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(Vapor + Liquid) Equilibrium for Mixtures Ethanol + Biodiesel from Soybean Oil and Frying Oil

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Abstract

In order to provide new isobaric vapor−liquid equilibrium (VLE) data for ethanol-biodiesel systems, experimental boiling points for mixtures of ethanol + biodiesel from soybean oil (SB) and frying oil (FB) were measured. UNIFAC and NRTL models were used to predict and correlate the data, in order to better represent the VLE in process simulation. VLE data were measured with a Fischer type ebulliometer. Reliability and reproducibility were evaluated with VLE data for ethanol + water system at 101.32 kPa. These data also proved to be thermodynamically consistent based on the area test. The boiling temperatures for ethanol + biodiesel systems agreed with other results reported at same pressure for SB and for biodiesel from sunflower seed oil (SSB). The thermodynamic modelling using the NRTL model obtained lower AAD values than those from UNIFAC, assuring better safety in the design and simulations steps of a biodiesel production plant.

Keywords: *Biodiesel; Ethanol; NRTL; UNIFAC; Vapor−liquid equilibrium.*

1. Introduction

Biodiesel is a renewable fuel composed of alkyl esters produced mainly from vegetable oils and animal fats through the transesterification reaction applied to the triacylglycerols [1]-[3]. However, the products of these reactions incorporate many impurities, requiring the use of secondary purification processes, especially, processes related to liquid-liquid extraction, distillation and separation by gravity present in the most used alkali-catalyzed transesterification route [4],[5].

In order, to improve the performance of the biodiesel production and purification processes, the equilibrium stages conditions involved in each step of these processes must usually be identified. Therefore, the study of vapor-liquid equilibrium (VLE) found in the alcohol recovery and biodiesel purification processes are very important in order to obtain a successful design and operation analysis for the distillation column. This separation is governed by the difference in boiling point between the alcohol and the alky esters [6],[7].

When ethanol is used instead of methanol close attention is required in the biodiesel purification process, mainly, due to the greater solubility of ethyl alcohol in the biodiesel phase. Because the ethanol is more soluble than methanol in this phase, it is more difficult to meet the market specifications after the vapor-liquid separation [8]-[11]. For this reason many biodiesel producers prefer to use methanol, which is also cheaper than ethanol [12],[13]. On the other hand, some countries such as Brazil, produce a large amount of ethanol, so prefer to use the cheaper ethanol as a way to make the biodiesel production more economically competitive than diesel [7],[14]. In addition, the use of ethanol instead methanol is encouraged since it is a renewable fuel and also lesser toxic. Furthermore, few studies have reported on the use of VLE data for ethanol-biodiesel systems close to ambient pressure [6],[11],[15]. These studies are not only important for a better representation of the VLE data in distillation columns and flash drums, but also as they show that a blend alkyl esters-ethanol can be used as a fuel to reduce the ignition delay and combustion problems due to low ethanol vapor pressure [15].

In order to increase the amount of VLE data for ethanol-biodiesel systems, we decided to measure experimental boiling points for mixtures of ethanol + biodiesel from soybean oil (SB) and from frying oil (FB), both at 101.32 kPa. In addition, two models, the Universal Functional-Group Activity Coefficient (UNIFAC) for prediction and the Non-Random Two-Liquid (NRTL) for thermodynamic modelling, were used in order to represent the VLE for these mixtures in the process simulation [16],[17].

2. Materials and Methods

2.1 Chemicals and Biodiesel Preparation

Merck supplied the methanol and ethanol, with purity of 99.9% (mass fraction). Distilled water was also used. Soybean oil (Lisa) was bought from a supermarket and was used to produce fatty acid methyl esters (FAME). An alkali-solid catalyzed (6% of CaO related to oil mass) transesterification reaction was carried out with a methanol to oil molar ratio of 14:1 at 333.15 K for 2 hours [18]. A 99% mass purity of esters was obtained for the soybean

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biodiesel. Frying oil was obtained from the restaurant at a local school and was used to produce the fatty acid ethyl esters (FAEE) that compose the FB. The transesterification reaction was carried out in a reactive distillation column using ethanol to oil molar ratio of 6:1, 1% of KOH catalyst (mass purity of 98%) related to oil mass [19]. The mass purity of esters obtained was higher than 96.5%.

The esters produced and ethanol compositions obtained from the VLE measurements were analyzed by Gas Chromatography (GC) with Flame Ionization Detection IN - GC/FID. The GC was from a model CG-2010 with Auto Injector AOC-5000 from Shimadzu as shown in Table 1.

Table 1. GC conditions for esters produced and compositions obtained in VLE measurements (standard EN 14103).

Variable	Esters analysis|VLE analysis
Column	Stabilwax |RTX-1
Column dimensions	(30 m x 0.25mm x 0.25 µm)|(30m x 0,32mm x 3 µm)
Detector type	FID
Detector temperature	553.15 K|423.15 K
Injector temperature	523.15 K|423.15 K
Carrier gas	Helium
Flow rate|injection volume	1.77 mL/min (constant)|500 µL
Oven temperature	484.15 K (40 min)|323.15 K (10 min)
Split ratio	1:50

2.2 VLE Apparatus and Procedures

The vapor–liquid equilibrium data were measured using a Fischer-type ebulliometer (model 602), as shown in Figure 1 [20, 21]. This is a dynamic measuring cell of VLE data suitable for non-electrolyte systems and allows the study of mixtures containing substances with a high boiling point. The operation is based on the circulation of the liquid and vapor phases in contact with each other until they reach equilibrium. The temperatures of equilibrium were measured using a PT-100 thermometer with a resolution of ± 0.05 K. Samples from the liquid and vapor phases were taken, both through activation of valves, when the equilibrium temperature had been attained. These compositions were analyzed by GC. The Itajaí Basin Alert System Operation Center, located at the Regional University of Blumenau, determined the system ambient pressure (101.32 kPa) at a resolution of ± 0.10 kPa.

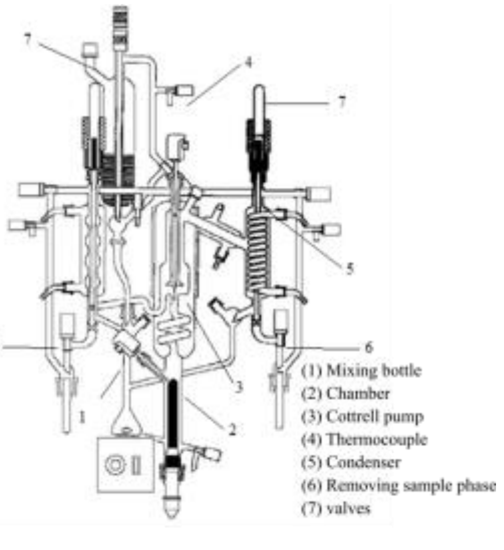

Figure 1. Fisher ebulliometer model 602.

2.3 Prediction and Thermodynamic Modelling

A modified Raoult's law was adopted to represent the VLE equilibrium at low pressures according to Eq. (1)

$$y_i P = x_i \gamma_i P_i^{vap} \tag{1}$$

where y_i and x_i are component vapor and liquid compositions (mole fraction), while P and P_i^{vap} represent total and component vapor pressures. The liquid-phase non-idealities were calculated based on the component activity coefficient (γ_i) [22].

The UNIFAC and NRTL models were used to predict and correlate the γ_i from the VLE data. The experimental (exp) and calculated (cal) results involved in VLE systems were compared using the average absolute deviation (AAD) according to Eq. (2).

$$AAD(V) = \frac{1}{N}\sum_{n=1}^{N}\left|V_n^{exp} - V_n^{cal}\right| \tag{2}$$

In Eq. (2) V represents the variable temperature (T), pressure (P) or vapor phase composition (y) in mole fraction; N are the total number of experimental points n.

The NRTL binary interaction parameters were obtained by minimization of the objective function (OF) adapted from Stragevitch and d'Ávila [23] according to Eq. (3),

$$OF = \sum_{n=1}^{N}\left[\left(\frac{P_n^{exp} - P_n^{cal}}{\sigma_P}\right)^2 + \sum_{i=1}^{I}\left(\frac{y_{i,n}^{exp} - y_{i,n}^{cal}}{\sigma_y}\right)^2\right] \tag{3}$$

where σ_P and σ_y are uncertainties observed in P and y_i; I are the total number of components i.

3. Results and Discussion

The experimental procedure was evaluated regarding reliability and reproducibility through measurements of VLE data for ethanol + water system at 101.32 kPa. These data also proved to be thermodynamically consistent based on the area test from Aspen Plus V8.8 [24],[25]. In addition, these VLE data were compared with other data measured at 101.3 kPa, as reported by Iwakabe and Kosuge [26], so that the methodology was validated given the close agreement with the experimental data as depicted in Figure 2a. The temperature (T) results were plotted against ethanol molar fractions in the liquid (x_1) and vapor (y_1) phases.

In addition, a thermodynamic modelling using NRTL model was carried out in order to compare the data to VLE diagrams. Agreement between experimental and calculated VLE was satisfactory for the region of measured data, based on AAD values as shown in Table 2. These results were attained using component vapor pressure parameters from Aspen Plus V8.8. Figure 2b also shows that the boiling points measured appear to fit better than the dew points. Therefore, we only took into account the data from the saturated liquid phase, since it appears to be more reliable than the data from the dew points.

The boiling temperatures for mixtures of ethanol + SB and ethanol + FB at 101.32 kPa were measured as shown in Table A1 of the Appendix. These data were compared with the VLE data from mixtures ethanol + SB and ethanol + biodiesel from sunflower seed oil (SSB) reported by Silva et al. [11] and Guo et al. [15] at 91.4 kPa and 100 kPa, respectively, as shown in Figure 3a and 3b. These results demonstrate that the VLE data trend agrees with the reported data plotted in ethanol mass fraction in the liquid phase (w_1). Despite the small differences in pressure, the temperature values are so close as to be in agreement with the small temperature changes found when the pressure was increased from 64.4 to 91.4 kPa in the VLE data, as reported by Silva et al. [11] Furthermore, temperatures for the VLE data were expected to be lower than those studied by Guo et al. [15], since the SSB was composed of ethyl esters, while the SB adopted in our work and by Silva et al. [11] were composed of FAME.

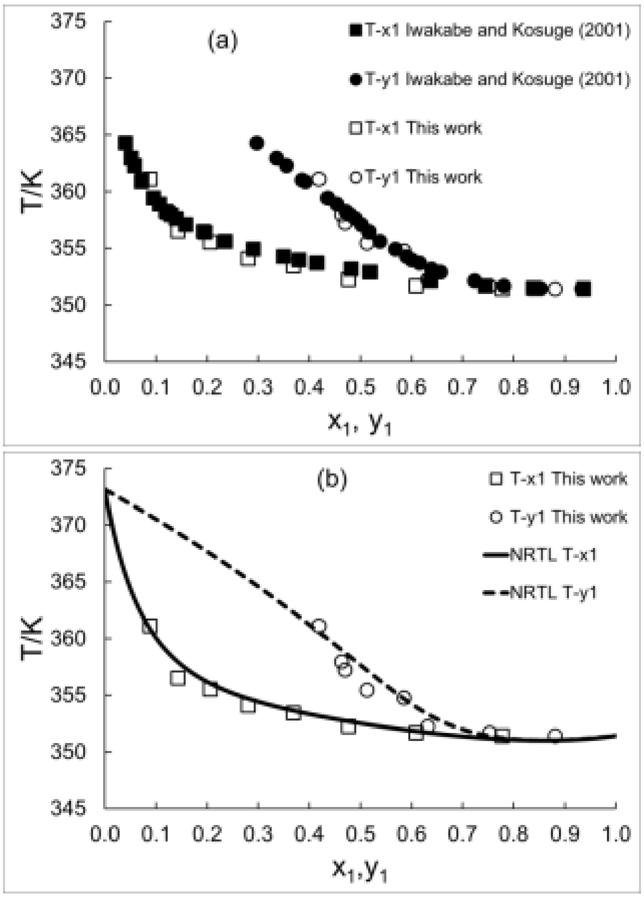

Figure 2. Comparison of VLE data measured for ethanol + water system at 101.32 kPa (a) with reported data [26] at same pressure and (b) with VLE obtained by NRTL model.

Table 2. AAD[a] values obtained in the VLE prediction and correlation with UNIFAC and NRTL models.

EtOH[b] plus	UNIFAC		NRTL		
	AAD(P)	*AAD(T)*	*AAD(P)*	*AAD(y)*	*AAD(T)*
Water	N/A[c]	N/A[c]	1.29	0.05	0.34
SB	6.81	1.79	1.09	N/A[c]	0.27
FB	6.93	1.81	2.44	N/A[c]	0.61

[a] *AAD(P)* and *AAD(T)* are average absolute deviations in kPa and K;
[b] EtOH represents Ethanol;
[c] N/A means not applied.

Figure 3b shows that despite the FB adopted to be composed of ethyl esters, some temperature values were lower compared to the experimental data reported. This behavior occurred because the frying oil had been used to fry food before it was used to produce biodiesel. Therefore, the combination of high temperatures and presence of air may have produced more volatile undesired products. In addition, some FAEE may have changed their geometry from cis to trans, since the frying oil was derived from soybean oil that contains a high amount of these unsaturated ethyl esters. Temperature values for higher biodiesel content were not shown because the FB had already reached the degradation step.

To better predict and model the VLE data obtained, we calculated the vapor pressures of pure and mixture of esters using the model proposed by Ceriani et al. [27] instead the updated Aspen Plus databank. Our choice was based on three reasons. First this model can be applied to a wide range of temperatures in satisfactory agreement with experimental data. Second the same model is also easily applied to mixtures as shown in Figures 4a and 4b. Third binary interactions parameters are not available for all components in Aspen Plus. The composition of fatty acids as specified by Silva et al. [11] and Alcantara et al. [28] for SB and FB, respectively, were used to calculate the vapor pressure of the alkyl ester mixtures, as shown in Table 3.

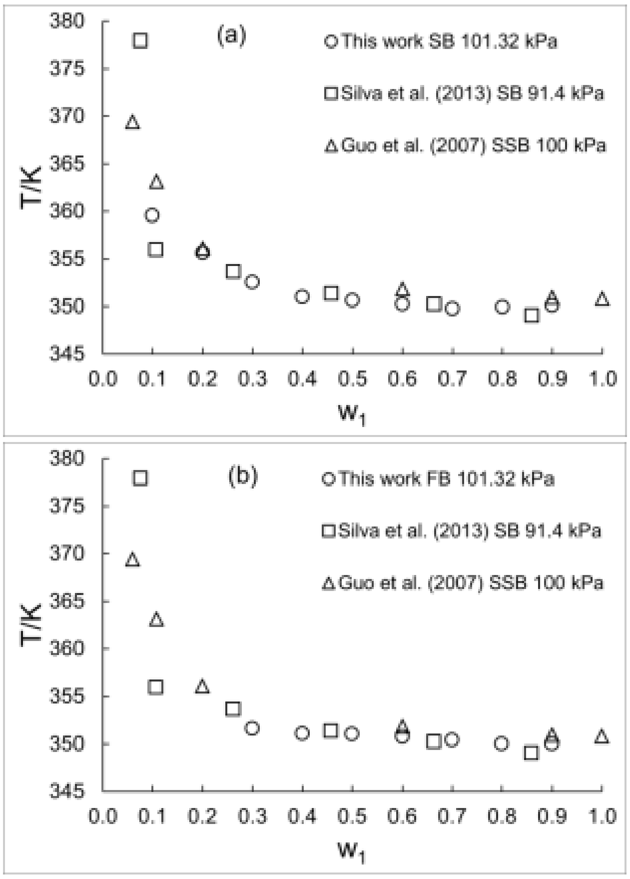

Figure 3. Agreement of VLE data for systems ethanol + biodiesel from soybean oil (SB) and (b) ethanol + biodiesel from frying oil (FB) with boiling points reported [11],[15].

The prediction of the VLE data using UNIFAC was poor for both systems based on *AAD* values, as shown in Table 2, despite a satisfactory representation in Figure 5.

On the other hand, the thermodynamic modelling using the NRTL model fitted relatively well with only few deviations. In addition, the NRTL curves show the expected behavior of a mixture of components with a high difference in boiling points. Table 2 and Table 4 show the deviations and the NRTL interaction parameters, respectively, including the ethanol + water system. The few deviations using the NRTL model assure more safety in the design and simulation steps of vapor-liquid contact equipment, including that one used for ethanol + biodiesel systems.

Table 3. Fatty acid composition adopted for biodiesel from soybean oil (SB) and frying oil (FB).

FAAE[a]	SB (FAME)[a]		FB (FAEE)[a]	
	M/(g/mol)[c]	w/%[d]	M/(g/mol)[c]	w/%[d]
C16:0[b]	270.45	11.1	284.48	12.0
C16:1	-	-	282.46	0.8
C18:0	298.5	4.0	-	-
C18:1	296.49	23.2	310.51	53.1
C18:2	294.47	53.6	308.50	33.1
C18:3	292.46	8.1	306.48	1.0
	Overall:	100	Overall:	100

[a] FAAE, FAME and FAEE mean fatty acid alkyl, methyl and ethyl esters;
[b] The two numbers separated by a colon stand for the carbon chain length and number of double bonds;
[c] M is molar mass;
[d] w is mass fraction.

Table 4. NRTL binary interaction parameters for the systems studied.

System ethanol (1) plus	A_{ij}/K	A_{ij}/K	$\alpha_{ij} = \alpha_{ji}$
Water (2)	120.17	471.34	0.47
Soybean oil biodiesel (3)	4122.70	618.56	0.34
Frying oil biodiesel (4)	2510.70	486.55	0.45

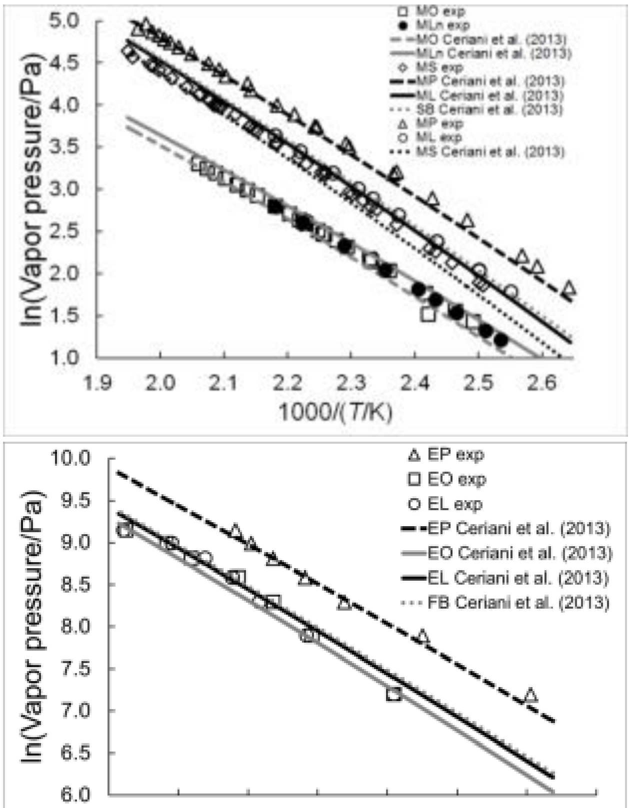

Figure 4. Experimental [29-33] and predicted vapor pressure for (a) methyl esters and biodiesel from soybean oil (SB); (b) ethyl esters and biodiesel from frying oil (FB).

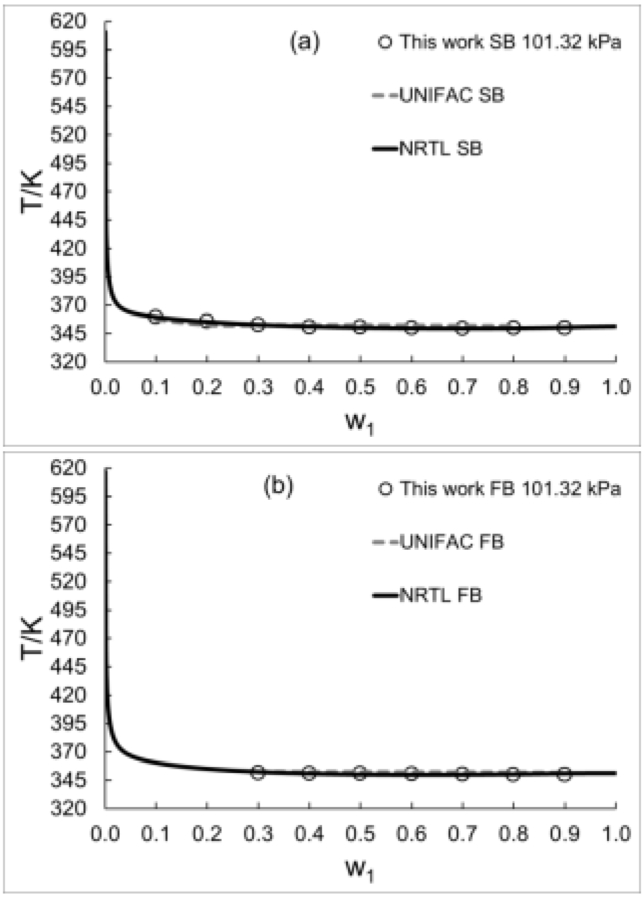

Figure 5. Experimental and calculated bubble-point temperature data using UNIFAC and NRTL models for systems (a) ethanol + SB and (b) ethanol + FB.

4. Conclusions

Vapor–liquid equilibrium data for mixtures of ethanol + biodiesel from soybean oil and ethanol + biodiesel from frying oil at 101.32 kPa were measured. The boiling temperatures in composition charts showed that VLE data agreed with reported data for soybean oil and sunflower seed oil under similar pressure conditions. The thermodynamic modelling using the NRTL model obtained low AAD values, assuring better safety conditions in the design and simulation steps for a biodiesel production plant.

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Nomenclature

AAD	Average absolute deviation
$AAD(P)$	AAD relative to pressure (kPa)
$AAD(T)$	AAD relative to temperature (K)
$AAD(y)$	AAD relative to vapor composition (mole fraction)
A_{ij}	NRTL binary interaction parameter between i-th and j-th components (K)
M	Molar mass (g/mol)
N	Number of experimental points
OF	Objective function

P	Total pressure (kPa)
P_i^{vap}	Vapor pressure of i-th component (kPa)
P_n	Pressure at n-th experimental point
T	Temperature (K)
V_n	Variable at n-th experimental point
V_n^{exp}	Experimental value of variable at n-th experimental point
V_n^{cal}	Calculated value of variable at n-th experimental point
w_i	Mass fraction of i-th component
x_i	Liquid mole fraction of i-th component
y_i	Vapor mole fraction of i-th component
$y_{i,n}$	Vapor mole fraction of i-th component at n-th experimental point

Greek symbols

α_{ij}	NRTL non-randomness parameter between i-th and j-th components
γ_i	Component activity coefficient
σ_P	Uncertainty observed in P (kPa)
σ_T	Uncertainty observed in T (K)
σ_x	Uncertainty observed in x
σ_y	Uncertainty observed in y

Abbreviations

EL	Ethyl linoleate
EO	Ethyl oleate
EP	Ethyl palmitate
FAAE	Fatty acid alkyl esters
FAEE	Fatty acid ethyl esters
FAME	Fatty acid methyl esters
FB	Biodiesel from frying oil (frying biodiesel)
FID	Flame ionization detector
GC	Gas Chromatography
ML	Methyl linoleate
MLn	Methyl linolenate
MO	Methyl oleate
MP	Methyl palmitate
MS	Methyl stearate
NRTL	Non-Random Two-Liquid
SB	Biodiesel from soybean oil (soybean biodiesel)
SSB	Biodiesel from sunflower seed oil (sunflower seed biodiesel)
UNIFAC	Universal Functional-Group Activity Coefficient
VLE	Vapor-liquid equilibrium

Appendix

Table A1. Experimental isobaric boiling points for mixtures of ethanol + biodiesel from soybean oil and frying oil.

Ethanol + SB		Ethanol + FB	
T/K[a]	x_1[b]	T/K[a]	x_1[c]
359.55	0.4134	351.61	0.7402
355.63	0.6132	351.08	0.8159
352.55	0.7310	351.04	0.8692
351.01	0.8088	350.76	0.9088
350.65	0.8638	350.43	0.9394
350.24	0.9049	350.03	0.9638
349.75	0.9367	350.02	0.9836
349.94	0.9621		
350.11	0.9828		

[a] Temperature uncertainty was σ_T = 0.10 K [34];
[b] Component liquid mole fractions were $\sigma_{x_1} = \sigma_{x_2}$ = 0.0023 [34];
[c] Component liquid mole fractions were $\sigma_{x_1} = \sigma_{x_2}$ = 0.0017 [34].

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