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Ternary Phase Equilibrium Data for Acetic Acid-Water-Solvent Systems and Separation of Acetic Acid from Aqueous Solution

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Abstract: Ternary phase equilibrium data for acetic acid with water and solvent (n-butyl alcohol, isobutyl alcohol and amyl alcohol) are presented and used to evaluate the possibility of employing distribution of acetic acid between water and these alcohols as a means of separation of acetic acid from its aqueous solution. Mutual solubility curves, tie-line data, distribution coefficient, selectivity diagrams and separation factor data were determined for these systems. From these data it has been concluded that of these solvents amyl alcohol offers the best hope of achieving separation of acetic acid by distribution between amyl alcohol and water as it has the highest separation factor value than those of other two alcohols.

Key words: Ternary phase, equilibrium data, aqueous solution, separation factor, distribution coefficient

INTRODUCTION

Extraction of constituents by solvent is a very common practice in chemical processing where a mixture containing separable components is treated with a solvent in which one or more of the desired components are preferentially soluble. This type of extraction is called liquid-liquid extraction in which a solution is brought into contact with a second liquid (solvent) essentially immiscible with the first one in order to bring about transfer of one or more components from the solution into the second solvent. It is an effective alternative process to distillation process. Liquid-liquid extraction process is the most useful method for purification, enriching and separation of components of very close volatility and of heat sensitive.

In many cases, acetic acid is found in its aqueous form. Acetic acid is an important laboratory reagent as well as solvent for carrying reactions since it remains unchanged by oxidizing and reducing agents. It is used for coagulation rubber latex, for curing fish and meat, for the manufacture of vinyl acetate, polyvinyl acetate and other laboratory reagents. Thus, the study of acetic acid extraction from its aqueous solution is fascinating all the time. In this study, in order to find out the proper solvent for the separation of acetic acid from aqueous solution, phase equilibrium data and mutual solubility data were therefore obtained experimentally by using solvents such as n-butyl alcohol, isobutyl alcohol and amyl alcohol.

Separation factor which is a quantitative index of effectiveness of separation has been computed from these data.

MATERIALS AND METHODS

All the materials used were of laboratory reagent grade, are shown in Table 1. The method that was used for the determination of mutual solubility and tie-line data of the present work is the "titration method of Othmer *et al.*^[1]. All the measurements were carried out at 29-30°C. The mutual solubility curves for each system were obtained by titrating known mixture of two components with the third one to the point of first appearance of permanent turbidity and the refractive index of the mixture at this point is measured by Atago Precision Abbe Refractometer Type-3. To add tie-line data, various mixtures of known compositions within the heterogeneous region were prepared, brought to equilibrium and the compositions of the conjugate phases were determined by measurement of refractive index, in conjunction with the calibration curves prepared with the

Table 1: Densities (ρ) and refractive indices (n_D) of the used materials at 20°C

Materials	Suppliers	Purity (%)	ρ (g mL ⁻¹)	n_D
Acetic acid	E. Merck, Germany	99.5	1.048	1.371
n-butyl alcohol	E. Merck (India) Ltd.	99.0	0.808	1.398
Isobutyl alcohol	BDH, England	98.0	0.806	1.396
n-amylalcohol	BDH, England	99.0	0.810	1.400

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systems of known compositions on the solubility curves^[2].

RESULTS AND DISCUSSION

Determination of mutual solubility curves: Twenty milliliter of water (diluent) was measured into a 150 mL Erlenmeyer flask and n-butyl alcohol (solvent) was added from a burette and the content of the flask was constantly agitated until the solution became just turbid. The amounts added were recorded as the maximum solubility of solvent in the diluent, since the appearance of the turbidity indicated the formation of second phase, the solvent layer. The compositions of the mixture at the maximum solubility were expressed in weight percent. Now 2 drops of water was added to this mixture and because of its consolute effect, a clear solution resulted. Then an arbitrarily chosen amount of acetic acid (solute) was added so that the compositions of the resulting mixture moved out side the solubility curve and the solvent was again added until turbidity resulted, a second point on the mutual solubility curve was obtained. Two drops of diluent was again added and the above operation of successive addition of solute and solvent were repeated, gave the diluent branch of the mutual solubility curve. To obtain the solvent branch of the mutual solubility curve, 20 mL of solvent was taken into a flask and the diluent was added from the burette until turbidity was obtained. Successive addition of solute and diluent in a manner similar to that for diluent branch gave solvent branch of the mutual solubility curve. Diluent branch and solvent branch of the mutual solubility curve together appeared as a hump and described the complete mutual solubility curve for the system acetic acid-water-n-butyl alcohol.

Similarly, mutual solubility curves for acetic acid-water-isobutyl alcohol and acetic acid-water-amyl

alcohol systems were also obtained. The mutual solubility curves for each of these systems are shown in the Fig. 1.

Ternary phase equilibrium data for acetic acid-water-solvent (n-butyl alcohol, isobutyl alcohol and amyl alcohol) systems at 25-26°C are listed in Table 2.

Construction of calibration curves: Each of the points on the mutual solubility curves in Fig.1 for the systems acetic acid-water-n-butyl alcohol, acetic acid-water-isobutyl alcohol and acetic acid-water-amyl alcohol, represent a mixture whose compositions in weight percent is known. The refractive indices of the mixtures corresponding to these points were determined by Atago Precision Abbe Refractometer Type-3. These refractive indices were plotted against the respective weight percent of solvent and diluent. These plots serve as the calibration curves shown in Fig. 2, for determining the unknown

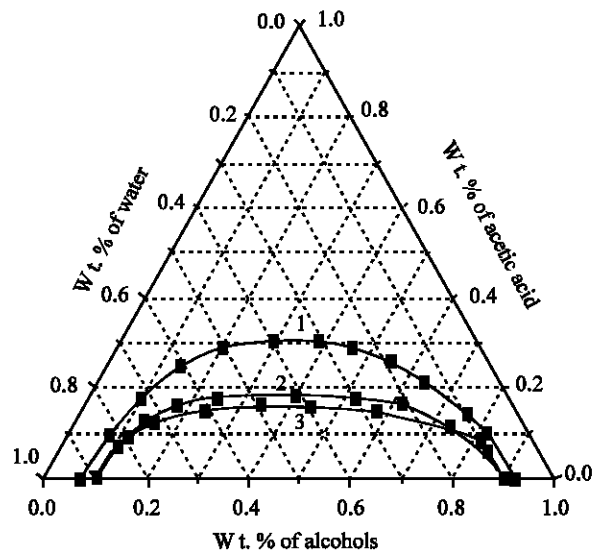


Fig. 1: Mutual solubility curves for acetic acid-water-alcohol systems, n-amyl alcohol (1), isobutyl alcohol (2) and n-butyl alcohol (3)

Table 2: Mutual solubility data and their corresponding refractive indices at 25-26°C (wt.%)

AA-W-n-butyl system				AA-W-isobutyl alcohol system				AA-W-amyl alcohol system			
AA	W	BTA	Refractive index (n_D)	AA	W	IBTA	Refractive index (n_D)	AA	W	AMA	Refractive index (n_D)
90	10.00	00.00	1.392 0	90.50	9.50	00.00	1.3890	93.28	7.720	00.00	1.3964
81.6	10.40	08.00	1.3884	83.61	10.56	5.83	1.3852	81.37	8.180	10.45	1.3940
73.24	15.45	11.36	1.3841	74.20	14.45	11.35	1.3822	75.64	10.15	14.21	1.3914
57.53	27.54	14.93	1.3773	61.50	21.54	16.96	1.3784	64.46	14.94	21.60	1.3872
44.59	40.42	15.84	1.3693	51.64	30.42	17.94	1.3741	55.13	19.07	25.80	1.3825
34.25	49.55	16.20	1.3653	40.25	42.05	18.70	1.3681	46.08	24.92	29.00	1.3790
23.50	61.05	14.94	1.3590	25.05	57.05	17.90	1.3601	38.21	30.79	31.00	1.3754
14.95	72.36	12.69	1.3532	18.15	66.16	15.69	1.3554	29.83	39.70	30.47	1.3698
11.41	79.16	09.43	1.3486	13.12	74.53	12.35	1.3501	20.56	50.86	29.58	1.3642
9.80	90.20	00.00	1.3396	11.23	82.56	6.71	1.3452	13.98	60.77	25.25	1.3594
				10.00	90.00	00.00	1.3400	10.12	71.88	18.00	1.3550
								8.180	83.13	8.690	1.3502
								6.790	93.21	00.00	1.3400

AA: Acetic Acid, W: Water

Table 3-5: Tie-line data (wt.%) at 25-26°C
3: Acetic acid (AA)-water(W)-n-butyl alcohol (BTA) system

Alcohol phase				Water phase			
AA	W	BTA	n_D	AA	W	BTA	n_D
4.5	10.0	85.5	1.389	3.5	86.0	10.5	1.343
10	12.5	77.5	1.386	7.5	81.0	11.5	1.347
14.5	17.0	68.5	1.383	10.5	77.5	12.0	1.350
15.5	24.0	60.5	1.379	13.5	69.0	17.5	1.355

4: Acetic acid (AA)-water (W)-isobutyl alcohol (IBTA) system

Alcohol phase				Water phase			
AA	W	IBTA	n_D	AA	W	IBTA	n_D
3	10.0	87.0	1.387	2.5	87.5	10.0	1.342
9.5	12.5	78.0	1.383	7.0	81.5	11.5	1.346
15	18.0	67.0	1.380	12.0	73.0	15.0	1.351
18	24.5	57.5	1.377	15.5	64.0	20.5	1.357

5: Acetic acid (AA)-water (W)-amyl alcohol (AMA)

Alcohol phase				Water phase			
AA	W	AMA	n_D	AA	W	AMA	n_D
5	8.0	87.0	1.395	2.0	90.5	7.5	1.343
14	9.5	76.5	1.392	6.0	86.0	8.0	1.348
18	12.0	70.0	1.389	16.0	74.5	9.5	1.354
25	15.0	60.0	1.385	23.5	64.0	12.5	1.358

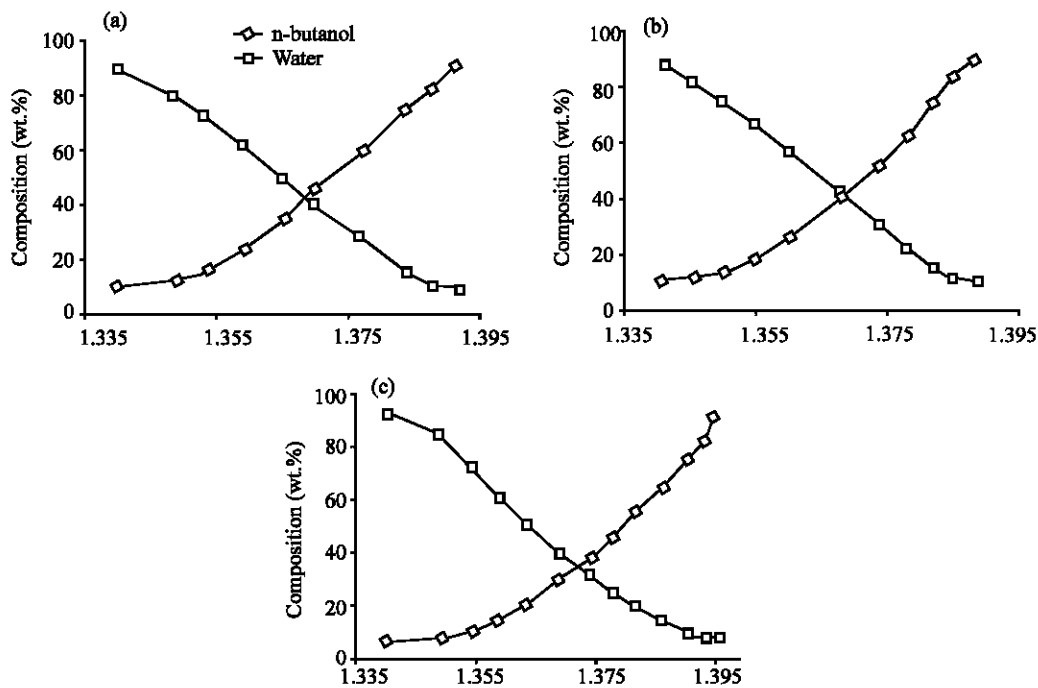


Fig.2: Correlation between refractive index and composition of equilibrium mixtures, acetic acid-water-n-butanol (a), acetic acid-water-isobutanol (b) and acetic acid-water-n-amyl alcohol (c)

compositions of coexisting phases needed for constructing tie-lines data and distribution data.

Determination of tie-line data: Tie-line data of each of these systems were obtained by taking about 20 mL of solvent and 20 mL of diluent in a flask and 2 to 5 mL of solute was added to give a ternary mixture of known

compositions within the heterogeneous region of the mutual solubility curve in Fig. 1, brought to equilibrium and the refractive indices of these two coexisting phases were measured. Comparing with these refractive indices, the compositions of the two coexisting phases in the ternary mixture were read off from the previously constructed calibration curves. Successive addition of

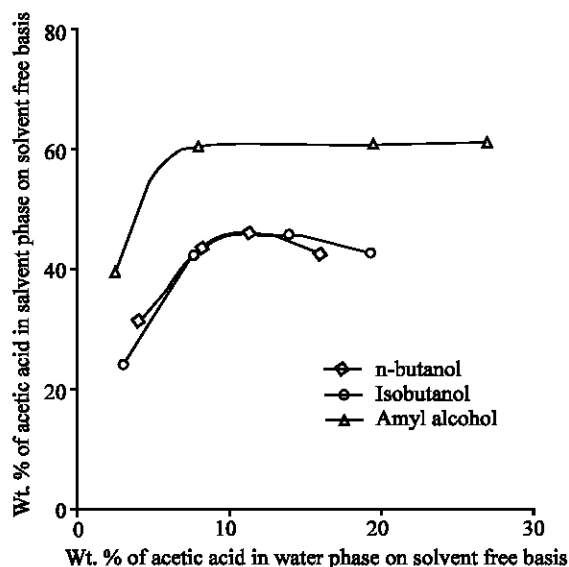


Fig. 3: Selectivity diagram for acetic acid-water-alcohols systems

solute in this ternary mixture gave the other tie-line data for these systems and continued until a single phase existed. Tie-line data for these systems are shown in the Table 3-5.

Determination of separation factor and selectivity diagram: The separation factor and the distribution coefficient of acetic acid between water and alcohols were determined numerically^[3] from the tie-line data, shown in the Table 6-8. The ratio of weight fraction of acetic acid in solvent phase to the weight fraction of acetic acid in diluent phase for each tie-line gives the distribution coefficient of acetic acid (D_s), which is greater than 1 for all the systems but acetic acid-water-amyl alcohol system shows the highest value (2.5). This indicates that acetic acid has preferential solubility between water and alcohol which is the key factor in the extraction process.

Separation factor is a ratio of distribution coefficient of solute (acetic acid) to the distribution coefficient of diluent (water). Hence the separation factor, $S = D_s/D_d$. The distribution coefficient of diluent, D_d is the ratio of the weight fraction of diluent in solvent phase to that in diluent phase. Selectivity diagrams were constructed by plotting weight percent of acetic acid in the solvent phase against that in the diluent phase both on solvent free basis. Selectivity diagrams of all the systems are shown in Fig. 3, which reveals that amyl alcohol shows the highest selectivity and n-butyl alcohol and isobutyl alcohol give almost the same selectivity but less than that of amyl alcohol.

Separation factor is a measure of the solubility of alcohols to separate acetic acid from aqueous solution. It

Table 6-8: Separation factor data for acetic acid-water-alcohols systems
6:Acetic acid-water- n-butyl alcohol system

Distribution coefficient of acetic acid (D_s)	Distribution coefficient of diluent (D_d)	Separation factor ($S_f = D_s/D_d$)
1.290	0.116	11.05
1.333	0.154	8.64
1.381	0.293	6.29
1.148	0.348	3.30

7: Acetic acid-water-isobutyl alcohol system

Distribution coefficient of acetic acid (D_s)	Distribution coefficient of diluent (D_d)	Separation factor ($S_f = D_s/D_d$)
1.20	0.114	10.50
1.357	0.123	11.00
1.25	0.246	5.07
1.161	0.383	3.03

8: Acetic acid-water-amyl alcohol system

Distribution coefficient of acetic acid (D_s)	Distribution coefficient of diluent (D_d)	Separation factor ($S_f = D_s/D_d$)
2.5	0.088	28.28
2.333	0.110	21.12
1.125	0.161	6.94
1.063	0.234	4.54

is also seen from the Table 6-8 that the separation factor for the system acetic acid-water-amyl alcohol is the highest, 28 that is about 2.5 times higher than that of other two systems.

CONCLUSIONS

For the separation of acetic acid from its aqueous solution by using solvents (n-butyl alcohol, isobutyl alcohol and amyl alcohol), the mutual solubility data, distribution coefficient, separation factor data and selectivity diagram of these systems were performed and thus it is to be said after considering all the findings, among these three solvents amyl alcohol is the most selective for the separation of acetic acid from its aqueous solution.

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