

# Characterization of soybean biodiesel: Influence of alcohol type and molar ratio on speed of sound

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## ABSTRACT

Ultrasound techniques are widely used for monitoring chemical reactions and characterizing liquids. While the speed of sound in pure fatty acid esters (methyl and ethyl) is reasonably well established, the influence of molar ratio and alcohol type on the speed of sound in biodiesel across different frequencies remains unexplored. This study investigates the ultrasonic characterization of soybean biodiesel produced using different alcohols and molar ratios at frequencies of 1 MHz, 5 MHz, 7.5 MHz, and 10 MHz. The speed of sound in biodiesel samples was measured using the pulse-echo technique, with the expanded uncertainty determined at a 95 % confidence level. The results show that biodiesel samples produced with different alcohols at the same molar ratio exhibit distinct speed of sound values. Additionally, biodiesel synthesized at a 6:1 molar ratio demonstrated the lowest speed of sound. The findings confirm that ultrasound effectively differentiates biodiesel samples based on speed-of-sound measurements. This study highlights ultrasound as a promising technique for identifying biodiesel production pathways, offering a non-invasive and efficient analytical tool for quality assessment.

Section: RESEARCH PAPER

Keywords: biodiesel; speed of sound; metrology; measurement; ultrasound

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## 1. INTRODUCTION

The global search for alternative energy sources to meet the growing demand in the industrial and transport sectors has driven a significant increase in biofuel production. Biodiesel emerges as a promising substitute for conventional diesel, offering reduced environmental impact [1]. Also known as fatty acid methyl ester (FAME), biodiesel can be produced from animal fat, vegetable oil, and waste oil via the transesterification process [2]. It is typically produced from vegetable oils and animal fats, with transesterification being the most common production route [3]. Biodiesel is fully miscible with petrodiesel and can be used in modern diesel engines without significant modifications. The injection and combustion processes are crucial for engine efficiency and are strongly influenced by thermophysical properties such as density, viscosity, and speed of sound [4]. Therefore, accurate knowledge of these properties in biofuel and raw materials is of great importance for process optimization and performance prediction.

Quality control of biodiesel is essential, as several physical properties directly affect fuel performance and, consequently, injection and combustion efficiency [5], [6]. To ensure proper

engine operation, biodiesel must comply with established quality standards [7], [8]. Thus, identifying compositional variations in biodiesel is fundamental to ensure compliance with the regulatory specifications.

Ultrasound-based methods have gained relevance in both industrial and research settings due to their non-destructive, real-time, and low-cost nature. Ultrasonic techniques have been applied to assess the physical and acoustic properties of products and raw materials, identifying adulterations and monitoring chemical reactions [9]-[12]. In recent years, ultrasound techniques have been extensively used for monitoring reactions and characterizing liquids, with several studies focused on oils [9], [13], [14] and biodiesel [4], [15], [16].

Several studies have already explored the use of ultrasound to characterize biodiesel and related compounds. For instance, Freitas et al. [4] measured and predicted the speed of sound of biodiesel fuels, providing valuable reference data and thermodynamic insights. Similarly, Lopes et al. [16] investigated the speed of sound in pure fatty acid methyl esters (FAMEs) and biodiesel, highlighting the relationship between molecular structure and acoustic properties. More recently, Baesso et al.

[15] demonstrated the potential of ultrasonic parameter measurements as a practical tool for assessing biodiesel production quality, reinforcing the applicability of ultrasound as a low-cost and non-destructive technique.

Although these works have advanced the understanding of ultrasonic characterization of biodiesel, gaps remain regarding the influence of production conditions (alcohol type and molar ratio) on the speed of sound across different ultrasonic frequencies. Addressing these aspects is crucial to improve the use of ultrasound in biodiesel quality control and to establish stronger correlations with production variables.

Moreover, metrological aspects are rarely considered. Since no measurement result is completely free of uncertainty, a metrological approach is essential to provide a quantitative indication of result reliability. Measurement uncertainty is a key criterion for determining how close the obtained results are to the expected value. Incorporating uncertainty analysis and normalized error further enhances the reliability of biodiesel differentiation [17]-[19].

Therefore, the present study aims to evaluate the influence of alcohol type (methanol and ethanol) and molar ratio (6:1 and 8:1) on the speed of sound of biodiesel at frequencies of 1 MHz, 5 MHz, 7.5 MHz, and 10 MHz. By including a metrological treatment of the measurements, this work also contributes to improving the reliability and reproducibility of ultrasonic characterization methods.

## 2. MATERIAL AND METHODS

### 2.1. Biodiesel samples

Biodiesel samples were produced through the transesterification of soybean oil. Methanol and ethanol were used at different molar ratios (6:1; 8:1, and 10:1), while potassium hydroxide (KOH) was employed as a catalyst at a fixed concentration of 1.5 %. The reaction was carried out at 40 °C under mechanical stirring at 400 rpm throughout the entire transesterification process.

Table 1 summarizes the six production routes used in this study, designated as samples I, II, III, IV, V, and VI.

### 2.2. Speed of sound

The speed of sound (SoS) in biodiesel samples obtained with different alcohols and molar ratios (samples I, II, III, IV, V, and VI) was measured at frequencies of 1 MHz, 5 MHz, 7.5 MHz, and 10 MHz. Measurements were performed using the experimental setup described in [14] (Figure 1).

The measurement setup includes a thermal bath [a], a cell containing the biodiesel sample [b], and an ultrasonic transducer corresponding to each frequency [c], with an active element diameter of 12.7 mm (NDT-Panametrics, Olympus Corporation, Japan). The pulse-echo method was used for measuring SoS in biodiesel samples, in which the ultrasonic transducer acted as both transmitter and receiver, excited by a

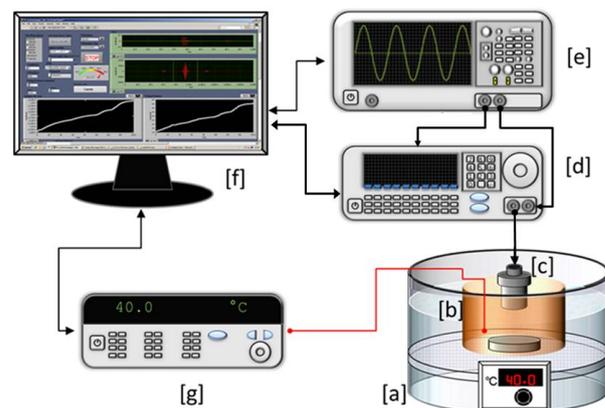


Figure 1. Experimental setup: (a) thermal bath, (b) glass vessel containing oil, (c) ultrasonic transducer, (d) waveform generator, (e) oscilloscope, (f) computer with a program developed in LabView™, and (g) data acquisition unit to measure sample temperature.

wave generator [d] model 33250A (Agilent Technologies, CA, USA). The acquired signals were digitized using an oscilloscope [e] model DSO-X 3012A (Agilent Technologies, CA, USA) and transferred to a computer [f] through a program developed in LabView (National Instruments, TX, USA). A data acquisition unit coupled to a thermocouple was used to continuously monitor the temperature of the sample [g]. Five replicates were performed under repeatability conditions.

### 2.3. Uncertainty analysis

The expanded uncertainty was calculated according to the Guide of the Expression of Uncertainty in Measurements [17], using a coverage factor  $k$  based on the t-distribution with a probability of 0.95 and the effective degrees of freedom. Measurement uncertainty was determined according to the type A (random) and type B (systematic) evaluation methods.

The speed of sound results and their associated uncertainties for the produced biodiesel were statistically compared by calculating the normalized error [18].

The  $E_n$  was calculated according to equation (1):

$$E_n = \frac{x_1 - x_2}{\sqrt{U_{x_1}^2 + U_{x_2}^2}}, \quad (1)$$

where  $x$  are the parameters of interest (e.g., SoS in sample) and  $U_x$  is the respective expanded uncertainty. If the absolute amount of the normalised error  $|E_n|$  is lower than and equal to one, the compared results are statistically equivalent. If  $|E_n|$  is larger than one, the results are not statistically equivalent.

## 3. RESULTS AND DISCUSSION

### 3.1. Comparison of different frequencies

The results of SoS as a function of frequency for the investigated samples, as well as their respective expanded ( $U$ ) and relative ( $U_{rel}$ ) uncertainties obtained from five replications, are presented in Table 2 and Table 3.

When increasing the frequency from 1 MHz to 5 MHz, the SoS decreases significantly in all investigated samples. From 5 MHz onwards, the changes are less accentuated. For the frequencies studied, the SoS values varied between 1422.1 m·s<sup>-1</sup> and 1392.2 m·s<sup>-1</sup> for methyl biodiesel (I, II, and III), and between 1409.6 m·s<sup>-1</sup> and 1384.8 m·s<sup>-1</sup> for ethyl biodiesel (IV, V, and VI). The uncertainties found were lower than 2.1 m·s<sup>-1</sup> (0.15 %).

Table 1. Biodiesel samples.

Sample	Alcohol type	Molar ratio
I	Methanol	6:1
II	Methanol	8:1
III	Methanol	10:1
IV	Ethanol	6:1
V	Ethanol	8:1
VI	Ethanol	10:1

### 3.2. Effect of molar ratio on speed of sound

Table 2 and Table 3 show the results of speed of sound as a function of frequency for methyl and ethyl biodiesel, respectively.

Analysing Table 2, it is noted that for all evaluated frequencies, the sample that presented the lowest speed of sound was sample I, being equal to  $1417.7 \text{ m}\cdot\text{s}^{-1} \pm 1.8 \text{ m}\cdot\text{s}^{-1}$  at 1 MHz and reaching  $1392.2 \text{ m}\cdot\text{s}^{-1} \pm 2.0 \text{ m}\cdot\text{s}^{-1}$  at 10 MHz.

Studies in the literature [20], [21] show that biodiesel with higher rates of conversion of triglycerides into esters have a lower speed of sound as a consequence of its composition. Based on this, biodiesel produced with an alcohol:oil molar ratio of 6:1 has a lower speed of sound and possibly a higher conversion rate. It is important to highlight that the 6:1 molar ratio is the most described and studied in literature, due to its optimal conversion rate.

Table 4 shows the results of the normalized error to evaluate the equivalence between the results presented in Table 2 and Table 3. According to the evaluation criteria, pairs that presented a normalized error of less than 1 were considered equivalent.

In addition, from Table 2 and Table 4, it is observed that at a frequency of 1 MHz, it was not possible to statistically differentiate the speed of sound between samples I and III ( $|E_n| = 0.88$ ) and between samples II and III ( $|E_n| = 0.93$ ). However, samples I (6:1) and II (8:1) were considered non-equivalent ( $|E_n| = 1.7$ ).

Table 2. Speed of sound as a function of frequency for biodiesel samples produced with methanol and different molar ratios.

Samples Methyl Biodiesel	Frequency in MHz	SoS	U	U <sub>rel</sub>
I [6:1]	1	1417.7	1.8	0.13
	5	1399.1	1.6	0.11
	7.5	1398.5	1.0	0.07
	10	1392.2	2.0	0.14
II [8:1]	1	1422.1	1.8	0.12
	5	1412.9	1.7	0.12
	7.5	1404.7	1.1	0.08
	10	1404.3	2.1	0.15
III [10:1]	1	1419.8	1.6	0.11
	5	1403.4	1.6	0.11
	7.5	1402.2	0.8	0.05
	10	1397.7	1.6	0.11

Table 3. Speed of sound as a function of frequency for biodiesel samples produced with ethanol and different molar ratios.

Samples Ethyl Biodiesel	Frequency in MHz	SoS	U	U <sub>rel</sub>
IV [6:1]	1	1395.6	1.6	0.11
	5	1390.4	1.7	0.12
	7.5	1388.6	1.6	0.12
	10	1387.8	1.7	0.12
V [8:1]	1	1401.8	1.7	0.12
	5	1395.0	2.0	0.14
	7.5	1387.8	1.7	0.12
	10	1385.3	1.1	0.08
VI [10:1]	1	1409.6	1.6	0.11
	5	1397.9	2.0	0.14
	7.5	1387.0	2.1	0.15
	10	1384.8	1.1	0.08

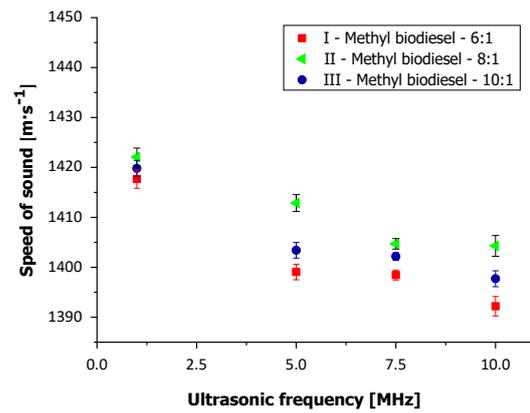


Figure 2. Speed of sound as a function of frequency for methyl biodiesel

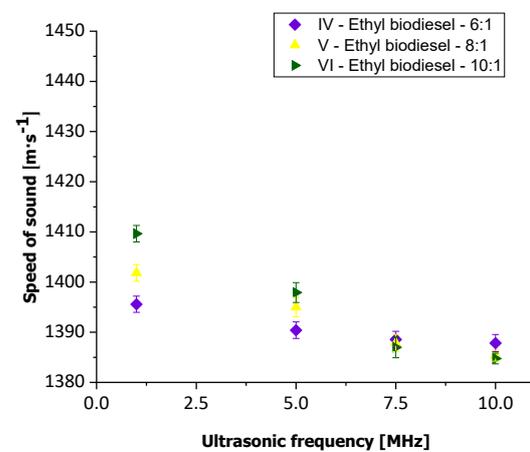


Figure 3. Speed of sound as a function of frequency for ethyl biodiesel.

At frequencies of 5 MHz, 7.5 MHz, and 10 MHz, it was possible to observe that samples I, II, and III were considered non-equivalent ( $|E_n| > 1$ , for all pairs), demonstrating that the ultrasonic technique is capable of distinguishing samples of methyl biodiesel produced from different molar ratios at frequencies higher than 5 MHz.

Unlike the results presented for methyl biodiesel, Figure 3 shows that at a frequency of 1 MHz, it was possible to observe a statistical difference in the speed of sound between samples IV, V, and VI ( $|E_n| > 1$ , for all pairs). At frequencies of 5 MHz, 7.5 MHz, and 10 MHz, most samples were considered statistically equivalent ( $|E_n| < 1$ , Table 4).

The obtained results demonstrated that it is possible to differentiate the three samples of ethyl biodiesel produced from different molar ratios only at 1 MHz. In the same way as observed in methyl biodiesel, for ethyl biodiesel, the sample that presented the lowest speed of sound was the sample produced with a molar ratio of 6:1 (IV).

### 3.3. Effect of alcohol type on speed of sound

Figure 4 shows the speed of sound as a function of frequency for all biodiesels produced (methyl and ethyl) at different molar ratios.

Normalized error values were calculated between pairs of biodiesels produced from different types of alcohol, to

Table 4. Normalized error for methyl (I, II, and III) and ethyl (IV, V, and VI) biodiesel samples.

$ E_n $	1 MHz	5 MHz	7.5 MHz	10 MHz
[I - II]	1.7	6.0	4.0	4.4
[I - III]	<u>0.88</u>	2.0	3.0	2.2
[II - III]	<u>0.93</u>	4.1	1.6	2.7
[IV - V]	4.9	<u>0.23</u>	<u>0.30</u>	1.2
[IV - VI]	8.5	<u>0.90</u>	<u>0.60</u>	1.5
[V - VI]	3.4	1.0	<u>0.33</u>	<u>0.36</u>

Table 5. Normalized error for biodiesel samples produced from different alcohols.

Frequency MHz	$ E_n $ [I - IV]	$ E_n $ [II - V]	$ E_n $ [III - VI]
1	11.1	8.2	4.5
5	1.5	6.9	2.2
7.5	5.2	8.1	6.9
10	1.7	8.3	6.7

statistically identify which samples can be considered equivalent in terms of propagation velocity values (Table 5).

Analysing the data in Table 5, it is observed that the biodiesel samples produced from different alcohols and the same molar ratio (I-IV), (II-V), and (III-VI) presented a normalized error greater than 1 for all the investigated frequencies. That is, even after the purification process, biodiesel produced with ethanol showed a lower speed of sound. This could be explained by the decrease in viscosity.

These results demonstrate that the proposed technique is a promising tool for distinguishing biodiesel produced through different routes. Furthermore, the method revealed that biodiesels produced from different alcohols at the same molar ratio exhibit distinct characteristics. The speed of sound results obtained in this study using methanol and a 6:1 molar ratio are consistent with previous ultrasonic characterization of biodiesel [12], [14]. The results for the other production routes and frequencies could not be compared with the literature, due to the lack of available studies. Compared to traditional analytical methods, the ultrasonic approach is faster, non-invasive, and does not require the use of solvents, highlighting its potential for real-time quality control in biodiesel production.

#### 4. CONCLUSIONS

Biodiesel samples obtained from different types of alcohol and molar ratios were characterized using low-power ultrasound. The speed of sound was measured at frequencies of 1 MHz, 5 MHz, 7.5 MHz, and 10 MHz. Methyl biodiesel samples were best distinguished at frequencies of 5 MHz, 7.5 MHz, and 10 MHz, whereas ethyl biodiesel samples were most clearly differentiated at a frequency of 1 MHz. The results demonstrated that biodiesels produced with different alcohols have non-equivalent speed of sound. Regarding the molar ratio, for both methanol and ethanol, the sample produced at a 6:1 molar ratio presented the lowest speed of sound.

Overall, ultrasound proved capable of differentiating biodiesel samples based on the speed of sound parameter. This approach could serve as a solvent-free technique for biodiesel quality assessment. This study highlights ultrasound as a non-invasive and efficient tool for biodiesel quality assessment, offering a potential alternative to traditional chemical analysis.

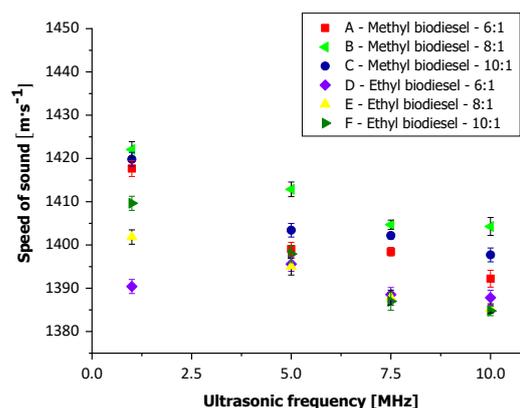


Figure 4. Speed of sound as a function of frequency for all biodiesels produced (I-VI).

Furthermore, the method shows potential for industrial applications, such as real-time monitoring and quality control during biodiesel production, offering a fast, efficient, and reliable alternative to traditional chemical analyses.

Future studies could expand this approach to biodiesel produced from alternative feedstocks, investigate the effect of additional ultrasonic frequencies, and explore correlations between ultrasonic parameters and conventional chemical characteristics, to enhance process monitoring and deepen the understanding of biodiesel properties.

#### AUTHORS' CONTRIBUTION

Pâmella A. Oliveira: conceptualization, formal analysis, investigation, methodology, and writing – original draft.

Raphaella M. Baesso: conceptualization, methodology, and writing – review & editing.

Rodrigo P. B. Costa-Félix: project administration, software, supervision, and writing – review & editing.

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