Enhancement of Solubility and Mass Transfer Coefficient Through Hydrotropy

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Abstract: This paper presents a comprehensive study on the effect of hydrotropes on the solubility and mass transfer coefficient of ethyl acetate in water. The solubility and mass transfer studies were performed using hydrotropes such as tri-sodium citrate, urea, sodium benzoate, and sodium salicylate under a wide range of concentrations (0 to 3.0 mol/L) and system temperatures (303 to 333 K). The performance of the hydrotropes was measured in terms of the Setschenow constant (K_s). It was found that the solubility of ethyl acetate increases with increase in hydrotrope concentration and also with system temperature. A Minimum Hydrotrope Concentration (MHC) in the aqueous phase was found required to initiate significant solubilization of ethyl acetate. Consequent to the increase in solubilization of ethyl acetate, the mass transfer coefficient was also found to increase with increase in hydrotrope concentration. All hydrotropes used in this work showed an enhancement in the solubility and mass transfer coefficient to different degrees. The maximum enhancement factor, which is the ratio of the value in presence and absence of a hydrotrope, has been determined for both solubility and mass transfer coefficient. These characteristics would be much useful in increasing the rate of output of the desired product made from ethyl acetate. In addition, the separation of ethyl acetate from any liquid mixture which is found to be difficult can be carried out effectively using this technique.

Keywords: hydrotropy; solubilization; mass transfer co-efficient; Separation

1. Introduction

Hydrotropes are a class of chemical compounds that effect a several fold increase in the solubility for sparingly soluble solute under normal conditions. This phenomenon, termed hydrotropy can be considered to be a unique and unprecedented solubilization technique. The origin of hydrotropy dates back to 1916 when Neuberg identified this pioneering technique for effecting very large solubility enhancements for a variety of sparingly soluble organic compounds [1]. Hydrotropes in general are water-soluble and surface-active compounds, which can significantly enhance the solubility of organic solutes such as esters, alcohols, aldehydes, ketones, hydrocarbons, and fats [2, 3, 4, 5].

Hydrotropes are widely used in drug solubilization, detergent formulation, health care, and household applications [6] as well as for being an extraction agent for fragrances [7]. This potentially attractive technique can also be adopted to separate close boiling point isomeric and non-isomeric mixtures. At the same time, the problem of emulsification,

which is normally encountered with conventional surfactant solution is not found with hydrotrope solutions [8]. Hydrotropes have been used to increase the rate of heterogeneous reactions and also for the separation of close boiling liquid mixtures through extractive distillation and liquid-liquid extraction.[9, 10]

Each hydrotrope has a selective ability towards a particular component in the mixture which facilitates easy recovery of the hydrotrope solution by controlled dilution with distilled water [10, 11]. The solubility enhancement in the organic compounds could be due to the formation of molecular structures in the form of complexes [12]. Previous experimental findings have concluded that hydrotropy is a process which goes beyond conventional solubilization methods such as miscibility, co-solvency, and the salting-in effect, since the solubilization effected by hydrotropy was higher and more selective compared to other solubilization techniques [13, 14]. The effect of hydrotropes on the solubility and mass transfer coefficient for a series of organic esters such as butyl acetate, ethyl benzoate, amyl acetate, methyl Salicylate and benzyl acetate were studied in our earlier publications [15-19].

For many binary systems involving sparingly soluble organic compounds such as ethyl acetate, mass transfer coefficient in the presence of a hydrotrope is probably due to the difference between the binary diffusivity (solute + solvent) and the diffusivity of the solute in the solution (solute + solvent + hydrotrope). Ethyl acetate serves as a raw material and intermediate for a wide variety of chemicals, drugs, and allied products such as scabicide. In addition, the separation of ethyl acetate from any liquid mixture is found to be difficult; hence, this hydrotropic technique can be adopted to increase the solubility as well as to separate such mixtures effectively. The hydrotropes used in this work are freely soluble in water and practically insoluble in ethyl acetate. All are non-reactive, non-toxic, and do not produce any significant heat effect when dissolved in water.

2. Experimental Section

All the chemicals used in this work were manufactured by Himedia Chemicals, Mumbai with the manufacturer's stated purity of 99%.

The experimental setup used for the determination of solubility values consisted of a thermostatic bath and a separating funnel. For each solubility test, about 100 ml of ethyl acetate previously saturated with distilled water was taken in a separating funnel and 100 ml of a hydrotrope solution of known composition was added. The separating funnel was sealed to avoid evaporation of the solvent at higher temperatures. The hydrotrope solutions of different concentrations were prepared by dilution with distilled water.The separating funnel was immersed in a constant temperature bath fitted with a temperature controller that could control the temperature within ± 0.1 °C. The setup was kept overnight for equilibration. After the equilibrium was attained, the aqueous layer was carefully separated from the ester layer and transferred into a beaker. The ester concentration was estimated by the addition of excess NaOH and titrated against standardized HCl solution and phenolphthalein as an indicator. All the solubility experiments were conducted in duplicate to check the reproducibility. The observed error in the reproducibility is <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 rpm 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 four blades. The baffle is 40 cm high with a diameter of 1.5 cm. There are about four baffles that rotate at a speed of 600 rpm.

For each run to measure the mass-transfer coefficient, 250 ml of the ester 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 seconds. After the end of fixed time t, the entire mixture was transferred to a separating funnel. After allowing to stand for some time, the aqueous layer was carefully separated from the ester layer. The concentration of the solubilized organic ester 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 C_b is the concentration of methyl benzoate at time t and C^* is the equilibrium solubility of methyl benzoate at the same hydrotrope concentration [2]. The slope of the graph gives $k_La/2.303$, from which k_La, the mass-transfer coefficient was determined. Duplicate runs were made to check the reproducibility. The observed error was <2%.

3. Results and Discussion

3.1. Solubility

The solubility of ethyl acetate in water at 303 K is 0.92 mol/L Tables 1 to 4, which is in close agreement with earlier reported values [20, 21].

Experimental data on the effect of hydrotropes, i.e. tri-sodium citrate, sodium benzoate, sodium salicylate and urea on the solubility of ethyl acetate are presented in Tables 1 to 4 and are plotted in Figures 1 to 4. Urea is a potential hydrotrope which has both polar and non-polar groups. It is one of the hydrotropes used in this study. It was observed that the aqueous solubility of ethyl acetate did not show any appreciable increase even after the addition of 0.30 mol/L of urea in the aqueous phase. Upon subsequent increase in the concentration of urea above 0.30 mol/L the solubility of ethyl acetate in water was found to increase significantly. This concentration of urea in the aqueous phase, 0.3 mol/L, is termed the Minimum Hydrotrope Concentration (MHC) which is the minimum required amount of urea (hydrotrope) concentration in the aqueous phase to cause a significant increase in the solubility of ethyl acetate in water. It has been observed that the MHC of urea in the aqueous phase does not vary even at high temperatures i.e. 303, 313, 323, 333 K. A similar trend in the MHC requirement in the aqueous phase has been observed for other hydrotropes also. Therefore it is evident that hydrotropic solubilization is displayed only above the MHC irrespective of the temperature. Hydrotropy does not seem to be operative below the MHC, which may be a characteristic of a particular hydrotrope with respect to each solute.

The solubilization effect varies with the concentration of hydrotropes Tables 1 to 4. In the present case, a clear increasing trend in the solubility of ethyl acetate was observed above the MHC of urea. This increase in solubility is maintained only up to a certain concentration of urea in the aqueous phase beyond which there is no appreciable increase in the solubility of ethyl acetate.

This concentration of urea (hydrotrope) in the aqueous phase is referred to as the maximum hydrotrope concentration (C_{max}). As can be seen from Table 5, the C_{max} values of urea, sodium Salicylate, tri-sodium citrate, and sodium benzoate with respect to ethyl acetate are (2.00, 2.25, 2.00, 2.50) mol/L respectively. From the analysis of the experimental data, it is observed that further increase in the hydrotrope concentration beyond C_{max} does not cause any appreciable increase in the solubility of ethyl acetate even up to 3.00 mol/L urea is the aqueous phase. Similar to the MHC values, the C_{max} values of hydrotropes also remained unaltered with an increase in the

system temperature.

Table 1. Effect of Tri sodium citrate concentration(C) on solubility(S) of ethyl acetate in water.

SI. No	C, mol/L	S, mol/L			
		T = 303 K	T = 313 K	T = 323 K	T = 333 K
1	0.00	0.92	0.72	0.73	0.71
2	0.10	0.83	0.78	0.74	0.75
3	0.20(MHC)	1.01	0.79	0.74	0.78
4	0.30	1.56	1.70	1.77	1.82
5	0.40	1.65	2.67	3.58	3.2
6	0.50	2.05	3.11	4.22	5.95
7	0.60	2.29	3.57	5.16	7.32
8	0.70	2.84	4.48	6.13	8.78
9	0.80	3.29	4.94	7.00	10.00
10	0.90	4.00	6.13	8.00	11.16
11	1.00	4.66	7.13	9.15	12.80
12	1.20	5.67	9.05	12.07	15.91
13	1.40	7.16	11.07	15.38	19.21
14	1.60	8.41	13.63	18.93	22.67
15	1.80	10.15	17.38	21.13	24.36
16	2.00(C_{max})	11.16	19.39	22.31	24.59
17	2.25	11.44	19.6	22.38	24.67
18	2.50	11.46	19.63	22.32	24.63
19	2.75	11.48	19.66	22.29	24.68
20	3.00	11.47	19.69	22.29	24.65

Table 2. Effect of Sodium Salicylate concentration(C) on solubility(S) of ethyl acetate in water.

SI. No.	C, mol/L	S, mol/L			
		T = 303 K	T = 313 K	T = 323 K	T = 333 K
1	0.00	0.92	0.87	0.81	0.82
2	0.10	0.94	0.89	0.81	0.82
3	0.20	1.04	0.90	0.84	0.82
4	0.30	0.87	0.92	1.13	0.93
5	0.40	0.9	0.94	1.07	0.93
6	0.50	0.88	0.96	1.10	0.96
7	0.60	0.93	0.90	1.18	1.07
8	0.70	0.90	0.93	1.32	1.04
9	0.80	0.94	0.92	1.54	1.18
10	0.90	0.94	1.07	1.67	1.34
11	1.00(MHC)	0.93	1.15	1.98	1.43
12	1.20	1.10	1.45	2.60	1.87
13	1.40	1.34	1.89	3.38	2.60
14	1.60	1.69	2.40	4.58	3.35
15	1.80	2.20	3.32	5.80	4.45
16	2.00	2.72	4.36	6.80	5.54
17	2.25(C_{max})	3.40	5.05	7.54	6.45
18	2.50	3.51	5.10	7.60	6.48
19	2.75	3.54	5.10	7.65	6.50
20	3.00	3.54	5.08	7.63	6.50

Table 3. Effect of Urea concentration(C) on solubility(S) of ethyl acetate in water.

SI. No.	C, mol/L	S, mol/L			
		T = 303 K	T = 313 K	T = 323 K	T = 333 K
1	0.00	0.92	0.87	0.81	0.72
2	0.10	0.94	0.85	0.81	0.77
3	0.20	0.91	0.89	0.84	0.74
4	0.30(MHC)	0.95	0.91	0.89	0.93
5	0.40	1.14	1.20	1.38	2.30
6	0.50	1.43	1.84	2.58	2.91
7	0.60	1.65	2.14	2.96	3.84
8	0.70	2.03	2.52	3.68	4.17
9	0.80	2.25	3.24	4.24	5.05
10	0.90	2.63	3.68	4.72	5.93
11	1.00	2.96	4.17	5.71	7.20
12	1.20	3.72	5.71	7.20	8.89
13	1.40	4.84	7.20	9.27	11.85
14	1.60	6.23	9.27	11.20	14.05
15	1.80	7.41	10.98	13.12	15.04
16	2.00(C_{max})	8.14	12.00	14.32	15.78
17	2.25	8.09	12.07	14.40	15.87
18	2.50	8.15	11.91	14.21	15.89
19	2.75	8.15	11.96	14.21	15.97
20	3.00	8.15	11.91	14.32	15.95

Table 4. Effect of Sodium benzoate concentration(C) on solubility(S) of ethyl acetate in water

SI. No	C, mol/L	S, mol/L			
		T = 303 K	T = 313 K	T = 323 K	T = 333 K
1	0.00	0.92	0.87	0.81	0.83
2	0.10	0.96	0.87	0.79	0.86
3	0.20	0.99	0.90	0.85	0.88
4	0.30	0.95	0.89	0.88	0.92
5	0.40	0.98	0.91	0.87	0.95
6	0.50(MHC)	1.01	0.94	0.91	1.65
7	0.60	1.32	1.56	1.78	3.11
8	0.70	1.63	2.19	2.04	4.57
9	0.80	2.07	2.65	3.20	5.67
10	0.90	2.29	3.11	4.30	7.00
11	1.00	2.67	3.93	5.30	8.00
12	1.20	3.59	5.60	7.32	10.43
13	1.40	4.94	7.32	9.42	12.62
14	1.60	6.31	8.87	11.62	15.09
15	1.80	7.64	11.25	14.00	18.20
16	2.00	9.24	13.63	17.20	21.68
17	2.25	11.00	17.47	21.49	25.43
18	2.50(C_{max})	11.82	18.81	22.5	26.25
19	2.75	11.79	18.84	22.59	26.34
20	3.00	11.89	18.93	22.59	26.25

Table 5. Minimum Hydrotrope Concentration (MHC) and Maximum Hydrotrope Concentration (C_{max}) values for Hydrotropes

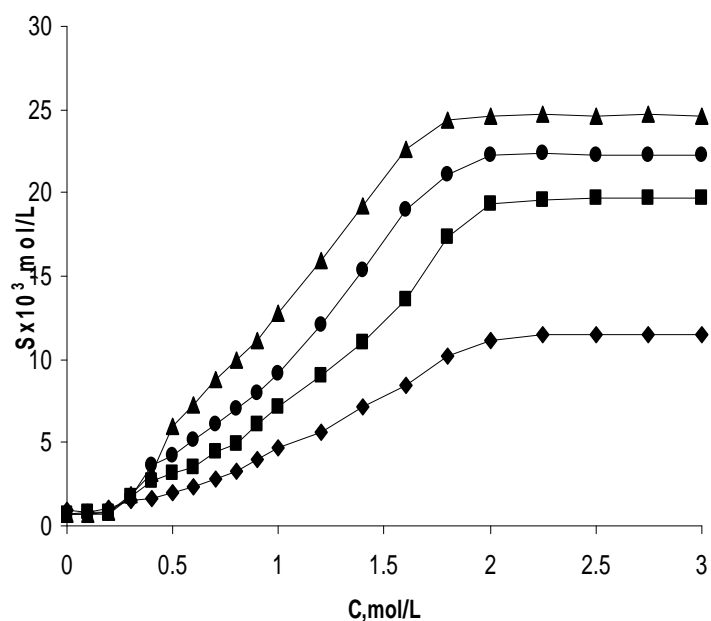| Hydrotrope	MHC mol/L	C_{max} / mol/L
Tri sodium citrate	0.2	2.00
Sodium salycilate	1.0	2.25
Urea	0.3	2.00
Sodium benzoate	0.5	2.50

Figure 1. Effect of Tri Sodium Citrate concentration (C) on the solubility (S) of Ethyl Acetate in water at
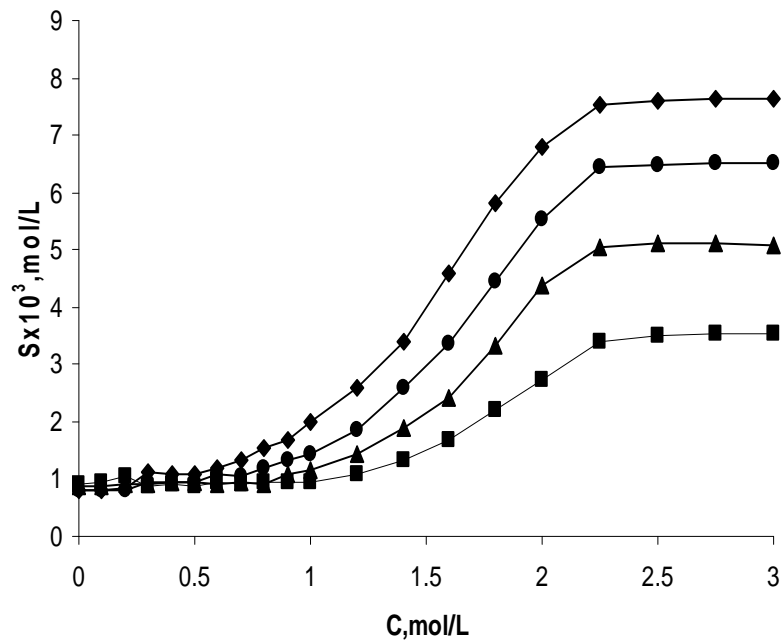303k 313k 323k 333k

Figure 2. Effect of Sodium Salicylate concentration (C) on the solubility (S) of Ethyl acetate in water at
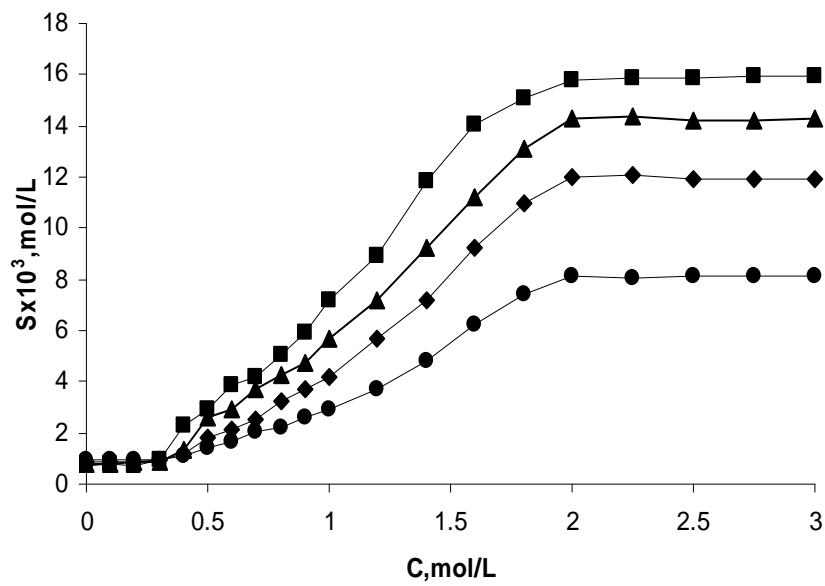—■— 303k —▲— 313k —◆— 323k —●— 333k

Figure 3. Effect of Urea concentration (C) on the solubility (S) of Ethyl acetate in water at
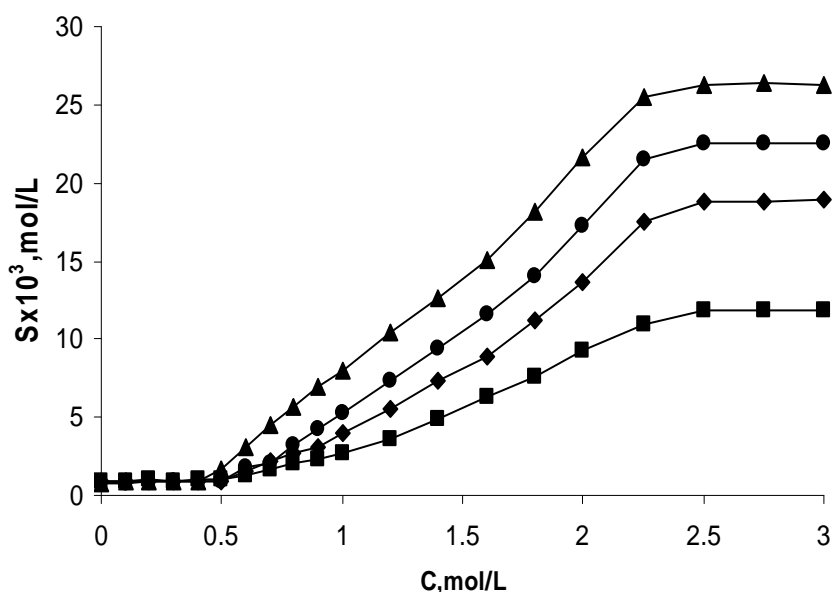—●— 303k —◆— 313k —▲— 323k —■— 333k

Figure 4. Effect of Sodium Benzoate concentration (C) on the solubility (S) of Ethyl acetate in water at

■ 303k   ◆ 313k   ● 323k   ▲ 333k

It appears that the solute molecules after finding their way through the interface of hydrotrope aggregates are held hidden in the hydrotropic stock; however, this arrangement seems to be a purely temporary one, because the solute particles contained within the hydrotrope stack can be brought out by simple dilution with distilled water, which alters the solution properties of the hydrotrope stack. This causes the dissociation of hydrotropic molecules and the properties of hydrotrope solutions with decrease in hydrotrope concentration approach to that of water, similar to the situation below MHC. This phenomenon was observed experimentally by release of the dissolved solute from the hydrotrope solution at any concentration between the MHC and C_{max} by simple dilution with distilled water and hence possible reuse of the hydrotrope solution. This is the unique advantage of the hydrotropic solubilization technique.

In the concentration range of urea between (0.00 and 3.00) mol/L there different regions [first region, (0.00 to 0.3) mol/L. second region, (0.30 to 2.00) mol/L third region, (2.00 to 3.00) mol/L] were obtained when using urea as a hydrotrope. It was inactive below an MHC of 0.30 mol/L, above which an appreciable increase in the solubility of ethyl acetate was found up to a C_{max} value of 2.00 mol/L, beyond which there is no further solubilization effect of the hydrotrope. Therefore, urea was found to be an effective hydrotrope in the concentration range between (0.30 to 2.00) mol/L towards ethyl acetate. It has also been observed that the solubilization effect of urea was not having a linear function with the concentration of the urea solution. The solubilization effect of urea increase with an increase in hydrotrope concentration and also with system temperature. The maximum enhancement factor (Φ_s, ratio between the maximum solubility value (S_{max}) in the presence and the solubility value in the absence (S_0) of a hydrotrope) values observed for the ethyl acetate + water system in presence of urea as the hydrotrope at (303, 313, 323, 333) K are 8.86, 13.69, 17.68 and 22.15 respectively Table 6.

A similar trend has been observed in the solubilization effect of other hydrotropes, namely, tri-sodium citrate, sodium benzoate

and sodium Salicylate. It has also been observed that the MHC values of the hydrotropes used in this work range between (0.20 to 1.00) mol/L, which seem to depend on the hydrophilic nature of the hydrotrope. The C_{max} values of hydrotropes range between (2.00 to 2.50) mol/L Table 5 in most cases.

The maximum solubilization enhancement factor (Φ_s) effected by various hydrotropes for ethyl acetate range between 3.85 and 34.72. The highest value for Φ_s (34.72) has been observed in this case of tri sodium citrate at system temperature of 333 K Table 6.

Table 6. Maximum Solubility Enhancement Factor (Φ_s) of ethyl acetate.

Hydrotrope	Maximum enhancement factor for solubility (Φ_s)			
	T = 303 K	T = 313 K	T = 323 K	T = 333 K
Tri sodium citrate	12.47	27.35	30.53	34.72
Sodium Salicylate	3.85	5.83	9.42	7.93
Urea	8.86	13.69	17.68	22.15
Sodium benzoate	12.92	21.76	27.89	31.63

3.2. Mass Transfer Coefficient

The mass transfer coefficient for the ethyl acetate + water system in the absence of any hydrotrope was determined to be 1.32×10^{-4} s^{-1} at 303 K Table 7. The effect of different hydrotropes on the mass transfer coefficient of ethyl acetate at different hydrotrope concentrations is also presented in the same Table 7. It can be seen that a threshold value of 0.20 mol/L is required for significant enhancement in the mass transfer coefficient of the ethyl acetate + water system, as observed in the case of the solubility determinations. The mass transfer coefficient of the ethyl acetate + water system increases with an increase in the hydrotrope concentration. Beyond a C_{max} value of 2.00 mol/L, there is no appreciable increase in the mass transfer coefficient of ethyl acetate as observed in the case of the

solubility determinations. The observed increase of the mass transfer coefficient in the presence of hydrotrope is probably due to the difference between the binary diffusivity (solute + solvent) and the diffusivity of the solute in the solution (solute + solvent + hydrotrope). A similar trend in the mass transfer coefficient enhancement (Φ_{mtc}) of ethyl acetate has been observed for other hydrotropes also. The maximum enhancement factor for the mass transfer coefficient (Φ_{mtc}, ratio between the mass transfer coefficient (K_{La}) values in the presence and absence of hydrotropes) values observed for the ethyl acetate + water system in the presence of various hydrotropes at 303 K are reported in Table 7. The highest values of Φ_{mtc} (22.19) has been observed in the presence of tri-sodium citrate as the hydrotrope at a C_{max} value of 2.00 mol/L.

Table 7. Enhancement Factor for the Mass Transfer Coefficient (C_b) on the Mass Transfer Coefficient (k_{La}) of ethyl acetate at a Temperature of T = 303 K.

Hydrotrope	Concentration C mol.L^{-1}	Mass transfer Coefficient (k_{La}) x 10^4 S^{-1}	Enhancement (Φ_{mtc})
Tri sodium citrate	0.00	1.32	-
	0.20(MHC)	1.46	1.10
	0.40	3.86	2.92
	0.80	8.16	6.18
	1.60	15.23	11.54
	2.00(C_{max})	29.30	22.19
	3.00	29.13	22.07
Sodium benzoate	0.00	1.32	-
	0.20	1.56	1.18
	0.50(MHC)	2.30	1.74
	0.80	4.44	3.36
	1.60	10.42	7.89
	2.50(C_{max})	21.82	16.53
	3.00	21.96	16.64
Sodium Salicylate	0.00	1.32	-
	0.20	1.43	1.08
	0.40	1.98	1.50
	1.00(MHC)	2.85	2.16
	1.60	5.22	3.95
	2.25(C_{max})	5.40	4.09
	3.00	5.50	4.17
Urea	0.00	1.32	-
	0.30(MHC)	1.43	1.08
	0.40	1.97	1.49
	0.80	6.01	4.55
	1.60	8.23	6.23
	2.00(C_{max})	13.42	10.17
	3.00	13.39	10.15

3.3. Effectiveness of Hydrotropes

  The effectiveness factor of each hydrotrope with respect to ethyl acetate 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 in ref [1], as given by the equation

$$\log[S/S_m] = K_s [C_s - C_m]$$

where S and S_m are the solubility values of ethyl acetate at any hydrotrope concentration

(C_s) and minimum hydrotrope concentration (MHC), respectively.The Setschenow constant (K_s) can be considered as a measure of the effectiveness of a hydrotrope at any given condition of hydrotrope concentration and system temperature. The Setscheow constant values of the hydrotropes, namely tri-sodium citrate, sodium benzoate, sodium Salicylate and urea, for the ethyl acetate + water system at different system temperatures are listed in Table 8.The highest value has been observed as 0.833 in case of tri-sodium citrate as the hydrotrope at 333 K.

Table 8. Setschenow Constant (K_S) of Hydrotropes with respect to ethyl acetate.

Hydrotrope	Setschenow Constant (K_S)			
	T = 303 K	T = 313 K	T = 323 K	T = 333 K
Tri sodium citrate	0.638	0.775	0.823	0.833
sodium Salicylate	0.450	0.514	0.465	0.523
Urea	0.549	0.659	0.710	0.723
Sodium benzoate	0.541	0.651	0.696	0.721

4. Conclusions

From the literature, the solubility data for ethyl acetate show that the ester is practically insoluble in water, which has been increased to a maximum value of 22.19 in the presence of tri-sodium citrate as the 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 ethyl acetate. The separation of ethyl acetate from any liquid mixture, which is found to be difficult, can be carried out effectively using this technique. The MHC and C_{max} values of the hydrotrope with respect to ethyl acetate can be used for the recovery of the dissolved ethyl acetate and hydrotrope solutions at any hydrotrope concentration between 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 ethyl acetate 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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