



Electrical and ultrasonic properties of vegetable oils and biodiesel



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HIGHLIGHTS

- Electrical and ultrasonic properties of vegetable oils and biodiesel were determined.
- Measurements allow to distinguish between biodiesel and feedstocks.
- Permittivity and speed of sound are correlated, in biodiesel and in feedstocks.
- Conductivity follows an Arrhenius law.

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ABSTRACT

Electrical properties (permittivity and conductivity) and speed of sound were determined in commercial vegetable oils from sunflower, corn, soybean, grape, cotton, olive, canola and chia, and in biodiesel from these oils. Electrical properties were measured in the frequency range from 20 Hz to 2 MHz at temperatures between 300 K and 343 K. The calibration uncertainty of the measuring system was below 1%.

The speed of sound was determined at the frequencies of 1.53 MHz, 5.66 MHz and 9.43 MHz. The uncertainty of the results was below 0.05%.

In all the measurements in this work, the temperature of the samples was stabilized within ± 0.1 K.

The experimental data for the speed of sound and permittivity of vegetable oils and biodiesel samples fit very satisfactorily to a linear dependence with temperature, whereas the conductivities follow an Arrhenius law.

In all the samples, the fitting parameters of the real part of the relative permittivity, the activation energy of the conductivity and the speed of sound allow to distinguish between the vegetable oil and the biodiesel obtained from it.

Within the temperature range studied in this work, a linear correlation is found between the speed of sound and the permittivity of vegetable oils and biodiesel.

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1. Introduction

Biodiesel (BD) is an alternative fuel obtained from the transesterification of vegetable oils (VO) or animal fats, and a short-chain alcohol [1,2]. After the chemical reaction there are two phases: the higher density phase (glycerin) must be separated and the lighter phase must be purified to comply with BD standards [3,4]. There is an ample variety of feedstocks according to local conditions in different countries [2,5].

Abbreviations: BD, biodiesel; EP, electrical properties; VO, vegetable oils; FAME, fatty acid methyl esters.

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There are two very important issues in BD production: the determination of the progress of the transesterification reaction, and the detection of contaminants in the final product. For BD producers it is desirable to ascertain in real time the progress of the reaction. Moreover, for users the presence of VO in the final product is undesirable since it can damage engines and reduce their performance.

According to international standards [3], the conversion efficiency is determined using chromatographic methods, by measuring the content of mono-, di-, and triglycerides after the purification of the light phase. However, on-line implementation of these measurements it is not easy; also, for small- and medium-scale plants, cost is an additional obstacle. In consequence, alternative methods for the determination of the transesterification progress and the detection of VO are of technological interest.

There are two techniques that satisfy these requirements: dielectric spectroscopy [6] and ultrasonic interferometry [7,8].

The results presented in this work show that dielectric spectroscopy and ultrasonic interferometry are potentially useful for these purposes. Both techniques are fast, non-destructive and of low-cost.

Dielectric spectroscopy has been successfully used in our research group for the production and characterization of BD [9]. It is used to characterize feedstocks from different origins [10], to detect alcohol in the light phase after the transesterification process [11], during the purification process and in the final product [12,13], and also for the characterization of FAME [14].

The increasing availability of multiparametric sensors makes possible the determination of electrical and acoustic properties in real time, at reasonable cost.

2. Materials and methods

2.1. Samples

Commercial VO samples from sunflower, olive, chia, canola, corn, grape and soybean were purchased in supermarkets [10]; BD was produced from these VO as described in Ref. [14].

The ester content was higher than 98.5% in all samples. Free and total glycerol content, water content, viscosity and acid value, flash point and methanol content, were all within the values specified in ASTM and EN standards [3,4]. It is known that methanol content and flash point are strongly correlated [9,11].

The composition of methyl esters in the biodiesel samples was determined according to EN 14103 [15], using a Perkin Elmer Clarus 500 chromatograph with injector split/splitless and a FID detector.

2.2. Measurement system

2.2.1. Electrical properties

Electrical properties (EP) of the samples were determined from isothermal dielectric spectra in the frequency range from 20 Hz to 2 MHz, and at temperatures between 303 K and 343 K [9].

The automatic EP measuring system was described in detail in a previous article [16]. The measuring cell had platinized electrodes to reduce the effects of electrode polarization. The temperature of the samples was controlled within ± 0.1 K with a thermostat (Lauda). The calibration of the system was carried out using cyclohexane (pro analysi) as reference liquid with an uncertainty below 1%.

2.2.2. Speed of sound

Speed of sound was determined by the interferometric method, using an instrument of the type described by Hubbard [8] and Pierce [17] and in more detail in Refs. [7,18]. The calibration was made with water of very high purity (resistivity above 10 M Ω cm), according to Refs. [19,20]. The calibration uncertainty was less than 0.05%. The measurements were made at the frequencies of 1.53 MHz, 5.66 MHz and 9.43 MHz and at temperatures between 303 K and 323 K.

3. Theory

3.1. Electrical properties

The electrical properties of a substance may be described in the frequency domain by the complex relative permittivity, $\varepsilon_r(\omega, T)$. The real part, $\varepsilon_r'(\omega, T)$, represents electrical polarization and the imaginary part, $\varepsilon_r''(\omega, T)$, is related to energy dissipation:

$$\varepsilon_r(\omega, T) = \varepsilon_r'(\omega, T) - i\varepsilon_r''(\omega, T) \quad (1)$$

The relative permittivity is determined by comparing the electrical capacitance of the empty measuring cell with its value when filled with the sample, as described in previous works [9,16].

In the frequency range measured in this work, there are no relaxation effects in the samples. Therefore, ε_r' is practically independent of ω , and ε_r'' describes only the energy dissipation associated the charge transport, i.e., it is directly proportional to conductivity and inversely proportional to frequency. In consequence, the imaginary part or permittivity is only relevant at low frequencies. In summary, for the liquids studied in this work, the complex relative permittivity is modeled by:

$$\varepsilon_r(\omega, T) = \varepsilon_r'(T) - i\sigma(T)/\varepsilon_0\omega \quad (2)$$

The value of $\varepsilon_r'(T)$ decreases with temperature. This is due to the dilatation of the fluid and also to the reduction in molecular polarization. The real part of the relative permittivity, ε_r' , fits very well to a linear dependence with temperature, in the range measured in this work:

$$\varepsilon_r'(T) = \varepsilon_r'(T_0) + d\varepsilon_r'/dT(T - T_0) \quad (3)$$

where T is the sample temperature expressed in Kelvin (K), $\varepsilon_r'(T_0)$ is the relative permittivity of the sample at the reference temperature $T_0 = 318$ K and $d\varepsilon_r'/dT$ is the temperature coefficient of the relative permittivity expressed in K^{-1} at the reference temperature, T_0 .

Conductivity, σ , increases steeply with temperature; experimental data may be fitted to an Arrhenius law:

$$\sigma(T) = \sigma_0 \exp(d/T) \quad (4)$$

where the units of σ are S/m , d is proportional to the conductivity activation energy ΔE ($d = \Delta E/kT$), σ_0 is the pre-exponential factor expressed in S/m and k is the Boltzmann constant ($1.3806503 \times 10^{-23}$ J K^{-1}). It is important to remark that the conductivity activation energy, ΔE , is an intrinsic property of the substance under study. On the other hand, the pre-exponential factor, σ_0 , strongly depends on moisture content and other contaminants [9,14]. This makes difficult to compare directly the results of conductivity measurements. However, it is possible to refer conductivity measurements to a common temperature by means of the Arrhenius dependence. Then, it is possible to compare quantitatively the degree of contamination of the samples (related to σ_0) at the reference temperature. This is important since conductivity measurements may be made during the production, storage and transport of VO and BD, at widely different temperatures.

3.2. Acoustic properties

Speed of sound was measured at 1.53 MHz, 5.66 MHz and 9.43 MHz, for each sample and at each temperature. Since the measured speed of sound showed no variation with frequency, within the experimental uncertainty, the results presented in this work correspond to 9.43 MHz. The acoustic signal during measurements was kept at a level low enough for linear propagation theory to be applicable, and also to ensure that the transducer remained always within its linear range. Moreover, the samples studied in this work may be considered homogeneous.

In all the samples studied in this work, the speed of sound decreases with temperature; the experimental data fit very well to a linear dependence:

$$v(T) = v(T_0) + dv/dT(T - T_0) \quad (5)$$

where the units of $v(T)$ are ms^{-1} , $v(T_0)$ is the speed of sound at the reference temperature ($T_0 = 318$ K) expressed in ms^{-1} , and dv/dT is the temperature coefficient of the speed of sound, expressed in $ms^{-1} K^{-1}$, at the reference temperature T_0 .

4. Results and discussion

4.1. Electrical properties

Fig. 1 shows ϵ'_r the experimental data for samples of sunflower VO and BD as a function of temperature (symbols). The continuous lines correspond to the linear model (Eq. (3)) for each sample and the dashed lines represent the root mean squared uncertainty.

From Fig. 1 it may be seen that BD permittivity is higher than in VO, within the measured temperature range. Although Fig. 1 corresponds to sunflower VO and BD samples, all samples studied in this work show the same behavior.

In Tables 1 and 2, the fitting parameters of the permittivity ($\epsilon'_r(T_0)$ and $d\epsilon'_r/dT$) are shown for VO and BD samples. The parameter uncertainties ($\Delta\epsilon'_r(T_0)$ and $\Delta d\epsilon'_r/dT$), the root mean squared uncertainty ($\Delta\epsilon'_r(T)$) and the determination coefficient (R^2) are also given. As mentioned above, the reference temperature is $T_0 = 318$ K.

From Tables 1 and 2 it may be seen that $\epsilon'_r(T_0)$ values of VO samples are between 3.03 (canola) and 3.18 (chia), while, for BD, $\epsilon'_r(T_0)$ is between 3.12 (soybean) and 3.26 (chia).

The determination coefficients, R^2 , show that in all cases the experimental data fit very well to the proposed model (Eq. (3)).

A plot of the fitting parameters, $\epsilon'_r(T_0)$ and $d\epsilon'_r/dT$, for olive, sunflower and corn VO and BD samples are shown in Fig. 2 (symbols). Dashed lines correspond to the uncertainty bands of the parameters (two standard deviations).

From Fig. 2 it is clear that the magnitudes of $\epsilon'_r(T_0)$ and $d\epsilon'_r/dT$ for BD samples are higher than those of VO of the same origin. As shown in previous works [9–14], this is to be expected, since the polarity of BD molecules is higher than in VO. Also, traces of polar substances (water, alcohol, etc.) might be present in BD samples. Although Fig. 2 shows the parameters of three species (olive, sunflower and corn), all the samples studied in this work show the same behavior.

The results of VO and BD electrical properties show that $\epsilon'_r(T_0)$ values can be used to discriminate between VO and BD samples with a common origin. Therefore, the measurement of electrical properties could be relevant to the on-line monitoring of transesterification, and also to detect non-transesterified VO in the final product.

The conductivity activation energy values, ΔE , of VO and BD samples are shown in Fig. 3. The representative values are given together with the uncertainty bands (one standard deviation).

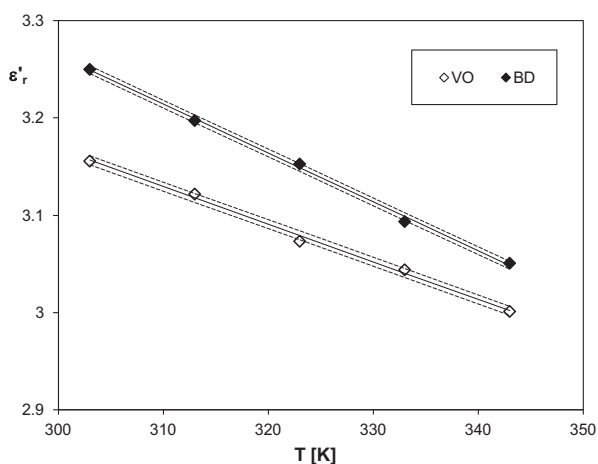


Fig. 1. Real part of permittivity (ϵ'_r) of samples of sunflower VO and BD as a function of temperature.

Table 1

Fitting parameters ($\epsilon'_r(T_0)$ and $d\epsilon'_r/dT$) with their uncertainties ($\Delta\epsilon'_r(T_0)$ and $\Delta d\epsilon'_r/dT$) for the permittivity in VO samples; the root mean squared uncertainty ($\Delta\epsilon'_r(T)$) and determination coefficient (R^2) are given for each fitting.

Origin	$\epsilon'_r(T_0 = 318 \text{ K})$	$\Delta\epsilon'_r(T_0)$	$d\epsilon'_r/dT$	$\Delta d\epsilon'_r/dT$	$\Delta\epsilon'_r(T)$	R^2
Sunflower	3.10	<0.01	-0.0039	0.0002	<0.01	0.996
Olive	3.07	<0.01	-0.0036	0.0002	<0.01	0.998
Chia	3.18	<0.01	-0.0043	0.0003	<0.01	0.996
Canola	3.03	<0.01	-0.0036	0.0002	<0.01	0.996
Corn	3.09	<0.01	-0.0042	0.0001	<0.01	0.999
Soybean	3.08	<0.01	-0.0039	0.0002	<0.01	0.997
Grape	3.15	<0.01	-0.0039	0.0001	<0.01	0.999

Table 2

Fitting parameters ($\epsilon'_r(T_0)$ and $d\epsilon'_r/dT$) with their uncertainties ($\Delta\epsilon'_r(T_0)$ and $\Delta d\epsilon'_r/dT$) for the permittivity in BD samples; the root mean squared uncertainty ($\Delta\epsilon'_r(T)$) and determination coefficient (R^2) are given for each fitting.

Feedstock	$\epsilon'_r(T_0 = 318 \text{ K})$	$\Delta\epsilon'_r(T_0)$	$d\epsilon'_r/dT$	$\Delta d\epsilon'_r/dT$	$\Delta\epsilon'_r(T)$	R^2
Sunflower	3.17	<0.01	-0.0050	0.0003	<0.01	0.998
Olive	3.13	<0.01	-0.0060	0.0001	<0.01	0.998
Chia	3.26	<0.01	-0.0049	0.0003	<0.01	0.998
Canola	3.17	<0.01	-0.0044	0.0003	<0.01	0.997
Corn	3.13	<0.01	-0.0052	0.0003	<0.01	0.997
Soybean	3.12	<0.01	-0.0044	0.0001	<0.01	0.999
Grape	3.17	<0.01	-0.0052	0.0007	<0.01	0.980

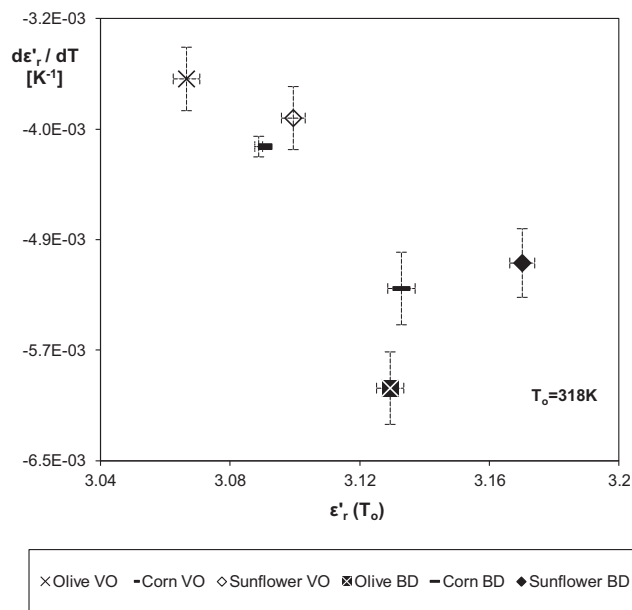


Fig. 2. Fitting parameters of permittivity ($\epsilon'_r(T_0)$ and $d\epsilon'_r/dT$) with their uncertainty bands, for VO and BD samples. Reference temperature is $T_0 = 318$ K.

As it can be seen from Fig. 3, there is a reduction of ΔE (approximately 60%) after the transesterification of VO into BD. The activation energy of BD samples is between 0.21 eV (chia) and 0.26 eV (soybean), while for VO it is between 0.32 eV (canola) and 0.46 eV (olive and soybean). The uncertainty of the activation energy of canola is higher because its conductivity is very close to the detection limit of the measuring system.

It is very important to remark that in this work dielectric spectroscopy was used to determine intensive properties that are intrinsic to the substance (permittivity and activation energy of conductivity). The calibration procedure separates the effects of the characteristics of the measuring cell from the electrical properties of the sample [9]. This is a fundamental difference with

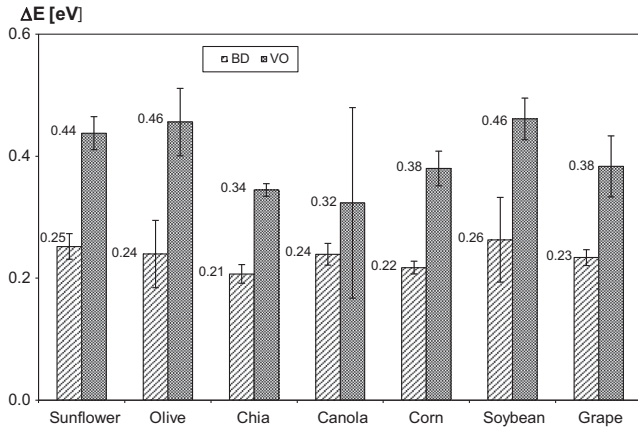


Fig. 3. Conductivity activation energy values (ΔE) of VO and BD samples.

impedance spectroscopy, where the geometry and characteristics of the electrodes influence directly the measured values of impedance (an extensive magnitude). This distinction is especially important at low frequencies and also for higher conductivity samples, where electrode polarization effects are appreciable, particularly when steel electrodes are used. Also, for measurements at higher frequencies (above 1 MHz), a careful calibration is necessary to correct the systematic errors that would be introduced by the connection cables.

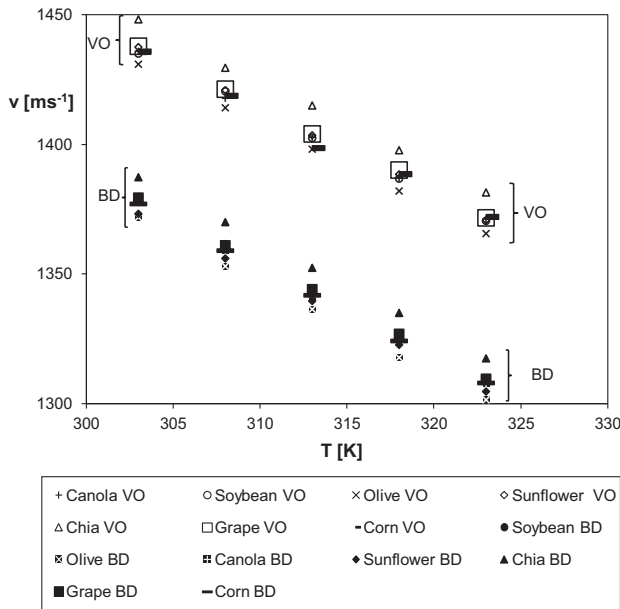


Fig. 4. Experimental values of the speed of sound (v) of VO and BD samples as a function of temperature. The measuring frequency was 9.3 MHz.

Table 3

Fitting parameters ($v(T_0)$ and dv/dT) with their uncertainties ($\Delta v(T_0)$ and $\Delta dv/dT$) for the speed of sound in VO; the root mean squared uncertainty ($\Delta v(T)$) and determination coefficient R^2 are given for each fitting.

Origin	$v(T_0 = 318 \text{ K}) \text{ (ms}^{-1}\text{)}$	$\Delta v(T_0) \text{ (ms}^{-1}\text{)}$	$dv/dT \text{ (ms}^{-1} \text{ K}^{-1}\text{)}$	$\Delta(dv/dT) \text{ (ms}^{-1} \text{ K}^{-1}\text{)}$	$\Delta v(T) \text{ (ms}^{-1}\text{)}$	R^2
Soybean	1386.7	0.4	−3.3	0.1	0.8	0.999
Olive	1381.9	0.1	−3.3	<0.1	0.2	0.999
Canola	1386.9	0.5	−3.2	0.1	0.9	0.999
Sunflower	1387.6	0.4	−3.3	<0.1	0.6	0.999
Chia	1397.8	0.5	−3.3	0.1	1.0	0.999
Grape	1388.6	0.6	−3.3	0.1	1.1	0.999
Corn	1386.9	1.5	−3.2	0.2	2.8	0.999

4.2. Speed of sound

Fig. 4 shows experimental values of the speed of sound, v , as a function of temperature for VO and BD samples (symbols). The measuring frequency was 9.3 MHz.

From Fig. 4 it is clear that the speed of sound for VO and BD decreases with temperature. Moreover, at all the measured temperatures, the speed of sound of VO is higher than for BD, independently of the origin. This behavior is relevant for the study of the progress of the transesterification reaction.

In Fig. 4, it may be seen that the speed of sound of VO from sunflower, soybean, canola, corn and grape are very close to each other, so their symbols appear superimposed. In the same fashion, the speed of sound of BD from canola, soybean, grape and corn are also very close to each other; therefore, in the plot of Fig. 4, their symbols are overlaid.

Experimental data were fitted to a linear model according to Eq. (5). Tables 3 and 4 show the fitting parameters ($v(T_0)$ and dv/dT), and their uncertainties ($\Delta v(T_0)$ and $\Delta dv/dT$) together with the determination coefficient (R^2), and the root mean squared uncertainty ($\Delta v(T)$), for each fitting.

From Tables 3 and 4 it can be seen that experimental data fit very well to the proposed linear model (Eq. (5)). The speed of sound, $v_0(T_0)$, at the reference temperature ($T_0 = 318 \text{ K}$) of VO samples is between 1398 ms^{-1} (chia) and 1382 ms^{-1} (olive). The relative uncertainty is below 0.1% for all the samples. The temperature coefficient, dv/dT , was between $-3.1 \text{ ms}^{-1} \text{ K}^{-1}$ (corn) and $-3.3 \text{ ms}^{-1} \text{ K}^{-1}$ (sunflower) with relative uncertainties below 6%.

The speed of sound, $v_0(T_0)$, at the reference temperature ($T_0 = 318 \text{ K}$) of BD samples was between 1335 ms^{-1} (chia) and 1318 ms^{-1} (olive) with relative uncertainties below 0.1%. The temperature coefficient dv/dT , was between $-3.4 \text{ ms}^{-1} \text{ K}^{-1}$ (sunflower) and $-3.5 \text{ ms}^{-1} \text{ K}^{-1}$ (olive) with relative uncertainties below 2%. It is worth noting that the speed of sound for BD at the reference temperature, $v_0(T_0)$, is about 5% lower than for VO samples, independently of the origin.

Fig. 5 shows $v_0(T_0)$ and of dv/dT of VO and BD samples and their uncertainties.

The fitting parameters of the speed of sound of VO and BD (Tables 3 and 4) are comprised in a narrow range. Then, it is possible to use speed of sound measurements, v , to discriminate between VO and BD samples, independently of their origin. This could be used to determine the transesterification progress. In contrast to EP, the fitting parameters of the speed of sound of VO and BD are grouped in two distinct sets, independently of the origin of the samples.

Measured values are compatible with those reported in literature [21–23] within experimental errors.

4.3. Correlation between electrical and acoustic properties

As shown in Sections 4.1 and 4.2, both the permittivity, ϵ_r' , and the speed of sound, v , of VO and BD may be assumed to depend

Table 4
Fitting parameters ($v(T_o)$ and dv/dT) with their uncertainties ($\Delta v(T_o)$ and $\Delta dv/dT$) for the speed of sound in BD; the root mean squared uncertainty ($\Delta v(T)$) and determination coefficient R^2 are given for each fitting.

Feedstock	$v(T_o = 318 \text{ K}) \text{ (ms}^{-1}\text{)}$	$\Delta v(T_o) \text{ (ms}^{-1}\text{)}$	$dv/dT \text{ (ms}^{-1} \text{ K}^{-1}\text{)}$	$\Delta(dv/dT) \text{ (ms}^{-1} \text{ K}^{-1}\text{)}$	$\Delta v(T) \text{ (ms}^{-1}\text{)}$	R^2
Soybean	1326.0	0.6	-3.5	0.1	1.0	0.999
Olive	1318.5	0.4	-3.5	0.1	0.7	0.999
Canola	1324.7	0.2	-3.4	0.1	0.4	0.999
Sunflower	1322.2	0.3	-3.4	0.1	0.5	0.999
Chia	1335.0	0.0	-3.5	0.1	0.1	0.999
Grape	1326.8	0.2	-3.5	0.1	0.4	0.999
Corn	1324.7	0.3	-3.5	0.1	0.5	0.999

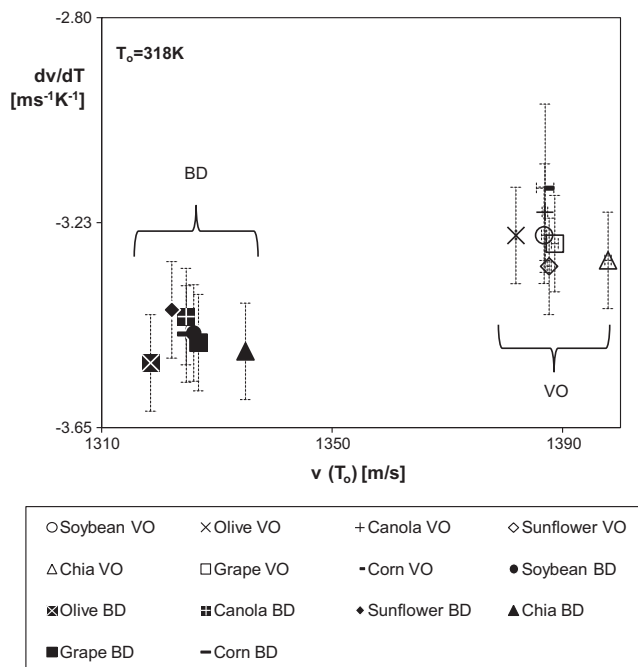


Fig. 5. Fitting parameters of the speed of sound ($v(T_o)$ and dv/dT) for VO and BD samples.

linearly on temperature, within the range studied in this work. Therefore, it is possible to write:

$$\varepsilon'_r(T) = \alpha + \beta v(T) \quad (6)$$

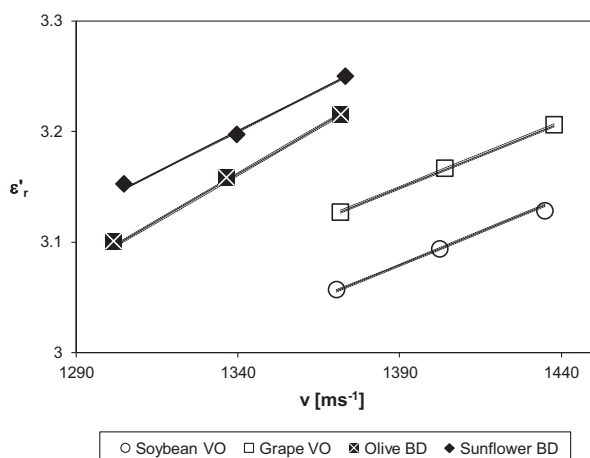


Fig. 6. Correlation between speed of sound (v) and permittivity (ε'_r) for VO and BD samples.

To verify this correlation, experimental values of ε'_r and v at 303 K, 313 K and 323 K were plotted in Fig. 6 (symbols), together with the linear dependence from Eq. (6) (continuous lines). The limits of the uncertainty bands were not shown in the graph, since they practically overlap with the continuous line. The constants α and β were obtained from the linear models of Eqs. (3) and (5), for soybean and grape VO, and olive and sunflower BD.

It can be seen that the correlation between ε'_r and v given by Eq. (6) is very satisfactory in the temperature range studied. This result is of interest for the development of sensors and control system applications.

5. Conclusions

Dielectric spectroscopy and ultrasonic interferometry techniques allow the determination of intrinsic properties of liquids in an accurate, fast and non-destructive way. In this work, soybean, corn, sunflower, olive, grape and chia vegetable oils and biodiesel samples were studied. This aims to ascertain the expected ranges of the electrical and ultrasonic properties in biodiesel and vegetable oils, and also the correlations between them. The results extend and generalize those reported in previous works and the literature.

The permittivity and the speed of sound in the samples fit very well to a linear dependence with temperature. When comparing the permittivities of biodiesel and vegetable oil feedstock at the reference temperature (318 K), the values of the former are about 1–5% higher. Also, the speed of sound in biodiesel is about 5% lower than in vegetable oils.

The conductivity in all the studied samples increases with temperature, following an Arrhenius law. The activation energy of conductivity for biodiesel is about 60% lower than in vegetable oils.

Measurements of permittivity and speed of sound allow discriminating between vegetable oils and biodiesel samples. Moreover, there is a linear relation between permittivity and speed of sound for vegetable oils and biodiesel, within the temperature range studied.

The determination of the activation energy of the conductivity is relevant for process control applications. In fact, conductivity measurements are often used in production plants to detect contaminants. However, for conductivity results to be meaningful in biodiesel and vegetable oil, it is necessary to refer measured values to the same temperature. The Arrhenius dependence of the conductivity may be used to compare measurements at different temperatures. This is useful for the detection of contaminants in the feedstock, during the production process and in the final product.

The results presented in this work suggest that measurement of electrical and acoustical properties could be used to monitor and control the transesterification process. This will be the subject of future articles.

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