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New findings of edible oil characterization by ultrasonic parameters

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ABSTRACT

The basic objective of the study was to confirm the usefulness of non-destructive ultrasonic testing in evaluating different edible oil samples. The experimental study was carried out for three types of edible oils (olive, sunflower, and corn) in which a 1.0 MHz ultrasound transducer was immersed. Density and viscosity values of the samples were determined simultaneously with the ultrasound tests. By themselves, ultrasound inspection, density, and viscosity, were able to characterize and distinguish each type from the others, but only the ultrasound inspection has a non-destructive nature. Moreover, significant correlations among density and viscosity with the acoustic parameters were found. The results postulate that ultrasound inspection is a fast and non-destructive tool to characterize and discriminate different types of edible oils.

1 Introduction

Olive oils command a premium price in the market, leading to great temptation to adulterate them with vegetable seed oils as they look more or less identical in colour and their presence cannot be easily identified by simple visual inspection (Ben-Ayed et al., 2013). Thus, binary and ternary mixtures of olive, sunflower and corn oils, appear to be fraudulent attempts in the market. Adulteration of olive oil may produce adverse effect which raises a serious issue to the consumer as well as to the oil agencies (Gurdeniz & Ozen, 2009). Therefore, characterizing it and determining its quality, as well as being able to distinguish between types, is of utmost importance to protect both, the producer and the consumer. Since adulterations are becoming more and more sophisticated, it is necessary to use advanced methods to detect these fraudulent practices. There are many analytical methods used to check the authenticity of oils (Ben-Ayed et al., 2013; Mavromoustakos et al., 2000). However, most are destructive techniques. Thus it is necessary to evaluate the capability of different techniques to analyse quality characteristics of oils, as can be seen in recent studies on the NIRs (Correa et al., 2019; Kaufmann et al., 2019, Kaufmann, Sampaio, García-Martín, & Barbin, 2022), or image analysis application (Alamprese et al., 2021).

Ultrasound inspection has been widely used to analyse different foodstuffs due to its non-destructive nature. Particularly, the ultrasound pulse velocity (UPV) and attenuation are the most used acoustic parameters to determine the physicochemical and mechanical properties of oil (Ali & Ali, 2014; Alouache, Laux, Hamitouche, Bachari, & Boutkedjirt, 2018; Baêsso, Oliveira, Morais, Alvarenga, & Costa-Félix, 2016; Rashed & Felföldi, 2016). The ability of ultrasound inspection has been proven to control the olive oil quality (Alouache et al., 2015), to investigate the adulteration (Azman & Abd Hamid, 2017), even to evaluate the quality of frying oils (Benedito et al., 2007). Other studies have focused on optimization of the ultrasound inspection of edible oils (Yan et al., 2019). Nevertheless, there are other parameters that can be extracted from ultrasound measurements and some of these can be linked to the study of the signal in the frequency domain, which have not been subject to assessment in the scientific literature.

Density and viscosity properties are of great importance in distinguishing different types of oils (Sahasrabudhe et al., 2017) and are essential in the design of unit operation processes such as distillation, heat exchangers, piping, and reactors (Rodenbush et al., 1999). However, characterizing an oil based on these variables is a difficult task and, unfortunately, they are not anymore practical to detect adulteration (Rukke & Schüller, 2017).

The objective of the present study was to use ultrasound inspection to

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characterize and differentiate between three different types of edible oils commonly consumed: Olive, Sunflower, and Corn oils, all from Spain, at two temperatures (25 and 29 °C). For this aim, the ultrasound parameters considered were the ultrasound pulse velocity (UPV), different variables extracted from the Fast Fourier Transform (FFT), and the attenuation. With this research, it is intended to contribute to knowledge about the acoustic properties of the three edible oils studied, focusing on the authenticity and identification of these oils, and providing new parameters for study that had not been considered previously for this product (FFT parameters). Additionally, a study of density and viscosity determinations were carried out, and its results were correlated with the ultrasound parameters.

2 Materials and methods

2.1. Samples

The oil samples were purchased in a Spanish supermarket, belonged to the "Coosur" brand (Jaén, Spain). These samples correspond to three types: olive, sunflower, and corn. Four one-litre samples of each type were acquired so a total of twelve samples were considered in the experiment.

2.2. Density and viscosity

According to previous works, ultrasound inspection is mainly influenced by the physicochemical parameters, achieving significant correlations among these parameters and acoustic ones (González-Mohino et al., 2019, 2021). Density (ρ) and viscosity (η) were considered as valuable parameters of oils. It is important to note that both parameters are dependent on the temperature (T), showing its influence by the following model equations (1, 2) (Ghosh et al., 2017):

$$\rho = A \bullet \mathrm{T} + \rho_0 \quad (1)$$

$$\eta = \eta_0 \bullet e^{\frac{B}{T}} \quad (2)$$

where A, ρ_0, η_0, and B, are constant characteristics of the oil type.

Density was determined using a glass and normalized density meter DIN 12,791 with a thermometer (Laboquimia, Spain). Viscosity was measured using a Selecta STS-2011 viscometer (J. P. SELECTA, Spain). A spindle TL5 with APM adaptor was used at 20 rpm for viscosity determination in 5 ml of sample. Both analyses were performed in triplicate and at two different temperatures, 25 °C and 29 °C, using an incubator FTC E90 (VELP Scientifica Srl, Italy). The mentioned temperatures were selected since they were common storage temperatures and, at the same time, can result in statistically significant differences between the density and viscosity parameters.

2.3. Ultrasound inspection

Samples, with a quantity of 725 ml, were introduced in a beaker of 800 ml capacity. The oils were heated to 29 °C in a thermostatic bath (JP SELECTA, Barcelona, Spain). Beginning from this temperature (29 °C), ultrasound measurements were obtained as the sample gradually cooled until 25 °C. Thus, a total of eight measurements were made for each type. Samples were inspected using immersion techniques in pulse-echo (PE) mode. Supplementary Material 1 (SM1) shows the set-up used for these measurements. Olympus Panametrics-NDT Model V314-SU piezoelectric transducer was used to transmit the signals with a frequency of 1 MHz. Supplementary Material 2 (SM2) lists the main characteristics of this transducer, including the near-field zone and the beam-spread angle. The transducer was mounted on a custom-designed metal structure so that its emitting surface was parallel to the bottom of the beaker. A mirror at the bottom of the beaker reflected the ultrasound waves. The transducer was placed at the top of the beaker, submerged approximately 1.5 cm in oil, whence it transmits the wave. Use of this inspection mode or that of through-transmission (TT) is irrelevant since they give similar results (Jiménez et al., 2017). The separation between the surface of the transducer and the mirror was 455.00 mm, a value much higher than that of the near field, which ensured the consistency of the measurements. Pulser-Receiver Panametrics-NDT Model 5077PR was used for transmitting and receiving the ultrasound signal. Pulser-receiver was connected to an InfiniiVision DSO-X 3032A oscilloscope (KEYSIGHT) for the acquisition of the signals. Of each measurement, three A-scans were stored, each one corresponding to a different time interval (*TI*) on the oscilloscope: (i) $TI = 5000$ μs, which included the trigger pulse and the first six echoes; (ii) $TI = 800$ μs, which included the first two echoes; and (iii) $TI = 120$ μs, which included only the first echo. The A-scans were recorded in csv files with 10 000 points. Supplementary material 3 (SM3) shows three typical A-scans (from corn samples at 25 °C), corresponding to (a) $TI = 5000$ μs, (b) $TI = 800$ μs, and (c) $TI = 120$ μs. Thereafter the preparation of this equipment, several acoustic parameters were determined.

2.3.1 Acoustic parameters

As aforementioned, the ultrasound parameters determined were the ultrasound pulse velocity UPV (or time-of-flight, *TOF*), various percentiles (25th, 50th, and 75th) of the received signal in the FFT cumulative frequency periodograms, and the attenuation measured from the echoes observed on the A-scan which originate from reflections in the mirror. In particular, UPV was determined in two ways. The first calculation was obtained from the times observed for the consecutive echoes of the receiver's A-scan and the second one, by means of the so-called cepstrum method (Gudra & Opielinski, 2004).

2.4. Data analysis

Significant differences between the three edible oil types, sunflower, olive, and corn were studied. The normality of density, viscosity, and ultrasound parameters were evaluated with the Shapiro-Wilk test. Depending on the normality tests results, Student-T test (p greater than 0.05) or U Mann-Whitney test ($p < 0.05$) were applied. IBM SPSS v.22 (IBM Co., U.S.A) statistic software package was used to carry out the former analysis. Pearson's correlation coefficient (R) among density and viscosity, and ultrasound parameters, was carried out using the XLSTAT software package (Addinsoft Pearson Edition, France).

3 Results and discussion

3.1. Density and viscosity results

Table 1 shows mean values of density and viscosity results for the three oil types. Corn oil presented higher density values than sunflower, and likewise this oil had higher density than olive oil, for both measured temperatures (25 and 29 °C). As previously reported, emulsion systems are highly influenced by the thermal behaviour (Rukke & Schüller, 2017). Thus, as occurred in other works, density decreases as temperature increases (Sahasrabudhe et al., 2017). The results of density were

Table 1
Density (ρ) and viscosity (η) results (means ± SD) for olive, sunflower, and corn for 25 and 29 °C. Letters in each column mean the significant differences between oil type. Lowercase letters mean type effect; capital letters mean temperature effect.

	ρ_{25}(kg/m^3)	ρ_{29}(kg/m^3)	η_{25} (cP)	η_{29} (cP)
Olive	909.0 ± 0.9^{cA}	906.0 ± 0.9^{cB}	74 ± 4^{aA}	59.9 ± 1.5^{aB}
Sunflower	915.00 ± 0.21^{bA}	912.00 ± 0.13^{bB}	61.9 ± 2.2^{bA}	50.0 ± 1.5^{bB}
Corn	917.0 ± 0.7^{aA}	915.0 ± 0.8^{aB}	59.0 ± 1.6^{bA}	48.0 ± 1.8^{bB}

consistent, according to previous studies, and therefore showing similar values and trends (Alouache et al., 2018; Azman & Abd Hamid, 2017; Esteban et al., 2012). On the other hand, olive oil showed the highest viscosity values in both temperatures, presenting significant differences in comparison with sunflower and corn oils. As expected (Sahasrabudhe et al., 2017), viscosity showed the same behaviour as density; decreasing when the temperature is increasing. Once again, our results are in concordance with the literature, being similar to those results from other studies (Azman & Abd Hamid, 2017; Benedito et al., 2002; Sahasrabudhe et al., 2017).

3.2. Ultrasound inspection results

3.2.1 Some previous particularities

As can be seen in SM3a, i consecutive echoes caused by reflections between the transducer facing mirror are observed. The first UPV calculation was obtained from the times TOF_{ij} observed for the six consecutive echoes of the receiver's A-scan, detecting the value achieved on the time axis at the points where the signal reaches a maximum ($j = \mathrm{M}$) and a minimum ($j = \mathrm{m}$) in each echo ($i = 1, 2,\ldots, 6$). Then, we obtained the velocity UPV_{lr} from the slope of the linear regression fit corresponding to Eq. (3) (Rufo et al., 2014):

$$d_i = UPV_{lr}\hat{\mathrm{A}}\cdot TOF_{ij} + C(\mathrm{for}\, i = 1, 2, \cdots, 6 \mathrm{and}\, j = M, m) \tag{3}$$

where d_i are the known distances that are travelled by the waves corresponding to the first six echoes. In this study, the errors in the determination of UPV_{lr} were of the order of 0.21%.

The second UPV calculation is based on the Fourier transform. Given that the procedure followed is identical to that used by the authors in previous works (González-Mohino et al., 2019; Jiménez et al., 2017), we shall limit ourselves here to briefly summarize it below. Supplementary material 4 (SM4) shows the FFTs obtained from the A-scans of SM3. The influence of a periodic excitation (here multiple echoes) shows up in the form of equidistant maxima in the spectrum. The TOF between the reflections is obtained from the distances of the maxima. By means of the so-called cepstrum method (Gudra & Opielinski, 2004), the spectrum can be smoothed, and the length of the period determined directly. As can be seen in the example of SM5 which shows the cepstrum created from the FFT of SM4a, the period t is read directly from the first maximum. Since the transducer and the mirror are separated by a distance d = 455.00 mm, spectral evaluation methods provide a measurement of the velocity UPV_c from Eq. (4):

$$UPV_c = \frac{2d}{t} \tag{4}$$

In the present study, the errors in the cepstrum determination of UPV_c were<0.16%.

Other parameters considered in this study were the frequencies of the 25th, 50th, and 75th percentiles of the FFT. First of all, we must remember that, according to Nyquist sampling theorem, the sampling frequency f' in time domain must be at least twice of frequency range f in frequency domain as shown in Eq. (5) (Zhong & Zhang, 2021):

$$f' \geq 2f \tag{5}$$

In our study, for the FFT obtained in SM4a, $f' = 10000/(5000\cdot10^{-6}) = 2\cdot10^6$. Similarly, $f'=10000/(800\cdot10^{-6}) = 12.5\cdot10^6$ considering SM4b, and $f'=10000/(120\cdot10^{-6}) = 83.3\cdot10^6$ considering SM4c. Taking into account that the central frequency of the transducer used is 10^6 Hz, it seems evident that the sampling frequency from which the FFT of SM4a has been obtained does not satisfy this theorem, especially considering that there are frequency components emitted by the transducer with a value greater than 10^6 Hz. In fact, the FFT obtained and shown in said SM seems to "cut off" abruptly. Consequently, we will exclusively use the parameters extracted from the FFTs obtained in the inspections where the first two echoes and only the first echo are shown.

Taking either of SM4b or SM4c as an example, it can be seen that the frequency distributions are not close to Gaussian, hence that the largest amplitude of the FFT is not a clear value to characterize the oil samples. Furthermore, even though the transducer has a central frequency of 10^6 Hz and a −6 dB bandwidth of 61.9%, the FFT obtained does not have a maximum amplitude at that frequency but at clearly lower ones. Consequently, instead of considering the nominal frequency of the transducer, it made more accurate to take the real wave propagation frequencies inside the oil samples. Therefore, it was constructed the cumulative frequency periodograms, showing the 25th, 50th, and 75th percentiles (FFT_{25}, FFT_{50} and FFT_{75}, respectively) of the frequencies of the received signals. SM6 shows the cumulative frequency periodogram created from the FFT of SM4b.

Another parameter considered was the ultrasound pulse attenuation. This parameter is a measure of the progressive energy loss undergone by the signals as they propagate through the material (Cerrillo et al., 2014). The attenuation coefficient α (in neper/m) was computed as Eq. (6):

$$\alpha = \frac{1}{2d}\ln\left(\frac{\mathrm{A}_i}{\mathrm{A}_j}\right) \tag{6}$$

where A_i and A_j are the (peak-to-peak) amplitudes of echoes i and j, respectively, and $2d$ represents the space covered by the ultrasound wave between them. Six echoes from A-scan corresponding to TI = 5000 μs were considered, and α was determined from the slope of the plot of ln (A_i/A_j) vs. $2d$ (Koc & Ozer, 2008).

3.2.2. Acoustic parameters

Fig. 1 displays the representation of UPV_{lr} and UPV_c results for the three oil types and for both measured temperatures. Firstly, it should be noted that the results of the two ways of calculating the UPV are very similar, showing the suitability of both determinations to obtain velocity values. In fact, the error bars clearly overlap when we consider each of the oil types. It is also observed that when temperature increases, UPV_{lr} and UPV_c decrease, as it has been previously described for these three oils (Azman & Abd Hamid, 2017), thus showing the same behaviour as density and viscosity. UPV was not significantly different for corn and sunflower oils at 25 °C. On the contrary, olive oil had significantly lower UPV values than the other two oil types at 25 °C. Regarding the measurements carried out at 29 °C, a dissimilar trend was found. The oil types were significantly different for both UPV at 29 °C. The highest UPV values were found for corn, followed by sunflower and then by olive oil. According to Rubalya Valantina et al, (2013), the ultrasonic velocity in oils depends on the fatty materials, hence the discrimination among our edible oils was probably due to the differences in the fatty acid profiles of each type (Codex alimentarius, 2019; CodexAlimentarius, 2017). Given that the behaviour of velocity with temperature is similar to that exhibited by density and viscosity, it is foreseeable that there is a relationship between these properties and the acoustic parameters.

The UPV results obtained are consistent with the literature values. Alouache et al. (2018) determined a velocity of 1469 m/s in olive oil, being slightly higher than our results probably due to the measured temperature (20 °C). In concordance with this study, the velocity was 1465 m/s for olive oil, 1471 m/s for sunflower oil, and 1469 m/s for corn oil, as collected by McClements & Povey (1992) using 1.2 MHz transducer. Lower values found in other studies (Azman & Abd Hamid, 2017) were reported as ultrasound velocity of 1430 m/s (at 25 °C) and 1412 m/s (at 30 °C) for olive oil type. Baêsso Costa-Felix Miloro & Zeqiri (2019) estimated the velocity of ultrasound of 1473 m/s in sunflower and 1470 in corn oil at 20 °C using 1 MHz transducer.

Table 2 shows attenuation results for the three oil types and both temperatures. In the first place, the fact that the attenuation results at 29 °C were superior to the results obtained at 25 °C stands out. Understanding viscosity as a measure of friction between the particles of a

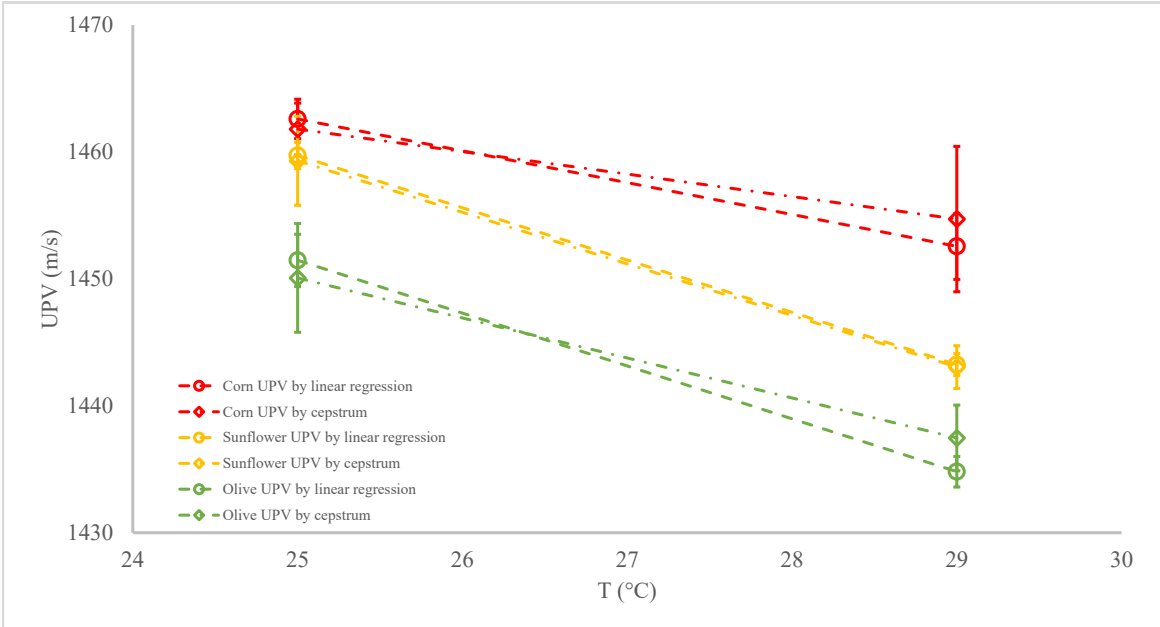

Fig. 1. UPV_{lr} and UPV_c mean values (±SE) for olive, sunflower, and corn oil types.

Table 2
FFT_{xx} and attenuation results (mean values ± SD) for olive, sunflower, and corn types at 25 and 29 °C (FFT_{xx} 1–2 $_T$ = FFT_{xx} corresponding to the first two echoes at temperature T = 25 or 29 °C; FFT_{xx} 1 $_T$ = FFT_{xx} corresponding to the first echo at temperature T = 25 or 29 °C; α_T = attenuation at temperature T = 25 or 29 °C). Letters in each row mean significant differences between oil type.

	Olive	Sunflower	Corn
α_{25}	26.26 ± 0.07^b	23.6 ± 0.4^c	27.2 ± 0.3^a
α_{29}	27.4 ± 0.8	28.4 ± 0.3	29.1 ± 1.3
FFT_{25} 1–2$_{25}$	723000 ± 7000^c	793000 ± 10000^a	756000 ± 3000^b
FFT_{50} 1–2$_{25}$	834000 ± 7000^b	1000000 ± 70000^a	891000 ± 22000^b
FFT_{75} 1–2$_{25}$	1050000 ± 50000^c	1300000 ± 70000^a	1170000 ± 30000^b
FFT_{25} 1–2$_{29}$	760000 ± 22000	784000 ± 3000	779000 ± 4000
FFT_{50} 1–2$_{29}$	910000 ± 60000	946000 ± 12000	936000 ± 8000
FFT_{75} 1–2$_{29}$	1170000 ± 130000	1230000 ± 30000	1201000 ± 23000
FFT_{25} 1$_{25}$	735000 ± 11000^c	809000 ± 17000^a	768000 ± 7000^b
FFT_{50} 1$_{25}$	857000 ± 17000^b	1070000 ± 70000^a	937000 ± 15000^b
FFT_{75} 1$_{25}$	1100000 ± 50000^b	1340000 ± 70000^a	1230000 ± 50000^a
FFT_{25} 1$_{29}$	777000 ± 20000^b	813000 ± 17000^a	803000 ± 7000^a
FFT_{50} 1$_{29}$	960000 ± 80000	991000 ± 15000	982000 ± 15000
FFT_{75} 1$_{29}$	1230000 ± 110000	1283000 ± 11000	1260000 ± 30000

fluid (Serway & Jewett, 2018), oil in our case, this result is logical since a higher friction (higher viscosity) would mean a greater binding force between the particles, which would translate into a lower loss of energy when the wave is transmitted from one particle to another and, consequently, a lower attenuation. Secondly, the attenuation values were significantly different at 25 °C between the three oil types studied. Corn oil was the type with the highest attenuation, followed by olive, and then by sunflower. Regarding the attenuation measures at 29 °C, no significant differences were found. This order in the attenuation value of the three inspected oils, without contradicting the explanation given for the viscosity-attenuation relationship, does suggest the influence of other parameters, such as density or others not studied here, on the viscosity of the samples.

Parameters extracted from FFT results were shown on Table 2 for the three types and both temperatures. The FFT_{25}, FFT_{50}, and FFT_{75} from two firsts echoes A-scans at 25 °C showed that the sunflower type significantly let higher frequencies pass, than the corn and olive types. Likewise, corn samples obtained higher values of FFT_{25} and FFT_{75} than the olive samples, without presenting significant differences in FFT50 between these two types. This uneven behaviour of the frequency components compared to that shown by the attenuation for oil types, indicates that the attenuation experienced by the signal depends not only on its frequency, but on oil considered as well. Thus, sunflower oil would be the one that proportionally attenuates the highest frequencies the least and olive oil the one that most. When temperature rises to 29 °C, no significant differences are found between the oils for the frequency percentiles. Considering only the first echo at 25 °C, once again the sunflower oil was the sample that let the highest frequencies pass for FFT25 and FFT50, however it was not significantly higher than the corn type for FFT75. Regarding the frequency percentiles considering the first echo at 29 °C, only FFT25 showed significant differences between the sunflower and corn oils with the olive oil, which has lower frequency components. As can be deduced, the explanation for these results is identical to that given above. Finally, FFT_{25}, FFT_{50}, and FFT_{75} mean values experimented an increment at 29 °C, that is, if we have already verified that the attenuation increases with temperature, this result now implies that this attenuation increase is proportionally less for the higher frequencies.

3.3. Correlation study

Table 3 displays coefficients of linear correlations between density, viscosity, and acoustic parameters for the three types at 25 and 29 °C. Several significant correlations were found, especially those related to UPV which were particularly high. When density increases the UPV increases, and this positive correlation is observed for both measurement temperatures. This result was highly predictable since both, density and velocity, are linearly related to temperature, so the relationship between both variables must also be. On the other hand, viscosity presented an opposite behaviour compared to density. When viscosity decreases, UPV_{lr} and UPV_c increase, and vice versa. For this point a clarification is necessary considering that, in our work, the relationship obtained between the velocity of propagation and the temperature is linear, as claimed by other authors (Azman & Abd Hamid, 2017; Rubalya Valantina et al., 2013). From Eqs. (1) and (2) it is unequivocally deduced that the relationship between velocity and viscosity must be of the type shown in Eq. (7). However, for the range of temperatures and viscosities considered in our work, a linear fit containing η (instead of ln η) or UPV (instead of $1/UPV$) also works correctly, as evidenced by the

Table 3
Coefficients of linear correlations among non-acoustic and acoustic parameters at 25 and 29 °C. Values in **boldface** mean statistical significance ($p < 0.05$).

	ρ_{25}	η_{25}	$\ln\eta_{25}$		ρ_{29}	η_{29}	$\ln \eta_{25}$
UPV_{lr} 25	**0.95**	**−0.97**	**−0.97**	UPV_{lr} 29	**0.94**	**−0.88**	**−0.89**
UPV_c 25	**0.91**	**−0.77**	**−0.79**	UPV_c 29	**0.85**	**−0.82**	**−0.83**
$1/UPV_{lr}$ 25	–	**−0.97**	**−0.97**	$1/UPV_{lr}$ 29	–	**−0.86**	**−0.89**
$1/UPV_c$ 25	–	**−0.86**	**−0.79**	$1/UPV_c$ 29	–	**−0.82**	**−0.83**
α_{25}	−0.07	0.06	0.05	α_{29}	0.60	**−0.72**	**−0.73**
FFT_{25} $1–2_{25}$	**0.71**	−0.65	−0.64	FFT_{25} $1–2_{29}$	0.60	−0.65	−0.64
FFT_{50} $1–2_{25}$	0.54	−0.46	−0.44	FFT_{50} $1–2_{29}$	0.41	−0.42	−0.42
FFT_{75} $1–2_{25}$	0.67	−0.58	−0.57	FFT_{75} $1–2_{29}$	0.27	−0.29	−0.29
FFT_{25} 1_{25}	0.68	−0.60	−0.59	FFT_{25} 1_{29}	**0.74**	**−0.76**	**−0.76**
FFT_{50} 1_{25}	0.60	−0.53	−0.51	FFT_{50} 1_{29}	0.34	−0.31	−0.31
FFT_{75} 1_{25}	**0.71**	−0.62	−0.61	FFT_{75} 1_{29}	0.34	−0.36	−0.36

high correlation coefficients also shown in Table 3.

$$\frac{1}{UPV} = C\ln\eta + D \tag{7}$$

It should be noted the significant and negative correlation coefficients obtained between viscosity and attenuation at 29 °C, in the sense that more viscous samples attenuate less. This result would be in accordance with what was pointed out above for these two parameters, a higher viscosity (higher friction) implies a greater binding force between the particles, which implies a lower loss of energy when the wave is transmitted from one particle to another and, consequently, a lower attenuation.

Finally, it should be highlighted that, FFT25 corresponding to firsts two echoes and first echo for both temperatures obtained high coefficients among parameters. In the case of density, the results suggest that denser samples allow higher frequency component waves to pass. In the case of viscosity, the more viscous samples attenuate these high frequencies more. These conclusions are also repeated for the relationships with FFT50 and FFT75, although with less significance.

4. Conclusions

Ultrasonic inspection allowed to distinguish the oil types of olive, sunflower, and corn at different temperatures, 25 and 29 °C. This discrimination was due to the novel ways to calculate UPV (based on linear regression and cepstrum), the ultrasonic variables extracted from the Fast Fourier Transform (FFT), and the attenuation. Likewise, significant correlations between density and viscosity with these acoustic parameters were found, showing a promising predictive character for these determinations in the edible oils studied.

The results of the present work showed new contributions and findings related to the ability of ultrasound to characterize and distinguish different types of oil from acoustic parameters. This opens up a new path not just to characterize and identify different types of oils with ultrasound inspection, but also to use it as a non-destructive, fast, and cost-effective method with which to ensure the authenticity of the oil and thus avoid possible frauds in the market. Further works should enlarge the number of samples to construct classification and predictive models, and moreover consider the ability of ultrasound inspection as a discrimination tool with the aim to detect fraudulent practices in the industry.

CRediT authorship contribution statement

A. Jiménez: Conceptualization, Methodology, Validation, Formal analysis, Resources, Data curation, Investigation, Writing – original draft, Writing – review & editing, Supervision, Resources, Funding acquisition. **M. Rufo:** Formal analysis, Investigation, Data curation, Writing – review & editing, Validation, Resources, Funding acquisition, Supervision. **J. Paniagua:** Investigation, Validation, Data curation, Writing – review & editing, Resources, Funding acquisition, Supervision. **A. González-Mohino:** Conceptualization, Validation, Formal analysis, Investigation, Data curation, Writing – original draft, Writing – review & editing. **L.S. Olegario:** Investigation, Formal analysis, Data curation, Validation, Writing – review & editing, Supervision.

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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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.foodchem.2021.131721.

References

Alamprese, C., Grassi, S., Tugnolo, A., & Casiraghi, E. (2021). Prediction of olive ripening degree combining image analysis and FT-NIR spectroscopy for virgin olive oil optimisation. *Food Control, 123*, Article 107755. https://doi.org/10.1016/j.foodcont.2020.107755

Ali, S. K. M., & Ali, B. (2014). *Acoustics Impedance Studies in Some Commonly Used Edible Oils, 1*(9), 563–566.

Alouache, B., Khechena, F. K., Lecheb, F., & Boutkedjirt, T. (2015). Characterization of olive oil by ultrasonic and physico-chemical methods. *Physics Procedia, 70*, 1061–1065. https://doi.org/10.1016/j.phpro.2015.08.226

Alouache, B., Laux, D., Hamitouche, A., Bachari, K., & Boutkedjirt, T. (2018). Ultrasonic characterization of edible oils using a generalized fractional model. *Applied Acoustics, 131*, 70–78. https://doi.org/10.1016/j.apacoust.2017.10.014

Azman, N. A., & Abd Hamid, S. B. (2017). Determining the time of flight and speed of sound on different types of edible oil. *IOP Conference Series: Materials Science and Engineering, 260*, 012034. https://doi.org/10.1088/1757-899X/260/1/012034

Baêsso, R. M., Costa-Felix, R. P. B., Miloro, P., & Zeqiri, B. (2019). Ultrasonic parameter measurement as a means of assessing the quality of biodiesel production. *Fuel, 241*, 155–163.

Baêsso, R. M., Oliveira, P. A., Morais, G. C., Alvarenga, A. V., & Costa-Félix, R. P. B. (2016). Use of ultrasound to monitor physical properties of soybean oil. *Journal of Physics: Conference Series, 733*, 012042. https://doi.org/10.1088/1742-6596/733/1/012042

Ben-Ayed, R., Kamoun-Grati, N., & Rebai, A. (2013). An overview of the authentication of olive tree and oil. *Comprehensive Reviews in Food Science and Food Safety, 12*(2), 218–227. https://doi.org/10.1111/crf3.2013.12.issue-210.1111/1541-4337.12003

Benedito, J., García-Pérez, J. V., Carmen Dobarganes, M., & Mulet, A. (2007). Rapid evaluation of frying oil degradation using ultrasonic technology. *Food Research International, 40*(3), 406–414. https://doi.org/10.1016/j.foodres.2006.10.017

Benedito, J., Mulet, A., Velasco, J., & Dobarganes, M. C. (2002). Ultrasonic assessment of oil quality during frying. *Journal of Agricultural and Food Chemistry, 50*(16), 4531–4536.

Cerrillo, C., Jimenez, A., Rufo, M., Paniagua, J., & Pachon, F. T. (2014). New contributions to granite characterization by ultrasonic testing. *Ultrasonics, 54*(1), 156–167. https://doi.org/10.1016/j.ultras.2013.06.006

Codex Alimentarius. (2019). Codex Stan 210-1999 STANDARD FOR NAMED VEGETABLE OILS. In *Codex Alimentarius* (Vol. 8, Issue 5).

CodexAlimentarius. (2017). Codex Stan 33-1981. STANDARD FOR OLIVE OILS AND OLIVE POMACE OILS. In *Codex Alimentarius* (Vol. 91, Issue 5).

Correa, E. C., Roger, J. M., Lleó, L., Hernández-Sánchez, N., Barreiro, P., & Diezma, B. (2019). Optimal management of oil content variability in olive mill batches by NIR spectroscopy. *Scientific Reports, 9*(1), 13974. https://doi.org/10.1038/s41598-019-50342-6

Esteban, Bernat, Riba, Jordi-Roger, Baquero, Grau, Rius, Antoni, & Puig, Rita (2012). Temperature dependence of density and viscosity of vegetable oils. *Biomass and Bioenergy, 42*, 164–171.

Ghosh, S., Holmes, M., & Povey, M. (2017). Temperature dependence of bulk viscosity in edible oils using acoustic spectroscopy. *Journal of Food Processing & Technology, 08* (06). https://doi.org/10.4172/2157-7110.1000676

González-Mohino, Alberto, Jiménez, Antonio, Paniagua, M. Jesús, Perez-Palacios, Trinidad, & Rufo, Montaña (2019). New contributions of ultrasound inspection to the characterization of different varieties of honey. *Ultrasonics, 96*, 83–89. https://doi.org/10.1016/j.ultras.2019.02.010

González-Mohino, Alberto, Jiménez, Antonio, Rufo, Montaña, Paniagua, Jesús M., Olegario, Lary S., & Ventanas, Sonia (2021). Correlation analysis between acoustic and sensory technique data for cooked pork loin samples. *LWT, 141*, 110882. https://doi.org/10.1016/j.lwt.2021.110882

Gudra, T., & Opielinski, K. J. (2004). Applying spectrum analysis and cepstrum analysis to examine the cavitation threshold in water and in salt solution. *Ultrasonics, 42* (1–9), 621–627. https://doi.org/10.1016/j.ultras.2003.11.016

Gurdeniz, G., & Ozen, B. (2009). Detection of adulteration of extra-virgin olive oil by chemometric analysis of mid-infrared spectral data. *Food Chemistry, 116*(2), 519–525. https://doi.org/10.1016/j.foodchem.2009.02.068

Jiménez, A., Rufo, M., Paniagua, J. M., Crespo, A. T., Guerrero, M. P., & Riballo, M. J. (2017). Contributions to ultrasound monitoring of the process of milk curdling. *Ultrasonics, 76*, 192–199. https://doi.org/10.1016/j.ultras.2017.01.007

Kaufmann, Karine Cristine, Favero, Flávia de Faveri, de Vasconcelos, Marcus Arthur Marçal, Godoy, Helena Teixeira, Sampaio, Klicia Araujo, & Barbin, Douglas Fernandes (2019). Portable NIR spectrometer for prediction of palm oil acidity. *Journal of Food Science, 84*(3), 406–411.

Kaufmann, K. C., Sampaio, K. A., García-Martín, J. F., & Barbin, D. F. (2022). Identification of coriander oil adulteration using a portable NIR spectrometer. *Food Control, 132*, Article 108536. https://doi.org/10.1016/j.foodcont.2021.108536

Koc, A. B., & Ozer, B. (2008). Nondestructive monitoring of renetted whole milk during cheese manufacturing. *Food Research International, 41*(7), 745–750. https://doi.org/10.1016/j.foodres.2008.05.008

Mavromoustakos, T., Zervou, M., Bonas, G., Kolocouris, A., & Petrakis, P. (2000). Novel analytical method to detect adulteration of virgin olive oil by other oils. *JAOCS, Journal of the American Oil Chemists' Society, 77*(4), 405–411. https://doi.org/10.1007/s11746-000-0065-x

McClements, D. Julian, & Povey, Malcolm J. W. (1992). Ultrasonic analysis of edible fats and oils. *Ultrasonics, 30*(6), 383–388.

Rashed, Mahmoud Said, & Felfoldi, Jozsef (2016). Ultrasonic method for identifying oil types and their mixtures. *Progress in Agricultural Engineering Sciences, 14*(s1), 111–119. https://doi.org/10.1556/446.14.2018.s1.11

Rodenbush, C. M., Hsieh, F. H., & Viswanath, D. S. (1999). Density and viscosity of vegetable oils. *JAOCS, Journal of the American Oil Chemists' Society, 76*(12), 1415–1419. https://doi.org/10.1007/s11746-999-0177-1

Rubalya Valantina, S., Chandiramouli, R., & Neelamegam, P. (2013). Detection of adulteration in olive oil using rheological and ultrasonic parameters. *International Food Research Journal, 20*(6), 3197–3202.

Rufo, M. M., Jiménez, A., & Paniagua, J. M. (2014). Comparative Study of Different Methods to Assess Ultrasonic Velocities of Waves in a Liquid Medium. *11th European Conference on Non-Destructive Testing (ECNDT 2014)*, https://www.ndt.net/search/docs.php3?id=16557.

Rukke, E.-O., & Schüller, R. B. (2017). Viscosity of a selection of edible oils and blends of oils at different temperatures. *Annual Transactions - The Nordic Rheology Society, 25*, 287–292.

Sahasrabudhe, S. N., Rodriguez-Martinez, V., O'Meara, M., & Farkas, B. E. (2017). Density, viscosity, and surface tension of five vegetable oils at elevated temperatures: Measurement and modeling. *International Journal of Food Properties, 20*(sup2), 1965–1981.

Serway, R. A., & Jewett, J. W. (2018). *Physics for scientists and engineers* ((10th ed.).). Cengage learning.

Yan, J., Wright, W. M. D., O'Mahony, J. A., Roos, Y., Cuijpers, E., & van Ruth, S. M. (2019). A sound approach: Exploring a rapid and non-destructive ultrasonic pulse echo system for vegetable oils characterization. *Food Research International, 125* (June), Article 108552. https://doi.org/10.1016/j.foodres.2019.108552

Zhong, Xu, & Zhang, Dong (2021). Latency prediction of earmuff using a lumped parameter model. *Applied Acoustics, 176*, 107870. https://doi.org/10.1016/j.apacoust.2020.107870