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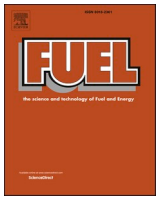

Full Length Article

Ultrasound is a powerful tool for monitoring real-time online transesterification reaction

P.A. Oliveira [a,c,d,e], R.M. Baesso [b], M.I. Nunes [c], J.A.P. Coutinho [d], R.P.B. Costa-Félix [a,e,*]

[a] Laboratory of Ultrasound, National Institute of Metrology, Quality, and Technology (Inmetro), Duque de Caxias, RJ, Brazil
[b] Ultrasound and Underwater Acoustics Group (UUA), National Physical Laboratory (NPL), Hampton Rd, Teddington TW11 0LW, UK
[c] Centre for Environmental and Marine Studies, Department of Environment and Planning, University of Aveiro, Portugal
[d] Centre for Research in Ceramics and Composite Materials (CICECO), Department of Chemistry, University of Aveiro, Portugal
[e] Post-graduation Program on Biotechnology, National Institute of Metrology, Quality and Technology, Duque de Caxias, RJ 25250-020, Brazil

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ABSTRACT

Biodiesel is an increasingly crucial alternative energy source, requiring efficient monitoring of production processes to ensure quality and economic viability. Conventional techniques for monitoring the transesterification reaction are often time-consuming, destructive, and unsuitable for real-time applications. This study demonstrates the potential of quantitative ultrasound as a real-time, non-destructive, and cost-effective approach for monitoring biodiesel production from soybean–castor oil blends (80:20, 70:30, 60:40, and 50:50 wt%). An ultrasonic pulse-echo method at 1 MHz was employed, and the speed of sound (SoS) was measured alongside fatty acid methyl ester content, viscosity, and density. A strong correlation was observed between SoS and FAME content ($R2 \approx 0.97$), supporting the method's applicability for real-time monitoring. SoS values ranged from 1365.7 ± 4.4 m·s^{-}1 to 1403.7 ± 1.8 m·s^{-}1, increasing with higher castor oil content, with statistically significant differences observed between the studied blends ($p < 0.05$). In contrast, viscosity (4.2 – 11.6 mPa•s) and density (897.0 – 927.7 kg•m$^{-3)}$ distinguish pure oils but could not differentiate between the blends. These findings highlight quantitative ultrasound as a reliable, sensitive and innovative tool for real-time biodiesel monitoring, with clear advantages over conventional methods and potential for industrial implementation, as it can support quality control while reducing analysis time and operational costs.

1. Introduction

Biodiesel is one of the most important liquid biofuels for partially replacing petroleum-based diesel fuels. In recent years, it has attracted international attention due to its favourable fuel properties, biodegradability, and compatibility with diesel engines [1,2]. Biodiesel is typically produced through the catalytic transesterification of vegetable oils or animal fats with short-chain alcohols [3]. Despite the increasing focus on second and third-generation biofuels, edible oils such as soybean, palm, and rapeseed remain dominant in industrial biodiesel production. However, large-scale use of edible oils can lead to imbalances in the global food market. To address these concerns, non-edible oilseeds have been increasingly investigated as alternative biodiesel feedstocks [4–7].

The castor oil plant (*Ricinus communis L.*) is one of the most promising non-edible crops due to its high annual seed production and oil yield. Its seeds contain 40 –55 % oil, higher than most oil-producing seeds [8]. Moreover, castor oil is cost-effective and requires minimal agricultural inputs [9]. The primary component of castor oil is ricinoleic acid (80 –90 %), an unsaturated hydroxy–fatty acid, accompanied by approximately 10 % non-hydroxylated fatty acids such as oleic and linoleic acids. The hydroxyl group (–OH) in ricinoleic acid increases intermolecular hydrogen bonding, leading to higher oil viscosity and posing a challenge for its direct use in biodiesel production [10,11]. Nevertheless, biodiesel derived from castor oil exhibits excellent lubricity, making it a valuable component when used in appropriate concentrations [11,12].

Castor oil can be blended with edible oils such as soybean oil to overcome viscosity limitations and explore industrially relevant applications. Soybean oil was chosen because it remains one of the most

* Corresponding author.
E-mail address: rpfelix@inmetro.gov.br (R.P.B. Costa-Félix).

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widely used industrial feedstocks for biodiesel, serving as an appropriate benchmark for evaluating monitoring methods. Blending castor oil with soybean oil produces biodiesel with lower viscosity, meeting standard specifications while reducing the need for post-production blending [13]. This approach enables the study of compositional effects on biodiesel production. It aligns with industrial interest in reducing reliance on edible oils, enhancing feedstock sustainability, and facilitating cost-effective large-scale biodiesel production.

In industrial biodiesel production, continuous monitoring of the transesterification reaction is crucial for optimising chemical and energy usage and facilitating purification to ensure product quality [14–17]. Ensuring reliable and traceable measurements is essential for process control, highlighting the need for methods integrating metrological principles in real-time monitoring. Traditional analytical techniques, including gas chromatography (GC) [18], nuclear magnetic resonance (^{1}H NMR) [19–23], and infrared (IR) spectroscopy [22–26], are highly precise but time-consuming and unsuitable for real-time process monitoring. Other approaches, such as density measurements [27] and laser spectroscopy [28], also require offline analysis over extended periods. These limitations hinder adaptation to industrial online implementation and rapid quality control [29].

Ultrasound techniques have emerged as a promising alternative for characterising and monitoring the physical and acoustic properties of liquids and chemical reactions[30–33]. Ultrasound offers non-destructive, rapid, cost-effective and user-friendly analysis, making it suitable for real-time process integration. Changes in ultrasonic parameters, such as the speed of sound (SoS), attenuation, and acoustic impedance, are directly influenced by fundamental physical properties of the medium, such as density, viscosity and compressibility. The SoS increases with higher density and decreases with greater compressibility, while attenuation reflects energy dissipation due to viscous and structural losses [34,35]. Variations in these parameters allow in situ quality assessment and provide a non-invasive method for process control [36–39].

While previous research has demonstrated the potential of ultrasound in monitoring transesterification reactions, most of these studies have focused on pure oils. The evaluation of blends of edible and non-edible feedstocks, particularly those of industrial relevance, remains limited. There is a lack of comprehensive studies on its application to various oils and their blends. Furthermore, few studies address metrological aspects and measurement uncertainty, which are essential for process reliability and traceability [31–41].

This study aims to evaluate the performance and reliability of quantitative ultrasound for real-time monitoring of transesterification reactions. Methanol was used as the alcohol, with soybean and castor oils as the sources of triglycerides, and potassium hydroxide as the catalyst. Reactions were conducted using pure oils and their blends, and the progress was monitored using offline and online techniques. By adopting a metrological approach, this research seeks to demonstrate the performance and reliability of ultrasound for real-time biodiesel monitoring, thereby contributing to more sustainable and efficient production technologies.

2. Materials and methods

2.1. Materials

The refined soybean oil supplied by Prio Energy (Portugal) was measured and found to have a free fatty acid (FFA) content of 0.27 % ± 0.02 %. The commercially available castor oil, as a refined commercial product (Arganour), is expected to have a similarly low FFA content (<3%), so no esterification step was required before transesterification. Potassium hydroxide, magnesium sulphate and methanol were purchased from Sigma-Aldrich (St Louis, MO, USA). For the sample derivatisation for characterisation in GC, the following reagents were used: pyridine and N,O-Bis(trimethylsilyl) acetamide (Panreac Aplichem, Barcelona, ES), chlorotrimethylsilane (Acros Organics, Geel, BE), and Methyl heptadecanoate (Sigma-Aldrich).

Pure soybean and castor oils and four blends (80:20, 70:30, 60:40 and 50:50 wt%) were selected for the study (Table 1). Soybean oil is edible and widely available, whereas castor oil is non-edible and highly viscous. The blend ratios (from 20 to 50 wt% castor oil) were chosen to provide a stepwise increase in non-edible oil content, enabling systematic evaluation of QUS performance and sensitivity. Blend ratio exceeding 50 wt% castor oil was not used, as it produces biodiesel with excessively high viscosity, exceeding standard specifications [13,42]. Additionally, a reaction using 100 % castor oil was performed to confirm that the speed of sound (SoS) method remains reliable for high-viscosity biodiesel products.

2.2. Monitoring of transesterification by QUS

Transesterification reactions were performed in a 450 mL glass batch reactor equipped with a mechanical stirrer (Mod 713D – Fisatom, SP, Brazil) and a thermal bath (Mod 557 – Fisatom, SP, Brazil) set at 40 °C. Methanol was employed at a 6:1 oil-to-methanol molar ratio with potassium hydroxide as the catalyst (1.5 wt% relative to oil mass), which chemically activates the reaction by forming methoxide ions that promote transesterifications. The excess methanol (relative to the 1:3 stoichiometric ratio) was applied to shift the equilibrium towards biodiesel and improve conversion. At the same time, the chosen catalyst concentration lies within the optimal range reported in the literature, providing high efficiency and avoiding excessive soap formation [43]. These widely used and industrially relevant conditions were not further optimised, as this study focused on evaluating quantitative ultrasound for real-time monitoring.

The oil blend was loaded into the reactor, and once it reached 40 °C, the pre-heated alcohol-catalyst mixture (also at 40 °C) was added. Ultrasound monitoring was started with the stirring at 400 rpm for 40 min, a reaction time selected from the literature [44] to ensure sufficient triglyceride conversion. The reaction was conducted at 40 °C to provide effective kinetics while minimising methanol evaporation and side reactions [7]. The temperature was maintained constant (standard deviation within ± 0,24 °C) using the thermostated bath and monitored with a thermocouple to ensure that variations in the speed of sound (SoS) reflect changes in the reaction medium rather than temperature fluctuations.

The transesterification reactions of the different routes described in Table 1 were monitored by ultrasound to evaluate the effect of oil type on the SoS. For each reaction route, five successive repetitions were performed under repeatability conditions.

An ultrasonic transducer with a nominal frequency of 1 MHz and a diameter of 12.7 mm, model A303S (Olympus Panametrics – Olympus Corporation, Japan), was used for monitoring. This frequency was selected based on previous studies [32], as it offers an optimal compromise between minimising ultrasonic signal attenuation and maintaining sufficient temporal resolution for accurate biodiesel measurements. Ultrasound transducer calibration is not necessary for the method proposed in this paper. For other applications, however, the

Table 1
Composition of soybean and castor oil blends used in this work.

Route	Blend of soybean/castor oils reference	Percentage of soybean oil [wt.%]	Percentage of castor oil [wt. %]
A	SC-80	80	20
B	SC-70	70	30
C	SC-60	60	40
D	SC-50	50	50
E	S-100	100	0
F	C-100	0	100

S = soybean oil; C = castor oil; SC = blends of soybean and castor oil.

transducer and the excitation system can be calibrated in terms of effective radiation area [45], ultrasonic field [46], and power [47,48]. The arbitrary function generator (model 33250A, Agilent Technologies, CA, USA) excited the transducer with a pure tone using a 3-cycle burst at 20 V peak-to-peak amplitude. The echo signal was digitised by an oscilloscope (model DSO-X 3012A, Agilent Technologies, CA, USA), and the data were transferred to a computer running a program developed in LabVIEW^{TM} (Laboratory Virtual Instrument Engineering Workbench) (National Instruments, TX, USA), as illustrated in Fig. 1.

The experiments were conducted using the pulse-echo method, in which the transducer emits an ultrasonic pulse that propagates through the medium, reaches a reflector target, and returns to the same transducer. This configuration allows measurements directly within the reaction vessel, using a single-sided setup that simplifies transducer alignment and avoids the need for two-sided access, which is required in through-transmission techniques. The time taken for the pulse to complete this round trip is defined as the time of flight (ToF). ToF was assessed using a cross-correlation approach, where the measured waveform was correlated with the first echo, and the time difference between the maximum peaks was used to calculate ToF. Uncertainty in ToF was estimated from repeated measurements under identical conditions (Type A), while Type B uncertainty was obtained from the calibration certificate of the oscilloscope (0.0009 %). These uncertainties were propagated to determine the corresponding uncertainty in the (SoS) measurements.

The speed of sound in the reaction medium (SoS_{RM}) was calculated according to Eq. (1):

$$SoS_{RM} = \frac{d}{\frac{ToF}{2}} \quad (1)$$

In Eq. (1), d is the distance between the transducer face and the reflector target, in meters [m], and ToF is the time of flight in the reaction mixture under analysis, measured in seconds [s]. The distance d was determined as a function of the transmission delay, using distilled water as the reference propagation medium, according to Eq. (2):

$$d = SoS_W \bullet \frac{ToF_W}{2} \quad (2)$$

where SoS_w is the speed of sound in water [m·s^{-1}] and (ToF_w) is the time of flight of the ultrasonic wave in water, measured in seconds [s]. Fig. 1 discloses schematically the experimental setup.

2.3. Monitoring of transesterification by offline techniques

Transesterification reactions were conducted in a 500 mL batch reactor that was closed and without a condenser. The reactor was jacketed with a thermal insulator to keep the temperature of the reaction mixture. The reactor lid featured two-hole openings: one for inserting the temperature sensor and the other for sampling aliquots at different reaction times without opening the reactor. The reactions were carried out with stirring at 400 rpm.

Initially, vegetable oil or an oily blend was added to the reactor, and magnetic stirring and heating commenced. Once the oil reached the predetermined temperature, the potassium methoxide mixture (pre-

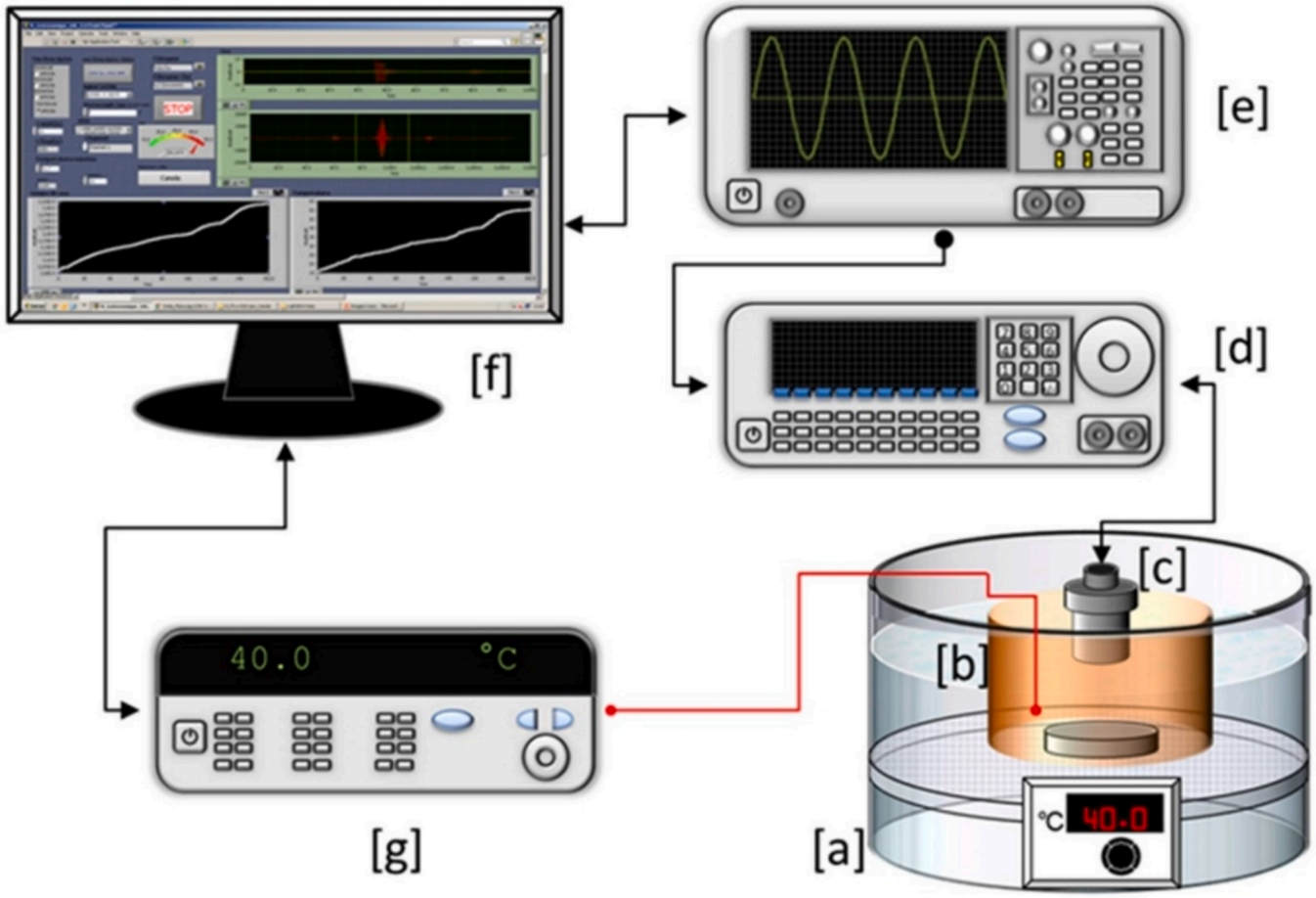

Fig. 1. An experimental setup was used for online monitoring of the transesterification reactions. [a] thermal bath, [b] glass batch reactor, [c] ultrasonic transducer, [d] function generator, [e] oscilloscope, [f] computer, and [g] data acquisition unit to monitor temperature.

heated to 40 °C) was added over a 3 to 5 s interval. Time zero was defined as when the alcohol/catalyst mixture was introduced into the reactor. The assays were performed under the same operating conditions used for ultrasound monitoring: 40 °C, 400 rpm and 40 min. This experimental procedure preceded each characterisation (viscosity, density, and GC analysis). Sample collection and processing details are described in the following sections.

2.3.1. Viscosity and density measurements

In biodiesel production, viscosity (η) and density (ρ) may change as the reactions progress, depending on the feedstock used. The viscosity and density of soybean–castor oil blends were measured during the transesterification reaction to monitor these variations and compare them with the data obtained from online ultrasound monitoring.

Viscosity and density were determined at 40 °C using an automated viscometer-densimeter (model SVM 3000, Anton Paar). Aliquots (4 mL) were collected from the reaction medium using a syringe and immediately injected into the viscometer-densimeter. The apparatus components (tube and rotor) were cleaned with ethanol and acetone before each measurement. Due to the time required for the cleaning, aliquots were collected and analysed at 8-minute intervals.

The instrument, previously employed for characterising various fluids, including biodiesel, exhibited good repeatability and reproducibility [49]. According to the manufacturer, the precision for viscosity measurements is 0.1 % and 0.35 % for repeatability and reproducibility, respectively, while for density measurements it is 0.00005 g·cm^{-}3 and 0.0001 g·cm^{-}3, respectively. In the present study, the measurements were conducted directly in the reaction mixtures. Three repetitions were performed for each route to determine the reaction medium's viscosity and density measurement uncertainty.

These measurements were carried out to evaluate the behaviour of viscosity and density during the reactions and to compare them with speed of sound (SoS) monitoring results, rather than to establish an explicit mathematical correlation with SoS.

2.3.2. Gas chromatography analysis

Offline monitoring of the transesterification reaction was performed using GC according to EN 14103:2020 [18] standard, a widely applied method for FAME analysis in biodiesel research. Analyses were carried out on a Trace 1300 GC Chromatograph (Thermo Scientific) equipped with a Varian CP Select™ Biodiesel for FAME capillary column (30 m, 0.32 mm, 0.10 μm) and a flame ionisation detector (FID). The capillary injection was maintained at 250 °C with a split ratio of 20:1, and a sample volume of 0.5 μL, using helium as the carrier gas. The column temperature program was as follows: initial temperature of 120 °C, ramped to 200 °C at 5 °C•min^{-1}.

FAME reference materials were used to prepare calibration standards, which were diluted in n-hexane to cover the expected concentration range of the samples. Calibration curves were constructed from these standards, and an internal standard solution (10 kg m^{-3} in n-hexane) was added to each sample to correct for variations in sample preparation and injection.

During the transesterification reactions, aliquots of 30 μL were collected at different times (2, 5, 10, 20, and 40 min). Each aliquot was derivatised with 100 μL of pyridine, 100 μL of N_2O-Bis(trimethylsilane) trifluoroacetamide and 50 μL dichlorotrimethylsilane. This silylation of hydroxyl groups halts the reaction, increases the volatility of hydroxy compounds, and improves their detection [50]. After derivatisation, 50 μL of the sample was transferred to a vial, weighed, and 100 μL of internal standard solution was added. The final solution was injected into the GC, and the peak areas were integrated.

This method allowed the determination of both total FAME content and individual FAME components; however, in this study, the analysis focused on the total FAME content to monitor the overall progress of the transesterification process. GC/FID data were processed using the Xcalibur software (version 2.0, Thermo Fisher Scientific, Massachusetts, USA).

2.4. Uncertainties and statistical analyses

Measurement uncertainty plays a crucial role in validating the ultrasound technique for analysing the transesterification of oil blends. All measurement uncertainties were evaluated according to the Guide to the Expression of Uncertainty in Measurement (GUM), using both type A (random) and type B (systematic) evaluation methods [51]. Reported results correspond to the standard uncertainty multiplied by a coverage factor, ensuring a coverage probability of 95 % (corresponding to a significance level of $\alpha = 0.05$).

To assess the equivalence of results, the statistical analysis of the normalised error (E_n) was employed [52]. The method was applied to compare the parameters obtained under different experimental conditions, allowing us to determine whether the results can be considered statistically equivalent while taking the measurement uncertainty into account, and consequently to identify whether the speed of sound can distinguish the different reaction pathways. The normalised error is assessed according to Eq. (3):

$$E_n = \frac{(x_1 - x_2)}{\sqrt{{U_{x_1}}^2 + {U_{x_2}}^2}} \tag{3}$$

where x_1 and x_2 are the values of the speed of sound and U_{x1} and U_{x2} are their expanded uncertainties (confidence level p = 0.95). According to this criterion, results are not considered statistically equivalent when $E_n > 1$, and statistically equivalent when $E_n \leq 1$. This approach inherently incorporates measurement uncertainty, thereby enabling rigorous comparison of results without the need for post-hoc adjustments for multiple testing.

3. Results and discussion

3.1. Speed of sound for transesterification monitoring

The effect of oil type on SoS was evaluated for the routes listed in Table 1, and the results are presented in Table 2 and Table 3, as well as in Fig. 2.

See Fig. 2 shows that at "time 0," the SoS is higher because the reaction medium initially consists of pure oil or oil blends. One can note that the similar SoS values observed for specific blends at this stage (e.g., 50–60 % or 70–80 % castor oil) reflect the initial physical properties of the mixtures. Although castor oil has higher viscosity than soybean oil, the SoS depends primarily on density and compressibility, which are very similar between blends with close compositions. As the reaction progresses, the decrease in SoS reflects compositional changes within the reaction medium. For routes involving oil blends (Routes A to D), a higher concentration of castor oil in the mixture corresponds to increased oil solubility. After 40 min of reaction, SoS = 1369.0 ± 3.9 m•s^{-1} for Route A and SoS = 1388.2 ± 1.4 m•s^{-1} for Route D, resulting in a Δ SoS ≈ 19 m•s^{-1}, as shown in Table 2. The SoS measurements for different blends were statistically distinct ($E_n > 1$), as shown in Table 4.

When comparing Route A (80 % soybean oil and 20 % castor oil) with Route E (100 % soybean oil), no significant statistical difference in SoS was observed at any reaction times ($E_n \leq 1$). At the end of the reaction, the SoS values were 1369.0 ± 3.9 m•s^{-1} for Route A (Table 2) and 1365.7 ± 4.4 m•s^{-1} for Route E (Table 3), resulting in a difference of only $\Delta v \approx 3.3$ m•s^{-1}. In contrast, Routes B, C and D showed statistically significant differences compared to Route E (E_n = 2.04, 2.94 and 4.87). These results indicate that ultrasound measurements of SoS can effectively differentiate transesterification reactions with varying oil mixtures, specifically highlighting the impact of incorporating 30 wt% castor oil into soybean oil. The observed differences in SoS can be attributed to the fatty acid composition of the triglycerides, which affects the density and compressibility of the reaction medium. Variations

Table 2
Results of the speed of sound measurements for reaction mixtures with various soybean and castor oil blends.

Reaction time[min]	Route A SC-80 SoS [m•s^{-1}]	U* [m•s^{-1}]	Route B SC-70 SoS [m•s^{-1}]	U* [m•s^{-1}]	Route C SC-60 SoS [m•s^{-1}]	U* [m•s^{-1}]	Route D SC-50 SoS [m•s^{-1}]	U* [m•s^{-1}]
0	1443.3	1.5	1441.1	2.6	1460.7	3.4	1461.7	3.4
1	1374.3	3.6	1377.5	3.8	1389.3	2.1	1385.9	3.2
5	1371.4	3.1	1377.1	2.5	1384.6	3.5	1388.6	2.5
10	1370.5	3.1	1376.4	2.2	1383.2	2.2	1388.6	2.4
20	1369.9	3.1	1376.0	2.5	1382.3	1.8	1388.4	1.9
40	1369.0	3.9	1376.0	2.5	1381.5	3.1	1388.2	1.4

* U = expanded uncertainty (p = 0.95); Temperature monitored during reactions: 40.0 ± 0.24 °C (standard deviation).

Table 3
Results of the speed of sound measurements for reaction mixtures with pure soybean (S-100) and castor oils (C-100).

Reaction time [min]	Route E S-100 SoS [m•s^{-1}]	U* [m•s^{-1}]	Route F C-100 SoS [m•s^{-1}]	U* [m•s^{-1}]
0	1419.0	2.6	1492.3	2.9
1	1369.4	5.4	1397.4	1.4
5	1367.7	4.8	1401.1	1.6
10	1367.3	4.9	1402.9	1.6
20	1366.8	5.0	1403.7	1.4
40	1365.7	4.4	1403.7	1.8

* U = expanded uncertainty (p = 0.95); Temperature: 40.0 ± 0.24 °C (standard deviation).

in chain length and degree of unsaturation influence intermolecular interactions and molecular packing, leading to measurable changes in the medium's acoustic properties. That comment is supported by Daridon et al. [53], who observed a linear increase in the molar compressibility with molecular weight for FAMEs and fatty acid ethyl esters (FAAE). Consequently, the SoS reflects the specific composition of the oil mixture and provides a means to differentiate the reaction routes. Although free fatty acids (FFAs) can influence ultrasonic propagation, their content remained low and within standard limits in this study and thus did not significantly affect the observed SoS variations.

For the routes with pure oil, at the end of the reaction, v = 1365.7 m•s^{-1} ± 4.4 m•s^{-1} was registered for soybean oil (Route E), and v = 1403.7 m•s^{-1} ± 1.8 m•s^{-1} for castor oil (Route F) (Table 3). It demonstrates that reactions involving different oils exhibit distinct ultrasonic properties, likely due to the varying types and concentrations of fatty acids in the oils, as castor oil contains over 88 % ricinoleic acid (C18:1) while soybean oil has more than 53 % linoleic acid (C18:2) [54]. Notably, the speed of sound (SoS) method reliably monitored reactions even for 100 % castor oil, which has a much higher viscosity than the blends. It confirms the technique's robustness across a wide range of viscosities, while the selected blend ratios ensure industrial relevance.

To the best of the authors' knowledge, there are no existing SoS measurements in the literature for transesterification reactions of

Table 4
Normalised error for blends (Route A to D) listed in Table 1.

Routes	Normalised Error (E_n)
A – B	1.51
A – C	2.51
A – D	4.63
B – C	1.38
B – D	4.26
C – D	1.97

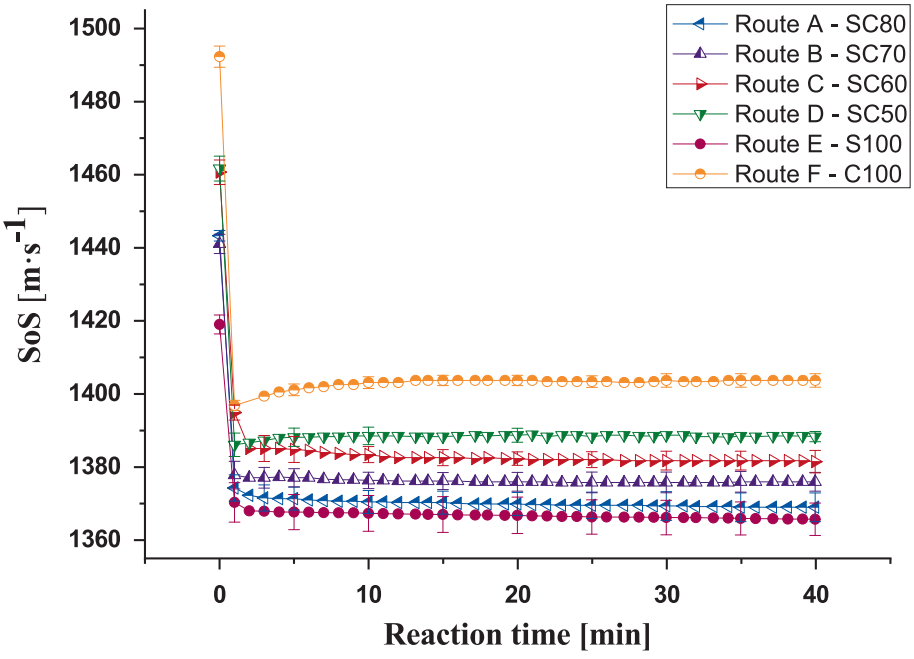

Fig. 2. Speed of sound (SoS) over time during the transesterification of soybean and castor blends at different ratios (SC80 to SC50), and pure soybean (S100) and castor oils (C100).

soybean and castor oil blends at different ratios to compare with the results obtained in this study. Quantitative ultrasound effectively monitored the transesterification reaction of soybean and castor oil blends across the studied ratios (ranging from 20 wt% to 50 wt% castor oil). It successfully differentiated between reactions produced from various oil types (S100 and C100).

3.2. Profiles of speed of sound, viscosity and density during transesterification

See Fig. 3 presents the temporal evolution of viscosity and density during the transesterification of oil blends (Route A –D), as well as for pure soybean (Route E) and castor (Route F) oils.

As shown in Fig. 3, both viscosity and density increase with higher castor oil content in the initial feedstock. Fig. 3a reveals a slight decrease in viscosity during the first 9 min of the reaction, with no significant changes thereafter. Viscosities for Routes A – D are considered metrologically equivalent, whereas density measurements for the pure oils (Routes E and F) are not metrologically equivalent to those of the blends. Route F, which employs pure castor oil, exhibits substantially higher dynamic viscosity, attributed to its elevated ricinoleic acid (C18:1) content. The hydroxyl groups in ricinoleic acid will likely promote hydrogen bonding with neighbouring molecules, thereby increasing viscosity [55,56].

Regarding density (Fig. 3b), Routes A–D are statistically equivalent. After 40 min of reaction, the density of Route E, (soybean oil, $\rho = 897.0$ kg•m^{-3}) is lower compared to Route F (castor oil, $\rho = 927.7$ kg•m^{-3}). Consistent with the viscosity trend, Route F shows distinctly higher density, reflecting the intrinsic physicochemical properties of castor oil.

To demonstrate the applicability of ultrasound in monitoring chemical reactions, the SoS was compared with the viscosity of each reaction route, considering each blend. Fig. 4 compares the variations of SoS and viscosity over time for Routes A–D (soybean-castor oil blends).

As shown in Fig. 4, both SoS and viscosity values stabilised for all routes after approximately 10 min of reaction. The E_n values calculated between the different reaction times for both SoS and viscosity were below 1, statistically confirming that the reactions reached a stable state. When comparing the other routes, an increase in castor oil content corresponds to higher viscosity, which was accompanied by a rise in SoS. Specifically, for Route A (Fig. 4a), the final viscosity and SoS were 4.5 mPa·s and 1369.0 m·s^{-1}, respectively. For Route B (30 wt% castor oil, Fig. 4b), viscosity increased to 5.1 mPa·s, and SoS to 1376.0 m·s^{-1}. For Route C (40 wt% castor oil, Fig. 4c), 5.4 mPa·s and 1381.5 m·s^{-1} were obtained, while for Route D (50 wt% castor oil – Fig. 4d), $\eta = 6.4$ mPa·s and SoS = 1388.4 m·s^{-1} were achieved.

Although viscosity varied slightly among Routes A –D (Fig. 3), the measurement uncertainty represented by the error bars indicates that these values are metrologically equivalent, preventing differentiation of the blends based solely on viscosity. In contrast, ultrasound measurements provide a sensitive tool capable of distinguishing changes in the reaction medium as castor oil content increases.

For the routes involving pure oils, Fig. 5 compares the variations of SoS and viscosity over time.

Consistent with the blends, both SoS and viscosity values stabilised after 10 min. After 40 min, pure soybean oil (Route E – Fig. 5a) presents a viscosity of 4.2 mPa·s and SoS of 1365.7 m·s^{-1}, whereas pure castor oil (Route F, Fig. 5b) showed a viscosity of 11.6 mPa·s and SoS of 1403.7 m·s^{-1}. It indicates that the speed of sound increases with the viscosity of the reaction medium, which is consistent with previous reports showing that ultrasonic propagation is influenced by density and intermolecular interactions, and that higher viscosity liquids generally exhibit higher speeds of sound. Tat and Van Gerpen (2000) measured SoS and viscosity of pure biodiesel and its esters, finding higher SoS in more viscous liquids [37], whilst Costa-Felix et al. (2018) showed similar trends for diesel–biodiesel mixtures [57]. These findings support the present study's trends for pure oils and their blends.

While both viscosity and ultrasound methods can differentiate between pure soybean and castor oil routes, ultrasound monitoring offers the advantage of in-line, real-time measurement without sampling, injection, or cleaning the measurement cell.

A similar analysis was performed to compare the evolution of SoS and density across the different blends (Fig. 6).

For example, the density of Route B (Fig. 6b) reached 912.7 kg•m^{-3}, whereas for Route D (50 wt% castor oil – Fig. 6d) it was 912.9 kg•m^{-3}. Considering measurement uncertainty, no significant differences were observed among the blends, confirming metrological equivalence. Additionally, the E_n values for density were all bellow 1. Notably, SoS increased significantly with castor oil concentration despite the constant density.

For pure oils, the evolution of SoS and density is shown in Fig. 7, confirming the trends observed for the blends.

Comparing Routes E and F highlights clear distinctions after 40 min of reaction. The reaction with soybean oil (Route E – Fig. 7a) exhibited a density of 897.0 kg•m^{-3} and a speed of sound of 1365.7 m·s^{-1}. In contrast, the reaction with castor oil (Route F – Fig. 7b) reached a density of 927.7 kg·m^{-3} and a speed of sound of 1403.7 m·s^{-1},

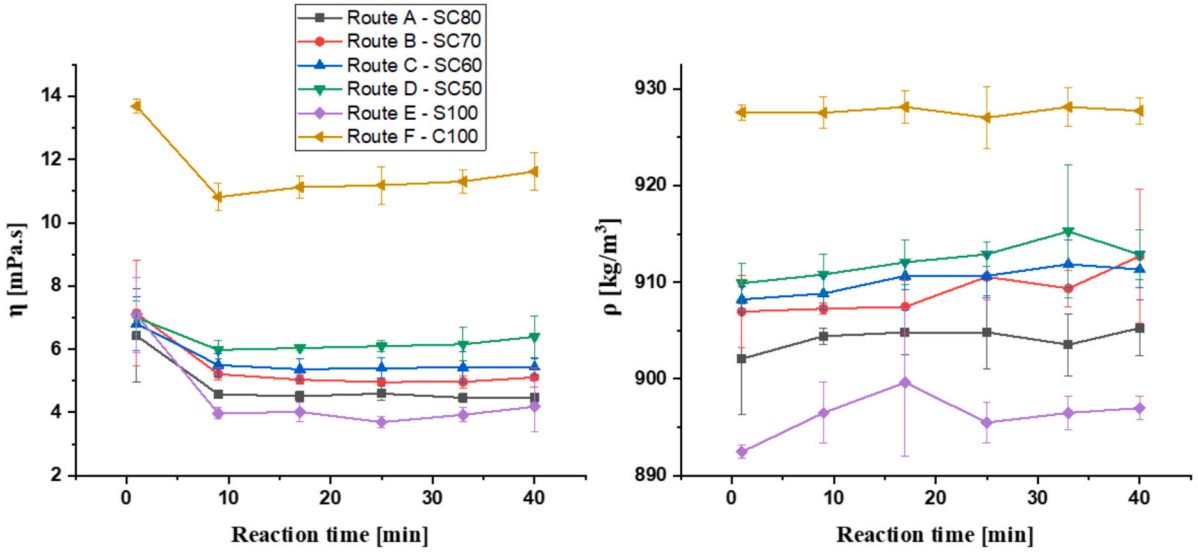

Fig. 3. Variation of viscosity and density during the transesterification of soybean and castor blends at different ratios and pure soybean and castor oils.

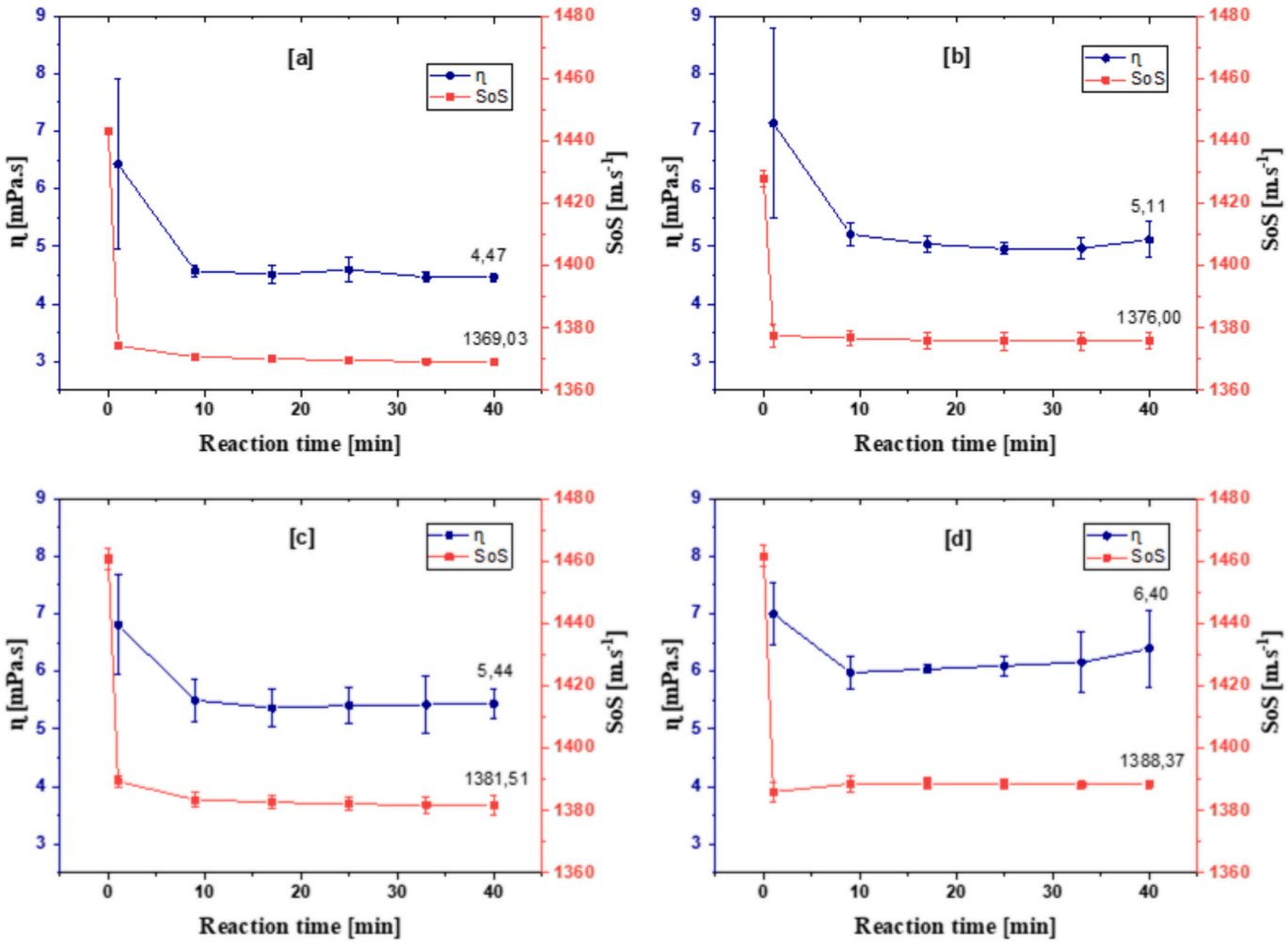

Fig. 4. Viscosity and speed of sound during the homogeneous transesterification of soybean (S) and castor (C) blends at different ratios. (a) Route A: S80% – C20 % (b) Route B: S70% – C30%; (c) Route C: S60% – C40%; and (d) Route D: S50% – C50%.

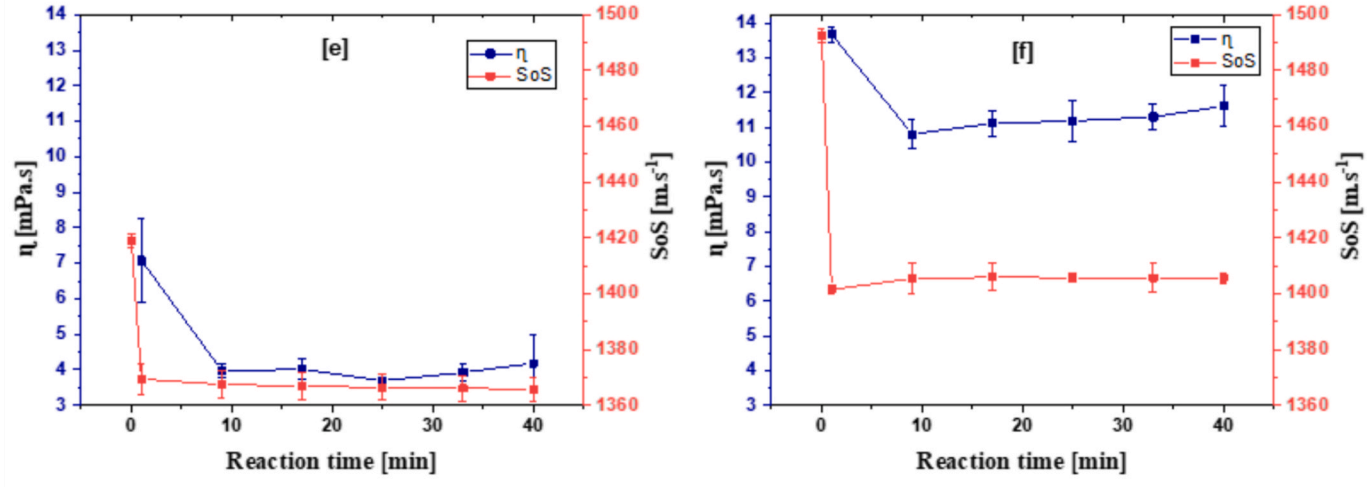

Fig. 5. Viscosity and speed of sound during the homogeneous transesterification of soybean (S) and castor (C) pure oils. (e) Route E: S100% and (f) Route F: C100%.

demonstrating that sound speed increases with the reaction medium's density. Similarly, viscosity measurements differentiate the reactions using pure soybean and castor oil. However, density measurements alone were insufficient to distinguish between the reactions of the different blends.

While the viscometer-densimeter method is cost-effective, it presents several limitations compared to ultrasound. It cannot provide real-time monitoring, nor can it effectively differentiate between reaction routes

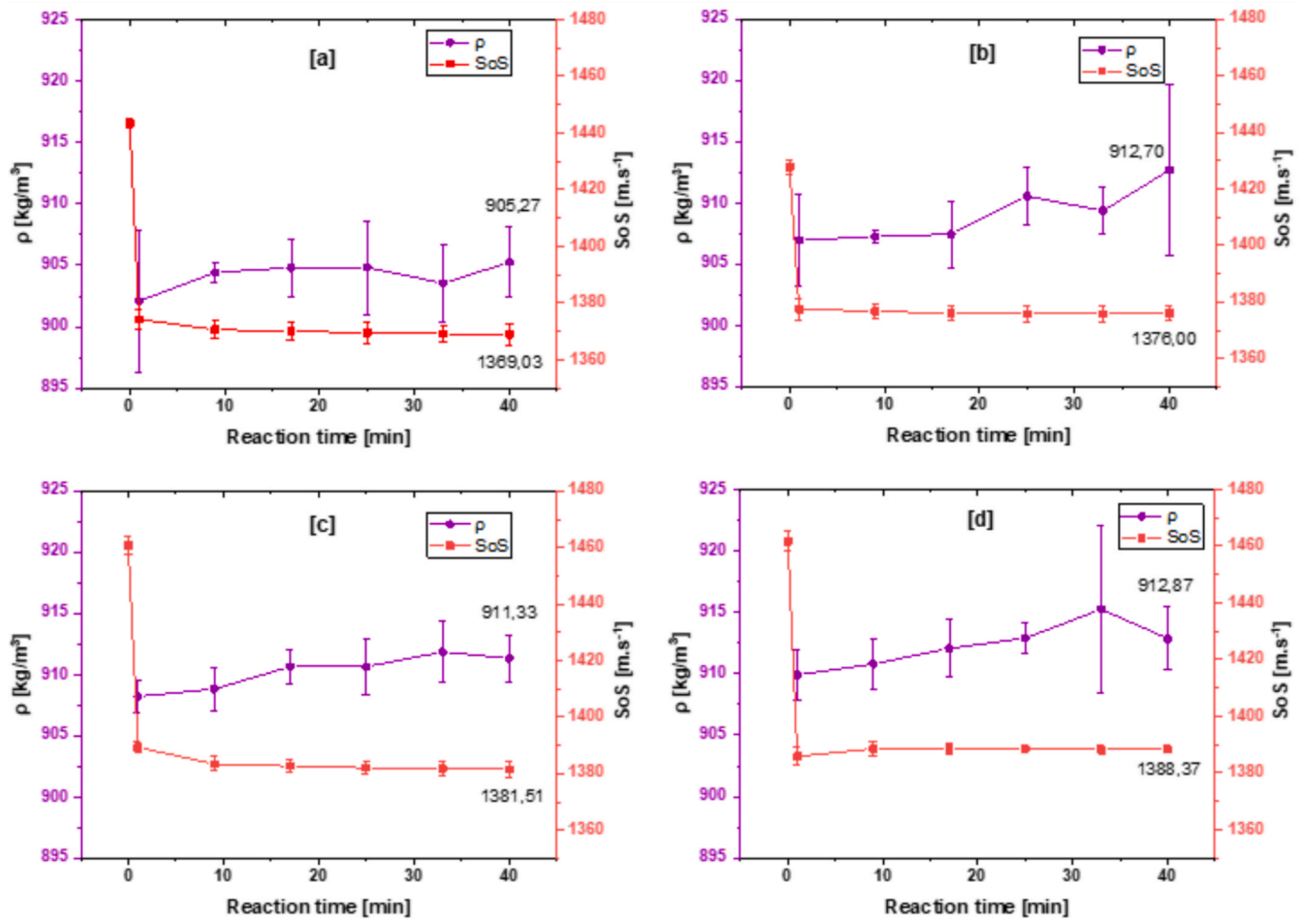

Fig. 6. Density and speed of sound during the homogeneous transesterification of soybean (S) and castor (C) blends at different concentrations. (a) Route A: S80% – C20 % (b) Route B: S70% – C30%; (c) Route C: S60% – C40%; and (d) Route D: S50% – C50%.

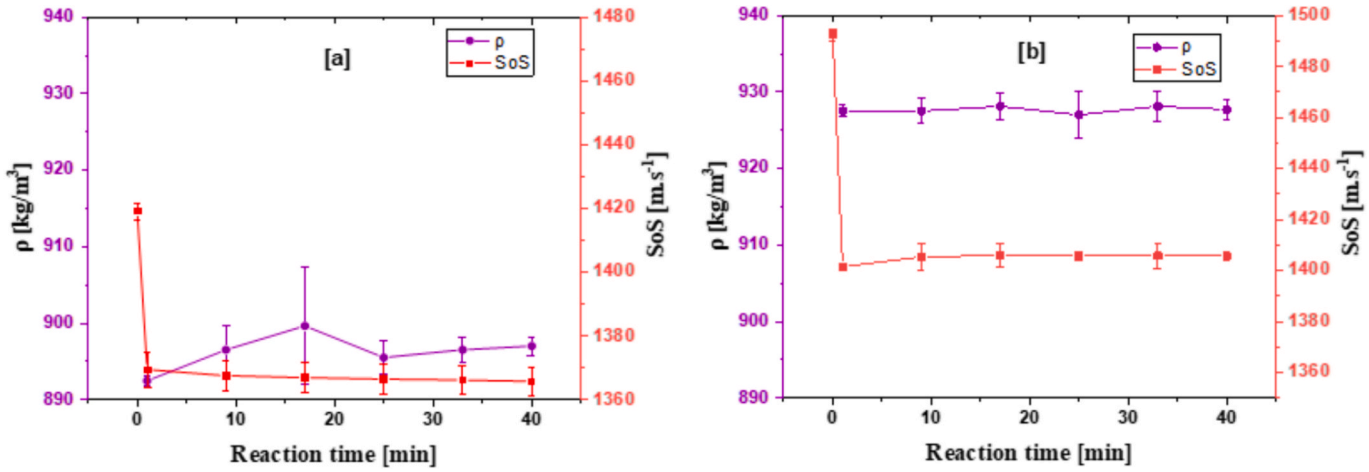

Fig. 7. Density and speed of sound during the homogeneous transesterification of soybean (S) and castor (C) pure oils. (e) Route E: S100% and (f) Route F: C100%.

or determine blend compositions. In contrast, ultrasound offers notable advantages, including real-time applicability, cost efficiency, and savings in both time and energy.

3.3. Influence of the type of oil and the composition on fatty acid methyl ester content

Fatty acid methyl ester contents at various transesterification reaction times of soybean, castor, and their blends (as described in Table 1) were determined using GC-FID. All routes were carried out under identical conditions. Fig. 8 presents the kinetic profiles for the

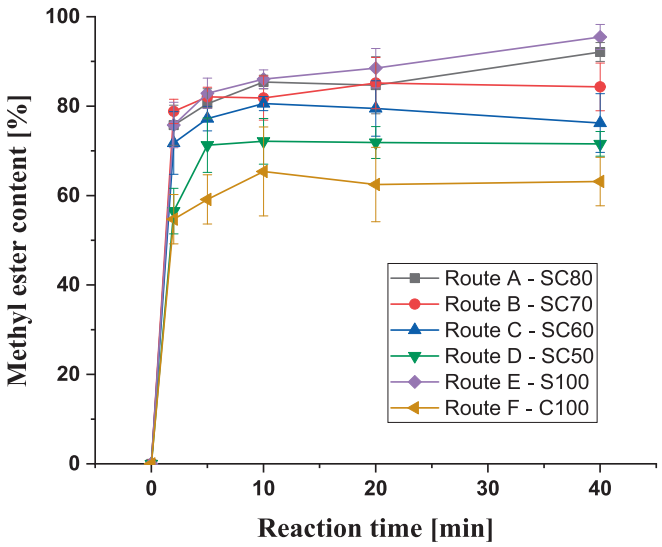

Fig. 8. Fatty acid methyl ester content in transesterification reaction mixtures of soybean oil, castor oil and their blends in different ratios.

transesterification of the soybean oil (S), soybean and castor oils blends (SC), and castor oil (C). Corresponding data are provided in Table 5.

See Fig. 8 shows the initial reaction rates for the different oil compositions, determining how quickly each system approaches equilibrium. Higher concentrations of soybean oil accelerate the early reaction, with reactions following Routes B, C, D, and F reaching equilibrium approximately 20 min into the process. Routes A and E, which have higher concentrations of soybean oil (80 wt% and 100 %, respectively), still vary in ester content even after 20 min. Significant differences in FAME content between different routes, with the exceptions of Route C (SC60/40) and D (SC50/50), which were statistically equivalent (En < 1).

Table 5 indicates that the FAME content in Route F (pure castor oil) after 40 min was only 63.1 %, the lowest among all other blends and pure soybean oil. Incorporating 50 wt% of soybean oil (Route D) increased the FAME content to 71.6 %. Further expanding the soybean oil concentration resulted in a higher FAME content, reaching 92.1 % in Route A. The highest FAME content was achieved with Route E (100 % soybean oil), at 95.5 %.

These results demonstrated that the composition of the vegetable oil blends significantly affects the FAME content, with a gradual increase as the concentration of soybean oil rises. The low yield observed with pure castor oil (around 2.0 % in oleic acid equivalents) is likely due to free fatty acids, which can neutralise part of the catalyst, reduce methoxide formation and promote saponification. This reaction decreases the FAME content [58,59].

Although FAME contents ranged from 63.1 % to 95.5 %, all values were below the 96.5 % required by EN 14103 [18]. However, the primary aim of this study was to correlate reaction yield with ultrasonic measurements, evaluating the sensitivity of the ultrasonic technique to changes in FAME content.

Table 5
Fatty acid methyl ester contents obtained by GC-FID for the transesterification reaction of a blend of soybean oil and castor oil employing different concentrations (1 wt% of KOH and molar ratio methanol:oil of 6:1).

Reaction time [min]	Fatty acid methyl ester content [%]					
	Route A SC 80/ 20[a]	**Route B SC 70/ 30**[a]	**Route C SC 60/ 40**[a]	**Route D SC 50/ 50**[a]	**Route E S100**	**Route F C 100**
0	0	0	0	0	0	0
2	75.7	78.8	71.8	56.5	75.8	54.7
5	80.5	82.1	77.2	71.3	82.9	59.2
10	85.4	81.8	80.6	72.1	86.0	65.4
20	84.7	85.2	79.5	71.9	88.5	62.5
40	92.1	84.3	76.2	71.6	95.5	63.1

[a] Blends of soybean (S) and castor (C) oils.

3.4. Speed of sound and FAME content

The ultrasonic speed of sound was related to the methyl ester content for each blend studied. Fig. 9 discloses the relationship between variation in speed of sound and methyl ester content for the routes using blends (A to D) and pure oils (E and F).

See Fig. 9 reveals that at "time 0″, the SoS varies among the samples due to their different compositions. It indicates that the ultrasound technique can detect differences between the tested routes even before the reaction begins. Only the blends corresponding to Routes C (60 % soybean oil and 40 % castor oil) and D (50 % soybean oil and 50 % castor oil) showed equivalent SoS at "time 0″.

In the first minute of the reaction, all routes show a decrease in SoS as the FAME content increases. It demonstrates that ultrasound can detect the conversion of triglycerides into esters and the differences in reaction rates across different blends as the reactions proceed. Ultrasound continuously monitors these changes in real time while GC analysis provides offline data. Fig. 10 shows the transesterification reaction over 40 min predefined for each route.

It is evident that for Routes B, C, D and F, both the FAME content and speed of sound reach equilibrium after 10 min of reaction. However, for Routes A and E, although the speed of sound stabilises after 10 min, the FAME content continues to vary between 20 and 40 min of reaction. While the ultrasound method did not show sensitivity to identify the variation in FAME content after 20 min for Routes A and E, comparing the different routes yielded promising results.

Routes A and E, which have the highest soybean oil concentration, exhibit the lowest SoS of 1369.0 $m{\cdot}s^{-1}$ and 1365.7 $m{\cdot}s^{-1}$, respectively, after 40 min, reaching a reaction. These routes also achieved the FAME content at the end of the experiments, reaching 92.1 % and 95.5 %, respectively. In contrast, Routes D and F, which have higher concentrations of castor oil, showed higher SoS of 1388.2 $m{\cdot}s^{-1}$ and 1403.7 $m{\cdot}s^{-1}$ but lower FAME contents, reaching 71.6 and 63.1 %, respectively.

Table 6 presents the FAME content and SoS results after 40 min of reaction for all tested routes.

In Fig. 10 and Table 6, it is evident that as the concentration of soybean oil increases, the FAME content rises while the SoS in the reaction medium decreases, indicating an inverse relation between speed of sound and methyl ester content. Although establishing explicit mathematical correlations was not the focus of this study, the measured data showed a strong positive correlation ($R^2 \approx 0.97$) between SoS and FAME content across the tested blends, supporting the consistency of ultrasound measurements with standard analytical techniques. The results confirm that variations in SoS consistently reflect changes in FAME content across the studied blends. Specifically, lower SoS values correspond to higher FAME content. This observation aligns with literature reports [31–41], which evaluated pure biodiesel (B100) and blends with methanol, glycerol, and triglycerides, highlighting that samples with lower FAME content exhibit higher SoS.

Previous studies have shown that the speed of sound varies systematically with the molecular structure of individual FAMEs and pure biodiesel fuels [60,61]. In contrast, the present work evaluates SoS during the transesterification reaction, where the medium is a complex mixture of methanol, glycerol, triglycerides, and esters in formation. Thus, the observed SoS reflects the overall FAME content and the contributions of the specific FAME species, alongside the unreacted reactants and by-products.

The in-line application of the ultrasound method provides the significant advantage of acquiring real-time information about the reaction

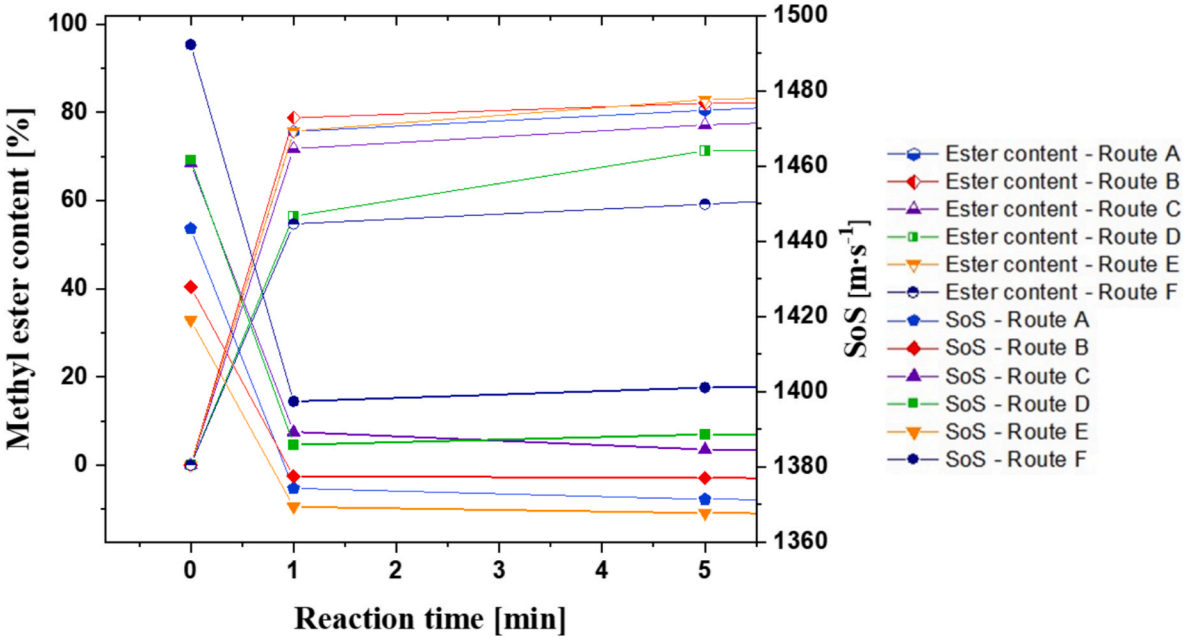

Fig. 9. FAME content and speed of sound during the initial 5 min of transesterification for all routes tested.

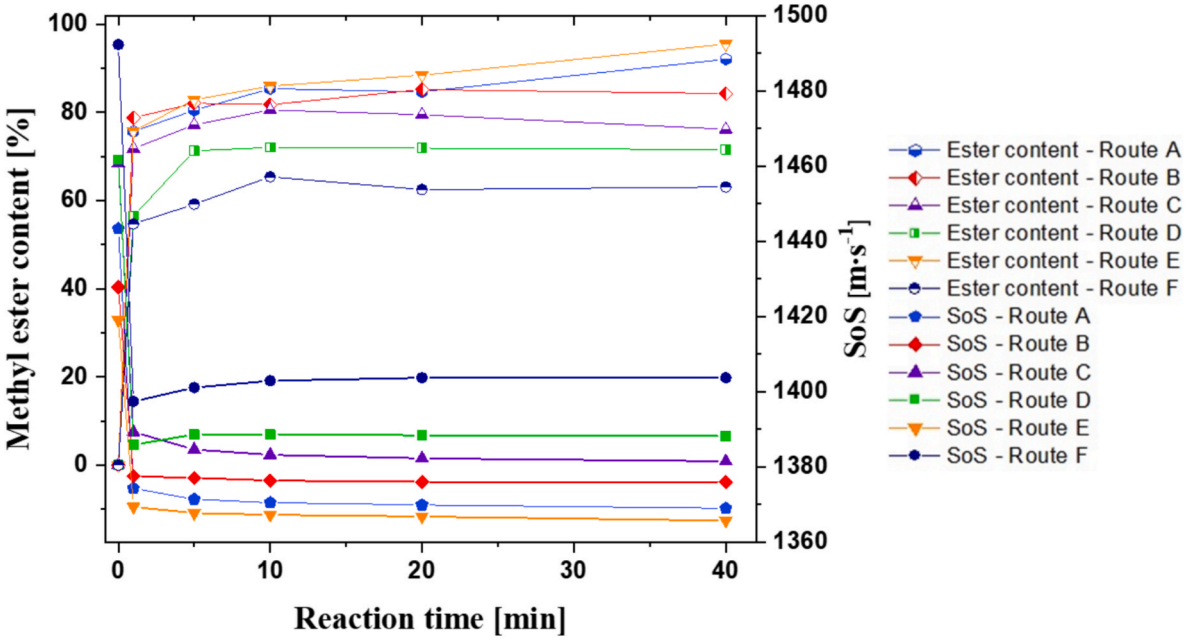

Fig. 10. Temporal evolution of FAME content and speed of sound over 40 min for routes tested.

Table 6
Speed of sound and FAME content for all routes tested after 40 min of transesterification reaction.

Routes	Concentration	FAME [%]	SoS [m·s^{-1}]
Route E	S100	95.5	1365.7 ± 4.4
Route A	SC80/20[a]	92.1	1369.0 ± 3.9
Route B	SC70/30[a]	84.3	1376.0 ± 2,.5
Route C	SC 60/40[a]	76.2	1381.5 ± 3.1
Route D	SC 50/50[a]	71.6	1388.2 ± 1.4
Route F	C100	63.1	1403.7 ± 1.8

[a] Blends of soybean (S) and castor (C) oil.

medium during transesterification. Such monitoring enables timely decision-making in the early stages of the reaction, reducing potential delays and the need for batch reprocessing. Additionally, it allows the evaluation of how fatty acid composition influences reaction kinetics and ultrasonic propagation. In this study, both soybean and castor oils and their blends were evaluated, demonstrating that SoS is sensitive to differences in the physicochemical properties of the reaction medium. Future studies could investigate the applicability of SoS monitoring for transesterification using oils with a broader range of fatty acid compositions to further assess the method's robustness and general applicability across different feedstocks.

In addition to soybean and castor oils, the applicability of SoS monitoring can extend to other biodiesel feedstocks. Literature reports

that SoS values vary across biodiesels depending on their fatty acid composition. For example, linseed oil biodiesel exhibits an SoS of 1422 $m \cdot s^{-1}$ at 293.15 K, close to the value reported for castor oil biodiesel (1464 $m \cdot s^{-1}$), while coconut oil biodiesel presents the lowest reported value (1362 $m \cdot s^{-1}$) [62]. Despite the relatively narrow range among most biodiesels, the ultrasound technique's high precision and low measurement uncertainty can ensure sufficient sensitivity to differentiating feedstocks and monitoring compositional changes during transesterification.

When monitoring transesterification using oil blends and different pure oils, ultrasound demonstrated its capability to detect subtle variations in oil composition and FAME content at the end of the reaction. These results highlight its potential for industrial implementation as a reliable tool for ensuring product quality and process efficiency.

4. Conclusions

This study evaluated the monitoring of transesterification reactions of soybean oil, castor oil, and their blends (80/20, 70/30, 60/40, and 50/50 wt%) using quantitative ultrasound (QUS). Complementary offline analyses of viscosity, density, and FAME content were performed to assess their effect on the speed of sound (SoS). While viscosity and density effectively distinguished reactions involving pure oils, they were less effective for blends, whereas QUS demonstrated higher sensitivity, with SoS decreasing as FAME content increased. These results confirm that ultrasound can track triglyceride conversion into FAME and detect subtle variations in oil composition.

Although ultrasound has previously been applied to real-time transesterification monitoring, this study systematically evaluates its performance for oil blends. The results demonstrate the feasibility of using QUS to monitor biodiesel production from soybean and castor oils and their blends. The method proved reliable, sensitive, and precise, distinguishing reactions involving different feedstocks. As a simple, cost-effective, and energy-efficient tool, ultrasound holds strong potential for industrial integration, supporting real-time process monitoring, quality control, and overall cost reduction in biodiesel production.

However, this study was conducted at a laboratory scale; the method's performance under industrial-scale conditions remains to be validated. Future work should focus on scaling up the ultrasonic monitoring system and testing its applicability across various oils and feedstocks with different fatty acid compositions, as well as under diverse operational conditions, to further assess the robustness and generality of the method.

CRediT authorship contribution statement

P.A. Oliveira: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **R.M. Baesso:** Writing – review & editing, Visualization, Methodology, Investigation, Formal analysis, Conceptualization. **M.I. Nunes:** Writing – review & editing, Validation, Supervision, Resources, Methodology, Investigation, Formal analysis, Data curation. **J.A.P. Coutinho:** Writing – review & editing, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis. **R.P.B. Costa-Félix:** Writing – review & editing, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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