

Volumetric and Viscometric Studies of Copper Surfactant Derived from Karanj (*Pongamia pinnata*) Oil in Methanol-benzene Mixture at 298.15 K

Shema Khan,¹ Rashmi Sharma² and Arun Kumar Sharma^{3*}

¹Department of Chemistry, S. R. K. P. Govt. P. G. College, Kishngarh-305801, Rajasthan, India

²Department of Chemistry, Smrat Prathviraj Chauhan Government College, Ajmer-305001, Rajasthan, India

³Department of Chemistry, Government Post Graduate College, Jhalawar-326001, Rajasthan, India

*Corresponding author: sharmaarun423@gmail.com

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ABSTRACT: *The density, molar volume, apparent molar volume, viscosity and other parameters of Cu(II) surfactant derived from karanj oil in non-aqueous solvents of varying compositions have been determined at constant temperature 298.15 K. The results were used to determine the critical micelle concentration (CMC), soap-solvent interactions and the effect of chain length of the surface active molecule on various parameters. The CMC values of copper karanj soap solutions decreased with the increase in the concentration of polar solvent methanol in the solvent mixture. These studies were done to study the solution behaviour, micellar features and various interactions of the derived biologically active surfactant with polar and non-polar solvents to understand the applied part of the molecule. The apparent molar volume has been examined in terms of Masson equation, and the limiting apparent molar volume has been interpreted in terms of solute-solvent interaction. The detailed study of aforesaid compounds clearly indicates that the solute-solvent interaction decreases with the increase in ring strain and size of the synthesised complexes. The conclusions with regard to solute-solute and solute-solvent interaction have been discussed in terms of well-known Moulík's and Jones-Dole equations. This vital information plays an important role in the selection of the synthesised molecule for various industrial and biological applications.*

Keywords: Cu(II) soap, non-edible oils, soap-solvent interaction, CMC, viscosity

1. INTRODUCTION

Cu(II) surfactants have found various applications in many industrial uses and play an important role in the colloid chemical phenomena. The exact information on the nature and structure of these compounds are of great significance in explaining their characteristics under different conditions. Recently, work on polymetallic complexes and transition metal complexes of heterocyclic ligands has been done, and their structure and biological characteristics have also been discussed.^{1,2} Additionally, researchers studied the molar volume and other parameters of various soaps.^{3,4} Many nitrogen and sulphur containing compounds were synthesised and investigated for a wide range of pharmacological and biological activities.⁵ They have been used as tranquilisers, sedatives, anti-inflammatory agents, antiviral, anti-malarial fungicide, bactericide, etc.⁶ As apparent molar volume of a solute reflects the cumulative effects of solute-solute and solute-solvent interactions, such data are expected to highlight the role of the cation and anion of a weak electrolyte in influencing its apparent molar volumes at infinite dilution on mixed solvent systems.^{7,8} The present work deals with the study of density, viscosity and other parameters derived from various equations of copper soap synthesised from (*Pongamia pinnata*) or karanj oil in a ternary system. The effect of soap concentration on viscosity of the solution in polar and non-polar solvent has been discussed in terms of Jones-Dole equations.⁹

The density parameters are important to understand the colloidal behaviour, critical micelle concentration (CMC) characteristics and nature of the complexes. These studies indirectly help in identifying the structural insight, physical and biochemical properties of these class heterocyclic complexes.¹⁰⁻¹² The studies will definitely help future researchers to apply these novel binuclear complexes in multiple interlinked fields based on their widest applicability.^{13,14} Overall, the present course of study helps us to understand the characteristic nature and application of biological active metal soaps.¹⁵ The study definitely generates a new hope in biological, pharmacological and therapeutic testing.¹⁶ It is anticipated that it will generate a new hope in various industrial and analytical applications.¹⁷ This information is of fundamental importance for understanding solute-solute, solvent-solvent interactions in solutions below and above CMS.¹⁸

2. EXPERIMENTAL

All the chemicals used were of LR/AR grade. Copper soap was prepared by refluxing the non-edible oil, i.e., karanj oil (extracted from kernels and purified) with ethyl alcohol and 2N KOH solutions for 3–4 h (Direct Metathesis).¹⁹ The neutralisation of excess KOH present was done by slow addition of 0.5N HCl.²⁰

Saturated solution of copper sulphate was then added to it to convert neutralised potassium soap into their corresponding copper soap. Copper soap obtained was then washed with warm water and 10% alcohol at 50°C and recrystallised using hot benzene. Molecular weights of copper soaps were determined from saponification value.²¹ The copper soap is abbreviated as copper-karanj (CK). The physical and analytical data given in Table 1.

Table 1: Analytical and physical data of CK soap.

Name of soap	Colour	M.P. (°C)	Metal content		S.V.	S.E.	Av. Mol.wt.
			Observed	Calculated			
CK	Dark green	51	9.4996	9.3426	181.5	309.091	679.682

2.1 Characterisation of CK Soap

In order to study the structure of soap, the infrared absorption spectra of compound was obtained on a Fourier transform infrared (FTIR) spectrophotometer, Shimadzu 821PC (4000–400 cm^{-1}) from analytical instrument facility, CDRI, Lucknow, India. Proton NMR spectra were also recorded at SAIF, CDRI, Lucknow, on NMR spectrometers, Bruker DRX-300 at 300 K using C_6D_6 (deuterated benzene) as solvent for soap.

2.1.1 IR spectral analysis

The absorption bands observed at 2927.6 cm^{-1} and 2855.7 cm^{-1} correspond to asymmetric and symmetric stretching of methylene ($-\text{CH}_2$) group in the IR spectra investigation of pure copper karanj soap, with the overlapping of CH_3 anti-symmetrical C-H (ν_{as}) at 2962 cm^{-1} and symmetrical C-H (ν_{s}) stretching at 2872 cm^{-1} , respectively. The presence of absorption band at 3018.4 cm^{-1} corresponds to olefinic $=\text{C}-\text{H}$ stretch and the un-conjugated $\text{C}=\text{C}$ stretching peaks are very much distinguished at 1655.8 cm^{-1} with two medium bands at 3018.4 cm^{-1} and 1655.8 cm^{-1} , and a strong band at 759.1 cm^{-1} confirms the cis-nature of alkenes. This out of plane C-H bending vibration of alkene is the most characteristic vibrational mode of alkene.²² It is clear from the spectra of CK soap that it usually is the strongest in the spectra of alkenes. The absorption peak at 1419.2 cm^{-1} is representative of symmetric C-H deformation bands of $=\text{C}-\text{H}$ group.

The absorption band near 1377.3 cm^{-1} is very stable in position arising from the symmetric bending of the methyl C-H showing that the methyl group is attached to another carbon atom. The in-plane bending (δ_{s}) scissoring band of C-H of methylene group is positioned at 1462.8 cm^{-1} . This peak may also be assigned to

C-H asymmetrical bending vibration of methyl group as it generally overlaps the scissoring vibrations of methylene group.

The peaks 1609.3 cm^{-1} and 1216.2 cm^{-1} may be assigned to carboxylate ion COO^- , C–O anti-symmetric and symmetric stretching, respectively. Also, a very clear $>\text{C}=\text{O}$ stretching is also observed at 1736 cm^{-1} , while a small peak at 1345 cm^{-1} corresponds to CH_2 twisting and wagging and 669.2 cm^{-1} to C-H rocking.²³ Copper-oxygen (Cu–O) stretching bands have been distinguished at 480 cm^{-1} (Table2).

Table 2: IR spectral data for CK soap.

Absorption bands	CK (cm^{-1})
Olefinic =C–H stretching	3018.4
-CH ₃ and -CH ₂ , -C–H antisymmetric stretching (d_{as})	2927.6
-CH ₃ and -CH ₂ , -C–H symmetric stretching (d_s)	2855.7
$>\text{C}=\text{O}$ stretching	1736.0
-C=C stretching (cis unconjugated)	1655.8
-COO ⁻ , -C–O antisymmetric stretching	1609.3
-CH ₂ , -C–H in plane bending (d_s) (scissoring) and asymmetric vibration of -C–H (-CH ₃) overlapping the former	1462.8 and ~1445
-C–H def. =C–H (rocking)	1419.2
-CH ₃ , -C–H symmetric bending (d_s)	1377.3
-CH ₂ , -C–H bending (δ twisting and wagging)	1345
-COO ⁻ , -CC(=O)-O symmetric stretching	1216.2
=C–H, out of plane bending of -C–H (strong)	759.1
-CH ₂ , -C–H rocking	669.2
Cu–O stretching	480

2.1.2 NMR Spectral analysis

The CK showed signal as a triplet at 0.918δ , 0.969δ , 0.987δ and 1.290δ representing the aliphatic -CH₃ proton and CH₂ proton attached to -CH₂-R group.²⁴ While the peak at 2.093δ represents the proton of -CH₂ group attached to C=C group. Vinylic proton gave signal at 5.474δ . The peaks at 2.233δ and 1.590δ correspond to CH₂ proton on α and β carbon linked to $(-\text{COO})_2\text{Cu}$ group. All the peaks are due to the

long chain fatty acid content (R) of the soap molecule $[(RCOO)_2Cu]$, shown in Table 3.

Table 3: NMR spectral data for CK soap.

Peak/signal	CK
$-CH_3-CH_2-R$	0.918
$-CH_2-CH_2-R$	1.229 1.290
$-CH_2-C=C-$	2.093 2.107
$-C=C-H$ (vinylic proton)	5.254 5.474 5.489 5.507 5.549
$-CH_2-C(=O)OCu$	2.233
$-CH_2-CH_2-C(=O)OCu$	1.590

2.2 Measurement of Density

Ostwald's modification of Sprengel pycnometer with a volume of about 10 ml was used for measuring the density of the soap solution in the thermo stated bath at 298.15 K. The density of the solutions was calculated by the following relationship. The measured values of density compared with literature values.²⁵ The possible error in the density was estimated to be less than. The density ρ was reproducible to within 0 kg m^{-3} :

$$\rho = \frac{w}{w_0} \quad (1)$$

where w and w_0 are the weights (same volume) of solution and water, respectively.

2.3 Evaluation of Molar Volume

The molar volume of the complex solution V has been calculated by the following relationship:²⁶

$$\nabla = \frac{M_1X_1 + M_2X_2 + M_3X_3}{\rho} \quad (2)$$

where X_1 is the mole fraction of the soap of molecular weight M_1 whereas X_2 is the mole fraction of benzene of molecular weight M_2 , and X_3 is the mole fraction of

methanol of molecular weight M_3 . On the other hand, ρ stands for density of the solution. The molar volume was reproducible to within $0 \text{ m}^3 \text{ mol}^{-1}$.

2.4 Evaluation of Apparent Molar Volume

The apparent molar volume has been calculated from the density data using the following equation:²⁷

$$\phi_v = \frac{M}{\rho^0} + \frac{1000(\rho_0 - \rho)}{c \cdot \rho^0} \quad (3)$$

where ρ_0 represents the density of the solvent, ρ is the density of the soap solution, M is the molecular weight of the soap, and c is the concentration of solution in mol l^{-1} . The A.M.V. was reproducible to within $0 \text{ m}^3 \text{ mol}^{-1}$.

2.5 Evaluation of Viscosity

The viscosity of the soap solutions was calculated by the following relationship:²⁸

$$\frac{\eta_0}{\eta} = \frac{\rho_0 \cdot t_0}{\rho \cdot t} \quad (4)$$

where η_0 , η , ρ_0 , ρ , t_0 and t are the viscosity, density and time of flow for the known and unknown solutions, respectively. The viscosity η was reproducible to within mPa s^{-1} .

3. RESULTS AND DISCUSSION

3.1 Density

The effect of varying soap concentrations on the density of Cu (II) karanj soap solution in 100% benzene, 80% benzene-methanol and 60% benzene-methanol in methanol benzene solvent were studied and the solutions were abbreviated as CK₁₀₀, CK₈₀ and CK₆₀, respectively. The density of CK soap solutions initially increases and then decreases and shows a sudden increase again. The plot of density ρ against concentration c (mol l^{-1}) is characterised by an intersection of two convex curves with reference to X-axis. The point of intersection of possible two straight lines at a definite concentration corresponds to the CMC (Figure 1) suggests the sudden change in density, i.e., behaviour of the solution.²⁹ The CMC values of copper karanj soap solutions decreases with the increase in methanol the polar solvent. The CMC of the CK soap solution:

$$\text{CK}_{100} > \text{CK}_{80} > \text{CK}_{60}$$

This difference clearly demonstrates that agglomeration of soap molecules initiates at a slightly lower soap concentration with the increase of methanol the polar solvent in benzene. This fact suggests that micellar entities are formed a little more easily and efficiently at a much lower soap concentration in the system with higher percentage of polar solvent. It is known that the CMC decreases by about one third per methylene group in aqueous solutions because the energy required to transfer a methylene group from micelle to bulk in small as intermolecular forces are stronger in polar solvents like water and methanol, whereas in non-polar solvents like benzene, these intermolecular forces are weaker.

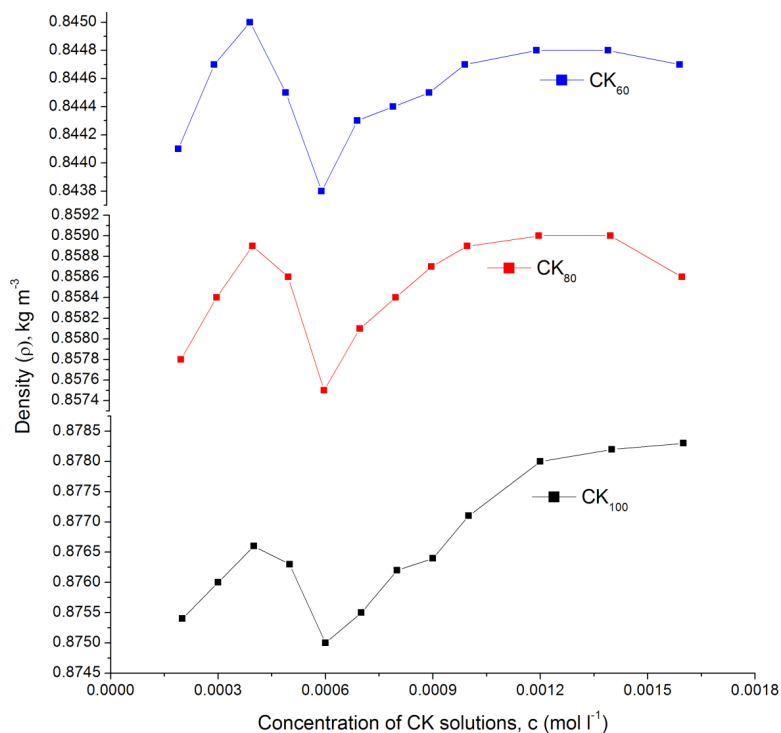


Figure 1: Plot of density versus concentration for CK soap solutions in CK₁₀₀, CK₈₀ and CK₆₀ at 298.15 K.

3.2 Molar Volume

The effect of varying soap concentrations on the molar volume of CK soap solution in benzene, 80% benzene-methanol and 60% benzene-methanol is observed and mentioned below:

$$CK_{100} > CK_{80} > CK_{60}$$

The molar volume of CK soap solutions also initially decreases and then increases and shows a sudden decrease again. The plot of molar volume \bar{V} against concentration is characterised by an intersection of two concave curves (Figure 2).

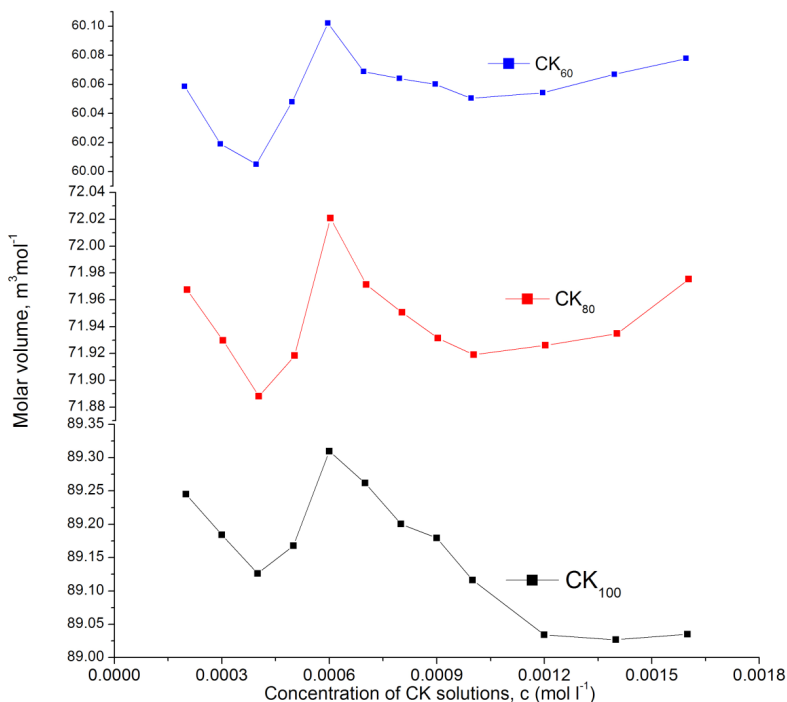


Figure 2: Plot of molar volume versus concentration for CK soap solutions in CK₁₀₀, CK₈₀ and CK₆₀ at 298.15 K.

3.3 Apparent Molar Volume

The effect of varying soap concentrations on the apparent molar volume of CK soap solution in 100% benzene, 80% benzene-methanol and 60% benzene-methanol is observed and mentioned herewith:

$$\text{CK}_{100} > \text{CK}_{80} > \text{CK}_{60}$$

The plot of molar volume \bar{V} against concentration is characterised by an intersection of two concave curves, shown in Figure 3.

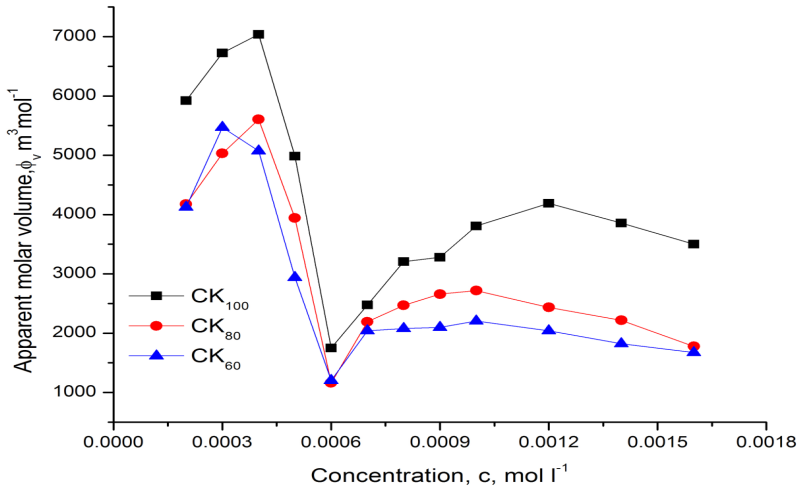


Figure 3: Plot of apparent molar volume versus concentration of CK soap solutions in CK₁₀₀, CK₈₀ and CK₆₀ at 298.15 K.

To explore these observations of various researchers in our referred systems the data has been analysed in terms of Masson's equation:³¹

$$\phi_v = \phi_v^0 + S_v \sqrt{c} \quad (5)$$

Here, ϕ_v represents apparent molar volume, whereas ϕ_v^0 and S_v represent limiting apparent molar volume and limiting apparent slope, respectively. ϕ_v^0 and S_v have been regarded as the measure of solute-solvent interactions and solute-solute interactions, respectively.

According to our observation of ϕ_v volume for CK soap, their some negative volume could be ascribed to the earlier study done on the basis of group additivity.¹⁶ Some trends that are observed can be explained, at least partially, in terms of structural ion-ion interactions, i.e., hydrophilic-hydrophilic, hydrophilic-hydrophobic and hydrophobic-hydrophobic interactions. The solute which is studied is CK soap with both hydrophilic and hydrophobic features, i.e., COO⁻ of fatty acid chain is hydrophilic in nature and long alkyl chain is hydrophobic in nature. On the other hand, the solvents, i.e., non-polar solvent, benzene is hydrophobic and polar solvent methanol is hydrophilic in nature. Thus, the various interactions occurring between solute and solvent have been analysed.³⁰

The plot of ϕ_v against \sqrt{c} is characterised by an intersection of two convex curves with reference to X-axis (Figure 4). The point of intersection of possible two straight lines in the curves at a definite concentration corresponds to the critical micelle concentration. Of course, this is the maximum concentration of molecular dispersion at which there is balancing of internal forces causing the formation of micelles. The value of apparent molar volume ϕ_v shows a sharp decrease below CMC and increases above CMC in CK soap solution in pure benzene, 80% benzene-methanol and 60% benzene-methanol system.

The CMC obtained from plot of ϕ_v against \sqrt{c} has also been confirmed by the density and molar volume measurements. The CMC obtained in this study, is in good agreement with the physical properties like viscosity and various ultrasonic parameters.

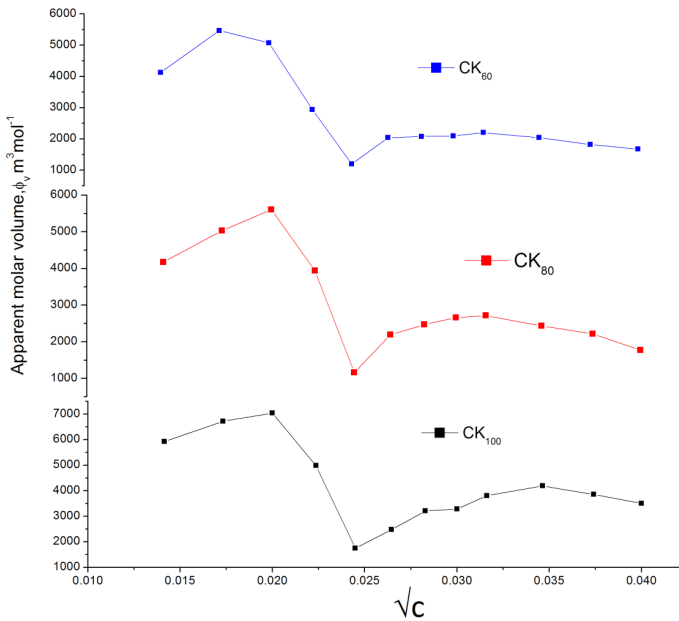


Figure 4: Plot of apparent molar volume versus \sqrt{c} of CK soap solutions in CK₁₀₀, CK₈₀ and CK₆₀ at 298.15 K (Moulik's equation).

Masson's equation (Equation 5) given earlier fits well both below and above CMC as the plot is characterised by an intersection of possible two straight lines. The value of limiting apparent molar volume (ϕ_v^0) for the soap is obtained by the extrapolation of ϕ_v against \sqrt{c} plot to $C \rightarrow 0$. Two values of ϕ_v^0 are obtained which are referred to as ϕ_{v1}^0 (below CMC) and ϕ_{v2}^0 (above CMC) as the Masson's equation is applicable to the two intersecting straight lines.

It is obvious from Table 4 that $\phi_{v1}^0 > \phi_{v2}^0$ for CK soap solutions. Thus, for CK soap solute-solvent interaction is more pronounced below CMC as compared to above. This may be ascribed to the favourable interactions between soap-solvent molecules before formation of the micelles.

Table 4: Computed parameters of Masson's equation for CK soap solutions in CK₁₀₀, CK₈₀ and CK₆₀ at 298.15K.

Soap solution	f_{v1}^0	f_{v2}^0	S_{v1}	S_{v2}
CK ₁₀₀	30692.70	-7680.71	-1171254.33	384608.81
CK ₈₀	25483.56	-1286.09	-983334.15	131929.94
CK ₆₀	22287.34	+1182.15	-862279.80	32192.21

Similarly, the parameter S_v in Masson's equation represents the limiting apparent slope and is indicative of solute-solute interactions. A greater magnitude of S_v shall be referred to as greater solute-solute (i.e., soap-soap) interactions. It is reasonable to determine two values of parameters S_v , i.e., below and above CMC designated as S_{v1} and S_{v2} , respectively. It is evident from Table 4 that values of S_v parameters follow the order $S_{v1} < S_{v2}$. Therefore, it can be concluded that solute-solute interactions become more pronounced above CMC with respect to solvents the observed order for ϕ_{v2}^0 and S_{v2} , i.e., above CMC:

$$\phi_{v2}^0(\text{CK}_{60}) > \phi_{v2}^0(\text{CK}_{80}) > \phi_{v2}^0(\text{CK}_{100})$$

$$S_{v2}(\text{CK}_{100}) > S_{v2}(\text{CK}_{80}) > S_{v2}(\text{CK}_{60})$$

Thus, it is obvious that above CMC, the soap-solvent interaction becomes more pronounced with more percent of polar-solvent methanol while the soap-soap interaction decreases, shown in Table 5.

Table 5: Values of CMC for CK soap solutions in CK₁₀₀, CK₈₀ and CK₆₀ at 298.15 K.

Parameter	CK ₁₀₀	CK ₈₀	CK ₆₀
ρ vs. c	0.00061	0.00057	0.00054
V vs. c	0.00061	0.00057	0.00054
φ_v vs. c	0.00061	0.00057	0.00054
η vs. c	0.00058	0.00053	0.00051
h_{sp} vs. c	0.00058	0.00053	0.00051
f_v vs. c	0.00058	0.00053	0.00051
(h_{sp}/c) vs. c	0.00058	0.00053	0.00051
(ψ/\sqrt{c}) vs. \sqrt{c}	0.00058	0.00053	0.00051
$(\eta/h_0)^2$ vs. c^2	0.00058	0.00053	0.00051
(I/c) vs. $\{I/\log(\eta/h_0)\}$	0.00058	0.00053	0.00051
(C/h_{sp}) vs. c	0.00058	0.00053	0.00051

3.4 Viscosity

Flow characterisation of soap solutions in terms of viscometric measurements has been employed as a tool for finding out the CMC of copper soaps in benzene and two compositions of benzene-methanol systems.

The plot of viscosity η against concentration c is shown in Figure 5 for CK soap solution in benzene-methanol mixture. The intersection of the two possible straight lines corresponds to the CMC of the CK soap. Before CMC, the plot is of the curve nature and after CMC, the viscosity value increases almost linearly. Interestingly, Moulik's equation also fits equally well to our referred systems:³²

$$\left(\frac{\eta}{\eta_0}\right)^2 = M + Kc^2 \quad (6)$$

where M and K are constants. Thus, the values of M and K could be evaluated both below and above CMC and are designated as M_1 , M_2 , K_1 and K_2 , respectively.

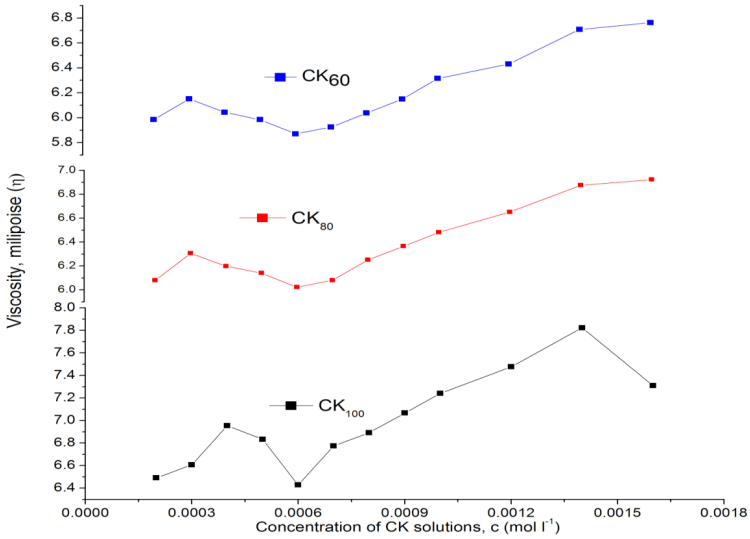


Figure 5: Plot viscosity versus concentration for CK soap solutions in CK₁₀₀, CK₈₀ and CK₆₀ at 298.15 K.

The viscosity data have also been interpreted in the light of Jones-Dole equation.³³

$$\left(\frac{\eta}{\eta_0}\right)^2 - 1 = A + B\sqrt{c} \tag{7}$$

For convenience, the equation may be expressed as:

$$\frac{\psi}{\sqrt{c}} = A + B\sqrt{c} \tag{8}$$

where the coefficient A and B refer to the solute-solute and solute-solvent interactions, respectively. The values of A and B are evaluated from the plot of (ψ/\sqrt{c}) vs. \sqrt{c} , which is characterised by an intersection of two straight lines. Thus, Jones-Dole equations fit well both below and above CMC. The value of CMC obtained is in close agreement with the CMC obtained from η vs. c , η_{sp} vs. c , and ϕ_v vs. c plots, shown in Figure 6.

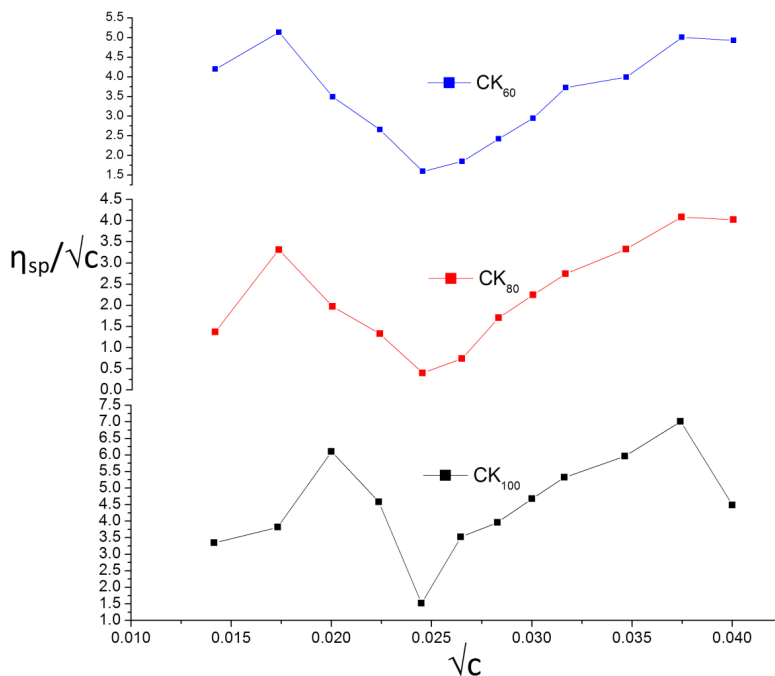


Figure 6: Plot of η_{sp}/\sqrt{c} against square root of concentration for CK soap solutions in CK₁₀₀, CK₈₀ and CK₆₀ at 298.15 K (Jones-Dole equation).

The values of A and B evaluated are recorded in Table 6. Literature survey reveals that positive value of A suggests a strong solute-solute interaction.

Table 6: Computed parameters derived from different equations for CK soap solutions in CK₁₀₀, CK₈₀ and CK₆₀ at 298.15 K.

Soap	Moulik's equation				Jones-Dole equation			
	M ₁	M ₂	K ₁	K ₂	A ₁	A ₂	B ₁	B ₂
CK ₁₀₀	1.04	0.9107	-0.9152	0.4639	26.48	-12.03	-1019.00	551.90
CK ₈₀	1.15	1.0281	-0.3660	0.1077	10.07	-3.52	-395.33	189.91
CK ₆₀	1.21	1.0740	-0.3746	0.1390	13.66	-2.54	-492.75	186.33

B co-efficient measures the structural modification induced by solute-solvent interaction. Earlier studies show that the increase in concentration of solute in solution contributes positively to the viscosity of B-coefficient. On the other hand, the breaking of the solvent structures by solute causes a decrease in viscosity.

This contributes negatively to the B-coefficient. Thus, B coefficient is the resultant of these two opposite forces. Therefore, the systems/molecules exhibiting negative B-coefficient have been assumed to exert a structure breaking effect by the solute on the solvent molecules.³⁴

It is suggested that solute-solute interaction is greater below CMC as compared to above CMC for our referred CK₁₀₀, CK₈₀ and CK₆₀ systems as $A_1 > A_2$, and A_1 is positive while A_2 is negative for all the systems. Literature survey also reveals that B-coefficient measures the structural modification induced by solute-solvent interaction, and the molecules exhibiting negative B-coefficient have been assumed to exert a structure-breaking effect by the solute on the solvent molecules and it has been observed for our CK₁₀₀, CK₈₀ and CK₆₀ systems that the B-coefficient below CMC, i.e., B_1 is negative. This suggests that structure-breaking effect is more below CMC as $B_1 < B_2$. It can be suggested that the structure breaking effect by the solute on the solvent molecules is more prominent below CMC as compared to above CMC, i.e., after the formation of the micelles.³⁵

4. CONCLUSION

The present research work attempts to prepare surface active organic compounds from natural non-edible oil and to study their solution behaviour, micellar features and various interactions with polar and non-polar solvents to understand the applied part of the solute. The beneficial effects of the synthesised pharmaceuticals and their biological applications may be explored in the further researches. The current topic will not only strengthen relation between industries, private sectors and research laboratories on the focal theme of biology, physics and environment, but also will also play a significant role in forth coming scientific development through the information related to micellar behaviour of surfactants.

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