Solubility and Mass Transfer Coefficient Enhancement of Benzyl Acetate in Water through Hydrotropy

N. Meyyappan and N. Nagendra Gandhi*

Department of Chemical Engineering, Alagappa College of Technology, Anna University,
Chennai − 600 025, India

A comprehensive investigation of the solubility and mass transfer coefficient enhancement of benzyl acetate through hydrotropy has been undertaken. The solubility studies have been carried out with hydrotropes such as citric acid, urea, sodium benzoate, and sodium salicylate for a wide concentration range from 0 to 3.0 mol/L^{-1} along with system temperature from $T = 303$ to 333 K. The effectiveness of hydrotropes was measured by the determination of Setschenow constant "K_s". The solubility of benzyl acetate increases with increasing hydrotrope concentration and also with system temperature. A minimum hydrotrope concentration (MHC) in the aqueous phase was required to initiate significant solubilization of benzyl acetate. Consequent to the increase in solubilization of benzyl acetate, the mass transfer coefficient was also found to increase with increasing hydrotrope concentration. A threshold value of MHC is to be maintained to have an appreciable enhancement in the 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 experiments.

1. Introduction

Hydrotropy is the phenomenon by which certain compounds (hydrotropes) can be used to effect a several fold increase in the solubility of solutes that are sparingly soluble in water under normal conditions. 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 that can significantly enhance the solubility of organic solutes such as esters, alcohols, aldehydes, ketones, hydrocarbons, and fats.[1−3]

Hydrotropes are widely used in various industries for applications such as drug solubilization, detergent formulation, health care, and household applications[4] besides being an extraction agent for fragrances.[5] This potentially attractive technique can also be adopted to separate isomeric and nonisomeric mixtures with similar boiling points. At the same time, the problem of emulsification, which is normally encountered with conventional surfactant solutions, is not found with hydrotrope solutions.[6] Hydrotropes have been used to increase the rate of heterogeneous reaction and are also used for the separation of liquid mixtures with similar boiling points and narrow melting points through extractive distillation and liquid−liquid extraction.[7,8]

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.[9,10] The solubility enhancement in the organic compounds could be due to the formation of molecular structures in the form of complexes.[11] Previous experimental findings have concluded that hydrotropy is a process that goes beyond miscibility or cosolvency or the salting-in effect or other conventional solubilization methods because the solubilization effected by hydrotropy was higher and more selective compared to that of other solubilization methods.[12] The effect of hydrotropes on the solubility and mass transfer coefficient of a series of organic esters such as butyl acetate, ethyl benzoate, amyl acetate, and methyl salicylate was studied in our earlier publications.[13−16]

For many binary systems involving a sparingly soluble organic compound such as benzyl acetate, the mass transfer coefficient in the presence of a hydrotrope is probably due to the difference between binary diffusivity (solute + solvent) and the diffusivity of the solute in the solution (solute + solvent + hydrotrope). Benzyl acetate serves as a raw material and intermediate for a wide variety of chemicals and allied products. Its separation from any liquid mixture can 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 benzyl acetate. All are nonreactive and nontoxic and do not produce any significant heat effect when dissolved in water. The easy availability and low cost are two other factors considered in the selection of these hydrotropes.

2. Experimental Section

The chemicals used were checked for purity using gas chromatography and refractometry (i.e., benzyl acetate was 99.9% pure and hydrotropes citric acid, urea, sodium salicylate, and sodium benzoate were of analar grade and were procured from Chemical Dug House (CDH), Mumbai). 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 benzyl acetate, previously saturated with distilled water, was taken in a separation funnel, and 100 mL of a hydrotrope solution of known concentration was added. The

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

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separation funnel was sealed to avoid the evaporation of the solvent at higher temperatures. Hydrotrope solutions of different concentrations were prepared by dilution with distilled water. The separation funnel was immersed in a constant-temperature bath fitted with a temperature controller that could control the temperature to 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 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 consist of a vessel equipped 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 vessel has a height of 40 cm and an inner diameter of 15 cm. The turbine impeller has a diameter of 5 cm, width of 1 cm, and length of 1.2 cm. It has four blades and rotates at 600 rpm. The baffle has a height of 40 cm with a diameter of 1.5 cm and four baffles arranged 90° to each other.

For each run, to measure the mass transfer coefficient, about 250 mL of benzyl acetate 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, or 2400 s. After the end of fixed time "*t*", the entire mixture was transferred to a separating funnel. After allowing the sample 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 in the same way as for solubility determinations. A plot of log $[1 - C_b/C^*]$ versus t is drawn, where C_b is the concentration of benzyl acetate at time t and C^* is the equilibrium solubility of benzyl acetate at the same hydrotrope concentrations. The slope of the graph 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 error was less than 2%.

3. Results and Discussion

3.1. Solubility. The solubility of benzyl acetate standard in water is 9.2×10^{-3} mol L^{-1} at 303 K, compared to "insoluble" as reported in the literature.[17,18] Thus, the solubility values we determined are in close agreement with the earlier reported values.

Experimental data on the effect of hydrotropes (i.e., citric acid, sodium benzoate, sodium salicylate, and urea) on the solubility of benzyl acetate 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 acetate, which is used as one of the hydrotropes in this study. The solubility of benzyl acetate in water at 303 K, in the absence of any hydrotrope, is 9.2×10^{-3} mol L^{-1} (Table 4). It was observed that the aqueous solubility of benzyl acetate did not show any appreciable increase even after the addition of 0.40 mol L^{-1} of urea in the aqueous phase. On subsequent increase in the concentration of urea above 0.40 mol L^{-1}, the solubility of benzyl acetate in water was found to increase significantly. This concentration of urea in the aqueous phase, 0.40 mol L^{-1}, is called the minimum hydrotrope concentration (MHC), which is the minimum required urea (hydrotrope) concentration in the aqueous phase to cause a significant increase in the solubility of benzyl acetate 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 MHC requirements in the aqueous phase has also been observed for other hydrotropes. Therefore, it is evident

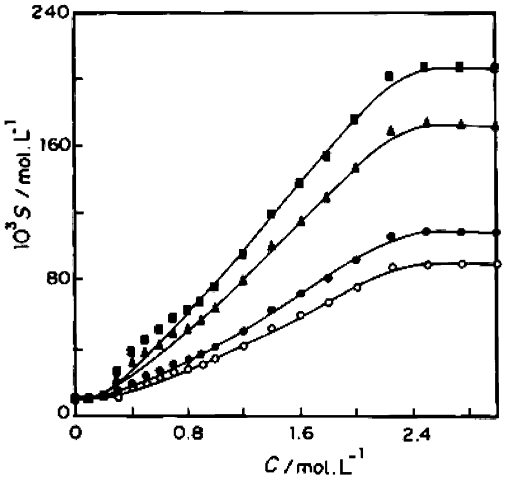

Figure 1. Effect of citric acid concentration on the solubility of benzyl acetate in water: ■, 333 K; ▲, 323 K; ●, 313 K; ○, 303 K.

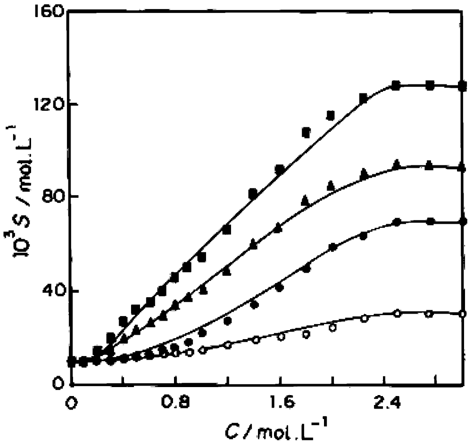

Figure 2. Effect of sodium benzoate concentration on the solubility of benzyl acetate in water: ■, 333 K; ▲, 323 K; ●, 313 K; ○, 303 K.

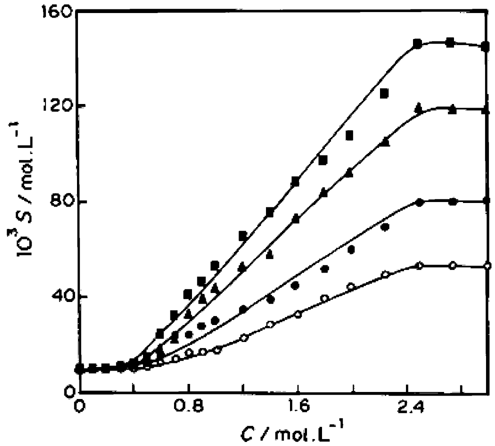

Figure 3. Effect of sodium salicylate concentration on the solubility of benzyl acetate in water: ■, 333 K; ▲, 323 K; ●, 313 K; ○, 303 K.

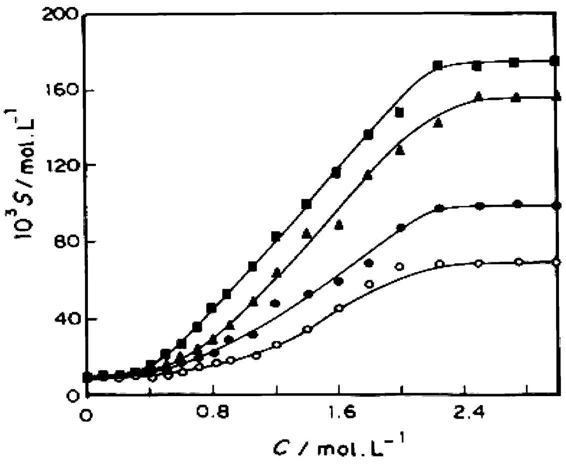

Figure 4. Effect of urea concentration on the solubility of benzyl acetate in water: ■, 333 K; ▲, 323 K; ●, 313 K; ○, 303 K.

Table 1. Effect of Citric Acid Concentration *C* on the Solubility *S* of Benzyl Acetate in Water

C	$10^3 S$ mol L^{-1}			
mol L^{-1}	$T = 303$ K	$T = 313$ K	$T = 323$ K	$T = 333$ K
0.00	9.184	9.360	9.792	9.904
0.10	9.981	10.063	10.072	10.146
0.20	10.012	10.311	10.403	10.234
0.30	11.134	12.079	14.610	16.208
0.40	16.423	18.178	19.460	21.909
0.50	19.475	25.187	33.175	42.706
0.60	21.946	35.284	49.424	59.505
0.70	25.109	42.465	58.475	87.404
0.80	26.944	48.946	88.163	106.201
0.90	29.415	51.355	116.425	126.958
1.00	33.272	55.914	119.546	147.755
1.20	41.561	63.171	128.386	169.951
1.40	51.913	74.487	139.618	178.450
1.60	59.765	81.348	147.279	189.850
1.80	67.129	89.216	158.314	194.659
2.00	76.587	98.375	164.566	198.456
2.25	79.142	103.563	169.798	201.305
2.50	85.313	109.271	173.251	207.432
2.75	85.311	109.270	173.251	207.454
3.00	85.311	109.270	173.265	207.464

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 (Table 4). In the present case, a clear increasing trend in the solubility of benzyl 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 benzyl 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, $C_{\max}$ values of urea, citric acid, sodium benzoate, and sodium salicylate with respect to benzyl acetate are 2.00, 2.25, 2.50, and 2.50 mol L^{-1}, respectively. From the analysis of the experimental data, it is observed that further increases in hydrotrope concentration beyond $C_{\max}$ do not bring any appreciable increase in the solubility of benzyl acetate even up to 3.00 mol L^{-1} of urea in the aqueous phase. Similar to the MHC values, the $C_{\max}$ values of hydrotropes also remained unaltered with increases in system temperature.

Table 2. Effect of Sodium Benzoate Concentration *C* on the Solubility *S* of Benzyl Acetate in Water

C	$10^3 S$ mol L^{-1}			
mol L^{-1}	$T = 303$ K	$T = 313$ K	$T = 323$ K	$T = 333$ K
0.00	9.184	9.361	9.792	9.904
0.10	9.281	9.462	10.075	10.146
0.20	9.301	9.612	10.456	10.234
0.30	9.830	10.012	14.612	21.203
0.40	10.220	11.314	19.463	34.902
0.50	11.472	12.516	23.176	46.708
0.60	11.943	13.014	25.424	49.560
0.70	12.409	14.570	27.478	51.403
0.80	12.948	16.096	33.169	65.203
0.90	13.461	18.341	36.423	69.560
1.00	14.525	21.912	39.540	73.752
1.20	16.263	23.178	48.389	88.954
1.40	18.923	34.483	59.612	98.453
1.60	20.463	41.349	67.278	109.852
1.80	21.626	49.215	78.316	114.653
2.00	24.589	58.374	84.266	118.459
2.25	28.146	63.562	89.569	121.564
2.50	30.369	69.278	93.275	127.436
2.75	30.369	69.278	93.276	127.456
3.00	30.369	69.285	93.275	127.456

Table 3. Effect of Sodium Salicylate Concentration *C* on the Solubility *S* of Benzyl Acetate in Water

C	$10^3 S$ mol L^{-1}			
mol L^{-1}	$T = 303$ K	$T = 313$ K	$T = 323$ K	$T = 333$ K
0.00	9.184	9.36	9.792	9.904
0.10	9.538	9.521	9.816	9.981
0.20	9.781	9.873	9.918	9.994
0.30	10.482	10.683	10.526	10.521
0.40	11.020	10.936	10.870	11.610
0.50	11.143	11.871	10.619	11.940
0.60	13.389	15.526	18.564	25.865
0.70	15.780	23.618	25.461	39.362
0.80	18.340	28.560	35.200	56.123
0.90	21.620	33.910	49.951	62.764
1.00	26.540	36.430	53.752	75.893
1.20	28.560	43.120	68.950	90.321
1.40	31.390	50.420	88.453	101.562
1.60	33.170	58.190	99.858	123.952
1.80	39.740	68.060	104.651	130.163
2.00	44.380	74.310	108.451	134.320
2.25	49.360	78.160	111.300	145.110
2.50	53.190	80.130	118.900	145.180
2.75	53.200	80.140	118.900	145.280
3.00	53.210	80.160	118.900	145.210

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 the hydrotrope solution with decreasing hydrotrope concentration approach those of water, similar to the situation below the MHC. This phenomenon was observed experimentally by the release of the dissolved solute from hydrotrope solutions at any concentration between the MHC and $C_{\max}$ by simple dilution with distilled water and hence the possible reuse of hydrotrope solutions. 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}, four different regions were obtained using urea as a hydrotrope. It was inactive below the MHC of 0.40 mol L^{-1}, above which an appreciable increase in the

Table 4. Effect of Urea Concentration *C* on the Solubility *S* of Benzyl Acetate in Water

C	$10^3 S$ mol L^{-1}			
mol L^{-1}	$T = 303$ K	$T = 313$ K	$T = 323$ K	$T = 333$ K
0.00	9.184	9.360	9.792	9.904
0.10	9.280	9.510	10.011	10.162
0.20	9.506	9.810	10.021	10.948
0.30	9.732	9.908	10.612	11.784
0.40	9.624	10.848	11.700	15.744
0.50	10.776	12.528	15.792	20.208
0.60	12.564	16.872	23.976	26.460
0.70	14.532	20.544	26.688	28.956
0.80	16.512	21.684	28.500	38.496
0.90	18.360	28.836	36.048	50.328
1.00	20.244	31.572	48.750	68.028
1.20	26.040	47.400	86.156	103.156
1.40	33.672	53.156	108.932	111.988
1.60	45.230	59.368	119.468	133.404
1.80	57.600	68.772	139.680	157.760
2.00	67.224	96.576	155.344	171.828
2.25	67.980	97.428	155.320	172.260
2.50	68.348	98.460	155.860	172.416
2.75	68.398	98.460	155.908	173.700
3.00	68.400	98.466	155.956	173.796

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

hydrotropes	MHC mol L^{-1}	C_{max} mol L^{-1}
citric acid	0.30	2.00
sodium benzoate	0.40	2.50
sodium salicylate	0.50	2.50
urea	0.40	2.00

Table 6. Solubility Enhancement Factor of Benzyl Acetate

	maximum enhancement factor for solubility (ϕ_s)			
hydrotropes	$T = 303$ K	$T = 313$ K	$T = 323$ K	$T = 333$ K
citric acid	9.29	11.67	17.7	20.94
sodium benzoate	3.30	7.40	9.53	12.87
sodium salicylate	5.80	8.57	12.14	14.66
urea	7.45	10.52	15.93	17.56

Table 7. Effect of Hydrotrope Concentration on Mass Transfer Coefficient of Benzyl Acetate

hydrotrope	C_b mol L^{-1}	$10^4 k_L a$ s^{-1}	ϕ_{mtc}
citric acid	0.00	4.606	
	0.20	5.46	1.05
	0.30	8.49	2.81
	0.80	14.04	3.05
	1.40	31.23	6.78
	2.00	46.80	10.16
	3.00	46.93	10.18
sodium benzoate	0.00	4.606	
	0.70	6.909	1.50
	0.80	9.212	2.05
	1.40	13.818	3.22
	2.00	17.564	4.50
	2.50	18.424	4.80
	3.00	18.424	
sodium salicylate	0.00	4.606	
	0.80	9.212	2.00
	0.90	11.515	2.50
	1.40	13.818	3.26
	2.00	16.121	3.39
	3.00	18.321	4.09
urea	0.00	4.606	
	0.30	5.012	1.08
	0.40	9.543	2.07
	1.00	15.062	3.27
	1.40	22.364	4.85
	1.80	39.888	8.66
	3.00	39.921	8.92

solubility of benzyl acetate was found up to C_{max} of 2.00 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 0.40 and 2.00 mol L^{-1} toward benzyl acetate. It has also been observed that the solubilization effect of urea was not a linear function of the concentration of the citric acid solution. The solubilization effect of citric acid increases with increasing hydrotrope concentration and also with system temperature. The maximum enhancement factor (ratio of the solubility values in the presence and absence of a hydrotrope) values observed for the benzyl acetate + water system in the presence of urea as a hydrotrope at 303, 313, 323, and 333 K are 7.45, 10.52, 15.93, and 17.56, respectively (Table 6).

A similar trend has been observed in the solubilization effect of other hydrotropes, namely, citric acid, sodium benzoate, and sodium salicylate. It has also been observed that the MHC values of the hydrotropes used in this work range from 0.30 to 0.50 mol L^{-1}, depending on the hydrophilic nature of the hydrotrope. The C_{max} values of hydrotropes range from 2.00 to 2.50 mol L^{-1} (Table 5) in most cases. The maximum solubilization enhancement factor (ϕ_s) effected by various hydrotropes for benzyl acetate range from 3.30 to 20.94 The highest value of ϕ_s (20.94) has been observed in the case of citric acid at a system temperature of 333 K (Table 6).

3.2. Mass Transfer Coefficient. The mass transfer coefficient for the benzyl acetate + water system in the absence of any hydrotrope was determined to be 4.606×10^{-4} s^{-1} at 303 K (Table 7). The effect of different hydrotropes on the mass transfer coefficient of benzyl acetate at different hydrotrope concentrations is also presented in 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 acetate + water system as observed in the case of solubility determinations. The mass transfer coefficient of the benzyl acetate + water system increases with increasing hydrotrope concentration. Beyond a C_{max} of 2.00 mol L^{-1}, there is no appreciable increase in the mass transfer coefficient of benzyl acetate, as observed in the case of solubility determinations. The observed increase in the mass transfer coefficient in the presence of a hydrotrope is probably due to the difference between 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 acetate has also been observed for other hydrotropes. The highest value of ϕ_{mtc} (10.16) has been observed in the presence of citric acid as the hydrotrope at a C_{max} of 2.00 mol L^{-1}.

4. Effectiveness of Hydrotropes

The effectiveness factor of each hydrotrope with respect to benzyl 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 by[19] as given by the equation

$$\log_{10}[S/S_m] = K_S[C_s - C_m] \tag{1}$$

where S and S_m are the solubility values of benzyl acetate at any hydrotrope concentration C_s and the minimum

Table 8. Setschenow Constant K_s of Hydrotropes with Respect to Benzyl Acetate

	K_s / L mol^{-1}			
T / K	citric acid	sodium benzoate	sodium salicylate	urea
303	0.443	0.218	0.353	0.426
313	0.493	0.375	0.455	0.486
323	0.576	0.494	0.592	0.598
333	0.602	0.632	**0.682**	0.562

hydrotrope concentration MHC, respectively. The Setschenow constant K_S can be considered to be 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, sodium benzoate, sodium salicylate, and urea, for the benzyl acetate + water system at different system temperatures are listed in Table 8. The highest value has been observed as 0.682 in the case of sodium salicylate as a hydrotrope at 333 K.

5. Conclusions

From the literature, the solubility data for benzyl acetate show that the ester is practically insoluble in water; the solubility has been increased to a maximum value of 20.94 in the presence of citric acid as a 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 acetate. The separation of benzyl 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 hydrotropes with respect to benzyl acetate can be used for the recovery of the dissolved benzyl acetate 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 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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