ARTICLE

Densities and Viscosities of Minority Fatty Acid Methyl and Ethyl Esters Present in Biodiesel

Maria Jorge Pratas,[†] Samuel Freitas,[†] Mariana B. Oliveira,[†] Sílvia C. Monteiro,[‡] Álvaro S. Lima,[§] and João A.P. Coutinho*,[†]

[†]CICECO, Chemistry Department, University of Aveiro, Campus de Santiago, 3810−193 Aveiro, Portugal
[‡]Polytechnic Institute of Leiria, Leiria, Morro do Lena — Alto Vieiro, 2411-901 Leiria, Portugal
[§]Universidade Tiradentes, Av. Murilo Dantas 300, Farolândia, Aracaju-SE, Brasil

ABSTRACT: Biodiesels have several known components in their composition. The majority of components is well described in the literature, but a minority of components are poorly characterized. These are however required to develop reliable models to predict the biodiesel behavior. This work considers minor components of biodiesel: the polyunsaturated compounds (in C18), the monounsaturated (in C16, C20, and C22), and the long-chain saturated esters. In this work, densities and viscosities of pure fatty acid ester minor components of biodiesel fuel were measured (three ethyl esters and seven methyl esters), at atmospheric pressure and temperatures from (273.15 to 373.15) K. Correlations for the densities and viscosities with temperature are proposed. Three predictive models were evaluated in the prediction of densities and viscosities of the pure ethyl and methyl esters here reported. The GCVOL group contribution method is shown to be able to predict densities for these compounds within 1.5 %. The methods of Ceriani et al. (CM) and of Marrero et al. (MG) were applied to the viscosity data. The first show a better predictive capacity to provide a fair description of the viscosities of the minority esters here studied.

INTRODUCTION

Nowadays, biodiesel fuel is seen as an alternative to the conventional petroleum-based fuels, reducing the dependency on fossil fuels and controlling greenhouse gas emissions.[1] Biodiesel fuel advantages and applications are well established.[2−9] This biofuel is comprised of monoalkyl esters of fatty acids derived from vegetable oils, animal fats, or mixtures of them. It is usually produced by the transesterification reaction of triglycerides with a short-chain alcohol, usually methanol or ethanol, in the presence of a catalyst, leading to the formation of mixtures of fatty acid methyl esters (FAMEs) or fatty acid ethyl esters (FAEEs), respectively.[7,9] The main components of biodiesel fuel depend on the raw materials used, and consequently, a wide range of esters can be present.[10] Knowing the profile of methyl or ethyl esters in biodiesel is of great importance as it controls its main properties.[10]

The fuel density influences the amount of mass injected at the injection systems, pumps, and injectors.[11,12] An amount of fuel precisely adjusted is necessary to provide proper combustion.[13] Combustion is initialized through atomization of the fuel. The use of a viscous fuel leads to a poor atomization which is responsible for premature injector cooking and poor fuel combustion.[11,14]

Density and viscosity data are well established for the more important biodiesel compounds; however, some of the minor components have received little attention in the past. However, they may have a non-negligible influence on the biodiesel fuel properties, and depending on the raw material used, these components can be present in a significant concentration.

The main goal of this work is to present new density and viscosity data for the minority components of biodiesel fuel such as methyl palmitoleate, methyl linolenate, methyl arachidate, methyl gadoleate, methyl behenate, methyl erucate, methyl lignocerate, ethyl linoleate, ethyl linolenate, and ethyl arachidate, at atmospheric pressure and

temperatures from (273.15 to 363.15) K. Some of these esters can be found in biodiesel fuel from peanut, rapeseed, or canola oils.[9]

Among the studied esters, density data with temperature have been found only for methyl linolenate. Ott el al.[15] compiled the available density data. Besides the seven points reported by them, only Gouw and Vlugter[16] measured two other data points, in 1964. For the other esters, some isolated density data were obtained from the compound's supplier.

As experimental measurements are time-consuming and expensive, especially for these minority biodiesel ester components, new models are necessary to predict these properties.

Several models have been proposed in the literature to estimate biodiesel fuel density and viscosity. The most important among them rely on the accurate knowledge of the properties of the pure compounds.[17] However, the scarcity of density and viscosity data available in the literature restricts the use of these models to predict properties for biodiesel fuel. In a previous work,[17] the densities and viscosities of common pure methyl and ethyl esters were measured and used to evaluate the performance of three predictive models. For prediction of density, the group contribution method GCVOL[18] (group contribution method for predicting saturated liquid density) model was evaluated, while the models of Ceriani et al.[19] and Marrero et al.[20] were tested for the viscosity. The behavior of these models is here compared against the data for the compounds here studied.

EXPERIMENTAL SECTION

Materials and Procedure. Three ethyl ester and seven methyl esters were used in this study. Table 1 reports the name, purity,

Table 1. Methyl and Ethyl Esters Studied in This Work

compound IUPAC name	fatty acid chain	common name	purity mass %	source	CAS
(Z,Z)-9,12-octadecadienoic acid, ethyl ester	C18:2	ethyl linoleate	99	Sigma	544-35-4
(Z,Z,Z)-9,12,15-octadecatrienoic acid, ethyl ester	C18:3	ethyl linolenate	99	Sigma	1191-41-9
eicosanoic acid, ethyl ester	C20:0	ethyl arachidate	98	Sigma	18281-05-5
(Z)-9-hexadecenoic acid, methyl ester	C16:1	methyl palmitoleate	99	Fluka	1120-25-8
(Z,Z,Z)-9,12,15-octadecatrienoic acid, methyl ester	C18:3	methyl linolenate	99	Sigma	301-00-8
eicosanoic acid, methyl ester	C20:0	methyl arachidate	99.5	Fluka	1120-28-1
cis-11-eicosenoic acid, methyl ester	C20:1	methyl gadoleate	98	Sigma	2390-09-2
docosanoic acid, methyl ester	C22:0	methyl behenate	99	Fluka	929-77-1
(Z)-13-docosenoate acid, methyl ester	C22:1	methyl erucate	99	Fluka	1120-34-9
tetracosanoic acid, methyl ester	C24:0	methyl lignocerate	99	Fluka	2442-49-1

Table 2. Experimental Density, in $kg \cdot m^{-3}$, for Ethyl and Methyl Esters

T/K	ethyl			methyl						
	linoleate	linolenate	arachidate	palmitoleate	linolenate	arachidate	gadoleate	behenate	erucate	lignocerate
278.15	893.8	904.6		880.4	913.2		884.8		881.6	
283.15	890.0	900.8		876.6	909.5		881.1		877.9	
288.15	886.3	897.0		872.8	905.7		877.5		874.3	
293.15	882.6	893.3		869.0	901.9		873.8		870.7	
298.15	878.8	889.5		865.2	898.2		870.2		867.1	
303.15	875.2	885.8		861.4	894.5		866.6		863.6	
308.15	871.5	882.0		857.6	890.7		863		860.0	
313.15	867.8	878.3		853.8	887.0		859.5		856.5	
318.15	864.2	874.6	841.2	850.0	883.3		855.9		853.0	
323.15	860.6	870.9	837.7	846.3	879.6	842.3	852.3		849.4	
328.15	856.9	867.2	834.1	842.5	876.0	838.7	848.8		845.9	
333.15	853.3	863.5	830.5	838.7	872.3	834.9	845.2	834.5	842.4	
338.15	849.7	859.9	827.0	834.9	868.6	831.4	841.7	831.0	839.0	830.5
343.15	846.0	856.2	823.5	831.1	864.9	827.8	838.2	827.5	835.5	827.1
348.15	842.4	852.5	820.0	827.3	861.2	824.3	834.6	824.1	832.0	823.7
353.15	838.8	848.9	816.6	823.5	857.6	820.8	831.1	820.6	828.6	820.3
358.15	835.2	845.3	813.2	819.8	853.9	817.3	827.6	817.2	825.1	816.9
363.15	831.6	841.6	809.7	816.0	850.3	813.8	824.1	813.7	821.7	813.5
368.15		838.0	806.1			810.4	820.7	810.3		810.1
373.15		834.5	802.4			807.0	817.3	806.9		806.8

supplier, and CAS number of each compound used in this study. Compound purity was confirmed by gas chromatography/flame ionization detection (GC-FID).

Experimental Measurements. Measurements of viscosity and density were performed in the temperature range of (273.15 to 373.15) K, or above melting point for saturated compounds, at atmospheric pressure using an automated SVM 3000 Anton Paar rotational Stabinger viscometer−densimeter. The viscometer is based on a tube filled with the sample in which floats a hollow measuring rotor. Because of its low density, the rotor is centered in the heavier liquid by buoyancy forces. Consequently, a measuring gap is formed between the rotor and the tube. The rotor is forced to rotate by shear stresses in the liquid and is guided axially by a built-in permanent magnet, which interacts with a soft iron ring. The rotating magnetic field delivers the speed signal and induces eddy currents in the surrounding copper casing. These eddy currents are proportional to the speed of the rotor and exert a retarding torque on the rotor. Two different torques influence

the speed of the measuring rotor. At the equilibrium, the two torques are equal, and the viscosity can be traced back to a single speed measurement. The SVM 3000 uses Peltier elements for fast and efficient thermostability. The temperature uncertainty is 0.02 K from (288.15 to 378.15) K. The absolute uncertainty of the density is 0.0005 $g \cdot cm^{-3}$, and the relative uncertainty of the dynamic viscosity obtained is less than 0.5 % for the standard fluid SHL120 (SH Calibration Service GMbH), in the range of the studied temperatures. Further details about the equipment and method can be found elsewhere.[21] This viscometer was previously tested for other compounds and presented a very good reproducibility.[17,22]

■ RESULTS AND DISCUSSION

Density. The experimental data obtained are reported in Table 2. For ethyl and methyl arachidate, methyl behenate, and methyl

lignocerate, the measurements were only carried out at temperatures above the melting point of these compounds.

In Figure 1, deviations from experimental values and available data were presented. Methyl linolenate is the only compound for which data are available in a wide temperature range[15] presenting a deviation of 0.3 % against the experimental data here reported. For some of the other compounds, only the supplier data were available to compare with measured data.[23] A few isolated points for erucate methyl ester[16] and linoleate ethyl ester[24] were also identified. The relative deviations of these data are less than 2 %.

A linear temperature dependency using an optimized algorithm based on the least-squares method was used to correlate the experimental density data measured, and the parameter values along with their confidence limits are reported in Table 3. This approach was already adopted previously for common pure methyl and ethyl esters.[17]

$$\rho/\text{kg}\cdot\text{m}^{-3} = b\cdot T/\text{K} + a \qquad (1)$$

A number of models to describe the density can be found in the literature, often based on the Racket equation,[12,25−27] but since they require experimental data adjusted parameters they are not predictive.
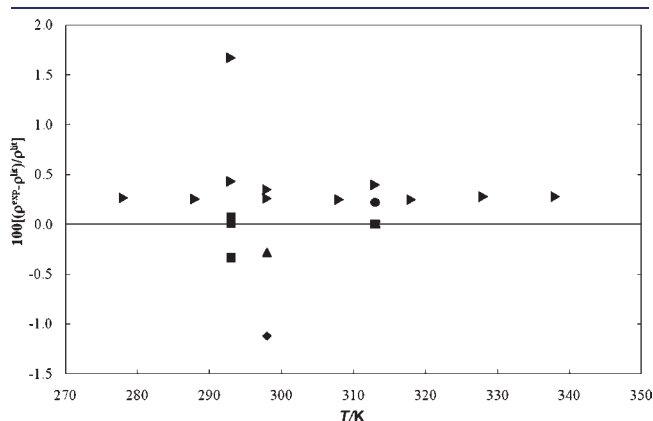
The group contribution method GCVOL model[18] was here used to predict the molar volumes and the densities of the compounds studied in this work. Figures 2 and 3 show GCVOL deviations on the density property predicted for FAEEs and FAMEs, respectively, where unsaturated esters are represented by full symbols and saturated by empty symbols. The results reported
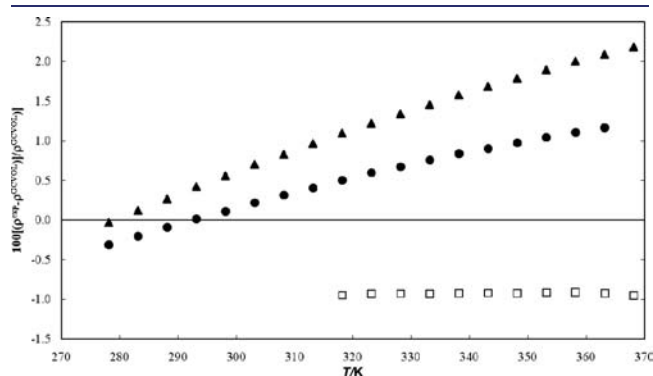
Figure 2. Relative deviations between density of ethyl esters predicted by GCVOL and this work's experimental data as a function of temperature: ●, ethyl linoleate; ▲, ethyl linolenate; and □, ethyl arachidate.
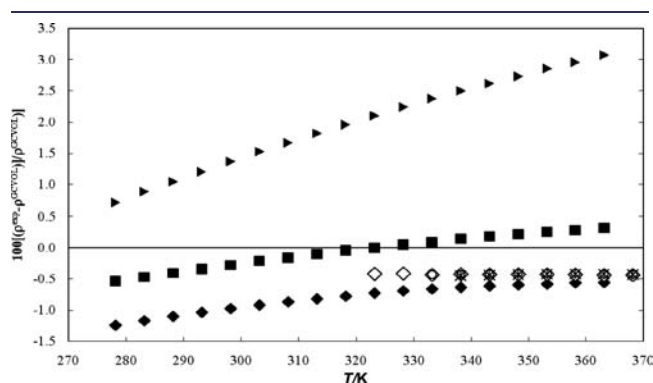
Figure 3. Relative deviations between density of methyl esters predicted by GCVOL and this work's experimental data as function of temperature: ♦, methyl palmitoleate; solid triangle pointing right, methyl linolenate; ◇, methyl arachidate; ★, methyl gadoleate; ○, methyl behenate; ■, methyl erucate; and ∗, methyl lignoceric.

Figure 1. Relative deviation of methyl and esters density data available in the literature[15,16,23,24] as a function of temperature: ●, ethyl linoleate; ▲, ethyl linolenate; ♦, methyl palmitoleate; solid triangle pointing right, methyl linolenate; and ■, methyl erucate. Zero line is this work's experimental data.

Table 3. Density Linear Temperature Correlation Constants (Equation 1) for Pure Methyl and Ethyl Esters over the Temperature Range (278.15 to 373.15) K and Corresponding 95 % Confidence Limits[a]

	$b/\text{kg}\cdot\text{m}^{-3}\cdot\text{K}^{-1}$	±	$t\cdot S_b$	$a/\text{kg}\cdot\text{m}^{-3}$	±	$t\cdot S_A$	t	AAD %
ethyl linoleate	−0.7307		0.002	1096.8		0.7	2.1	0.01
ethyl linolenate	−0.7406		0.003	1109.7		0.7	2.1	0.08
ethyl arachidate	−0.7015		0.004	1064.3		1.3	2.4	0.01
methyl palmitoleate	−0.7577		0.001	1091.1		0.2	2.1	0.003
methyl linolenate	−0.7401		0.002	1118.9		0.6	2.4	0.009
methyl arachidate	−0.7117		0.002	1070.2		0.8	2.1	0.24
methyl gadoleate	−0.7133		0.001	1082.2		0.2	2.4	0.09
methyl behenate	−0.6900		0.003	1064.3		0.9	2.1	0.004
methyl erucate	−0.7038		0.003	1077.0		1.0	2.1	0.01
methyl lignocearate	−0.6783		0.002	1059.9		0.8	2.2	0.006

[a] S, standard deviation.

that the densities of FAME and FAEE can be predicted within a deviation of $\pm$ 1.5 %, with the exception of linolenate esters for higher temperatures due to a poor model description of the polyunsaturation effect on the densities.

The isobaric expansivity coefficient at constant pressure (α_p) is defined as

$$\alpha_p = -\left(\frac{\partial \ln \rho}{\partial T}\right)_p \qquad (2)$$

The logarithm of density exhibits a linear behavior with temperature in the studied temperature range. The isobaric expansivities estimated from the experimental data are reported in Table 4.

Viscosity. The experimental data of viscosity of several minority esters here studied are reported in Table 5. As observed in a previous work,[17] the viscosity of all esters increases with the ester chain length and decreases with the level of unsaturation.

To better ascertain the experimental data of viscosity here measured, these were compared with literature data as shown in Figure 4. Unfortunately, the comparison was limited due to the lack of viscosity data in the literature for the esters here studied. Only viscosities of four FAMES and one FAEE[28−31] were found and used on this comparison. The relative deviations observed reached a maximum deviation of 15 % against the data by Meirelles et al.[19] and as discussed in a previous work.[17] These deviations must be due to experimental problems on the data reported by the authors.

The Vogel−Tammann−Fulcher equation (eq 3) was used to describe the experimental viscosities as a function of temperature.

$$\mu/\text{mPa}\cdot\text{s} = \exp\left(A + \frac{B}{(T/\text{K} - T_0)}\right) \qquad (3)$$

where A, B, and T_0 are fitting parameters whose values were estimated using an optimization algorithm based on the least-squares method. The values of the parameters in conjunction with their uncertainty and the average absolute deviations (AAD %) of the correlation are reported in Table 6. The results show that the VTF equation provides a good description of the experimental data of viscosity with a maximum value of AAD of 0.52 %.

The Ceriani et al.[19] (CM) and the Marrero et al.[32] (MG) group contribution models were evaluated against the viscosity data here measured. The deviations between the experimental and predicted viscosities are shown in Table 7. The CM model presented an overall deviation of 11.9 % for all minority esters studied with the maximum deviations of 21.7 % for ethyl linolenate and 25.9 % for methyl linolenate, respectively. These deviations denote some limitations of the CM model in predicting viscosity of the unsaturated esters. The MG method is much less accurate with an overall AAD of 25.7 % and large deviations for all minority esters.

Table 4. Isobaric Expansivities, α_p, for the Studied Fatty Acid Esters and Corresponding 95 % Confidence Limits[a]

	$\alpha_p \cdot 10^3/\text{K}^{-1}$	$\pm$	$t \cdot s(\alpha_p) \cdot 10^3$
ethyl linoleate	0.847		0.002
ethyl linolenate	0.850		0.002
ethyl arachidate	0.854		0.003
methyl palmitoleate	0.894		0.005
methyl linolenate	0.840		0.002
methyl arachidate	0.860		0.002
methyl gadoleate	0.836		0.001
methyl behenate	0.841		0.001
methyl erucate	0.827		0.001
methyl lignocerate	0.829		0.001

[a] S, standard deviation.

Table 5. Experimental Viscosities, in mPa·s, for Fatty Acid Ethyl and Methyl Esters

T/K	ethyl			methyl						
	linoleate	linolenate	arachidate	palmitoleate	linolenate	arachidate	gadoleate	behenate	erucate	lignocerate
278.15	8.1875	6.2820		6.1685	6.3612		14.340		18.087	
283.15	7.0842	5.5379		5.3667	5.6183		11.975		14.943	
288.15	6.1652	4.9210		4.6617	5.0032		10.131		12.556	
293.15	5.4231	4.4014		4.1075	4.4844		8.6667		10.657	
298.15	4.8073	3.9606		3.6471	4.0429		7.4879		9.1414	
303.15	4.3074	3.5831		3.2886	3.6665		6.5284		7.9069	
308.15	3.8539	3.2578		2.9430	3.3405		5.7379		6.9171	
313.15	3.4060	2.9750		2.6162	2.9253		5.0803		5.9575	
318.15	3.1608	2.7281	5.6573	2.4218	2.6750		4.5289		5.4021	
323.15	2.8291	2.5114	4.9733	2.1751	2.4725	4.8319	4.0624		4.7602	
328.15	2.6411	2.3204	4.5070	2.0304	2.3030	4.3226	3.6649		4.3306	
333.15	2.4287	2.1511	4.0577	1.8697	2.1234	3.8888	3.3231	4.7493	3.9100	
338.15	2.2414	2.0004	3.6714	1.7275	1.9659	3.5170	3.0278	4.2736	3.5480	5.1392
343.15	2.0753	1.8658	3.3373	1.5945	1.8165	3.1964	2.7709	3.8657	3.2344	4.6279
348.15	1.9270	1.7450	3.0462	1.4822	1.6878	2.9177	2.5461	3.5133	2.9609	4.1894
353.15	1.7727	1.6362	2.7799	1.3656	1.5827	2.6745	2.3484	3.2066	2.7070	3.8102
358.15	1.6757	1.5382	2.5690	1.2898	1.4877	2.4612	2.1736	2.9392	2.5097	3.4797
363.15	1.5685	1.4491	2.3716	1.2070	1.4021	2.2726	2.0186	2.7087	2.3223	3.1895
368.15		1.3684	2.1961		1.3272	2.1066	1.8807	2.5020		2.9327
373.15		1.2950	2.0395		1.2527	1.9591	1.7576	2.3169		2.7066

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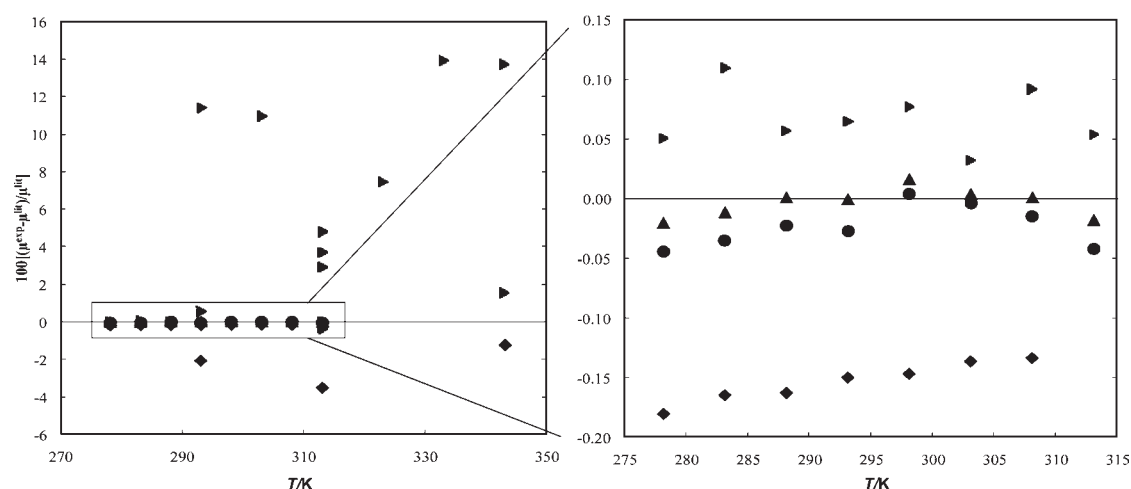dx.doi.org/10.1021/je1012235 |J. Chem. Eng. Data 2011, 56, 2175–2180

Figure 4. Relative deviation of methyl and esters dynamic viscosity data available in the literature[28−31] as a function of temperature: ●, ethyl linoleate; ▲, ethyl linolenate; ♦, methyl palmitoleate; solid triangle pointing right, methyl linolenate; and ■, methyl erucate. Zero line is this work's experimental data.

Table 6. Vogel−Tammann−Fulcher Equation Constants for Pure Ethyl and Methyl Esters Over the Temperature Range (278.15 to 373.15) K and Corresponding 95 % Confidence Limits[a]

	A	± $t \cdot S_A$	B/K	± $t \cdot S_B$	T_0/K	± $t \cdot S_{T_0}$	AAD[b] (%)
ethyl linoleate	−2.54	0.23	715.05	88.3	124.13	11.7	0.49
ethyl linolenate	−2.67	0.46	795.17	202	101.67	24.5	0.053
ethyl arachidate	−2.90	0.23	906.95	92.0	122.33	12.0	0.22
methyl palmitoleate	−2.87	0.35	748.28	159	118.44	19.8	0.52
methyl linolenate	−3.00	0.12	904.38	48.4	91.88	6.42	0.74
methyl arachidate	−2.72	0.02	806.47	7.20	135.35	0.938	0.0070
methyl gadoleate	−2.54	0.02	733.80	7.22	137.19	0.877	0.071
methyl behenate	−2.53	0.15	768.64	52.3	145.06	6.17	0.036
methyl erucate	−2.41	0.12	715.39	52.4	143.27	6.26	0.38
methyl lignocerate	−2.87	0.022	951.53	9.76	127.00	1.32	0.021

[a] S, standard deviation. [b] AAD $= 1/N_p \sum_{i=1}^{N_p} ABS[(\exp_i - \text{calc}_i)/\text{calc}_i] \cdot 100$.

Table 7. Absolute Average Deviation between the Measured Viscosity of Pure Ethyl and Methyl Esters over the Temperature Range (278.15 to 373.15) K and Those Estimated by CM and MG Models

	CM (%)	MG (%)
ethyl linoleate	7.84	29.5
ethyl linolenate	21.7	31.0
ethyl arachidate	17.3	23.5
methyl palmitoleate	5.95	24.1
methyl linolenate	25.9	18.4
methyl arachidate	11.8	13.4
methyl gadoleate	9.30	25.9
methyl behenate	18.2	44.3
methyl erucate	0.250	27.5
methyl lignocerate	0.270	19.5
overall AAD	**11.9**	**25.7**

CONCLUSIONS

New experimental data for the densities and viscosities of pure saturated and unsaturated methyl and ethyl esters from minority biodiesel fuel composition, in the temperature range (273 to 363) K and at atmospheric pressure, are presented. New correlations of the density and viscosity dependency with temperature are also proposed. The experimental data here reported were used to test density and viscosity predictive models.

The GCVOL model predictions were compared with the experimental liquid densities to show that it is able to describe the FAMEs and FAEEs densities with deviations smaller than 1 % for saturated compounds and 2 % for unsaturated.

The Ceriani et al. method showed to be superior to the Marrero et al. method in terms of predictive ability for viscosities, presenting an overall average deviation of 11.9 % for all minority esters here studied.

■ AUTHOR INFORMATION

Corresponding Author
*E-mail: jcoutinho@ua.pt. Phone: +351 234401507. Fax: +351 234370084.

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