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# Density, Excess Volumes, and Partial Volumes of Binary Mixtures of Soybean Biodiesel + Diesel and Soybean Biodiesel + *n*-Hexadecane at Different Temperatures and Atmospheric Pressure

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Densities, $\rho$, of binary mixtures of diesel + soybean biodiesel and n-hexadecane + soybean biodiesel have been measured as a function of the composition at different temperatures (293.15, 313.15, 333.15, 353.15, and 373.15) K and atmospheric pressure, over the entire composition range, using an Anton Paar SVM 3000 oscillating U-tube densimeter. The excess molar volume, $V^E$, and excess partial molar volume, $\bar{V}^E$, of the binary mixtures were calculated from the density data. These data have been correlated using the Redlich−Kister type function in terms of mass fraction ($w$), with a maximum standard deviation of 0.152 $\text{cm}^3\cdot\text{mol}^{-1}$. The excess volumes are positive for the soybean biodiesel + diesel system. The values of the excess volume for the system soybean biodiesel + *n*-hexadecane are also positive and an inversion of sign in $V^E$ is observed at higher molar fractions of soybean biodiesel (around $x_1 = 0.60$ to 0.80). The excess partial volume at infinite dilution has been also calculated.

## Introduction

In Brazil, renewable energy has gained attention during the last decades first with the PROALCOOL Program, involving production of ethanol fuel from sugar cane, and now with the BIODIESEL Program. In Brazil, a biodiesel content of B5 [in other words, mass fraction ($w$) = 0.05 of biodiesel] on the diesel blends commercialized since January 2010 was established by the Brazilian Regulatory Agency (Agência Nacional do Petróleo, Gás Natural e Biocombustíveis, ANP).[1] The production of biodiesel from vegetable oils is one of the alternatives for a world strategy to expand renewable energy sources. Several methods exist for producing biodiesel. The most significant is the transesterification method using basic homogeneous catalyst. Biodiesel has many advantages, and it has been increasingly used as a substitute for conventional diesel fuel, once it may be directly used in diesel vehicles with little or no modification.[2,3]

The knowledge of the fluid properties is important for many industrial applications. Among these properties, density plays an important role. Those properties are relevant from the stand point of both equipment design and process control. It should be remembered that density is a fuel property which has a direct effect on the engine performance characteristics. Many performance characteristics such as cetane number and heating value are related to the density.[4] This property also influences the efficiency of fuel.[5,6]

For diesel engines, one of the major performance characteristics is the ignition quality, related to the diesel fuel chemical structure. This quality is expressed by the cetane number. The behavior of the diesel fuel is compared to that of two pure hydrocarbons selected as a reference, *n*-cetane or *n*-hexadecane ($CH_3{-}(CH_2)_{14}{-}CH_3$) which is given the number 100 and $\alpha$-methylnaphthalene which is given the number 0.[7] Although hexadecane is a slightly larger molecule than what is thought of as the average for petrodiesel, it is commonly used as a reference molecule for modeling diesel fuel thermodynamics properties.

The excess properties of binary mixtures containing biodiesel are useful in the study of molecular interactions and arrangements, and such data are scarce in the literature for these systems. For this reason, a set of experiments have been carried out on density for binary mixtures of soybean biodiesel + diesel and soybean biodiesel + hexadecane, at different temperatures (293.15, 313.15, 333.15, 353.15, and 373.15) K and atmospheric pressure. Excess volume has been derived from these data. It should be noticed that biodiesels and diesel have a multicomponent composition. Nevertheless, it was treated here as a pseudopure component (e.g., the molecular weight of biodiesel was considered as a weighed average in terms of mass fraction) and their mixtures as pseudobinary mixtures only for the purpose of parameter estimations.

## Experimental Section

***Materials.*** In this study, commercially grade edible soybean oil was used in biodiesel production. It should be noticed that due its low acidity number (less than 0.5 mg $\text{KOH}\cdot\text{g}^{-1}$) no further purification was needed. The biodiesel samples were obtained by alkaline-catalyzed (potassium methoxide, mass fraction ($w$) = 0.08) transesterification of soybean oil using methanol (supplied from J. T. Baker) as transesterificant alcohol, without any pretreatment to the feedstocks due to their lower acidity number. Methanol in excess, about volume fraction ($\varphi$) = 100 %, was used in those reactions. Transesterification reactions were carried out homogeneously in liquid phase, in a laboratory scale apparatus, consisting of a 2000 mL reaction flask equipped with a reflux condenser, magnetic stirrer, and thermometer. The reaction procedure consisted of initial addition of the oil and alcohol phases, followed by the catalyst loading under mechanical agitation (1250 rpm), at 298 K during 1 h. After 12 h of transesterification reaction, the glycerin layer was

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**Table 1. Fatty Acids Methyl Esters (FAME) Profile of Soybean Biodiesel**

| fatty acid | mass fraction ($w$) |
|---|---|
| methyl palmitate (C16:0; $C_{17}H_{32}O_2$) | 0.1132 |
| methyl stearate (C18:0; $C_{19}H_{34}O_2$) | 0.0313 |
| methyl oleate (C18:1; $C_{19}H_{36}O_2$) | 0.2387 |
| methyl linoleate (C18:2; $C_{19}H_{34}O_2$) | 0.5494 |
| methyl linolenate (C18:3; $C_{19}H_{32}O_2$) | 0.0494 |
| methyl behenate (C22:0; $C_{23}H_{46}O_2$) | 0.0181 |

**Table 2. Physicochemical Properties of Pseudopure Biodiesel Produced from Soybean, Diesel Fuel Properties**

| physical property | soybean biodiesel[a] | diesel |
|---|---|---|
| density/kg·m$^{-3}$ | 884.30 | 845.00 |
| pour point/K | 270.15 | |
| flash point/K | 437.15 | 341.15 |
| ester content/$w$ | >0.991 | |
| cold filter plugging point/K | 267.15 | |
| acidity number/mg KOH·g$^{-1}$ | 0.1186 | |
| kinematic viscosity, at 313.15 K/mm$^2$·s$^{-1}$ | 4.674 | 3.161 |
| free glycerin content | 0.004 | |
| total glycerin | 0.1540 | |
| distillation/% | | |
| 10 | | 488.75 |
| 50 | | 548.05 |
| 90 | | 612.25 |
| molar mass/gmol·g$^{-1}$ | 291.85 | 184.43 |

[a] This analysis respects the limits imposed by regulations from the U.S.A., European Union, and Brazil.

**Table 3. Density Experimental Data for Pseudopure Compounds, at Different Temperatures**

| | $\rho$/g·cm$^{-3}$ | | |
|---|---|---|---|
| $T$/K | soybean biodiesel | $n$-hexadecane | diesel |
| 293.15 | 0.8853 | 0.7743 | 0.8468 |
| 313.15 | 0.8707 | 0.7605 | 0.8332 |
| 333.15 | 0.8562 | 0.7466 | 0.8189 |
| 353.15 | 0.8416 | 0.7327 | 0.8047 |
| 373.15 | 0.8272 | 0.7186 | 0.7904 |

**Table 4. Density, $\rho$, and Excess Molar Volume, $V^E$, for the Pseudobinary Mixtures ($w_1/x_1$ Soybean Biodiesel + $n$-Hexadecane ($1 - w_1/x_1$)), at Different Temperatures**

| | $\rho$/g·cm$^{-3}$ | | | | |
|---|---|---|---|---|---|
| $w_1$ | $T$/K = 293.15 | $T$/K = 313.15 | $T$/K = 333.15 | $T$/K = 353.15 | $T$/K = 373.15 |
| 0.1099 | 0.7848 | 0.7709 | 0.757 | 0.7426 | 0.7287 |
| 0.2202 | 0.7961 | 0.7821 | 0.768 | 0.7536 | 0.7394 |
| 0.3268 | 0.8071 | 0.7932 | 0.7792 | 0.7646 | 0.7502 |
| 0.4319 | 0.8183 | 0.8042 | 0.7902 | 0.7758 | 0.7611 |
| 0.5309 | 0.8290 | 0.8150 | 0.8010 | 0.7864 | 0.7719 |
| 0.6315 | 0.8406 | 0.8264 | 0.8123 | 0.7980 | 0.7833 |
| 0.7248 | 0.8516 | 0.8374 | 0.8232 | 0.8088 | 0.7941 |
| 0.8208 | 0.8633 | 0.849 | 0.8347 | 0.8203 | 0.8056 |
| 0.9129 | 0.8746 | 0.8602 | 0.8458 | 0.8315 | 0.8167 |
| | $V^E$/cm$^3$·mol$^{-1}$ | | | | |
| $x_1$ | $T$/K = 293.15 | $T$/K = 313.15 | $T$/K = 333.15 | $T$/K = 353.15 | $T$/K = 373.15 |
| 0.087 | 0.1200 | 0.1278 | 0.1025 | 0.2817 | 0.1835 |
| 0.180 | 0.0694 | 0.0788 | 0.1029 | 0.2469 | 0.2580 |
| 0.274 | 0.1058 | 0.0437 | −0.0016 | 0.1886 | 0.2660 |
| 0.371 | 0.1237 | 0.1067 | 0.0371 | 0.1132 | 0.3009 |
| 0.468 | 0.1931 | 0.1089 | 0.0152 | 0.1441 | 0.2323 |
| 0.571 | 0.1042 | 0.0632 | −0.0158 | −0.0417 | 0.1131 |
| 0.671 | 0.0370 | −0.0347 | −0.0984 | −0.1120 | 0.0301 |
| 0.780 | −0.0645 | −0.1298 | −0.1778 | −0.2203 | −0.0905 |
| 0.891 | −0.0811 | −0.1356 | −0.1630 | −0.2702 | −0.1055 |

separated in a separating funnel. The ester layer was washed in three steps. The first step was accomplished with a twice washing procedure with pure water ($\varphi = 10$ %) to remove catalyst, soap, and glycerol excess. After that, it was washed once with chloridric acid (0.1 M HCl solution), $\varphi = 10$ %, to neutralize the medium. It should be noticed that after each washing procedure the dense phase was separated by settling. The conversion of oil into biodiesel using this technique was 98.1 ± 1.2 %. The chemical compositions of the oils as its equivalent fatty acid composition are presented in Table 1.

All biodiesel samples were characterized following laboratory procedures and standards indicated by the ANP,[3] before blends preparation. The physical parameters are listed in Table 2. All blends were prepared in the biodiesel mass fraction range between $w = (0.09$ and $0.9)$, at 298.15 K.

The methyl esters compositions were determined by gas chromatography with FID detector (GC-FID), using a Varian CP-3800 gas chromatograph system equipped with a flame ionization detector (FID; $T_{\text{detector}} = 523.15$ K) and automated split injector ($T_{\text{injection}} = 473.15$ K). The column was a CP WAX 52CB 30 m × 0.25 mm × 0.05 $\mu$m DB ($T_{\text{column}} = 483.15$ K).

$n$-Hexadecane was purchased from Merck with a stated minimum $w$ of 0.99, confirmed by chromatography analysis. No further purification of the product was carried out.

Diesel fuel was kindly furnished by Lubrificantes e Derivados de Petróleo do Nordeste, LUBNOR, Petrobras S/A. This sample was characterized by determining its viscosity, density, pour point, distillation temperatures, flash point, acid number, and calculated cetane index. Diesel fuel tested was distilled following the procedure established by ASTM D86.[8] The fuel properties of the diesel fuel are presented in Table 2.

***Apparatus and Procedure.*** The densities, $\rho$, of pseudopure liquids and their binary mixtures were determined using an Anton Paar SVM 3000 digital oscillation U-tube. The measuring cell contains a tube filled with sample, which rotates at constant speed. Binary mixtures were performed using an electronic balance (Technal Mark 210 A) accurate to 0.1 mg. The uncertainty of reported density data is estimated to be ± 0.0005 g·cm$^{-3}$. The temperature in the cell was regulated to ± 0.01 K. The uncertainty in mole fractions is estimated to be lower than ± 1.8·10$^{-3}$. Further details about technical procedures or manipulation could be found in earlier works.[9,10]

## Results and Discussion

Experimental values of the density of pseudopure and pseudobinary mixtures of soybean biodiesel + diesel and soybean biodiesel + $n$-hexadecane at $T = (293.15, 313.15, 333.15, 353.15$, and $373.15)$ K, at atmospheric pressure, and composition are shown in Tables 3, 4, and 5. Each measurement is the result of an average of (at least) three concurrent measurements. It can be observed that densities decrease with increasing temperatures for all blends and pseudopure compounds studied.

The estimated values of the parameters of the first-order polynomial equation (eq 1) are given in Table S1 (in the Supporting Information), where $A_0$ and $A_1$ are constants specific for a temperature.

$$\rho = A_0 + A_1 T \tag{1}$$

The excess molar volume on mixing values was calculated using eq 2 from experimental data

$$V^{E} = \sum_{i=1}^{n} x_i M_i (\rho^{-1} - \rho_i^{-1}) \tag{2}$$

where $\rho$ is the density on mixing. The $\rho_i$ is the density of pseudopure chemicals and $n$ is the number of components in the mixture. It should be noticed that the molar mass for diesel was obtained by VPO (vapor pressure osmometry), whereas for biodiesel it was by methyl esters content, Table 2.

The excess volumes have been fitted to the Redlich−Kister[11] polynomial equation given below

$$V^{E} = x_1(1 - x_1) \sum_{j=1}^{k} A_j (1 - 2x_1)^j \tag{3}$$

where $V^E$ is the excess molar volume, $x_1$ is the mole fraction, $A_j$ is a parameter, and $k$ is the degree of the polynomial equation. $A_j$ values were obtained by the method of least-squares using Marquardt algorithm to derive the binary coefficients. The corresponding standard deviations are given by

$$\sigma(V^{E}) = \sqrt{\frac{\sum V^{E}_{\text{exp}} - V^{E}_{\text{adj}}}{(n - p)}} \tag{4}$$

where subscript "adj" refers to adjusted data and $n$ and $p$ are the number of experimental points and number of parameters retained in the respective equation. The adjustable parameters, $A_k$, along with standard deviations $\sigma$ calculated using eq 4 for excess volume for the binary mixtures are listed in Tables S2 and S3 (in the Supporting Information).

The estimated values of excess mole volume, $V^E$, with molar fraction $x_1$ of soybean biodiesel are summarized in Tables 4 and 5 and displayed in Figure 1. For the mixtures of soybean biodiesel + diesel, the $V^E$ are positive over the entire composition range, with a maximum value of approximately 0.9 mol·cm$^{-3}$ at $x_1 = 0.45$ and $T = 373.15$ K. This mixture presents an expansive trend in terms of molecular interactions. This trend diminishes toward infinite dilution. Positive $V^E$ values can be attributed to the weak interactions between different molecules. Nevertheless, the $V^E$ curves for the mixtures of soybean biodiesel + $n$-hexadecane are sigmoidal and tend to change to negative values at higher molar fractions (around $x_1$ = 0.60 to 0.80). Negative $V^E$ arises from several contributions, which may involve specific interactions between unlike molecules in the mixture (at high content of soybean biodiesel in the mixture).

**Table 5. Density, $\rho$, Excess Molar Volume, $V^E$, for the Pseudobinary Mixtures ($w_1/x_1$ Soybean Biodiesel + Diesel Fuel ($1 - w_1/x_1$)), at Different Temperatures**

| | $\rho$/g·cm$^{-3}$ | | | | |
|---|---|---|---|---|---|
| $w_1$ | $T$/K = 293.15 | $T$/K = 313.15 | $T$/K = 333.15 | $T$/K = 353.15 | $T$/K = 373.15 |
| 0.1017 | 0.8497 | 0.8358 | 0.8218 | 0.8074 | 0.7934 |
| 0.2050 | 0.8539 | 0.8399 | 0.8257 | 0.8112 | 0.7970 |
| 0.3054 | 0.8578 | 0.8435 | 0.8293 | 0.8149 | 0.8005 |
| 0.4097 | 0.8618 | 0.8477 | 0.8355 | 0.8191 | 0.8047 |
| 0.5134 | 0.8656 | 0.8513 | 0.8370 | 0.8227 | 0.8082 |
| 0.6079 | 0.8694 | 0.8552 | 0.8409 | 0.8265 | 0.8122 |
| 0.7103 | 0.8737 | 0.8594 | 0.8450 | 0.8307 | 0.8158 |
| 0.8088 | 0.8777 | 0.8633 | 0.8490 | 0.8347 | 0.8200 |
| 0.9071 | 0.8816 | 0.8671 | 0.8527 | 0.8386 | 0.8240 |

| | $V^E$/cm$^3$·mol$^{-1}$ | | | | |
|---|---|---|---|---|---|
| $x_1$ | $T$/K = 293.15 | $T$/K = 313.15 | $T$/K = 333.15 | $T$/K = 353.15 | $T$/K = 373.15 |
| 0.081 | 0.4320 | 0.4972 | 0.4216 | 0.4816 | 0.4027 |
| 0.167 | 0.5121 | 0.5779 | 0.5537 | 0.6364 | 0.6188 |
| 0.254 | 0.6059 | 0.7341 | 0.7066 | 0.7512 | 0.7983 |
| 0.350 | 0.6826 | 0.7223 | 0.0476 | 0.7154 | 0.7579 |
| 0.450 | 0.7763 | 0.8506 | 0.8417 | 0.8288 | 0.9079 |
| 0.546 | 0.7222 | 0.7326 | 0.7120 | 0.7155 | 0.7151 |
| 0.655 | 0.5585 | 0.5638 | 0.5646 | 0.5110 | 0.7285 |
| 0.766 | 0.3973 | 0.3999 | 0.3511 | 0.2736 | 0.4075 |
| 0.883 | 0.2198 | 0.2203 | 0.1950 | 0.0156 | 0.1010 |

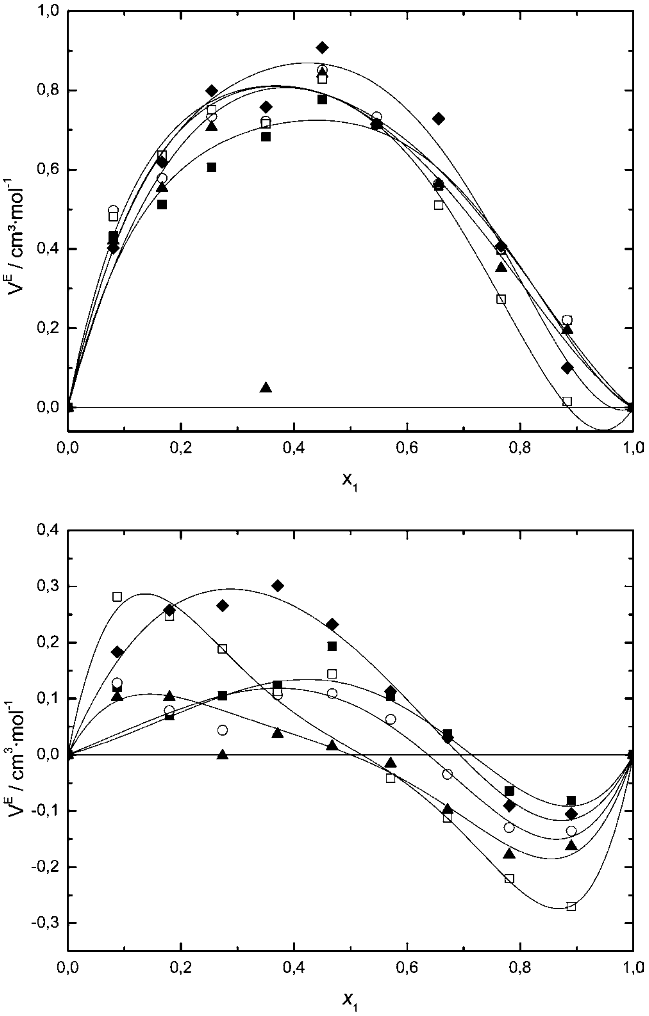

**Figure 1.** Temperature influence in excess molar volume for binary mixtures. Top: binary mixtures of $x_1$ soybean biodiesel + $(1 - x_1)$ diesel in the temperature range $T$ = (293.15 to 373.15) K. ■, 293.15 K; ○, 313.15 K; ▲, 333.15 K; □, 353.15 K; ♦, 373.15 K. Bottom: binary mixtures of $x_1$ soybean biodiesel + $(1 - x_1)$ $n$-hexadecane in the temperature range $T$ = (293.15 to 373.15) K. ■, 293.15 K; ○, 313.15 K; ▲, 333.15 K; □, 353.15 K; ♦, 373.15 K. All lines were obtained from Redlich−Kister polynomial fit.

It can be observed from Figure 1 that the effect of temperature on excess molar volume for these systems shows an erratic behavior. This phenomenon can be attributed to the multicomponent composition of soybean biodiesel and diesel, treated here as binaries systems only for purposing of parameters estimations. Therefore, it can be observed in Figure 1 that, in general, for the mixtures of soybean biodiesel + diesel there is an expansion due to increase in temperature. For the soybean biodiesel + n-hexadecane mixtures there is an expansion/contraction due to the positive/negative values for $V^E$ above $x_1 \geq 0.50$, as mentioned before.

The excess partial molar volumes $\bar{V}_i^E$ should be written as

$$\bar{V}_i^{E} = \bar{V}_i - V_i^{*} \tag{5}$$

where $V_i^{*}$ represent the molar volumes of the component 1 (soybean biodiesel) or 2 (*n*-hexadecane or diesel) and $\bar{V}_i$ can be determined from excess molar volumes using the relations[12]

$$\bar{V}_1^{E} = V^{E} + x_2\frac{dV^{E}}{dx_1} \tag{6}$$

and

$$\bar{V}_2^{E} = V^{E} + x_1\frac{dV^{E}}{dx_1} \tag{7}$$

The excess partial molar volume of soybean biodiesel ($\bar{V}_1^{E}$) in soybean biodiesel + *n*-hexadecane mixtures is positive at lower soybean biodiesel. Inversion of sign is temperature dependent and occurs at different compositions, $0.25 \leq x_1 \leq 0.55$. The ($\bar{V}_2^{E}$) values are negative after $x_1 \approx 0.7-0.9$, as indicated in Figures 2 and 3. Nevertheless, for soybean biodiesel + diesel mixtures, the excess partial molar volume of soybean biodiesel and diesel is positive, except at higher biodiesel concentration, around $x_1 \approx 0.9$ for $\bar{V}_1^{E}$.

The excess partial volume at infinite dilution can be determined by partial derivative of excess partial volume of the mixture at $x_i$ equal to zero, at operational conditions ($P$ and $T$). The influence of temperature in the partial excess molar volume at infinite dilution is graphically represented in Figure 4. It can be observed that the excess partial molar volumes at infinite dilution for soybean biodiesel in the soybean biodiesel + diesel mixtures; and, soybean biodiesel in the soybean biodiesel + *n*-hexadecane mixtures are positive for all temperatures studied in this work. Moreover, for *n*-hexadecane in the soybean biodiesel + *n*-hexadecane mixtures, the $\bar{V}_1^{E\infty}$ are negative for the entire temperature range studied. Otherwise, for diesel in the soybean biodiesel + diesel mixtures the $\bar{V}_1^{E\infty}$ are positive for temperatures below 333.15 K.

## Conclusions

Densities for binaries mixtures of soybean biodiesel + diesel, and soybean biodiesel + *n*-hexadecane have been experimentally determined at $T$ = (293.15 to 393.15) K and atmospheric pressure, over the entire mole fraction range. The excess molar volume was correlated using the Redlich−Kister equation. The excess volumes are positive for soybean biodiesel + diesel system. For soybean biodiesel + n-hexadecane mixtures, the $V^{E}$ is positive with an inversion of sign at higher molar fractions of soybean biodiesel. The effect of temperature in the excess partial volume at infinite dilution has been also calculated, showing different values (positive or negative) for the systems studied.

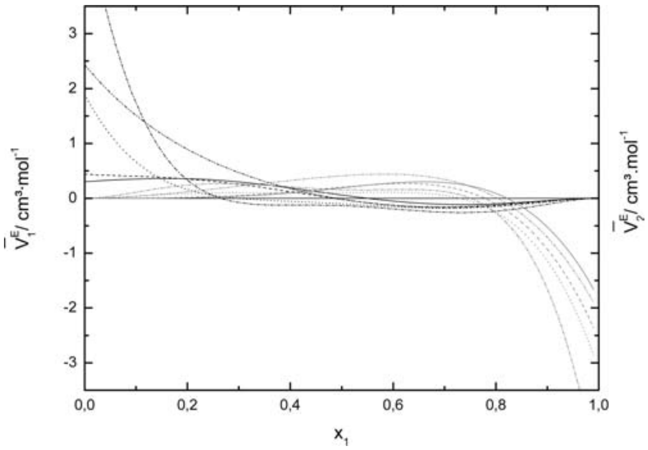

**Figure 2.** Partial excess volume for binary mixtures, cm$^3$·mol$^{-1}$, $\bar{V}_1^{E}$ (dark lines) and $\bar{V}_2^{E}$ (gray lines), of $x_1$ soybean biodiesel + $(1 - x_1)$ *n*-hexadecane at different temperatures (−, 293.15 K; −−, 313.15 K; ---, 333.15 K; −•−, 353.15 K; −••−, 373.15 K).

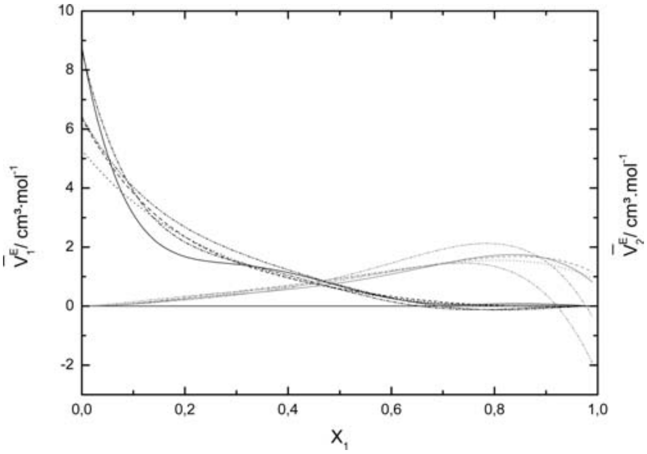

**Figure 3.** Partial excess volume for binary mixtures, cm$^3$·mol$^{-1}$, $\bar{V}_1^{E}$ (dark lines) and $\bar{V}_2^{E}$ (gray lines), of $x_1$ soybean biodiesel + $(1 - x_1)$ diesel at different temperatures (−, 293.15 K; −−, 313.15 K; ---, 333.15 K; −•−, 353.15 K; −••−, 373.15 K.).

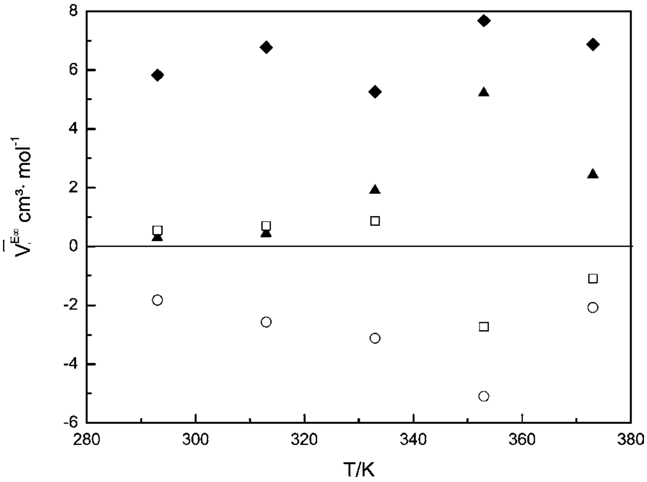

**Figure 4.** Influence of temperature in the partial excess molar volume at infinite dilution for each compound in binary mixtures systems. ▲, soybean biodiesel in the soybean biodiesel + *n*-hexadecane mixtures; ♦, soybean biodiesel in the soybean biodiesel + diesel mixtures; ○, *n*-hexadecane in the soybean biodiesel + *n*-hexadecane mixtures; □, diesel in the soybean biodiesel + diesel mixtures.

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### Supporting Information Available:

Tables detailing adjusted parameters for first-order polynomial fit for binary mixtures and standard deviation (Table A) and Redlich−Kister equation parameters for binary mixtures of soybean biodiesel + *n*-hexadecane (Table B) and soybean biodiesel + diesel

(Table C). This material is available free of charge via the Internet at http://pubs.acs.org.

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