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Liquid-Liquid Equilibria of Water + Lacticacid + Butylacetate

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Abstract

Experimental liquid-liquid equilibria of the ternary system of (water + lactic acid + butyl acetate) were studied at T=(298.15, 303.15 and 308.15) K under atmospheric conditions. Complete phase diagrams were obtained by determining solubility and tie-line data. The ternary system exhibits type-1 behavior of LLE. The reliability of the experimental tie-line data was ascertained by using both Hand and Othmer-Tobias correlations. The UNIFAC group contribution method was used to predict the observed ternary liquid-liquid equilibrium data. Distribution coefficients and separation factors were evaluated to evaluate the extracting capability of the solvent. Separation factors were more than unity and the UNIFAC prediction was satisfactory.

Keywords: Lactic acid; Liquid-liquid equilibria; Butyl acetate; Separation factor; UNIFAC

Introduction

Fermentation is one of the viable processes in the production of some chemicals in manufacturing industries. Two of the major obstacles of fermentation are purity of the product and efficiency of the medium. Industrially, lactic acid is produced through the fermentation of sugar, starch, molasses or cheese whey in the presence of micro-organisms such as Lactobaccilus delbrueckii [1-8]. The pure lactic acid is used and applied; in the food industry (as food additives and preservatives), in pharmaceutical industry (as calcium source in calcium lactate), as solvent for lacquers and moisturizers in hand skin lotion or for the production of biodegradable polymers [1,2]. The main obstacle or problem of its industrial production, just like other fermentation processes, is the accumulation of the acid. During the fermentation process, the lactic acid accumulation leads to the simultaneous decrease in the pH of the medium and this causes the process inhibition. Therefore there is need to either remove the acid in situ or add a neutralizer that will optimize the fermentation process, in order to increase the productivity of the acid and the performance of the bioreactor [3-8]. This has therefore caused an increase in interest towards the acid recovery from fermentation broth. Different processes have been applied for this purpose which includes precipitation with calcium hydroxide, use of ion exchange resins and electrodialysis [9-14]. Some of the methods are either complicated, yield low product, have low purity, cause disposal and environmental problems, or costly, which at the end makes about 50% of the total cost spent on separation and purification [15,16]. In lieu of all these separation processes, distillation and extraction processes can also be used to separate waterbased products, the former being non-applicable in the case of lactic acid because the acid can be deformed at high temperature [2,17]. Research has been carried out on the probable use of some groups of extractants (such as alcohols, acetates, ketones, etc.) for the latter process; long chain alcohols were found suitable for this purpose and their liquid-liquid equilibrium data were also determined [18,19]. Also, the equilibrium condition between the organic and aqueous phases was found to be a very important aspect of the liquid-liquid extraction process and variables most generally considered for the process are: physical properties (density difference between liquid phases and boiling point), chemical properties (distribution coefficient, selectivity and flammability), availability and cost [17,20,21].

This study is a part of research program on the recovery of lactic acid from fermentation broth using organic solvents. Several studies have been carried out to obtain LLE data on the extraction of lactic acid from its dilute aqueous solution [19,22,23]. Ternary liquid-liquid equilibria are usually determined by either the titration method, continuous measurement or cell equilibration method [21,24]. In the cell equilibration method, the solubility data is first established using the cloud point method and the tie lines are determined with the aid of a gas chromatograph (GC) [25,26] or a refractometer, which has been initially calibrated [20]. Based on hydrogen bonding interactions, butyl acetate is considered as a probable solvent for the removal of lactic acid from fermentation broth [18]. The LLE results for (water + lactic acid + butyl acetate) at three different temperatures (T = 298.15K, 303.15K and 308.15K) are reported here. The reliability of the experimental data was ascertained by using both the Othmer-Tobias and Hand correlating equations [27].

The liquid-liquid equilibrium data were predicted by the UNIFAC (universal functional group activity coefficient) method [24-29]. The UNIFAC method uses the activity coefficient, γ_i for the prediction. At equilibrium, the activities of the component ion in both phases are equal and the mole fractions x_i^E and x_i^R of the conjugate phases are calculated using the following equation:

$$\gamma_i^{\ E} X_i^E = \gamma_i^{\ R} X_i^R \tag{1}$$

where γ_i^{E} and γ_i^{R} are the corresponding activity coefficients of component i in extract and raffinate phases. The interaction parameters between each of the main groups were used to calculate activity coefficients of component i. The values of the UNIFAC parameters for LLE predictions were taken from a UNIFAC table [29].

Experimental Section

Materials and equipment's

Analytical grade butyl acetate and lactic acid (purities >90%) were purchased and used without further purification. Deionized

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W ₁	W ₂	W ₃	Brix
1.0000	0.0000	0.0000	0.0
0.9950	0.0000	0.0050	0.7
0.9400	0.0525	0.0075	3.5
0.9150	0.0740	0.0110	5.0
0.8685	0.1085	0.0230	8.0
0.7575	0.1825	0.0600	15.0
0.6800	0.2100	0.1100	20.0
0.5900	0.2400	0.1700	25.9
0.5100	0.2600	0.2300	30.3
0.4500	0.2700	0.2800	32.8
0.4000	0.2700	0.3300	34.2
0.1200	0.0000	0.8800	33.3
0.1250	0.0150	0.8600	33.4
0.1300	0.0300	0.8400	33.5
0.1245	0.0475	0.8100	33.6
0.1580	0.0620	0.7800	33.8
0.1785	0.0915	0.7300	34.0
0.2025	0.1375	0.6600	34.2
0.2200	0.1800	0.6000	34.4
0.2550	0.2150	0.5300	34.6
0.2700	0.2400	0.4900	34.7

Table 1: Solubility Data for Water + Lactic acid + Butyl acetate at 298.15K.

W ₁	W ₂	W ₃	Brix
1.0000	0.0000	0.0000	0.0
0.9950	0.0000	0.0050	0.7
0.9425	0.0475	0.0070	3.9
0.9190	0.0725	0.0110	5.4
0.8850	0.0975	0.0175	7.9
0.8550	0.1195	0.0255	10.2
0.7700	0.1800	0.0500	15.1
0.6900	0.2200	0.0900	20.2
0.5950	0.2750	0.1300	25.1
0.5000	0.3000	0.2000	29.2
0.3800	0.3080	0.3120	33.0
0.1050	0.0000	0.8950	33.4
0.1080	0.0220	0.8700	33.5
0.1165	0.0285	0.8550	33.6
0.1325	0.0425	0.8250	33.8
0.1480	0.0700	0.7820	34.0
0.1550	0.0938	0.7512	34.2
0.1640	0.1160	0.7200	34.4
0.2015	0.1485	0.6500	34.6
0.2200	0.1780	0.6020	34.9
0.2550	0.2110	0.5340	35.0

Table 2: Solubility Data for Water + Lactic acid + Butyl acetate at 303.15K.

and redistilled water was used throughout the experiment. Setra digital balance (410S model) was used to measure the masses of the materials. The stated accuracy of the balance is 10⁻³ g. Brix values were measured with Digital Refractometer (203X model) by MISCO Limited London. The surface was cleaned with water before and after every measurement. The refractometer has the variation of \pm 0.1 brix value. Rotary evaporator (52A model) was used to stir the mixture at a constant temperature. Variation of the rotary evaporator is \pm 0.1 °C. Boiling points were measured with Fischer boiling point apparatus.

Experimental procedure

The binodal curves for the (water + lactic acid + butyl acetate) system at T=(298.15, 303.15 and 308.15) K were determined by the cloud point method [14,24,25]. The major central part of the solubility curve was obtained by titrating the heterogeneous mixture of (water + butyl acetate) with lactic acid until the turbidity disappeared. For the water and butyl acetate-side limited regions, binary mixtures of (water + lactic acid) were titrated with butyl acetate and binary mixture of (butyl acetate + lactic acid) titrated with water until the transition from homogeneity to turbidity was observed. The corresponding brix values were recorded. Mutual solubility values of (water + butyl acetate) binaries were also measured using the method based on cloud point detection. The transition point between homogeneity and heterogeneity zones was also determined visually. The titration was performed by adding in minute drops the material and the visual inspection of the transition was achieved by waiting approximately 5 minutes at the transition point and observed the heterogeneity. The graphs of variation of brix value with corresponding mass fractions of water (W_1) and butyl acetate (W_2) were plotted at (T=298.15, 303.15 and 308.15) K for calibration.

End point determinations of tie-lines were based upon the independent analysis of the conjugate phases which were regarded as being in equilibrium. Mixtures of known masses of water, lactic acid and butyl acetate lying within the heterogeneous gap were introduced into rounded bottom flask attached to a rotary evaporator and were stirred rigorously for about 2 hours. The mixture was then transferred into a vertical settler to settle down into water and butyl acetate layers. The brix values of the resulting two phases were measured using the refractometer [20]. The composition of each phase was determined from the calibration graph.

Results and Discussion

Experimental LLE data

The experimental solubility data of the phase equilibria of (water + lactic acid + butyl acetate) at T=(298.15, 303.15 and 308.15) K (Tables 1-3), while the corresponding binodal curves (Figures 1-3). The ternary system exhibits type-1 behavior of LLE [21]. This type is characterized by the total miscibility between two pairs and partial miscibility in the third pair as shown in the figures. The heterogeneous area (that

W ₁	W ₂	W ₃	Brix
1.0000	0.0000	0.0000	0.0
0.9950	0.0000	0.0050	0.7
0.9700	0.0245	0.0055	1.7
0.9500	0.0440	0.0060	3.2
0.9200	0.0685	0.0115	4.5
0.8840	0.0980	0.0180	6.8
0.8300	0.1350	0.0350	10.6
0.7900	0.1600	0.0500	13.1
0.6800	0.2200	0.1000	20.1
0.5400	0.2700	0.1900	30.0
0.4500	0.2800	0.2700	34.3
0.1050	0.0000	0.8950	33.1
0.1225	0.0200	0.8575	33.4
0.1265	0.0250	0.8485	33.5
0.1310	0.0310	0.8380	33.6
0.1475	0.0500	0.8025	33.8
0.1825	0.0875	0.7300	34.3
0.1950	0.1050	0.7000	34.7
0.2275	0.1425	0.6300	35.2
0.2550	0.1850	0.5600	35.7
0.2600	0.2350	0.5050	35.8
0.2700	0.2600	0.4700	36.0

Table 3: Solubility Data for Water + Lactic acid + Butyl acetate at 308.15K.

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Figure 1: Solubility curve (circles), experimental tie lines (squares) and predicted tie-lines (triangles) for water (1) + lactic acid (2) + butyl acetate (3) at T = 298.15K.



Figure 2: Solubility curve (circles),experimental tie lines (squares) and predicted tie-lines (triangles) for water (1) + lactic acid (2) + butyl acetate (3) at T = 303.15K.



Figure 3: Solubility curve (circles),experimental tie lines (squares) and predicted tie-lines (triangles) for water (1) + lactic acid (2) + butyl acetate (3) at T = 308.15K.

is area under the curve) increased with temperature in the order of 298.15K<303.15K<308.15K. This due to the fact that solubility increases with temperature which has in turn increased the heterogeneous area [2].

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Calibration curves were determined for the ternary system at T=(298.15, 303.15 and 308.15) K. The resulting curves are reported in Figures 4-6 respectively. The tie-lines data of the ternary system at T = (298.15, 303.15 and 308.15) K are eventually evaluated from the resulting curves (Tables 4-6). The tie-lines for the system at the three temperatures are shown in Figures 1-3, respectively. The slope of the tie-lines obtained in this work shows that lactic acid is more soluble in water than in butyl acetate.

Distribution coefficient and separation factor

To indicate the ability of the solvent in the recovery of lactic acid, distribution coefficients for water ($D_1 = w_{13}/w_{11}$) and lactic acid ($D_2 = w_{23}/w_{21}$) and separation factors (S) were calculated from experimental data. w_{13} and w_{23} are the mass fractions of water and lactic acid in









	Water	phase		Butyl acetate phase				
Brix	W ₁₁	W ₂₁	W ₃₁	Brix	W ₁₃	W ₂₃	W ₃₃	
3.5	0.9400	0.0525	0.0075	33.6	0.1425	0.0475	0.8100	
3.9	0.9300	0.0615	0.0085	33.7	0.1500	0.0550	0.7950	
5.0	0.9150	0.0740	0.0110	33.8	0.1580	0.0620	0.7800	
6.4	0.8950	0.0885	0.0165	33.9	0.1675	0.0750	0.7575	
8.0	0.8685	0.1085	0.0230	34.0	0.1785	0.0915	0.7300	
10.1	0.8500	0.1200	0.0300	34.1	0.1900	0.1025	0.7075	

 Table 4: Experimental Tie-line Data for Water (1) + Lactic acid (2) + Butyl acetate (3) at 298.15K.

	Water	phase		Butyl acetate phase			
Brix	W ₁₁	W ₂₁	W ₃₁	Brix	W ₁₃	W ₂₃	W ₃₃
3.8	0.9425	0.0505	0.0070	33.5	0.1080	0.0220	0.8700
4.8	0.9300	0.0610	0.0090	33.6	0.1165	0.0285	0.8550
5.4	0.9190	0.0700	0.0110	33.7	0.1250	0.0350	0.8400
6.5	0.9050	0.0810	0.0140	33.8	0.1325	0.0425	0.8250
7.9	0.8850	0.0975	0.0175	33.9	0.1405	0.0545	0.8050
10.2	0.8550	0.1170	0.0280	34.0	0.1480	0.0700	0.7820

 Table 5: Experimental Tie-line Data for Water (1) + Lactic acid (2) + Butyl acetate (3) at 303.15K.

	Water	phase		Butyl acetate phase			
Brix	W ₁₁	W ₂₁	W ₃₁	Brix	W ₁₃	W ₂₃	W ₃₃
3.2	0.9500	0.0440	0.0060	33.9	0.1225	0.0200	0.8575
3.6	0.9390	0.0535	0.0075	34.0	0.1265	0.0250	0.8485
4.2	0.9275	0.0640	0.0085	34.1	0.1310	0.0310	0.8380
5.6	0.9100	0.0765	0.0135	34.2	0.1395	0.0390	0.8215
6.8	0.8840	0.0980	0.0180	34.3	0.1475	0.0500	0.8025
8.7	0.8650	0.1100	0.0250	34.4	0.1550	0.0600	0.7850

 Table 6: Experimental Tie-line Data for Water (1) + Lactic acid (2) + Butyl acetate (3) at 308.15K.



Figure 7: Variation of distribution coefficient of lactic acid with mass fraction of lactic acid in water-rich phase for water + lactic acid + butyl acetate ternary system at 298.15, 303.15 and 308.15K.



organic-rich phase, respectively. w_{11} and w_{21} are the mass fractions of water and lactic acid in aqueous phase, respectively. The separation factor is defined as the ratio of distribution coefficients of lactic acid (1) to water (2), $S = D_2/D_1$.

The variation of distribution coefficient and separation factor for

lactic acid as a function of the solute mass fraction in the aqueous phase for the ternary system at different temperatures are shown in Figures 7 and 8.

The experimental results indicate that butyl acetate has a high separation factor, which shows the ability of this solvent to extract lactic acid from water. This quantity is greater than one (separation factors varying between 3.0438 and 5.9683) for the system reported in this work, which means the extraction of lactic acid by butyl acetate is possible. The separation factors and distribution coefficients are not constant over the whole two-phase region.

In order to compare the effectiveness of extraction of lactic acid by butyl acetate to those of long-chain alcohols, the separation values measured at 298.15 K in this work are compared with those of octanol, nonanol and decanol. The separation values for the three long-chain alcohols (varying between 4.43 and 9.19) were higher than that of butyl acetate, which shows that long-chain alcohols were better extractants compared to butyl acetate [20], though the efficiency of the acetates could be better off using those of long chains [2].

Reliability of the tie-line data

The reliability of experimentally measured tie-line data can be ascertained by applying Othmer-Tobias and Hand correlations [27] at each temperature as shown in equations 4 and 5 respectively.

$$\ln\left(\frac{1-w_{11}}{w_{11}}\right) = a_1 + b_1 \ln\left(\frac{1-w_{33}}{w_{33}}\right) \tag{4}$$

$$\ln\left(\frac{w_{21}}{w_{11}}\right) = a_2 + b_2 \ln\left(\frac{w_{23}}{w_{33}}\right)$$
(5)

Where w_{11} the mass fraction of water in water-rich is phase; w_{21} is the mass fraction of lactic acid in water-rich phase; w_{23} is the mass fraction of lactic acid in solvent-rich phase; and w_{33} is the mass fraction of butyl acetate in solvent-rich phase.

Othmer-Tobias plots were constructed of $\ln[(1-w_{33})/w_{33}]vs$ $\ln[(1-w_{11})/w_{11}]$; and Hand plots were constructed of $\ln(w_{23}/w_{33})vs$ $\ln(w_{21}/w_{11})$. The correlations are shown in Figures 9 and 10 at the three different temperatures.

The linear plots of the correlations were used to determine the coefficients a_i and b_i shown in Table 7. Standard deviations were calculated to evaluate the deviations of the experimental values from the linear plots, according to equation 6. These are reported in Table 8.



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	298.15			303.15		308.15		
D ₁	D ₂	S	D ₁	D ₂	S	D ₁	D ₂	S
0.1516	0.9048	5.9683	0.1146	0.4356	3.8010	0.1289	0.4545	3.5260
0.1613	0.8943	5.5443	0.1253	0.4672	3.7296	0.1347	0.4673	3.4692
0.1727	0.8378	4.8512	0.1360	0.5000	3.6765	0.1412	0.4844	3.4306
0.1872	0.8475	4.5270	0.1463	0.5247	3.5840	0.1533	0.5098	3.3255
0.2055	0.8433	4.1036	0.1588	0.5590	3.5209	0.1669	0.5102	3.0569
0.2235	0.8542	3.8219	0.1731	0.5983	3.4564	0.1792	0.5455	3.0438

D1 and D2 are the distribution coefficients of water and lactic acid respectively.

Table 7: Distribution coefficients (Di) and Separation factors (S) for Water + Lactic acid + Butyl acetate at 298.15K, 303.15K and 308.15K.

Temperature (in K)	in K) 298.15				303.15			308.15				
	a _i	b _i	δ_1	δ_2	a _i	b _i	δ_1	δ_2	a _i	b _i	δ_1	δ_2
Othmer – Tobias	0.042	0.544	0.003	0.002	-0.167	0.617	0.002	0.002	-0.457	0.460	0.002	0.002
Hand	-0.176	0.919	0.025	0.019	0.283	1.350	0.007	0.008	-0.124	1.184	0.016	0.016

Table 8: Coefficients of Othmer–Tobias and Hand Equations and Standard Deviations for water + lactic acid + butyl acetate at 298.15, 303.15 and 308.15K.

Group	Group P	arameters
	R _k	Q _k
CH3	0.9011	0.8480
CH ₂	0.6744	0.5400
H ₂ O	0.9200	1.4000
CH3CO	1.6724	1.4880
CH ₃ COO	1.9031	1.7280
CH ₂ O	0.9183	0.7800
СООН	1.3013	1.2240

Table 9: UNIFAC Group Parameters.

$$\delta_i = \left(\frac{\sum_{k=1}^n \left(w_{ji}^{\ calc} - w_{ji}^{\ exp}\right)^2}{N}\right)^{\frac{1}{2}}$$
(6)

Where N is the number of the tie lines and i=j=1 and 3. δ_1 and δ_2 (i = 1 or 2) are the standard deviations of the mass fractions of water in the raffinate phase and butyl acetate in the extract phase respectively. The standard deviations calculated were low. These give credence to the experimental data obtained.

Prediction of the experimental tie-line data

The UNIFAC method was also used to predict the equilibrium data of the ternary mixtures; the method using the group parameters and group-group interaction parameters of CH, CH₂ CH₃ OH, COOH and

H₂O functional groups obtained by Poling et al. [29], given in Tables 9 and 10, respectively.

A FORTRAN programme was written to predict the LLE data according to UNIFAC equations 6, 7 and 8. The

Predicted values (W_{ij}^{pre} , i=component, j=phase) are compared with the experimental values at the respective temperatures (Tables 11-13).

$$\ln \gamma_i = \ln \gamma_i^C + \ln \gamma_i^R \tag{6}$$

$$\ln \gamma_{i}^{C} = \ln(\varphi_{i} / x_{i}) + 1 - \varphi_{i} / x_{i} - (z / 2)q_{i}[\ln(\varphi_{i} / \theta_{i}) + 1 - \varphi_{i} / \theta_{i}]$$
(7)

$$\ln \gamma_i^R = \sum_k v_{ki} (\ln \Gamma_k - \ln \Gamma_{ki})$$
(8)

The quality of the prediction was evaluated with the residual function (F) calculated from the difference between the experimental value and predicted value for each ternary system according to equation 9; where n is the number of tie-lines, w_{iexp} is the experimental mass fraction and w_{ical} is the predicted mass fraction of component i. j refers to phases and k=1,2,3,4....n(tie lines).

$$F = \left\{ \sum_{k} \min \left[\sum_{i} \sum_{j} \left(w_{i_{exp}} - w_{i_{cal}} \right)^{2} \right] / 6n \right\}^{\frac{1}{2}}$$
(9)

The residual function (F) calculated for (water + lactic acid + butyl acetate) system at T = 298.15K, 303.15K and 308.15K were 0.0271, 0.0387and 0.0332, respectively.

Main group	-CH3	-CH ₂	H ₂ O	–CH ₃ CO	-CH ₃ COO	-CH ₂ O	-соон
-CH ₃	0	0	1318	476.4	232.1	251.5	663.5
-CH ₂	0	0	1318	476.4	232.1	251.5	663.5
H ₂ O	300	300	0	-195.4	72.87	540.5	-14.09
-CH ₃ CO	26.76	26.76	472.5	0	-37.36	-103.6	669.4
-CH ₃ COO	114.8	114.8	200.8	372.2	0	-235.7	660.2
–CH₂O	83.36	83.36	-314.7	191.1	461.3	0	664.6
-COOH	315.3	315.3	-66.17	-297.8	-256.3	-338.5	0

Table 10: UNIFAC group-group interaction parameters (a_{mn}) [29].

	Water phase		Butyl acetate phase			
W ₁₁ pre	W ₂₁ pre	W ₃₁ pre	W ₁₃ pre	W ₂₃ pre	W ₃₃ pre	
0.9231	0.0276	0.0071	0.1451	0.0903	0.8632	
0.9119	0.0342	0.0090	0.1530	0.0989	0.7891	
0.8991	0.0430	0.0113	0.1608	0.1070	0.7572	
0.8623	0.0590	0.0160	0.1738	0.1125	0.7833	
0.8139	0.0815	0.0234	0.1963	0.1218	0.7157	
0.7910	0.0972	0.0300	0.2042	0.1265	0.7086	

Table 11: The predicted liquid-liquid equilibrium data for water + lactic acid + butyl acetate at 25 $^{\circ}$ C.

	Water phase		Butyl acetate phase			
W ₁₁ pre	W ₂₁ pre	W ₃₁ pre	W ₁₃ pre	W ₂₃ pre	W ₃₃ pre	
0.8667	0.0151	0.0071	0.1174	0.0737	0.8813	
0.8682	0.0213	0.0133	0.1243	0.0815	0.8533	
0.8747	0.0278	0.0111	0.1313	0.0881	0.8355	
0.8647	0.0366	0.0141	0.1387	0.0940	0.8183	
0.8318	0.0515	0.0191	0.1495	0.1031	0.7370	
0.7746	0.0768	0.0292	0.1634	0.1067	0.7505	

Table 12: The predicted liquid-liquid equilibrium data for water + lactic acid + butyl acetate at 30 $^\circ\text{C}.$

Water phase			Butyl acetate phase		
W ₁₁ pre	W ₂₁ pre	W ₃₁ pre	W ₁₃ pre	W ₂₃ pre	W ₃₃ pre
0.9471	0.0064	0.0060	0.1228	0.0775	0.8539
0.9347	0.0159	0.0076	0.1271	0.0842	0.8347
0.9187	0.0216	0.0100	0.1323	0.0917	0.8366
0.9047	0.0310	0.0138	0.1403	0.0961	0.8394
0.8673	0.0458	0.0197	0.1504	0.1070	0.7329
0.8378	0.0603	0.0259	0.1600	0.1200	0.7568

Table 13: The predicted liquid-liquid equilibrium data for water + lactic acid + butyl acetate at 35 $^{\circ}\text{C}.$

Conclusion

The binodal and tie-line data for (water +lactic acid + butyl acetate) ternary systems were determined at temperatures of 298.15K, 303.15K and 308.15K. The tie-line data of the system were satisfactorily ascertained by the Othmer-Tobias and Hand correlating equations. Butyl acetate was able to recover lactic acid from aqueous lactic acid solution. The UNIFAC method used predicted the experimental data satisfactorily.

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