Densities and Viscosities of Binary Mixtures of Babassu Biodiesel + Cotton Seed or Soybean Biodiesel at Different Temperatures

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Density ρ and viscosity η were measured for binary mixtures of cotton seed + babassu and soybean + babassu biodiesel over the composition range at several temperatures of (293.15, 313.15, 333.15, 353.15, and 373.15) K and atmospheric pressure. The viscosity deviation ($\Delta\eta$) was determined and fitted by a Redlich−Kister type function in terms of mass fraction (w). The Joback's method combined with Lee−Kesler and Rackett equations were used to calculate the densities of the binary mixtures of cotton seed + babassu and soybean + babassu biodiesel. The experimental and estimated density values gave almost identical values with a relative differences of less than 0.21 %. The Ceriani's group contribution method was used to predict the viscosity, with a maximum relative deviation of 31 %.

Introduction

The search for renewable fuels has been extensively studied by several fields of science. One of these renewable fuels, biodiesel, has been of great interest to researchers. The production of biodiesel brings many advantages because it is a biodegradable fuel and nontoxic and produces less particulate matter.[1] It can be produced from a variety of fats and vegetable oils, including oilseeds such as canola and soybean. In recent years, there has been a scientific effort to study the production of biodiesel from different raw materials. Several countries are already implementing a particular composition of biodiesel in diesel fuel. Nowadays, in Brazil, a large number of plants that are used in the production of biodiesel have been cultivated: babassu, cotton seed, soybean, sunflower, and others. Nevertheless, the most important raw material sources used for the production of biodiesel are soybean seeds (77.13 %), followed by bovine fat (17.07 %) and cotton seeds (4.62 %), data from the last Bulletin of the Brazilian Regulatory Agency (Agência Nacional do Petróleo, Gás Natural e Biocombustíveis - ANP).[2]

Biodiesel is a group of monoalkyl esters of long chain fatty acids derived from renewable feedstock, which has been increasingly used as a substitute of conventional diesel fuels. Several methods exist for producing biodiesel. The most significant is the transesterification method using a basic homogeneous catalyst. In this process, vegetable oil is added to a monohydroxyl alcohol (ethanol or methanol) in the presence of a basic homogeneous catalyst to form alcohol esters and glycerol.[1] Babassu, cotton seed, and soybean are examples of vegetable oils that can be used in the production of biodiesel by transesterification process. The ANP established a biodiesel content of B5 (in other words, $w = 0.05$ of biodiesel) in the diesel blends commercialized since January 2010.[3]

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Table 1. Fatty Acid Methyl Ester (FAME) Profile of Babassu, Soybean, and Cotton Seed Oils

	mass fraction (w) of FAME		
fatty acid	babassu	soybean	cotton seed
C10:0	0.0510	0.0000	0.0000
C12:0	0.2811	0.0000	0.0000
C14:0	0.2556	0.0000	0.0062
C16:0	0.1541	0.1129	0.2409
C18:0	0.0504	0.0396	0.0256
C18:1	0.2079	0.1998	0.1574
C18:2	0.0000	0.5839	0.5699
C18:3	0.0000	0.0586	0.0000
C22:0	0.0000	0.0052	0.0000

The thermodynamic properties of the biodiesel (pure and blended) have a great significance in the understanding of intermolecular interactions between different molecules of biodiesel produced by some vegetable oils. For this reason, a set of experiments have been carried out on the density and viscosity of different biodiesels.[4] This study reports the viscosity η and density ρ at T = (293.15, 313.15, 333.15, 353.15, and 373.15) K for binary mixtures of cotton seed + babassu and soybean + babassu biodiesel over the whole composition range. It should be noticed that biodiesels and diesel have a multicomponent composition. Nevertheless, it was treated here as binary systems only for the purpose of parameter estimations.

Experimental Section

Materials. Commercial edible grade soybean oil seed and babassu were obtained from Bunge Alimentos S.A. (Ipojuca/PE, Brazil) and Campestre Óleos Vegetais Ltd. (São Paulo/SP, Brazil), respectively. The chemical compositions of the oils as its equivalent fatty acid composition are presented in Table 1.

The methanol used in this work was obtained from Synth (Diadema/SP, Brazil) with a stated minimum mass fraction (w) purity of 0.99. Sodium hydroxide, used as a catalyst, was obtained from Grupo Química (Rio de Janeiro/RJ, Brazil), with a minimum purity of $w = 0.96$. It should be noticed that the

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Table 2. Physicochemical Properties of Pure Biodiesel Produced from Babassu, Soybean, and Cotton Oils, Together with the Biodiesel Regulations from USA, European Union, and Brazil

	biodiesel			limits		
physical property	babassu	soybean	cotton	USA ASTM D 6751	EU EN 14214	Brazil ANP 42
flash point/K	385.15	441.15	359.15	403.15 min	393.15 min	373.15 min
ester content/w	> 0.981	> 0.981	>0.981		96.5 min	96.5
cold filter plugging point/K	267.15	267.15	278.15		5 max (grade A) 0 max (grade B) −5 max (grade C) −10 max (grade D) −15 max (grade E) −20 max (grade F)	
acidity number/mg KOH·g^{-1}	0.0778	0.0953	0.1262	0.5 max	0.5 max	0.5 max
kinematic viscosity, at 313.15 K/mm^2·s^{-1}	3.180	4.674	3.990	1.9−6.0	3.5−5.0	3.00−6.00
density/kg·m^{-3}	874.4	884.8	880.3		860−900	850−950
free glycerin content/w	0	0.004	0	0.02 max	0.02 max	0.02 max
total glycerin/w	0.1399	0.1540	0.0345	0.24 max	0.25 max	0.38 max

samples were used as received, in other words, no purification procedure was used.

Transesterification Reaction. Commercial edible grade oils and a previously prepared solution of methanol and sodium hydroxide were fed into a glass vessel at a molar ratio of methanol to oil of 9.0, with a mass fraction (w) = 0.002 of sodium hydroxide. The amount of methanol, soybean oil, and sodium hydroxide used in the reactions were calculated, on the basis of their molar concentration, to give 250 mL of the reaction mixture. The vessel was placed inside an ultrasonic bath (Marconi model Unique USC 40 kHz; internal dimensions: 14 × 24 × 9 cm; volume: 2.7 L). The reaction was carried out at a temperature of 30 °C and atmospheric pressure. Low-frequency ultrasound (40 kHz) was applied at a 4870 W·m^{-2} intensity.[5] Temperature was controlled circulating running water through the ultrasonic bath. The reaction was carried out during 60 min. The conversion of oil into biodiesel using this technique was 98.1 ± 1.2 %. After the transesterification, the resulting glycerin was removed by decantation after 12 h, and the resulting ester phase was washed in three steps. The first step was accomplished with a twice-washing procedure with pure water (φ = 100 %) to remove catalyst, soap, and excess glycerol. After that, it was washed once with chloridric acid (0.1 M), φ_1 = 10 %, to neutralize the medium. It should be noticed that after each washing procedure the dense phase was separated by settling.[4] The procedure described above was used for the production of biodiesel from cotton seed and soybean. Nevertheless, biodiesel from babassu oil used the following conditions: a molar ratio of methanol to oil of 6.0, and sodium hydroxide, volume fraction (φ_1) = 0.05. The reaction conditions reported herein were found to be optimum in the production of biodiesel from soybean oil, cotton seed oil, and babassu oil.

Biodiesel Characterization. Biodiesel samples were characterized according to the procedures and standards indicated by the Brazilian Regulatory Agency (ANP).[3] The characteristics of the biodiesel produced from soybean, cotton seed, and babassu are presented in Table 2.

Gas chromatography was used for the determination of ester content using a Varian CP-3800 gas chromatograph system equipped with a FID (flame ionization detector). The detector temperature was set at 523.15 K, and the injector temperature was set at 473.15 K. The column used for separation was a CP WAX 52CB 30 m × 0.25 mm × 0.05 μm DB. The oven temperature was set at 483.15 K.

Biodiesel Blends. All blends were prepared in the mass fraction range between w = (0.09 and 0.9), at 298.15 K.

Viscosity and Density Measurements of the Blends. The measurements of viscosity and density were carried out in an Anton Paar SVM 3000 digital oscillation U-tube apparatus; a detailed description of the apparatus has been described elsewhere.[4,6] A volume of 5 mL of sample was injected into the equipment to measure the density (ρ) and dynamic viscosity (η) of pure biodiesel and their binary mixtures. Both measurements were carried out simultaneously. The viscosity was determined in a cell containing a tube filled with sample, which rotates at constant speed. The density was determined by a density cell using the oscillating U-tube principle. Density and viscosity measurements have an uncertainty of ± 0.0005 g·cm^{-3} and ± 0.35 % of the measured value within the works adjustment range, respectively. The temperature in the cell was regulated to ± 0.01 K. The uncertainty in mass fraction reported on this work was estimated to be lower than ± 1.8·10^{-3}. The binary mixtures were prepared by mass using an electronic balance (Tecnal model Mark 210A) accurate to 0.0001 g.

Mathematical Model. The method proposed by Ceriani et al.[7] was used for viscosity prediction. This method uses the concept of group contribution, in which a compound or a mixture of compounds is considered as a solution of groups and its properties are the sum of the contribution of each group. The model is described by eqs 1 to 5.

$$\ln(\eta_i) = \sum_k N_k\left(A_{1k} + \frac{B_{1k}}{T} - C_{1k}\ln T - D_{1k}T\right) + M\sum_k N_k \left(A_{2k} + \frac{B_{2k}}{T} - C_{2k}\ln T - D_{2k}T\right) + Q \quad (1)$$

$$Q = \xi_1 q + \xi_2 \quad (2)$$

$$q = \alpha + \frac{\beta}{T} - \gamma \ln T - \delta T \quad (3)$$

$$\xi_1 = f_0 + N_c f_1 \quad (4)$$

$$\xi_2 = s_0 + N_{cs} s_1 \quad (5)$$

where η_i is the dynamic viscosity; T is the temperature; N_k is the number of groups k in the molecule i; M is the component molecular weight that multiplies the "perturbation term"; A_{1k}, A_{2k}, B_{1k}, B_{2k}, C_{1k}, C_{2k}, D_{1k}, and D_{2k} are parameters obtained for the regression of experimental data by Ceriani et al.;[7] Q is a correction term; ξ_1 and ξ_2 are related to each class of compounds, where ξ_1 is a function of N_c, the total number of carbon atoms in the molecule, and ξ_2 describes the differences between the vapor pressure of isomer esters at the same temperature and is related to the number of carbons of the substitutive fraction N_{cs}, which, in the fatty esters, is mainly used to account for the effect of the alcoholic potion; α, β, γ, δ, f_0, f_1, s_0, and s_1 are the optimization parameters.

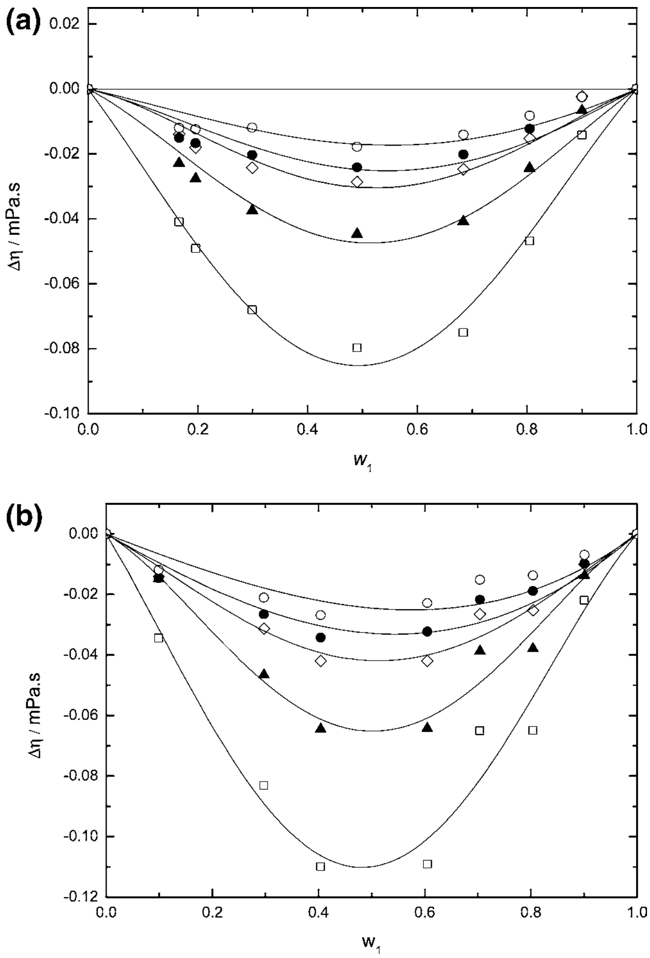

Figure 1. (a) Viscosity deviation of the binary mixtures of cotton seed + babassu biodiesel as a function of mass fraction of cotton seed biodiesel at different temperatures. (b) Viscosity deviation of the binary mixtures of soybean + babassu biodiesel as a function of mass fraction of soybean biodiesel at different temperatures. Lines calculated from Redlich−Kister polynomials. □, 293.15 K; ▲, 313.15 K; ◇, 333.15 K; ●, 353.15 K; ○, 373.15 K.

Table 3. Density, ρ, and Dynamic Viscosity, η, for Cotton Seed, Soybean, and Babassu Biodiesel at Temperatures Ranging from T = (293.15 to 373.15) K

biodiesel	T/K	ρ/g·cm^{-3}	η/mPa·s
cotton seed	293.15	0.8816	5.9440
	313.15	0.8672	3.6653
	333.15	0.8528	2.5005
	353.15	0.8382	1.8257
	373.15	0.8234	1.3931
soybean	293.15	0.8858	6.5754
	313.15	0.8714	4.0381
	333.15	0.8569	2.7449
	353.15	0.8424	1.9990
	373.15	0.8280	1.5218
babassu	293.15	0.8762	4.3823
	313.15	0.8608	2.7490
	333.15	0.8454	1.9007
	353.15	0.8303	1.4052
	373.15	0.8146	1.0786

The Rackett equation was used to estimate liquid density. It was considered that the liquid density is constant and also that it is not a function of temperature and pressure. For this, the Joback method of group contributions and the Lee−Kesler mixing rules were used to estimate the critical properties (T_c,

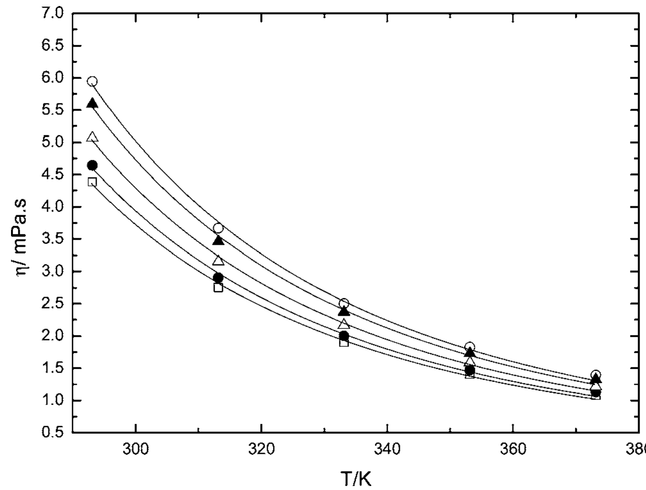

Figure 2. Temperature effect on the dynamic viscosity of biodiesel blends as a function of temperature and the mass fraction of biodiesel produced from cotton seed + babassu oils. □, pure babassu biodiesel; ●, w = 0.1960 of cotton seed biodiesel; △, w = 0.4900 of cotton seed biodiesel; ▲, w = 0.8050 of cotton seed biodiesel; ○, pure cotton seed biodiesel. Data fitted to the Andrade's type equation.

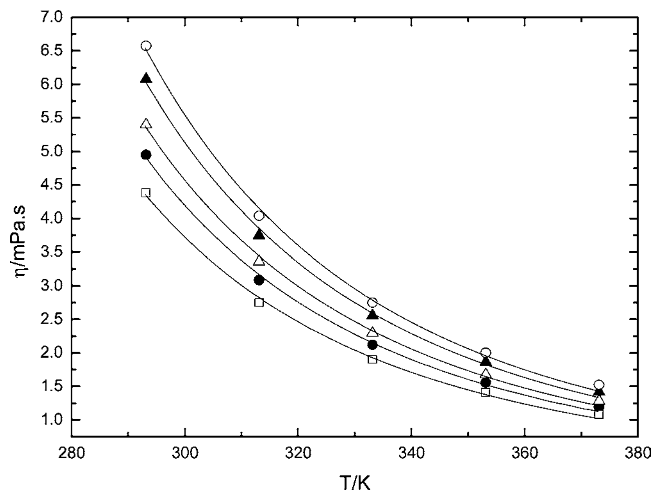

Figure 3. Temperature effect on dynamic viscosity of biodiesel blends as a function of temperature and the mass fraction of biodiesel produced from soybean + babassu oils. □, pure babassu biodiesel; ●, w = 0.2970 of soybean biodiesel; △, w = 0.5010 of soybean biodiesel; ▲, w = 0.8040 of soybean biodiesel; ○, pure soybean biodiesel. Data fitted to the Andrade's type equation.

P_c, and V_c) and normal boiling point (T_b) of biodiesel, as recommended by Knapp et al.[8]

Results and Discussion

Table 2 lists the basic physicochemical properties (flash point, ester content, cold filter plugging point, acidity number, dynamic viscosity, and density) of the pure biodiesel samples of babassu, cotton seed, and soybean. Viscosity values (η) and densities values (ρ) for pure biodiesel from soybean, cotton seed, and babassu oils at T = (293.15, 313.15, 333.15, 353.15, and 373.15) K are reported in Table 3.

The dependence of viscosity, viscosity deviation, and density on temperature is also illustrated in Figures 1 to 3 and Tables 4 and 5. The behavior of viscosity and density was respectively exponential and linear as expected, in view that the reduced temperatures of the experiments were lower than 0.7.[9] The biodiesel mixtures also did not exhibit any anomalous viscosity behavior.

Table 4. Dynamic Viscosity, η, Viscosity Deviation, $\Delta\eta$, and Density, ρ for the Binary Mixtures (w_1 Cotton Seed Biodiesel + (1 − w_1) Babassu Biodiesel) at Different Temperatures

w_1	T/K = 293.15	T/K = 313.15	T/K = 333.15	T/K = 353.15	T/K = 373.15
			η/mPa·s		
0.166	4.6006	2.8783	1.9863	1.4599	1.1188
0.196	4.6392	2.9010	2.0003	1.4709	1.1278
0.299	4.7812	2.9855	2.0558	1.5106	1.1607
0.490	5.0678	3.1533	2.1660	1.5871	1.2149
0.684	5.3754	3.3349	2.2863	1.6726	1.2796
0.805	5.5926	3.4623	2.3683	1.7314	1.3235
0.900	5.7737	3.5671	2.4383	1.7811	1.3592
			$\Delta\eta$/mPa·s		
0.166	−0.04094	−0.02281	−0.01397	−0.01510	−0.01201
0.196	−0.04919	−0.02759	−0.01796	−0.01672	−0.01244
0.299	−0.06805	−0.03747	−0.02424	−0.02033	−0.01194
0.490	−0.07973	−0.04469	−0.02860	−0.02415	−0.01780
0.684	−0.07510	−0.04085	−0.02466	−0.02022	−0.01412
0.805	−0.04687	−0.02432	−0.01524	−0.01230	−0.00827
0.900	−0.01413	−0.00657	−0.00222	−0.00255	−0.00245
			ρ/g·cm^{-3}		
0.166	0.8772	0.8621	0.8470	0.8318	0.8157
0.196	0.8774	0.8624	0.8471	0.8317	0.8160
0.299	0.8780	0.8629	0.8478	0.8327	0.8172
0.490	0.8789	0.8640	0.8489	0.8338	0.8186
0.684	0.8799	0.8653	0.8507	0.8357	0.8203
0.805	0.8809	0.8662	0.8516	0.8369	0.8219
0.900	0.8815	0.8668	0.8521	0.8373	0.8227

Table 5. Dynamic Viscosity, η, Viscosity Deviation, $\Delta\eta$, and Density, ρ for the Binary Mixtures (w_1 Soybean Biodiesel + (1 − w_1) Babassu Biodiesel) at Different Temperatures

w_1	T/K = 293.15	T/K = 313.15	T/K = 333.15	T/K = 353.15	T/K = 373.15
			η/mPa·s		
0.099	4.5650	2.8618	1.9702	1.4493	1.1106
0.297	4.9505	3.0852	2.1201	1.5549	1.1891
0.404	5.1583	3.2053	2.1998	1.6108	1.2307
0.501	5.3982	3.3626	2.2921	1.6776	1.2822
0.605	5.6001	3.4646	2.3695	1.7321	1.3239
0.804	6.0806	3.7475	2.5542	1.8637	1.4212
0.901	6.3363	3.8967	2.6513	1.9304	1.4710
			$\Delta\eta$/mPa·s		
0.099	−0.0344	−0.0148	−0.0141	−0.0147	−0.0119
0.297	−0.0832	−0.0467	−0.0313	−0.0267	−0.0211
0.404	−0.1100	−0.0645	−0.0420	−0.0343	−0.0270
0.501	−0.0828	−0.0322	−0.0315	−0.0251	−0.0184
0.605	−0.1090	−0.0643	−0.0419	−0.0323	−0.0228
0.804	−0.0650	−0.0379	−0.0252	−0.0189	−0.0137
0.901	−0.0220	−0.0138	−0.0100	−0.0098	−0.0069
			ρ/g·cm^{-3}		
0.099	0.8773	0.8620	0.8465	0.8308	0.8157
0.297	0.8793	0.8642	0.8490	0.8335	0.8186
0.404	0.8802	0.8653	0.8503	0.8350	0.8201
0.501	0.8814	0.8665	0.8516	0.8362	0.8215
0.605	0.8824	0.8676	0.8530	0.8381	0.8226
0.804	0.8844	0.8699	0.8552	0.8404	0.8255
0.901	0.8853	0.8709	0.8563	0.8418	0.8721

The density values of biodiesel decreased following the sequence: soybean > cotton seed > babassu. The same order was also observed for decreasing viscosity values: soybean > cotton seed > babassu. These results are influenced by the composition of fatty acid methyl esters (e.g., average carbon number, the position of the insaturation, and glycerin content) of the biodiesel.

Biodiesel from soybean oil is characterized by long chain methyl esters (C18) and insaturation resulting in a denser product. The density of cotton seed oil biodiesel is only slightly lower than soybean oil biodiesel given the higher amount of C16:0 instead of C18:1. Babassu oil biodiesel is a nonconventional biodiesel that is characterized by short methyl ester chains, mostly C12:0 and C14:0, which results in a less dense biodiesel. The lower total glycerin content of babassu oil biodiesel also contributes to the lower density of this biodiesel.

Chain length has a greater effect on viscosity than in density. A longer chain length results usually in higher viscosity. As such, the viscosity observed for soybean oil biodiesel was much higher than for babassu oil biodiesel. Lower temperatures also showed to pronounce the differences in viscosity caused by longer chain lengths.

The parameters of the first-order polynomial equation for density prediction (eq 6) are presented in Table A (in the Supporting Information, SI). An excellent agreement between the measured and the estimated values was obtained. A maximum standard deviation of 0.319 was obtained for all studied blends.

$$\rho = A_0 + A_1 T \qquad (6)$$

The Andrade's equation for dynamic viscosity can be used when the ratio of temperature to the liquid critical temperature

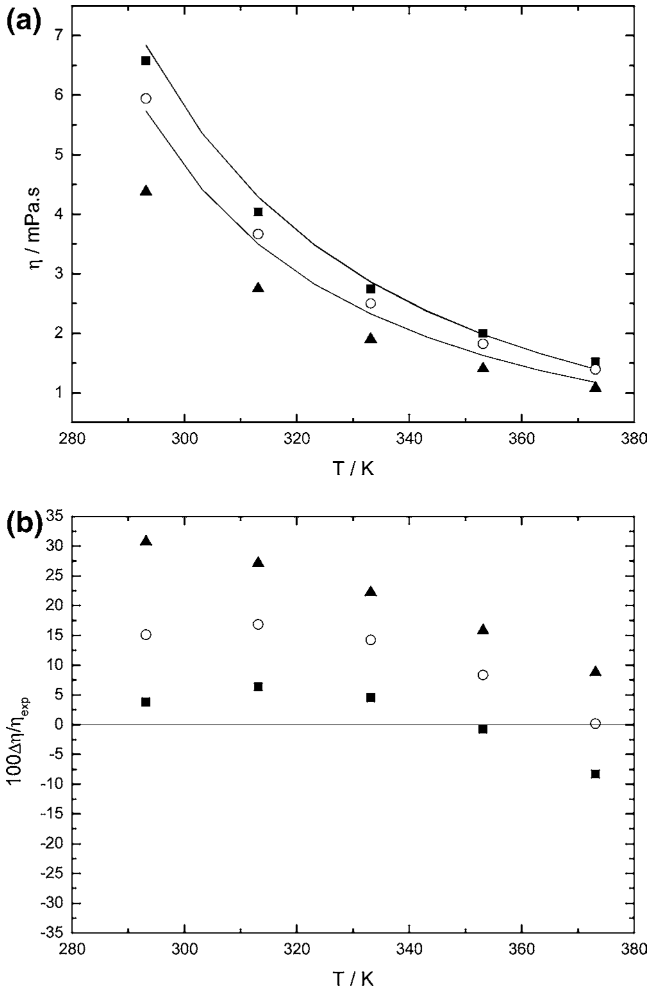

Figure 4. (a) Viscosity of the pure soybean biodiesel, ■; cotton seed biodiesel, ▲; and babassu biodiesel, ○. Lines calculated from group contribution method for the prediction of viscosity of fatty compounds (Ceriani et al., 2008[7]); (b) Deviation between experimental and predicted viscosity using Ceriani's method for soybean biodiesel, ■; cotton seed biodiesel, ▲; and babassu biodiesel, ○.

Table 6. Critical Properties of the Mixtures of Fatty Acid Methyl Esters Estimated Using Lee−Kesler Mixing Rules

	T_{cm}	P_{cm}	V_{cm}
biodiesel	K	bar	$cm^3 \cdot mol^{-1}$
babassu	722.30	13.9	924.436
soybean	759.46	12.1	1067.55
cotton seed	756.31	12.2	1058.05

Table 7. Rackett Compressibility Factor (Z_{RA}) for Methyl Esters Estimated Using the Spencer and Danner Method

biodiesel	Z_{RA}
babassu	0.219
soybean	0.219
cotton seed	0.221

(T_r) is less than 0.7. A modified form of the Andrade's equation proposed by Reid et al.[9] (eq 7) was used to predict the dynamic viscosity of the biodiesel mixtures.

$$\ln(\eta) = A_0 + \frac{A_1}{T} \qquad (7)$$

Table B (in the SI) presents the results of the regressions obtained by fitting eq 7 to the experimental data. A maximum standard deviation of 0.323 was obtained for the regressions, which indicate the equation provided a good fit and representation of the data.

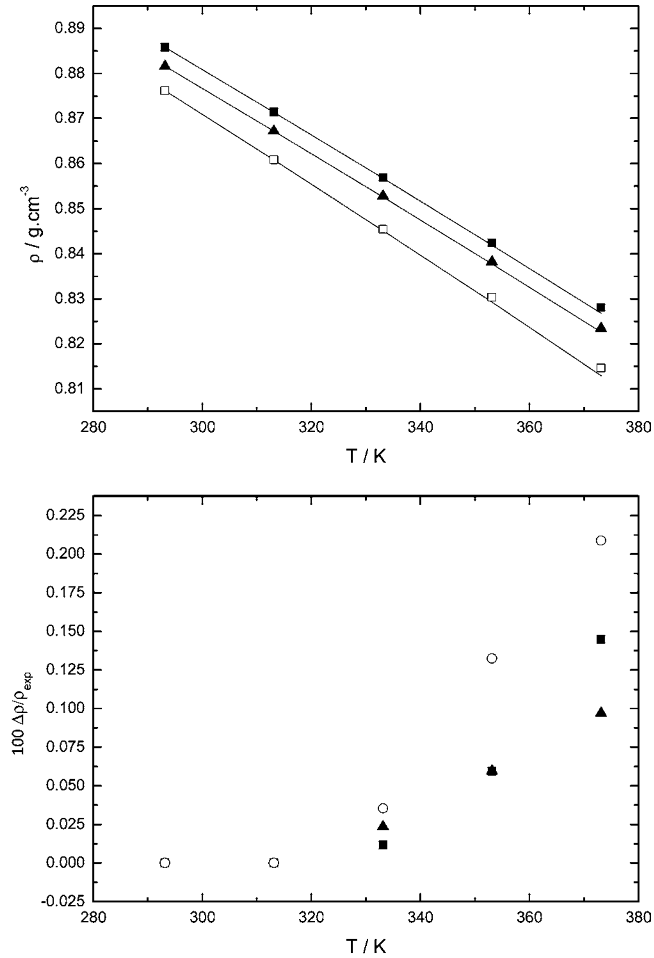

Figure 5. Top: density of the pure soybean biodiesel, ■; cotton seed biodiesel, ▲; and babassu biodiesel, ○. Bottom: prediction error for the Rackett equation for pure soybean biodiesel, ■; cotton seed biodiesel, ▲; and babassu biodiesel, ○.

Both biodiesel mixture systems, cotton seed + babassu and soybean + babassu, showed that the viscosity deviation decreased with increasing temperature. It can be observed that values for viscosity deviation are slightly negative for all temperatures and systems studied in this work and decrease with increasing temperature.

Viscosity deviation values were correlated using the Redlich−Kister polynomial equation[10] (eq 8).

$$\Delta\eta = w_1(1 - w_1)\sum_{j=1}^{k} A_j(1 - 2w_1)^j \qquad (8)$$

where w_1 is the mass fraction, k is the degree of the polynomial expansion, A_j is a parameter, and $\Delta\eta$ is the viscosity deviation. The viscosity deviation for both biodiesel mixtures are presented in Table C (in the SI).

Table D (in the SI) lists the group contribution constants for the prediction of viscosity of fatty methyl esters propose by Ceriani et al. (2008).[7] Figure 4 shows deviations of results predicted by using Ceriani's method to our dynamic viscosity data for fatty acid methyl esters. The figure illustrates that deviations are positive for all biodiesel studied, except for soybean biodiesel at temperatures above 353.15 K, with a maximum relative deviation of 30.7 % for cotton seed biodiesel at 293.15 K. It is important to mention that similar results were obtained in our previous work.[4]

Table E (in the SI) shows critical parameters and normal boiling points of the fatty methyl esters obtained using Joback's method.

Table 6 shows critical properties of the mixtures of fatty acid methyl esters estimated using the Lee−Kesler mixing rules. In Table 7, the values of the Rackett compressibility factor (Z_{RA}) for methyl esters estimated using the Spencer and Danner method is shown.[9]

Figure 5 depicts measured values of density for the pure biodiesel compared with those predicted by the Rackett equation. It can be observed that the maximum relative deviation is 0.21 % for babassu biodiesel at 373.15 K.

Conclusions

The density and viscosity of binary blends of cotton seed + babassu and soybean + babassu biodiesel were determined at temperatures ranging from T = (293.15 to 373.15) K. Density and viscosity data were correlated with the first-order polynomial equation and modified Andrade's equation, respectively, and presented a good agreement with the data. The viscosity deviation was calculated using experimental data and fitted using the Redlich−Kister polynomial equation. All systems measured exhibited slightly negative viscosity deviation values, which decreased with an increase in temperature. The experimental and estimated density values gave almost identical values with relative differences less than 0.21 %. The Ceriani's group contribution model was used to predict the viscosity with a maximum relative deviation of 31 %.

Supporting Information Available:

Estimated parameters for density, viscosity, and viscosity deviation of the binary mixtures (cotton seed + babassu and soybean + babassu biodiesel) are shown in Tables A to C. Table D illustrates the constants of the groupr contribution method proposed by Ceriani et al.[7] In addition, Table E lists critical properties and normal boiling point of the fatty acid methyl esters obtained using Joback's method. This material is available free of charge via the Internet at http://pubs.acs.org.

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