J. Chem. Eng. Data **2000,** *45,* 419−423

Effect of Hydrotropes on Solubility and Mass Transfer Coefficient of Methyl Salicylate

M. Dharmendira Kumar and N. Nagendra Gandhi*

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

This paper presents a comprehensive study on the effect of citric acid, sodium benzoate, sodium salicylate, and urea (hydrotropes) on the solubility and mass transfer coefficient for the extraction of methyl salicylate in water. A Minimum Hydrotropic Concentration (MHC) in the range between 0.20 and 0.90 mol/L was found essential to show a significant increase in the solubility and mass transfer coefficient for the methyl salicylate−water system. The solubility of methyl salicylate has been increased to a maximum value of 22.84 in the presence of citric acid as hydrotrope at concentration of 2.00 mol/L and temperature of 333 K. The maximum enhancement factor for the mass transfer coefficient was found to be 7.88 in the presence of citric acid as hydrotrope at a concentration of 2.00 mol/L at 303 K at 600 rpm. The Setschenow constant, K_s, a measure of the effectiveness of the hydrotrope, has been determined for each case, and the highest value has been observed as 0.743 in the case of citric acid.

Introduction

Hydrotropes are a class of chemical compounds used to effect a severalfold increased aqueous solubility to certain solutes which are sparingly soluble in water under normal conditions (Neuberg, 1916; Saleh and El-Khordagui, 1985). This increase in solubility in water is probably due to the formation of organized assemblies of hydrotrope molecules at critical concentrations (Badwan et al., 1983; Balasubramanian et al., 1989). This phenomenon, termed as hydrotropy, can be considered to be a potentially and industrially attractive technique, since the observed increase in solubility is generally higher than that effected by other known solubilization methods. Hydrotropes have been used to solubilize various sparingly soluble organic compounds (Booth and Everson, 1948, 1949, 1950; Korenman, 1974). The role of hydrotopes in enhancing rates of heterogeneous reactions has also been brought out (Janakiraman and Sharma, 1985; Pandit and Sharma, 1987). Hydrotropes have been effectively used in the development of extractive separations and in distillation as extractive solvents for the separation of close boiling point mixtures (Gaikar and Sharma, 1986; Mamata Agarwal and Gaikar, 1992; Mahapatra et al., 1988). The effects of hydrotropes on the solubility and the mass-transfer coefficient of esters such as butyl acetate and ethyl benzoate were studied in our earlier publications (Nagendra Gandhi et al., 1998a, 1998b). Easy recovery of the dissolved solute and the possible reuse of hydrotrope solutions make this method the most effective one, particularly at industrial levels (Mckee, 1946; Balasubramanian et al., 1989; Mamta Aggarwal and Gaikar, 1992; Nagendra Gandhi et al., 1998a, 1998b; Dharmendira Kumar and Nagendra Gandhi, 2000).

The advantage of certain properties, such as the solvent character being independent of pH, high selectivity, and the absence of emulsification, makes this technique superior to other solubilization methods, such as micellar solubilization, miscibility, cosolvency, salting-in, and so forth. Because of the solubilizing effect of these hydrotropes, the mass transfer coefficient of two-phase systems can be considerably enhanced.

It has been observed that, in many two-phase reaction systems involving a sparingly soluble organic compound like methyl salicylate, the mass-transfer coefficient was found to be very low solely due to the poor solubility of methyl salicylate in the aqueous phase. Since methyl salicylate serves as a raw material/intermediate for a wide variety of chemicals and allied products and the separation of methyl salicylate from any liquid mixture seems to be difficult, 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 methyl salicylate. All are nonreactive and nontoxic and do not produce any temperature effect when dissolved in water. The easy availability and cheapness of hydrotropes are the other factors considered in the selection of hydrotropes. Data on various aspects of hydrotropic study on the solubility and mass-transfer coefficient for the methyl salicylate−water system are reported for the first time.

Experimental Section

All the chemicals used in this work were manufactured by Aldrich Chemical Co., with a manufacturer's stated purity of 99%.

The experimental setup for the determination of solubility values consisted of a thermostatic bath and a separating funnel. For each solubility test, about 100 mL of methyl salicylate, 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

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* To whom correspondence should be addressed. Phone: (91 44) 235 1126 Ext. 3515. Fax: + 91 44 235 2642/235 0240. E-mail: nngandhi@annauniv.edu.

10.1021/je9901740 CCC: $19.00

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 using a standardized HCl solution and phenolpthalein 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 sizes of the vessel, the baffle, and the turbine impeller are given below.

The vessel 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 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 it was allowed 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 salicylate at time *t* and C^* is the equilibrium solubility of methyl salicylate at the same hydrotrope concentration. 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 <2%.

Results and Discussion

(a) Solubility. The solubility of the methyl salicylate standard in water is 4.11×10^{-3} mol/L at 303 K, compared to 4.14×10^{-3} as reported by Dean (1987). Thus, the solubility values in water are in excellent agreement with the earlier value.

Experimental data representing the average of duplicate determinations on the effect of hydrotropes, that is, citric acid, sodium benzoate, sodium salicylate, and urea, on the solubility of methyl salicylate are presented in Tables 1−4 and are plotted in Figures 1−4. Citric acid is one of the hydrotropes used in this study. The solubility of methyl salicylate in water at 303 K in the absence of any hydrotrope is 4.11×10^{-3} mol/L (Table 1). A minimum hydrotope concentration (MHC), that is, 0.20 mol/L, was found to be required to effect a significant increase in the solubility of methyl salicylate in water. It has been observed that the solubility of methyl salicylate in water did not show any appreciable increase even after the addition of 0.20 mol/L of citric acid in the aqueous phase.

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 the 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 methyl salicylate on dilution with water below the MHC. The knowledge of MHC values is necessary especially at industrial levels, as it ensures ready recovery of the hydrotrope for reuse.

Table 1. Effect of Citric Acid Concentration (*C*) on the Solubility (*S*) of Methyl Salicylate in Water

	10^3S/mol L^{-1}			
C/mol L^{-1}	$T = 303$ K	$T = 313$ K	$T = 323$ K	$T = 333$ K
0.00	4.11	4.39	4.78	5.06
0.10	4.13	4.42	4.85	5.21
0.20	4.18	4.45	4.90	5.33
0.30	4.51	4.77	6.32	7.18
0.40	4.83	5.90	8.13	10.62
0.50	5.61	8.21	10.78	14.05
0.60	6.23	8.12	14.90	18.08
0.70	8.93	10.05	17.21	23.91
0.80	8.09	10.83	17.00	26.12
0.90	8.89	11.78	19.05	30.37
1.00	9.14	15.57	25.86	31.89
1.20	11.24	18.11	30.32	43.83
1.40	12.05	23.19	37.41	54.26
1.60	13.28	30.87	45.06	61.12
1.80	18.73	39.79	58.52	88.03
2.00	27.21	52.15	84.70	116.08
2.25	27.30	52.42	84.80	115.85
2.50	27.34	52.39	84.86	116.01
2.75	27.37	52.51	84.90	115.96
3.00	27.35	52.58	84.96	115.98

Table 2. Effect of Sodium Benzoate Concentration (*C*) on the Solubility (*S*) of Methyl Salicylate in Water

	10^3S/mol L^{-1}			
C/mol L^{-1}	$T = 303$ K	$T = 313$ K	$T = 323$ K	$T = 333$ K
0.00	4.11	4.39	4.78	5.06
0.10	3.89	4.23	4.78	5.10
0.20	3.92	4.26	4.71	5.13
0.30	3.99	4.36	4.75	5.11
0.40	4.08	4.38	4.71	5.10
0.50	4.13	4.40	4.18	5.14
0.60	4.29	4.51	4.98	5.73
0.70	4.34	4.87	6.56	8.19
0.80	4.45	5.52	7.12	10.82
0.90	4.56	5.29	8.17	13.56
1.00	5.14	7.54	8.05	20.67
1.20	5.69	6.78	12.10	22.31
1.40	5.83	9.12	13.38	27.58
1.60	6.10	10.46	15.89	35.37
1.80	6.58	12.76	18.64	40.96
2.00	7.75	14.08	22.41	52.57
2.25	8.55	19.23	35.80	60.47
2.50	8.59	19.29	35.75	60.56
2.75	8.50	19.25	35.79	60.50
3.00	8.64	19.34	35.84	60.53

The solubilization effect varies with concentration of hydrotropes (Table 1). In the present case, a clear increasing trend in the solubility of methyl salicylate was observed above the MHC of citric acid. This increasing trend is maintained only up to a certain concentration of citric acid in the aqueous phase, beyond which there is no appreciable increase in the solubility of methyl salicylate. This concentration of citric acid (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 the hydrotrope concentration beyond C_{max} does not bring any appreciable increase in the solubility of methyl salicylate even up to 3.00 mol/L of citric acid in the aqueous phase. Similar to the MHC values, the C_{max} values of hydrotropes also remained unaltered with an increase in system temperature.

The knowledge of MHC and C_{max} values of each hydrotrope with respect to a particular solute assumes greater

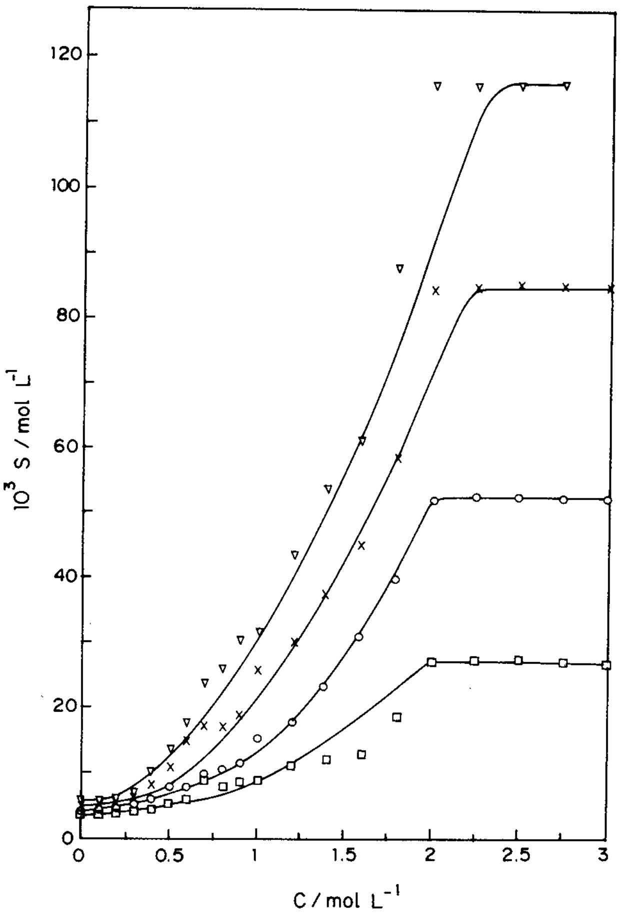

Figure 1. Effect of citric acid concentration (C) on the solubility of Methyl salicylate in water (S) at T = 303 K (□), 313 K (○), 323 (×), and 333 K (▽).

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

	10^3S/mol L^{-1}			
C/mol L^{-1}	T = 303 K	T = 313 K	T = 323 K	T = 333 K
0.00	4.11	4.39	4.78	5.06
0.10	3.99	4.27	4.69	4.94
0.20	3.99	4.25	4.72	4.96
0.30	4.03	4.25	4.75	4.98
0.40	4.07	4.29	4.77	5.02
0.50	4.01	4.32	4.80	5.05
0.60	4.09	4.30	4.81	5.01
0.70	4.05	4.35	4.78	5.01
0.80	4.05	4.37	4.80	5.06
0.90	4.12	4.39	4.81	5.08
1.00	4.28	4.49	5.09	5.26
1.20	5.26	5.23	6.44	6.78
1.40	5.35	6.14	6.75	10.12
1.60	6.13	7.05	7.83	10.84
1.80	6.54	9.33	9.22	15.56
2.00	8.18	8.80	10.67	20.96
2.25	9.78	11.94	13.96	31.37
2.50	9.89	11.35	14.17	31.59
2.75	9.85	11.59	14.10	31.67
3.00	10.53	11.73	14.34	31.63

significance in this study, since it indicates the beginning and saturation of the solubilization effect of hydrotropes. The values of MHC and C_{max} of a hydrotrope with respect to methyl salicylate may be useful in determining the recovery of the dissolved methyl salicylate even to an extent of the calculated amount from hydrotrope solutions at any concentration between the MHC and C_{max} by simple dilution with distilled water. This is the unique advantage of the hydrotropic solubilization technique.

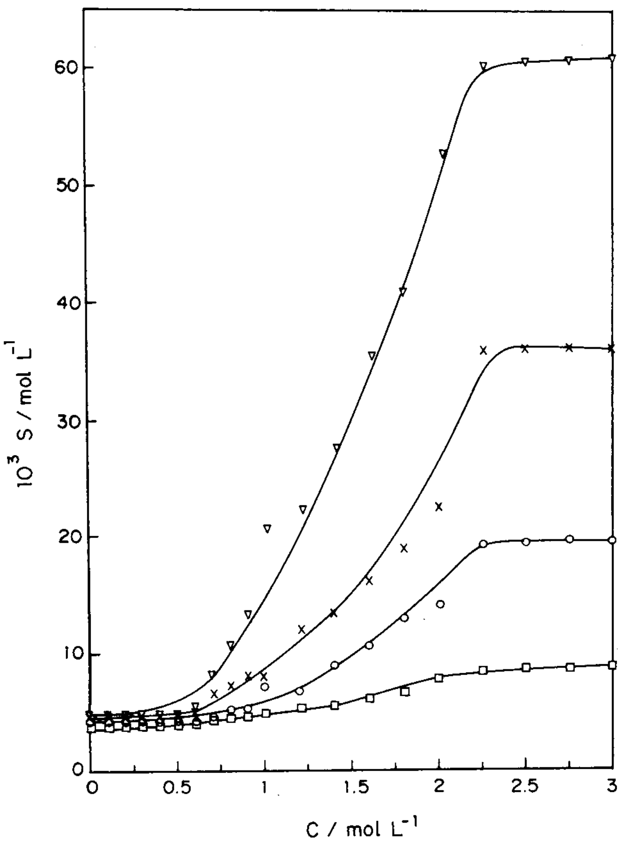

Figure 2. Effect of Sodium benzoate concentration (C) on the solubility of methyl salicylate in water (S) at T = 303 K (□), 313 K (○), 323 K (×), and 333 K (▽).

Table 4. Effect of Urea Concentration (C) on the Solubility (S) of Methyl Salicylate in Water

	10^3S/mol L^{-1}			
C/mol L^{-1}	T = 303 K	T = 313 K	T = 323 K	T = 333 K
0.00	4.11	4.39	4.78	5.06
0.10	4.15	4.36	4.81	5.12
0.20	4.18	4.33	4.85	5.17
0.30	4.14	4.38	4.85	5.21
0.40	4.14	4.41	4.87	5.19
0.50	4.89	4.62	4.18	6.54
0.60	5.54	5.03	9.11	9.13
0.70	6.45	5.79	10.04	12.15
0.80	7.01	6.24	10.38	15.21
0.90	7.88	7.38	11.24	16.65
1.00	8.13	8.87	14.09	22.53
1.20	10.09	12.91	28.17	30.05
1.40	11.67	14.03	27.13	38.73
1.60	13.30	15.36	33.24	49.61
1.80	15.21	21.59	40.08	60.08
2.00	17.49	24.00	55.27	73.88
2.25	21.37	36.35	69.12	92.24
2.50	21.14	36.40	68.89	92.28
2.75	21.19	36.32	68.99	92.34
3.00	21.25	36.45	69.15	92.47

From the experimental data plotted in Figure 1, it can further be observed that, in order to achieve the particular solubility of methyl salicylate to a value of 20 × 10^{-3} mol/L, the citric acid concentration should be 1.80 mol/L at 303 K, 1.20 mol/L at 313 K, 0.91 mol/L at 323 K, and 0.65 mol/L at 333 K in the aqueous phase. Thus, it can be seen that as the system temperature increases, the concentration of citric acid required in the aqueous phase to achieve a particular solubility of methyl salicylate decreases. A similar trend has been observed for other systems also.

In the concentration range of citric acid between 0.00 and 3.00 mol/L, four different regions of citric acid as

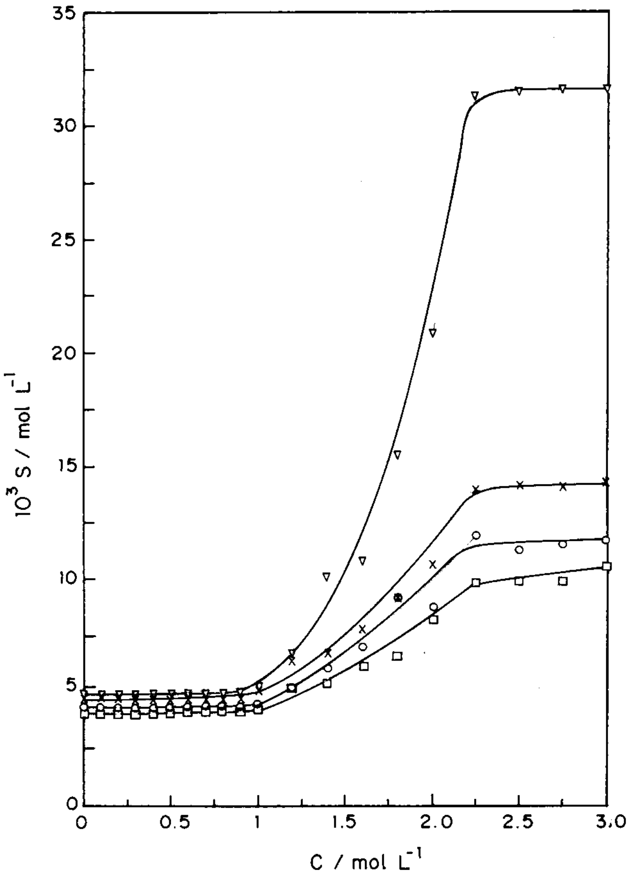

Figure 3. Effect of Sodium salicylate concentration (C) on the solubility of methyl salicylate in water (S) at T = 303 K (□), 313 K (○), 323 K (×), and 333 K (▽).

hydrotrope were observed. It was inactive below MHC values of 0.20 mol/L, above which an appreciable increase in the solubility of methyl salicylate was found up to 0.90 mol/L. Therefore, citric acid was found to be an effective hydrotrope in the concentration range between 0.20 and 2.00 mol/L toward methyl salicylate. It has also been observed that the solubilization effect of citric acid was not a linear function of the concentration of the citric acid solution. The solubilization effect of citric acid increases with an increase in hydrotrope concentration and also with system temperature.

A similar trend has been observed in the solubilization effect of other hydrotropes, namely sodium benzoate, sodium salicylate, and urea. It has also been observed that the MHC values of hydrotrope used in this work range between 0.20 and 0.90 mol/L (Table 5), which seem to depend on the hydrophilicity of a hydrotrope. The C_{max} values of hydrotropes range between 2.00 and 2.25 mol/L (Table 5) in most cases. The maximum solubilization enhancement factors (ϕ_s), which is the ratio of solubility values in the presence and absence of a hydrotrope, affected by various hydrotopes for methyl salicylate, range between 2.10 and 22.84. The highest value of ϕ_s (22.84) has been observed in the case of citric acid at a system temperature of 333 K (Table 6).

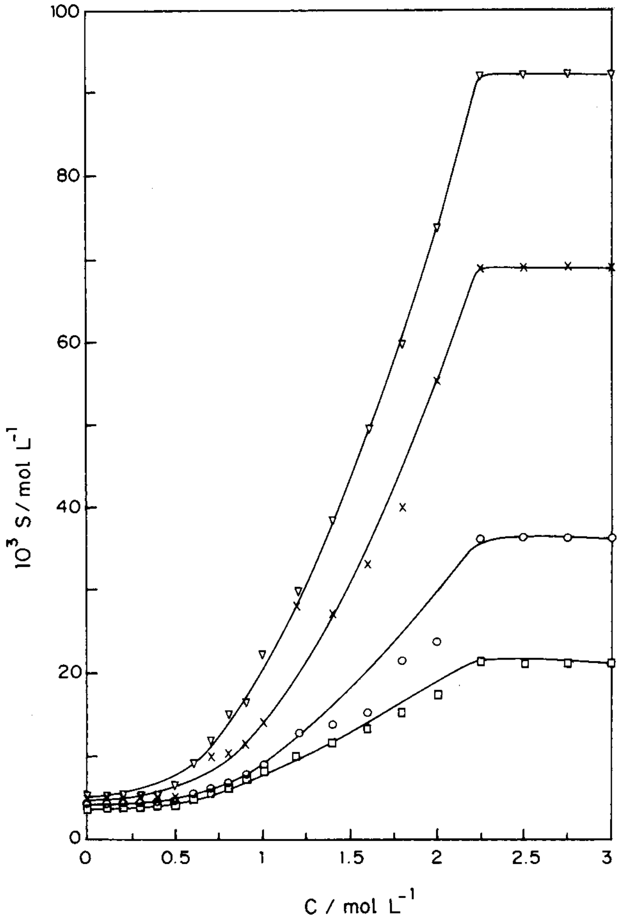

Figure 4. Effect of urea concentration (C) on the solubility of methyl salicylate in water (S) at T = 303 K (□), 313 K (○), 323 K (×), and 333 K (▽).

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

	C/mol L^{-1}	
hydrotrope	MHC	C_{max}
citric acid	0.20	2.00
sodium benzoate	0.50	2.25
sodium salicylate	0.90	2.25
urea	0.40	2.25

Table 6. Maximum Solubilization Enhancement Factor of Methyl Salicylate

	maximum enhancement factor for solubility (ϕ_s)			
hydrotrope	T = 303 K	T = 313 K	T = 323 K	T = 333 K
citric acid	6.66	11.98	17.77	22.84
sodium benzoate	2.10	4.41	7.50	11.97
sodium salicylate	2.56	2.67	3.00	6.25
urea	5.17	8.30	14.47	18.27

(b) Mass-Transfer Coefficient. The mass-transfer coefficient of the methyl salicylate + water system in the absence of any hydrotrope was determined to be 3.14 × 10^{-5} s^{-1} at 30 °C (Table 7). The effect of different hydrotropes on the mass-transfer coefficient of methyl salicylate at different hydrotope concentrations is also given in the same table. 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 methyl salicylate + water system, as observed in the case of solubility determinations. The mass-transfer coefficient of methyl salicylate + water system increases with an increase in citric acid concentration. Beyond a C_{max} of 2.00 mol/L there is no appreciable increase in the mass-transfer coefficient of methyl salicylate, as observed in the case of solubility determinations. The maximum enhancement in the mass-transfer coefficient of the methyl salicylate + water system in the presence of citric acid was found to be 7.88. These observations suggest that an increase in the mass-transfer coefficient is found to occur upon increased solubilization. A similar trend in the mass-transfer coefficient enhancement (ϕ_{mtc}) of methyl salicylate has been observed for other

Table 7. Effect of Hydrotrope Concentration on the Mass-Transfer Coefficient of Methyl Salicylate

hydrotrope	C/ mol L^{-1}	mass-transfer coefficient, $k_La/\times10^5$ S^{-1}	enhancement factor for the mass-transfer coefficient, ϕ_{MTC}
citric acid	0.00	3.14	
	0.20	3.23	1.03
	0.30	6.47	2.06
	0.60	8.67	2.76
	1.00	11.65	3.71
	1.60	16.27	5.18
	2.00	24.74	7.88
	3.00	24.65	8.23
sodium benzoate	0.00	3.14	
	0.50	3.26	1.04
	0.60	4.33	1.38
	1.00	7.91	2.52
	1.60	10.17	3.24
	2.25	15.10	4.81
	3.00	15.32	4.88
sodium salicylate	0.00	3.14	
	0.90	3.23	1.03
	1.00	3.96	1.26
	1.60	5.89	1.87
	2.25	10.61	3.38
	3.00	10.83	3.45
urea	0.00	3.14	
	0.40	3.31	1.05
	0.50	5.90	1.88
	1.00	8.23	2.62
	1.60	14.00	4.46
	2.25	21.48	6.84
	3.00	21.63	6.89

Table 8. Setschenow Constant (K_s) Values of Hydrotropes with Respect to Methyl Salicylate

	K_s			
T/K	citric acid	sodium benzoate	sodium salicylate	urea
303	0.452	0.181	0.278	0.385
313	0.593	0.366	0.322	0.495
323	0.688	0.498	0.343	0.623
333	0.743	0.609	0.586	0.676

hydrotropes, namely sodium benzoate, sodium salicylate, and urea. The highest value of ϕ_{mtc} (7.88) has been observed in the presence of citric acid as hydrotrope at the C_{max} 2.00 mol/L.

Effectiveness of Hydrotropes

The effectiveness factor of each hydrotrope with respect to methyl salicylate at different system temperatures has been determined by analyzing the experimental solubility data for each case, applying the model suggested by Setschenow (1951) and later modified by Pathak and Gaikar (1992), as given by the equation

$$\log[S/S_m] = K_s[C_s - C_m] \tag{1}$$

where S and S_m are the solubilities of methyl salicylate at any hydrotrope concentration C_s and the minimum hydrotrope concentration C_m, 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, sodium benzoate, sodium salicylate, and urea, for the methyl salicylate + water system at different system temperatures are listed in Table 8. The highest value has been observed as 0.743 in the case of citric acid as hydrotrope.

Conclusions

The solubility of methyl salicylate, which is practically insoluble in water, has been increased to a maximum value of 22.84 in the presence of citric acid as hydrotrope with a corresponding increase in the mass-transfer coefficient. This would be very useful in increasing the rate of output of the desired product made from methyl salicylate. The separation of methyl salicylate 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 methyl salicylate can be used for the recovery of the dissolved methyl salicylate 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 methyl salicylate from its solution.

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Received for review July 1, 1999. Accepted December 23, 1999. One of the authors (M.D.K.) thanks CSIR for the grant of a Senior Research Fellowship during the tenure of this research work.

JE9901740