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Characterization of Olive Oil by Ultrasonic and Physico-chemical Methods

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Abstract

Olive oil excels by its nutritional and medicinal benefits. It can be consumed without any treatment. However, its quality can be altered by inadequate storage conditions or if it is mixed with other kinds of oils. The objective of this work is to demonstrate the ability of ultrasonic methods to characterize and control olive oil quality. By using of a transducer of 2.25 MHz nominal frequency, in pulse echo mode, ultrasonic parameters, such as propagation velocity and attenuation, have been measured for pure olive oil and for its mixtures with sunflower oil at different proportions. Mechanical properties, such as density and viscosity, have also been determined. The results of ultrasonic measurements are consistent with those obtained by physico-chemical methods, such as rancidity degree, acid index, UV specific extinction coefficient and viscosity. They show that the ultrasonic method allows to distinguish between mixtures at different proportions. The study allows concluding that ultrasound techniques can be considered as a useful complement to existing physico-chemical analysis techniques.

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Keywords: Olive oil; Quality control; Ultrasonic velocity; Ultrasonic attenuation; Physico-chemical methods.

1. Introduction

Ultrasound is a noninvasive technique that was widely used in food industry. In the literature, many applications of this technique can be found for different types of products, ranging from liquids to solids (Jayani et al. (2012a, 2012b)). Ultrasound can be applied to solve some problems found in the oil related industries. In this regard, ultrasonic velocity has also been measured to determine the chemical structure of different oils including the

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chain length and degree of unsaturation (Javanaud and Rahalkar (1988)) and to assess oil composition and adulteration (Coupland and McClements (1997)). Although oil is among low attenuative materials, attenuation coefficient can also be considered. For that, Benedito et al. (2007) have used both velocity and attenuation to characterize frying oil degradation. Gradwell et al. (1985) have correlated them to rheological properties of many edible oils.
The objective of this work is to demonstrate the ability of the ultrasonic methods to characterize and control the olive oil quality. In this order, acoustic parameters such as velocity and attenuation and mechanical properties such as density and viscosity, for pure olive oil and for its mixtures with soya oil at different proportions have been measured.

2. Materials and Methods

Olive and soya oils were purchased from a local retailer and used without further treatment. Characterizations are done on 6 samples of olive oil, soya oil and 4 mixtures at different percentages of soya oil, that is: 20%, 40%, 60% and 80%.

2.1. Ultrasonic Measurements

The experimental setup consisted of an ultrasonic transducer (2.25 MHz, 0.5 in. diameter, Panametrics, Olympus), excited with a Sofranel 5073PR pulser-receiver. Electrical signals were sent to a Tektronix TDS 2012 digital oscilloscope connected to a computer via an IEEE-GPIB bus. The transducer was attached and coupled by an ultrasonic gel to the oil container. In this study, the samples were placed into two containers of respective dimensions ($75\times75\times40mm^3$) and ($75\times75\times30mm^3$). Measurements have been done in echo mode. Therefore, the propagation velocity of the ultrasonic wave can be given by the following expression:

$$V_g = {2\Delta z}/{\tau}. \tag{1}$$

Δz represents the difference in the distance traveled by the ultrasonic wave in two samples of different thicknesses and τ the corresponding time of flight the wave. This latter has been calculated by estimating the difference between the positions of the first zero crossings of the signals corresponding to the first echoes emanating from the rear faces of the two samples respectively. To determine the attenuation coefficient and phase velocity of the ultrasonic wave, a broadband spectroscopy method, detailed in (Peters and Petit (2003)), has been applied.
If we assume a planar front wave, z being the distance traveled by the ultrasonic wave in the sample, the acoustic pressure can be written as:

$$p(z,t) = \int_{-\infty}^{+\infty} P(f) e^{-\alpha(f)z} e^{2i\pi f\left(t-\frac{z}{c(f)}\right)} df, \tag{2}$$

where $\alpha(f)$ is the attenuation coefficient, $c(f)$ the phase velocity, $P(f)$ the Fourier transform of the acoustic pressure $p(z,t)$ and f the frequency. If we consider that z_1 and z_2 are two different positions in the sample, the velocity and attenuation coefficient can be calculated and written as:

$$c(f) = \frac{2\pi f \Delta z}{\Delta\varphi - 2\pi f \tau}, \tag{3}$$

$$\alpha(f) = \frac{1}{z_2 - z_1} \, log\left|\frac{P(z_1,f)}{P(z_2,f)}\right|, \tag{4}$$

where $\Delta\varphi = Arg\left(\left|\frac{P(z_2,f)}{P(z_1,f)}\right|\right)$ is the absolute phase and $\boldsymbol{\tau}$ the difference in the time of flight of the ultrasonic pulses.

2.2. Physico-chemical Analyses

2.2.1. Fatty acids

The content of fatty acids was analyzed by measuring the acid value. This value represents the necessary potassium quantity for the neutralization of the acidity of 1g of fats (oil). It allows to assess the state of deterioration of the free acid fats. In our case, it is determined according to the standard (AFNOR 1984), by dissolving a sample in a solvent mixture and the titration of free fatty acids by means of a hydrochloric acid solution.

2.2.2. Ultraviolet spectrophotometry (Specific extinction coefficient)

Ultraviolet spectrophotometry analysis corresponds to the maximum absorption of the conjugated dienes and trienes resulting from the decomposition of oils. The values of these absorbances are expressed as specific extinctions, conventionally denoted by K (extinction coefficient). The specific extinction was determined according to the standard (IOC/T.20/Doc No.19/Rev.3.) at 232 nm (K232) and 270 nm (K270), using a spectrophotometer Hελios UV / Vis.

2.2.3. Degree of rancidity

This test is used to evaluate the oxidative stability of oils. Specifying TIR (Induction time in Rancimat, expressed in hours) corresponds to the duration time for which the oil has withstood oxidative stress. The degree of rancidity was determined by a Rancimat 743 - Metrohm according to the standard (ISO 6886).

2.2.4. Viscosity

The viscosity of the samples was measured using a rotary viscometer (Nahita, Model 801), which can be used for edible oils and similar products. The measurements were made at room temperature by directly inserting a probe into the sample. The experiments were carried out in triplicate and the average was considered.

3. Results and Discussions

Fig. 1 shows the variations of the ultrasonic propagation velocity and the acidity index. Both increase with the percentage of added soya oil. Olive oil comprises triglycerides. Each of these elements consists of three fatty acids. When triglycerides are degraded, fatty acids are detached and become free in oil. Their percentage determines the acidity of the oil.

Initially, the rate of free fatty acids in olive oil is more important than in refined soya oil (which doesn't contain pigments, such as carotenoids and chlorophylls, and aromatic cells) (Fig.1). However, due to storage conditions (light, temperature...), this rate of free fatty acids increases rapidly in soya oil, lacking of antioxidants, unlike olive oil. This explains the increase in acidity according to the percentage of soya oil added to the mixture. The same trend is observed for the phase velocity and for the group velocity of ultrasonic waves in the oil mixtures.

From Fig.2, it can be noted that when the percentage of soya oil is greater, the values of the specific extinction K232 become important. This indicates that the number of hydro-peroxides detected by UV absorption at 232 nm is more important in this oil. We found the same tendency for the phase velocity measured at 2.25 MHz.

In Fig.3, the variations of the ultrasonic attenuation coefficient and the rancidity are represented. Both decrease according to the added soya oil percentage. This decrease of attenuation is related to the viscosity, which is less important for soya oil. The total oxidation time, which is due to the number of antioxidants, is high in olive oil. By the addition of soya oil, oxidation time is reduced, because the latter is a refined oil that does not contain antioxidants.

In addition to these measurements, the viscosity of olive oil and its mixtures has been determined. Its variations (Fig. 4) showed a quasi-linear decrease from (74,1± 0,7) mPa.s to (62,8± 0,6) mPa.s, when the percentage of added soya oil increased. This is consistent with the variations of the ultrasonic attenuation coefficient and denotes the smaller viscosity of soja oil compared to that of the olive oil.

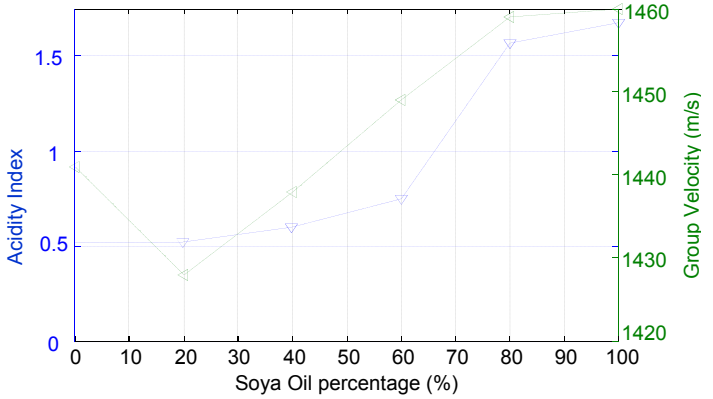

Fig. 1. Variations of the ultrasonic propagation velocity and the acidity value as a function of the percentage of soya oil in olive oil.

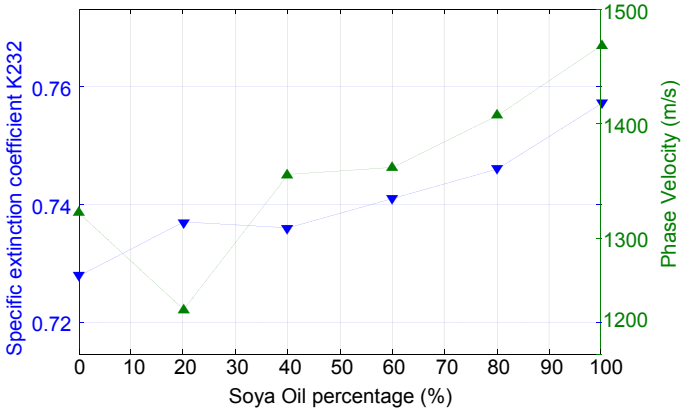

Fig. 2. Evolution of phase velocity and the specific extinction coefficient K232 as a function of the percentage of soya oil in olive oil.

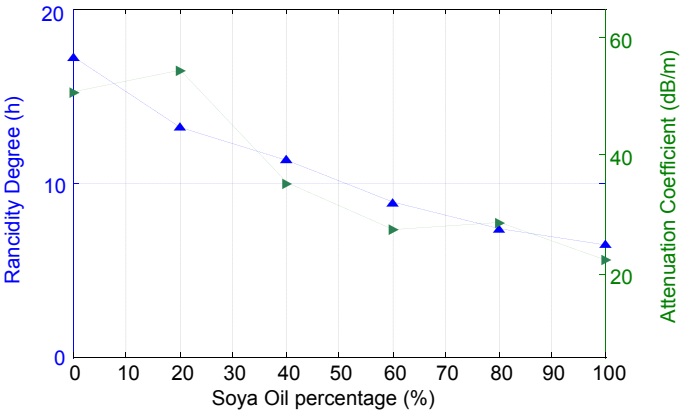

Fig. 3. Variations of ultrasonic attenuation coefficient and degree of rancidity versus soya oil percentage in olive oil.

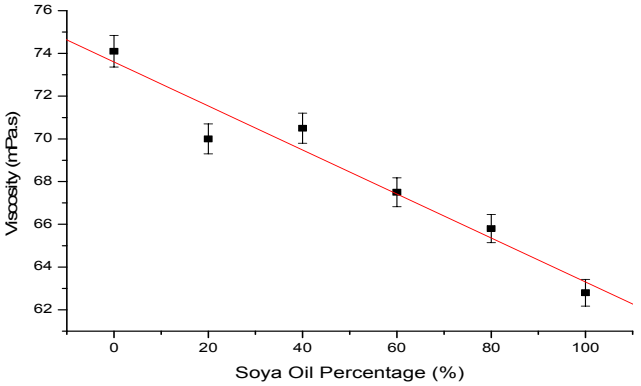

Fig. 4. Evolution of viscosity versus soya oil percentage in olive oil.

4. Conclusion

The physico-chemical control methods are relatively binding, because of their someway sophisticated implementation. For these methods, the tested material is unrecoverable with sometimes a high cost. But, in return, their use gives a guarantee of quality, purity and food safety for which consumers are increasingly attached.

Our study showed that there is a good correlation between the measured acoustic and mechanical parameters (an increase of the group velocity, phase velocity, density and a decrease of attenuation and viscosity according to the percentage of soya oil in the mixtures).

The results of physico-chemical analyzes (acid value, UV specific extinction, peroxide value and degree of rancidity) are also consistent with the increase in the percentage of soya oil (The increase of free fatty acids levels and hydroperoxides absorbance, the decrease of peroxides number and antioxidants).

As an example, the specific extinction and the phase velocity variations tendency as well as the acid index and the group velocity increasing are consistent. It is the same for the decrease of rancidity degree and of the attenuation coefficient as a function of the percentage of soya oil.

These results show the ability of ultrasound methods to characterize olive oil and to detect its possible adulterating by soya oil. Furthermore, they show that these methods may be a non-invasive, useful tool in addition to physico-chemical analysis used in the quality control of food oils.

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