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SOLUBILITY & EQUILIBRIUM DATA FOR PHENOL-AQUEOUS
SODIUM SALICYLATE- α -METHYL NAPHTHALENE SYSTEM

PRITHVI RAJ, K. DHANAMJAYA PRASAD, S. MOINUDDIN AHMED,

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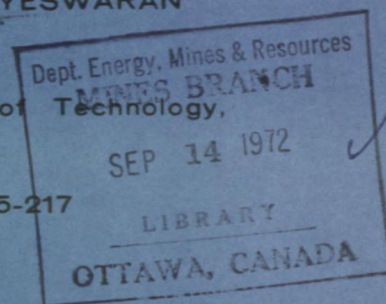
LABORATORY STUDIES ON SOLVENT EXTRACTION OF
LOW-TEMPERATURE TAR OILS BY AQUEOUS SODIUM SALICYLATE

P. RAJ, M. A. KHALEEL AKMAL, Y. V. SUBBA RAO,

S. MOINUDDIN AHMED AND R. VAIDYESWARAN

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Solubility & Equilibrium Data for Phenol-Aqueous Sodium Salicylate- α -Methyl Naphthalene System

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Manuscript received 29 September 1966

Solubility and equilibrium data for phenol-aqueous sodium salicylate (54 per cent)- α -methyl naphthalene system at 30°C. are reported. The system exhibits two types of curves — the ordinary bite type curve and a small convex loop with a solid and liquid phase — typical of systems containing water and an inorganic salt. The tie-line data fit with most of the empirical relationships reported in literature. The plait point for the system is at 36.0 per cent phenol concentration.

THE search for newer solvents for the extraction of phenols from tar oils obtained from low temperature carbonization of coal led to the consideration of aqueous sodium salicylate and sodium benzoate. Earlier experiments with aqueous solutions of sodium salicylate of different concentrations have shown 54 per cent (wt/vol.) sodium salicylate to be ideal for the extraction of tar acids from low temperature tar oils¹. For the successful development of a process based on this solvent, it is necessary to have an exact knowledge of the phase behaviour of the substances involved — neutral oil, phenols and the solvent. However, in view of the complex composition of the first two, a ternary system is selected with typical pure components closely representing those in the tar oils. For this purpose, phenol is considered to be typical of the tar acids and α -methyl naphthalene of the aromatic neutral compounds. The determination of solubility and equilibrium data was, therefore, undertaken to find out the suitability of aqueous sodium salicylate as solvent for the extraction of phenols from tar oils.

Materials and Methods

Chemically pure phenol (BDH) was dehydrated in a STPTC distillation apparatus and the material boiling in the range 178-178.5°C. at 715 mm. Hg was collected (n_D^{41} , 1.5390; purity, 99.9 per cent).

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Aqueous solution of sodium salicylate (B. P., Rhodia, France) of concentration 54 per cent (wt/vol.) (n_D^{30} , 1.4375) was prepared. α -Methyl naphthalene (technical grade, Theodor GmbH, Munchen) was fractionated in a Vigreux column equivalent to three theoretical plates and the portion boiling in the range 241-42°C. at 715 mm. Hg was used (n_D^{30} 1.612; lit., 1.61278).

Solubility data — The solubility data were determined by the turbidity end point method². In the solvent-rich region, mixtures of phenol and sodium salicylate solution were taken in 100 ml. stoppered conical flasks and maintained at 30°C. for 30 min. in a thermostat. α -Methyl naphthalene was added gradually from a burette until permanent turbidity appeared. Knowing the weight of α -methyl naphthalene added, the composition of the ternary mixture at 30°C. was calculated. In the aromatic-rich region sodium salicylate solution was added to mixtures of α -methyl naphthalene and phenol and titrated in a similar manner.

Equilibrium data — The equilibrium data were determined at 30°C. in an apparatus similar to that used by Smith and Bonner³. It consisted of a glass-jacketed cylindrical unit (length 16 cm. and int. diam. 40 mm.) with a stirring arrangement. In the jacket, water was circulated at 30°C. Provision was made for the removal of the sample by means of a stop-cock at the bottom. Mixtures containing known weights of phenol, α -methyl naphthalene and sodium salicylate solution were taken

in the unit and stirred for 1 hr to bring the phases to equilibrium. The mixture was allowed to settle for 30 min. and the two phases were collected separately, weighed and analysed. Phenol was estimated by bromination method⁴ after extraction with ethyl ether and fixing it with 10 per cent sodium hydroxide. Sodium salicylate was estimated by titration with barium hydroxide and water by Dean and Stark method⁵.

Results and Discussion

The data are presented in Tables 1 and 2 and Fig. 1. While plotting the binodal curve, it is assumed that sodium salicylate solution behaves as a single component. This supposition has been proved to be valid by previous workers⁶ with systems where aqueous solutions have been used as solvents. In the present study, it was found by analysis that the composition of the solvent was the same in both the phases in equilibrium.

The solubility data showed two curves—one of the ordinary bite type and the other a closed loop (Fig. 1). In the aromatic-rich phase, when

sodium salicylate solution was added, turbidity resulted, which on further addition of the solvent disappeared. When more of the solvent was added, turbidity reappeared. The points corresponding to the first appearance of turbidity and its disappearance gave rise to a convex loop touching the equilibrium curve at the lower concentrations of phenol and resembling a hysteresis loop. Small quantities of solid got separated on the first addition of sodium salicylate solution, perhaps due to salting out phenomenon. Such phenomena of solid and liquid phase region have been reported with systems containing inorganic salts and water as components in ternary systems⁷. The liquid after removal of solid by filtering was clear and homogeneous. Although island type of curves have been reported in the literature, this cannot be considered to be an island type as it is surrounded by homogeneous phase on all sides⁸.

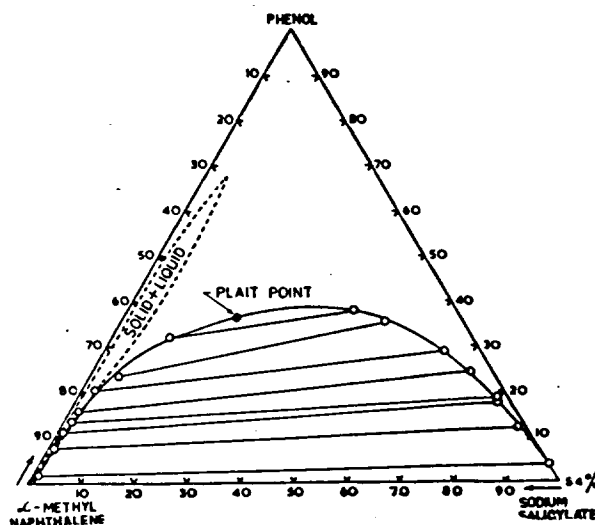


Fig. 1—Solubility and equilibrium diagram for phenol- α -methyl naphthalene-sodium salicylate system at 30°C.

TABLE 1—SOLUBILITY DATA FOR PHENOL- α -METHYL NAPHTHALENE-AQUEOUS SODIUM SALICYLATE (54%) SYSTEM—AQUEOUS PHASE

(Temp., 30°C.; values are in wt %)

Phenol	α -Methyl naphthalene	Sod. salicylate	Phenol	α -Methyl naphthalene	Sod. salicylate
2.50	0.10	97.40	33.00	12.20	54.80
3.70	0.30	96.00	35.50	16.20	48.30
11.50	1.30	87.20	36.12	18.27	45.61
12.80	1.50	85.70	36.45	37.87	25.68
14.95	1.72	83.33	36.60	36.00	27.40
15.70	1.80	82.50	36.67	20.37	42.96
17.69	2.45	79.86	37.10	22.16	40.74
21.05	3.44	75.51	37.50	23.90	38.60
28.41	7.48	64.11	37.80	26.20	36.00

TABLE 2—SOLUBILITY DATA—ORGANIC PHASE

(Temp., 30°C.; values are in wt %)

First turbidity			Clear point			Second turbidity		
Phenol	α -Methyl naphthalene	Sod. salicylate	Phenol	α -Methyl naphthalene	Sod. salicylate	Phenol	α -Methyl naphthalene	Sod. salicylate
6.019	93.65	0.33	—	—	—	6.003	93.42	0.58
10.52	81.02	8.46	—	—	—	10.32	79.50	10.18
19.28	78.86	1.86	—	—	—	18.91	78.34	2.75
23.00	76.30	0.70	22.80	75.00	2.20	22.40	74.50	3.10
23.80	75.90	0.30	23.40	74.50	2.10	23.39	74.45	2.16
24.28	74.90	0.82	23.79	73.10	3.11	23.70	73.00	3.30
24.50	74.78	0.72	23.86	72.85	3.29	23.78	72.57	3.65
24.75	75.10	0.15	24.15	73.25	2.60	24.09	73.00	2.91
28.90	70.10	1.00	28.36	68.74	2.90	27.80	67.36	4.84
30.48	69.40	2.12	29.94	66.10	3.96	28.81	63.60	7.59
31.82	67.50	0.68	30.66	65.10	4.24	29.28	62.10	8.62
35.10	62.40	2.50	34.50	61.30	4.20	31.70	56.30	12.00
43.10	56.00	0.90	41.00	53.30	5.70	34.65	45.15	20.20
48.95	50.41	0.64	48.10	46.00	5.90	—	—	—
53.38	45.62	1.00	52.50	42.50	5.00	—	—	—
68.16	28.77	3.07	—	—	—	—	—	—

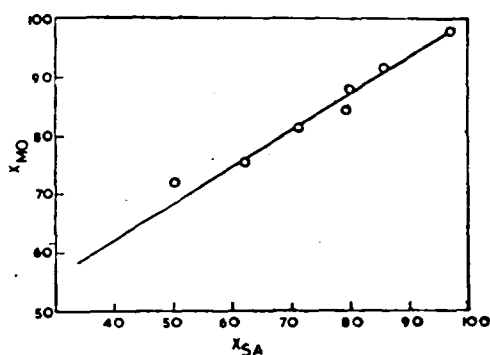


Fig. 2 — Branker-Hunt-Nash correlation of data
 $\left[\frac{x_{SA}}{x_{MO}} = \text{constant} \right]$

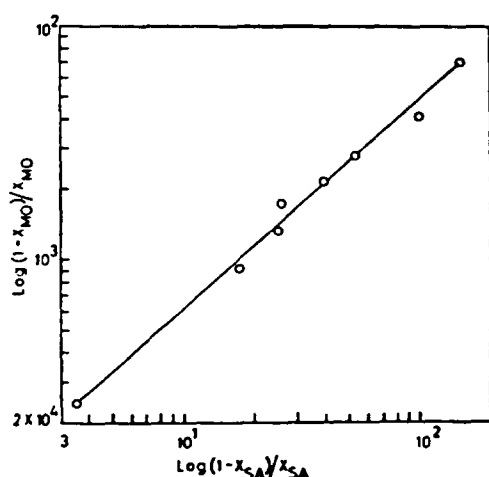


Fig. 3 — Othmer-Tobias correlation of data [Correlation equation, $\log \frac{1-x_{MO}}{x_{MO}} = C_1 \log \frac{1-x_{SA}}{x_{SA}} + C_2$]

The formation of the closed loop could be explained to be due to the phenol picking up the water preferentially, which results in the throwing out of some of the solid sodium salicylate. On further addition of the aqueous solvent, the precipitated solid gets into solution, resulting again into a single phase.

The tie-line data are also shown in Fig. 1. These data were also correlated by the methods of Branker *et al.*⁹, Bachman¹⁰, Othmer and Tobias¹¹, Hand¹² and Dryden¹³. The plots obtained by the first and third correlations are shown in Figs. 2 and 3. These and other plots except that obtained by Dryden's method¹³ were linear, lending further support to the assumption that sodium salicylate solution behaves more as a single component.

The plait point for the system determined by conjugate method of interpolation and extrapolation, Sherwood's method¹⁴ and distribution curve method¹⁵ showed 36.0 per cent phenol concentration in all the cases.

Conclusion

1. The phase boundary diagram for the system phenol-aqueous sodium salicylate (54 per cent wt/vol.)- α -methyl naphthalene exhibits two types

TABLE 3 — EQUILIBRIUM DATA

(Temp., 30°C.; values are in wt %)

Aqueous phase			Organic phase		
Phenol	α -Methyl naphthalene	Sod. salicylate	Phenol	α -Methyl naphthalene	Sod. salicylate
3.353	0.047	96.6	1.936	97.665	0.399
11.70	2.90	85.4	7.999	91.481	0.520
17.55	2.45	80.0	11.35	88.00	0.65
18.23	2.00	79.77	13.50	84.96	1.543
24.00	4.22	71.78	15.81	8.70	1.965
28.46	7.00	64.54	20.21	77.00	2.74
35.03	14.97	50.00	23.76	71.08	5.16
37.21	22.74	40.05	32.41	58.39	9.20

of curves, one the ordinary bite type curve with two liquid phases and the other a small convex loop with a solid and liquid phase, typical of certain systems containing water and inorganic salt.

2. The equilibrium data for the bite type curve show a plait point with 36.0 per cent phenol concentration and the tie-line data fit with most of the empirical relationships.

Acknowledgement

The authors wish to express their thanks to Dr G. S. Sidhu for his keen interest during the course of the present investigation and for his permission to publish this work.

Nomenclature

x_{PA} = wt fraction of phenol in aqueous phase
 x_{PO} = wt fraction of phenol in organic phase
 x_{MA} = wt fraction of α -methyl naphthalene in aqueous phase
 x_{MO} = wt fraction of α -methyl naphthalene in organic phase
 x_{SA} = wt fraction of sodium salicylate in aqueous phase
 C_1, C_2 = constants

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LABORATORY STUDIES ON SOLVENT EXTRACTION OF LOW-TEMPERATURE TAR OILS BY AQUEOUS SODIUM SALICYLATE

By P. RAJ, M. A. KHALEEL AKMAL, Y. V. SUBBA RAO, S. MOINUDDIN AHMED*
and R. VAIDYESWARAN

The use of aqueous saturated sodium salicylate for the extraction of tar acids from a low-temperature tar fraction is discussed. Solubility and equilibrium data for the system: neutral oil-tar acids-aqueous sodium salicylate when plotted on a ternary diagram showed two types of curves: an ordinary bite type and another a loop containing solid and liquid phases. The tie-line data agree with most of the empirical relationships reported in the literature. The plait point for the system had a tar acid concentration of 47%. The results have been compared with the earlier data for the system: phenol- α -methylnaphthalene-aqueous sodium salicylate.

Introduction

Several solvents have been tried for the recovery of tar acids from coal tar oils.^{1,2} The use of aqueous salt solutions of organic acids has attracted attention in recent years. A plant based on sodium phenolate solution is in operation at the industrial complex based on lignite at Neyveli³ for the extraction of phenols from the low-temperature tar fraction boiling up to 220°. Work has been carried out on the use of aqueous salt solutions in this laboratory and the use of aqueous sodium salicylate and sodium benzoate was covered in a patent.⁴ In an earlier communication, the solubility and equilibrium data of α -methylnaphthalene-phenol-aqueous saturated sodium salicylate were reported, the former two being considered typical of the components found in tar oil.⁵ In the present paper similar data for the system: tar acids-neutral oil-aqueous sodium salicylate are presented and the suitability of aqueous sodium salicylate is discussed.

Experimental

Materials

The light tar obtained from low-temperature carbonisation of a meta-lignite coal in the Lurgi-Spuel gas pilot plant in operation at the laboratory was distilled in a pot still and the fraction boiling between 175 and 230° was used for the extraction studies. The tar content of the fraction was 40% and the neutral oil content was 60% including 0.4% of tar bases.

For the solubility and equilibrium studies, the neutral oil and the tar acids (including the bases which were present in negligible amount) were separated by conventional methods from the tar oil fraction by the scheme shown in Fig. 1.

Sodium salicylate (B.P. Rhodia, France) was used for the preparation of 54% (w/v) aqueous sodium salicylate, (270 g of sodium salicylate dissolved in water to make up a solution of 500 ml) (n_D^{20} 1.4375). The same solution was used for solubility, equilibrium and extraction studies. This concentration represented the saturated solution at 30°. Other concentrations were also prepared in a similar manner.

Methods

Extraction

The tar distillate and the aqueous sodium salicylate of appropriate concentration were shaken in a separating

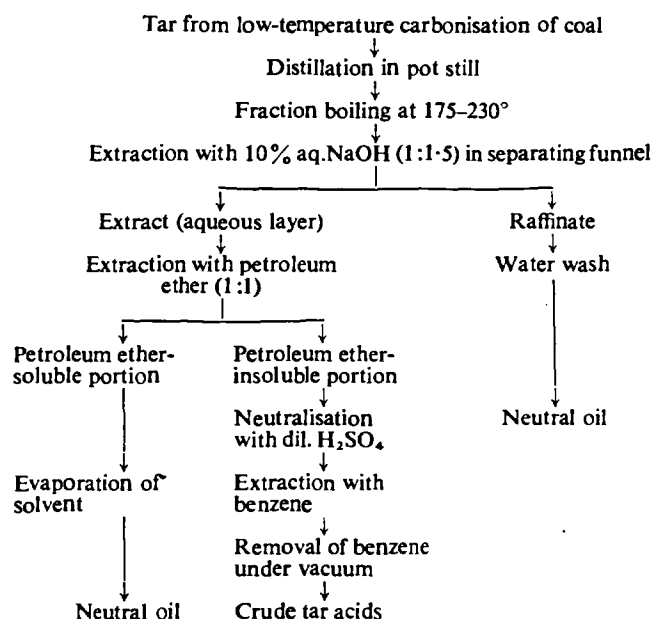


Fig. 1. Scheme for the separation of neutral oil and crude tar acids from the tar oil fraction

funnel at $28 \pm 2^\circ$ for ~ 5-6 min and allowed to settle for 20-25 min before the layers were separated. The extract and the raffinate layers were analysed for crude tar acids and neutral oil content by the following procedures:

Extract—An aliquot of the extract was re-extracted with diethyl ether to remove the acids and the neutral oil. The tar acids were fixed with 10% aqueous sodium hydroxide and then freed by sulphuric acid. The tar acids were extracted with benzene and then freed of solvent by vacuum distillation until of constant weight, finally weighed as dry, crude tar acids. The other portion from ether extraction was washed with water to remove all traces of alkali and dried over sodium sulphate. The solvent was removed and finally weighed for neutral oil content.

Raffinate—A portion of the raffinate was washed with water to remove any dissolved sodium salicylate and the organic layer was extracted with 10% hydroxide to remove

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tar acids which were estimated as described above. The neutral oil content was also determined as above.

Solubility data

The solubility data were determined by the turbidity end-point method.⁶ In the solvent-rich region (aqueous sodium salicylate), mixtures of known quantities of tar acid and solvent were taken in 100 ml stoppered conical flasks and maintained at 30° for 30 min in a thermostat. Neutral oil was added gradually from a burette until permanent turbidity appeared. The composition of the ternary mixture was calculated from the weight of the neutral oil added. In the organic-rich region, solvent was added from the burette to a mixture of known quantities of neutral oil and tar acid and the turbidity was determined in a similar manner.

Equilibrium data

The equilibrium data were determined at 30° in an apparatus similar to that used by Smith & Bonner.⁷ It consisted of a glass-jacketed cylindrical unit (length 16 cm, i.d. 40 mm) with a stirring arrangement. Water at 30° was circulated in the jacket from a thermostat. Provision was made to withdraw the sample by means of a stop-cock at the bottom. Mixtures of known quantities of tar acid, neutral oil and solvent were taken in the unit and stirred for 1 h to bring the phases to equilibrium. The mixture was allowed to settle for 30 min before withdrawing the two phases separately. The separated phases were weighed and analysed for their compositions according to the procedure described above. The sodium salicylate content in the extract was estimated by weighing the solid after evaporation of an aliquot of the extract treated with ether to remove the tar acids and neutral oil. The sodium salicylate content in the raffinate was estimated by titration with barium hydroxide. The water contents in the raffinate and extract were determined by azeotropic distillation benzene by the Dean & Stark method.

The plait point for the system, determined by the conjugate method of interpolation and extrapolation, showed a composition of 47% tar acids, 24% neutral oil and the rest aqueous sodium salicylate.

Results and discussion

Effect of concentration and solvent: feed ratio

In Figs 2 and 3 are shown the results of the effect of concentration of the solvent (sodium salicylate) and the ratio of solvent: tar oil on the percentage recovery of tar acids and their purity. The experiments were carried out in a separating funnel in three stages. The respective quantity of solvent was divided in a ratio of 2:2:1 and fed to each successive stage. As the concentration was increased, the yield of tar acids increased but the purity decreased. Pure (99.5%) tar acids could be obtained by the use of 10% sodium salicylate, but the recovery was only 21%. When a saturated solution of sodium salicylate was used, the recovery was 89% but the tar acids contained 10% of neutral oil. It can be seen from Fig. 3 that a solvent: feed ratio of 5:1 resulted in a satisfactory product. Further increase in the ratio increased the recovery, but affected the purity of the tar acids. The studies on solubility and equilibrium data were carried out with a 54% (w/v) solution using a solvent: feed ratio of 5:1.

Solubility and equilibrium data

Tables I and II show the solubility and equilibrium data

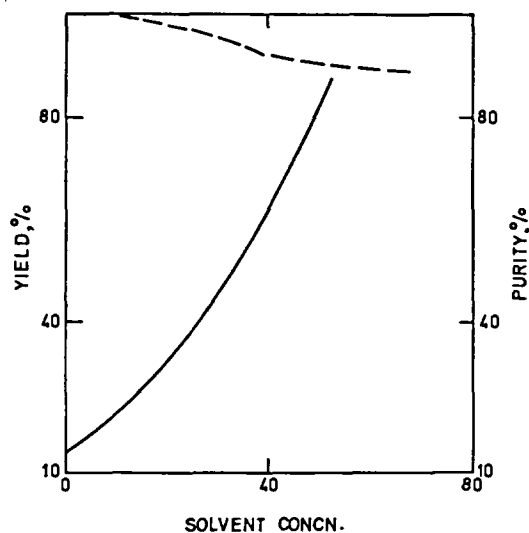


Fig. 2. Effect of solvent concentration on yield and purity for a solvent:feed ratio of 5:1
— yield, - - - - - purity

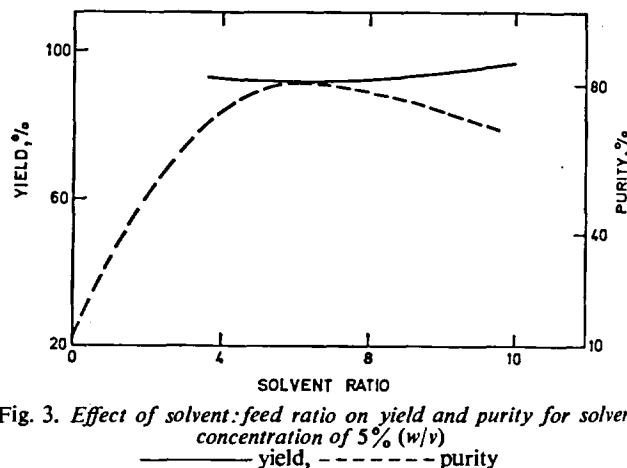


Fig. 3. Effect of solvent: feed ratio on yield and purity for solvent concentration of 5% (w/v)
— yield, - - - - - purity

for the system: tar acids–neutral oil–aqueous saturated sodium salicylate and the data are plotted in a ternary diagram in Fig. 4. When the binodal curve was plotted it was assumed that aqueous sodium salicylate behaves as a single component, the reasons for which have been explained in the previous paper.⁵

The same type of data were obtained as in the case of phenol- α -methylnaphthalene–aqueous sodium salicylate.⁵ An ordinary bite-type curve and a closed loop were observed. The separation of small quantities of solid near the organic-rich phase was noticed and resulted in the first turbidity. The turbidity point normally represents a composition which lies on the boundary of the binodal curve. In the present system, near the solid–liquid loop, a first turbidity point lying on the left-hand portion of the loop was noted. The turbidity disappeared with further addition of the solvent and the mixture corresponded to a point on the right-hand side of the loop. This was denoted as the clear point. The homogeneous mixture became turbid with continuing addition of the solvent, and the point corresponded to a composition on the main binodal curve. This formation of two turbidity points and a clear point was observed with mixtures containing

TABLE I
Solubility data

Organic-rich phase									Solvent-rich phase		
First turbidity			Clear point			Second turbidity					
Crude tar acids	Neutral oil	Aq. sodium salicylate	Crude tar acids	Neutral oil	Aq. sodium salicylate	Crude tar acids	Neutral oil	Aq. sodium salicylate	Crude tar acids	Neutral oil	Aq. sodium salicylate
5.48	94.00	0.52	—	—	—	—	—	—	4.93	94.30	0.77
13.10	84.60	2.30	—	—	—	—	—	—	10.14	89.10	0.76
21.59	77.61	0.81	—	—	—	—	—	—	18.77	79.51	1.72
25.00	73.60	1.41	—	—	—	—	—	—	27.70	68.25	4.05
34.15	63.65	2.20	—	—	—	—	—	—	32.42	62.2	5.38
39.95	57.50	2.55	—	—	—	—	—	—	41.20	45.8	13.0
44.90	52.50	2.60	—	—	—	—	—	—	45.50	36.44	18.06
47.20	50.10	2.70	—	—	—	—	—	—	48.60	26.87	24.53
50.10	46.90	3.00	—	—	—	—	—	—			
53.10	43.30	3.50	—	—	—	—	—	—			
56.90	39.20	3.90	—	—	—	—	—	—			
57.30	39.20	3.50	—	—	—	—	—	—			
61.60	33.40	5.00	51.50	30.63	12.87	49.80	26.78	23.42			
63.61	34.80	1.60	58.50	28.10	13.40	48.25	23.20	28.55			
66.60	27.61	5.79	60.80	25.80	14.12	46.40	19.21	34.30			
71.90	22.54	5.56	64.20	20.59	15.21	41.90	13.35	44.75			
73.10	19.34	7.56	65.60	17.30	17.10	39.80	10.48	49.72			
75.60	16.58	7.82	67.95	14.92	17.13	36.61	8.05	55.34			
78.20	8.6	9.40	73.80	7.73	18.43	—	—	—			

TABLE II
Equilibrium data

Aqueous phase			Organic phase		
Crude tar acids	Neutral oil	Aq. sodium salicylate	Crude tar acids	Neutral oil	Aq. sodium salicylate
8.00	1.10	90.90	22.38	77.25	0.37
11.20	0.80	88.00	33.11	64.78	2.11
14.58	1.50	73.90	36.60	62.00	1.40
17.55	2.01	80.44	40.80	56.50	3.70
27.41	4.50	69.00	47.00	50.03	3.00
30.82	5.50	63.68	51.60	45.00	3.40

Temperature, 30°C; values in wt.-%

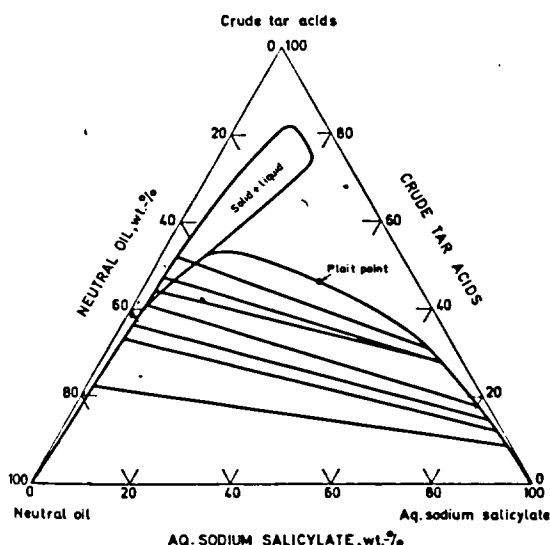


Fig. 4. Solubility and equilibrium diagram for the system: crude tar acids-neutral oil-aqueous sodium salicylate at 30°C

~ 55% and more of tar acids. With mixtures containing 40–55% of tar acids, the clear point coincided with a point on the main binodal curve but the solids found in the loop disappeared and the liquid separated into two layers. This phenomenon can be explained in the same way as before,⁵ i.e., it is due to the tendency of the tar acids to pick up water, a portion of the water from the solvent being taken up by the tar acids, resulting in salting-out of the solid, as the solvent was already a saturated solution. Such phenomena have been reported with systems containing inorganic salts and water as components in ternary systems.⁸ The area of the bite curve as well as the loop was greater in the present case than with pure compounds.

The tie-line data were correlated by the method of Branker *et al.*,⁹ Bachman,¹⁰ Hand¹¹ and Dryden.¹² The plots were linear except for those based on Bachman. The slopes of the tie lines were different from those with pure components. These inclined towards the solvent apex while the tie lines with pure components tended to incline towards the α -methylnaphthalene apex. Thus, for any particular feed with a particular solvent:feed ratio, the purity of the tar acids with respect to the neutral oil content was less in the present

system compared with the system: phenol- α -methylnaphthalene-aqueous sodium salicylate. Further, in the lower region of the acids, the slope of the tie lines is less than in the higher ranges, indicating that more stages are required for the extraction to obtain a pure product.

The tie lines do not interfere in the solid-liquid range and hence there may not be any practical problem. The maximum purity of tar acids obtainable in the extract (92%) can be found by drawing a tangent to the binodal curve from the solvent apex.

The laboratory results were compared with the results obtained by calculation under the same conditions from the phase diagrams of phenol- α -methylnaphthalene-aqueous sodium salicylate and crude tar acids-neutral oil-aqueous sodium salicylate systems. As expected, the laboratory results agreed more with those obtained from the latter diagram. The calculations showed that as the solvent: feed ratio was increased, the yield increased up to a certain ratio, and further increase in the ratio did not improve the yield of tar acids, as can be seen from the lower line in Fig. 3. This is in general agreement with the results shown in the solubility and equilibrium diagram (Fig. 4) from which it can be seen that any increase in solvent: feed ratio above 5:1 has no marked effect on the purity of the extracted tar acids, although laboratory extraction studies indicated that the purity of tar acids slightly decreased with increased solvent: feed ratio (Fig. 3).

Countercurrent extraction and recovery of tar acids

The number of extraction stages required for the countercurrent operation of the system tar acids-neutral oil-54% (w/v) aqueous sodium salicylate was calculated by the method of Branker *et al.*⁹

Using a feed of tar oil containing 40% tar acids, for a tar acids recovery of 92% and a maximum purity of 92%, the solvent: feed ratio was found to be 2.45:1 and the number of stages was eight. In the pheno-raffin process³ using aqueous sodium phenolate as solvent, it is reported that a solvent: feed ratio of 6-7:1 with a 10-stage countercurrent extraction is necessary for a 95% recovery of tar acids from a low-temperature lignite tar (Neyveli Lignite Corporation, private communication). The purity of the tar acids is reported to be low and the extract is further washed with hexane to remove neutral hydrocarbons and bases and then steam-stripped. In an earlier patent,⁴ it was shown that the recovery and purification of the tar acids could be obtained from the aqueous sodium salicylate by steam stripping alone, and it thus appears that sodium salicylate is a better solvent than the aqueous phenolate currently used in India.

Selectivity of the solvent

The efficiency of extraction of tar acids using different feed compositions was also studied. Tar oils with different tar acid compositions were treated with 30% (w/v) aqueous sodium salicylate using a solvent:oil ratio of 5:1. With tar oils boiling between 170° and 200° which contain mostly phenol and cresols, the recovery of tar acids was 62-63%. With a feed containing 80% xlenols and 20% cresols (boiling range 200-230°), the recovery fell to 28%, and with tar oil of boiling range 230-290°, the recovery was only 5%. With the light tar produced in the Lurgi-Spuel gas plant (boiling range 170-350°), the recovery was 22.8%.

Recovery of tar acids from extract

Experiments were also carried out on the recovery of tar acids from the extract and are covered in the patent.⁴ One method used was steam distillation from which it was found that the first 30% (v/v) of this steam distillate contained the major portion of the neutral hydrocarbons. After removal of the hydrocarbons, the rest of the steam distillate up to 200% (v/v) contained pure tar acids.

Conclusions

The optimum concentration of aqueous sodium salicylate as a solvent is 54% (w/v) at 30°. The nature of the phenols in the tar distillate has an effect on the efficiency and purity of recovered tar acids.

Results from phase equilibria described in this communication are more closely in agreement with experimental results than those derived from the study of the phenol- α -methylnaphthalene-aqueous sodium salicylate systems, and hence these results should be used for plant design purposes.

Increase of solvent feed ratio reduces the number of stages required and increased the yield, but the purity remains almost constant.

Fractional recovery of tar acids from extract is possible by steam distillation. Maximum purity possible is 92% and the plait point showed a composition of 47% tar acids, 24% neutral oil and the rest 54% (w/v) aqueous sodium salicylate.

The solvent shows considerable promise for the extraction of tar acids from tar oils.

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