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Effect of Hydrotropes on the Solubility and Mass Transfer Coefficient of Benzyl Benzoate in Water

N. Meyyappan[†] **and N. Nagendra Gandhi***[,‡]

Department of Chemical Engineering, Sri Venkateswara College of Engineering, Sriperumbudur 602 105, India,
and Department of Chemical Engineering, Alagappa College of Technology, Anna University,
Chennai 600 025, India

This work presents a comprehensive study on the effect of hydrotropes on the solubility and mass transfer coefficient of benzyl benzoate in water. The solubility studies were performed using hydrotropes such as tri-sodium citrate, urea, sodium benzoate, and sodium salicylate for various concentrations in the range from (0 to 3.0) mol·L^{-1} and system temperatures ranging from T = (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 benzyl benzoate increases with an increase in hydrotrope concentration and also with system temperature. A minimum hydrotrope concentration (MHC) in the aqueous phase was required to initiate significant solubilization of benzyl benzoate. Consequent to the increase in solubilization of benzyl benzoate, the mass transfer coefficient was also found to increase with an 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 between the presence and absence of a hydrotrope, has been determined for both cases. However, a threshold value of the MHC was found essential to initiate significant increase in hydrotrope concentration.

1. Introduction

Hydrotropes are a class of chemical compounds that effect a severalfold increase in the solubility for sparingly soluble solutes 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.[2] 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.[1−4]

Hydrotropes are widely used in drug solubilization, detergent formulation, health care, and household applications[5] as well as for being an extraction agent for fragrances.[6] This potentially attractive technique can also be adopted to separate close boiling point isomeric and nonisomeric mixtures. At the same time, the problem of emulsification, which is normally encountered with conventional surfactant solution, is not found with hydrotrope solutions.[7] Hydrotropes have been used to increase the rate of heterogeneous reactions and have also been used for the separation of close boiling liquid mixtures through extractive distillation and liquid−liquid extraction.[8,9]

Each hydrotrope has a selective ability toward 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, cosolvency, 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 was studied in our earlier publications.[15−19]

For many binary systems involving a sparingly soluble organic compound such as benzyl benzoate, the 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). Benzyl benzoate 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 any liquid mixture is found 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 benzyl benzoate. All are nonreactive, are nontoxic, and do not produce any significant heat effect when dissolved in water.

2. Experimental Section

The chemicals used were checked for purity using gas chromatography and with a refractometer; that is, benzyl benzoate of 99.9% and hydrotropes such as tri-sodium citrate, urea, sodium salicylate, and sodium benzoate were of high purity grade and procured from HiMedia Chemicals, Mumbai. The experimental setup used for the determination of solubility values consisted of a thermostatic

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* Corresponding author. Fax: (91)-44-22352642. E-mail: n_nagendra2002@yahoo.com.

[†] Sri Venkateswara College of Engineering.

[‡] Alagappa College of Technology.

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Table 1. Effect of Tri-sodium Citrate Concentration (C) on the Solubility (S) of Benzyl Benzoate in Water

	10^3S/mol·L^{-1}			
C/mol·L^{-1}	T = 303 K	T = 313 K	T = 323 K	T = 333 K
0.0	6.96	7.02	7.15	7.23
0.1	7.23	7.64	7.42	7.37
0.2	7.25	7.82	7.66	8.89
0.3	8.10	9.18	10.83	13.49
0.4	11.90	13.81	14.22	16.49
0.5	14.20	17.60	24.28	28.06
0.6	15.68	27.09	35.98	43.37
0.7	18.12	32.24	45.70	61.48
0.8	19.56	36.94	52.30	77.38
0.9	21.16	39.18	60.90	92.24
1.0	24.52	43.23	69.12	105.52
1.2	30.16	48.45	81.64	123.16
1.4	37.89	57.58	92.53	129.66
1.6	43.56	61.74	99.96	137.49
1.8	48.69	68.06	106.94	141.25
2.0	55.52	74.90	112.86	144.19
2.25	57.68	87.40	120.61	146.67
2.50	62.05	94.00	126.65	150.89
2.75	62.08	94.80	126.45	151.86
3.0	61.18	94.88	126.98	151.23

Table 2. Effect of Urea Concentration (C) on the Solubility (S) of Benzyl Benzoate in Water

	10^3S/mol·L^{-1}			
C/mol·L^{-1}	T = 303 K	T = 313 K	T = 323 K	T = 333 K
0.0	6.96	7.02	7.15	7.23
0.1	7.09	7.18	7.28	7.56
0.2	7.85	7.89	8.15	8.69
0.3	8.89	9.98	10.84	11.76
0.4	11.56	12.56	14.48	15.71
0.5	15.54	17.75	24.77	26.86
0.6	17.06	26.34	36.76	39.81
0.7	21.67	33.41	46.62	50.56
0.8	24.85	38.23	53.35	57.89
0.9	28.88	44.53	62.13	67.38
1.00	32.78	50.51	70.49	76.45
1.20	38.72	59.65	83.24	90.29
1.40	43.87	67.62	88.62	102.35
1.60	47.38	73.03	94.63	110.53
1.80	50.72	78.15	101.91	118.28
2.00	53.51	82.46	109.96	124.81
2.25	57.25	88.17	117.39	133.44
2.50	60.07	92.59	123.61	140.56
2.75	60.11	92.66	123.89	140.26
3.00	60.23	92.85	123.96	140.54

Table 3. Effect of Sodium Salicylate Concentration (C) on the Solubility (S) of Benzyl Benzoate in Water

	10^3S/mol·L^{-1}			
C/mol·L^{-1}	T = 303 K	T = 313 K	T = 323 K	T = 333 K
0.0	6.96	7.02	7.15	7.23
0.1	7.05	7.12	7.38	7.42
0.2	7.23	7.46	7.51	7.68
0.3	8.58	9.58	10.25	10.85
0.4	9.45	10.19	12.09	14.35
0.5	11.39	17.52	20.76	24.65
0.6	17.05	26.22	31.08	36.89
0.7	21.54	33.12	39.24	46.58
0.8	24.29	37.35	44.29	52.56
0.9	28.31	43.53	51.65	61.25
1.00	31.75	48.82	57.87	68.69
1.20	38.02	58.46	69.29	82.25
1.40	43.27	66.53	78.86	93.60
1.60	46.18	71.07	84.16	99.89
1.80	49.35	75.86	89.93	106.74
2.00	52.49	80.71	95.67	113.56
2.25	56.15	86.32	102.32	121.45
2.50	58.76	90.36	107.25	127.12
2.75	58.82	90.44	107.21	127.25
3.00	58.96	90.65	107.45	127.54

bath and a separating funnel. For each solubility test, ~100 mL of benzyl benzoate, 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 hydrochloric acid.

The experimental setup used for the determination of the mass transfer coefficient consists of a vessel provided with baffles and a turbine impeller, run by a motor to agitate the mixture. The speed of the impeller 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 well-known. The glass vessel is 40 cm in height and 15 cm in inner diameter. The turbine impeller has a diameter of 5 cm, a width of 1 cm, and a length of 1.2 cm. It has four blades and is made to rotate at 600 rpm. The baffle is made of stainless steel and has a diameter of 1.5 cm, having four baffles arranged 90° to each other.

For each run, to measure the mass transfer coefficient, ~250 mL of benzyl benzoate, previously saturated with distilled water, was added to the aqueous solution of the hydrotrope 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, the entire mixture was transferred to a separating funnel. After allowing it to stand for some time, the aqueous layer was carefully separated from the ester layer (C_b). The composition of the solubilized organic ester in aqueous hydrotrope solutions at time t was analyzed as was done for the solubility determinations.

A plot of $-\log[1 - C_b/C^*]$ versus t is drawn where C^* is the equilibrium solubility of benzyl benzoate at the same hydrotrope concentration (taken from the solubility determinations part). The slope of the above plot gives $k_La/2.303$, from which k_La, the mass transfer coefficient, was calculated. Duplicate runs were made to check the reproducibility. The observed deviation was <2%.

3. Results and Discussion

3.1. Solubility. The solubility of benzyl benzoate in water at 303 K is 6.96×10^{-3} mol·L^{-1} (Tables 1−4), which is in close agreement with earlier reported values.[20,21]

Experimental data on the effect of hydrotropes, that is, tri-sodium citrate, sodium benzoate, sodium salicylate, and urea, on the solubility of benzyl benzoate are presented in Tables 1−4 and are plotted in Figures 1−4. Urea is a typical component which has both polar and nonpolar groups, so it acts as a potential hydrotrope for organic compounds such as benzyl benzoate, which is used as one of the hydrotropes in this study. It was observed that the aqueous solubility of benzyl benzoate did not show any appreciable increase even after the addition of 0.20 mol·L^{-1} urea in the aqueous phase. Upon subsequent increase in

Table 4. Effect of Sodium Benzoate Concentration (C) on the Solubility (S) of Benzyl Benzoate in Water

	10^3S/mol·L^{-1}			
C/mol·L^{-1}	T = 303 K	T = 313 K	T = 323 K	T = 333 K
0.0	6.96	7.02	7.15	7.23
0.1	6.98	7.03	7.26	7.77
0.2	7.02	7.29	7.85	8.06
0.3	7.29	8.44	8.89	9.33
0.4	10.71	15.75	16.25	21.40
0.5	12.78	16.29	20.01	28.20
0.6	14.12	20.50	26.54	35.06
0.7	16.38	22.60	61.52	37.60
0.8	17.64	24.80	34.68	42.38
0.9	19.04	27.80	38.64	44.80
1.0	22.06	32.00	42.25	50.41
1.2	27.14	37.20	47.58	57.76
1.4	34.11	44.12	56.58	65.35
1.6	39.24	47.40	61.25	69.62
1.8	43.82	54.70	65.57	76.49
2.0	49.97	61.50	72.25	84.15
2.25	51.92	74.70	82.51	93.12
2.50	55.85	80.56	91.65	96.24
2.75	55.87	80.72	91.75	96.45
3.0	55.96	80.76	91.88	96.47

the concentration of urea above 0.20 mol·L^{-1}, the solubility of benzyl benzoate in water was found to increase significantly. This concentration of urea in the aqueous phase, 0.20 mol·L^{-1}, 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 benzyl benzoate in water. It has been observed that the MHC of urea in the aqueous phase does not vary even at temperatures of (313, 323, and 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.

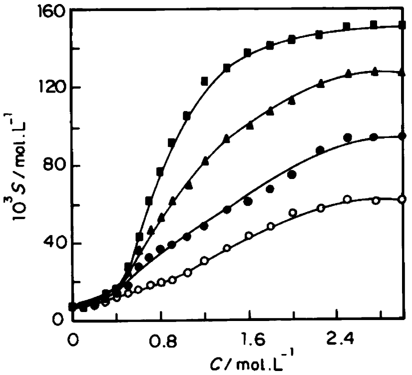

Figure 1. Effect of tri-sodium citrate concentration (C) on the solubility (S) of benzyl benzoate in water: ■, 333 K; ▲, 323 K; ●, 313 K; ○, 303 K.

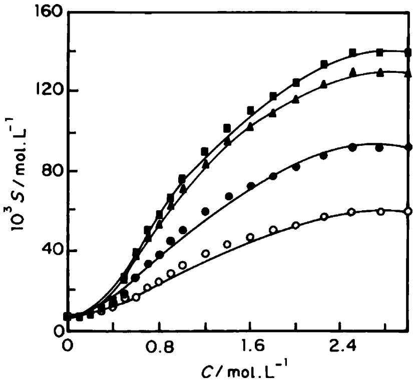

Figure 2. Effect of urea concentration (C) on the solubility (S) of benzyl benzoate in water: ■, 333 K; ▲, 323 K; ●, 313 K; ○, 303 K.

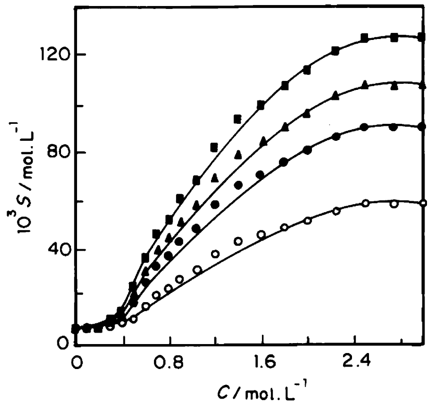

Figure 3. Effect of sodium salicylate concentration (C) on the solubility (S) of benzyl benzoate in water: ■, 333 K; ▲, 323 K; ●, 313 K; ○, 303 K.

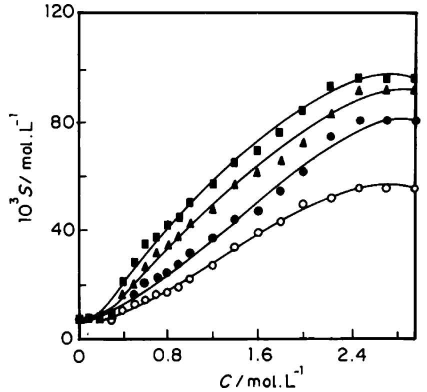

Figure 4. Effect of sodium benzoate concentration (C) on the solubility (S) of benzyl benzoate in water: ■, 333 K; ▲, 323 K; ●, 313 K; ○, 303 K.

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

hydrotrope	MHC/mol·L^{-1}	$C_{\max}$/mol·L^{-1}
tri-sodium citrate	0.25	2.25
urea	0.2	2.3
sodium salicylate	0.3	2.4
sodium benzoate	0.27	2.35

The solubilization effect varies with the concentration of hydrotropes (Tables 1−4). In the present case, a clear increasing trend in the solubility of benzyl benzoate 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 benzyl benzoate.

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 benzyl benzoate are (2.30, 2.40, 2.25, and 2.35) mol·L^{-1}, 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 benzyl benzoate even up to 3.00 mol·L^{-1} urea in the aqueous phase. Similar

Table 6. Maximum Solubility Enhancement Factor (ϕ_s) of Benzyl Benzoate

	$T = 303$ K, $S_0 = 6.96 \times 10^{-3}$ mol·L^{-1}		$T = 313$ K, $S_0 = 7.02 \times 10^{-3}$ mol·L^{-1}		$T = 323$ K, $S_0 = 7.15 \times 10^{-3}$ mol·L^{-1}		$T = 333$ K, $S_0 = 7.23 \times 10^{-3}$ mol·L^{-1}	
hydrotrope	10^3S_{max}/ mol·L^{-1}	ϕ_s	10^3S_{max}/ mol·L^{-1}	ϕ_s	10^3S_{max}/ mol·L^{-1}	ϕ_s	10^3S_{max}/ mol·L^{-1}	ϕ_s
tri-sodium citrate	62.08	8.91	94.88	13.51	126.98	17.76	151.86	**21.00**
urea	60.11	8.63	92.85	13.23	123.96	17.34	140.56	19.44
sodium salicylate	58.96	8.47	90.65	12.91	107.45	15.03	127.54	17.64
sodium benzoate	55.96	8.04	80.76	11.50	91.88	12.85	96.47	13.34

to the MHC values, the C_{max} values of hydrotropes also remained unaltered with an increase in the system temperature.

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^{-1}, three different regions [first region, (0.00 to 0.30) mol·L^{-1}; second region, (0.30 to 2.25) mol·L^{-1}; third region, 2.25 mol·L^{-1}] were obtained when using urea as a hydrotrope. It was inactive below an MHC of 0.30 mol·L^{-1}, above which an appreciable increase in the solubility of benzyl benzoate was found up to a C_{max} value of 2.25 mol·L^{-1}, 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 and 2.25) mol·L^{-1} toward benzyl benzoate. 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 increases 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 benzyl benzoate + water system in the presence of urea as the hydrotrope at (303, 313, 323, and 333) K are 8.63, 13.23, 17.34, and 19.44, 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 and 0.30) mol·L^{-1}, which seem to depend on the hydrophilic nature of the hydrotrope. The C_{max} values of hydrotropes range between (2.25 and 2.40) mol·L^{-1} (Table 5) in most cases. The maximum solubilization enhancement factor (ϕ_s) effected by various hydrotropes for benzyl benzoate range between 8.08 and 21.00. The highest value of ϕ_s (21.00) has been observed in the case of tri-sodium citrate at a system temperature of 333 K (Table 6).

Table 7. Effect of Hydrotrope Concentration (C_b) on the Mass Transfer Coefficient (k_La) of Benzyl Benzoate at a Temperature of T = 303 K

hydrotrope	C/mol·L^{-1}	10^4k_La/s^{-1}	ϕ_{mtc}
tri-sodium citrate	0.00	3.70	
	0.20	6.24	1.68
	0.40	8.76	2.36
	0.80	16.15	4.35
	1.60	28.76	7.76
	2.40	44.09	11.96
	3.00	44.42	11.97
urea	0.00	3.70	
	0.20	4.07	1.09
	0.40	12.31	3.32
	0.80	18.95	5.10
	1.60	29.38	7.92
	2.40	35.09	9.46
	3.00	36.33	9.79
sodium salicylate	0.00	3.70	
	0.20	5.80	1.56
	0.40	8.56	2.31
	0.80	15.32	4.14
	1.60	17.39	4.70
	2.40	19.98	5.40
	3.00	20.09	5.43
sodium benzoate	0.00	3.70	
	0.20	5.64	1.52
	0.40	8.21	2.21
	0.80	12.74	3.43
	1.60	15.65	4.22
	2.40	17.12	4.62
	3.00	17.63	4.75

3.2. Mass Transfer Coefficient. The mass transfer coefficient for the benzyl benzoate + water system in the absence of any hydrotrope was determined to be 3.70×10^{-4} s^{-1} at 303 K (Table 7). The effect of different hydrotropes on the mass transfer coefficient of benzyl benzoate at different hydrotrope concentrations is also presented in the same table (Table 7). It can be seen that a threshold value of 0.2 mol·L^{-1} is required for significant enhancement in the mass transfer coefficient of the benzyl benzoate + water system, as observed in the case of the solubility determinations. The mass transfer coefficient of the benzyl benzoate + water system increases with an increase in the hydrotrope concentration. Beyond a C_{max} value of 2.40 mol·L^{-1}, there is no appreciable increase in the mass transfer coefficient of benzyl benzoate, 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 benzyl benzoate 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 benzyl benzoate + water system in the presence of various hydrotropes at 303 K are reported in Table 7. The highest value of ϕ_{mtc} (11.97)

Table 8. Setschenow Constant (K_S) of Hydrotropes with Respect to Benzyl Benzoate

	K_S/L·mol^{-1}			
T/K	tri-sodium citrate	sodium benzoate	sodium salicylate	urea
303	0.4356	0.3579	0.4272	0.4238
313	0.5094	0.3400	0.5070	0.5021
323	0.5648	0.3773	0.5322	0.5547
333	**0.5676**	0.3349	0.5609	0.5647

has been observed in the presence of tri-sodium citrate as the hydrotrope at a C_{max} value of 2.25 mol·L^{-1}.

4. Effectiveness of Hydrotropes

The effectiveness factor of each hydrotrope with respect to benzyl benzoate 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 22, as given by the equation

$$\log[S/S_m] = K_S[C_s - C_m] \qquad (1)$$

where S and S_m are the solubility values of benzyl benzoate 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 conditions of hydrotrope concentration and system temperature. The Setschenow constant values of the hydrotropes, namely, tri-sodium citrate, sodium benzoate, sodium salicylate, and urea, for the benzyl benzoate + water system at different system temperatures are listed in Table 8. The highest value has been observed as 0.5676 in the case of tri-sodium citrate as the hydrotrope at 333 K.

5. Conclusions

From the literature, the solubility data for benzyl benzoate show that the ester is practically insoluble in water, which has been increased to a maximum value of 20.92 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 benzyl benzoate. The separation of benzyl benzoate 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 benzyl benzoate can be used for the recovery of the dissolved benzyl benzoate 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 benzyl benzoate 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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