

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

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## Abstract

In order to provide new isobaric vapor–liquid equilibrium (VLE) data for ethanol-biodiesel systems, experimental boiling points for ethanol + soybean oil biodiesel (SB) and for frying oil biodiesel (FB) mixtures 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 passed a consistency thermodynamic area test. The boiling temperatures for ethanol + biodiesel systems agreed with other results reported at same pressure for SB and sunflower seed oil biodiesel (SSB). The thermodynamic modelling using the NRTL model obtained lower RMSD 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, animal fats or residual oil and fats through the transesterification or esterification reactions applied to the triacylglycerols or free fatty acids [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 at 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, since this separation is governed by the difference in boiling point between the alcohol and the alkyl 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 the United States and Brazil have high ethanol production, so that they prefer

to use the cheaper ethanol as a way to make the production of biodiesel more economically competitive than diesel [7,14]. Few studies have reported on the use of VLE data for ethanol-biodiesel systems [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 through the 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 ethanol + soybean oil biodiesel and for frying oil biodiesel systems, 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) model 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-wt%. Distilled water was also used. Soybean oil 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 during 2 hours. A 99% mass purity of esters was obtained for the soybean biodiesel. Frying oil was obtained from the restaurant at a local school and used to produce the fatty acid ethyl esters (FAEE) that compose the frying oil

biodiesel. 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. The mass purity of esters obtained was greater 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 [18, 19]. 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  $\pm 0.05$  K. Samples of liquid and vapor phases were taken, both through activation of valves, when the equilibrium temperature were attained. Those 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  $\pm 0.10$  kPa.

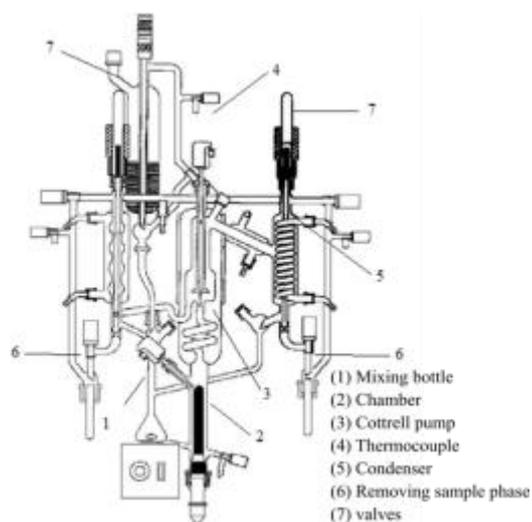


Figure 1. Fisher ebulliometer model 602.

## 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)

$$y_i P = x_i \gamma_i P_i^{vap} \quad (1)$$

where  $y_i$  and  $x_i$  are component vapor and liquid compositions (mole fraction), while  $P$  and  $P_i^{vap}$  represents total and component vapor pressures. The liquid-phase non-idealities were calculated based on the component activity coefficient ( $\gamma_i$ ) [20].

The UNIFAC and NRTL models were used to predict and correlate the activity coefficients from the VLE data. The experimental and calculated compositions involved in VLE systems were compared using the root mean square deviation (*RMSD*) for a variable  $V$  according to Eq. (2)

$$RMSD(V) = 100 \sqrt{\frac{1}{N} \sum_{n=1}^N (V_n^{exp} - V_n^{cal})^2} \quad (2)$$

where  $V$  represents the variable temperature ( $T$ ), pressure ( $P$ ) or vapor ( $y_i$ ) phase composition (mole fraction);  $N$  is the number of experimental points. The superscripts *exp* and *cal* denote experimental and calculated compositions.

The NRTL binary interaction parameters were obtained by minimization of the objective function (*OF*) adapted from Stragevitch and d'Ávila [21]

$$OF = \sum_{n=1}^N \left[ \left( \frac{P_n^{exp} - P_n^{cal}}{\sigma_P} \right)^2 + \left( \frac{y_n^{exp} - y_n^{cal}}{\sigma_y} \right)^2 \right] \quad (3)$$

where  $\sigma_P$  and  $\sigma_y$  are uncertainties observed in  $P$  and  $y_i$ .

## 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 passed a consistency thermodynamic area test using Aspen Plus V8.8 [22, 23]. In addition, these VLE data were compared with other data measured at 101.3 kPa reported by Iwakabe and Kosuge [24], so that the methodology was validated given the close agreement with the experimental data as depicted in Figure 2a.

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, based on *RMSD* 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 isobaric boiling temperatures for ethanol + soybean oil biodiesel (SB) and ethanol + frying oil biodiesel (FB) systems at 101.32 kPa were measured and are shown in Table 3. These data were compared with the VLE data of ethanol + soybean oil biodiesel (SB) and ethanol + sunflower seed oil biodiesel (SSB) systems 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. 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, boiling 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 soybean biodiesel adopted in our work and by Silva et al. [11] were composed of methyl esters.

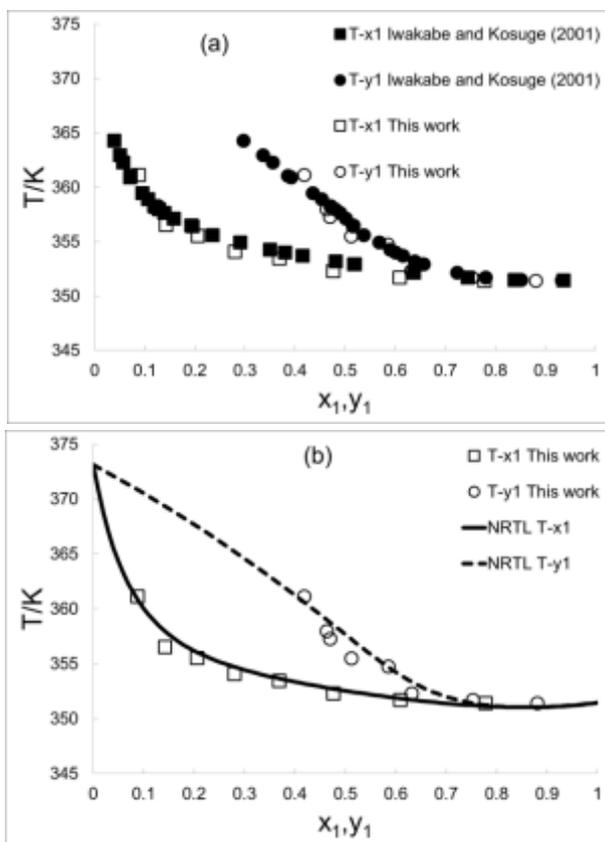


Figure 2. Comparison of VLE data measured for ethanol + water system at 101.32 kPa (a) with reported data [24] at same pressure and (b) with VLE obtained by NRTL model.

Table 2. RMSD obtained in the VLE prediction and correlation with UNIFAC and NRTL models

Ethanol plus	UNIFAC		NRTL		
	$RMSD_p^a$	$RMSD_T^a$	$RMSD_p$	$RMSD_y^a$	$RMSD_T$
Water	N/A <sup>b</sup>	N/A	1.29	0.05	0.34
SB	6.81	1.79	1.09	N/A	0.27
FB	6.93	1.81	2.44	N/A	0.61

<sup>a</sup> $RMSD_p$ ,  $RMSD_y$  and  $RMSD_T$  are RMSD relative to pressure (kPa), vapor molar fraction and temperature (K).

<sup>b</sup>N/A means not applied.

Figure 3b shows that despite the fact that the frying oil biodiesel adopted was composed of ethyl esters, some temperature values were lower compared to the experimental data reported. This behavior occurred because the frying oil was used to fry food before it was used to produce biodiesel. Therefore, some ethyl esters may have changed their geometry from cis to trans, probably, as ethyl linoleate and ethyl linoleate by an isomerization reaction, since the frying oil was derived from soybean oil that contains a high amount of these unsaturated ethyl esters. In addition, due to the high temperatures during the frying of food and the presence of air, it is possible that other reactions may have occurred producing other more volatile

undesired products. Temperature values for higher biodiesel content were not presented because the FB had already reached the degradation step.

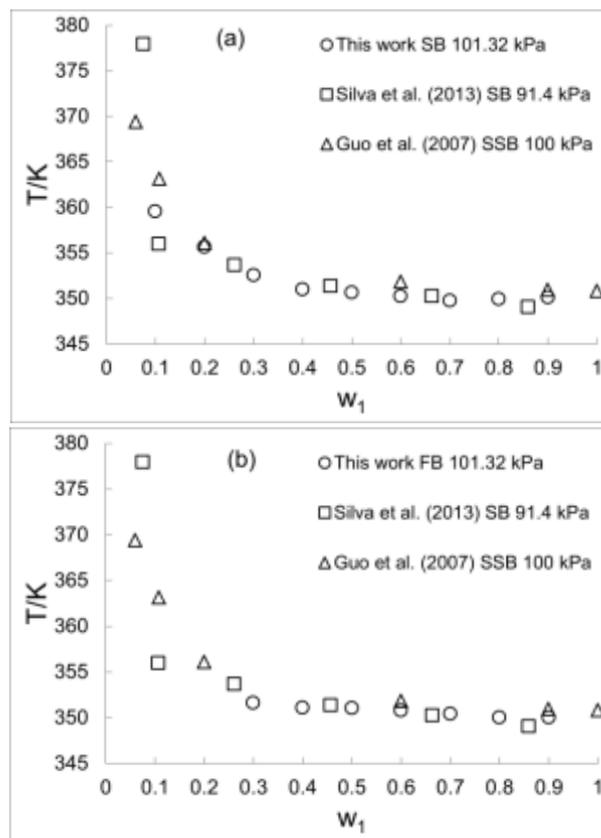


Figure 3. Agreement of VLE data for systems (a) ethanol + soybean oil biodiesel (SB) and (b) ethanol + frying oil biodiesel (FB) with boiling points reported [11],[15].

To better predict and model the VLE data obtained, we calculated pure and mixtures of ester vapor pressure using the model proposed by Ceriani et al. [26], since this model applies wide range of temperatures in satisfactory agreement with experimental data. Furthermore, the same model is also easily applied to mixtures as shown in Figure 4a and 4b. The fatty acids composition specified by Silva et al. [11] and Alcantara et al. [27] for soybean oil biodiesel and frying oil biodiesel, respectively, were used to calculate the vapor pressure of the alkyl ester mixtures, as shown in Table 4.

Table 3. Experimental isobaric boiling points for ethanol+biodiesel systems from soybean and frying oils.

Ethanol + soybean oil biodiesel		Ethanol + frying oil biodiesel	
$T/K^a$	$x_l^b$	$T/K^a$	$x_l^c$
359.55	0.4134	351.61	0.7402
355.635	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		

<sup>a</sup> Temperature uncertainty was  $\sigma_T = 0.10$  K [25].

<sup>b</sup> Component liquid mole fractions were  $\sigma_{x_1} = \sigma_{x_2} = 0.0023$  [25].

<sup>c</sup> Component liquid mole fractions were  $\sigma_{x_1} = \sigma_{x_2} = 0.0017$  [25].

Table 4. Fatty acid composition adopted for soybean and frying oil biodiesel

FAAE <sup>a</sup>	Soybean biodiesel (FAME) <sup>a</sup>		Frying biodiesel (FAEE) <sup>a</sup>	
	<i>M</i> /(g/mol) <sup>c</sup>	<i>w</i> / % <sup>c</sup>	<i>M</i> /(g/mol) <sup>c</sup>	<i>w</i> / % <sup>c</sup>
C16:0 <sup>b</sup>	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

<sup>a</sup>FAAE, FAME and FAEE mean fatty acid alkyl esters, methyl esters and ethyl esters;

<sup>b</sup>The two numbers separated by a colon stand for the carbon chain length and number of double bonds;

<sup>c</sup>*M* is molar mass and *w* is mass fraction.

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

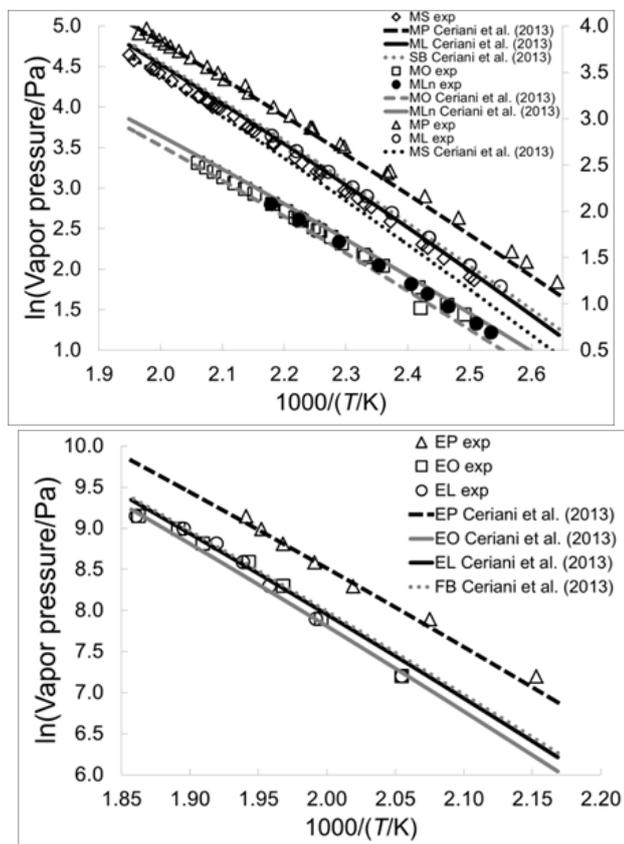


Figure 4. Experimental [28-32] and predicted vapor pressure for (a) methyl esters and soybean oil biodiesel adopted; (b) ethyl esters and frying oil biodiesel adopted.

On the other hand, the thermodynamic modelling using the NRTL model fitted relatively well with only few deviations. Table 2 and Table 5 show the *RMSD* values and the NRTL interaction parameters, respectively, including the ethanol + water system. The few deviations using NRTL model assure more safety in the design and simulation steps of vapor-liquid contact equipment, including that used with ethanol + biodiesel systems.

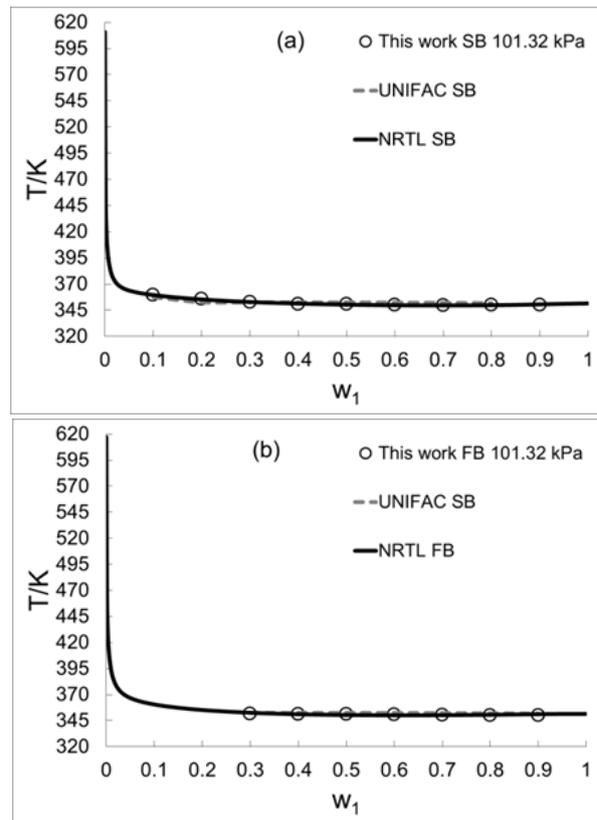


Figure 5. Experimental and calculated bubble-point temperature data using UNIFAC and NRTL models for systems (a) ethanol + SB and (b) ethanol + FB.

Table 5. NRTL binary interaction parameters for the systems studied

System ethanol (1) plus	<i>A</i> <sub>12</sub> /K	<i>A</i> <sub>13</sub> /K	$\alpha_{12} = \alpha_{13}$
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

#### 4. Conclusions

Vapor-liquid equilibrium data for systems of ethanol + soybean oil biodiesel and ethanol + frying oil biodiesel 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 *RMSD* values, assuring better safety conditions in the design and simulation steps for a biodiesel production plant.

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#### Nomenclature

<i>A</i> <sub><i>ij</i></sub>	NRTL binary interaction parameter between <i>i</i> -th and <i>j</i> -th components (K)
<i>M</i>	Molar mass (g/mol)
<i>N</i>	Number of experimental points
<i>OF</i>	Objective function
<i>P</i>	Total pressure (kPa)
<i>P</i> <sub><i>i</i></sub> <sup>vap</sup>	Vapor pressure of <i>i</i> -th component (kPa)

$P_n$	Pressure at $n$ -th experimental point
$RMSD$	Root mean square deviation
$RMSD_P$	RMSD relative to pressure (kPa)
$RMSD_T$	RMSD relative to temperature (K)
$RMSD_x$	RMSD relative to liquid molar fraction
$RMSD_y$	RMSD relative to vapor molar fraction
$T$	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
$y_n$	Vapor mole fraction at $n$ -th experimental point

#### Greek symbols

$\alpha_{ij}$	NRTL non-randomness parameter between $i$ -th and $j$ -th components
$\gamma_i$	Component activity coefficient
$\sigma_P$	Uncertainty observed in $P$ (kPa)
$\sigma_T$	Uncertainty observed in $T$ (K)
$\sigma_x$	Uncertainty observed in $x$
$\sigma_y$	Uncertainty observed in $y$

#### Abbrev. Definitions

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	Frying oil 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	Soybean oil biodiesel
SSB	Sunflower seed oil biodiesel
UNIFAC	Universal Functional-Group Activity Coefficient
VLE	Vapor-liquid equilibrium

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