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LIQUID-LIQUID EQUILIBRIA OF THE WATER-ACETIC ACID-ETHYL ACETATE SYSTE

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Abstract— **Liquid-Liquid equilibrium composition data for the systems (Water-Acetic acid- ethyl acetate) were measured at 298K and atmospheric pressure and used to evaluate the possibility of employing distribution of acetic acid solute between the organic phase and aqueous phase was investigated. The reliability of the experimental tie-line data was ascertained by using: Othmer–Tobias, Ishida and Hand correlations, which was found to fit the data very well, producing high value of coefficient (R2). (0.995, 0.9923, 0.9994) respectively, Distribution coefficients and separation factors were evaluated for the immiscibility region.**

Keywords— **Liquid-Liquid equilibrium, Tie- line Correlation, tie-line data prediction.**

I. INTRODUCTION

In this study acetic acid was chosen as the solute in the system as it one of the most widely used carboxylic acids in industry, it is used in many reactions, for example, the synthesis of acetic esters, or it can be used as a solvent, for example, in the manufacture of cellulose acetate or pharmaceutical products. In these processes, aqueous solutions of acetic acid are produced which can be economically recovered. The separation of acetic acid and water by simple rectification very difficult, requiring a column with many stages and a high reflux ratio[1], thus incurring high running costs.

In practice other processes are used depending on the concentration of acetic acid present in the feed. Between 50 and 70% w/w acetic acid, extractive distillation is used [2]. By adding a third component, the volatility of water is increased and the separation can be achieved with less energy. Below 40% acetic acid, liquid-liquid extraction is most appropriate [2]. Acetic acid is extracted from water by a suitable solvent in order to obtain substantially pure acetic acid. In order to minimize energy cost in the solvent recovery stage, lower boiling point solvent is usually chosen [3]. In practice, ethyl acetate is usually preferred

Recovery of organic acids from dilute solutions resulting from fermentation processes is important, and many solvents have been tried in attempts to improve recovery[7] (Upchurch and Van Winkle, 1952;Correa et al., 1989; Sayar et al., 1991; Kirk and Othmer, 1992; Dramur and Tatlı, 1993; Briones etal., 1994; Arce et al., 1995; Fahim et al., 1996;Fahim et al., 1997; SÛlimo et al., 1997; Colombo etal., 1999; Aljimaz et al., 2000; Taghikhani et al.2001).

A lot of solvents have been examined for extraction of acetic acid. Selected distribution ratios (D) and selectivities (S) taken from several papers are shown in Table 1 below [9].

Table 1: Distribution ratios (D) and separation factors (S) for extraction of acetic acid from dilute solution in water with several different solvents at 298K.

Ternary system	D	S	Literature
Water + Acetic Acid + Ethyl acetate	0.94	13.64	This study
Water + Acetic Acid + Cyclohexyl Acetate	0.318	18.71	Sayar et al., 1991
Water + Acetic Acid + Dimethyl Phthalate	0.138	6.27	Dramur and Tatlı , 1993
Water + Acetic Acid + Diethyl Phthalate	0.117	10.64	Dramur and Tatlı , 1993
Water + Acetic Acid 1-Pentanol	4.033	9.86	Fahim et al., 1996

Water + Acetic Acid +Ethylcyhlohexane	0.036	11.25	Higashiuchi et al., 1993
Water + Acetic Acid+ + Isophorone	3.164	9.15	Colombo et al., 1999
Water + Acetic Acid + Methyl Isopropyl Ketone	3.444	18.03	Correa et al., 1989

II. MATERIAL AND METHODS

A. **Materials:–**

Water, acetic acid, ethyl acetate and sodium hydroxide (0.1N, 0.5N) were obtained from Red Sea University (chemical engineering lab)

Water: the distilled water was used with 0.997g/cm3 at room temperature Boiling Point 100°C

Acetic acid: was 99.8% purity, density 1.05 g/cm3, Boiling Point 118.5°C

Ethyl acetate: was 95% purity, density 0.902 g/cm3, Boiling Point 77.1°C

B. Procedure

The binodal curves (mutual solubility curves) for acetic acid distributed between water and an organic solvent (ethyl acetate) were obtained by mixing known volumes of two components (water and ethyl acetate) with the third component (acetic acid) and shacked after each addition of drop wise of acetic acid to the point of first appearance of permanent turbidity(observed by the cloud point) and finds the volume of the acetic acid [5]. Ten to twenty milliliter of water (diluent) was measured into a 125 mL Erlenmeyer flask and solvent ethyl acetate was added from a burette and agitated until the solution became just turbid. The amounts are recorded as the maximum solubility of solvent in diluents, since 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. Two drops of the water was added to this mixture and because of its consulate effect, a clear solution was resulted. Then an arbitrarily chosen amount of solute acetic acid was added so that the composition of resulting mixture moved outside the solubility curve and the solvent was again added until turbidity results and second point on the binodal curve was obtained. Two drops of the water was again added and the above operation of successive addition of solute and solvent were repeated. Finally, turbidity was no longer result from the addition of solvent and only a single phase exists and thus obtained the diluents branch of the mutual solubility curve. For the determination of points on the solvent side, say to complete the binodal curve, the same procedure was applied, starting with an initial measured quantity of solvent in the flask, Diluents and solute were then successively added to obtain the points representing all possible compositions of the solvent phase. Thus the mutual solubility curve for acetic acid-water-ethyl acetate system was obtained as shown in table 2 and fig 1.Tie line data of each of these systems were obtained by taking about 20 milliliter of solvent and an equal amount of diluents (water) in a flask and 2 to 5 cubic milliliter of acetic acid (solute) was added to give ternary mixture of known composition within the heterogeneous region of the mutual solubility curve, brought to equilibrium and separated by separation funnel, the absorbance of these two co-existing phases were measured. Comparing with this absorbance, the composition of the two co-existing phases in the ternary mixtures was read off from the previously determined calibration curve. By the successive addition of solute in this ternary mixture, the other tie line data for these systems are obtained until a single phase existed the results are shown in table 3 and Figure 1

III. EXPERIMENT AND RESULT

The experimental binodal curves and tie-line data on water-acetic acid- ethyl acetate at 298.15±0.20K, is given in Tables 2 and 3, respectively. The binodal curve and tie lines were plotted and shown in Figure1

Table 2: Solubility data for the system Water (A) - Acetic acid (C) – ethyl acetate (S) at 298K and 1 atm (weight fraction)

Water rich phase (aqueous phase)			Solvent rich phase (organic phase)		
X_{AA}	X_{CA}	X_{SA}	X_{AS}	X_{CS}	X_{SS}
0.311	0.217	0.472	0.035	0.000	0.965
0.344	0.220	0.436	0.064	0.049	0.887
0.489	0.218	0.293	0.061	0.049	0.890

0.496	0.218	0.286	0.140	0.124	0.736
0.803	0.093	0.104	0.155	0.148	0.697
0.859	0.058	0.083	0.224	0.193	0.583
0.929	0.000	0.071	0.262	0.208	0.530

Table 3: Tie line data for the system (water-acetic acid-ethyl acetate) in weight fraction at at 298K and 1 atm

Water rich phase (aqueous phase)			Solvent rich phase (organic phase)		
X_{AA}	X_{CA}	X_{SA}	X_{AS}	X_{CS}	X_{SS}
0.877	0.043	0.08	0.06	0.039	0.901
0.572	0.198	0.23	0.25	0.2	0.55
0.804	0.096	0.10	0.10	0.096	0.804
0.868	0.052	0.08	0.06	0.049	0.891
0.710	0.150	0.14	0.15	0.15	0.700

Where:
A: water C: acetic acid S: ethyl acetate
XAA: weight fraction of water in aqueous phase
XCA: weight fraction of acetic acid in aqueous phase
XSA: weight fraction of ethyl acetate in aqueous phase
XAS: weight fraction of water in organic phase
XCS: weight fraction of acetic acid in organic phase
XSS: weight fraction of ethyl acetate in organic phase

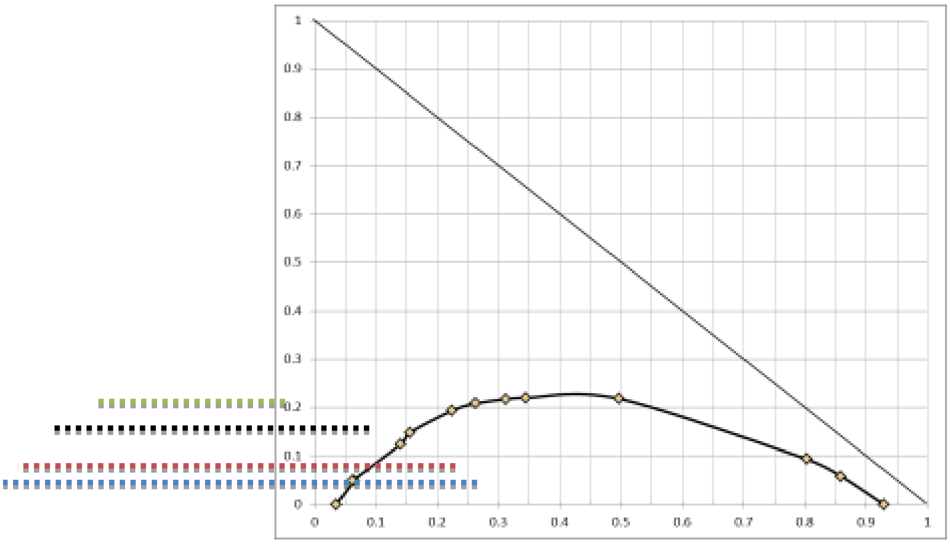

FIG 1 : BIONDAL CURVE AND TIE LINE PLOT

The distribution coefficients and separation factor:

To show the selectivity and strength of the solvent in extracting the acid, distribution coefficients (Di) for the acetic acid (i=2) and water (i=1) and the separation factor (S) is determined as follows:

$$D_i = \frac{\text{Weight fraction in aqueous phase } (X_{iA})}{\text{Weight fraction in solvent phase } (X_{iS})}$$

$S = D2/D1$

The distribution coefficients and separation factor were given in Table 4

Table 4: Distribution coefficients (Di) of water (1) and acetic acid (2) and separation factors S

X_{CS}	X_{CA}	$D_2=X_{CS}/X_{CA}$	X_{AS}	X_{AA}	$D_1=X_{AS}/X_{AA}$	$S=D_2/D_1$
0.039	0.043	0.907	0.060	0.877	0.068	13.257
0.150	0.150	1.000	0.150	0.710	0.211	4.733
0.200	0.198	1.010	0.250	0.572	0.437	2.311
0.096	0.096	1.000	0.100	0.804	0.124	8.040
0.049	0.052	0.942	0.060	0.868	0.069	13.632

THE EXTRACTION POWER OF THE SOLVENT, PLOTS OF D_2 VS. X_{CA} AND S VS. X_{CA}, ARE GIVEN IN FIGURE 2 AND 3, RESPECTIVELY

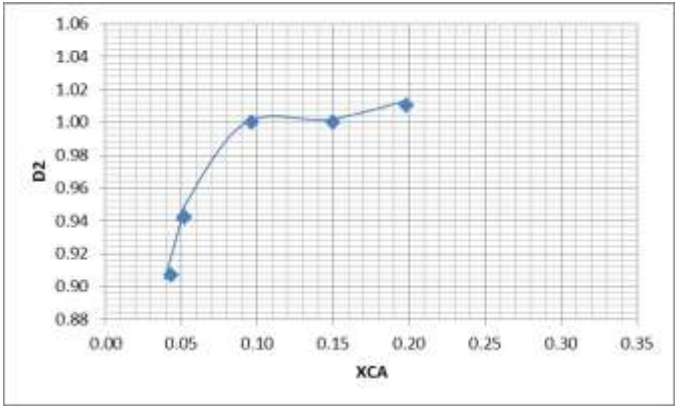

Fig 3: distribution coefficients of acetic acid D2 as a function of the mass fraction of acetic acid in the aqueous phase, X_{CA} for the systems water, acetic acid and ethyl acetate.

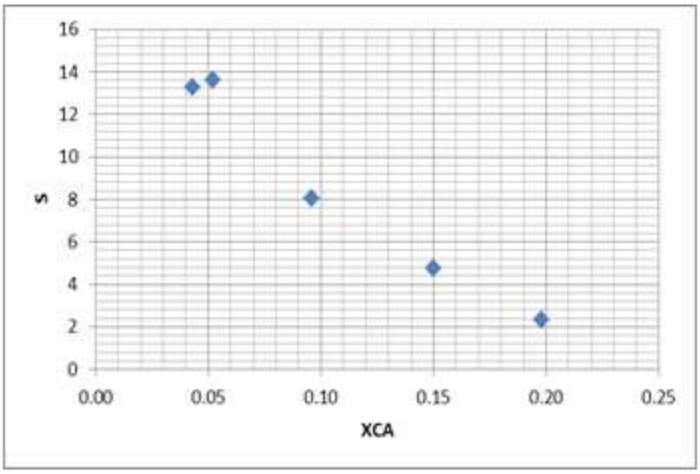

Fig 4: Separation factor, S, as a function of the mass fraction of acetic acid in the aqueous phase, X_{CA} for the systems of water + acetic acid + ethyl acetate

The effectiveness of extraction of acetic acid by ethyl acetate is given by its separation factor (S), which is an indication of the ability of ethyl acetate to separate acetic acid from water. This quantity is greater than one (separation factors varying between 2.3 and13.6) for the system reported here, which means that extraction of acetic acid by ethyl acetate is possible. It is, however, not constant over the whole two-phase region.

Correlation methods[4,8]

1.Hand:

$$\frac{x_{CS}}{x_{SS}} = k\left[\frac{x_{CA}}{x_{AA}}\right]^n$$

The results and plot are in table 5 and fig 5

Table 5: experimental data of water+ acetic acid+ ethyl acetate for hand correlation method

X_{CS}	X_{SS}	lnXcs/Xss	X_{CA}	X_{AA}	$\ln X_{CA}/X_{AA}$
0.039	0.901	-3.13994	0.043	0.877	-3.01531
0.15	0.55	-1.29928	0.15	0.572	-1.3385
0.2	0.804	-1.39128	0.198	0.804	-1.40133
0.096	0.891	-2.228	0.096	0.868	-2.20184
0.049	0.7	-2.65926	0.052	0.71	-2.61402

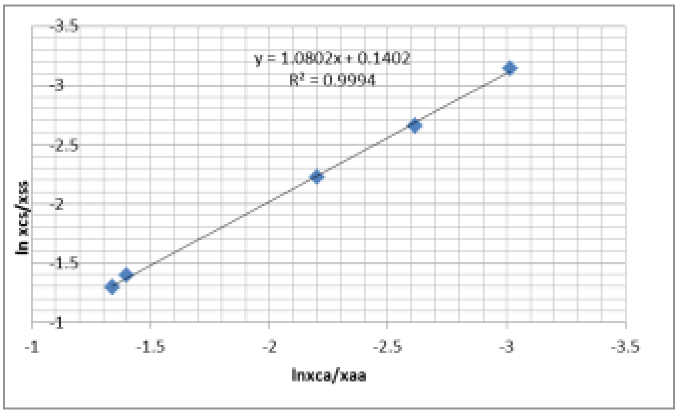

Fig 5: experimental data plot of water+ acetic acid+ ethyl acetate system for hand correlation method

2.Othmer-Tobias:

$$\frac{1 - x_{SS}}{x_{SS}} = k\left[\frac{1 - x_{AA}}{x_{AA}}\right]^n$$

The results and plot are in table 6and fig 6

Table 6: experimental data of water+ acetic acid+ ethyl acetate for Othmer-Tobias correlation method

X_{SS}	$1-X_{SS}$	$\ln(1-X_{SS})/Xss$	X_{AA}	$1-X_{AA}$	$Ln(1-X_{AA})/X_{AA}$
0.901	0.099	-2.208385	0.877	0.123	-1.96432
0.55	0.45	-0.200671	0.572	0.428	-0.29002
0.804	0.196	-1.411485	0.804	0.196	-1.41148
0.891	0.109	-2.100997	0.868	0.132	-1.88339
0.7	0.3	-0.847298	0.71	0.29	-0.89538

3.Ishida:

$$\frac{x_{SA}.x_{CS}}{x_{CA}.x_{SS}} = k\left[\frac{x_{AS}.x_{SA}}{x_{AA}.x_{SS}}\right]^n$$

The results and plot are in table 7 and fig 7

Table 7: experimental data of water+ acetic acid+ ethyl acetate for Ishida correlation method

X_{SA}	X_{CS}	X_{CA}	X_{SS}	$X_{SA}*X_{CS}/X_{CA}*X_{SS}$ ln		X_{AS}	X_{SA}	X_{AA}	X_{SS}	$X_{AS}*X_{SA}/X_{AA}*X_{SS}$ ln	
0.08	0.04	0.04	0.90	0.08	-2.52	0.06	0.08	0.88	0.90	0.01	-5.10
0.23	0.15	0.15	0.55	0.42	-0.87	0.25	0.23	0.57	0.55	0.18	-1.70
0.10	0.20	0.20	0.80	0.13	-2.07	0.10	0.10	0.80	0.80	0.02	-4.17
0.08	0.10	0.10	0.89	0.09	-2.41	0.06	0.08	0.87	0.89	0.01	-5.08
0.14	0.05	0.05	0.70	0.19	-1.67	0.15	0.14	0.71	0.70	0.04	-3.16

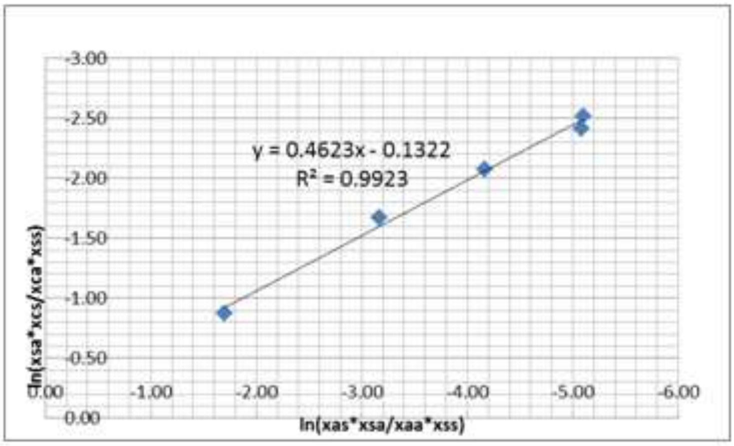

Fig 7: experimental data plot of water+ acetic acid+ ethyl acetate system for Ishida correlation method

IV. CONCLUSION

An intensive literature survey on the models and the models predicting this equilibrium data were cited on many different systems. Experimental studies concerning the separation of acetic acid from its aqueous solution using ethyl acetate were performed the results illustrate the possibility to use ethyl acetate as specific solvents for this separation technique .Mutual solubility data, distribution coefficient, separation factor data and selectivity diagrams have been studied and correlated via several correlations methods. Hand’s Correlation proved to be the most appropriate with the highest value of regression coefficient R2 Hand's Correlations for ternary system need to be extended for multi component systems.

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