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Measurement and prediction of speeds of sound of fatty acid ethyl esters and ethylic biodiesels

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HIGHLIGHTS

- ▶ Measurement of speed of sound for several fatty acid ethyl esters was carried.
- ▶ Speed of sound data for ethylic biodiesels is reported for the first time.
- ▶ Prediction of speed of sound was carried using the Wada's group contribution method.
- ▶ The Wada's model provides a good description of speed of sound.

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ABSTRACT

Speed of sound of fatty esters provides important information about biodiesel injection characteristics and enables the estimation of many other important properties of biodiesels. Nevertheless, the experimental speeds of sound of fatty esters are very scant. This work provides new data on speed of sound for nine fatty acid ethyl esters and four ethylic biodiesels, measured at atmospheric pressure and temperatures ranging from 293.15 to 343.15 K. These new data is used to evaluate the ability of the Wada's group contribution method to predict the biodiesel speed of sound. It is here shown that this model provides excellent description of the experimental data, with overall average relative deviations (OARDs) of 0.25% for the ethyl esters and between 0.45% and 0.59% for the biodiesels.

1. Introduction

Worldwide researchers, fuel industries and policy-makers have placed biofuels at the forefront of renewable energies, through various policies and incentives. Among the biofuels, in Europe the biodiesel has been playing a key-role in the promotion of energy sustainability, especially for the transport sector. This tendency is expected to keep increasing in the coming years when new feedstocks for biodiesel production are developed from agricultural residues and non-edible oils. Blends of biodiesel with diesel and ethanol with gasoline are expected to account for 54% of the growth in liquids fuel consumption between 2009 and 2035 [1]. This happens because biodiesel are obtained from vegetable oils, animal fats and greases, and its use as fuel is economically viable, technically compatible and environmentally friendly. Besides being produced from renewable sources, it contributes less to greenhouse gases emissions and can replace petrodiesel totally or partially in conventional diesel engines without modification [2–4].

The aforementioned advantages have fed the academic interest in biodiesel in the last few years. While some works focused on establishing novel approaches to improve the production and purification [5–7] others have oriented their line of research to the

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study of thermophysical properties of fatty esters and biodiesels [8–16] to enhance the quality of biodiesels, i.e., their properties in accordance with the Norm CEN EN 14214 [17] in Europe and the Norm ASTM D6751 in United States of America [18].

Speed of sound is one important acoustic property that affects directly the fuel injection characteristics, especially for injectors activated with pressure, and the amount of NOx emissions in the mechanically controlled in-line injection systems by simply changing the combustion temperature. High speeds of sound increase the combustion temperature and consequently the amount of NOx emissions [19]. This property also permits the estimation of other thermodynamic properties like isentropic and isothermal compressibilities, isobaric thermal expansion coefficient, thermal pressure coefficient and the reduced bulk modulus [20,21].

As biodiesels are composed of fatty esters, the knowledge of the properties of these pure compounds enables the prediction of the properties of those fuels as we have shown elsewhere for other biodiesel properties [10,14,15,22]. Unfortunately there are but a few data available in the literature for fatty acid methyl esters (FAMEs) [13,21,12,23–27] being the experimental speeds of sound of fatty acid ethyl esters (FAEEs) even more scant [28], although some studies were already done for the shorter methyl and ethyl esters [29]. There at, this work aims at providing new experimental data of speed of sound for nine saturated and unsaturated FAEE and four ethylic biodiesels, measured at atmospheric pressure and temperatures from 293.15 to 343.15 K, and then using them to evaluate the predictive ability of Wada's group contribution method [28].

2. Experimental section

2.1. Esters samples

The nine ethyl esters here studied were ethyl butyrate (98% quoted purity from Fluka), ethyl caprylate (>99% quoted purity from Aldrich), ethyl caprate (99% quoted purity from Fluka), ethyl laurate (99% quoted purity from Sigma), ethyl myristate (99% quoted purity from Aldrich), ethyl palmitate (>99% quoted purity from Sigma), ethyl stearate (>99.0% quoted purity from Fluka), ethyl oleate (>98% quoted purity from Aldrich), ethyl linoleate (>99% quoted purity from Sigma). These compounds were used as received without any further purification.

2.2. Biodiesel samples: synthesis and analysis

Three of the four biodiesel samples studied here were synthesized by a transesterification reaction of vegetable oils, such as soybean (S), sunflower (Sf) and palm (P), performed on a laboratory scale. The transesterification reaction was carried out with ethanol using sodium hydroxide (NaOH) as the catalyst. The amount of NaOH used was 1.0 wt.% of the oil. Oil and ethanol with a mole ratio of 1:6 reacted at 323.15 K for 180 min.

A fourth sample consisting of ethylic biodiesel derived from soybean oil and beef tallow (S+B) was also investigated. This sample was an industrial one kindly supplied by Fertibom (Catanduva, SP, Brazil), a Brazilian company that produces ethylic biodiesel in industrial scale.

The fatty acid ethyl esters (FAEEs) compositions for all biodiesel samples were determined in triplicate by gas chromatography. The chromatographic analyses were carried out using a GC capillary gas chromatograph system (Agilent, 6850 Series GC System, Santa Clara, CA, USA) under the following experimental conditions: Elite 225 capillary column (PERKIN ELMER, 50% Cyanopropylphenyl–Phenylmethylpolysiloxane, 0.25 μm × 29 m × 0.25 mm); helium as carrier gas at a flow rate of 2.17×10^{-8} m^3/s; injection temperature of 523 K; column temperature of 373 K for 120 s, 373–503 K (rate of 7 K/60 s), 503 K for 600 s; detection temperature of 523 K; and injection volume of 1.0 μL. The fatty acid ethyl esters were identified by comparison with external standards purchased from Nu Check Prep (Elysian, MN, USA). Quantification was done by internal normalization.

2.3. Measurement of density and speed of sound

Experimental measurements of density and speed of sound were made concurrently using an Anton Paar vibrating tube densimeter and ultrasound speed meter, model DSA 5000M, with an automatic temperature control within ±0.01 K. All measurements were made at ambient pressure. According to the procedure already described elsewhere [30], calibration of the speed of sound cell was made with degassed Millipore ultra-quality water. Measurement and comparison with literature values of speed of sound of toluene and cyclohexane at 25 °C leads us to assume an accuracy of 0.5 m s^{-1}, as claimed by the manufacturer. In the case of density, besides the usual method recommended by the manufacturer of using dry air and degassed ultra-pure water at 293.15 K as reference fluids, a new calibration procedure thoroughly described elsewhere [31] was performed. The calibrants used were ultra-pure water and dodecane with certified density values issued by H§D Fitzgerald, with expanded uncertainties of 0.01 kg m^{-3} (coverage factor $k = 2$, providing a 95% level of confidence). The use of this pair of calibrating fluids allowed a close bracketing of the densities measured, the importance of which has recently been emphasized by Fortin et al. [32] As the temperature range of certified density values for dodecane does not cover values higher than 323.15 K, an extrapolation of those values had to be made. However, a careful analysis of results based on comparison between direct density values (taken from direct readings of the densimeter) and final values obtained from the calibration procedure allowed a reassurance about the validity of that extrapolation.

Every day before starting the measurements, the usual routine procedure of performing a water and air check was invariably adopted. Before injection all samples were pre-heated, and degassed, at the maximum experimental temperature. Then, for the same single sample injection a complete series of measurements was made, decreasing the temperature from 343.15 K to 293.15 K in decrements of 5 K. At each temperature three to seven data readings were taken and some measurements were repeated with a new injection, allowing asserting an estimate for the repeatability and standard uncertainty for density values lower than 0.0006% and 0.005%, respectively, and for speed of sound of 0.01% and 0.02%, respectively. After each set of measurements the instrument was flushed several times with *n*-heptane at 333 K and with acetone at 313 K, sequentially, and then dried at 343 K during at least 1 h, with a stream of forced room air. To assess the effectiveness of these cleaning actions, new air and water checks were done and whenever deviations higher than 0.002% for density and 0.013% for sound speed were found, a new cycle of cleaning steps was executed.

3. Prediction of speed of sound

The prediction of speed of sound for ethyl esters and ethylic biodiesels here studied was done by using the Wada's Group Contribution Method previously proposed by us [28] to predict the speed of sound of alkyl esters. In a previous work, we showed that this model could provide a good prediction of the speed of sound of methyl esters and the corresponding methylic biodiesels [13]. This model simply relates speed of sound (u in m s^{-1}) with density (ρ in kg m^{-3}), molecular mass (M_w in g mol^{-1}) and molecular compressibility (κ_m) according to the following equation:

$$u = \rho^3 \left(\frac{\kappa_m}{M_w}\right)^{7/2} \quad (1)$$

The molecular compressibility (κ_m) is also known as Wada's constant and its value can easily be decomposed in groups [28] allowing for the establishment of a group contribution model as presented in the following equation:

$$K_m(T) = \sum_{j=1}^{n_G} N_j K_{m,j}(1 - \chi(T - T_0)) \quad (2)$$

where $K_{m,j}$ connotes the Wada's constant of the group j which occurs N_j times in the given molecule and χ is a constant parameter used to take into account the influence of temperature.

As described in our previous works [13,28], to carry out the predictions of speed of sound using the Wada's model, the ester molecule must be split into five main groups: —CH_3— and —CH_2— to account the linear and saturated alkyl chain, —CH=CH— to describe the contribution of the unsaturation of the alkyl chain and —CH_3COO— and —CH_2COO— to take into account the ester contribution from methyl and ethyl esters, respectively. Then the corresponding Wada's constants reported in Daridon et al. [28], are used to estimate the speed of sound for each ethyl ester in the range of temperatures investigated.

For biodiesels, the application of Wada's model can be carried using two different approaches. The first approach (Wada 1) follows exactly the method described above, i.e., splits the biodiesel molecules into the main groups aforementioned, whose Wada's constants are already known, then predicts their speeds of sound using either the experimental or predicted densities of biodiesels using a linear mixing rule of the densities of the pure fatty acid esters as the two approaches present only ca. 0.1% of difference. Since there are no experimental data for some of the less common FAEEs, a pseudo-component approach is used, where the biodiesel composition is modified by adding C16:1 toC16:0 and C20:1, C22:0 and C24:0 to C20:0.

The second approach (Wada 2) consists of using a linear mixing rule to predict the speed of sound of biodiesels from that of their pure constituents (FAEE) according to Eq. (3), as suggested previously for methylic biodiesels [12]:

$$u_{BD} = \sum_i x_i u_i \quad (3)$$

where u_{BD} (m s^{-1}) is the speed of sound of biodiesels, x_i is the molar fraction and u_i (m s^{-1}) is the speed of sound of pure ethyl esters predicted with the Wada's model. For this approach the estimation of the densities for pure FAEEs were the same as those used in Wada 1.

To assess the predictive ability of the model, the relative deviations (RDs) between the predicted and experimental data of speed of sound were calculated according to the following equation:

$$RD(\%) = \frac{u_{calc_i} - u_{exp_i}}{u_{exp_i}} \times 100 \quad (4)$$

Then the average relative deviation (ARD) was calculated as a summation of the modulus of RD over N_p experimental data points. The overall average relative deviation (OARD) was calculated by Eq. (5), where N_s is the number of systems studied:

$$\text{OARD}(\%) = \frac{\sum_n ARD}{N_s} \quad (5)$$

4. Results and discussion

The experimental densities and speeds of sound for nine fatty acid ethyl esters and four ethylic biodiesels, measured at atmospheric pressure and temperatures from 293.15 to 343.15 K, are presented in Tables 1a and 1b and 2. The FAEEs compositions of the studied biodiesels are reported in Table 3.

For pure ethyl esters, the magnitude of densities is in very good agreement with that reported by Pratas et al. [9] Their speeds of sound decrease with the temperature and increase with the ester

Table 1a
Experimental speed of sound of fatty acid ethyl esters.

T (K)	Butyrate	Caprylate	Caprate	Laurate	Myristate	Palmitate	Stearate	Oleate	Linoleate
u (m s^{-1})									
293.15	1195.13	1280.73	1313.21	1339.55	1361.05			1396.59	1405.24
298.15	1173.94	1261.39	1294.31	1320.91	1342.60			1378.54	1387.18
303.15	1152.95	1242.35	1275.62	1302.49	1324.34	1342.65		1360.67	1369.28
308.15	1132.16	1223.50	1257.14	1284.26	1306.23	1324.67		1342.98	1351.56
313.15	1111.52	1204.60	1238.82	1266.16	1288.32	1306.90		1325.49	1334.05
318.15	1091.01	1186.02	1220.66	1248.26	1270.61	1289.33	1304.71	1308.17	1316.70
323.15	1070.67	1167.59	1202.67	1230.54	1253.08	1271.96	1287.43	1291.03	1299.47
328.15	1050.49	1149.35	1184.87	1213.00	1235.76	1254.78	1270.34	1274.04	1282.49
333.15	1030.43	1131.23	1167.20	1195.61	1218.51	1237.76	1253.46	1257.24	1265.67
338.15	1010.55	1113.31	1149.72	1178.43	1201.52	1220.91	1236.80	1240.64	1249.03
343.15	990.85	1095.65	1132.43	1161.44	1184.74	1204.31	1220.46	1224.26	1232.62

Table 1b
Experimental density of fatty acid ethyl esters.

T (K)	Butyrate	Caprylate	Caprate	Laurate	Myristate	Palmitate	Stearate	Oleate	Linoleate
ρ (kg m^{-3})									
293.15	878.96	866.48	863.97	862.15	860.95			868.87	880.49
298.15	873.68	862.16	859.90	858.25	857.18			865.24	876.83
303.15	868.38	857.84	855.83	854.35	853.39	852.48		861.62	873.17
308.15	863.07	853.52	851.76	850.45	849.62	848.79		858.00	869.53
313.15	857.73	849.17	847.69	846.56	845.84	845.10		854.39	865.88
318.15	852.38	844.84	843.61	842.65	842.06	841.42	841.02	850.77	862.23
323.15	847.00	840.49	839.53	838.74	838.28	837.74	837.42	847.16	858.59
328.15	841.60	836.14	835.45	834.84	834.51	834.07	833.82	843.56	854.95
333.15	836.17	831.78	831.36	830.94	830.73	830.40	830.23	839.95	851.31
338.15	830.71	827.41	827.26	827.03	826.95	826.73	826.65	836.35	847.67
343.15	825.23	823.04	823.16	823.12	823.18	823.06	823.06	832.75	844.04

Table 2
Experimental density and speed of sound of ethylic biodiesels.

T (K)	ρ (kg m^{-3})				u (m s^{-1})			
	S	Sf	S+B	P	S	Sf	S+B	P
293.15	876.64	875.65	875.44	866.65	1402.10	1402.40	1400.00	1390.27
298.15	872.99	872.01	871.79	862.97	1384.09	1384.20	1381.98	1372.07
303.15	869.36	868.37	868.13	859.31	1366.24	1365.85	1364.13	1354.08
308.15	865.72	864.74	864.49	855.65	1348.59	1347.97	1346.48	1336.28
313.15	862.09	861.11	860.84	851.99	1331.13	1330.55	1328.93	1318.66
318.15	858.46	857.49	857.20	848.34	1313.83	1313.36	1311.61	1301.24
323.15	854.83	853.85	853.55	844.68	1296.70	1296.12	1294.38	1283.98
328.15	851.20	850.23	849.92	841.04	1279.74	1279.22	1277.38	1266.90
333.15	847.56	846.61	846.28	837.39	1262.96	1262.49	1260.55	1249.99
338.15	843.94	843.00	842.65	833.74	1246.38	1245.97	1243.93	1233.24
343.15	840.32	839.38	839.02	830.09	1230.04	1229.69	1227.55	1216.68

Table 3
Composition of the biodiesels studied, in mass percentage.

Fatty acid ethyl esters	Biodiesel			
	S	Sf	S+B	P
Octanoate	–	–	–	0.03
Decanoate	–	–	–	0.03
Laurate	–	–	0.03	0.42
Myristate	0.07	0.09	0.30	0.72
Palmitate	10.92	5.66	11.81	38.67
Palmitoleate	0.08	0.09	0.16	0.15
Stearate	2.93	3.11	3.23	4.49
Oleate	27.45	35.32	27.53	44.51
Linoleate	52.65	54.46	49.90	10.29
Linolenate	4.96	0.28	5.87	0.26
Arachidate	0.29	0.20	0.31	0.25
Eicosenoate	0.18	0.13	0.20	0.10
Behenate	0.37	0.49	0.44	0.04
Erucate	–	0.04	0.08	0.03
Lignocerate	0.099	0.14	0.15	0.02

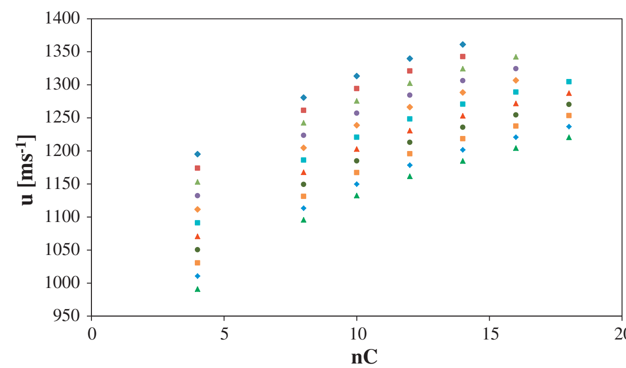

Fig. 1b. The dependency of speed of sound of FAEE on carbon chain length at different temperatures in Kelvin. 293.15, 298.15, 303.15, 308.15, 313.15, 318.15, 323.15, 328.15, 333.15, 338.15, 343.15.

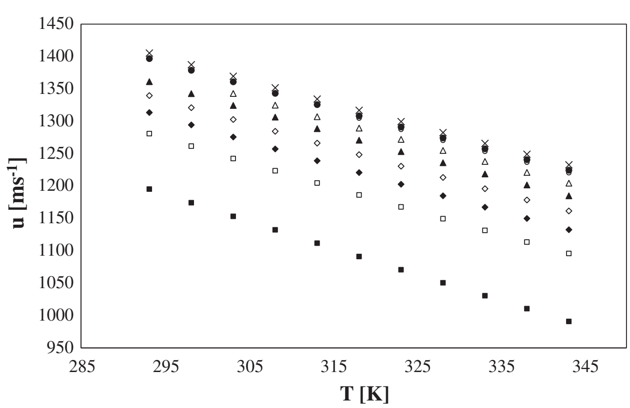

Fig. 1a. The dependency of speed of sound of FAEE on temperature. ■ Butyrate, □ Caprylate, ♦ Caprate, ◇ Laurate, ▲ Myristate, △ Palmitate, ° Stearate, ● Oleate and × Linoleate.

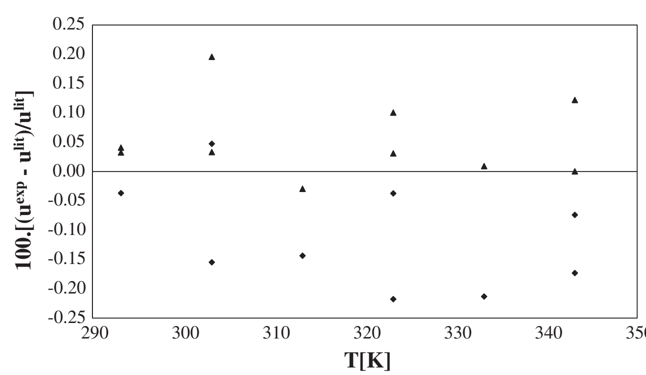

Fig. 2. RDs for ethyl esters available in the literature ♦ Caprate [28,33] and ▲ Myristate [28,34].

chain length as seen in Figs. 1a and 1b. Moreover, for the same chain length, the presence of unsaturated bonds in the ester molecule increases the magnitude of speed of sound as expected since this property also depends directly on the density. Due to the lack of experimental data for ethyl esters, our experimental data were only compared to those reported by Daridon et al. [28] and Ndiaye et al. [33,34] the data showed to be in very good agreement, presenting a deviation below ±0.20% as shown in Fig. 2.

For the ethylic biodiesels, the difference of densities between the fluids is mainly expressed by the difference of FAEEs compositions. Moreover, since the FAMEs present a higher value for density than the corresponding FAEEs with the same number of carbon atoms in acid moiety, as already shown in Pratas et al. [9] the magnitude of the densities for ethylic biodiesels is expected to be lower than that of the corresponding methylic biodiesels. Regarding the speed of sound, as previously observed for methylic biodiesels [13], a difference in the speed of sound of only ca. 1.0% is observed between the four types of biodiesels studied. The same observation is valid for its temperature dependency.

The experimental data here reported was used to test the predictive ability of the Wada's model previously proposed [28]. The results reported in Tables 4 and 5 suggest that the Wada's model provide a very good description of the experimental speeds of

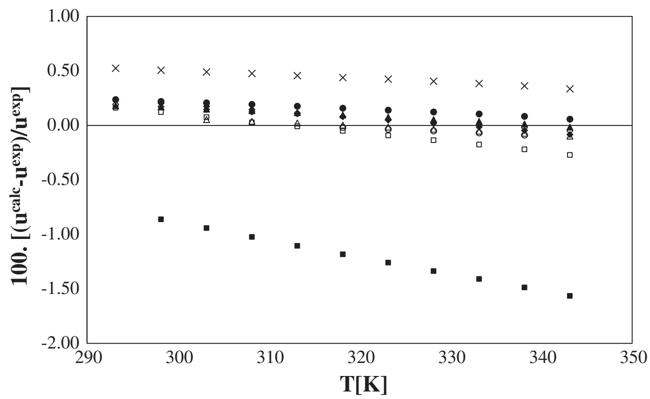

Fig. 3. RDs between experimental and predicted speed of sound of FAEE using Wada's group contribution method. ■ Butyrate, □ Caprylate, ♦ Caprate, ◇ Laurate, ▲ Myristate, △ Palmitate, ◦ Stearate, ● Oleate and ×Linoleate.

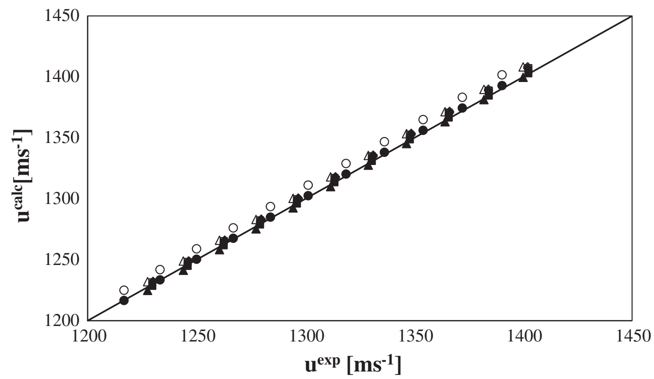

Fig. 4. Experimental and predicted speed of sound of biodiesel fuels using Wada 1 (close symbols) and Wada 2 (open symbols) ♦ S, ■ Sf, ▲ S+B and ● P.

Table 4
Average relative deviations of speed of sound estimated by Wada's model for the fatty acid ethyl esters.

Ethyl ester	ARD (%)
Caprylate	0.12
Caprate	0.11
Laurate	0.089
Myristate	0.094
Palmitate	0.043
Stearate	0.053
Oleate	0.15
Linoleate	0.44
OARD, %	0.14

Table 5
Average relative deviations for ethylic biodiesels.

Biodiesel	ARD (%)	
	Wada 1	Wada 2
S	0.81	0.31
Sf	0.65	0.26
S+B	0.60	0.46
P	0.30	0.75
OARD, %	0.59	0.45

sound for both the ethyl esters and the ethylic biodiesels respectively. For the nine ethyl esters studied the model tends to slightly overpredict the experimental speed of sound. Moreover, the deviations are very stable in the range of temperatures studied, except for the short-chain esters like ethyl butyrate where the model presents larger deviations as seen in Fig. 3. This limitation might be related with the inadequacy of the Wada's constants here used for description of speed of sound of the short-chain esters. Further work to overcome this problem is being undertaken but it does not impact on the systems of interest for the biodiesel industry. By excluding the ethyl butyrate of the remaining ethyl esters due to the larger deviations, the Wada's model presents only an OARD of 0.14% (Table 4). For the biodiesels, the Wada 1 approach presents an OARD of 0.59% (Table 5). Using the Wada 2 approach the OARD obtained was of only 0.45%. The predictions presented in Fig. 4 show that the deviations are temperature independent. Therefore, the Wada's model applied directly or through the mixing rules can be extended to other biodiesel fuels provided that the composition of fatty esters is well known.

Finally since the fuel injection systems operate at high injection pressures, the prediction of high pressure speeds of sound would be of importance. But, unlike for methylic biodiesels, there are yet no data for FAEE to extend the atmospheric pressure model here proposed to high pressures as previously done for methylic biodiesels [12,13]. The measurement of high pressure speed of sound for fatty acid ethyl esters and ethylic biodiesels is being carried in our laboratories and will be object of future works.

5. Conclusions

New experimental data of speed of sound for nine fatty acid ethyl esters and four ethylic biodiesels, measured at atmospheric pressure and temperature from 293.15 to 343.15 K, were here reported and were used to assess the predictive ability of the Wada's model. It is shown that this method describes very well the experimental data of speed of sound for pure esters and biodiesel fuels, presenting only OARDs of 0.25% and 0.45%, respectively. This good description of the data suggests that the Wada's model can be extended to the prediction of the acoustic properties of other biodiesel fuels provided that their FAEEs composition is known.

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