J. Chem. Eng. Data 1997, 42, 631−635

Separation of Eutectics of Chloronitrobenzenes through Hydrotropy

Narayan S. Tavare*,† **and Edecio J. Colonia**‡

Department of Chemical Engineering, University of Bradford, West Yorkshire BD7 1DP, U.K.

The hydrotrope sodium butyl monoglycol sulfate for the separation of mixtures of *o*-, *m*-, and *p*-chloronitrobenzenes was explored at the eutectic compositions. Three ternary diagrams were constructed using the single- and two-component equilibrium compositions of binary mixtures at different hydrotrope concentrations together with eutectic and two-component saturation tie lines. It was possible to separate two of the three binary simple and the ternary eutectics because sodium butyl monoglycol sulfate was able to shift their equilibrium curves away from the eutectic tie lines.

Introduction

Hydrotropes have been used for the separation of difficult-to-separate solid mixtures. For this purpose, a simple technique is employed which involves either solubilization and precipitation (i.e., the solubilization of the mixture in a hydrotrope solution and subsequent selective precipitation of a desired component by controlled dilution with water) or leaching (i.e., controlled partial solubilization so that one of the components remains in the solid phase with an improved purity). Both these techniques can be conveniently employed to effect the separation of the component at higher purity and reasonable recovery. Geetha *et al.* (1991) obtained *o*-chloronitrobenzene (CNB) with >99% purity and ∼70% recovery from its mixture with *p*-CNB using sodium butyl monoglycol sulfate (NaBMGS) and demonstrated its effectiveness and simplicity over the other methods based on solvent extraction (Hanson and Patel, 1966) and extractive crystallization (Dikshit and Chivate, 1970; Tare and Chivate, 1976). Conventional methods based on a combination of fractional crystallization and vacuum distillation appear attractive in terms of separation duty (i.e., thermal load and theoretical stages) but have major limitations on their ability to separate pure components when the eutectic, molecular addition compound, or azeotrope is encountered.

Employing the solubilization and precipitation technique, Raynaud-Lacroze and Tavare (1993) used sodium cumenesulfonate (NaCS) for typical product mixtures from industrial reactors to separate 2-naphthol with >99% purity (starting from 85%) and 80% mass recovery from 1-naphthol. Using sodium cumenesulfonate, Colonia *et al.* (1993) were able to separate *p*-chlorobenzoic acid from both ortho−para mixtures of the typical industrial product (42% mass *p*-chlorobenzoic acid) and eutectic composition (14% mass *p*-chlorobenzoic acid) with 92 and 82% mass purities and ∼70−75% mass recoveries, respectively. Dixit *et al.* (1996a,b, 1997) extended the work to look systematically at the use of the NaBMGS hydrotrope for the separation of *o*- and *p*-chlorobenzoic acids from their mixtures. In order to examine the efficacy of the technique, precipitation experiments were performed with the mixtures of *o*- and *p*-chlorobenzoic acids over a wide range of composition mixtures. The location of the initial composition and the path followed by the process on the ternary phase equilibrium diagram decided the precipitating component. The effects of two important process variables on the final product crystal size distribution and the influence on the relative nucleation kinetics were investigated. The specificity in solubilization has been successfully employed not only for the separation of close boiling components from binary mixtures forming simple eutectics (Colonia and Tavare, 1994) but also both close boiling and close melting components like *m*- and *p*-aminoacetophenones (Jadhav *et al.*, 1995) and for achieving improved quality and purity of pharmaceutical intermediates such as 6-aminopenicillanic acid (Tavare and Jadhav, 1996).

Table 1. Eutectics between *o*-, *m*-, and *p*-Chloronitrobenzenes: Composition and Melting Points, t_m (An, 1977; Dikshit and Chivate, 1970)

composition, mol %			
o-CNB	*m*-CNB	*p*-CNB	t_m °C
Binary Simple Eutectics			
	62.2	37.8	25.3
66.9		33.1	14.6
59.0	41.0		10.5
Ternary Eutectic			
42.5	36.6	20.9	6.1

The separation of eutectic mixtures, however, is particularly difficult, requiring unconventional techniques such as adductive or extractive crystallization and a strategy of selective reversible reactions (Tavare, 1995). Since the technique of selective precipitation from hydrotropic solutions for separation purposes is relatively simple, it was employed for eutectic mixtures. Raynaud-Lacroze and Tavare (1993) showed that the eutectic composition of 1- and 2-naphthols could not be broken with NaCS, while Colonia *et al.* (1993) demonstrated the feasibility of separating that of *o*- and *p*-chlorobenzoic acids and *o*- and *p*-CNBs using NaCS (see also Colonia and Tavare, 1994).

Large quantities of CNBs are produced for the manufacture of organic intermediates, *o*- and *p*-CNBs being of technical importance. The boiling points of *o*-, *m*-, and *p*-CNBs are ∼246, 236, and 242 °C and the melting points 32.5, 44.4, and 83.5 °C, respectively. All these isomers form three binary simple eutectics and ternary eutectics as shown in Table 1. The ternary phase equilibrium for *o*-, *m*-, and *p*-CNBs is reproduced in Figure 1 (An, 1977; Dikshit and Chivate, 1970). The purpose of this paper is to explore further the possibility of separation of mixtures of *o*-, *m*-, and *p*-CNBs at the eutectic composition, i.e., the three binary and the ternary mixtures) using the commercially available NaBMGS hydrotrope. These eutectic compositions of solid mixtures are chosen due to the difficulties associated in their separation by conventional methods.

[†] E-mail address: n.s.tavare@bradford.ac.uk. Fax number: ++44 1274 385700.

[‡] Deceased.

S0021-9568(96)00405-0 CCC: $14.00

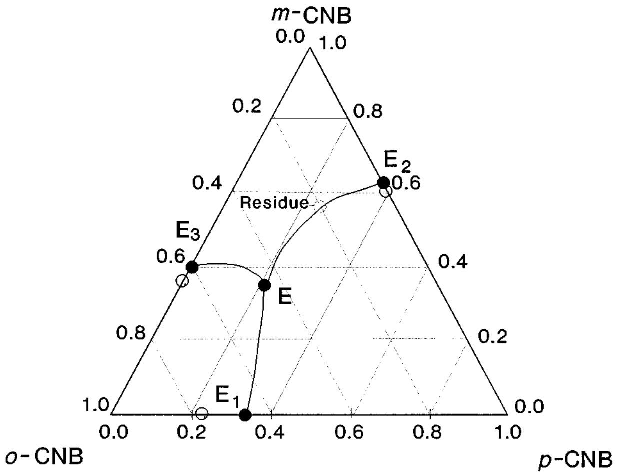

Figure 1. Ternary phase diagram. Simple ternary eutectics of chloronitrobenzenes (Table 1): (○) two-component saturation point with the NaBMGS hydrotrope; (●) eutectic; (◒) residue from leaching experiment at 3 °C.

Ternary Phase Equilibrium Diagrams

The construction of the ternary phase equilibrium diagram is essential for the prediction of the efficacy of a given hydrotrope to separate the eutectic composition of the two solubilizates. Solubility of a single, pure isomeric component was determined by both mass disappearance and spectroscopic methods and the solubility curves for *o*- and *p*-CNBs as a function of hydrotrope concentration were constructed at 12 °C (Geetha *et al.*, 1991). The mass disappearance method was used in this study. All Analar grade CNBs were further purified by the sweating process (Matsuoka *et al.*, 1986). In order to measure the mass of the disappeared component in the solution phase during the equilibration step in the mass disappearance method, a known and excess amount of solubilizate was equilibrated in the hydrotrope solution of known concentration for several hours (at least 6 h; usually overnight (~24 h)) in a magnetically stirred and jacketed vessel (of ~100 mL capacity) maintained at a constant temperature (within ±0.1 deg) by circulating water from a thermostatic water bath. The bath was also provided with a freeze unit for operations below the ambient temperature. A commercial aqueous solution of NaBMGS50 (with 50 mass %) was supplied by Hüls (U.K.) Ltd., Milton Keynes. All other hydrotrope concentrations were achieved by dilution of the neat hydrotrope with water. All hydrotrope concentration percentages were expressed in a mass/mass basis. The slurry was then filtered under vacuum through filter paper (Whatman no. 1) at the same temperature as that of the bath. When the operating temperature was not in the range of ambient, a jacketed filter coupled to the thermostatic bath was employed. The vessel containing the slurry was disconnected from the water circulating system but its jacket was kept full of water at the operating temperature and the vessel submerged in the bath throughout the filtration. The residue on the filter paper (Whatman no. 1) was dried in an oven kept at 60−65 °C and weighed on an electronic balance with 0.001 g precision and the difference in masses between the initial charge and final residue determined. The solubility was calculated as the amount dissolved per unit mass of water, thus requiring us to evaluate the amount of water present in the hydrotrope solution of a given concentration. The amounts of solids in both the solid and solution phases were kept at the same order of magnitude. Generally, the undissolved solids were in the range of 25−30 mass % of the solids initially charged into the vessel. The solubility data (expressed in grams of component/grams of water) were used to construct solubility curves. These typical sigmoidal curves indicated that the solubility increased rapidly above the critical hydrotrope concentration until a point of inflection. Beyond this point the rate of increase of the solubility with the hydrotrope concentration steadily decreased so that the solubility eventually approached a straight line parallel to the abscissa asymptotically.

In this study, for a data point on a saturation line with respect to both components at any hydrotrope concentration, an excess amount of the mixture of two components (i.e., the amount of each of them was more than the solution could dissolve) was added to the NaBMGS solution of desired concentration. Again, the undissolved solids should account for a reasonable fraction of the dissolved solids. The solution was then stirred at the desired temperature for ~24 h using a magnetic stirrer in a similar jacketed glass vessel for equilibration. Undissolved solids were separated by filtration of the entire slurry through (Whatman no. 1) filter paper, and the residue on the filter paper was dried, weighed, and subsequently used for component analysis. The composition of *o*-, *m*-, and *p*-CNB in the dry solids was determined by gas chromatography. At the start of each session, a set of standards was always analyzed prior to the analysis of any sample. The majority of the analyses were made at least in duplicate and only results close enough to each other were recorded as an average during the initial stages of standardization. In most cases results were reproducible within 1%. The dry CNB solids were diluted in methanol to 10 mg of total solids/g of methanol and analyzed in a Perkin-Elmer gas chromatograph provided with a BP10 medium polarity column initially using an oven temperature of 118 °C, injection temperature of 168 °C and detector temperature of 300 °C. These conditions were slightly altered to match the actual compositions of the standards. The mother liquor compositions were not analyzed in all the cases but instead determined by difference. Only in a few initial cases was the material balance check performed by analyzing CNB mother liquor after extracting solid hydrotrope from the solution by methanol. It was satisfactory within 5−10%. The two-component saturation tie line was drawn by joining the water apex with the data points representing solutions saturated with respect to both solutes for different hydrotrope concentrations. The eutectic tie line, on the other hand, was obtained by simply linking up the water apex with any data point representing the simple eutectic composition. Table 2 shows all the compositions (milligrams of component/gram of water) of solutions saturated with respect to two components as a function of NaBMGS hydrotrope concentration for the three binaries, viz. *o*- and *m*-, *m*- and *p*-, and *p*- and *o*-CNBs, at 7, 17, and 12 °C, respectively. The data point representing the composition of solution saturated with respect to two components in a binary component system provides a characteristic limit on the solubility of the components in the mixture and is invariant when the temperature and hydrotrope concentration are fixed.

In addition, the solubility data were obtained for binary mixtures of known CNB composition by the crystal disappearance method. In this method, the amount of known hydrotrope solution required just for complete disappearance of solid crystalline phase by visual observation was determined. The total amount of the hydrotrope solution slowly added to the slurry up to the disappearance point, where no crystal (undissolved solids) could any longer be seen by the naked eye, was used to determine a data point on an *isoplethal* solubility curve. Unless the initial com-

Table 2. Composition of Solution Saturated with Two Respective Chloronitrobenzenes

hydrotrope concn, g of NaBMGS/g of H_2O	composition, mg/g of H_2O					
	o- + *m*-CNB (t = 7 °C)		*m*- + *p*-CNB (t = 17 °C)		*p*- + *o*-CNB (t = 12 °C)	
	o	*m*	*m*	*p*	*p*	*o*
1.0	83.9	43.7	37.9	26.9	27.5	97.5
0.7	56.2	27.3	24.9	14.4	14.2	48.3
0.5	37.0	18.3	15.0	8.9	4.8	15.6
0.4	18.5	11.8	11.7	8.4	4.2	14.8
0.3	12.6	7.8	10.1	6.5	1.5	5.5

Table 3. Solubility of a Saturating Component in Binary Mixtures of Chloronitrobenzenes in Aqueous NaBMGS Solutions

hydrotrope concn, g of NaBMGS/g of H_2O	composition, mg/g of H_2O					
	o- + *m*-CNB (t = 7 °C)		*m*- + *p*-CNB (t = 6 °C)		*p*- + *o*-CNB (t = 12 °C)	
	o	*m*	*m*	*p*	*p*	*o*
1.0	13.7	0.0	12.0	0.0	0.0	13.7
	13.6	4.5	13.5	4.5	4.5	13.6
	9.5	9.5	12.3	12.3	9.5	9.5
	3.6	10.9	3.9	11.5	10.9	3.6
	0.0	11.3	0.0	11.4	11.3	0.0
0.7	11.0	0.0	0.0	9.9	0.0	11.0
	9.8	3.2	3.9	11.6	3.2	9.8
	5.5	5.5	8.9	8.9	5.5	5.5
	2.2	6.6	7.1	2.4	6.6	2.2
	0.0	6.6	6.6	0.0	6.6	0.0
0.5	7.1	0.0	0.0	3.5	0.0	7.1
	9.3	3.1	1.9	5.7	3.1	9.3
	4.2	4.2	5.0	5.0	4.2	4.2
	1.1	3.4	3.4	1.2	3.4	1.1
	0.0	4.0	4.0	0.0	4.0	0.0

position happened to be that of the two-component saturation point, the resulting solution was assumed to be saturated with respect to only one of the components. In order to measure the mass of the hydrotrope solution added in the solution phase during the equilibration step, a near saturated solution was prepared by adding a known amount of NaBMGS solution to a binary solid CNB mixture and maintained in a magnetically stirred and jacketed vessel (of ~100 mL capacity) at a constant temperature (within ±0.1 deg) by circulating water from a thermostatic water bath. The remaining small amount of known NaBMGS hydrotrope solution was then added very slowly till the last crystal disappeared and a homogeneous solution was achieved. The results of such measurements for the three mixtures at three different hydrotrope concentrations are reported in Table 3. The solubility was initially calculated in milligrams of solute/gram of water. The results were quite reproducible within ±0.2 mg/g of water. The solubility data obtained by this method were, however, slightly less than those obtained by the weight disappearance method under otherwise similar conditions. These data were converted into mol % (i.e., in the form of relative concentrations of solute 1−solute 2−water, all in mol %) using molecular weights of all species to construct the hydrotrope *isoplethal* solubility curves of the saturating component in the binary mixture, i.e., hydrotrope *isopleths* for CNB equilibrium concentrations in the ternary phase equilibrium diagrams. These smooth curves were drawn through the data points. These *isopleths* have a threshold or characteristic point, usually at minimum, as represented by the two-component saturation data point. This point splits the *isopleths* into two primary parts, each being a saturation curve with respect to the primary component and undersaturated with respect to the other component of the binary mixture. Naturally, the primary component will precipitate out first in the region, as the solution will be supersaturated with respect to this component. The locus of such two-component saturation points for different hydrotrope concentrations is referred to as the two-component saturation tie line. Also plotted in these ternary diagrams are the eutectic tie lines, representing the locus of the points (solutions) whose solubilizate composition is that of the simple binary eutectic. This two-component saturation curve divides the hydrotrope *isopleths* into two sections. Thus, the single- and two-component equilibrium compositions (pure and binary mixtures) for different hydrotrope concentrations, together with eutectic and two-component saturation tie lines, are all the data used for the construction of the three ternary diagrams shown in Figures 2−4. For the sake of clarity only the enlarged triangular section of the water apex of the ternary diagram depicting three hydrotrope *isoplethal* solubility curves are shown. Also included in Figure 1 are the two-component saturation data points for these binary systems.

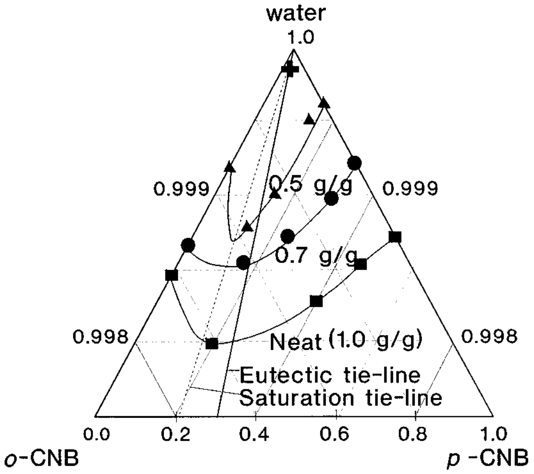

Figure 2. Ternary phase diagram for the *o*-CNB−*p*-CNB−water system with the NaBMGS hydrotrope (data from Tables 2 and 3). Solubilization and precipitation experiment (Table 4): (**x**) charge (feed) (not shown); (**+**) mother liquor.

Separation Feasibility

Two types of equilibration experiments, i.e., solubilization and precipitation, and leaching, were performed at

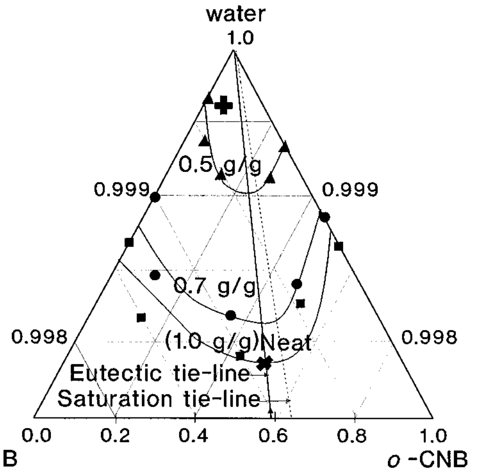

Figure 3. Ternary phase diagram for the *o*-CNB−*m*-CNB−water system with the NaBMGS hydrotrope (data from Tables 2 and 3). Solubilization and precipitation experiment (Table 4): (**x**) charge (feed); (+) mother liquor.

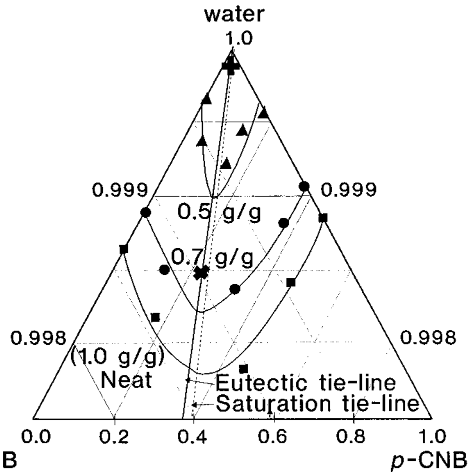

Figure 4. Ternary phase diagram for the *m*-CNB−*p*-CNB−water system with the NaBMGS hydrotrope (data from Tables 2 and 3). Solubilization and precipitation experiment (Table 4): (**x**) charge (feed) (+) mother liquor.

temperatures at least 2.5 deg below the melting point of each mixture. Geetha *et al.* (1991) employed a simple technique of solubilization and precipitation for the separation of mixtures of ordinary composition. Their equilibrium precipitation carried out as a single stage batch operation indicated that the optimal amount of dilution water for their system, *o*- and *p*-CNBs with NaBMGS hydrotrope, was about twice that of the initial neat hydrotrope solution. A similar procedure was used in the solubilization and precipitation technique.

In this work, the solid mixtures at the eutectic composition were prepared in 100−200 mL jacketed vessels, magnetically stirred, and maintained at constant temperature by means of a water bath. In these solubilization and precipitation experiments, known amounts of eutectics were dissolved in NaBMGS solutions of different hydrotrope concentrations. The method of crystal disappearance point was employed, whereby the solvent is slowly added up to the point where no crystal can be seen any longer. The resulting solution was diluted with an amount of water equaling twice that of the hydrotrope solution consumed, and the suspension, after being allowed to settle, was filtered in a jacketed filter maintained at the same temperature as the vessels. The solids were dried overnight at 60−65 °C in an oven, weighed, and dissolved in methanol. Both solutions of solids and mother liquor in methanol were analyzed by gas chromatography. In the leaching experiments, as excess amount (with respect to all components) of the eutectic composition was charged into a known amount of hydrotrope solution. After equilibration at constant temperature, the suspensions were treated just like in the previous technique.

Table 4. Solubilization and Precipitation Experiments at Operating Temperature, t_o, with Neat NaBMGS at Binary Eutectic Composition of Chloronitrobenzenes

			composition, mol %		
mixture	t_m, °C	t_o, °C	eutectic (charge)	precipitate	mother liquor
o/*m*	10.5	7	59.0 *o*	59.8 *o*	59.8 *o*
m/*p*	25.3	17	62.2 *m*	62.3 *m*	61.3 *m*
p/*o*	14.6	12	33.1 *p*	31.8 *p*	35.7 *p*

In such instances, the system ternary phase equilibrium diagram is of fundamental importance. The relative position of the eutectic tie line and the two-component saturation tie line would suggest whether the eutectic composition could somehow be broken. The degree to which the separation could be effected would depend on the distance between these two tie lines. Consequently, when these tie lines coincided, it would be impossible to break the eutectic using the particular hydrotrope for which the valid ternary diagram was available.

In all the solubilization and precipitation experiments the composition of the CNBs in the precipitate and mother liquor were roughly the same as that of the eutectic point (Table 4); therefore no separation was achieved. For eutectic compositions the solubilization and precipitation technique was not effective. The separation technique employed for the eutectic composition was an operation that consisted of leaching the excess solids of a suspension. It involved the partial solubilization of the mixture of solutes so that one of them remained in the solid phase with an improved purity. The experiments were conducted in 100−250 mL magnetically stirred jacketed vessels kept at a constant temperature by circulating water from a water bath. The precipitated solids obtained from dilution of the equilibrated solutions, as well as the mother liquors, were analyzed by gas chromatography in order to check the closure of the material balance. When an excess amount of mixture at the eutectic composition was charged into a hydrotrope solution, one of the following possibilities would occur: (a) the excess solids, which constituted the solid phase (slurry) would precipitate out with the eutectic composition, thus resulting in a solution phase (mother liquor) with that same solute composition and therefore not effecting any separation; (b) the mother liquor would have the same composition as that of the two-component saturated solution and, consequently, the solid phase has a composition different from those of the eutectic and of two-component saturation one. This meant that the slurry got enriched in one of the components while the mother liquor got enriched in the other and vice versa. The leaching of the excess solids at the eutectic composition, however, resulted in some separation, particularly for two (*o* and *p*, and *o* and *m*) binaries (Table 5) and for the ternary. Indeed, in the binary eutectics where the separation was achieved, the saturation tie line with respect to both components in NaBMGS solutions clearly shifted away from the eutectic tie line in the ternary phase equilibrium diagrams (see Figures 2−4). Also included in the figures are the data points representing the feed and mother liquor of the solubilization and precipitation experiments. For the ternary eutectic point (42.5 mol % *o*, 36.6 mol % *m*, 20.9 mol % *p*, and 6.1 °C), a typical leaching experiment

Table 5. Leaching Experiments at Operating Temperature, t_o, with Neat NaBMGS at Binary Eutectic Composition

amt of charge, mg	t_o °C	amt of NaBMGS, g	residue mass, mg	residue composition, mol %
3299 *o*/*m*	7	40.2	642	34.1 *o*
3004 *m*/*p*	17	67.7	668	47.9 *m*
2563 *p*/*o*	12	39.4	264	8.8 *p*

at 3 °C with 1504 mg of the mixture and 31.0 g of neat NaBMGS (50 mass %) produced 649 mg of residue (undissolved solids, see Figure 1) containing 18.4 mol % *o*, 58.3 mol % *m*, and 23.3 mol % *p*, and indicating a change in composition. In such experiments, the recovery and enrichment of a particular isomer depend on the relative amounts of initial solid mixture and hydrotrope solution.

Conclusions

In this study the use of the hydrotrope sodium butyl monoglycol sulfate (NaBMGS) was extended for the separation of eutectics of *o*-, *m*-, and *p*-chloronitrobenzenes (CNBs) at the eutectic compositions. Three phase equilibrium diagrams for the two of three CNBs components and water systems with NaBMGS as a hydrotrope were conveniently depicted on a concentration triangular diagram. In addition to solubility data of the pure components, solubility data for a component in the presence of the other component and hydrotrope at different concentration levels were used to construct three hydrotrope *isoplethal* (i.e., the same hydrotrope concentration) solubility curves. The two component saturation tie line was drawn through all data points at different hydrotrope concentrations and the eutectic tie line joins the binary eutectic point and water apex. The ternary diagrams indicated the possibility of separating two of the three binary simple and the ternary eutectics because sodium butyl monoglycol sulfate (NaBMGS) was able to shift their equilibrium curves away from the eutectic tie lines. The exploratory feasibility study does warrant further systematic investigation on the separation of eutectics through the phenomenon of hydrotropy.

Acknowledgment

The experimental work reported in this paper was performed at the Department of Chemical Engineering, UMIST, Manchester, M60 1QD, England. The free supply of hydrotrope from Hüls (U.K.) Ltd. is appreciated.

Literature Cited

An, So Song. Studies on the separation of *o*- and *p*-chloronitrobenzene Part 2. Solid-liquid equilibrium of a three component system. *Hwahak Kwa Hwahak Kongopi* **1977**, *20*, 289−294.

Colonia, E. J.; Tavare, N. S. Separation of eutectics through hydrotropy. Proceedings of the 1994 IChemE Research Event; Institution of Chemical Engineers: London, 1994; 749−751.

Colonia, E. J.; Raynaud-Lacroze, P. O.; Tavare, N. S. Separation of isomers: Hydrotropy and precipitation. In *Industrial Crystallization '93*; Rojkowski, Z. H., Ed.; Politechnika: Warszawska, Poland, 1993; 3-153-3-159.

Dikshit, R. C.; Chivate, M. R. Separation of ortho and para nitrochlorobenzenes by extractive crystallization. *Chem. Eng. Sci.* **1970**, *25*, 311−317.

Dixit, A. B.; Colonia, E. J.; Tavare, N. S. Separation of *o*- and *p*-chlorobenzoic acids: hydrotropy and precipitation. *J. Cryst. Growth* **1996a**, *166*, 976−980.

Dixit, A. B.; Colonia, E. J.; Tavare, N. S. Selective precipitation of *p*-chlorobenzoic acid. In Proceedings of the 13th Symposium on Industrial Crystallization; Biscans, B., Garside, J., Eds.; PROGEP: Florence, 1996b; 1, 135−140.

Dixit, A. B.; Colonia, E. J.; Tavare, N. S. Use of a hydrotrope in selective precipitation of *o*- or *p*-chlorobenzoic acids. Proceedings of the 1997 IChemE Research Event; Institution of Chemical Engineers: Rugby, 1977 (to be presented).

Geetha, K. K.; Tavare, N. S.; Gaikar, V. G. Separation of *o*- and *p*-chloronitrobenzenes through hydrotropy. *Chem. Eng. Commun.* **1991**, *102*, 211−224.

Hanson, C.; Patel, A. N. Separation of *o*- and *p*-chloronitrobenzene by solvent extraction. *J. Appl. Chem.* **1966**, *16*, 341−344.

Jadhav, V. K.; Dixit, B. A.; Tavare, N. S. Solubilities of *m*- and *p*-aminoacetophenones in hydrotrope solutions. *J. Chem. Eng. Data* **1995**, *40*, 669−673.

Matsuoka, M.; Ohishi, M.; Kasama, S. Purification of *p*-dichlorobenzene and *m*-chloronitrobenzene crystalline particles by sweating. *J. Chem Eng. Jpn.* **1986**, *19*, 181−185.

Raynaud-Lacroze, P. O.; Tavare, N. S. Separation of 2-naphthol: Hydrotropy and Precipitation. *Ind. Eng. Chem. Res.* **1993**, *32*, 685−691.

Tare, J. P.; Chivate, M. R. Separation of close boiling isomers by adductive and extractive crystallization. *AIChE Symp. Ser.* **1976**, *72*, No. 153, 95−99.

Tavare, N. S. *Industrial crystallization process simulation analysis and design*; Plenum: New York, 1995.

Tavare, N. S.; Jadhav, V. K. Solubilities of 6-aminopenicillanic acid and phenoxyacetic acid in hydrotrope solutions. *J. Chem. Eng. Data* **1996**, *41*, 1196−1202.

Received for review December 18, 1996. Accepted February 25, 1997.[⊗]

JE960405S

[⊗] Abstract published in *Advance ACS Abstracts,* April 15, 1997.