_K is the experimental slope, a measure of solute–solute interactions. The plot of ϕ_k *versus* √c is characterized by an intersection of two straight line thus Masson's equation fits well both below an above CMC (Figure 6). Correspondingly, two values of both ϕ_k^0 and S_k are obtained, i.e. below and above CMC and designated as ϕ_{k1}^0 and ϕ_{k2}^0 and S_{k1}, S_{k2}. A close study of Table 4–5 reveals that negative values of ϕ_{k1}^0 and positive value of ϕ_{k2}^0 are obtained. The orders of the two parameters are observed to be as follows:

$$\phi_{k1}^0 < \phi_{k2}^0 \text{ and } S_{k1} > S_{k2}$$

Literature survey [25] revealed that negative values of ϕ_{k1}^0 indicate electrostriction and hydrophobic interaction. The negative value of ϕ_{k1}^0 is also attributed to the loss of structural compressibility of solute molecules due to the increased population of hydrophobic non-polar solvent benzene. It shows that structural disruption is much in evidence in solution.

Since $\phi_{k1}^0 < \phi_{k2}^0$ thus it might be suggested that solute–solvent interaction was more prominent above CMC as compared to below CMC. It might also be suggested that above CMC, hydrophobic–hydrophobic group interactions, between the long alkyl chain fatty acids of CK soap derived from karanj oil and non-polar solvent benzene were more dominant as compared to below CMC in CK_{100} system and in CK_{80} and CK_{60} system the hydrophilic-hydrophilic interaction between COO^- and polar solvent also became prominent.

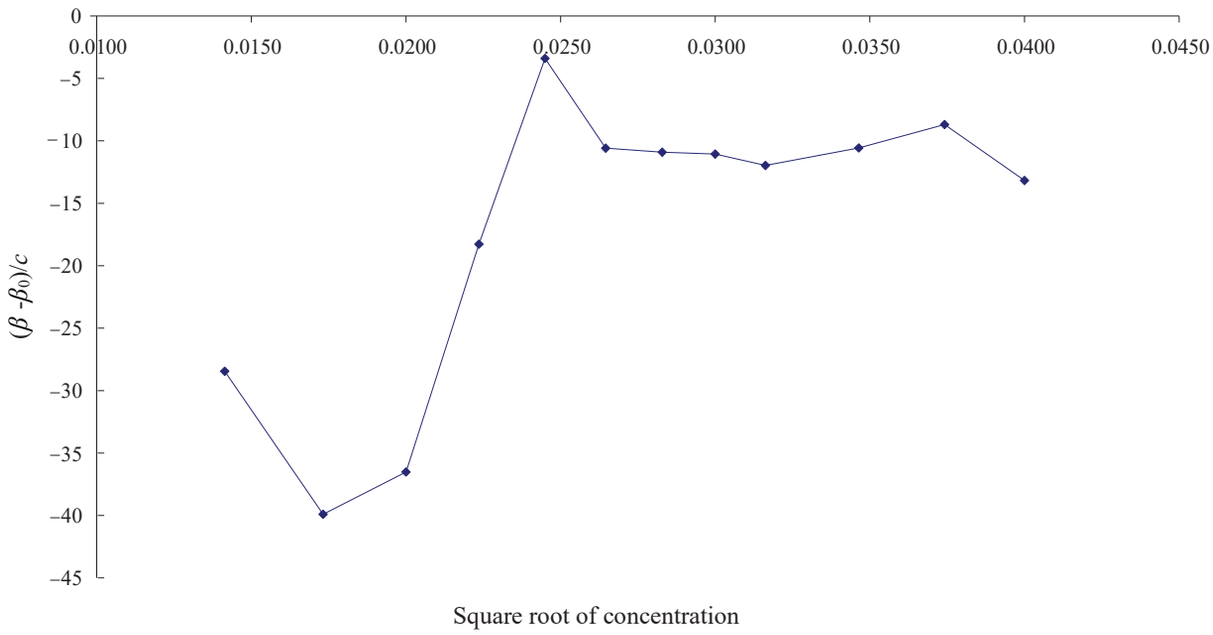

Figure 4. Plot for Bachem's relation for Cu (II) karanj soap solutions in 60% benzene-methanol.

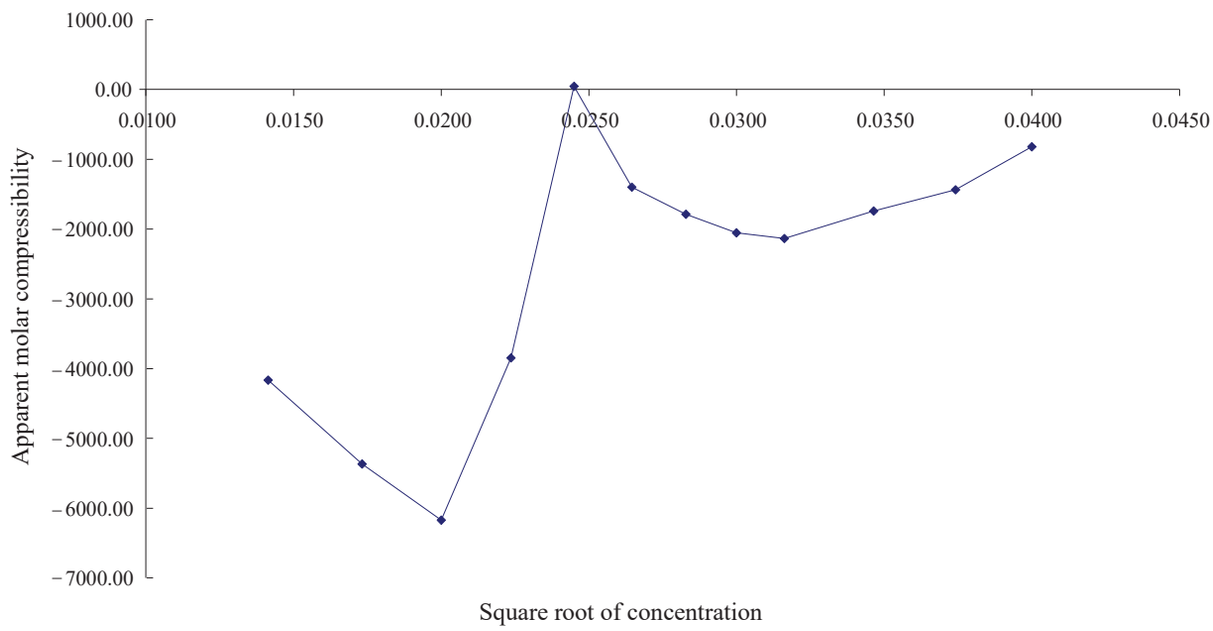

Figure 6. Plot of apparent molar compressibility *versus* square root of concentration for cu (II) karanj soap solutions in 80% benzene-methanol.

Table 5. Computed parameters for Bachem's relationship and Masson's equation for Cu (II) karanj soap solutions in 100% benzene, 80% benzene-methanol and 60% benzene-methanol system at 298.15 K.

Soap	Bachem's relationship				Masson's equation			
	A_1 X 10^7	A_2X 10^7	B_1	B_2	$\phi_{k1}{}^0$X 10^5	$\phi_k 2^0$ X 10^5	S_{k1}	S_{k2}
CK_{100}	– 179.32	20.38	7020.26	– 43696.71	– 35447.07	10619.08	1406062.93	– 461702.39
CK_{80}	– 172.72	14.61	6868.88	– 923.93	– 33986.10	3473.43	1375834.29	– 184828.41
CK_{60}	– 126.32	– 130.65	4945.77	– 7.16	– 35491.37	– 714.48	1452251.44	– 25888.25

CONCLUSION

The non-linear variation of ultrasonic velocity and acoustical parameters with the molar concentration of copper soaps derived from karanj oil in pure benzene and its mixture of 80% and 60% ratio with methanol, showed that there was sufficient intermolecular interaction between solute and solvent molecules. The decrease in adiabatic compressibility β indicated that the presence of solute–solvent interaction due to which micellar alignment was considerably affected. The decrease in the intermolecular free length L_f with the increase in the concentration of the soap indicated that there was significant interaction between solute and solvent molecules and that the structural arrangement was considerably affected. The types of micellar structures formed need to be studied further. The acoustic behaviour of newly synthesized natural fungicide was studied to understand and analyze the micellar features of the solute in ternary system which might provide valuable information for its applications in different fields.

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