

# Vapor-Liquid Equilibrium at Several Experimental Conditions of the Binary Mixtures Methyl Lactate with n-Alkanols

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**Abstract:** In this contribution the vapor-liquid equilibrium of the binary mixtures formed by methyl lactate and the first four lineal alkanols has been determined at constant pressure ( $p = 101.325$  kPa) and at constant temperature ( $T = 323.15$  K). The results have been adequately correlated with the Wilson equation. The obtained data have been satisfactorily checked for thermodynamic consistency using the van Ness method. Furthermore, we have applied the UNIFAC method to predict the vapor-liquid equilibrium and compared these predictions with the experimental results.

**Keywords:** n-Alkanols, Methyl lactate, Vapor-liquid equilibrium.

## 1. INTRODUCTION

Lactate esters have many applications in different fields; they are used as ingredients in chemical industry, as solvents for nitro and ethyl cellulose, oils, dyes, paints and in synthetic polymers. Moreover, lactate esters are also used as additives in biochemical, cosmetics and pharmaceuticals [1].

Among the methods for purification, the esterification of a lactate acid with a suitable alcohol and subsequent hydrolysis of the purified ester is widely accepted as highly efficient [2-5]. This complex process can be carried out by reactive distillation unit. The technology of reactive distillation offers a large number of advantages over the conventional sequential approach of reaction and separation [6]. For the correct design of a reactive distillation column, it is necessary to have knowledge of the phase equilibrium of the system to be separated according to the composition, temperature and pressure.

In this paper, the vapor-liquid equilibrium at isobaric ( $p = 101.325$  kPa) and isothermal conditions ( $T = 323.15$  K) of binary systems containing methyl lactate and a lineal alcohol (methanol, ethanol, 1-propanol and 1-butanol) has been measured over the whole composition range. The experimental data were correlated using the Wilson equation [7]. The obtained results have been checked for the thermodynamic consistency using the van Ness method [8, 9]. Finally, the UNIFAC group contribution method [10, 11] has been used to predict the vapor-liquid equilibrium of the systems studied here.

A survey of the literature shows that Sanz *et al.* [12] determined the isobaric vapor-liquid equilibrium for methanol + methyl lactate at three different pressures (33.33, 66.66 and 101.33 kPa).

## 2. EXPERIMENTAL

The specifications (source, purity and water content) about the chemicals employed in this study can be found in Table 1. Due to the high purities of these compounds (at least 0.997 mass fraction) they were used without further purification. The water content of the liquids was measured using an automatic titrator Crison KF 1S-2B.

An all-glass ebulliometer (Labodest model) manufactured by Fischer was used in the isobaric and isothermal VLE measurements. The equilibrium temperature inside the apparatus was determined using a thermometer (model F25) from Automatic Systems Laboratories with an uncertainty of 0.02 K while the equilibrium pressure was measured with an uncertainty of 0.05 kPa by means of a Digiquartz 735-215A-102 pressure transducer.

In each measurement, the pressure or the temperature were fixed and the liquid mixture was kept at the boiling point for 30 min. Then, samples of liquid and condensed vapor were analyzed by densitometry using a digital densimeter (DMA 5000 model) from Anton Paar, prior to this the corresponding density-calibration curves were obtained [13]; the uncertainty in the determination of mole fraction compositions is 0.002. The experimental equipment and operating procedures have been detailed in previous papers [14, 15].

The experimental normal boiling points and vapor pressures at  $T = 323.15$  K of pure components,  $T_b$  and

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**Table 1: Provenance, Purity and Water Content of the Compounds**

Compound	Source	Purity (mass fraction)	Analysis method	Water content / ppm
Methyl lactate	TCI	0.997	GC	177
Methanol	Sigma-Aldrich	0.998	GC	161
Ethanol	Acros	0.998	GC	152
1-Propanol	Sigma-Aldrich	0.998	GC	197
1-Butanol	Sigma-Aldrich	0.999	GC	188

**Table 2: Normal Boiling Point,  $T_b$ , and Vapor Pressure at  $T = 323.15$  K,  $p^0$ , of the Pure Compounds and Comparison with Literature Data**

Compound	$T_b$ / K		$p^0$ / kPa	
	Exptl.	Lit.	Exptl.	Lit.
Methyl lactate	416.27	417.95 <sup>a</sup> 416.93 <sup>b</sup>	2.400	2.399 <sup>k</sup>
Methanol	337.96	337.92 <sup>c</sup> 337.9 <sup>d</sup>	55.735	55.566 <sup>l</sup>
Ethanol	351.50	351.49 <sup>e</sup> 351.47 <sup>f</sup>	29.545	29.419 <sup>l</sup>
1-Propanol	370.24	370.26 <sup>g</sup> 370.20 <sup>h</sup>	12.185	12.132 <sup>m</sup>
1-Butanol	390.71	390.75 <sup>i</sup> 390.77 <sup>j</sup>	4.505	4.533 <sup>n</sup>

<sup>a</sup> [13]. <sup>b</sup> [17]. <sup>c</sup> [18]. <sup>d</sup> [19]. <sup>e</sup> [20]. <sup>f</sup> [21]. <sup>g</sup> [22]. <sup>h</sup> [23]. <sup>i</sup> [24]. <sup>j</sup> [25]. <sup>k</sup> [26]. <sup>l</sup> [27]. <sup>m</sup> [28]. <sup>n</sup> [29].

$p^0$ , were determined using the same circulating apparatus used for mixtures measurement and they are listed in Table 2 along with some literature data [12, 16-28].

$$\Lambda_{ij} = \frac{V_j^0}{V_i^0} \exp\left(-\frac{\lambda_{ij} - \lambda_{ii}}{RT}\right) \quad (2)$$

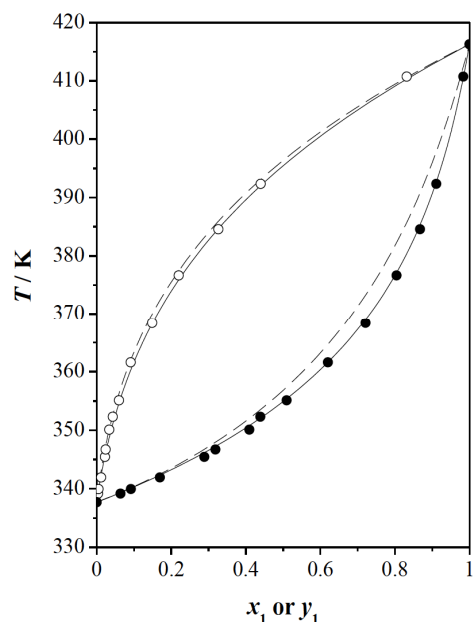
### 3. RESULTS AND DISCUSSION

The experimental data,  $T$ - $x_1$ - $y_1$  or  $p$ - $x_1$ - $y_1$ , along with correlated activity coefficients can be found in the supplementary material. The corresponding ( $T$ - $x_1$ - $y_1$  or  $p$ - $x_1$ - $y_1$ ) equilibrium diagrams can be seen in Figures 1 to 8.

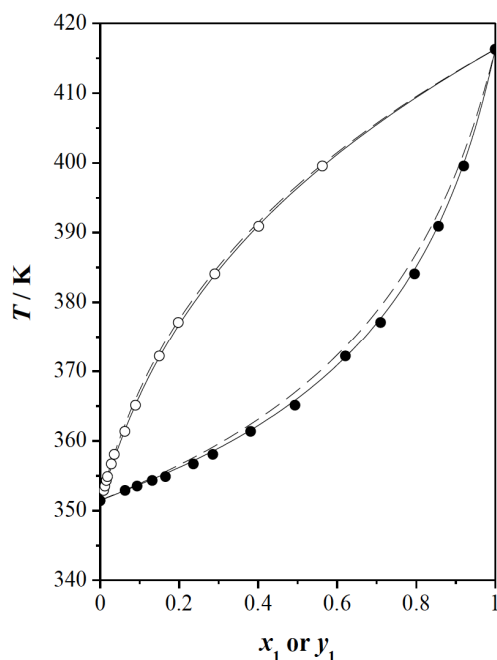
With respect to the comparison of our results at  $p = 101.325$  kPa with previously published ones [12], the agreement is not very good, the average deviation in temperature is 0.99 K while the average deviation in vapor phase composition is 0.0075.

The experimental vapor-liquid equilibrium data were correlated using the Wilson equation that correlates activity coefficients with the temperature,  $T$ , and the liquid phase mole fraction,  $x_i$ :

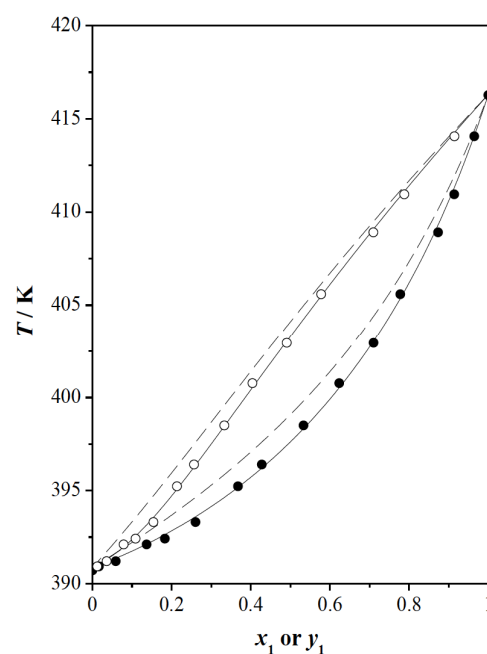
$$\ln \gamma_i = -\ln\left(\sum_j x_j \Lambda_{ij}\right) + 1 - \sum_k \frac{x_k \Lambda_{ki}}{\sum_j x_j \Lambda_{kj}} \quad (1)$$



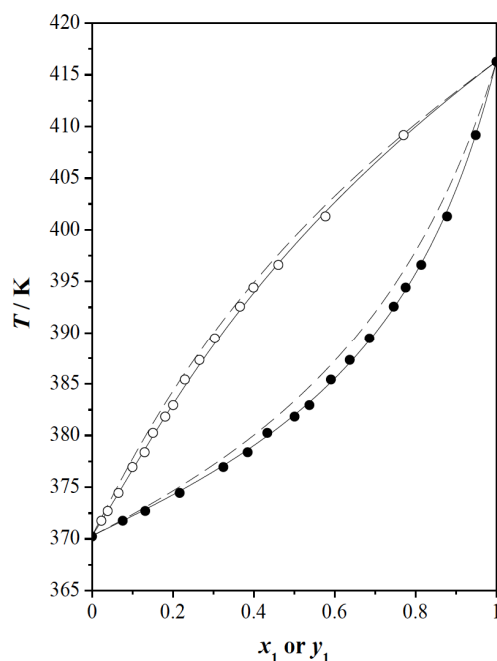
**Figure 1:**  $T$ - $x_1$ - $y_1$  diagram for the binary mixture methyl lactate (1) + methanol (2); (O, ●) experimental data at  $p = 101.325$  kPa; (—) Wilson correlation; (---) UNIFAC prediction.



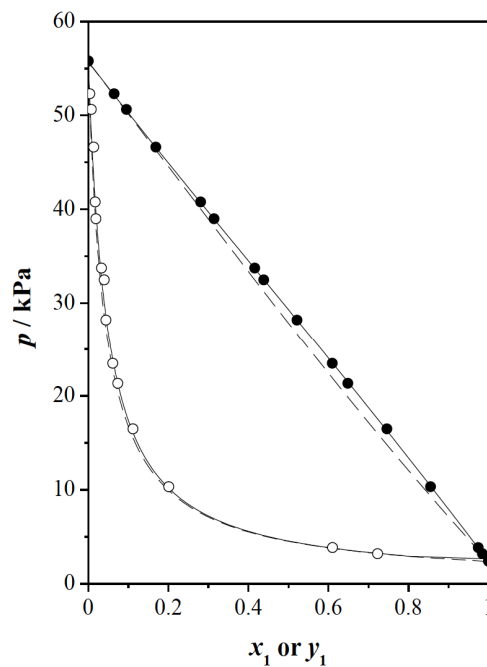
**Figure 2:**  $T$ - $x_1$ - $y_1$  diagram for the binary mixture methyl lactate (1) + ethanol (2): (○, ●) experimental data at  $p = 101.325$  kPa; (—) Wilson correlation; (---) UNIFAC prediction.



**Figure 4:**  $T$ - $x_1$ - $y_1$  diagram for the binary mixture methyl lactate (1) + 1-butanol (2): (○, ●) experimental data at  $p = 101.325$  kPa; (—) Wilson correlation; (---) UNIFAC prediction.



**Figure 3:**  $T$ - $x_1$ - $y_1$  diagram for the binary mixture methyl lactate (1) + 1-propanol (2): (○, ●) experimental data at  $p = 101.325$  kPa; (—) Wilson correlation; (---) UNIFAC prediction.

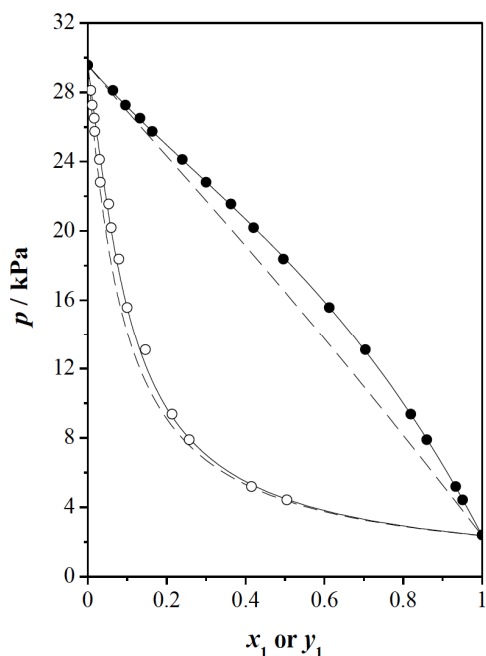


**Figure 5:**  $p$ - $x_1$ - $y_1$  diagram for the binary mixture methyl lactate (1) + methanol (2): (○, ●) experimental data at  $T = 323.15$  K; (—) Wilson correlation; (---) UNIFAC prediction.

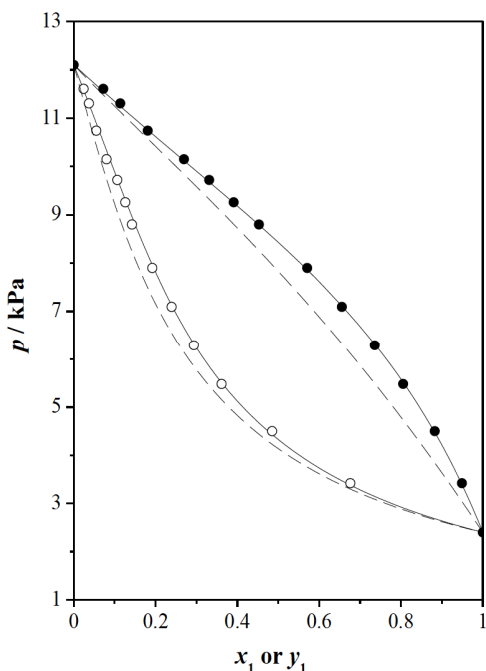
Where  $(\lambda_{ij} - \lambda_{ji})$  are the Wilson parameters,  $V_i^\circ$  is the liquid molar volume of component  $i$  at  $T = 298.15$  K,  $R$  and  $T$  are respectively the gas constant and the absolute temperature. The Wilson parameters have been obtained by minimizing the objective function in

terms of experimental and calculated pressures proposed by Silverman and Tassios [29]:

$$F = \sum_{i=1}^n \left( \frac{p^{\text{exp}} - p^{\text{cal}}}{p^{\text{exp}}} \right)_i^2 \quad (3)$$

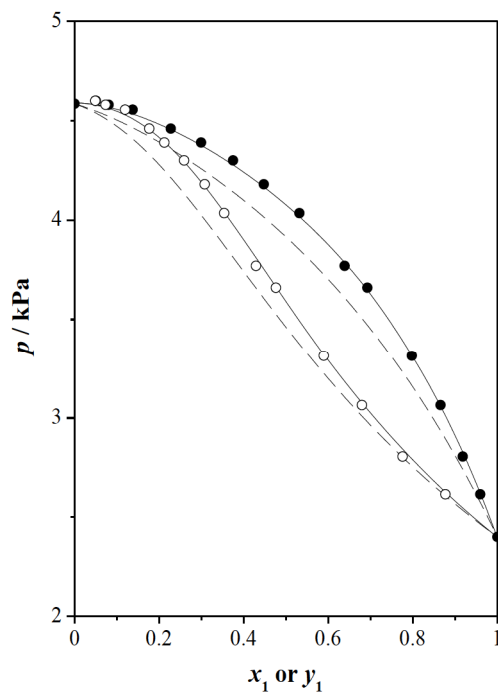


**Figure 6:**  $p$ - $x_1$ - $y_1$  diagram for the binary mixture methyl lactate (1) + ethanol (2): (○, ●) experimental data at  $T = 323.15$  K; (—) Wilson correlation; (---) UNIFAC prediction.



**Figure 7:**  $p$ - $x_1$ - $y_1$  diagram for the binary mixture methyl lactate (1) + 1-propanol (2): (○, ●) experimental data at  $T = 323.15$  K; (—) Wilson correlation; (---) UNIFAC prediction.

For the computation of calculated pressures both the non-ideality of the vapor phase and the variation of the Gibbs function of the pure compounds with pressure have been taken into account, the calculated pressure is obtained from the following equations:



**Figure 8:**  $p$ - $x_1$ - $y_1$  diagram for the binary mixture methyl lactate (1) + 1-propanol (2): (○, ●) experimental data at  $T = 323.15$  K; (—) Wilson correlation; (---) UNIFAC prediction.

$$p^{cal} = \sum_{i=1}^2 x_i \gamma_i p_i^{\circ} \exp \left[ \frac{(V_i^{\circ} - B_{ii})(p - p_i^{\circ}) - (1 - y_i)^2 p \delta_{ij}}{RT} \right] \quad (4)$$

$$\delta_{ij} = 2B_{ij} - B_{ii} - B_{jj} \quad (5)$$

where  $p$  and  $p_i^{\circ}$  are respectively the total pressure and the vapor pressure of the pure compound  $i$ ,  $y_i$  is the vapor phase mole fraction,  $V_i^{\circ}$  is the liquid molar volume of component  $i$  at the equilibrium temperature and  $B_{ii}$  and  $B_{ij}$  are respectively the second virial coefficient of component  $i$  and the cross second virial coefficient. The vapor pressures of the pure components as a function of the temperature were obtained by using the Antoine's equation, taking the constants from Riddick *et al.* [30] and Lomba *et al.* [25], the vapor pressure at  $T = 323.15$  K were measured in our laboratory. The second virial coefficients were calculated using the Tsonopoulos' method [31, 32], and the saturated liquid molar volumes as a function of the temperature were estimated by means of the Rackett equation [33], these molar volumes at  $T = 323.15$  K were experimentally determined.

Table 3 shows the estimated parameters of the Wilson equation at  $p = 101.325$  kPa or at  $T = 323.15$  K for all the studied systems along with the corresponding average deviations in temperatures,  $\Delta T$ ,

**Table 3: Parameters of the Wilson Equation,  $\lambda_{ij}$ - $\lambda_{ii}$ , Average Deviation in Temperature,  $\Delta T$ , or Pressure,  $\Delta p$ , and Average Deviation in Vapor Phase Composition,  $\Delta y$** 

Experimentals conditions	$\lambda_{ij} - \lambda_{ii} / \text{kJ}\cdot\text{mol}^{-1}$	$\lambda_{ij} - \lambda_{ii} / \text{kJ}\cdot\text{mol}^{-1}$	$\Delta T / \text{K}$	$\Delta p / \text{kPa}$	$\Delta y$
Methyl lactate (1) + methanol (2)					
$p = 101.325 \text{ kPa}$	-3262.46	3565.46	0.40		0.0053
$T = 323.15 \text{ K}$	-1592.40	1928.80		0.073	0.0026
Methyl lactate (1) + ethanol (2)					
$p = 101.325 \text{ kPa}$	825.39	274.92	0.37		0.0050
$T = 323.15 \text{ K}$	404.85	1291.09		0.044	0.0043
Methyl lactate (1) + 1-propanol (2)					
$p = 101.325 \text{ kPa}$	2617.14	-547.42	0.18		0.0030
$T = 323.15 \text{ K}$	1029.93	636.65		0.038	0.0044
Methyl lactate (1) + 1-butanol (2)					
$p = 101.325 \text{ kPa}$	993.09	114.15	0.24		0.0030
$T = 323.15 \text{ K}$	1374.69	340.91		0.025	0.0044

or in pressures,  $\Delta p$ , and in vapor phase compositions,  $\Delta y$ , between the experimental results and the calculated ones. As can be seen in Table 3, the correlation with the Wilson equation is quite good, the biggest deviation is shown by the system methyl lactate + methanol at  $T = 323.15 \text{ K}$ . The obtained data have been checked for thermodynamic consistency using the van Ness method [8], described by Fredenslund et al. [9], all the systems satisfied this test.

The systems deviate positively from Raoult's law, although these deviations are not high. Methyl lactate and alkanols have the possibility to form hydrogen bonds due to the presence of OH groups, these hydrogen bonds can be established with like or unlike molecules. The interactions between methyl lactate and an alkanol seems to be similar to those of the pure compounds [34], therefore the deviations from ideal behavior are small.

#### 4. UNIFAC PREDICTIONS

The UNIFAC group contribution method is an effective tool for predicting the vapor-liquid equilibrium of multicomponent mixtures at isobaric or isothermal conditions. In this method, the liquid phase is considered as a mixture of functional groups. Thus, the vapor-liquid equilibrium of the system of interest can be calculated by using limited number of parameters describing the interactions between the structural groups [10]. In this work, we have employed the modified UNIFAC method to predict both isobaric and isothermal vapor-liquid equilibrium of the studied binary mixtures.

The graphical comparison between experimental and predicted results is shown in Figures 1-8. On the other hand, Table 4 displays the average deviations in temperature,  $\Delta T$ , at  $p = 101.325 \text{ kPa}$  or in pressure,  $\Delta p$ ,

**Table 4: UNIFAC Predictions: Average Deviations in Temperature,  $\Delta T$ , or Pressure,  $\Delta p$ , and in Vapor Phase Composition,  $\Delta y$** 

System	Experimentals conditions	$\Delta T / \text{K}$	$\Delta p / \text{kPa}$	$\Delta y$
Methyl lactate (1) + methanol (2)	$p = 101.325 \text{ kPa}$	2.26		0.0179
	$T = 323.15 \text{ K}$		0.920	0.0075
Methyl lactate (1) + ethanol (2)	$p = 101.325 \text{ kPa}$	1.03		0.0079
	$T = 323.15 \text{ K}$		1.280	0.0192
Methyl lactate (1) + 1-propanol (2)	$p = 101.325 \text{ kPa}$	1.14		0.0113
	$T = 323.15 \text{ K}$		0.465	0.0230
Methyl lactate (1) + 1-butanol (2)	$p = 101.325 \text{ kPa}$	1.01		0.0112
	$T = 323.15 \text{ K}$		0.118	0.0117

at  $T = 323.15$  K together with the deviation in vapor phase composition,  $\Delta y$  between the experimental and predicted VLE data. These deviations indicate that for these mixtures the predictions are not very satisfactory. The overall average deviations in temperature and pressure are  $\Delta T = 1.36$  K and  $\Delta p = 0.695$  kPa, respectively. The worst results correspond to the systems: methyl lactate + methanol at  $p = 101.325$  kPa and methyl lactate + methanol at  $T = 323.15$  K.

## 5. CONCLUSIONS

In the present work, the vapor-liquid equilibrium for four binary mixtures: methyl lactate + methanol, methyl lactate + ethanol, methyl lactate + 1-propanol and methyl lactate + 1-butanol have been determined experimentally over the whole composition range at constant pressure 101.325 kPa and at constant temperature 323.2 K using a Fischer-Labodest ebulliometer. The data were considered thermodynamically consistent by passing the Van Ness test.

The correlation of the measured data was performed using the Wilson activity coefficient model, the correlation is adequate.

Moreover, we have also used the UNIFAC method to calculate the the vapor-liquid equilibrium for our systems, these predictions are not quite satisfactory.

## SUPPLEMENTAL MATERIALS

The supplemental materials can be downloaded from the journal website along with the article.

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