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Hydrotropic Aggregation Behavior of Butyl Stearate

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Abstract On investigation for the solubility and mass transfer coefficient of butyl stearate through hydrotropy has been studied. This study was carried out using hydrotropes such as citric acid, urea, nicotinamide, sodium salicylate under the influence of a wide range of hydrotrope concentrations [(0 to 3) mol $\cdot L^{-1}$] and different system temperatures [(303 to 333) K]. It has been distinctively observed and noted that the solubility of butyl stearate increases with an increase in hydrotrope concentration and also with system temperature likely similar to the several organic compounds and drugs A Minimum Hydrotrope Concentration (MHC) in the aqueous phase was required to initiate significant solubilization of butyl stearate. Consequent to the increase in the solubilization of butyl stearate, the mass transfer coefficient was also found to increase with increase in hydrotrope concentration at 303 K. A threshold value similar to MHC is to be maintained to have an appreciable enhancement in mass transfer coefficient. The maximum enhancement factor, which is the ratio of the value in the presence and absence of a hydrotrope, has been determined for all sets of experimentations. To ascertain the hydrotropic aggregation behavior of butyl stearate, thermodynamic parameters such as gibb's free energy, enthalpy, and entropy of butyl stearate were determined. The gibb's free energy decreases with an increase in system temperature. The aggregation of hydrotropes was found to be exothermic in nature and favored by a positive value of entropy.

Keywords Hydrotrope, Solubility, Butyl stearate, Thermodynamics

1. Introduction

Neuberg identified this pioneering technique and the first one who reported and introduced the terminology of Hydrotropy (Neuberg, 1916). Hydrotropy is considered as a unique and unprecedented solubilization technique because of the easy recovery of dissolved solute and possible re-use of hydrotrope solutions for effecting very large solubility enhancement for a variety of sparingly soluble organic compounds. However, the term has been used in the literature to designate non-micelle forming substances, either liquids or solids, organic or inorganic, capable of solubilizing insoluble compounds this increase in solubility in water is probably due to the formation of organized assemblies of hydrotrope molecules at critical concentrations.

The solubility enhancement in the organic compounds could be due to the formation of molecular structures in the form of complexes. (Gaikar.V.G.et.al: 1986, 1993, 1999).

The use of hydrotrope solutions in such industrial applications is attractive because of their easy availability, the ready recovery of the dissolved solutes by simple dilution with water or by solvent extraction, and the absence of any fire hazards (Perry, 1997) Hydrotropy does seem to be operative above a particular concentration termed critical or minimum hydrotrope concentration. Above this critical concentration, the solubilization rises remarkably and may level off to a plateau thus leading to a sigmoidal solubility profile with hydrotrope concentration (Gandhi et.al.2009, 2010, 2012)

In general the Hydrotropes are water-soluble and surface-active compounds which can significantly enhance the solubility of organic solutes such as esters, acids, alcohols, aldehydes, ketones, hydrocarbons, and fats (Bhat M.B., 1999).In many two-phase reaction systems involving a sparingly soluble organic compound like butyl stearate, the mass-transfer coefficient was found to be very low solely due to the poor solubility of solute in the aqueous phase. Since butyl stearate serves as raw material/intermediate for a wide variety of chemicals and allied products and its separation from any liquid mixture seems to be difficult, this hydrotropic phenomenon can be effectively used for this system. Hydrotropic agents are freely soluble organic compounds that, at a concentration sufficient to induce a stack-type aggregation, considerably enhance the aqueous solubility of organic substances practically insoluble under normal conditions (C.Jayakumar and N.Nagendra Gandhi; 2010, 2012)

Hydrotropes have many practical applications including separation process, development of pharmaceutical formations, painting and coatings, food, plastic additives, selective separation, increase of cloud points of detergent solutions, changes in reaction rate, preparation of drilling well fluids, and separation of water-oil emulsion. It has been

observed that the effect of hydrotropes on the solubility and mass transfer coefficient for a series of organic compounds such as benzamide, cyclohexane, L-tyrosine were studied (Wagle, 2007; Tanford, 1980; Friberg, 1994).With the considerations and the various aspects of hydrotropic behavior and based on its tremendous applications on the enhancement of solubility and mass transfer coefficient for the butyl stearate-water system is reported here for the first time.

Table 1. Effect of Citric acid Concentration (*c*) on the Solubility (*S*) of Butyl stearate in Water

C	10^6S/mol ·L^{-1}			
mol ·L^{-1}	T = 303 K	T = 313 K	T = 323 K	T = 333 K
0.2	3.97	4.27	4.47	4.34
0.4	4.05	4.38	4.49	4.43
0.6	4.36	4.67	5.02	5.68
0.8	4.58	5.06	5.45	6.24
1	4.96	5.42	5.67	7.54
1.2	5.13	5.78	5.98	8.68
1.4	5.38	6.15	6.75	9.55
1.6	5.92	6.48	6.78	10.55
1.8	6.15	6.92	7.35	11.82
2	6.32	7.45	7.87	12.31
2.2	6.35	8.34	8.99	13.01
2.4	7.52	9.1	10.94	14.02
2.6	7.53	9.12	10.98	14.12
2.8	7.77	9.23	11.02	14.44
3	7.91	9.43	11.04	14.52

2. Materials and Methods

All the chemicals used in this work are manufactured by SD Fine Chemicals Pvt. Ltd Mumbai, with a manufacturer's stated purity of 0.99 mol fraction. The thermostatic bath method was to determine solubility values. For each solubility test, about 100 mL of butyl stearate, previously saturated with distilled water, was taken in a separating funnel, and 100 mL of a solution of the hydrotrope of known concentration was added. The separating funnel was sealed to avoid evaporation of mixtures at higher temperatures. The solution of different concentrations of the hydrotrope was prepared by dilution with distilled water. The separating funnel was immersed in a constant-temperature bath fitted with a temperature controller which could control the temperature within ±0.1 °C. The setup was kept overnight for equilibration. After equilibrium was attained, the aqueous layer was carefully separated from the ester layer and the layer was analyzed by UV spectrophotometer at 375nm. All the solubility experiments were conducted in duplicate to check the reproducibility. The observed error was ≤2%. The experimental setup for the determination of the mass transfer coefficient consisted of a vessel provided with baffles and a turbine impeller run by a motor to agitate the mixture. The speed of the impeller in revolutions per minute was selected in such a way to get effective mixing, which was maintained at the same value for all experiments. The experimental procedure used for the determination of the transport coefficient is a well-adopted one. The vessel used for mass transfer studies is of height 40 cm and of inner diameter 15 cm. The turbine impeller diameter is 5 cm; the width is 1 cm; and the length is 1.2 cm. It has 4 blades. The baffle is 40 cm high with a diameter of 1.5 cm. There are 4 baffles that rotate at a speed of 600 rpm. For each run to measure the mass-transfer coefficient, 250 mL of the butyl stearate previously saturated with distilled water was added to the hydrotrope solution of known concentration. The sample was then agitated for a known time of (600, 1200, 1800, and 2400) s after the end of fixed time *t*.

Table 2. Effect of Urea Concentration (*c*) on the Solubility (*S*) of Butyl stearate in Water

C	10^6S/mol ·L^{-1}			
mol ·L^{-1}	T = 303 K	T = 313 K	T = 323 K	T = 333 K
0.2	4.77	5.36	5.62	5.79
0.4	4.96	6.18	6.13	6.88
0.6	5.12	6.47	6.98	7.37
0.8	6.11	6.92	7.42	7.87
1	5.87	7.18	7.69	8.33
1.2	6.12	7.67	8.22	9.88
1.4	6.92	8.18	9.13	10.89
1.6	7.18	8.92	10.45	11.78
1.8	7.67	9.06	10.98	12.98
2	9.26	10.58	11.77	13.12
2.2	9.72	11.42	12.08	14.56
2.4	10.06	11.56	12.77	15.33
2.6	10.21	11.98	12.93	15.41
2.8	10.34	11.99	12.95	15.49
3	10.39	12.03	12.99	15.67

Table 3. Effect of Nicotinamide Concentration (*c*) on the Solubility(*S*) of Butyl stearate in Water

C	10^6S/mol ·L^{-1}			
mol ·L^{-1}	T = 303 K	T = 313 K	T = 323 K	T = 333 K
0.2	5.01	5.65	5.89	6.29
0.3	5.13	5.92	6.04	6.34
0.4	5.47	6.13	6.75	7.33
0.6	6.32	7.86	7.99	8.33
0.8	7.18	8.43	8.88	9.23
1	8.52	9.52	10.52	11.022
1.2	9.26	10.55	11.55	13.56
1.4	10.12	11.02	14.58	15.78
1.6	11.36	13.56	16.12	19.34
1.8	11.92	15.67	17.45	23.84
2	12.56	16.77	18.26	26.78
2.2	13.12	18.55	21.88	29.51
2.4	15.17	21.02	25.89	32.47
2.6	15.19	21.33	25.97	32.61
2.8	15.22	21.88	26.01	32.67
3	15.37	21.99	26.1	32.73

The entire mixture was transferred to a separating funnel. After allowed to stand for 1 hour, the aqueous layer was carefully separated from the butyl stearate layer. The concentration of the solubilized butyl stearate in aqueous hydrotrope solutions at time *t* was analyzed as done for solubility determinations. A plot of -log [1-C_b/c^*] versus *t* is drawn, where $\mathbf{C_b}$ is the concentration of solute at time $\boldsymbol{t}$ and $\boldsymbol{c^*}$ is the equilibrium solubility of solute at the same hydrotrope concentration. The slope of the graph gives $\boldsymbol{k_{La}/2.303}$, from which $\boldsymbol{k_{La}}$, the mass-transfer coefficient, was determined. Duplicate runs were made to check the reproducibility. The observed error was ≤ 2 %.

Table 4. Effect of Sodium salicylate Concentration (*c*) on the Solubility (*S*) of Butyl stearate in Water

C mol ·L^{-1}	10^6S/mol ·L^{-1}			
	T = 303 K	T = 313 K	T = 323 K	T = 333 K
0.2	6.37	6.73	6.95	7.48
0.4	6.92	7.49	7.02	8.89
0.6	7.58	8.56	8.12	9.88
0.8	8.12	9.32	10.49	11.58
1	9.56	10.56	14.52	15.86
1.2	9.72	11.42	16.58	17.99
1.4	10.26	12.56	18.12	20.36
1.6	11.45	13.62	22.58	25.62
1.8	14.38	15.89	25.67	28.94
2	15.21	17.81	28.72	36.38
2.2	19.03	22.15	30.16	39.18
2.4	19.33	22.55	30.22	39.24
2.6	19.42	22.71	30.43	39.59
2.8	19.55	22.87	30.88	39.77
3	19.6	22.99	30.91	39.85

3. Results and Discussion

3.1. Solubility

The solubility of the butyl stearate standard in the absence of any hydrotrope in water is 0.84* 10^{-6} mol ·L^{-1} at 303 K, compared to 1.3*10^{-6}mol ·L^{-1} as reported by Dean (1987). Thus, the solubility values in water are in excellent agreement with earlier reported values.[24-25]

Experimental data representing the average of duplicate determinations on the effect of hydrotropes, i.e., Citric acid, urea, nicotinamide, sodium salicylate, on the solubility of butyl stearate are presented in Tables 1 to 4 and are plotted in Figures 1 to 4. Nicotinamide is one of the hydrotropes used in this study. It has been observed that the solubility values increase significantly only after the addition of 0.40 mol ·L-1 of nicotinamide in the aqueous phase. This concentration is referred to as the Minimum Hydrotrope Concentration (MHC).

Therefore, it is evident that hydrotropic solubilization is displayed only above the MHC, irrespective of system temperature. Hydrotropy does not seem to be operative below MHC, which may be a characteristic of a particular hydrotrope with respect to each solute. This MHC value assumes greater significance in the context of recovery of hydrotrope solutions. Since hydrotropy appears to operate only at significant concentrations of hydrotrope in water, most hydrotropic solutions release the dissolved solute on dilution with distilled water below MHC. The knowledge of MHC values is necessary especially at industrial levels, as it ensures ready recovery of hydrotrope for reuse. The MHC values remained unaltered even at increased system temperatures.

The solubilization effect varies with concentration of hydrotropes (Table 1). In the present case, a clear increasing trend in the solubility of butyl stearate was observed above the MHC of nicotinamide. This increasing trend is maintained only up to a certain concentration of nicotinamide in the aqueous phase, Beyond which there is no appreciable increase in the solubility of butyl stearate. This concentration of nicotinamide (hydrotrope) in the aqueous phase is referred to as the maximum hydrotrope concentration (*c*max). From the analysis of the experimental data, it is observed that a further increase in hydrotrope concentration beyond *c*max does not bring any appreciable increase in the solubility of butyl stearate even up to 3.00 mol ·L^{-1} of nicotinamide in the aqueous phase. Similar to the MHC values, the c_{max} values of hydrotropes also remained unaltered with an increase in system temperature.

Table 5. MHC and C_{max} Values of Hydrotropes

Hydrotropes	MHC mol ·L^{-1}	C_{max} mol ·L^{-1}
Citric acid	0.4	2.4
Urea	0.2	2.4
Nicotinamide	0.3	2.4
Sodium salicylate	0.2	2.2

Table 6. Maximum Enhancement Factor for Solubility (φs) of Butyl stearate

Hydrotropes	Maximum enhancement factor for solubility (φ_S)			
	303 K	313 K	323 K	333 K
Citric acid	1.97	2.338	2.787	3.553
Urea	2.636	2.97	3.254	3.888
Nicotinamide	3.917	5.4	6.596	8.228
Sod.salicylate	4.985	5.6911	7.684	9.929

The knowledge of MHC and cmax values of each hydrotrope with respect to a particular solute assumes greater significance in this study since it indicates the beginning and saturation of the solubilization effect of hydrotropes. The values of MHC and cmax of a hydrotrope with respect to butyl stearate may be useful in determining the recovery of the solute even to an extent of the calculated amount from hydrotrope solutions at any concentration between MHC and cmax by simple dilution with distilled water. This is the unique advantage of the hydrotropic solubilization technique.

From the experimental data plotted in Figure 1, it can further be observed that, to achieve the particular solubility of say 6.35*10^{-6} mol ·L^{-1}, the citric acid.

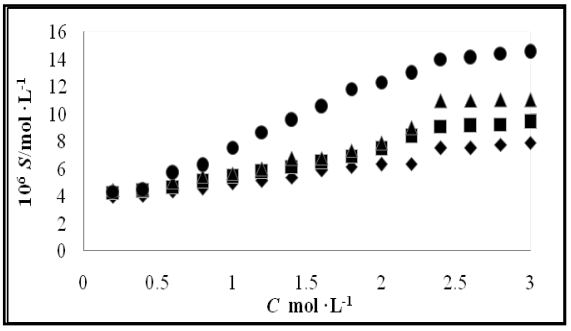

Figure 1. Effect of citric acid concentration (*c*) on the solubility (*S*) of butyl stearate in water at different temperatures: [●, T=303 K, ▲, T=313 K, ■, T=323 K, and ♦, T=333 K.]

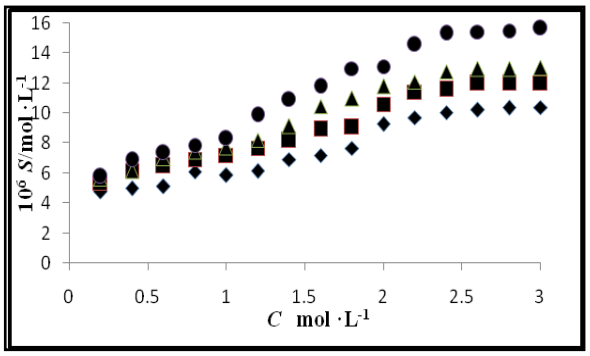

Figure 2. Effect of urea concentration (*c*) on the solubility (*S*) of butyl stearate in water at different temperatures: [●, T=303 K, ▲, T=313 K, ■, T=323 K, and ♦, T=333 K.]

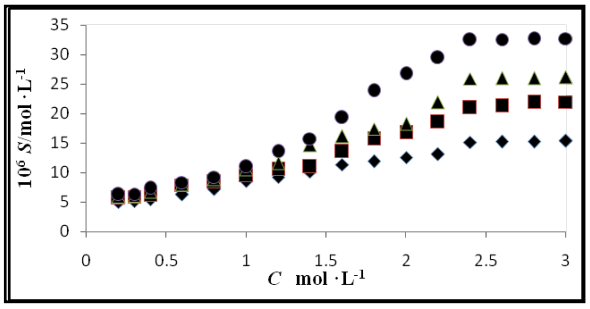

Figure 3. Effect of nicotinamide concentration (*c*) on the solubility (*S*) of butyl stearate in water at different temperatures: [●, T=303 K, ▲, T=313 K, ■, T=323 K, and ♦, T=333 K.]

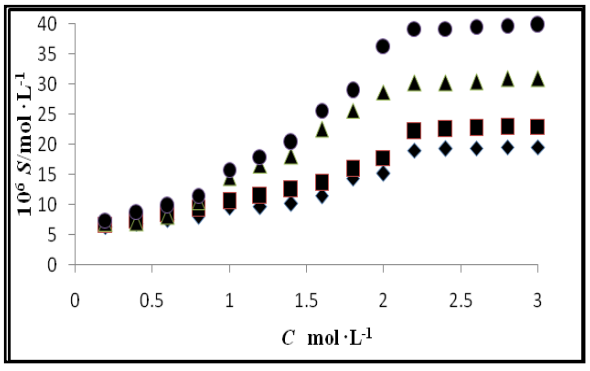

Figure 4. Effect of sodium salicylate concentration (*c*) on the solubility (*S*) of butyl stearate in water at different temperatures: [●, T=303 K, ▲, T=313 K, ■, T=323 K, and ♦, T=333 K

Concentration should be 2.60 mol ·L^{-1} at 303 K, 1.60 mol ·L^{-1} at 313 K, 1.40 mol ·L^{-1} at 323 K, and 1.00 mol ·L^{-1} at 333 K in the aqueous phase. Thus, it can be seen that as the system temperature increases the concentration of sodium salicylate required in the aqueous phase to achieve a particular solubility of butyl stearate decreases. A similar trend has been observed for other systems also.

In the concentration range of nicotinamide between (0.00 and 3.00) mol ·L^{-1}, three different regions of nicotinamide as hydrotrope were observed. It was inactive below MHC values of 0.40 mol ·L^{-1}, above which an appreciable increase in the solubility of butyl stearate was found up to 2.40 mol ·L^{-1} and beyond which there is no further increase in the solubility even up to 3.00 mol ·L^{-1}. Hence sodium salicylate was found to be an effective hydrotrope in the concentration range between (0.40 and 2.40) mol ·L^{-1} toward butyl stearate. It has also been observed that the solubilization effect of butyl stearate was not a linear function of the concentration of the nicotinamide solution. The solubilization effect of sodium salicylate increases with increase in hydrotrope concentration and also with system temperature.

A similar trend has been observed in the solubilization effect of other hydrotropes, namely, citric acid, urea, nicotinamide, and sodium salicylate. It has also been observed that the MHC values of hydrotrope used in this work range between (0.40 and 0.60) mol ·L^{-1} (Table 5) which seem to depend on the hydrophilicity of a hydrotrope. In most of the cases as reported in the recent articles the C_{max} values of hydrotropes are also viably ranges between (2.20 to 2.40) mol ·L^{-1} respectively (Table 5) . The highest value of solubilization enhancement factors φ_s, which is the ratio of solubility values in the presence and absence of a hydrotrope, has been observed in the case of sodium salicylate as 9.92 at a system temperature of 333 K (Table 6).

3.2. Mass-Transfer Coefficient

The mass-transfer coefficient of the butyl stearate + water system in the absence of any hydrotrope was determined to be 0.084*10^{-6} s^{-1} at 303 K (Table 7). The effect of different hydrotropes on the mass-transfer coefficient of butyl stearate at different hydrotrope concentrations is also given in the same table. It can be seen that a threshold value of 0.40 mol ·L^{-1} is required to effect significant enhancement in the mass transfer coefficient of the butyl stearate + water system, as was observed in the mass-transfer coefficient of the butyl stearate + water system in the presence of sodium salicylate was found to be 20.06 (Table 7). A similar trend in the mass-transfer coefficient enhancement (φ_{mtc}) of butyl stearate has been observed for other hydrotropes also, namely citric acid, urea, nicotinamide and sodium salicylate. The highest value of φ_{mtc} (20.06) has been observed in the presence of sodium salicylate under a wide range of hydrotrope concentrations [(0 to 3.0) mol ·L^{-1}] and different system temperatures [(303 to 333) K] have been determined and presented in Table 8.

3.3. Aggregation Characteristics of Hydrotropes

The change in enthalpy, entropy, and free energy accompanying the aggregation of hydrotropes such as nicotinamide, sodium salicylate, resorcinol, and sodium citrate under a wide range of hydrotrope concentrations [(0 to 3.0) mol $\cdot L^{-1}$] and different system temperatures [(303 to 333) K] have been determined and presented in Table 8. The calculations are based on MHC as determined in solubility studies.

$$\Delta G^{\circ} = RT \ln (X_{MHC}) \quad (1)$$

Where X_{MHC} = solubility of butyl stearate at MHC in mol $\cdot L^{-1}$.

Figure 5 and Figure 6 shows the relationship between the standard free energy of both the hydrotropes and the temperature effects respectively. The free energy decreases with increase in temperature as reported in Table 8.

The standard enthalpy (ΔH^{o}) of aggregation can be found by the Van't Hoff equation

$$\Delta H^{\circ} = -RT^{2} (\partial \ln X_{MHC}/\partial T) \quad (2)$$

The slope in the plot of ln (X_{MHC}) versus T at each temperature was taken as (∂ ln $X_{MHC}/\partial T$). A linear plot was observed for both the hydrotropes as shown in Figure 7. The values of enthalpy are negative which indicates the aggregation behavior of exothermic nature

The standard entropy (ΔS^{o}) of aggregation was calculated from

$$\Delta S^{\circ} = [(\Delta H^{\circ} - \Delta G^{\circ})/T] \quad (3)$$

The entropy change in all cases is positive which confirms that aggregation of hydrotropes is favored entropically. However, the values decrease with increasing temperature as seen from Table 8. This may be due to the fact that self-aggregation becomes poor at higher temperature because of enhanced molecular motion at increased temperatures.

3.4. Effectiveness of Hydrotropes

The effectiveness factor of each hydrotrope with respect to butyl stearate at different system temperatures has been determined by analyzing the experimental solubility data for each case applying the model suggested by Setschenow and later modified by Pathak and Gaikar (1992),as given by the equation as follows

$$\text{Log } [S/S_m] = K_s [C_s - C_m] \quad (4)$$

Where S, is the solubility of butyl stearate at any hydrotrope concentration c_s and S_m is the minimum hydrotrope concentration respectively. The Setschenow constant K_s can be considered as a measure of the effectiveness of a hydrotrope at any given conditions of hydrotrope concentration and system temperature. The Setschenow constant values of hydrotropes, namely citric acid, urea, nicotinamide, sodium salicylate for the butyl stearate + water system at different system temperatures is listed in Table 9. The highest value has been observed as 0.360 in the case of sodium salicylate as hydrotrope at 333 K.

Table 7. Effect of Hydrotrope Concentration (c) on the Mass Transfer Coefficient (k_{La}) of Butyl stearate at 303 K

Hydrotropes	C mol·L^{-1}	k_{La} 10^{6} s^{-1}	Enhancement factor for mass transfer coefficient(φ_{mtc})
Citric acid	0.0	0.0084	-
	1.0	0.0557	6.6
	2.0	0.076	9.40
	3.0	0.115	13.69
Urea	0.0	0.0084	-
	1.0	0.037	4.4
	2.0	0.044	5.38
	3.0	0.053	6.30
Nicotinamide	0.0	0.0084	-
	1.0	0.071	8.45
	2.0	0.122	14.52
	3.0	0.146	17.38
Sodium salicylate	0.0	0.0084	-
	1.0	0.0852	10.11
	2.0	0.1373	16.35
	3.0	0.1685	20.06

Table 8. Effect of Minimum Hydrotrope Concentration (MHC), Standard Gibbs free energy (ΔG^{o}), Standard Enthalpy (ΔH^{o}) and Entropy (ΔS^{o}) of Butyl stearate

Hydrotropes	T, K	ΔG KJ/mol	ΔH KJ/mol	ΔS KJ/mol
Citric acid	303	-31.28	-1.52	0.098
	313	-32.11	-4.58	0.097
	323	-33.07	-4.58	0.097
	333	-34.13	-3.82	0.097
Urea	303	-30.86	-1.62	0.086
	313	-31.59	-4.88	0.085
	323	-32.46	-4.90	0.084
	333	-33.39	-4.07	0.083
Nicotinamide	303	-30.68	-1.73	0.086
	313	-31.33	5.20	0.084
	323	-32.28	-5.21	0.083
	333	-33.14	-4.34	0.082
Sodium salicylate	303	-30.13	-1.84	0.086
	313	30.99	-5.53	0.086
	323	-31.90	-5.53	0.085
	333	-32.67	-4.61	0.084

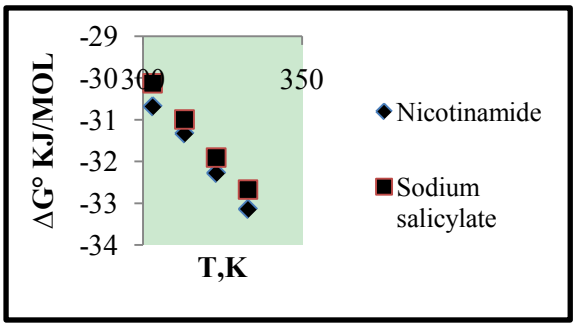

Figure 5. Temperature (T) versus standard Gibbs free energy (ΔG^{o}) ♦, nicotinamide; ■, sodium Salycilate

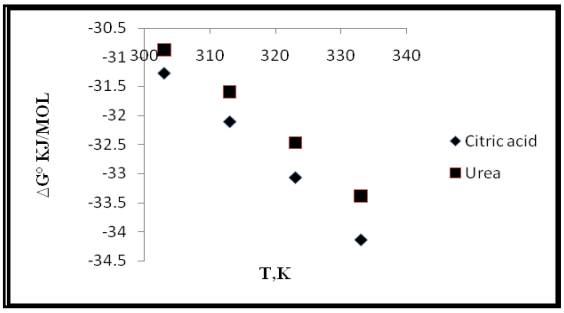

Figure 6. Temperature (T) versus standard Gibbs free energy (ΔG^{o}): ♦, urea; ■, citric acid

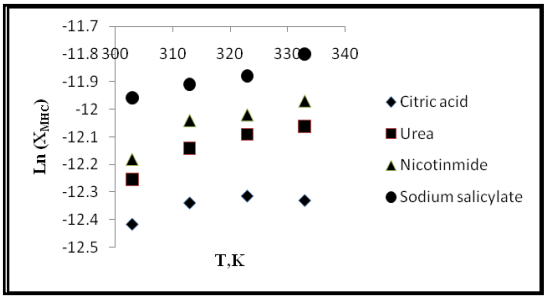

Figure 7. Temperature (T) versus ln (X_{MHC}): [♦, citric acid, ■ urea, ▲ nicotinamide, ● sodium salicylate]

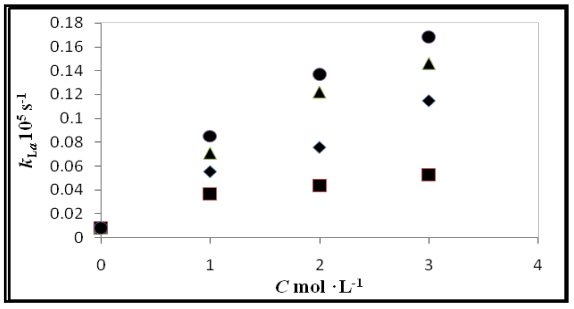

Figure 8. Effect of hydrotrope concentration (c) on the mass transfer coefficient (k_{La}) of butyl stearate in water at 303 K:[♦, citric acid, ■ urea, ▲ nicotinamide, ● sodium salicylate]

Table 9. Setschenow Constant (k_s) of Hydrotropes with Respect to Butyl stearate

Hydrotropes	Setschenow Constant (k_s)			
	T = 303 K	T = 313 K	T = 323 K	T = 333 K
Citric acid	0.1344	0.1588	0.1934	0.2502
Urea	0.1473	0.1512	0.162	0.1922
Nicotinamide	0.2242	0.2621	0.301	0.3378
Sodium salicylate	0.2376	0.2587	0.32187	0.3605

4. Conclusions

The solubility of butyl stearate which is practically insoluble in water has been increased to a maximum enhancement factor value of 20.06 in the presence of sodium salicylate as hydrotrope with a corresponding increase in the mass transfer coefficient. This would be useful in increasing the rate of output of the desired product made from butyl stearate. Solubility is found useful in the case of hydrotropes to study thermodynamic stability. From the data obtained by this study, it is found that hydrotrope concentration gives self-aggregation at higher minimum concentration compared to Micellar surfactants. The MHC and c_{max} values of the hydrotrope with respect to butyl stearate can be used for the recovery of the dissolved butyl stearate and hydrotrope solutions at any hydrotrope concentration between the MHC and c_{max} by simple dilution with distilled water. This will eliminate the huge cost and energy normally involved in the separation of the solubilized solute from its solution. The unprecedented increase in the solubilizing effect of hydrotropes is attributed to the formation of organized aggregates of hydrotrope molecules at a particular concentration.

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