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Effect of Inorganic Salts on Ternary Equilibrium Data of
Propionic Acid-Water-Solvents Systems

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Abstract: Liquid-Liquid Equilibrium (LLE) data were obtained for the pseudo-ternary systems of propionic acid (PA) + water + solvents (methyl isopropyl ketone and methyl isobutyl ketone) + salts (NaCl and KCl) at 25-26°C in order to study the effect of salts on extraction equilibrium of these systems by comparing with the same systems without salt. Mutual solubility curves, tie-line data, distribution coefficient, selectivity diagrams and separation factor data were determined for these systems. The salting-out effect was investigated by using inorganic salts NaCl or KCl at two concentrations of each salt within their solubility limits. The results obtained in these experiments showed that the salts NaCl and KCl significantly affect the solubility of PA in the organic solvents, as well as the distribution coefficients and separation factors for PA by changing their equilibrium composition and the selectivity of the solvents while the region of heterogeneity increases as compared to the same system of without salt. The thermodynamic consistency of the equilibrium tie-line data was ascertained by Othmer-Tobias plots as well as by Hand plots.

Key words: Liquid-liquid equilibrium, ternary system, salting-out effect, distribution coefficient, tie-line

INTRODUCTION

Liquid-Liquid Extraction (LLE) process is the most useful method for purification, enriching and separation of components of very close volatility and of heat sensitive. Extraction of constituents by using 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. LLE is a process in which a solution is brought into contact with a second liquid (solvent) essentially immiscible or partially miscible with the first one in order to bring about transfer of one or more components from the solution into the second solvent. The solvent phase is usually termed as extract phase and another phase from where the solute is extracted, called diluent or raffinate phase. It is an effective alternative method to distillation process.

With an increase in demand for environmentally friendly chemicals, the efficient separation of carboxylic acids from dilute solutions resulting from fermentation processes is an important where many solvents have been tested to improve such separation demonstrated by Briones *et al.* (1994), Dramur and Tatli (1993), Fahim and Al-Muhtaseb (1996) and Ozmen *et al.* (2004). The extractive recovery of propionic acid by selective solvent

systems from aqueous solutions, such as fermentation broth has received increasing interest. Liquid-liquid equilibrium data for propionic acid (PA)-water-solvents (methyl butyl ketone, methyl isobutyl ketone and methyl isopropyl ketone) was investigated in our previous work (Roy *et al.*, 2006). Propionic acid is an important laboratory reagent as well as solvent for carrying reactions since it remains unchanged by oxidizing and reducing agents. Moreover, it is used as a cellulose-solvent in pharmaceutical industries and also can be used to provide propionates used as fungicides (Kirk and Othmer, 1947).

The presence of dissolved salt changes significantly the phase equilibrium behavior of a mixture, this phenomenon is often referred to as salting-in or salting-out effect. It can be used to optimize separation processes such as rectification to shift the azeotropic conditions, extraction to alter the miscibility gaps and also absorption and fractional crystallization to change the distribution coefficients. During recent years, attempts have been made to generate the reliable as well as reproducible experimental data for the salt containing systems by Azner *et al.* (2000), Edwin and Desai (1971), Govindarjan and Sabarathinam (1997).

In this study, liquid-liquid equilibrium data as well as the mutual solubility data for the ternary systems of

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PA + water + methyl isopropyl ketone (MIPK) and PA + water + methyl isobutyl ketone (MIBK) were determined in the presence of NaCl and KCl as electrolytes at 25-26°C in order to study the salting-out effect on liquid-liquid equilibria of the ternary systems used in the experiments. Separation factor, distribution coefficient and selectivity those are the quantitative index of effectiveness of separation have been computed from the experimental data, those were compared with the same ternary systems without containing salts (Roy et al., 2006). The thermodynamic consistency of the tie-line data were correlated with plots demonstrated by Othmer and Tobias (1942) and by Hand (1930).

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. (1941). All the measurements were carried out at 25-26°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 (cloud point) and the tie-line data determination was performed by taking various mixtures of known compositions within the heterogeneous region were prepared, brought to equilibrium and the compositions of the conjugate phases were determined by analytical method.

Determination of mutual solubility curves: The determination of solubility data of a salt containing pseudo-ternary system was similar to the experimental procedure adopted for the system without salt (Roy et al., 2006), except that water was replaced by aqueous solutions of NaCl or KCl of 5 and 10% of each by weight, within the saturation limit at 25 C. The solubility curves near the solvent-rich region resulted in the precipitation of salt which hindered the disappearance of turbidity and as a result, a complete solubility curves could not be obtained for the entire range of compositions for the systems in this experiment.

Determination of equilibrium data: The determination of equilibrium tie-line data was conducted in an equilibrium cell equipped with magnetic stirrer and isothermal fluid jacket. The mixture temperature was maintained by a thermostatic bath with an accuracy of -0.1°C. The tie-lines were obtained by preparing ternary mixtures of known overall compositions (10 mL of solvent + 10 mL of salt solution +1 to 20 mL of solute) lying within the

Table 1: Physical properties of materials used in this experiment

Materials	Suppliers	Purity (%)	Boiling point (°C)	Density ρ (g mL^{-1})	Refractive index (n$_D$)
Propionic acid	Aldrich, Japan	99.5	141	0.993	1.381
Methyl isobutyl ketone	Aldrich, Japan	99	117	0.801	1.396
Methyl isopropyl ketone	Aldrich, Japan	99	94	0.805	1.388
Sodium chloride, NaCl	Aldrich, Japan				
Potassium chloride, KCl		Aldrich, Japan			

heterogeneous region and stirring vigorously for 1 h and allowed to attain the equilibrium at least 3 h. A complete disappearance of cloudiness in the both layers (solvent and water) ensured the attainment of equilibrium. The individual phases were separated and weighed very carefully. Three compositions of water phase were determined by analytical method and fourth one by material balance and complete compositions of solvent phase were computed on the basis of materials balance after knowing all the compositions of water phase. The composition of PA was analyzed by direct titration against 0.1N sodium hydroxide with phenolphthalein as indicator. Solvent compositions were determined by gas chromatograph (GC) with flame ionization detector (FID) and salt by gravitation method after evaporating the liquid.

The GC-FID analysis was performed using a Shimadzu GC-14A instrument (Shimadzu Inc., Tokyo, Japan) equipped with a J and W DB5-fused silica capillary column (15 m ×0.25 mm i. d. × 0.25 μm film) with He carrier gas: 380 kPa; H$_2$:120 kPa; air: 150 kPa; column pressure: 80 kPa; oven temperature 503 K; injection and detection temperatures: 553.

RESULTS AND DISCUSSION

Mutual solubility data and curves for liquid-liquid ternary systems of propionic acid(PA) + water + methyl isopropyl ketone + salts and propionic acid + water + methyl isobutyl ketone + salts are reported in Table 2 and 3 and Fig. 1a and b, respectively. Figure 1 also shows the mutual solubility curves for these systems without of salt (Roy et al., 2006). The compositions of mutual solubility data in Table 2 and 3 and in Fig. 1 are expressed in weight percent and in weight fraction, respectively. It is evident from the Fig. 1 that the area of heterogeneity for all systems with salts is larger than that of systems of without salts and this effect is observed higher in the higher concentration of salts. This is because of higher mutual solubility of water than that of salt-solution and solvent. It is also concluded that the system that shows

Table 2: Mutual solubility data (on a salt-free basis) for the pseudo-ternary systems of Propionic acid (1) + Water (2) + Methyl isopropyl ketone (3) + Salts (4) at 25-26°C

5% NaCl				10% NaCl			
W_1	W_2	W_3	W_4	W_1	W_2	W_3	W_4
0.00	98.71	1.29	4.94	0.00	98.64	1.36	9.93
11.82	85.04	3.14	4.27	8.33	89.82	1.85	9.05
21.12	71.00	7.88	3.60	21.24	72.00	6.76	7.27
30.10	57.90	12.00	2.96	31.20	58.80	10.00	5.95
37.85	41.80	20.35	2.19	38.90	46.20	14.90	4.67
43.00	29.00	28.00	1.57	46.62	34.30	19.08	3.47
44.82	20.27	34.91	1.15	51.75	26.00	22.25	2.67
40.32	17.27	42.41	1.00	56.43	18.02	25.55	1.87
35.92	13.78	50.30	0.83	50.67	15.16	34.17	1.58
27.95	11.19	60.86	0.70	44.29	11.88	43.83	1.25
5% Kcl				**10% KCl**			
0	95.79	4.21	4.80	0.00	98.20	1.80	9.89
8.06	87.16	4.78	4.38	16.47	82.39	2.14	8.30
24.15	69.75	6.10	3.53	28.14	67.86	4.00	6.85
32.00	60.00	8.00	3.06	40.23	54.50	5.27	5.52
39.21	49.90	10.89	2.58	48.00	42.00	10.00	4.27
44.89	38.16	16.95	2.01	50.91	29.44	19.65	3.01
43.48	26.55	29.97	1.45	52.11	21.34	26.55	2.20
38.22	20.00	41.78	1.13	47.46	17.69	34.85	1.84
32.60	15.49	51.91	0.91	42.26	15.05	42.69	1.57
27.30	11.92	60.78	0.74				

W = wt. percent of composition

Table 3: Mutual solubility data (on a salt-free basis) for the pseudo-ternary systems of Propionic acid (1) + Water (2) + Methyl isobutyl ketone (3) + Salts (4) at 25-26°C

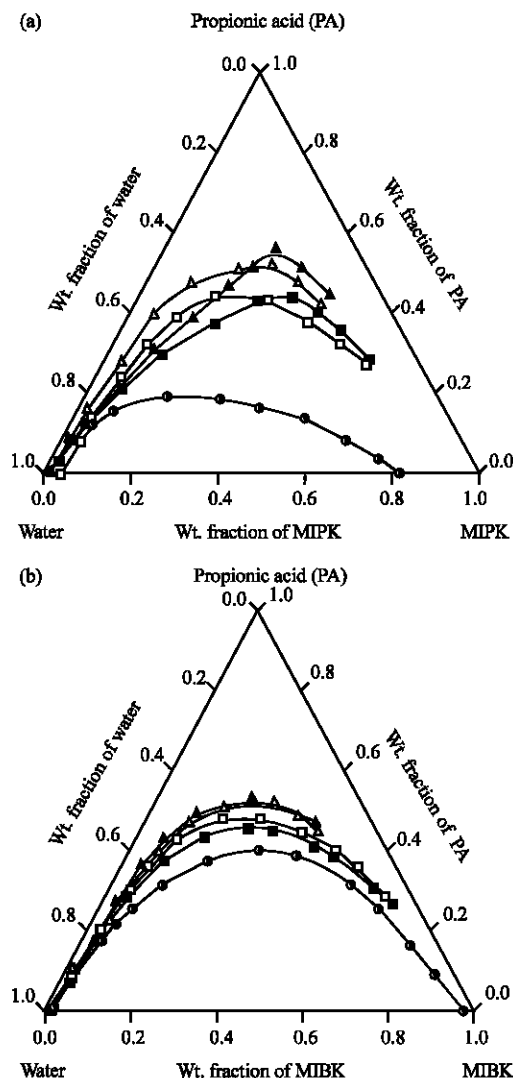| 5% NaCl				10% NaCl			
W_1	W_2	W_3	W_4	W_1	W_2	W_3	W_4
0	99.50	0.50	4.95	0	99.40	0.60	9.96
9.760	88.45	1.79	4.42	6.80	90.80	2.40	9.02
28.60	66.58	4.82	3.43	26.93	70.08	2.99	7.23
37.47	53.80	8.73	2.78	35.73	60.00	4.27	6.38
43.04	41.57	15.39	2.13	42.17	51.69	6.14	5.42
46.06	29.91	24.03	1.63	48.09	40.28	10.63	4.09
44.82	24.27	30.91	1.38	53.25	25.03	21.72	2.48
41.32	16.58	42.10	1.04	46.73	13.50	39.77	1.31
38.42	13.28	48.30	0.87				
30.15	7.99	61.86	0.53				
26.48	5.39	68.13	0.32				
5% KCl				**10% KCl**			
0	99.35	0.65		0	99.50	0.50	
8.42	89.98	1.60	4.510	9.21	89.19	1.60	8.99
19.89	77.71	2.40	3.920	16.52	82.02	2.80	8.27
36	58.00	6.00	2.970	30.00	65.00	5.00	6.57
42.5	48.00	9.50	2.480	39.20	54.00	6.80	5.47
47.75	34.75	17.50	1.850	47.00	43.00	10.00	4.37
48.6	25.95	25.45	1.420	50.00	33.00	17.00	3.37
44	18.00	38.00	1.035	52.31	20.25	27.44	2.09
39.84	12.14	48.02	0.752	48.32	16.90	34.78	1.76
35.49	9.15	55.36	0.610	44.75	13.93	41.31	1.46
28.28	6.38	65.34	0.465				

Fig. 1: Mutual solubility curves for (a) propionic acid-water-methyl isopropyl ketone (MIPK) and (b) propionic acid-water-methyl isobutyl ketone (MIBK), ● : White salt, ■: 5% NaCl, ▲: 10% NaCl, □: 5% KCl, △: 10% KCl

low solubility against water or salt-solution is the superior to use for the separation of propionic acid from its aqueous solution. In the solvent-rich region, precipitation of salt hindered the determination of the solubility curve, so complete solubility curves are not shown in Fig. 1.

Tie-line data: Tie-line data (on salt-free basis) for the systems PA + water + MIPK + salts and PA + water + MIBK + salts, are shown in the Table 4 and 5, respectively, on the basis of weight percent, which represent the equilibrium pseudo-ternary compositions of two phases (aqueous and solvent) while they are in equilibrium stage. According to these Table 4 and 5, the equilibrium distribution compositions as well as other properties of systems studied were computed and compared with the same systems of without salt.

Distribution coefficient and separation factor: The separation factor (S) and the distribution coefficient of

Table 4: Tie-line data (on a salt-free basis) for the pseudo-ternary systems of Propionic acid (1) + Water (2) + Methyl isopropyl ketone (3) + Salts (4) at 25-26°C

Solvent-rich phase (3)				Water-rich phase (2)			
W_{13}	W_{23}	W_{33}	W_{43}	W_{12}	W_{22}	W_{32}	W_{42}
Propionic acid (1) + Water (2) + Methyl isopropyl ketone (3) + 5% NaCl (4)							
9.86	4.95	85.19	0.05	0.91	96.12	2.97	6.9
21.5	8.37	70.13	0.06	2.30	94.30	3.40	7.12
27.95	11.19	60.86	0.1	3.26	92.54	4.20	7.32
33.97	13.78	50.30	0.26	4.89	90.67	4.44	7.93
40.32	17.27	42.41	0.35	6.29	89.06	4.65	8.39
Propionic acid (1) + Water(2) + Methyl isopropyl ketone (3) +10% NaCl (4)							
13.60	5.21	81.19	0.05	0.98	96.89	2.13	13.77
20.80	7.00	72.20	0.07	1.54	95.78	2.68	14.08
30.88	9.29	59.83	0.12	2.56	94.46	2.98	14.59
42.24	13.88	43.88	0.3	5.10	92.28	2.62	18.30
49.67	16.16	34.17	0.49	7.07	91.00	1.93	19.10
Propionic acid (1) + Water (2) + Methyl isopropyl ketone (3) + 5% KCl (4)							
9.98	6.32	83.70	0.02	1.18	96.48	2.34	7.08
18.90	10.02	71.08	0.04	2.18	93.97	3.85	7.32
27.20	11.92	60.78	0.1	3.76	92.07	4.17	7.76
32.60	15.49	51.91	0.35	5.25	90.47	4.28	8.21
38.22	22.00	39.78	0.5	8.06	87.16	4.78	8.74
Propionic acid (1) + Water (2) + Methyl isopropyl ketone (3) + 10% KCl (4)							
12.52	6.28	81.20	0.05	1.25	96.35	2.40	14.38
21.20	8.88	72.92	0.33	2.18	94.99	2.83	16.15
28.26	10.28	61.12	0.64	3.34	93.29	3.37	16.91
35.06	12.60	52.34	0.86	5.09	91.41	3.50	18.64
40.52	16.22	43.21	1.22	7.01	89.19	3.80	20.85

Table 5: Tie-line data (on a salt-free basis) for the pseudo-ternary systems of Propionic acid (1) + Water (2) + Methyl isobutyl ketone (3) + salts (4) at 25-26°C

Solvent-rich phase (3)				Water-rich phase (2)			
W_{13}	W_{23}	W_{33}	W_{43}	W_{12}	W_{22}	W_{32}	W_{42}
Propionic acid (1) + Water (2) + Methyl isobutyl ketone (3) + 5% NaCl (4)							
14.46	6.22	79.32	0.05	0.78	96.06	3.16	7.20
20.87	7.50	71.63	0.07	1.05	95.14	3.81	7.35
26.28	9.09	64.66	0.1	1.28	94.38	4.34	7.70
33.73	12.15	54.12	0.42	1.76	93.15	5.09	8.36
41.69	18.82	39.49	0.75	2.58	90.59	6.83	9.25
Propionic acid (1) + Water (2) + Methyl isobutyl ketone (3) +10% NaCl (4)							
15.12	5.65	79.23	0.06	0.72	97.52	1.76	13.79
26.31	7.86	65.83	0.18	1.12	96.59	2.29	14.04
34.52	9.32	56.16	0.36	1.55	95.66	2.79	14.55
45.22	15.01	39.77	0.52	2.43	94.30	3.27	18.50
53.25	27.03	19.72	0.85	3.82	90.61	5.57	19.40
Propionic acid (1) + Water (2) + Methyl isobutyl ketone (3) + 5% KCl (4)							
15.83	6.56	77.60	0.04	0.89	97.18	1.93	7.39
25.86	7.88	65.34	0.05	1.32	96.12	2.56	7.64
32.49	9.50	55.36	0.08	1.68	94.86	3.46	7.52
38.84	13.14	48.02	0.16	2.25	94.13	3.62	8.23
48.10	26.45	25.45	0.35	4.14	89.98	5.90	8.95
Propionic acid (1) + Water (2) + Methyl isobutyl ketone (3) + 10% KCl (4)							
16.60	5.69	77.71	0.04	0.84	96.16	3.00	14.03
26.92	6.84	66.24	0.06	1.21	94.68	4.11	14.63
33.92	8.38	57.70	0.11	1.57	93.29	5.14	15.10
41.75	12.15	46.10	0.28	2.15	91.41	6.44	16.13
47.82	17.40	34.79	0.51	2.89	89.19	7.92	18.26

propionic acid, (D_1) between water and solvents were determined numerically from the experimental tie-line data in Table 4 and 5. The ratio of weight percent of propionic acid in solvent phase (W_{13}) to the weight percent of propionic acid in diluent phase (W_{12}) for each tie-line gives the distribution coefficients of propionic acid (D_1) and separation factor (S) is a ratio of distribution coefficient of propionic acid (D_1) to the distribution coefficient of water (D_2). Hence the separation factor, $S = D_1/D_2$. The distribution coefficient of water, (D_2) is the ratio of the weight percent of water in solvent phase (W_{23}) to that in water phase (W_{22}). The experimental values of (D_1) and (S) are listed in Table 6 and 7 comprising the values of same parameters obtained in the same systems of without salts from our previous work (Roy *et al.*, 2006). The comparison results of distribution coefficients for

Table 6: Distribution coefficients of propionic acid (D) and separation factors (S) for the systems of propionic acid (1)-water (2)-methyl isopropyl ketone (3) with and without salt at 25-26°C

Without salt		5% NaCl		10% NaCl		5% KCl		10% KCl	
D*	S*	D	S	D	S	D	S	D	S
8.04	35.81	10.84	210.34	13.88	258.08	8.46	129.11	10.01	153.67
7.23	26.80	9.35	105.31	13.50	184.80	8.67	81.30	9.72	104.02
7.26	22.59	8.57	70.90	12.06	122.65	7.26	56.08	8.56	77.70
6.33	14.55	6.95	45.70	8.28	55.06	6.21	36.26	6.89	49.97
4.61	8.65	6.41	33.05	7.02	39.56	4.74	18.78	5.78	31.78

*Values obtained from literature (Roy *et al.*, 2006)

Table 7: Distribution coefficients of propionic acid (*D*) and separation factors (*S*) for the systems of propionic acid(1)-water(2)-methyl isobutyl ketone(3) with and without salt at 25-26°C

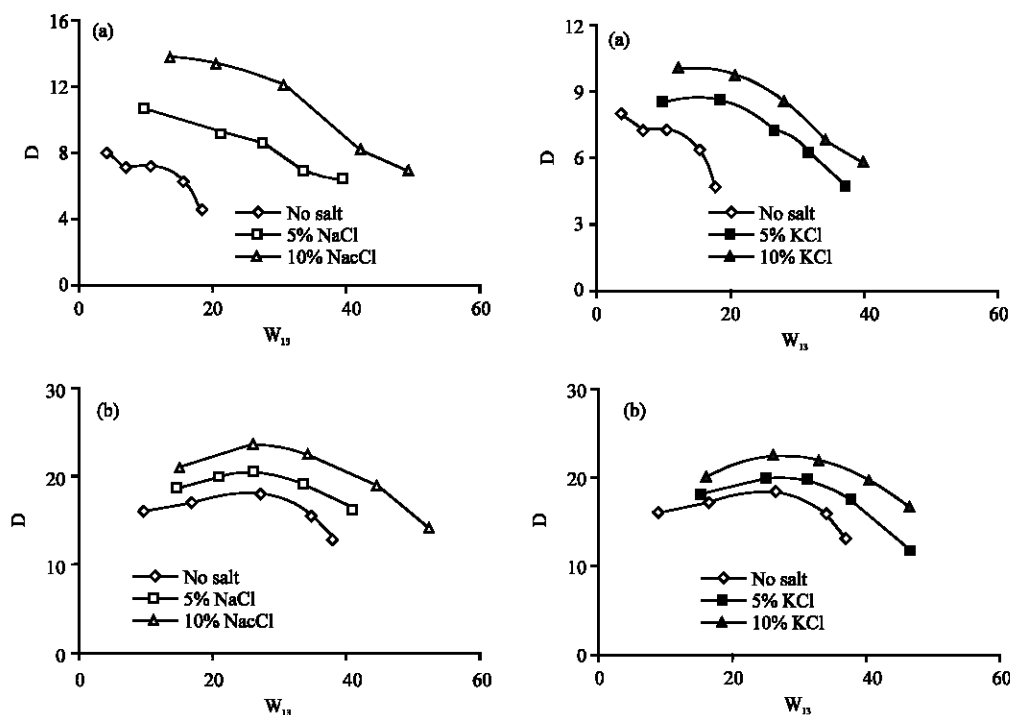| Without salt		5% NaCl		10% NaCl		5% KCl		10% KCl	
D*	S*	D	S	D	S	D	S	D	S
15.83	237.50	18.54	286.30	21.00	362.46	17.78	263.09	19.76	333.97
16.83	209.56	19.87	252.14	23.49	288.67	19.59	238.97	22.25	307.95
18.14	161.66	20.53	213.88	22.27	228.59	19.34	193.11	21.60	240.52
15.55	95.62	19.16	146.93	18.60	116.91	17.26	123.66	19.42	146.09
12.66	54.67	16.16	77.78	13.94	46.73	11.62	39.52	16.54	84.81

*Values obtained from literature (Roy *et al.*, 2006)

Fig. 2: Distribution coefficient, D, plotted against wt. percentage of propionic acid in solvent phase, W_{13}, on salt-free basis for (a) propionic acid-water-MIPK and (b) propionic acid-water-MIBK, with or without salts

the systems studied with or without salts are shown in Fig. 2a and b while it is observed that the distribution coefficient is higher in the system with salts and it increased with increase in concentration of salt for both salts. This indicates that both the salts could bring appreciable changes in the distribution coefficient of propionic acid between water and solvents and this is more prominent in the higher concentration of salts. The distribution coefficient is the key factor in the extraction process.

It is also observed from the Table 6 and 7 that the separation factors (S) for the systems propionic acid + water + MIBK + 10% NaCl and propionic acid + water + MIPK + 10% NaCl are 362.46 and 258, respectively, that is
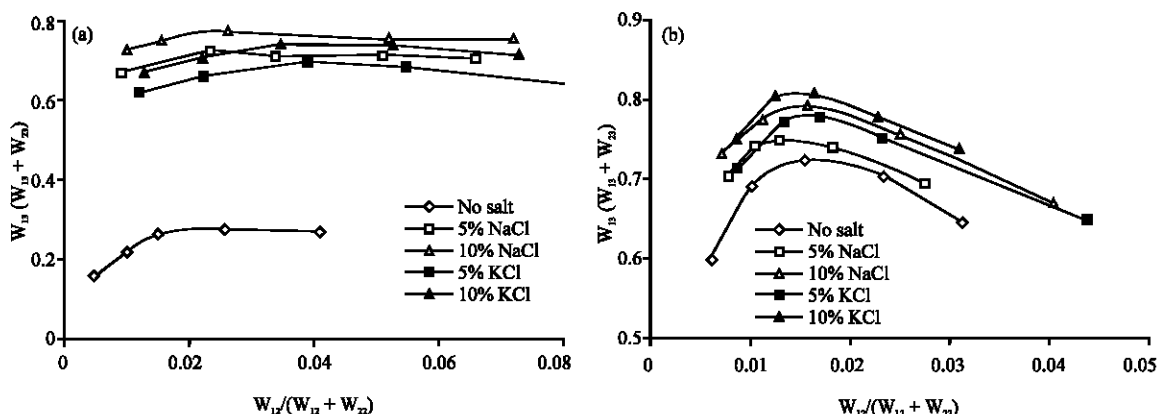
Fig. 3: Selectivity diagram for the systems of (a) propionic acid-water-MIPK and (b) propionic acid-water-MIBK with or without salts
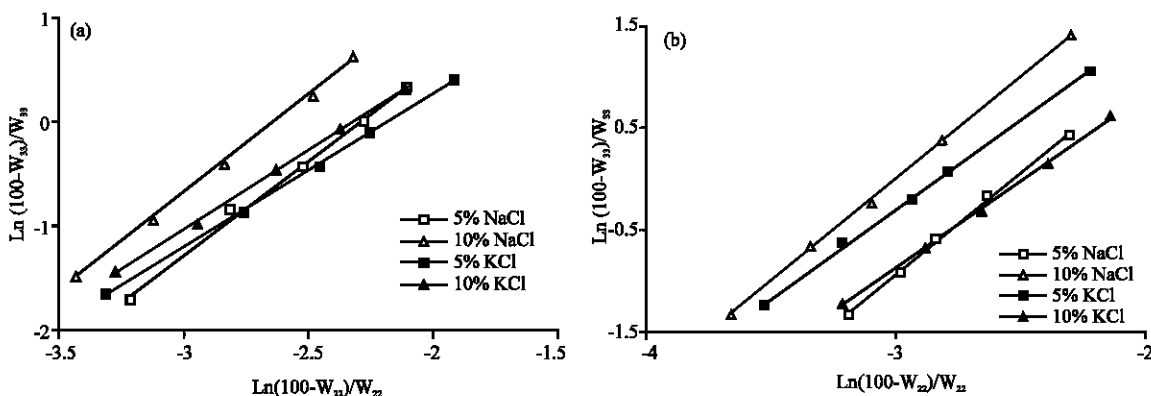

Fig. 4: Tobias correlations on salt-free basis for (a) propionic acid-water-MIPK-salts and (b) propionic acid-water-MIBK-salts of different concentrations

about 1.6 and 7 times higher than those of the same systems without salts. Separation factor is a measure of the solubility of solvents to separate propionic acid from aqueous solution.

Selectivity diagram: This was constructed by plotting weight percent of propionic acid in the solvent phase against that in the diluent phase both on solvent free basis. Selectivity diagrams of all these systems with or without salts are shown in Fig. 3, which reveals that 10% salt solution shows the highest selectivity in all cases and the lowest one is in without salt system while this difference is very significant for the solvent methyl isopropyl ketone.

Othmer-tobias and hand correlations: The reliability of experimentally measured tie line data on the basis of weight percent for these systems is ascertained by

applying Othemer-Tobias (Eq. 1) and Hand (Eq. 2) equations.

$$\ln\left(\frac{100-W_{33}}{W_{33}}\right) = a + b \ \ln\left(\frac{100-W_{22}}{W_{22}}\right) \qquad (1)$$

$$\ln\left(\frac{W_{13}}{W_{33}}\right) = a + b \ \ln\left(\frac{W_{12}}{W_{22}}\right) \qquad (2)$$

Othmer-Tobias plots and Hands plots for the correlations of experimental tie line data are shown in Fig. 4a, b and 5a, b, respectively, while the correlation coefficients (a) and (b) and correlation factor (R^2) values were calculated from their respective correlation curve by the least-squares method, given in Table 8. Going through Fig. 4 and 5, the linearity of the plot indicates the degree of consistency of the related data. It is also observed from

Table 8: Correlation coefficients and factors for the Othmer-Tobias and Hand Equations for Propionic acid (1)-Water (2)-Solvents (3) - Salts systems

Correlation	Solvents	Salts	5% salt			10% salt		
			a	b	R^2	a	b	R^2
Othmer-Tobias	MIPK,	NaCl	1.809	4.12	0.994	1.87	4.92	0.997
		KCl	1.48	3.23	0.996	1.50	3.47	0.998
	MIBK	NaCl	1.92	4.81	0.998	1.95	5.85	0.998
		KCl	1.71	4.82	0.998	1.67	4.13	0.998
Hand	MIPK,	NaCl	1.033	2.67	0.999	1.036	3.01	0.997
		KCl	1.02	2.43	0.994	0.994	2.49	0.996
	MIBK	NaCl	1.39	5.04	0.997	1.48	5.64	0.997
		KCl	1.36	4.86	0.994	1.40	5.16	0.996

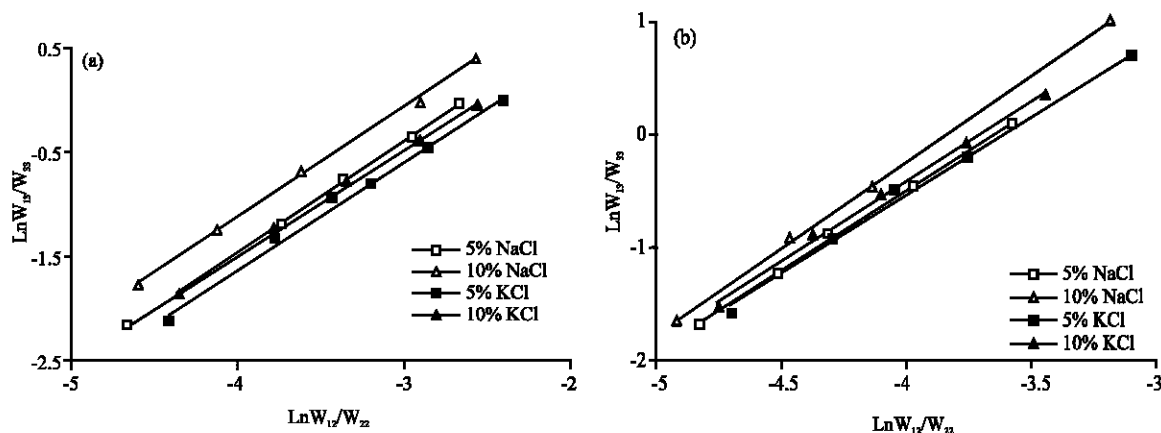MIBK: Methyl isobutyl ketone; MIPK: Methyl isopropyl ketone

Fig. 5: Hand correlations on salt-free basis for (a) propionic acid-water-MIPK-salts and (b) propionic acid-water-MIBK-salts of different concentrations

Table 8 that the correlation factor (R^2) close to unity in all cases, suggests a high degree of consistency of the related data.

CONCLUSIONS

Liquid-liquid extraction equilibrium data, mutual solubility data, distribution coefficient, separation factor data and selectivity diagram for the systems of propionic acid + water + solvents (methyl isobutyl ketone and methyl isopropyl ketone) + salts (NaCl or KCl) were determined at atmospheric pressure and at 25-26°C and compared with the values obtained in the same systems of without salts in order to investigate the effect of electrolytes (aqueous solution of NaCl or KCl) on the LLEE data of these systems. The results obtained from the experiments show that the electrolytes significantly affect the solubility of propionic acid in the organic solvent used. It is also observed that the distribution coefficient of propionic acid and the selectivity of the solvents in extracting propionic acid from its aqueous solution increase in the presence of electrolytes in the ternary system. The tie line data of

the ternary systems with and without salt were satisfactorily correlated by both the Othmer-Tobias and the Hand equations.

As a final conclusion, both solvents in the presence of dissolved electrolyte appears to be better solvents than without presence of dissolved electrolytes for extracting propionic acid from its aqueous solutions, in view of the high values of distribution coefficients and selectivity.

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