

## Ultrasonic Assessment of Oil Quality during Frying

JOSE BENEDITO,<sup>\*,†</sup> ANTONIO MULET,<sup>†</sup> J. VELASCO,<sup>‡</sup> AND  
 M. CARMEN DOBARGANES<sup>‡</sup>

Food Technology Department, Universidad Politécnica de Valencia, Camino de Vera s/n,  
 46022, Valencia, Spain, and Food Quality and Characterization Department,  
 Instituto de la Grasa (CSIC), Avda Padre García Tejero, 4, 41012 Sevilla, Spain

In this paper, changes in ultrasonic properties during thermoxidation of virgin olive oil were studied. Samples of virgin olive oil were heated over different periods of time from 2 to 16 h at 200 °C. Oil degradation was characterized by means of physical and chemical changes, i.e., viscosity, color, polar compounds, polymers, and polar fatty acids. Ultrasonic measurements were carried out while the oil sample was cooled from 35 to 25 °C. It was found that velocity and attenuation measurements were related to viscosity measurements through a classical equation for viscous liquids. The ultrasonic measurements were also related to the percentages of polar compounds and polymers, which shows the feasibility of using ultrasonic properties to monitor oil quality. Nevertheless, as long as the ultrasonic measurements are temperature dependent, this variable must be controlled in order to obtain repetitive and reliable measurements.

**KEYWORDS:** Frying oil; polar compounds; ultrasonics; viscosity

### INTRODUCTION

The use of ultrasonics can be divided into low- and high-intensity applications, depending on whether the purpose is to assess some material characteristics or affect a process by means of introducing ultrasonic energy in the system (1). Low-intensity equipment and techniques have been used in the past for many industrial applications. Specifically, in the food industry the applications have been extended to, among others, vegetables, fruits, meat, dairy products, and fats and oils (1, 2).

The use of ultrasonics for meat composition assessment (fat content and distribution) was one of the first applications in the food industry and currently has the highest number of literature references (3–6). For vegetables and fruits, ultrasonics have been used mainly to assess ripeness, by relating the ultrasonic parameters to textural or sugar content changes (7, 8). In the dairy industry, ultrasonics have been used, among other applications, to determine the optimum rennet cut time, to monitor the degree of cheese maturity, to assess cheese composition, and also to detect cracks within cheese pieces (9–11).

The ultrasonic velocity in a fatty material decreases as temperature increases showing three different regions (1). In the first region, the triglycerides are completely solid and therefore the decrease in velocity is due to the negative temperature coefficient of the ultrasonic velocity in the solid fat. In the second region, some of the triglycerides start melting which causes a faster velocity decrease. Finally, for higher

temperatures, only liquid oil is found (third region) and the decrease is due to the negative temperature coefficient of velocity as was the case for solid fat. Ultrasonic applications can be found for the three regions. Miles et al., (12) used ultrasonics to study the solid fat content in adipose tissue in order to establish the suitability for bacon manufacturing. The solid fat content has also been ultrasonically assessed because it has an important implication in the texture, spreadability, and consistency of many fatty materials such as margarine or butter (1).

The ultrasonic velocity has been measured to determine the chemical structure of different oils including the chain length and degree of unsaturation (13). Therefore, the velocity measurements can be used for oil composition and adulteration assessment (14, 15). Attenuation and velocity have also been correlated to rheological properties of edible (castor, olive, groundnut, sunflower, and rapeseed) oils (16).

Changes in ultrasonic velocity are also expected in oils and fats subjected to processes involving high temperatures. Deep frying is one of the most commonly used procedures for the preparation of foods, and, hence, different aspects of the process have been extensively studied for many years (17). During frying, a complex series of reactions takes place resulting in hydrolysis, oxidation, and polymerization of the oil. The quality of fried foods is affected by that of the oil, and, consequently, regulations or guidelines have been established in many countries to guarantee high-quality foods. Limits for polar compounds of around 25% have been established in most of the official regulations, limiting frying fats and oil degradation for human consumption, although polymers and free fatty acids have also been considered in some countries (18).

\* To whom correspondence should be addressed. Phone/fax: 34-963879366. E-mail: jjbenedi@tal.upv.es.

<sup>†</sup> Universidad Politécnica de Valencia.

<sup>‡</sup> Instituto de la Grasa (CSIC).

Unfortunately, studies carried out in different countries on samples collected by their inspection services indicate the need for improving the quality of frying oil to produce more nutritious fried foods (19). In this respect, efficiency of quality control is dependent on the existence of objective analytical methods, compatible with the needs of the fryer operators. However, it is known that official methods are applied only in public laboratories connected to inspection services and in research studies. The main reason is that they are expensive and time-consuming techniques, requiring technical personnel and laboratory facilities.

In general, methods for determining when to discard fats in fried food outlets are based on physical changes whose reliability depends on the skill of the fryer operator, and oil abuses occur because of the lack of correlation between these criteria and official methods (20). Consequently, more research is needed on rapid measurements, i.e., viscosity, color, ultrasonics, etc., to be applied *in situ* to define cutoff levels at which the fats should be replaced.

In a previous paper, the changes in ultrasonic velocity for corn oil subjected to different heating periods have been reported (21). Unfortunately, no information is given on the chemical degradation of the samples and, consequently, relationships between changes in ultrasonic properties and the main parameters limiting the use of frying fats for human consumption were not studied.

The objective of this paper was to study the changes in ultrasonic velocity and attenuation during thermoxidation of virgin olive oil in order to assess the feasibility of using ultrasonic techniques to monitor the quality of frying oils and to determine the point for discarding used frying fats and oils according to the present regulations. In this respect, relationships between ultrasonic velocity or attenuation and the main physical and chemical parameters measuring frying oil degradation have been defined.

## MATERIALS AND METHODS

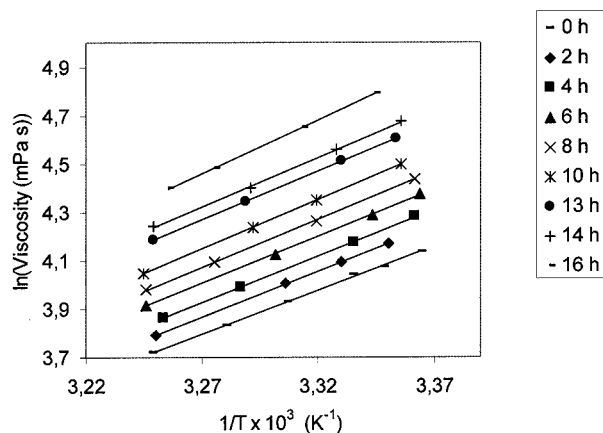
Extra virgin olive oil bought in a local supermarket (Valencia, Spain) was heated in a domestic fryer at 200 °C for 16 h. Samples of 150 mL were periodically removed and frozen at -20 °C until analysis.

**Ultrasonic Measurements.** The experimental setup used for the experiments consisted of two narrow-band ultrasonic transducers (1 MHz, 0.75" in. crystal diameter, A314S-SU model, Panametrics, Waltham, MA), attached to a cubic container (49 × 49 × 49 mm) where the oil samples were placed. The container was introduced into a temperature-controlled bath to maintain the sample temperature, and the oil was moderately stirred to avoid formation of bubbles. The ultrasonic measurements were carried out while the oil was cooled from 35 to 25 °C. The transducers were linked to a pulser-receiver (Toneburst Computer Controlled, model PR5000-HP, Matec Instruments, Northborough, MA) which sent the electrical signal to a digital storage oscilloscope (Tektronix TM TDS 420, Tektronix, Inc., Wilsonville, OR). For each ultrasonic measurement, five signal acquisitions were taken and averaged. The time-of-flight was computed on the averaged signal and the ultrasonic velocity was calculated taking into account the system delay (which was previously calibrated as described in Benedito et al. (10)).

The attenuation coefficient ( $\alpha$ ) was computed by fitting the experimental data to eq 1 (1).

$$\ln A = \ln A_0 - \alpha d \quad (1)$$

where  $d$  is the distance traveled by the wave,  $A_0$  is the initial amplitude of the signal measured as the peak to peak voltage, and  $A$  is the amplitude of the signal at distance  $d$ . Four ultrasonic echoes were considered to compute attenuation.



**Figure 1.** Relationship between viscosity and temperature; Arrhenius model.

**Viscosity.** The viscosity of the samples was measured using a falling ball viscometer (Haake falling ball viscometer C, Karlsruhe, Germany) for transparent liquids. The apparatus had a tempering jacket which allowed control of the temperature of the experiments in the range considered (25–35 °C). The temperature was measured directly by inserting a probe in the falling tube. The experiments were carried out by triplicate and the average was considered.

**Density.** The density of the samples was measured with a specific gravity hydrometer (Proton, model 72082, Barcelona, Spain) at 20 °C. The oil samples were placed into a glass container, which was introduced into a temperature controlled bath at 20 °C and allowed to reach temperature equilibrium for 2 h. Next, the hydrometer was inserted in the cylinder and the density was directly read on the scale.

**Color.** The color measurements were carried out by triplicate using a colorimeter equipped with a set of colored glasses, according to the British Lovibond Color Scale (Lovibond Scale). Lovibond Color Standards were followed as described in the Official Methods and Recommended Practices of the American Oil Chemists' Society (Cc 13e-92, AOCS, (22)). In preliminary experiments it was observed that yellow was the only color index varying consistently during frying. Therefore, only yellow measurements are reported in this paper.

**Chemical Analyses. Polar Compounds.** Polar compounds were quantified by adsorption chromatography following the IUPAC Standard Method 2.507 (23).

**Polymers.** Triacylglycerol polymers were quantified by high-performance size exclusion chromatography (HPSEC) following the IUPAC Standard Method (24)

**Fatty Acids.** Fatty acids were analyzed by GLC after derivatization to fatty acid methyl esters (FAME) with KOH 2 N in methanol, and triheptadecanoin as internal standard, according to the IUPAC Standard Method (23). A HP 6890 chromatograph on a HP Innowax capillary column (poly(ethylene glycol), 30 m × 0.25 mm i.d., film thickness 0.25  $\mu$ m) (Hewlett-Packard, Palo Alto, CA), was used under the following temperature program: 180 °C (4 min), 4 °C/min to 230 °C (15 min). Samples were introduced into the column via a split injector (split ratio 1:40) at 250 °C and the flow rate of hydrogen, used as carrier gas, was 1 mL/min. Temperature of both split injector and flame ionization detector was 250 °C.

## RESULTS AND DISCUSSION

**Changes in the Physicochemical Characteristics during Heating.** The variation of viscosity ( $\mu$ ) with temperature ( $T$ ) is usually described using an Arrhenius-type equation ( $\ln \mu = \ln(A+B)/T$ ), (25, 26). **Figure 1** shows the plot of the Neperian logarithm of viscosity versus the inverse of temperature for the different frying times. The close fit of the experimental data to the Arrhenius equation (continuous lines) indicates that the model can be used for the range of frying times and temperatures considered in this study.

**Table 1.** Physico-Chemical Characteristics of Olive Oil at Different Heating Times

heating time (h)	viscosity at 30 °C (mPa s)	color (yellow)	polar compounds (%)	polymers (%)
0	50.3	1.11	6.2	trace
2	54.4	1.20	11.3	2.6
4	58.0	1.68	22.1	6.0
6	63.1	1.70	25.8	8.0
8	67.5	1.88	29.2	11.9
10	72.9	2.04	33.1	15.0
13	82.1	2.36	37.9	19.1
14	84.8	3.62	40.5	21.1
16	100.4	3.90	45.7	25.8

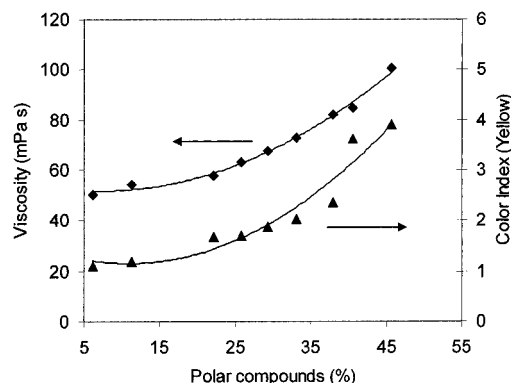
**Table 2.** Quantitation of Major Fatty Acids (%) in Samples of Olive Oil Heated at 200 °C

heating time (h)	C16:0	C16:1	C18:0	C18:1	C18:2	others	polar fatty acids
0	16.3	2.3	2.2	61.3	16.2	1.4	0.4
2	16.3	2.4	2.1	60.8	15.8	1.5	1.1
4	16.4	2.4	2.0	58.6	14.1	1.9	4.7
6	16.2	2.3	2.2	55.8	13.2	1.5	8.8
8	16.5	2.4	2.0	55.2	12.6	2.0	9.3
10	16.2	2.2	2.1	54.3	11.3	2.4	11.5
13	16.6	2.2	2.2	53.4	10.2	2.3	13.1
14	16.4	2.0	2.3	52.5	8.9	2.7	15.2
16	16.5	2.1	2.1	52.3	7.4	1.8	17.8

**Tables 1** and **2** show the results for the physicochemical analysis carried out on the oil samples at different frying times. The viscosity at 30 °C was interpolated using the Arrhenius-type equations. As can be observed from **Table 1** and **Figure 1**, viscosity increases during simulated frying for all the temperatures as has been previously found by other authors (27, 28). This increase in viscosity is considered a good indicator of the oil alteration. For example, in Belgium the oil is discarded when viscosity exceeds a specified limit (25 mPa s at 50 °C). Thus, the obtained equations could be used to interpolate viscosity measurements in the range of temperatures considered, to examine viscosity at a given temperature. On the other hand, the oil density (measured at 20 °C) varied from 914 to 925 kg/m<sup>3</sup> (0 to 16 h).

Another possible indicator of the oil alteration during frying was found to be the yellow color index (**Table 1**). Similar color changes have been found for other frying oils such as canola, soybean, and corn oil (29).

Both changes in viscosity and color with heating time fitted a quadratic equation ( $R^2 = 0.98$ ;  $p < 0.001$  and  $R^2 = 0.91$ ;  $p < 0.01$  respectively). The final columns in **Table 1** show the levels of the main compounds formed during thermoxidation and frying. First, polar compound percentages give information about the total new compounds formed through oxidative, thermal, and hydrolytic reactions, while polymers, one of the group of polar compounds, are the most representative compounds which originate at the highest temperature of the frying process. As can be observed, polar compound levels of samples heated for less than 6 h were compatible with the established regulations on frying fats and oils (around 25%) although it is not strange to find used frying oils and fats with polar compound percentages as high as those found for olive oil heated for 16 h (19). On the other hand, polymeric compounds are also the basis for discarding fats. A maximum level of 16% is admitted

**Figure 2.** Relationship between the percentage of polar compounds and physical measurements.

in The Netherlands, which is exceeded after 10 h in the case of virgin olive oil under the conditions used in this study.

**Table 2** summarizes the changes in fatty acids at the different heating periods. As expected, the degradation affected unsaturated fatty acids, with levels being higher for the most unsaturated fatty acid, while saturated fatty acids remained at the initial levels. In the last column, an estimation of polar fatty acids has been included. It has been calculated as the noneluting compounds under the chromatographic conditions applied (100 –  $\Sigma$  fatty acids). It is important to remark that polar compounds and polymeric compounds are quantified directly in the samples without previous derivatization, and consequently quantitative data refer to triglyceride molecules. In this regard, quantitation of polar fatty acids provides additional information on the fatty acyl groups attached to the glyceridic backbone undergoing thermal or oxidative degradation and contributing to modifying the nutritional properties of the initial oil. Percentages of polar fatty acids ranged from 0.4 to 17.8%, and were, as expected, much lower than those parallel values found for polar compounds (6.2 and 45.7%, respectively).

**Figure 2** shows the relationship between the percentage of polar compounds and viscosity and the color index. In both cases the experimental data was fitted closely using quadratic equations ( $R^2 = 0.99$ ;  $p < 0.001$  and  $R^2 = 0.93$ ;  $p < 0.01$  for viscosity and color respectively). The closer fit for viscosity could be due to the fact that the visual color measurements are subject to operator variability (30).

As long as the changes in viscosity and color are related to the variation in the percentage of polar compounds, which is widely considered to monitor the oil alteration including the Spanish legislation for olive oil (31, 32), they could be used to assess oil quality.

**Ultrasonic Measurements.** The acoustic parameters (velocity and attenuation) depend on the physicochemical properties of the medium and therefore are greatly influenced by temperature. Ultrasonic velocity increases in line with the temperature in water but decreases in fat, which is considered as the basis of the ultrasonic composition assessment (33, 6). The influence of temperature on attenuation is not so widely reported in the literature.

**Figure 3** shows the ultrasonic velocity at different temperatures for oil samples fried for 2, 10, and 16 h. As expected, the ultrasonic velocity decreases linearly with temperature in all cases, as in the case for other liquid and solid fats (1, 10, 12). **Table 3** shows the linear coefficients found for the different frying times. The slope ( $a$ ) ranges from  $-3.37$  to  $-3.54$  m s<sup>-1</sup> °C<sup>-1</sup> very close to the value reported by McClements (1) for liquid oils ( $-3.30$  m s<sup>-1</sup> °C<sup>-1</sup>). The temperature coefficient for

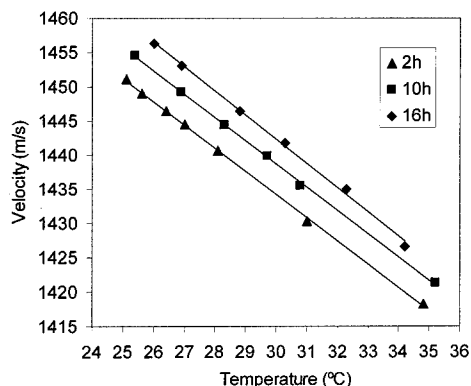


Figure 3. Variation of velocity with temperature for different frying times.

Table 3. Linear Correlations between Velocity and Temperature ( $v = aT + b$ )

heating time (h)	<i>a</i>	<i>b</i>	<i>R</i> <sup>2</sup>
0	-3.38	1535.4	0.9991
2	-3.39	1536.0	0.9995
4	-3.37	1537.2	0.9992
6	-3.38	1538.0	0.9993
8	-3.39	1539.2	0.9996
10	-3.40	1540.8	0.9995
13	-3.37	1541.2	0.9995
14	-3.40	1542.3	0.9989
16	-3.54	1542.3	0.9982

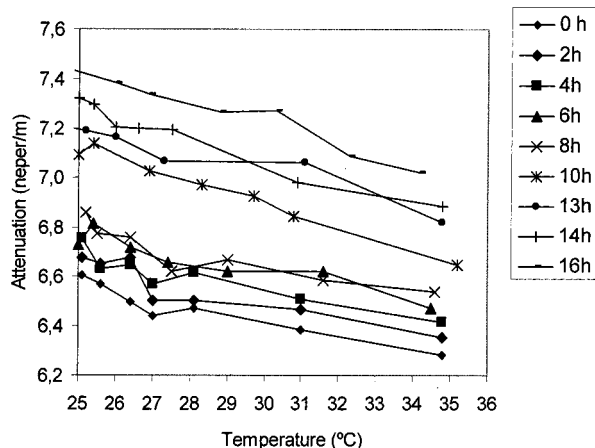


Figure 4. Variation of attenuation with temperature for different frying times.

ultrasonic velocity in solid fats decreases to  $-5 \text{ m s}^{-1} \text{ } ^\circ\text{C}^{-1}$  (6, 12). For mixtures of solid/liquid fats, the temperature coefficient for velocity is comprised within the values for solid and liquid fats.

The ultrasonic attenuation decreases as temperature increases (Figure 4) probably due to the greater influence of the decrease of viscosity compared to the decrease of velocity and density. Both velocity and attenuation increase during frying (Figures 3 and 4) due to the changes in the physicochemical properties of the oil. Lacey and Payne (21) also found an increase of the ultrasonic velocity at 30 °C during simulated frying of corn oil at 170 °C. They found a change of velocity from 1439 to 1447 m/s, which are very similar to those values obtained at 30 °C for olive oil (1433 to 1442 m/s). Interestingly, a significant change was produced at any temperature between samples heated for 8 and 10 h. These values, for polar compounds and polymers, were close to those established for discarding used frying oils (29.2 and 33.1% for polar compounds and 12.9 and

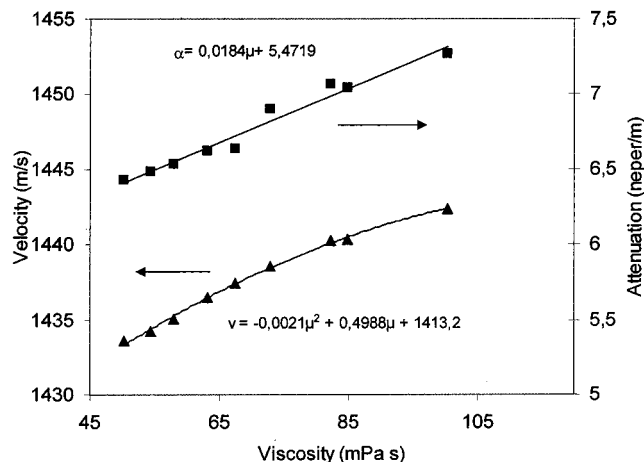


Figure 5. Relationship between viscosity and the ultrasonic parameters at 30 °C.

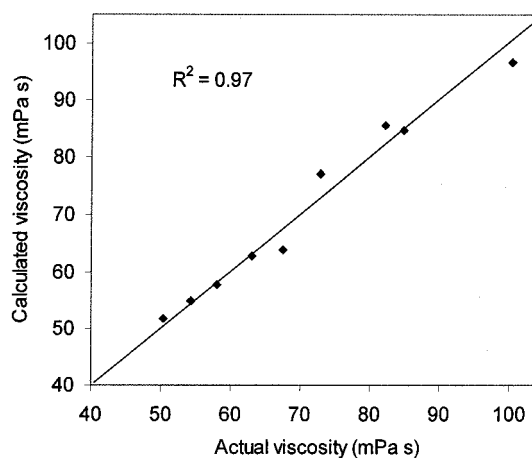


Figure 6. Actual versus calculated viscosity using eq 2.

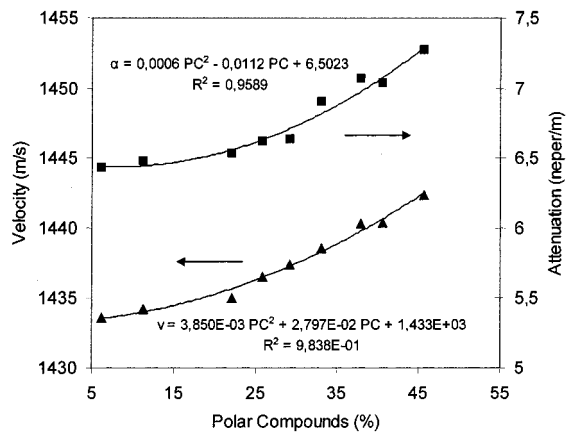
15.0% for polymeric compounds, respectively), suggesting that values for attenuation between 6.6 and 7.1 Neper/m depending on the temperature selected, would have been useful for discarding the oil. Figure 5 shows the relationship between viscosity and the ultrasonic parameters (velocity and attenuation). The attenuation coefficients at 30 °C were interpolated from the experimental data (Figure 4) using the Splines method (34). A close and significant fit was found when relating viscosity to velocity using a polynomial model of second order ( $R^2 = 0.99$ ;  $p < 0.0001$ ). On the other hand, viscosity was linearly related to attenuation ( $R^2 = 0.96$ ;  $p < 0.001$ ).

To examine the relationship between velocity, attenuation, and viscosity a model (eq 2) derived from the classical equation for viscous liquids (eq 1) was used, considering density as constant within the temperature range considered.

$$\alpha = \frac{c}{v^3}[\mu + d] \quad \mu = \frac{\alpha v^3}{c} - d \quad (2)$$

where  $c$  and  $d$  are two constants.

Viscosity was calculated from eq 2. For that purpose the constants  $c$  and  $d$  were computed using the tool Solver from Excel, minimizing the square differences between the experimental viscosity and viscosity calculated using eq 2. The regression coefficients were  $c = 0.0647$  (Neper/m) ( $\text{cm}^3/\text{s}^3$ ) / (mPa s) and  $d = 240.32$  (mPa s). Figure 6 shows the close fit between the calculated and experimental viscosities. It can be observed that the correlation including velocity and attenuation



**Figure 7.** Relationship between the percentage of polar compounds and the ultrasonic parameters at 30 °C.

is poorer than that including only velocity, which is probably due to the higher variability found for the attenuation measurements (**Figure 4**). Lacey and Payne (21) used a similar model which considered only viscosity and the ultrasonic velocity.

**Figure 7** shows the relationship between the percentage of polar compounds and the ultrasonic parameters. As long as the polar compound percentage is related to changes in physicochemical properties, such as viscosity and probably density and the bulk modulus, the ultrasonic velocity and attenuation will be linked to this chemical parameter, widely considered as an oil quality indicator.

These results show that ultrasonic measurements could be used to monitor the alteration of olive oil during heating and, consequently, during frying. The use of an automated ultrasonic system could replace traditional methods, providing a fast measurement.

## CONCLUSIONS

The change in the chemical composition during simulated olive oil frying, which determines oil quality, is linked to a change of viscosity and color. Velocity and attenuation increased during frying due to the changes in the physicochemical oil properties. Velocity and attenuation measurements were found to be related to viscosity measurements through a classical equation for viscous liquids. The ultrasonic measurements are also related to the percentage of polar compounds, which shows the feasibility of using a continuous and automated ultrasonic system to monitor the oil quality. As long as the ultrasonic measurements are temperature dependent, this variable must be controlled in order to obtain repetitive and reliable measurements. Additional research should be done in order to develop sufficiently precise correlations, which can lead to determining the discard point for different frying oils. The influence of different fried products should also be established.

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