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Effect of Hydrotropes on Solubility and Mass Transfer Coefficient of *p*-Nitrobenzoic Acid

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Abstract: The effect of various hydrotropes such as sodium benzoate, sodium salicylate and nicotinamide on the solubility and mass transfer coefficient of *p*-nitrobenzoic acid was investigated. The solubility studies were carried out under a wide range of hydrotrope concentrations 0 to 3.0 kmol m^{-3} and different system temperatures (303-333 K). It has been observed that the solubility and mass transfer coefficient of *p*-nitrobenzoic acid increases with increase in hydrotorpe concentration and also with system temperature. The maximum enhancement factor, which is the ratio of the value in the presence and absence of a hydrotrope, has been determined for all sets of experimentations. The effectiveness of hydrotropes was measured in terms of Setschenow constant K_s and reported for all hydrotropes used in this study.

Key words: Hydrotropy, solubilization, mass transfer coefficient, separation

INTRODUCTION

Hydrotropy is a unique and unprecedented solubilization technique in which certain chemical compounds termed as hydrotropes can be used to effect a several fold increase in the solubility of sparingly soluble solutes under normal conditions (Neuberg, 1916). This increase in solubility in water is probably due to the formation of organized assemblies of hydrotrope molecules at critical concentrations (Badwan *et al*., 1982; Balasubramanian *et al*., 1989).

Hydrotropes are water-soluble and surface-active compounds, which can significantly enhance the solubility of organic solutes such as esters, acids, alcohols, aldehydes, ketones, hydrocarbons and fats (Friberg and Brancewicz, 1994; Raynaud Lacroze and Tavare, 1993; Laxman and Sharma, 1990; Maheswari *et al*., 2006; Gaikar and Phatak, 1993; Colonia *et al*., 1998). The solubility enhancement in the organic compounds could be due to the formation of molecular structures in the form of complexes (Hodgdon and Kaler, 2007; Ooya *et al*., 2005; Rovetto *et al*., 2006). Easy recovery of dissolved solute and possible reuse of hydrotrope solutions makes this most attractive particularly at industrial levels.

Besides the advantage of certain properties such as the solvent character being independent of pH, non-flammability, easy availability of hydrotropes, inexpensive aqueous phase makes this method superior to other solubilization methods (Agarwal and Gaikar, 1992a, b; Gaikar and Sharma, 1986; Mahapatra *et al*., 1988; Friberg *et al*., 1996; Gaikar and Phatak, 1999).

Hydrotropy is a process which goes beyond other conventional solubilization methods such as miscibility, co-solvency, salting-in etc., since it offers high selectivity and unprecedented increase in solubility and mass transfer coefficient. The problem of emulsification, which is normally encountered with surfactant solution, is not found with hydrotrope solution (Hefa and David, 2007). 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 (Nagendra Gandhi *et al*., 1998a, b; Nagendra Gandhi and Dharmendira Kumar, 2000a, b; Meyyappan and Nagendra Gandhi, 2004, 2005).

It has been observed that, in many two-phase reaction systems involving a sparingly soluble organic compound like *p*-nitrobenzoic acid, the mass transfer coefficient was found to be very low solely due to the

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poor solubility of *p*-nitrobenzoic acid in the aqueous phase. Since, *p*-nitrobenzoic acid serves as raw material/intermediate for a wide variety of chemicals and allied products and the separation of *p*-nitrobenzoic acid from any liquid mixture seems to be difficult, this hydrotropic technique can be adapted to increase the solubility as well as to separate such mixtures effectively (Agrawal *et al*., 2004; Dandekar *et al*., 2008). Data on various aspects of hydrotropic study on the solubility and mass transfer coefficient for *p*-nitrobenzoic acid-water system are reported for the first time.

MATERIALS AND METHODS

This study was carried out from November 2008 to February 2009 in Department of Chemical Engineering, St. Peter's Engineering College, Chennai-600 054, Tamil Nadu, India. All the chemicals used in this study were procured from S.D. Fine-Chem Ltd., Mumbai with a manufacturer's stated purity of 99%.

The experimental setup for the determination of solubility values consists of a thermostatic bath and a separating funnel. For each solubility test, an excess amount of powdered solid was taken in a separating funnel and 100 mL hydrotrope solution of known concentration was added. The separating funnel was immersed in a constant-temperature bath fitted with a temperature controller which could control the temperature within +0.1°C. The setup was kept overnight for equilibration. After equilibrium was attained, the solution was filtered from the remaining solid. The concentration of the dissolved organic acid in aqueous hydrotrope solutions was analyzed by titrating against standardized sodium hydroxide solution using 0.2 mL phenolphthalein solution as an indicator. Blank titration was carried out and necessary correction was done to calculate the dissolved organic acid in aqueous hydrotrope solutions. All the solubility experiments were conducted in duplicate to check the reproducibility. The observed error in the reproducibility was <2%.

The experimental setup for the determination of the mass transfer coefficient consisted of a vessel provided with baffles and a turbine impeller run by a motor to agitate the mixture. The vessel used for mass transfer studies is of height 40 cm and 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 height with a diameter of 1.5 cm. There are about 4 baffles that rotate at a speed of 600 rpm (Meyyappan and Nagendra Gandhi, 2004, 2005).

For each run, to measure the mass transfer coefficient, an excess amount of powdered solid 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 sec. After the end of fixed time t, the entire mixture was transferred to a separating funnel. After allowing the mixture to stand for some time, the solution was filtered from the remaining solid. The concentration of the solubilized organic acid in aqueous hydrotrope solutions at time t was analyzed in the same way as done for solubility determinations. A plot of -log $(1-C_b/C^*)$ versus t is drawn, where C_b is the concentration of *p*-nitrobenzoic acid at time t and C* is the equilibrium solubility of *p*-nitrobenzoic acid at the same hydrotrope concentration. 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%.

RESULTS

Solubility: Experimental data representing the effect of hydrotropes, i.e., sodium benzoate, sodium salicylate and nicotinamide on the solubility of *p*-nitrobenzoic acid are presented in Table 1-3. The solubility of *p*-nitrobenzoic acid in water at 303 K in the absence of any hydrotrope is 2.09×10^{-3} kmol m^{-3} (Table 1). The solubilization effect varies with concentration of hydrotropes (Table 1). From Table 1-3 it is evident that hydrotropy was operational above the certain significant hydrotrope concentration called Minimum Hydrotrope Concentration (MHC)

Table 1: Effect of sodium benzoate concentration (C) on the solubility (S) of *p*-nitrobenzoic acid in water

	10^3S (kmol m^{-3})			
C (kmol m^{-3})	T = 303 K	T = 313 K	T = 323 K	T = 333 K
0.00	2.09	2.54	3.06	3.65
0.10	2.16	2.51	3.14	3.61
0.20	2.20	2.68	3.21	3.72
0.30 (MHC)	2.97	3.91	4.79	5.21
0.40	3.72	5.61	6.83	8.19
0.50	4.90	8.34	9.20	13.73
0.60	7.50	9.95	14.05	17.93
0.70	10.10	11.73	15.41	19.38
0.80	11.58	13.18	18.79	23.64
0.90	14.39	17.32	21.62	26.57
1.00	16.60	20.79	26.80	30.91
1.20	17.70	24.15	32.74	40.63
1.40	22.00	28.19	36.18	45.68
1.60	24.50	33.71	39.12	54.67
1.80	26.08	37.25	47.49	60.31
2.00	27.34	38.36	50.47	65.72
2.25	28.31	40.08	52.82	71.68
2.50 (C_{max})	29.54	41.76	56.91	76.71
2.75	29.69	41.87	57.16	76.87
3.00	29.74	42.01	57.21	76.94

Table 2: Effect of sodium salicylate concentration (C) on the solubility (S) of *p*-nitrobenzoic acid in water

	10^3 S (kmol m^{-3})			
C (kmol m^{-3})	T = 303 K	T = 313 K	T = 323 K	T = 333 K
0.00	2.09	2.54	3.06	3.65
0.10	2.13	2.71	3.13	3.72
0.20	2.28	2.86	3.18	3.85
0.30	2.31	2.92	3.24	4.02
0.40 (MHC)	3.78	4.79	6.87	8.63
0.50	5.91	8.03	10.78	12.56
0.60	7.28	10.67	13.74	17.62
0.70	8.62	13.51	18.56	23.89
0.80	9.54	14.78	23.61	29.58
0.90	12.36	16.83	27.47	34.04
1.00	13.24	19.67	30.72	37.71
1.20	14.76	21.09	33.49	43.75
1.40	16.07	25.71	35.36	49.09
1.60	17.43	27.62	36.98	52.86
1.80	18.18	28.14	38.83	56.31
2.00 (C_{max})	19.59	29.05	40.28	59.07
2.25	19.68	29.12	40.31	59.16
2.50	19.86	29.21	40.46	59.29
2.75	20.02	29.31	40.59	59.36
3.00	20.08	29.36	40.64	59.42

Table 3: Effect of nicotinamide concentration (C) on the solubility (S) of *p*-nitrobenzoic acid in water

	10^3S (kmol m^{-3})			
C (kmol m^{-3})	T = 303 K	T = 313 K	T = 323 K	T = 333 K
0.00	2.09	2.54	3.06	3.65
0.10	2.05	2.63	3.14	3.74
0.20	2.14	2.72	3.23	3.78
0.30	2.19	2.80	3.36	3.81
0.40	2.25	2.86	3.56	3.87
0.50 (MHC)	2.98	3.94	4.86	5.06
0.60	3.49	4.49	5.52	7.11
0.70	3.61	5.12	7.79	9.64
0.80	3.87	5.71	8.72	11.37
0.90	4.66	6.56	10.28	13.72
1.00	5.01	8.47	12.37	15.47
1.20	6.27	9.82	13.91	17.49
1.40	7.92	12.36	15.97	22.71
1.60	8.50	13.81	18.17	24.63
1.80	9.47	14.37	19.65	26.17
2.00 (C_{max})	10.41	16.17	21.23	29.04
2.25	10.49	16.21	21.36	29.11
2.50	10.53	16.35	21.47	29.20
2.75	10.64	16.58	21.59	29.38
3.00	10.70	16.83	21.83	29.47

of sodium benzoate (0.30 kmol m^{-3}), sodium salicylate (0.4 k mol m^{-3}) and nicotinamide (0.5 kmol m^{-3}) irrespective of system temperature. This MHC value assumes greater significance in the context of recovery of hydrotrope solutions. This increasing trend is maintained only up to a certain concentration of hydrotrope in the aqueous solution, beyond which there is no appreciable increase in the solubility of *p*-nitrobenzoic acid. This concentration of hydrotrope in the aqueous solution is referred to as the maximum (Table 4), which seem to depend on the hydrophilicity of a hydrotrope. As can be seen from Table 4, C_{max} values of sodium benzoate, sodium salicylate and nicotinamide are 2.50, 2.00 and 2.00 kmol m^{-3}, respectively. The maximum solubilization enhancement factor (ϕ_s) which is the ratio of solubility values in the presence and absence of hydrotrope, ranges between 5.12 and 21.08. The low value of ϕ_s 5.12 was observed in the presence of nicotinamide at a system temperature of 303 K. The highest value of ϕ_s 21.08 has been observed in the case of sodium benzoate at a system temperature of 333 K (Table 5).

Table 4: MHC and C_{max} values for Hydrotropes

Hydrotrope	MHC(kmol m^{-3})	C_{max}(kmol m^{-3})
Sodium benzoate	0.3	2.5
Sodium salicylate	0.4	2.0
Nicotinamide	0.5	2.0

Table 5: Maximum solubilization enhancement factor (ϕ_s) of *p*-nitrobenzoic acid

	Maximum enhancement factor for solubility (ϕ_s)			
Hydrotrope	T = 303 K	T = 313 K	T = 323 K	T = 333 K
Sodium benzoate	14.23	16.54	18.70	21.08
Sodium salicylate	9.61	11.56	13.28	16.28
Nicotinamide	5.12	6.63	7.13	8.07

Mass transfer coefficient: The mass-transfer coefficient of *p*-nitrobenzoic acid+water system in the absence of any hydrotrope was determined as 0.96×10^{-3} s^{-1} at 303 K (Table 6). The reported mass transfer coefficient enhancement (ϕ_{mtc}) of *p*-nitrobenzoic acid of hydrotrope solutions are 15.20 for sodium benzoate, 10.10 for sodium salicylate and 5.43 for nicotinamide at their maximum hydrotrope concentrations. The highest value of ϕ_{mtc} (15.20) has been observed in the presence of sodium benzoate as hydrotrope at C_{max} of 2.50 kmol m^{-3}.

Effectiveness of hydrotropes: The effectiveness factor of each hydrotrope with respect to *p*-nitrobenzoic acid 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:

$$\log[S / S_m] = K_s[C_s - C_m] \quad (1)$$

where, S and S_m are the solubility of *p*-nitrobenzoic acid at any hydrotrope concentration C_s and the minimum hydrotrope concentration C_m, respectively.

The Setschenow constant values of hydrotropes namely sodium benzoate, sodium salicylate and nicotinamide for p-nitrobenzoic acid+water system at different system temperatures are listed in Table 7.

Table 6: Effect of hydrotrope concentration (C) on the mass transfer coefficient (k_La) of *p*-nitrobenzoic acid

Hydrotrope	C (kmol m^{-3})	$k_La \times 10^3$ sec^{-1}	Enhancement factor for mass transfer coefficient (ϕ_{mtc})
Sodium	0.00	0.96	-
Benzoate	0.20	1.09	1.14
	0.30 (MHC)	1.57	1.63
	0.40	1.72	1.79
	0.60	3.08	3.21
	0.80	5.13	5.34
	1.00	8.13	8.47
	1.20	8.95	9.32
	1.40	10.27	10.70
	1.60	12.49	13.01
	1.80	13.16	13.70
	2.00	13.84	14.42
	2.25	14.07	14.66
	2.50 (C_{max})	14.59	15.20
	2.75	14.71	15.32
	3.00	14.83	15.45
Sodium	0.00	0.96	-
Salicylate	0.20	1.13	1.18
	0.40 (MHC)	1.91	1.99
	0.60	3.24	3.38
	0.80	4.49	4.68
	1.00	6.31	6.57
	1.20	7.08	7.38
	1.40	8.03	8.36
	1.60	8.59	8.95
	1.80	9.03	9.40
	2.00 (C_{max})	9.70	10.10
	2.25	9.81	10.22
	2.50	9.86	10.27
	2.75	9.98	10.40
	3.00	10.02	10.43
Nicotinamide	0.00	0.96	-
	0.20	1.06	1.04
	0.40	1.12	1.17
	0.50 (MHC)	1.53	1.59
	0.60	1.71	1.78
	0.80	1.96	2.04
	1.00	2.54	2.65
	1.20	3.10	3.23
	1.40	3.61	4.07
	1.60	4.17	4.34
	1.80	4.63	4.82
	2.00 (C_{max})	5.21	5.43
	2.25	5.24	5.46
	2.50	5.27	5.49
	2.75	5.38	5.60
	3.00	5.42	5.65

Table 7: Setschenow constant (K_s) of hydrotropes with respect to *p*-nitrobenzoic acid

	Setschenow constant (K_s)			
Hydrotrope	T = 303 K	T = 313 K	T = 323 K	T = 333 K
Sodium benzoate	0.455	0.468	0.489	0.531
Sodium salicylate	0.446	0.458	0.48	0.513
Nicotinamide	0.362	0.408	0.427	0.506

DISCUSSION

The solubility of *p*-nitrobenzoic acid in water is 2.09×10^{-3} kmol m^{-3} at 303 K, compared to 1.197×10^{-3} kmol m^{-3} at 288 K. Thus, the solubility values in water are in excellent agreement with the earlier reported values (John, 1987; Perry, 1997). Sodium benzoate is one of the hydrotropes used in this study. It was observed that the solubility of *p*-nitrobenzoic acid did not show any appreciable increase up to addition of 0.30 kmol m^{-3} of sodium benzoate. On subsequent increase in the concentration of sodium benzoate above 0.30 kmol m^{-3}, the solubility of *p*-nitrobenzoic acid in water was found to increase significantly. This concentration of sodium benzoate in the aqueous phase is termed as Minimum Hydrotrope Concentration (MHC), which is the minimum required sodium benzoate concentration in the aqueous phase to effect significant increase in the solubility of *p*-nitrobenzoic acid in water (Miguel *et al*., 2007). It has been observed that the MHC of sodium benzoate in the aqueous phase does not vary even at increased temperatures. A similar trend in MHC requirement in the aqueous phase has also been observed for other hydrotropes. In the present case, a clear increasing trend in the solubility of *p*-nitrobenzoic acid was observed above the MHC of sodium benzoate. This increase in solubility of *p*-nitorbenzoic acid is maintained up to 2.50 kmol m^{-3} of sodium benzoate in the aqueous phase, beyond which there is no appreciable increase in the solubility of *p*-nitrobenzoic acid. This concentration of sodium benzoate in aqueous phase is referred to as the maximum hydrotrope concentration (C_{max}). From the analysis of the experimental data, it is observed that further increase in hydrotrope concentration beyond C_{max} does not bring any appreciable increase in the solubility of *p*-nitrobenzoic acid even up to 3.00 kmol m^{-3} of sodium benzoate in the aqueous solution. Similar to the MHC values, C_{max} values of hydrotropes also remained unaltered at increased system temperatures.

The knowledge of MHC and C_{max} values of each hydrotrope with respect to a particular solute assumes greater significance in this study since, it indicates the beginning and saturation of the solubilization effect of hydrotropes. The values of MHC and C_{max} of a hydrotrope with respect to *p*-nitrobenzoic acid may be useful in determining the recovery of the dissolved *p*-nitrobenzoic acid by simple dilution with distilled water. This is the unique advantage of the hydrotropic solubilization technique. Since, hydrotropy appears to operate only at significant concentrations of hydrotrope in water, most hydrotropic solutions release the dissolved *p*-nitrobenzoic acid on dilution with water below MHC. The knowledge of MHC values is necessary especially at industrial levels, as it ensures ready recovery of the hydrotrope for reuse. It can further be observed from results that, in order to achieve the particular solubility of *p*-nitrobenzoic acid, say 16.6×10^{-3} kmol m^{-3}, the sodium benzoate concentration should be 1.00 kmol m^{-3} at 303 K,

0.90 kmol m^{-3} at 313 K, 0.70 kmol m^{-3} at 323 K and 0.60 kmol m^{-3} at 333 K in the aqueous solution. Thus, it can be seen that as the system temperature increases, the concentration of sodium benzoate required in the aqueous phase to achieve a particular solubility of *p*-nitrobenzoic acid decreases. A similar trend has been observed for other systems also. It has also been observed that the solubilization effect of sodium benzoate was not a linear function of the concentration of the sodium benzoate. The solubilization effect of sodium benzoate increases with increase in hydrotrope concentration and also with system temperature (Wagle *et al*., 2007).

The effect of different hydrotropes on the mass transfer coefficient of *p*-nitrobenzoic acid at different hydrotrope concentrations is also given in the Table 6. It can be seen that a threshold value of 0.30 kmol m^{-3} is required to effect significant enhancement in the mass transfer coefficient of *p*-nitrobenzoic acid+water system, as observed in the case of solubility determinations. The mass-transfer coefficient of *p*-nitrobenzoic acid+water system increases with increase in sodium benzoate concentration. Beyond a C_{max} of 2.50 kmol m^{-3} there is no appreciable increase in the mass transfer coefficient of *p*-nitrobenzoic acid, as observed in the case of solubility determinations. The maximum enhancement factor for mass transfer coefficient of *p*-nitrobenzoic acid+water system in the presence of sodium benzoate was found to be 15.20 (Table 6). A similar trend in the mass transfer coefficient enhancement (ϕ_{mtc}) of *p*-nitrobenzoic acid has been observed for other hydrotropes also namely sodium salicylate and nicotinamide. 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 highest value has been observed as 0.531 in the case of sodium benzoate as hydrotrope at 333 K. The order of effectiveness of various hydrotropes based on K_s values is given by sodium benzoate>sodiumsalicylate> nicotinamide.

CONCLUSION

The solubility of *p*-nitrobenzoic acid, which is practically insoluble in water, has been increased to a maximum of 21.08 times in the presence of sodium benzoate as hydrotrope. The mass transfer coefficient was also found to increase to a maximum value of 14.59×10^{-3} sec^{-1} and with an enhancement factor of about 15.20 times in the presence of sodium benzoate as hydrotrope. This would be useful in increasing the rate of output of the desired product made from *p*-nitrobenzoic acid. The MHC and C_{max} values of the hydrotrope with respect to *p*-nitrobenzoic acid can be used for the recovery of the dissolved *p*-nitrobenzoic acid 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 *p*-nitrobenzoic acid 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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