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Solvation Study of Neem Oil in Aqueous Sodium Dodecyl Sulphate at different Temperatures

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ABSTRACT

The plant diseases have significant role in agriculture in terms of reduction in yield and economy. In agriculture, for thousands of years, people have been using neem oil as a natural insecticide, fungicide and bactericide. Neem pesticides have been widely used to kill or manage the population of pest. Neem oil is hydrophobic in nature; in order to emulsify it in water for application purpose, it must be formulated with appropriate surfactants. In the present study, neem oil was mixed with sodium dodecyl sulphate (SDS). The density, viscosity and ultrasonic velocity of aqueous solutions of SDS with neem oil have been measured. The compressibility behaviour of solutions provides very useful information related to solute and solvent interactions. The solubility of neem oil has been investigated by the estimated values of solvation parameters. The experimental data have been used to calculate the solvation parameters such as adiabatic compressibility, molal hydration number, apparent molal compressibility, apparent molal volume, limiting apparent molal compressibility, limiting apparent molal volume and their constants and viscosity co-efficients A and B of Jones-Dole equation. Based on the results, the nature and types of interactions in the solutions are discussed. The growth performance of cowpea crop in pot culture experiment is also analysed. After manifestation, periodical records of symptoms of diseases due to insects are noted. The biometric observations of crops at frequent intervals are enumerated. The results obtained from ultrasonic method and pot culture experiment are in consonant with each other.

Keywords: *SDS; neem oil; adiabatic compressibility; hydration number; apparent molal compressibility; apparent molal volume; pot culture experiment*

I. Introduction

The study of propagation of ultrasonic waves in liquid system is now well established as an effective means for examining certain physical properties of the materials [1]. Most of the physical properties of liquids are actually controlled by the strengths of intermolecular attractive forces [2]. The nature and degree of molecular interactions in different solutions depend upon the nature of solvent, the structure of solute molecule and extent of solutes taking place in the solution [3, 4]. The ultrasonic study of intermolecular interactions plays an important role in the development of molecular science [5].

Pesticides are chemicals that control the population of pests. There are many different types of pesticides on the market today, but the most common are herbicides and insecticides, which destroy unwanted plants and insects. The benefits of pesticides include increased food production, increased profits for farmers and the prevention of diseases. Neem oil is an organic pest pressed from the fruits and seeds of neem. It has been used for hundreds of years to control pests and diseases. Components of neem oil can be found in many products today. These include toothpaste, cosmetics, soaps and pet shampoos.

Surfactant is an abbreviation for surface active agents, which literally means active at a surface [6]. Surfactants contain two distinct grouping in their structure, polar or charged group at one end of it is the 'head group" which is hydrophilic in nature and long chain of alkyl or aryl group is the "tail group" which is hydrophobic in nature. At low concentration, surfactants are dispersed as discrete molecules. However at a particular concentration, surfactant molecules get associated to form aggregates or micelles [7-9]. This concentration is known as critical micelles concentration (CMC) which is an important property of surfactant. Above CMC, the surfactants exist as aggregates or micelles. Anionic surfactants are usually chosen for surfactant based remediation procedures because of their lower degree of adsorption on soil than that by cationic and non-ionic surfactants [10]. Surfactants help by reducing the surface tension of water and since herbicide sprays are mostly water, this keeps the spray on the targeted weed rather than rolling off onto the soil.

In this research work, deliberate analyses were done with neem oil in SDS at 298K, 303K and 308K. The ultrasonic velocity data alone will not provide sufficient information about nature and types of interactions present in the solution. Hence their derived parameters such as adiabatic compressibility (β), molal hydration number (n_h'), apparent molal compressibility (ϕ_k), apparent molal volume (ϕ_v), limiting apparent molal compressibility (ϕ_k^0), limiting apparent molal volume (ϕ_v^0) and their constants (S_k, S_v) and viscosity B co-efficient have been found to know about the various types of interactions exist between solute and solvent.

In pot culture experiment, cowpea seeds are grown in pots at ambient temperature. Neem oil of different concentration in different molarities of SDS is sprayed on the leaves and stem of the crop. After spraying neem oil, the control of pests viz., aphids, leaf roller, leaf miner etc., is visualized. The biometric observations of the crops such as plant height, dry mass etc., are also found.

I. Experimental Study

Neem oil is used as an organic biopesticides repellent against insects. SDS used in the present study is of AR grade (99% of purify) and hence used without further purification. Deionised water was used for the preparation of solutions. Solutions of SDS of different concentrations were prepared by molarity scale using a Denver digital electrical balance with an accuracy $\pm$ 0.1 kg m^{-3}. An Ostwald's viscometer of 10ml capacity was used for the viscosity measurement. The ultrasonic velocity of solution has been measured using with an ultrasonic interferometer (Mittal Enterprises, New Delhi, Model F-81) working at a fixed frequency of 2MHz. A digital electrically operated constant temperature bath (Raaga Industries, Chennai) was used to maintain the desired temperature. The accuracy of the measurement of temperature was $\pm$ 0.1° C.

A. Pot culture experiment

The pot culture experiment was conducted to find out the effect of various concentrations of neem oil solutions on growth of cowpea crop [11]. The soil samples were prepared with different compositions of reddish brown soil, sand, farm yard manure (4:1:1). Healthy seeds of cowpea (vamban1) collected from National Pulses Research Centre, Vamban, Pudukkottai and sown in pots with 7 treatments of three replicate [12]. The pots were filled with $5\frac{1}{2}$ kg of soil, each sown with three seeds of cowpea. Water was applied when needed to keep the soil moist. The crops were grown and after 25 days, neem oil solution was sprayed carefully by using garden pressure spray pump so that the solutions fall on the crop surface.

B. Computational method

Using the measured values of density, viscosity and velocity of neem oil solutions, the compressibility, solvation parameters have been calculated using the standard relations,

1. Adiabatic compressibility $\beta = \frac{1}{U^2\rho}$
2. Molal hydration number $n_h' = \frac{n_1}{n_2}\left(1-\frac{\beta}{\beta_0}\right)$
3. Apparent molal compressibility $\phi_k = \frac{1000}{m\rho_0}(\rho_0\beta - \beta_0\rho) + \left(\frac{M\beta_0}{\rho_0}\right)$

where, β and β_0 are adiabatic compressibilities of solution and solvent. N denotes the number of solvent molecules. n_1 and n_2 are the number of moles of solvent and solute. ρ and ρ_0 are density of solution and solvent. M is the molecular weight of solute.
The apparent molal compressibility ϕ_k is the function of m as obtained by Gucker [13] from Debye Huckel theory [14]and is given by

$$\phi_k = \phi_k^0 + S_k\sqrt{m}$$

where ϕ_k^0 is the limiting apparent molal compressibility at infinite dilution and S_k is a constant.

4. Apparent molal volume $\phi_v = \frac{1000}{m\rho_0}(\rho_0 - \rho) + \frac{M}{\rho_0}$

The apparent molal volume ϕ_v has been found to differ with concentration. According to Masson's [15] empirical relation,

$$\phi_v = \phi_v^0 + s_v\sqrt{m}$$

Where ϕ_v^0 is the limiting apparent molal volume at infinite dilution and S_v is a constant.

5. A and B coefficients (η)

The importance of viscometric study of electrolytic solution in mixed solvent is well established by M S. Chauhan et al., in the year 2002 [16]. The entire viscosity data have been analysed in the light of Jones-Dole semi-emprical equation [17].

$$\frac{\eta}{\eta_0} = 1 + Am^{1/2} + Bm$$

Where η and η_o are the viscosities of the solution and solvent respectively and m is the molal concentration of the solute-solvent system. A and B are constants which are distinct for a solute-solvent system. A is known as the Falkenhagen coefficient which characterises the ionic interaction and B is the Jones-Dole or viscosity B-coefficient which depends on the size of the solute and nature of solute-solvent interactions [18,19].

II. Results and discussion

The experimentally measured values of density (ρ), viscosity (η) and ultrasonic velocity (U) for different concentrations of neem oil in SDS are given in table 1 and 2. The values of adiabatic compressibility (β), molal hydration number (n_h'), apparent molal compressibility (ϕ_k), apparent molal volume (ϕ_v), the limiting apparent molal compressibility (ϕ_k^0), limiting apparent molal volume (ϕ_v^0) and their constants, A and B co-efficients of Jones-Dole equation are shown in table 2-5.

The density values increases with neem oil concentration but it decreases with rise in temperature. This increasing behaviour implies a strong hydrophobic nature of solute neem oil which interacts with the hydrophobic part of SDS. The decrease in density with increase in temperature suggests decrease in cohesive force due to increase in the thermal energy of the system. This causes volume expansion and hence decreases in density.

It is observed from table 2, that the values of viscosity increases with increase in concentration of SDS as well as with neem oil (0.0014 - 0.0069 mM) and the same decreases with rise in temperature. The viscosity increases linearly with concentration of neemoil, this indicates that there exist a strong interaction between solute and solvent. The gradual increase in the value of viscosity with different concentrations of neem oil in SDS confirms the increase of cohesive forces because of strong interactions between solute and solvent molecules.

The values of ultrasonic velocities are increases with concentration of neem oil and temperature. It also increases with SDS concentration and also with temperature. The hydrophilic end of surfactant interacts with water whereas hydrophobic end interacts with neem oil. The addition of surfactant in neem oil will increase the cohesion between neem oil and hydrophobic part of surfactant. The cohesion increases with the increase of surfactant concentration in the solutions. The addition of surfactants will enhance the effectiveness of neem oil. It also increases in the order: 6mM < 7mM < 8mM.

From table 2, it was observed that the values of adiabatic compressibility (β) decreases with increase of SDS and neem oil. The compressibility of neem oil decreases with SDS concentration at all temperatures justifies the result drawn from velocity data. The decrease in adiabatic compressibility is due to the increase in electrostrictive compression of solvent around the neem oil molecules, which results in a large decrease in the compressibility of solution [20]. From the magnitude of β values, it is found that strong molecular association is noted in 8mM SDS solution.

The process of attraction and association of water molecules with solute molecules is termed as hydration. It is observed from table 3 that the values of n_h' are positive in all the systems and it is found to decrease with increasing the molarity of neem oil as well as SDS. The positive values of n_h' convey significant solvation of solutes [21]. It also denotes the existence of appreciable hydrophobic interactions between solute and SDS molecule. The positive value of n_h' is also validated for the structure making behaviour of solute. The hydration number decrease with increase in neem oil concentration indicates the strong interaction between co-solute-solute molecules.

But, however it increase with rise in temperature. This is because the rise in temperature causes the rearrangement of structure leads to a comparatively more ordered state that increases the ultrasonic velocity with temperature. The decrease in hydration number with increasing concentration of SDS points out that the addition of SDS cause dehydration effect on neem oil in solution.

The observation of ϕ_k and ϕ_v of surfactant solutions are noted as follows:

i. ϕ_k and ϕ_v values are negative over the entire range of molarity of neem oil.
ii. ϕ_v values various non-linearly with neem oil concentration at all temperatures as well as with increasing concentration of SDS. This indicates an existence of ion-solvent interactions. The negative values of ϕ_v indicate electrostrictive solvation of ion [22]. It can be concluded that for 8mM SDS solution, the molecular association is greater and hence the solvation of neem oil is effective in 8mM SDS at 298K and 303K temperatures.
iii. The values of ϕ_k increases linearly with neem oil and SDS concentration at all temperatures. This increasing behaviour of ϕ_k implies the existence of strong solute-solvent interaction in all the systems. The negative values of ϕ_k reveal the existence of hydrophobic and ionic interactions in the solutions.
iv. The variations in the values of ϕ_k are found to be similar to the variations of n_h values with opposite sign.

The limiting apparent molal compressibility ϕ_k^0 gives information regarding solute-solvent interactions and S_k that of solute-co-solute interactions in the solutions. It is noted from table 4 that ϕ_k^0 values are negative and it increases with increasing concentration of SDS and decreases with rise in temperature. The significant negative values of ϕ_k^0 in all the systems fortifies the persistance of solute-solvent interaction. The magnitude of ϕ_k^0 is in the order 8mM SDS > 7mM SDS > 6mM SDS. The positive values of S_k with increasing concentration of SDS reveals strong co-solute-solute interactions occurs in the solution. It also follows the same order as ϕ_k^0.

At infinite solution, the volume behaviour of a solute corresponds to ϕ_v^0. It is independent of ion-ion interactions and contributes information about ion-solvent interactions. Negative values of ϕ_v^0 symbolizes the persistance of ion-solvent interaction in all the systems. Further, it is evident from table 4 that, S_v values are positive indicating the strong solute-solute interaction at all temperatures. The positive values of S_v are connected with hydrophilic portion of SDS. The maximum value of S_v is found for 8mM SDS at all temperatures.

On observing the above variations of ϕ_k and ϕ_v in neem oil-surfactant solution, the following interactions may supposed to be taking place among solute (neem oil), co-solute (SDS) and water.

- Hydrophilic-dipolar interactions between the hydrophilic part of SDS and water.
- Hydrophobic-ionic interactions between the hydrophobic parts of neem oil and the non-polar (Na^+) parts of SDS.

The variation in the values of viscosity attributed to the structural changes due to the introduction of solute in a solvent. It is noticed from the table 5 that the viscosity values increases with increasing concentration of neem oil as well as SDS in the solution. The viscosity values are decreses with increase in temperature. This increasing behaviour signifies the existence of molecular interaction taking place in these solution. Viscosity A and B coefficients of Jones-Dole equation have been evaluated to get more information about solute-solvent interactions. It is viewed from table 5 that A and B values are positive at all temperatures. A values are decrease with increase in temperature and B values are increase with increase in temperature. A is the measure of solute-solute interactions. From the value of A, it is cleared that there exists strong solute-solute interactions in the solutions. The order and disorder introduced by the solute into the solvent is very well understood by B coefficient. It is also a measure of solute-solvent interaction and relative size of the solute and solvent molecules. The values of B coefficient clearly denotes the existence of solute-solvent interactions. The magnitude of B values follows the order: 8mM SDS > 7mM SDS > 6mM SDS. This result is in good aggrement with the result drawn from S_k and ϕ_k^0 data. The large values of B denote the structure promoting capacity of solute.

In pot culture experiment 7 treatments of three replicate are taken which contain the combination of two different concentration of neem oil in different molarities of SDS. In this experiment, biometric observation viz., plant height, is recorded on 8, 16, 24, 32nd DAS. A periodical record of diseases due to pests are noted. Different concentrations of neem oil in different molarities of SDS is sprayed on leaves and stem of the cowpea crops for 7 treatments. After the application of neem oil solution, the control of pests like aphid, leaf rollers, leaf miner are visvalized explicitly. Plant height is an important component, which increases plant growth by increasing the number and length of the internodes. The treatments T6 (8mM-SDS, 1.39mM-neem oil) and T7 (8mM-SDS, 6.94mM-neem oil) were found to be dominated in the growth of cowpea among the 7 treatments. The plant height in T7 treatment was greater than other treatments (table 6). The morphological characters of cowpea crop are given in table 7. It is detected from table 7, that the morphological characters of cowpea crop are elevatedin treatments T6 and T7.

Table 1: Values of Ultrasonic velocity and density of neem oil in SDS

Molarity (M)	Ultrasonic velocity (m/s)			Density (Kg/m^3)		
	298K	303K	308K	298K	303K	308K
6mM SDS with neem oil						
0	1520	1526	1532	998.51	997.97	996.09
1.39	1528	1535	1543	1000.35	999.94	998.03
2.78	1535	1544	1551	1002.19	1001.11	1000.71
4.16	1538	1549	1558	1006.71	1005.17	1004.26
5.55	1541	1555	1565	1007.81	1006.36	1005.94
6.94	1545	1561	1569	1008.91	1007.61	1006.03
7mM SDS with neem oil						
0	1530	1534	1538	999.694	998.577	996.887
1.39	1537	1542	1548	1001.79	1000.92	999.901
2.78	1544	1550	1556	1002.85	1001.43	1000.87
4.16	1547	1554	1560	1006.98	1005.28	1005.19
5.55	1550	1560	1567	1009.67	1007.94	1006.45
6.94	1554	1563	1571	1010.12	1008.95	1008.83
8mM SDS with neem oil						
0	1536	1540	1547	1000.86	999.757	997.058
1.39	1543	1548	1555	1002.57	1001.54	1000.16
2.78	1550	1556	1563	1003.80	1002.14	1001.28
4.16	1553	1560	1566	1007.29	1005.79	1005.57
5.55	1559	1567	1574	1006.45	1004.35	1003.30
6.94	1561	1571	1577	1008.66	1006.38	1005.94

Table 2: Values of viscosity (η) and adiabatic compressibility (β) of neem oil in SDS

Molarity (M)	viscosity (10^{-3} Nsm^{-2})			β (10^{-10} $Kg^{-1}ms^{-2}$)		
	298K	303K	308K	298K	303K	308K
6mM SDS with neem oil						
0	0.8972	0.8015	0.7245	4.335	4.303	4.278
1.39	0.9214	0.8224	0.7439	4.282	4.244	4.209
2.78	0.9466	0.8448	0.7621	4.235	4.190	4.154
4.16	0.9683	0.8618	0.779	4.199	4.146	4.102
5.55	0.9913	0.8831	0.7982	4.178	4.110	4.059
6.94	1.0177	0.9044	0.8178	4.152	4.073	4.038
7mM SDS with neem oil						
0	0.9052	0.8096	0.7281	4.273	4.256	4.241
1.39	0.9313	0.8314	0.7480	4.225	4.202	4.174
2.78	0.9591	0.8556	0.7688	4.183	4.156	4.127
4.16	0.9809	0.8742	0.7836	4.149	4.119	4.088
5.55	1.0048	0.8949	0.8049	4.123	4.077	4.046

6.94	1.0313	0.9162	0.8229	4.099	4.057	4.016
8mM SDS with neem oil						
0	0.9111	0.8144	0.7329	4.235	4.218	4.191
1.39	0.9466	0.8448	0.7590	4.189	4.167	4.135
2.78	0.9775	0.8745	0.7824	4.147	4.122	4.088
4.16	0.9994	0.8949	0.7994	4.116	4.086	4.055
5.55	1.0352	0.9221	0.8245	4.088	4.055	4.023
6.94	1.0587	0.9441	0.8423	4.069	4.026	3.997

Table 3: Values of molal hydration number, apparent molal volume and apparentmolar compressibility of neem oil in SDS

Temperature	Molal hydration number			
	Molarity (M)	6mM	7mM	8mM
298K	1.39	483.68	441.31	425.57
	2.78	455.09	417.53	413.38
	4.16	412.36	382.04	371.09
	5.55	357.15	349.84	343.81
	6.94	338.40	327.13	316.29
303K	1.39	537.56	499.78	478.09
	2.78	517.41	460.11	450.87
	4.16	480.36	422.81	414.20
	5.55	444.97	416.33	381.79
	6.94	429.52	375.06	364.97
308K	1.39	635.38	623.92	527.03
	2.78	569.83	529.16	484.19
	4.16	540.92	474.42	427.99
	5.55	506.77	452.59	395.51
	6.94	450.71	424.77	371.01
Temperature	Apparent molal volume (m^3 mol^{-1})			
298K	1.39	-593.24	-782.67	-498.43
	2.78	-595.85	-406.08	-329.39
	4.16	-1232.6	-1013.0	-809.99
	5.55	-942.12	-1060.4	-276.73
	6.94	-788.29	-789.82	-408.54
303K	1.39	-684.96	-951.14	-555.49
	2.78	-399.95	-299.53	-129.35
	4.16	-995.49	-876.53	-714.89
	5.55	-779.49	-953.04	-100.22

	6.94	-677.68	-782.94	-238.98
308K	1.39	-1101.6	-1435.9	-1502.4
	2.78	-1369.2	-702.26	-789.21
	4.16	-1664.8	-1259.9	-1308.8
	5.55	-1476.9	-989.51	-394.71
	6.94	-1157.0	-1012.5	-567.47
Temperature	Apparent molar compressibility (10^{-6} m^2 N^{-1})			
298K	1.39	-4.05	-3.75	-3.46
	2.78	-3.83	-3.40	-3.29
	4.16	-3.76	-3.38	-3.17
	5.55	-3.19	-3.14	-2.74
	6.94	-2.99	-2.86	-2.58
303K	1.39	-4.48	-4.26	-3.87
	2.78	-4.20	-3.67	-3.49
	4.16	-4.16	-3.62	-3.45
	5.55	-3.79	-3.60	-2.95
	6.94	-3.63	-3.21	-2.88
308K	1.39	-5.40	-5.41	-4.62
	2.78	-4.99	-4.37	-3.99
	4.16	-4.89	-4.17	-3.78
	5.55	-4.54	-3.89	-3.16
	6.94	-3.97	-3.68	-3.04

Table 4: Values of limiting apparent molal compressibility (ϕ°_k), limiting apparent molal volume (ϕ°_v) and their constants S_k and S_v of solution at different temperatures

Systems	$-\phi_k^0$ (10^{-6} m^2N^{-1})			S_k ($10^{-6}m^{-1}$ N^1mol^{-1})		
	298K	303K	308K	298K	303K	308K
6mM SDS	4.54	5.12	6.23	0.42	0.56	0.71
7mM SDS	4.45	5.08	5.89	0.61	0.71	0.84
8mM SDS	4.40	4.86	5.86	0.68	0.82	1.06
Systems	ϕ_v^0 (m^3 mol^{-1})			S_v ($mol^{-3/2}$)		
6mM SDS	25.57	26.27	33.85	6.64	7.90	8.23
7mM SDS	24.36	26.19	32.37	6.07	7.02	9.59
8mM SDS	21.99	23.50	26.85	6.81	8.02	9.77

Table 5: Values of A and B coefficients of Jones - Dole equation of solution at different temperatures

Systems	Viscosity (η)					
	A/($dm^{-3/2}mol^{-1/2}$)			B/(dm^3mol^{-1})		
	298K	303K	308K	298K	303K	308K
6mM SDS	19.06	18.12	17.76	0.0067	0.0215	0.0422
7mM SDS	19.17	18.18	17.80	0.0658	0.0659	0.0735
8mM SDS	20.08	19.71	18.10	0.2788	0.2864	0.2871

Table 6: Effect of the solutions on crop height of cowpea by pot culture experiment

Molarity of Neem oil	Dry mass in Kg/m^3				
	Treatment	Stem	Root	Leaf	Yield
	Control (T1)	3.405	1.486	6.756	7.759
1.39mM	6mM (T2)	2.177	0.838	3.672	9.371
	7mM (T4)	2.975	1.857	6.683	9.664
	8mM (T6)	4.723	1.976	8.643	11.563
6.94mM	6mM (T3)	3.745	1.891	8.627	10.399
	7mM (T5)	4.441	2.105	9.026	14.835
	8mM (T7)	4.909	2.121	9.148	16.140

Table 7: Effect of the solutions on morphological characters of cowpea by pot culture experiment

Molarity of Neem oil	Dry mass in Kg/m^3				
	Treatment	Stem	Root	Leaf	Yield
	Control (T1)	3.405	1.486	6.756	7.759
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	8mM (T6)	4.723	1.976	8.643	11.563
6.94mM	6mM (T3)	3.745	1.891	8.627	10.399
	7mM (T5)	4.441	2.105	9.026	14.835
	8mM (T7)	4.909	2.121	9.148	16.140

1.Aphid

2.Leaf Roller

3. Leaf Miner

4.Well stem of concentration crop

5.Damage stem of control crop

7 Treatments Randomized in three Replicates

CONCLUSION

The density (ρ), viscosity (η) and ultrasonic velocity (U) have been measured for different concentrations of SDS with neem oil at different temperatures, which have been used as biological pesticides. The values are used to calculate the adiabatic compressibility and hydration parameters. The evaluated parameters clearly suggest that neem oil is strong structure maker in SDS. The hydrophobic parts of surfactant molecules shed light on solute-solvent interaction with neem oil. From the solvation parameters, it was found that there exit a strong interaction of neem oil in 8mM SDS. Pot culture studies were carried out to find out the control of pests by neem oil sprayed on the crop. The crop height is increased with increase in concentration of neem oil and SDS. It is evident from the ϕ_k^0,S_k values and A, B coefficients of Jones-Dole equation of neem oil in 8mM SDS is large. Pot culture experiment results also show that the control of pests, plant height and morphological characters of cowpea crop are excellent in neem oil with 8mM SDS.

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