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Excess Volumes and Deviations of Viscosities of Binary Blends of Sunflower Biodiesel + Diesel and Fish Oil Biodiesel + Diesel at Various Temperatures

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Supporting Information

ABSTRACT: This paper presents the values of viscosity (η) and density (ρ) of binary blends of sunflower biodiesel + diesel and of fish oil biodiesel + diesel over the entire range of proportion at several temperatures. Temperatures of (293.15, 313.15, 333.15, 353.15, and 373.15) K at atmospheric pressure were studied. Excess volumes (V^E) and deviations of viscosities ($\Delta\eta$) have been calculated from the experimental data. These thermodynamic properties are essential in the understanding of intermolecular interactions between different molecules of biodiesel, and it is important in the design of several equipments used in the biodiesel industry. The excess viscosity has been fitted using the Redlich−Kister polynomial equation. A modified Rackett's equation was used to estimate the density. The viscosity for the biodiesels was estimated using three models: Thomas, Orrick−Erbar, and modified Sastri−Rao. The critical properties were estimated by the Rackett's equation, together with the Marrero−Gani method of group contribution.

INTRODUCTION

Biodiesel has become one of the most important alternatives to substitute energy provided by fossil sources. Biodiesel is nontoxic and biodegradable and produces less particular matter. The production of biodiesel has many economic and environmental advantages.[1,2]

The main compounds of biodiesel are groups of mono esters of long chain derived from a renewable source. There are many methods that can be used to produce biodiesel. The most important method uses homogeneous catalysis to promote the transesterification reaction. In this method, vegetable oil and mono hydroxyl alcohol (ethanol or methanol) are mixed in the presence of a catalyst, producing ester-rich and glycerol-rich phases. Biodiesel blends are used worldwide as fuel to vehicles.

Biodiesel can be produced from a variety number of vegetable oils and animal fat, for example, soybean, sunflower, oil fish, and animal fat. Sunflower oil is mainly used as food grade oil, but it is also important in biodiesel production because it may be cultivated between harvests, besides having large oil content ($w = 0.30$ to 0.50). Fish oil is produced from the viscera of the fish, which is a waste from the fish industry.

Biodiesel is sold blended from different resource materials. This biodiesel is added to diesel, in a final blend known as B5, B8, B20, and B100, indicating the volume fraction of biodiesel component in the blend with petro diesel.[2]

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 and in the project of several equipments of industry, and it has been recently object of several studies.[3−7] The determination of thermodynamics properties of biodiesel + biodiesel and biodiesel + diesel blends is part of a continuing study in our laboratory to provide data for the characterization of the molecular interactions for those systems.[8−10] This paper reports the viscosity (η) and density (ρ) at T = (293.15, 313.15, 333.15, 353.15, and 373.15) K for binary blends of sunflower biodiesel + diesel and fish oil biodiesel + diesel over the whole proportion range. From these experimental data excess volumes (V^E) and deviations of viscosities ($\Delta\eta$) have been calculated.

EXPERIMENTAL SECTION

Materials. Sunflower seed oil and fish oil were obtained respectively from Cargill Agrícola S.A. (Marinque, Brazil) and NUTEC (Fundação Núcleo de Tecnologia Industrial do Ceará, Fortaleza, Brazil). The diesel sample was kindly supplied by LUBNOR (Petrobras/LUBNOR, Lubrificantes e Derivados de Petróleo do Nordeste, Fortaleza, Brazil). Methanol 99 % was supplied by Synth (Diadema, Brazil). Sodium hydroxide, used as a catalyst, was obtained from Grupo Química (Rio de Janeiro, Brazil). Table 1 shows the fatty acid methyl ester (FAME) composition of methyl esters produced from fish and sunflower oils. Physicochemical properties of diesel fuel used in this work are presented in Table S1, in the Supporting Information (SI).

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Table 1. Fatty Acid Methyl Ester (FAME) Profile of Sunflower and Fish Oils

fatty acid	mass fraction (w) of FAME	
	fish	sunflower
C14:0	0.0376	-
C16:0	0.0566	0.0710
C16:1	0.2809	-
C18:0	0.0746	0.0480
C18:1	0.4229	0.2260
C18:2	0.1274	0.6550

Table 2. Densities (ρ) and Viscosities (η) for Diesel, Fish Oil, and Sunflower Biodiesel at Temperatures Ranging from T = (293.15 to 373.15) K

	T	ρ	η
biodiesel	K	$g\cdot cm^{-3}$	mPa·s
diesel	293.15	0.8270	3.8433
	313.15	0.8132	2.3898
	333.15	0.7990	1.6445
	353.15	0.7845	1.2074
	373.15	0.7708	0.9207
fish oil	293.15	0.8776	6.2801
	313.15	0.8632	3.8107
	333.15	0.8488	2.5688
	353.15	0.8341	1.8580
	373.15	0.8191	1.4044
sunflower	293.15	0.8830	6.0298
	313.15	0.8683	3.7244
	333.15	0.8537	2.5439
	353.15	0.8389	1.8558
	373.15	0.8247	1.4128

Transesterification Reaction. The fish oil or the sunflower seed oil was fed with methanol and sodium hydroxide into a glass reactor (500 mL), at a molar ratio of 9:1 (methanol/oil). Sodium hydroxide (with a mass fraction of $w = 0.002$) was used as a catalyst for the transesterification reaction. The reaction was carried out with the vessel placed inside an ultrasonic bath (Unique model USC 40 kHz; internal dimensions: 14 × 24 × 9 cm; volume: 2.7 L). The temperature in the bath was 303.15 K at atmospheric pressure. Low frequency ultrasound (40 kHz) was applied at a 4870 $W\cdot m^{-2}$ intensity for 60 min. The reaction reached a conversion into biodiesel of 98.1 %. After the transesterification, the resulting glycerin was removed by settling after 12 h, and the resulting ester phase was washed in three steps, as described elsewhere.[8−11] The biodiesel + diesel blends were prepared at 298.15 K, in the mass fraction range between w = (0.09 and 0.9).

Biodiesel Characterization. Brazilian Regulatory Agency (ANP) procedure has been used for the characterization of the biodiesel.[12] Gas chromatography system, coupled with an FID (flame ionization detector), has been used for the ester content analysis, by using a Varian CP-3800. The detector temperature was set at 523.15 K, and the injector temperature was set at 473.15 K. The separation column was CP WAX 52CB 30 m × 0.25 mm × 0.05 μm DB. The temperature was set at 483.15 K.

Table 3. Dynamic Viscosity, η (mPa·s), Viscosity Deviation, $\Delta\eta$ (mPa·s), and Density, ρ ($g\cdot cm^{-3}$), for the Blends of (w_1 Sunflower Oil Biodiesel + (1 − w_1) Diesel) 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		
0.105	3.9858	2.4786	1.7055	1.2493	0.9437
0.207	4.1272	2.5714	1.7720	1.3010	0.9968
0.314	4.3296	2.6948	1.8537	1.3608	1.0388
0.419	4.5054	2.8054	1.9297	1.4154	1.0787
0.518	4.7182	2.9382	2.0218	1.4832	1.1336
0.616	4.9981	3.1035	2.1126	1.5492	1.1945
0.716	5.2028	3.2274	2.2122	1.6189	1.2347
0.812	5.4941	3.4007	2.3304	1.7117	1.3074
0.903	5.7824	3.5751	2.4418	1.7850	1.3629
			$\Delta\eta$/mPa·s		
0.105	−0.0871	−0.0513	−0.0334	−0.0262	−0.0287
0.207	−0.1687	−0.0947	−0.0587	−0.0406	−0.0258
0.314	−0.2003	−0.1141	−0.0732	−0.0502	−0.0364
0.419	−0.2540	−0.1436	−0.0916	−0.0637	−0.0482
0.518	−0.2577	−0.1429	−0.8859	−0.0601	−0.0420
0.616	−0.1921	−0.1084	−0.0859	−0.0576	−0.0293
0.716	−0.2060	−0.1180	−0.0763	−0.0528	−0.0383
0.812	−0.1246	−0.0728	−0.0444	−0.0222	−0.0129
0.903	−0.0353	−0.0198	−0.0149	−0.0079	−0.0022
			$\rho/g\cdot cm^{-3}$		
0.105	0.8322	0.8184	0.8044	0.7901	0.7758
0.207	0.8376	0.8237	0.8097	0.7954	0.7809
0.314	0.8435	0.8296	0.8152	0.8008	0.7866
0.419	0.8490	0.8352	0.8207	0.8063	0.7920
0.518	0.8545	0.8406	0.8265	0.8118	0.7973
0.616	0.8601	0.8460	0.8316	0.8173	0.8027
0.716	0.8658	0.8516	0.8373	0.8225	0.8083
0.812	0.8722	0.8578	0.8432	0.8286	0.8140
0.903	0.8773	0.8631	0.8487	0.8341	0.8195

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, following the ASTM D7042-04 procedure. For this, a sample of 5 mL of was injected into a cell of measurements. It is important to mentioned that both measurements were carried out simultaneously. The uncertainty of the measurements is estimated to be 0.0005 $g\cdot cm^{-3}$ and 0.35 % for density and viscosity measurements, 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 $1.8\cdot 10^{-3}$. An electronic balance (Tecnal model Mark 210A) (accurate to 0.0001 g) has been used for the preparation of all blends.

Property Predictions. Three models (Thomas, Orrick−Erbar, and modified Sastri−Rao, as described by Anand et al.[13]) have been chosen in this work to predict the viscosity of the biodiesel. These models have been selected due to its simplicity of using and theoretical base−group contribution method. For instance, only critical properties are needed.

Critical temperature (T_c), critical pressure (P_c), critical volume (V_c), and normal boiling temperature (T_{nb}) were estimated using Marrero−Gani method.[14] This method presents better prediction

Table 4. Dynamic Viscosity, η (mPa·s), Viscosity Deviation, $\Delta\eta$ (mPa·s), and Density, ρ (g·cm^{-3}), for the Blends of (w_1 Fish Oil Biodiesel + (1 − w_1) Diesel) 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		
0.103	4.0000	2.4847	1.7077	1.2541	0.9572
0.207	4.1667	2.5854	1.7768	1.3032	0.9964
0.314	4.3883	2.7192	1.8668	1.3723	1.0493
0.419	4.6096	2.8469	1.9490	1.4255	1.0896
0.518	4.8809	3.0038	2.0491	1.4975	1.1406
0.616	5.1396	3.1516	2.1455	1.5638	1.1903
0.716	5.4564	3.3343	2.2618	1.6443	1.2496
0.812	5.7658	3.5108	2.3757	1.7234	1.3074
0.903	6.1640	3.7310	2.5130	1.8159	1.3743
			$\Delta\eta$/mPa·s		
0.103	−0.0844	−0.0452	−0.0248	−0.0168	−0.0109
0.207	−0.1810	−0.0985	−0.0590	−0.0389	−0.0244
0.314	−0.2153	−0.1139	−0.0661	−0.0381	−0.0223
0.419	−0.2425	−0.1312	−0.0782	−0.0512	−0.0313
0.518	−0.2149	−0.1163	−0.0705	−0.0443	−0.0287
0.616	−0.1950	−0.1078	−0.0647	−0.0418	−0.0264
0.716	−0.1219	−0.0672	−0.0408	−0.0263	−0.0155
0.812	−0.0586	−0.0342	−0.0203	−0.0129	−0.0065
0.903	0.1154	0.0553	0.0320	0.0197	0.0158
			ρ/g·cm^{-3}		
0.103	0.8317	0.8177	0.8037	0.7893	0.7755
0.207	0.8367	0.8228	0.8086	0.7942	0.7800
0.314	0.8416	0.8276	0.8136	0.7991	0.7848
0.419	0.8468	0.8327	0.8186	0.8042	0.7897
0.518	0.8523	0.8381	0.8235	0.8089	0.7948
0.616	0.8571	0.8430	0.8288	0.8142	0.7995
0.716	0.8628	0.8485	0.8338	0.8194	0.8049
0.812	0.8683	0.8538	0.8393	0.8248	0.8099
0.903	0.8737	0.8593	0.8448	0.8301	0.8155

accuracy when compared with the method proposed by Joback−Reid.[15] The acentric factor (ω) was estimated by using the Constantinou−Gani method[16] coupled with Lee−Kesler mixing rules, as recommended by Knapp et al.[17] The Rackett equation modified by Spencer−Danner was used to estimate liquid density (eq 1).

$$\rho = \frac{\rho_R}{Z_{RA}^{\varphi}} \tag{1}$$

where

$$\varphi = \left(1 - \frac{T}{T_c}\right)^{2/7} - \left(1 - \frac{T_R}{T_c}\right)^{2/7} \tag{2}$$

ρ_R and T_R are the reference density and temperature, respectively, and Z_{RA} is the Rackett compressibility factor. The reference temperature used was T_R = 333.15 K, and the density reference (ρ_R) was the density evaluated at 333.15 K.

■ RESULTS AND DISCUSSION

The viscosity and density of pure biodiesel and diesel were measured at different temperatures ((293.15 to 373.15) K in steps of 20 K). Also, the density and viscosity of biodiesel + diesel blends of nine different proportions were measured. Table 2 presents the values of density and viscosity of pure biodiesel and diesel at different temperatures.

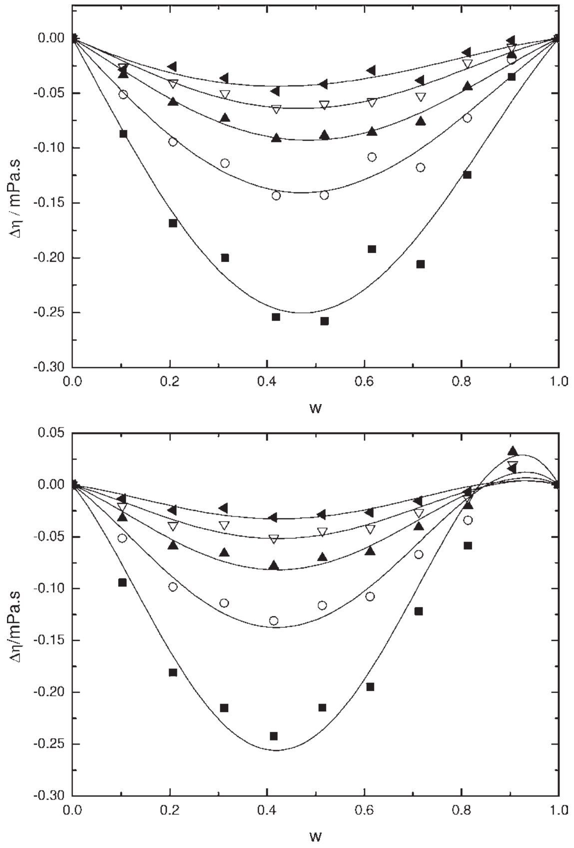

Figure 1. (a) Viscosity deviation of the blends as a function of mass fraction of sunflower biodiesel. (b) Deviation of dynamic viscosity as a function of mass fraction of fish oil biodiesel. Experimental results: w sunflower/fish oil biodiesel + (1 − w) diesel. Lines are calculated from Redlich−Kister polynomials. ■, 293.15 K; ○, 313.15 K; ▲, 333.15 K; ▽, 353.15 K; left-pointing triangle, 373.15 K.

The dependence of viscosity, viscosity deviation, and density on temperature for the systems biodiesel + diesel are presented in Tables 3 and 4 and illustrated in Figures 1 to 3.

The density, as expected, showed a linear behavior with temperature. The density data were fitted by the first-order polynomial equation (eq 3). The estimated parameters are shown in the Table S2 (in the SI). A good agreement was obtained by this equation between the experimental and the estimated values, with a maximum standard deviation of 0.0029, for all of the blends.

$$\rho = A_0 + A_1 T \tag{3}$$

The kinematic viscosity was fitted to Andrade's equation, having an exponential behavior. The equation that was used was a modified form of Andrade's equation proposed by Reid et al.,[18] see (eq 4)

$$\ln(\eta) = A + \frac{B}{T} \tag{4}$$

Table S3 (in the SI) shows the results of the regressions using eq 4 (where A and B are constants specific for a temperature). The maximum standard deviation of 1.4088 provides a good fit and representation of the data.

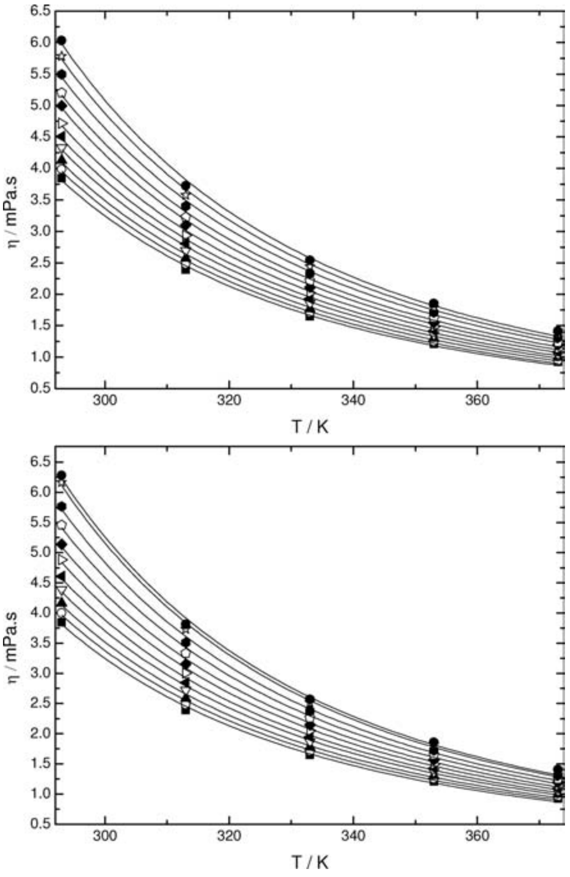

Figure 2. Temperature effect on dynamic viscosity for (a) blends of sunflower biodiesel + diesel and (b) blends of fish oil biodiesel + diesel. Experimental results: all ranges with (●) pure sunflower/fish oil biodiesel and (■) pure diesel; temperature ranging from (293.15 to 373.15) K. Data are fitted to an Andrade's type equation.

The values of viscosity deviation were correlated by the polynomial series of Redlich–Kister,[19] eq 5:

$$\Delta\eta = w_1(1 - w_1)\sum_{j=1}^{k} A_j(1 - 2w_1)^j \qquad (5)$$

where w_1 is the mole fraction, k is the degree of the polynomial expansion, A_j is a parameter, and $\Delta\eta$ is the viscosity deviation.

A_j values were obtained using a nonlinear least-squares fitting procedure. The corresponding standard deviations were given by eq 6:

$$\sigma(V^{\mathrm{E}}) = \sqrt{\frac{\sum V^{\mathrm{E}}_{\mathrm{exp}} - V^{\mathrm{E}}_{\mathrm{adj}}}{(n - p)}} \qquad (6)$$

where the subscript exp refers to experimental, adj to the adjusted values, n is the number of experimental points, and p is the number or parameters retained in the respective equation.

The adjustable parameters, A_j, and standard deviation, σ, calculated using eq 4 for viscosity deviations are listed in Table S4, in the SI. The results were based on a third-degree polynomial expansion, $k = 3$.

The values of excess volume V^{E} were fitted by eq 7. The excess volume data are shown in Tables 5 and 6 and Figure 4. For both systems (sunflower biodiesel + diesel and of fish oil biodiesel + diesel) the V^{E} are positive over the entire proportion range, with a maximum value around 0.5 mol·cm^{-3}. These blends present an expansive trend in terms of molecular interactions. This trend diminishes toward infinite dilution. It should be noticed that positive V^{E} values can be attributed to the weak interactions between different molecules.

$$V^{\mathrm{E}} = \sum_{i=1}^{n} w_i M_i(\rho^{-1} - \rho_i^{-1}) \qquad (7)$$

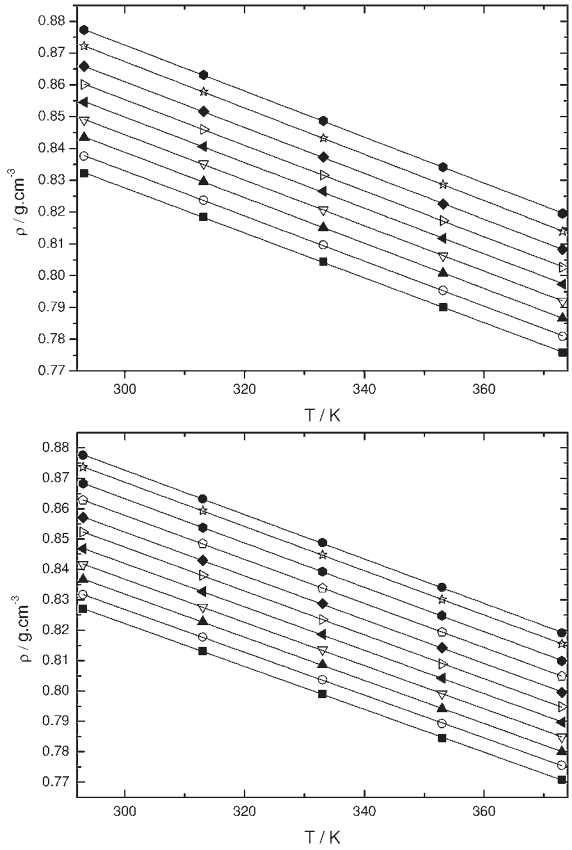

Figure 3. Temperature effect on density for (a) blends of sunflower biodiesel + diesel and (b) blends of fish oil biodiesel + diesel. Experimental results: all ranges with (●) pure sunflower/fish oil biodiesel and (■) pure diesel. Temperature ranging from (293.15 to 373.15) K. Data are fitted to first-order polynomial equation.

Table 5. Excess Molar Volume (V^{E}/cm^3·mol^{-1}) for the Blends of (w_1 Sunflower Biodiesel + (1 − w_1) Diesel) 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
0.000	0.0000	0.0000	0.0000	0.0000	0.0000
0.105	0.7835	0.7730	0.7219	0.6713	0.8649
0.207	1.3167	1.3185	1.2672	1.2203	1.4844
0.314	1.6660	1.6436	1.7128	1.6992	1.8622
0.419	1.9587	1.8791	1.9749	1.9564	2.1472
0.518	2.0143	1.9363	1.8844	1.9558	2.2054
0.616	1.9005	1.8549	1.8897	1.8059	2.0756
0.716	1.6682	1.6194	1.5970	1.6762	1.7753
0.812	1.0127	0.9854	1.0387	1.0136	1.2334
0.903	0.6185	0.4809	0.4321	0.3768	0.5644
1.000	0.0000	0.0000	0.0000	0.0000	0.0000

Table 6. Excess Molar Volume (V^E/cm$^3\cdot$mol^{-1}) for the Binary Blends of (w_1 Fish Oil Biodiesel + (1 − w_1) Diesel) 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
0.000	0.0000	0.0000	0.0000	0.0000	0.0000
0.103	0.7180	0.7752	0.7324	0.7177	0.7167
0.207	1.2401	1.2618	1.2868	1.2810	1.3772
0.312	1.6781	1.7220	1.6885	1.7202	1.8142
0.414	1.8408	1.9009	1.8963	1.8915	2.0141
0.514	1.7441	1.8144	1.9716	2.0321	1.9636
0.612	1.7210	1.7374	1.7450	1.7902	1.8945
0.712	1.3064	1.3605	1.5318	1.4863	1.4568
0.813	0.8479	0.9423	1.0212	0.9882	1.0574
0.905	0.1811	0.2019	0.2517	0.2650	0.1458
1.000	0.0000	0.0000	0.0000	0.0000	0.0000

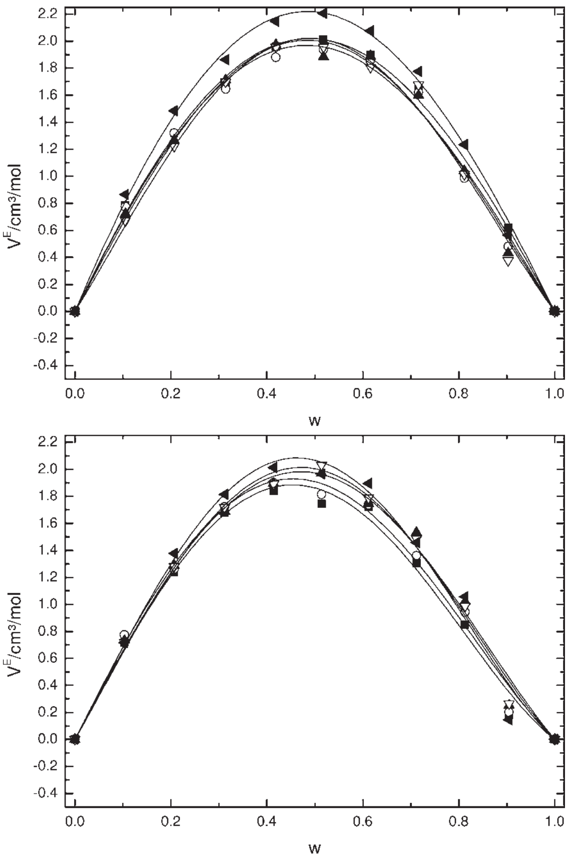

Figure 4. Excess volume of the binary blends vs mass fraction of (top) sunflower biodiesel and (bottom) fish oil biodiesel. ■, at 293.15 K; ○, at 313.15 K; ▲ at 333.15 K; ▽, at 353.15 K; left-pointing triangle, at 373.15 K. Experimental results: w sunflower/fish oil biodiesel + (1 − w) diesel. Lines are calculated from Redlich−Kister polynomials.

Tables 7 and 8 present the comparison between experimental and estimated density and viscosity for sunflower oil biodiesel and fish oil biodiesel, along with the SD (standard deviation), AAE (absolute average error), and ARE (average relative error). The critical properties estimated by the Marrero−Gani method (T_c, P_c, V_c) and the Lee−Kesler method (acentric factor) and the Lee−Kesler mixing rules for the two studied biodiesels are shown in Table S5, in the SI.

Table 7. Comparison between Experimental Density and Rackett's Model Modified by Spencer−Danner for Pure Biodiesel[a]

sample	SD	AAE	ARE
sunflower biodiesel	0.00	0.00	0.12
fish oil biodiesel	0.01	0.01	0.58

[a] SD = $(\Sigma(X_{\text{est}} - X_{\text{exp}})^2/N)^{1/2}$; AAE = $1/N\,\Sigma|X_{\text{est}} - X_{\text{exp}}|$; ARE (%) = $1/N\,\Sigma|(X_{\text{est}} - X_{\text{exp}})/X_{\text{exp}}|\cdot 100$.

Table 8. Comparison between Experimental Viscosity Data and Predictive Models for Pure Biodiesel[a]

sample	SD			AAE			ARE (%)		
	OEM[b]	TM[c]	SRMM[d]	OEM	TM	SRMM	OEM	TM	SRMM
sunflower	1.30	1.86	0.23	1.01	1.61	0.13	20.70	49.58	3.13
fish oil	1.41	2.64	0.27	1.09	2.26	0.17	22.10	68.10	4.28

[a] SD = $(\Sigma(X_{\text{est}} - X_{\text{exp}})^2/N)^{1/2}$; AAE = $1/N\Sigma|X_{\text{est}} - X_{\text{exp}}|$; ARE (%) = $1/N\Sigma|(X_{\text{est}} - X_{\text{exp}})/X_{\text{exp}}|\cdot 100$. [b] Orrick−Erbar model. [c] Thomas model. [d] Sastri−Rao modified model.

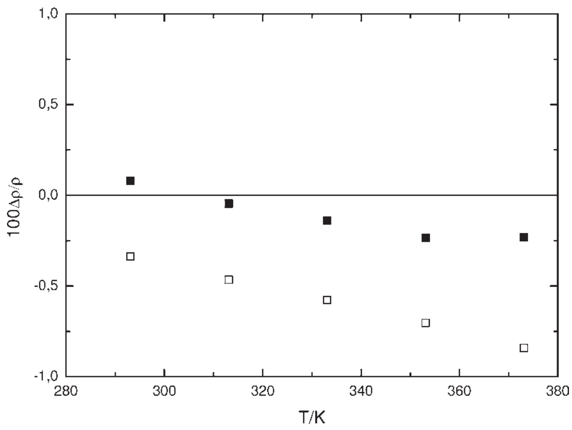

Figure 5. Deviation between experimental density and estimated density by the Rackett equation modified by the Spencer−Danner model for sunflower biodiesel (■) and fish oil biodiesel (□).

It should be noticed that the proportion of biodiesel was considered to behave as an ideal solution applying the Kay's rule to evaluate the viscosity (μ) of biodiesel. Kay's rule is expressed by eq 8:

$$\mu_m = \sum_i w_i \mu_i \tag{8}$$

The deviation between experimental data of sunflower and fish oil biodiesel and the estimated density by the modified Rackett's equation are shown in Figure 5. The deviation between experimental data of sunflower biodiesel and fish oil biodiesel and the estimated viscosity by the modified Sastri−Rao model are shown in Figure 6.

Figures 7 and 8 show the behavior of sunflower biodiesel and fish oil biodiesel as a function of temperature using the modified Sastri−Rao model and Kay's rules. The results show that the Sastri−Rao model predicted with good accuracy the viscosity of biodiesel.

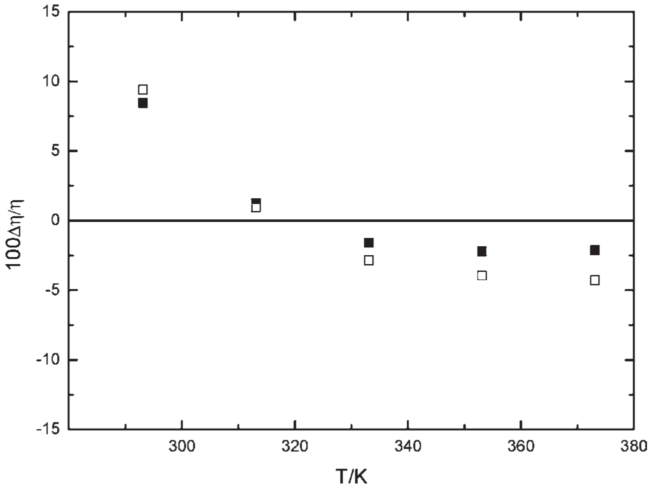

Figure 6. Deviation between experimental viscosity and estimated viscosity by the Sastri−Rao modified group contribution model for sunflower biodiesel (■) and oil fish biodiesel (□).

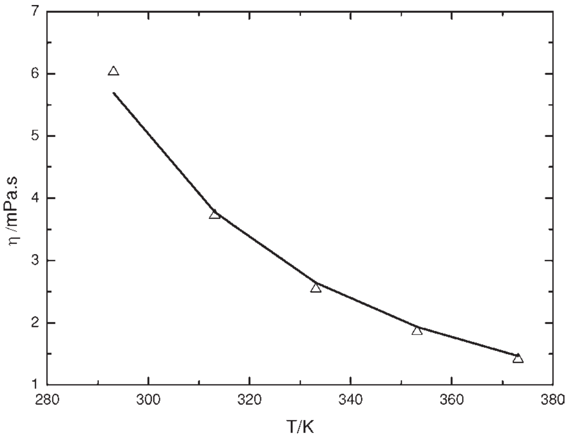

Figure 7. Comparison between experimental data (△) and estimated (solid line) viscosity using Sastri−Rao modified and Kay's mixture rules for sunflower biodiesel.

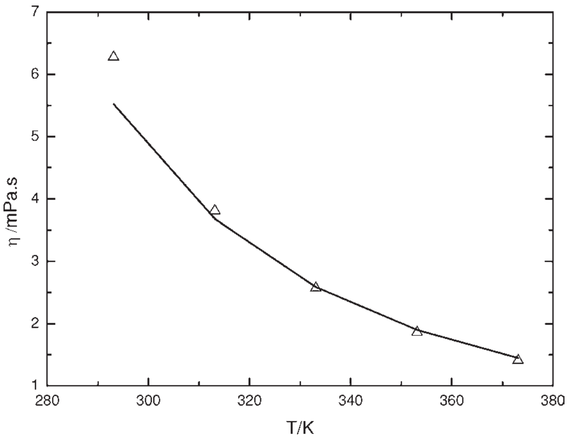

Figure 8. Comparison between experimental data (△) and estimated (solid line) viscosity using Sastri−Rao modified and Kay's mixture rules for oil fish biodiesel.

■ CONCLUSIONS

Density and viscosity of blends of sunflower or fish oil biodiesels + diesel were determined at temperatures ranging from (293.15 to 373.15) K in steps of 20.0 K. Density and viscosity data were correlated with good agreement with the first-order polynomial and the Andrade's equation, respectively. Viscosity deviation was calculated using experimental data and fitted using the Redlich−Kister polynomial equation. All systems measured exhibited slightly negative viscosity deviation values and decreased with the increase in temperature. The modified Sastri−Rao model used to predict the viscosity showed the best accuracy with a maximum relative deviation of 4.28 %. The experimental and estimated density values had relative differences of a maximum of 0.58 %.

■ ASSOCIATED CONTENT

Ⓢ **Supporting Information.** Data tables described in the text. This material is available free of charge via the Internet at http://pubs.acs.org.

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