Design and implementation of an ultrasonic sensor for rapid monitoring of industrial malolactic fermentations of wines.

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Abstract:

Ultrasound is an emerging technology that can be applied to monitor food processes. However, ultrasonic techniques are usually limited to research activities within a laboratory environment and they are not extensively used in industrial processes. The aim of this paper is to describe a novel ultrasonic sensor designed to monitor physical-chemical changes that occur in wines stored in industrial tanks. Essentially, the sensor consists of an ultrasonic transducer in contact with a buffer rod, mounted inside a stainless steel tube section. This structure allows the ultrasonic sensor to be directly installed in stainless steel tanks of an industrial plant. The operating principle of this design is based on the measurement of the ultrasonic velocity of propagation. In order to test its proper operation, the sensor has been used to measure changes of concentration in aqueous samples and to monitor the progress of a malolactic fermentation of red wines in various commercial wineries. Results show the feasibility of using this sensor for monitoring malolactic fermentations in red wines placed in industrial tanks.

Keywords: Sensor design; Industrial measurements; Process monitoring; Ultrasound, Malolactic fermentation.

Introduction

Full automation of processes in food and beverage industries is desirable as it can increase plant productivity, reduce wastage of raw materials and help to achieve a constant quality of the final product. However, in practice, many food and beverage plants are not fully automated. This can often be attributed to technical difficulties related to on-line sensing of food/beverage properties.

Several conventional measurement methods for monitoring food/beverage processes, such as Paper Chromatography (PC), Thin Layer Chromatography (TLC), High-Performance Liquid Chromatography (HPLC), enzymatic analysis, Fourier-transform Infrared Spectroscopy (FT-IR) and reflectance are described in the literature[1]. Most of these methods, do however, share the fact that they are expensive, rather complex, and require the taking of samples that need to be sent to an external lab for further analysis. Moreover, when these methods are used, obtaining accurate results tends to be a rather time consuming process. On top of all of that, these methods themselves are, generally speaking, not affordable to small companies.

Ultrasound is an emerging and promising technology that can be applied to food/beverage processing and property sensing. Unlike conventional methods, ultrasonic techniques are non-invasive, non-destructive, accurate, fast, inexpensive, on-line and suitable for process automation[2]. Online measurements of ultrasonic velocity of propagation can be used to monitor concentrations in solutions [3-4], process fermentation[5-12] and food composition [13-19]. Recently, some reviews related to the applications of ultrasound in analysis of food have been published [20-21]. However, these techniques are very sensitive to physical parameters such as temperature, and for this reason are usually limited to research activities within a laboratory environment [22-23], and are not used in-situ in industrial processes.

Most of the papers referenced above focus on the analysis method and the application, but the sensor design itself is not described. This is because the sensor used is generally a commercial off-the-shelf part suitable for a laboratory environment[24]. Unfortunately, commercial off-the-shelf ultrasonic sensors targeted at liquid measurements and specifically designed to be placed inside industrial tanks are not currently available. In these cases, the sensor has to be designed by the user.

Ultrasonic sensors are widely used in the industry for distance measurement, fill level monitoring and obstacle detection. However, they are rarely used to monitor food/beverage properties during a process. As a result, there are very few papers describing the design of ultrasonic sensors targeted at measurements of chemical changes that occur in liquids stored in industrial tanks. Some of these papers focus on the sensor operating principle and sensor setup[25]. Others focus on describing the ultrasonic sensor design but for sensors measuring temperature, density and viscosity[26-33]. But papers actually describing the design and industrial implementation of ultrasonic sensors used for the measurement of chemical properties of food in industrial processes are hardly found in the literature.

In a previous paper [12] the authors described an experimental study of the ultrasonic propagation velocity in laboratory mixtures of water–ethanol–malic acid and lactic acid. A good correlation was found between the ultrasonic velocity and malic and lactic acid concentrations. These results indicated the great potential of the ultrasonic technique to determine malic and lactic acid concentrations during the malolactic fermentation process.

In this paper, the ultrasonic- sensor targeted for food/beverage measurements is described. As a novelty, this sensor is specifically designed to monitor food/beverage processes in industrial environments. Design of the ultrasonic sensor, its operating principle, sensor dimensioning, materials used, error and uncertainty in the measurements and signal processing are described in this paper. In order to test its proper operation, this sensor has been used to measure changes of concentration in aqueous samples and to monitor a malolactic fermentation in red wines. The obtained results are also discussed and conclusions are given.

Material and methods.

Preliminary

The main purpose of this sensor design is the monitoring of physicochemical changes that occur in liquid media during an industrial process by measuring the ultrasonic velocity of propagation in the liquid. The main advantage of this sensor is that it has been designed to be easily used in industrial processes carried out within tanks. The following subsections give the details of the operating principle and construction of the sensor.

Operating principle

The velocity of an ultrasonic wave when propagating through a liquid can be easily obtained with the basic configuration depicted in Figure 1. This picture setup shows an ultrasonic transducer operating both as a transmitter and receiver, which is fixed to one side of a buffer rod. The liquid sample sits between the far side of the buffer rod and the reflector. The working principle is as follows: first, an electric pulsed signal is applied to the transducer which is then converted into an ultrasonic wave (A_0) that propagates across the buffer rod. At the far side of the buffer rod, i.e. the part of the rod that is in direct contact with the liquid, part of the incident wave is echoed back onto the ultrasonic transducer (A_{r1}) and part is transmitted through the liquid towards the wave reflector (A_{t1}). This transmitted ultrasonic signal travels through the liquid path length (L_{liquid}), and is echoed back at the wave reflector and, eventually, it reaches the liquid-buffer interface. At this point, part of the signal is reflected again onto the liquid (A_{t1r1}) and part is transmitted through the buffer rod to the ultrasonic transducer, where it is detected. This signal is often called the measurement signal (A_{m1}).

The time of flight of the ultrasonic signal through the liquid (TOF_{liquid}) is equal to the delay between A_{r1} and A_{m1}. The electrical signals at the piezoelectric transducer due to these ultrasonic waves appear represented in Figure 2 (top). Thus, the velocity of the ultrasonic wave propagating through the liquid (V_{liquid}) can be easily obtained by Eq.1.

$$V_{liquid} = (2 \cdot L_{liquid})/TOF_{liquid} \quad (1)$$

In Eq. 1, V_{liquid} refers to the velocity of the ultrasonic wave propagating through the liquid, L_{liquid} refers to the liquid path length and TOF_{liquid} refers to the time of flight of the ultrasonic wave through the liquid. Using this operating principle, both the buffer rod and the liquid path lengths should be dimensioned to allow the reception of A_{m1} after the arrival of A_{r1} but before the reception of the second echo from the liquid-buffer interface A_{r2}, see Figure 2 (top) [34].

Sensor dimensioning and shortening

As mentioned in the previous subsection, buffer rod dimensions are an important point to be determined in this sensor design. Eq.2 relates the buffer rod and liquid path lengths to the ultrasonic velocity of propagation within the liquid:

$$L_{BR} = (L_{liquid} \cdot V_{BR}) / \beta \cdot V_{liquid} \quad (2)$$

In Eq. 2, L_{BR} refers to the buffer rod length, L_{liquid} refers to the liquid path length, L_{BR} refers to the velocity of propagation along the buffer rod, and V_{liquid} refers to the velocity of the ultrasonic wave when it is propagated through the liquid. Here β is a parameter (which ranges from 0 to 1), that defines the relative position of the measurement signal with respect to two consecutive reference signals. When the β parameter is set to 0.5, the measurement signal will be received right in the middle of two consecutive reference signals.

According to Eq.2, if the ultrasonic velocity of propagation alongside the buffer rod is much higher than the velocity within the liquid, the buffer rod length needs to be rather long. In practice, buffer rods that satisfy the required dimensions and weights for these cases are sometimes difficult to adapt to the majority of the existing food tanks and pipes.

Said that, the buffer rod length can be reduced if A_{m1} is received in between consecutive reference signals that are further out in time, e.g. A_{r2} and A_{r3}, as shown in Figure 2 (middle). The general case is shown in Figure 2 (bottom). For this case, the buffer length can be obtained from Eq. 3.

$$L_{BR} = (L_{liquid} \cdot V_{BR}) / (n-\beta) \cdot V_{liquid} \qquad (3)$$

In Eq. 3, the length of the buffer rod for n = 1 is the same as that obtained with Eq. 2 for the conventional buffer design procedure, in which the reception of the measurement signal is fixed between the first reference signal and its echo, giving the longest possible buffer rod length (L_{BR1}). Similarly, for n = 2, 3,. . . and successive natural numbers, the measurement signal arrives after n reference signals, and thus the buffer rod can be therefore shortened to L_{BR2}, L_{BR3} and successive L_{BRn} buffer rod lengths.

Physical design of the sensor

An exploded view of the ultrasonic sensor is depicted in Figure 3. The most important component of the ultrasonic sensor is the transducer (numbered 1 in Figure 3). For this reason, a great deal of attention has been paid to selecting a proper transducer for the application. A B1F ultrasonic transducer manufactured by General

Electric has been selected. This is a general purpose wideband transducer with an element size of 20 mm and a resonant frequency of 1 MHz. This frequency has been chosen in order to avoid attenuation effects on the ultrasonic waves travelling both through the liquid media and the buffer rod. Also, a 1 MHz frequency is widely used for process characterization in liquid media.

The transducer is placed in contact with the buffer rod (numbered 2 in Figure 3). An ultrasonic couplant is placed between them. The buffer rod has a cylindrical shape as does the transducer. Since the sensor will be used in the monitoring of malolactic fermentation processes in red wines, a High-Density Polyethylene (HDPE) plastic material was chosen for the buffer rod because this material is allowed to be in direct contact with food by the food industry, and it is lighter than other alternatives such as stainless steel.

According to Eq.3 the selection of the buffer rod length (L_{BR}) and liquid path length (L_{liquid}) values are related to the ratio of ultrasonic velocities of propagation in both media. The sensor has been designed to be used in liquid media, with an ultrasonic velocity of propagation of about 1500m/s. On the other hand, the ultrasonic velocity of propagation alongside the High-Density Polyethylene buffer rod is 2430 m/s. According to Eq.3, it is possible to obtain the relationship L_{BR}/L_{liquid} if the n and β values are known. In this case, a value of 2 for the parameter n has been chosen (the measurement signal appears between echoes 1 and 2). For β, the selected value was 0.5. With these values, the relationship L_{BR}/L_{liquid} is 1.08. In order to accomplish this value, the buffer rod and liquid path lengths in our sensor are 50 mm and 46.6 mm, respectively.

The noise level has also been considered when dimensioning the buffer rod. Spurious ultrasonic echoes generated at different parts of the sensor boundaries are considered noise and are strongly related to its geometry. Careful design of the sensor geometry can reduce noise level and improve the accuracy and reliability of the measurements. The relationship between transducer size and buffer rod diameters must be considered as well. The diameter of the buffer rod should be a least 1.5 times the diameter of the transducer in order to reduce the acoustic interference caused at the sensor boundaries [35]. On the other hand, the diameter of the buffer rod should be dimensioned so that it can be easily introduced inside the food tanks using a standard 50 DIN thread. Accordingly, if the diameter of the transducer is 20 mm and the inner diameter of a standard 50 DIN thread is 53 mm, the diameter of the buffer rod needs to be set to 40 mm.

It is well known that ultrasonic velocity is highly sensitive to temperature [2, 36-37]. That is why most ultrasonic experiments are always taken under constant or controlled temperature in a laboratory environment. However, in the case of in-situ industrial processes it is very difficult to carry out the process under a controlled temperature. Therefore it is necessary to measure the temperature and compensate accordingly. For this reason, a cavity with a diameter of 2 mm (numbered 6 in Figure 3) is drilled into the buffer rod. A temperature sensor, a 5 kΩ thermistor, is placed inside the cavity. This cavity is deep enough so that the temperature sensor is placed at a very short distance from the liquid.

The stainless steel ring (numbered 3 in Figure 3) holds the buffer rod in place. Also it fits into the tank outlet and stops the tank from leaking liquid with the help of a silicone ring. The cylindrical tube, (numbered 4 in Figure 3) which has two open windows, covers the buffer rod. Its dimensions are 74 mm of length and 47.8 mm of diameter. The end of this cylindrical tube (numbered 5 in Figure 3) acts as a wave reflector. The wave reflector and cylindrical body are designed with a cylindrical shape in order to facilitate a smooth integration into the tank. The cylindrical tube with the reflector remains inside the tank, with the liquid, and with the buffer rod in contact with the liquid.

The sensor has been fitted with a standard 50 DIN screw thread which is compatible with the stainless steel tank outlet making it possible to fit the sensor inside the industrial tank. It fits perfectly within the tank outlet and stops the tank from leaking liquid with the help of a silicone ring.

The prototype of the sensor is shown in Figure 4. In Figure 4a, the buffer rod, the temperature sensor and the transducer are shown in detail. The transducer is fixed to the buffer rod structure by a small aluminium plate screwed to the buffer rod. Figure 4b shows the ultrasonic sensor shielded with a stainless steel cap in order to protect the transducer from accidental hits.

Two examples of the sensor coupled to an industrial tank using a standard 50 DIN screw thread can be seen in Figure 5.

Experimental setup

The experimental setup is shown in Figure 6. The ultrasonic transducer is excited at its 1 MHz resonant frequency with a sine-wave tone burst of 10 cycles and 20 V_{pp} of amplitude, using an Agilent 33522 function/Arbitrary Waveform Generator. The received waves are averaged 128 times and acquired at a sampling rate of 500 MS/s using a Tektronix DPO 2024 Digital Phosphor Oscilloscope. The resultant signal, which has a Signal-to-Noise Ratio (SNR) of approximately 34 dB, is then stored in a computer for processing using the Phase-Shift method (based on a fast Fourier transform algorithm). This is used to obtain the elapsed time variations between consecutive signals (A_{r1} and A_{m1}), which in turn allowed us to calculate absolute velocities, on the basis of an initial reference value.

In order to measure the temperature, the sensor was also equipped with a 5.0 kΩ thermistor. The electrical signal from the thermistor was measured using a data-logger (Agilent 34970A/34972A Data Acquisition).

As several liquid tanks are monitored at the same time, the electrical output voltage that comes from the signal generator is applied to the different transducers. Also the received signals are multiplexed by means of a USB controlled custom relay board. Data is acquired by the oscilloscope and monitored in the computer. This design provides feedback control to the industrial process. The process can be observed in the computer and the operator can be alerted.

Data acquisition.

Instruments described in the previous section (Waveform Generator, Multiplexer, Oscilloscope and Temperature Data-Logger) are connected to a PC using USB buses. Data acquisition is performed by a custom application developed using the LabVIEW® environment from National Instruments. The program interface is shown in Figure 7. Figure 8 schematically represents the program structure that has been developed.

According to Figure 8, first, the program opens communication with all connected instruments, such as the Oscilloscope, Temperature Data-Logger, Multiplexer and Waveform Generator. Also, a data file path is created.

The second step consists of the instruments configuration. A default configuration is automatically established for each instrument, but the user can modify it as many times as desired before the data acquisition begins. Sampling rate, scope channels selected and number of samples taken in each acquisition are established in this step.

Data acquisition begins manually when the "acquire" button is pressed by the user. Then, instruments configuration changes are no longer possible. During data acquisition period, data are automatically captured at the sampling rate previously established. Two data types are acquired at each sampling time: temperature (provided by the data-logger) and the ultrasonic waveform (provided by the scope).

Acquired data are saved in spreadsheet compatible files, one for each capture. Data file format equals to a spreadsheet one, where each column corresponds to a scope channel. For each channel, it is saved: the channel name, data and time when the acquisition took place, temperature, interval rate between correlative points and the series of the scope data points displayed.

The data acquisition process runs indefinitely until it is aborted manually by the user. Then, the communication with instruments is closed and program execution is finished.

Error and uncertainty in the measurement of ultrasonic velocity.

Using the measurement set-up described in the previous section and with the ultrasonic sensor working in pulse-echo mode, the acquired signals look like those in Figure 9.

As can be seen, each acquired signal is composed of the excitation pulse which excites the ultrasonic sensor and five echoes that the same sensor subsequently receives (pulse-echo mode). A_{r1} and A_{m1}, separated by the time of flight in the liquid medium (TOF_{liq}), are the signals of greater interest, i.e., they are the echoes that define the time taken by an ultrasonic wave to travel twice the path length through the liquid medium. This parameter will be subsequently used to calculate the propagation speed of ultrasounds in the medium of interest (V_{liquid}). It should be noticed that the A_{r2} echo lies intentionally in between the previous two signals in order to reduce, for convenience, the length of the buffer rod (see section “sensor dimensioning and shortening”). Finally, the ECHO3 and ECHO4 echoes which are a consequence of new reflections and transmissions of A_{m1} will not be taken into account in the ultrasonic velocity calculation.

All acquired signals from Figure 9 are processed using a fast Fourier transform (FFT) algorithm to obtain the time of flight in the liquid (TOF_{liquid}) [38]. Then, the ultrasonic propagation velocity in the liquid is calculated by dividing the distance travelled through the liquid by the time of flight, as stated in Eq. 1.

Eq. 1 provides the absolute ultrasonic velocity of the liquid medium. However, when monitoring a process it is usually more interesting to measure the speed variation over the time the process lasts. This variation is calculated with respect to a reference velocity (usually the ultrasonic velocity the liquid has at the beginning of the process). Therefore, Eq. 1 can be rewritten as Eq. 4.

$$V_{liquid} = \frac{2 \cdot L_{liquid}}{TOF_{liquid}} = \frac{D}{TOF_{liqRef} + \Delta TOF_{liquid}} = \frac{D \cdot V_{liqRef}}{D + V_{liqRef} \cdot \Delta TOF_{liquid}} \qquad (4)$$

where D equals $2 \cdot L_{liquid}$, V_{liqRef} is the reference velocity, TOF_{liqRef} is the time of flight of an ultrasonic wave at the beginning of the process and, ΔTOF_{liq} is the variation over time of the time of flight. As a consequence, Eq. 4 can be used to obtain the variation over time of the ultrasonic velocity with respect to the ultrasonic velocity of reference (Eq. 5).

$$\Delta V_{liquid} = V_{liquid} - V_{liqRef} = \frac{(V_{liqRef})^2 \cdot \Delta TOF_{liquid}}{D + V_{liqRef} \cdot \Delta TOF_{liquid}} \qquad (5)$$

Therefore, the absolute error in the variation over time of the ultrasonic velocity in the liquid medium $\varepsilon(\Delta V_{liquid})$ can be calculated from the absolute errors of each of the variables involved in the measurement. Then, according to propagation of errors, Eq. 6 states that:

$$\varepsilon \cdot \left(\Delta V_{liquid}\right) \approx \left|\frac{\partial \cdot \Delta V_{liquid}}{\partial \cdot V_{liqRef}}\right| \cdot \varepsilon\left(V_{liqRef}\right) + \left|\frac{\partial \cdot \Delta V_{liquid}}{\partial \cdot \Delta TOF_{liquid}}\right| \cdot \varepsilon\left(\Delta TOF_{liquid}\right) + \left|\frac{\partial \cdot \Delta V_{liquid}}{\partial \cdot D}\right| \cdot \varepsilon(D) \qquad (6)$$

Combining Eq. 5 and Eq. 6, the uncertainty in the ultrasonic velocity can be obtained by the following expression:

$$\varepsilon \cdot \left(\Delta V_{liquid}\right) \approx \frac{V_{liqRef} \cdot \Delta TOF_{liquid} \cdot \left(2 \cdot D + V_{liqRef} \cdot \Delta TOF_{liquid}\right)}{\left(D + V_{liqRef} \cdot \Delta TOF_{liquid}\right)^2} \cdot \varepsilon\left(V_{liqRef}\right) + $$
$$+ \frac{\left(V_{liqRef}\right)^2 \cdot D}{\left(D + V_{liqRef} \cdot \Delta TOF_{liquid}\right)^2} \cdot \varepsilon\left(\Delta TOF_{liquid}\right) + \frac{\left(V_{liqRef}\right)^2 \cdot \Delta TOF_{liquid}}{\left(D + V_{liqRef} \cdot \Delta TOF_{liquid}\right)^2} \cdot \varepsilon(D) \qquad (7)$$

In our experiments, $\varepsilon(V_{liqref})$, $\varepsilon(\Delta TOF_{liquid})$ and $\varepsilon(D)$ were measured with a precision of ±0,01 m/s, ±2 ns and ±0,01 mm, respectively. Then, from Eq. 7, the uncertainty in the variation of the ultrasonic velocity (worst case) in the liquid medium would be approximately of ± 0.1 m/s.

Results and discussion.

In order to experimentally examine the behaviour of the sensor to changes of concentration of solutions, the propagation velocity of ultrasound in aqueous solutions of malic acid and lactic acid was measured. The reason why these solutions where chosen is that the malic and lactic acids are substances involved in a malolactic fermentation, a process which is intended to be monitored with the described sensor[1,39-42] . This section shows the obtained results.

Ultrasonic propagation velocity in ternary mixtures water – lactic acid – malic acid was measured. For this purpose, thermostated aqueous samples of lactic acid in different concentrations (0, 2, 4 and 6 g/l) were prepared, which corresponds to concentration values for most wines, ranging from the least acidic (1 g/l) to the most (8 g/l). Aliquots of malic acid were added to the related samples of lactic acid, and ultrasonic propagation velocity was measured. Results are represented graphically in Figure 10.

Empirical equations from the data obtained in Figure 8 have been adjusted, using an order 2 polynomial model. The adjusted equations are shown in Table 1:

In Table 1, a good fit is observed in the empirical equations obtained from the experimental data, with a coefficient of determination R^2 higher than 0.99.

From the obtained results, it can be seen that the sensor shows good quadratic behaviour which makes this sensor suitable for measuring changes in concentration of liquid samples.

In addition, this sensor has been experimentally tested to monitor an industrial process, more specifically, the malolactic fermentation process of red wine in some pilot plants and wineries[12,37,43,44]. In Figure 11, the ultrasonic velocity of propagation (ΔVel) and the temperature variation (ΔT) were measured during the malolactic fermentation process of a “tempranillo” wine from Palencia (Spain). It is observed that the ultrasonic velocity and temperature profiles are similar. This is because the temperature has changed significantly during the malolactic fermentation process, and the ultrasonic velocity variation is mainly due to the temperature variation.

From results collected in Figure 11, it is possible to correlate ultrasonic velocity and temperature (Figure 12). In Figure 12, a good correlation between ultrasonic velocity and temperature is observed. So, a linear empirical equation is derived (Eq. 8).

$$v = 0.4176 + 0.8269 \cdot t \qquad (8)$$

In Eq. 8, v refers to ultrasonic velocity (in m/s) and t corresponds to temperature (in ºC). The coefficient of determination R^2 is 0.9885. The slope of the linear equation indicates that the ultrasonic velocity increases at a rate of 0.83 m/s for each degree Celsius of temperature. This coefficient will be used later to compensate the temperature effect in the ultrasonic velocity of propagation. Results are also shown in Figure 11 (ΔVel-compT). It is observed that the ultrasonic velocity variation calculated after applying the temperature compensation coefficient is significantly lower than the original measured values (ΔVel). This is because the temperature is the main factor that affects the ultrasonic velocity of propagation. This highlights that changes in temperature can seriously mask the variations due to chemical changes and makes it more difficult to monitor the process.

Also, malic and lactic acid concentrations are provided for this wine sample. In a previous paper[12], the authors described an empirical equation that correlates the ultrasonic velocity of propagation with the malic and lactic acid concentrations. For this purpose, ultrasonic velocity in quaternary mixtures water – ethanol - lactic acid – malic acid was measured. Thermostated samples of malic acid in different concentration (0, 2, 4, 6, 8 and 10 g/l), solved in ethanol 11.5% v/v, were prepared. Aliquots of lactic acid were added to the samples of malic acid, and the ultrasonic propagation velocity was measured. Results are represented graphically in a 3D plot (Figure 13), showing a good correlation between the ultrasonic velocity and the malic and lactic acid concentrations. From data represented in Figure 13, a linear empirical equation has also been derived (Eq. 8).

$$\Delta v = -0.2196 \cdot \Delta x_{malic\ acid} + 0.2359 \cdot \Delta x_{lactic\ acid} \qquad (8)$$

In Eq. 8, Δv refers to ultrasonic velocity variation (in m/s), $\Delta x_{\text{malic acid}}$ corresponds to the variation of malic acid concentration (in g/l) and $\Delta x_{\text{lactic acid}}$ corresponds to the variation of lactic acid concentration (in g/l). The coefficient of determination R^2 is 0.996.
During malolactic fermentation process, malic acid concentration decreases and lactic acid increases. So, according to Eq. 8, ultrasonic velocity should increase as malolactic fermentation process takes place.

Accordingly, and applying Eq. 8, it is possible to estimate the theoretical ultrasonic velocity of propagation from the malic and lactic acid concentrations. Results obtained are also shown in Figure 11 (ΔVel_Teo).

Experimentally obtained results after the removal of the temperature contribution (ΔVel-compT) show that the ultrasonic velocity variation initially increases, followed by a decrease and ending up increasing again until a new stable value is reached, which is higher than the initial value. These results are close to the expected theoretical ones (ΔVel_Teo), particularly after a time of 100h. Differences of both curves before this time are not due to changes in lactic and malic acid concentrations, but to different factors related with the lactic acid bacteria growth. In this phase, malic acid is not transformed to lactic acid, but other processes take place that result in changes in ultrasonic velocity[12].

Results have shown the suitability of using this sensor for online monitoring of malolactic fermentation processes. Indeed, a good correlation was found between ultrasonic velocity and both malic and lactic acid concentrations.

Conclusion

This paper describes a novel industrial ultrasonic sensor designed for online monitoring of food processes in a liquid medium within an industrial environment, including the operating principle and construction details. The ultrasonic sensor is based on the measurement of the evolution of the ultrasonic velocity of propagation during the process. One of the main advantages of the sensor is that its structure allows it to be directly installed in standard stainless steel tanks of an industrial plant. The sensor was tested by measuring the ultrasonic velocity of propagation in aqueous samples of malic and lactic acid, and also in an industrial process of a malolactic fermentation of red wine. The obtained results show the feasibility of using this sensor in all those processes in which physical-chemical changes occur in liquids stored in industrial tanks.

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Tables.

Table 1. Adjusted empirical functions and coefficients of determination (R^2), for ultrasonic propagation velocity (y-axis) as a function of the malic-acid concentration (x-axis), for aqueous samples of lactic acid. Units: x (g/l), y (m/s).

	Empirical function	**Coefficient of determination (R^2)**
Water	$y = -0.0005x^2 + 0.2944x – 0.0282$	0.9978
Water-lactic acid 2 g/l	$y = -0.0185x^2 + 0.4517x + 0.5994$	0.9983
Water-lactic acid 4 g/l	$y = -0.0254x^2 + 0.5346x + 1.1729$	0.9998
Water-lactic acid 6 g/l	$y = -0.0262x^2 + 0.4792x + 1.7988$	0.9917

Figures.

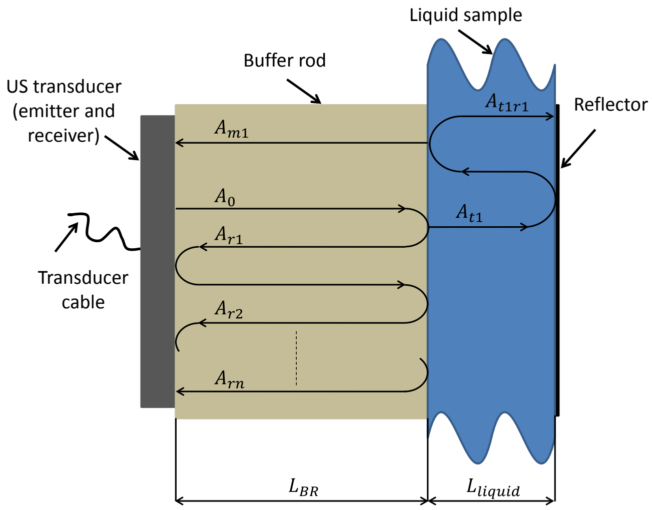

Figure 1. Description of the operating principle of the ultrasonic sensor. L_{BR} refers to the buffer rod length, L_{liquid} refers to the liquid path length, A_0 refers to the incident ultrasonic wave that propagates across the buffer rod, A_{r1} refers to the part of the incident wave that is echoed back onto the ultrasonic transducer, A_{t1} refers to the part that is transmitted through the liquid towards the wave reflector, A_{t1r1} refers to the transmitted ultrasonic signal that is reflected again onto the liquid and A_{m1} refers to the measurement signal that is transmitted to the ultrasonic transducer (where it is detected).

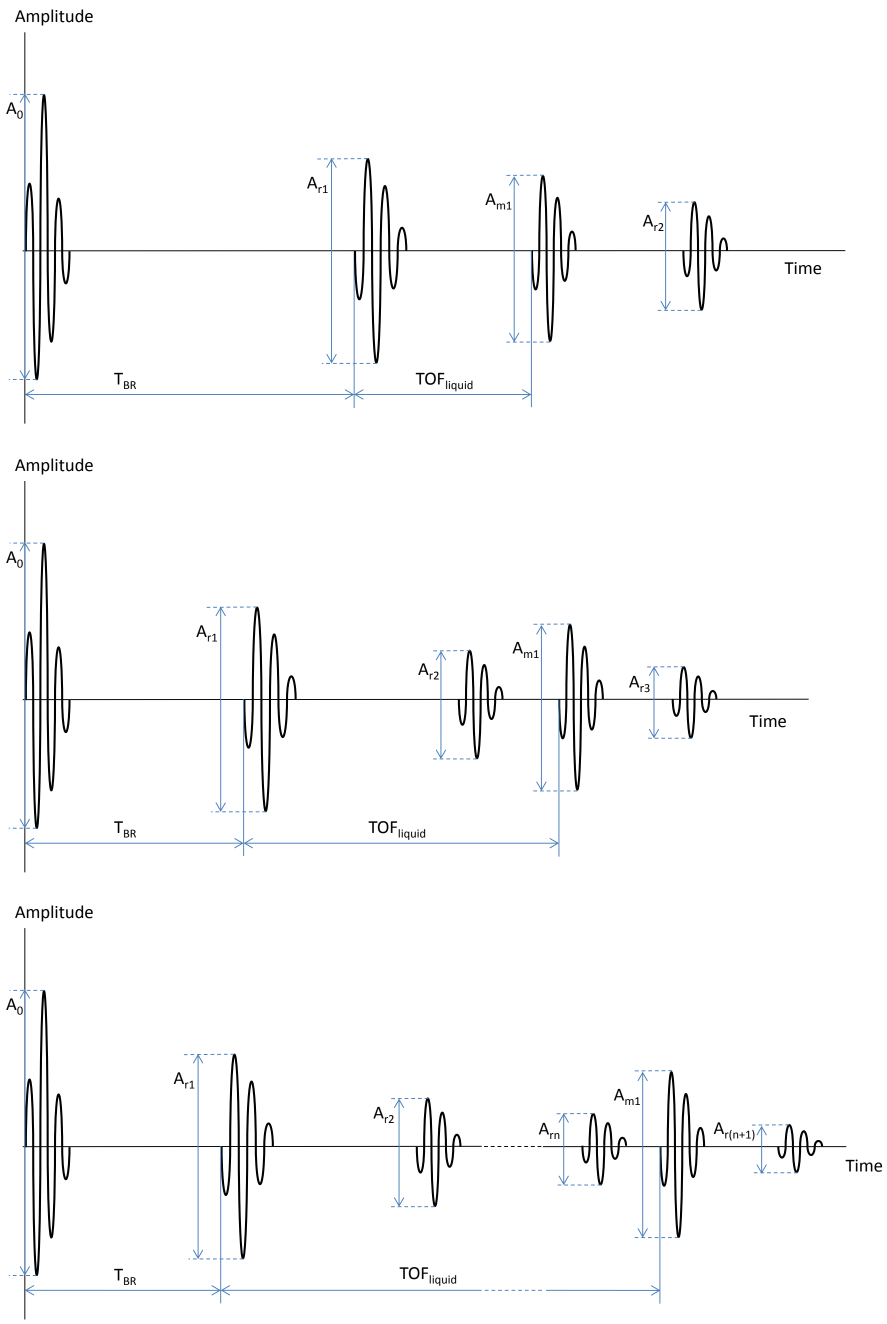

Figure 2. Representation of the electrical signals at the piezoelectric transducer for the conventional case of reception of the measurement signal A_m between the reference signal A_{r1} and the second echo A_{r2} (top), for the reception of the measurement signal between the echo of the reference signal A_{r2} and its echo A_{r3} (middle) and for the general case of reception of the measurement signal between two consecutives echoes of the reference signal, A_{rn} and $A_{(r(n+1))}$ respectively (bottom). T_{BR} refers to the time of flight of the ultrasonic signal through the buffer rod, and TOF_{liquid} refers to the time of flight of the ultrasonic signal through the liquid.

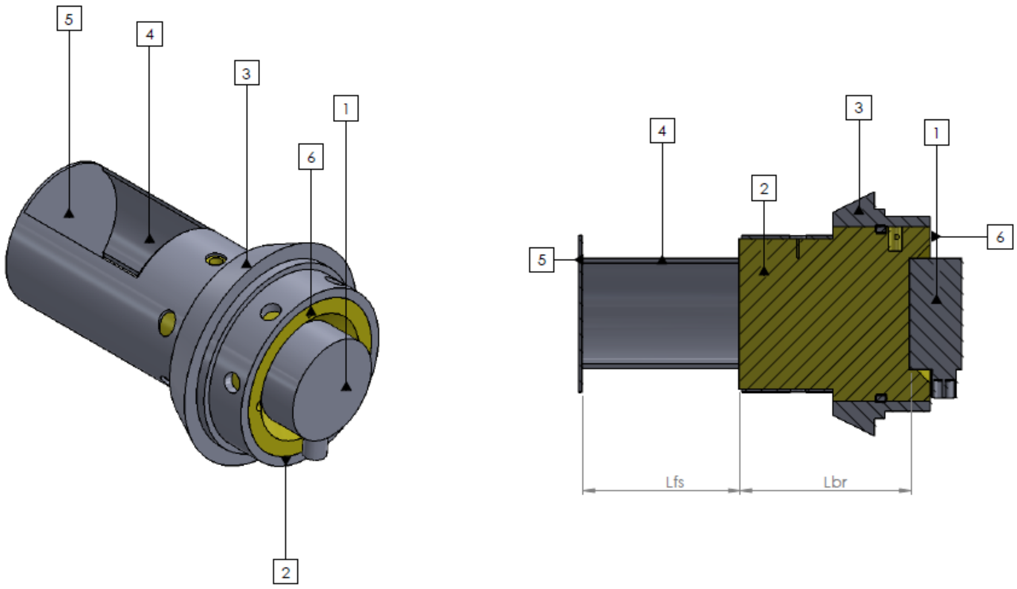

Figure 3. Exploded view of the sensor. 1- Transducer. 2- Buffer Rod. 3- Stainless steel ring. 4- Cylindrical body. 5 - Wave reflector. 6 - Temperature transducer. L_{br} refers to the buffer rod length, and L_{fs} refers to the liquid sample length.

(**a**) (**b**)

Figure 4. Sensor prototype.

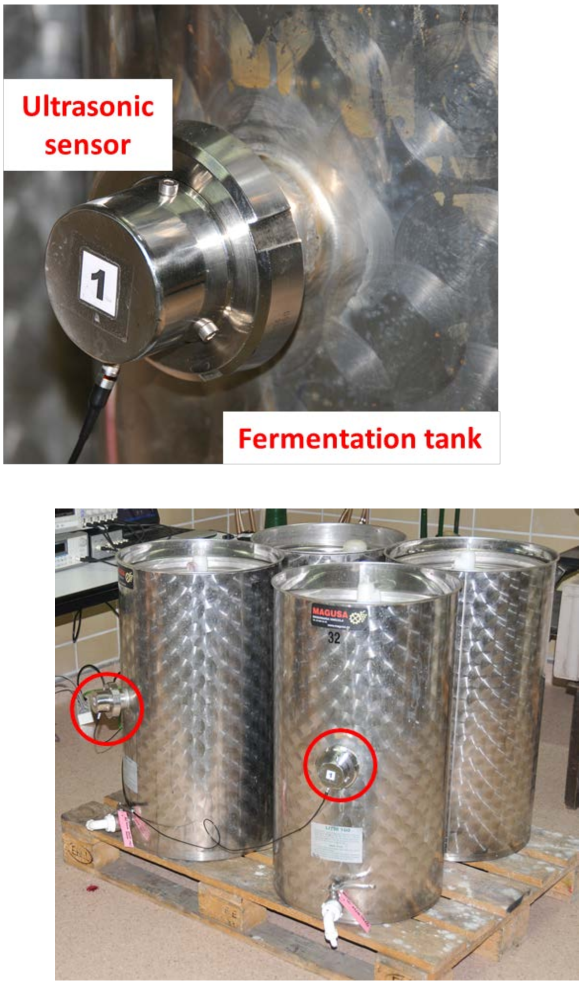

Figure 5. Images of the sensor coupled to an industrial tank using a standard 50 DIN screw thread.

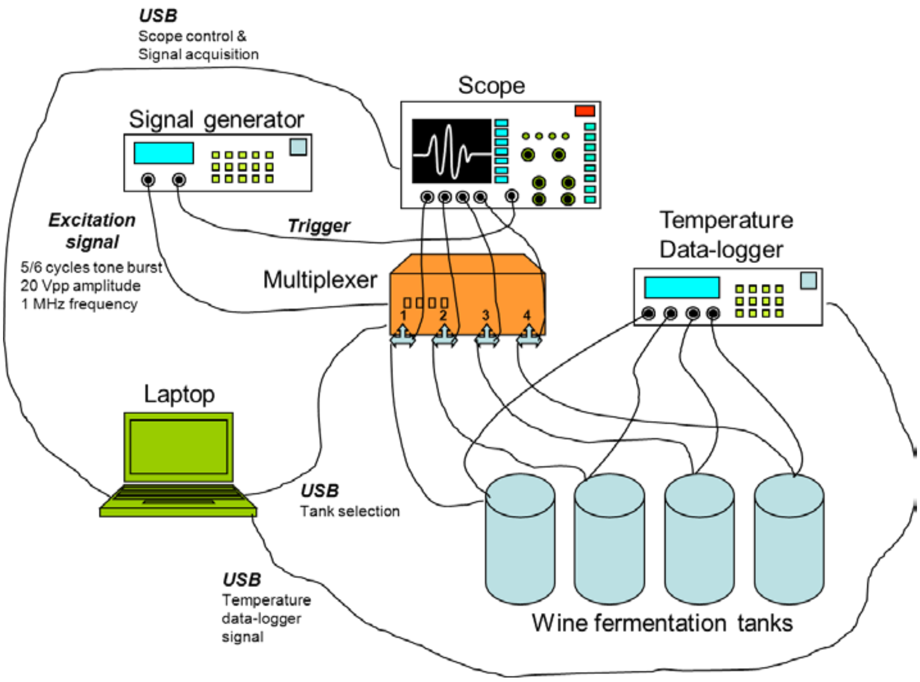

Figure 6. Experimental set-up.

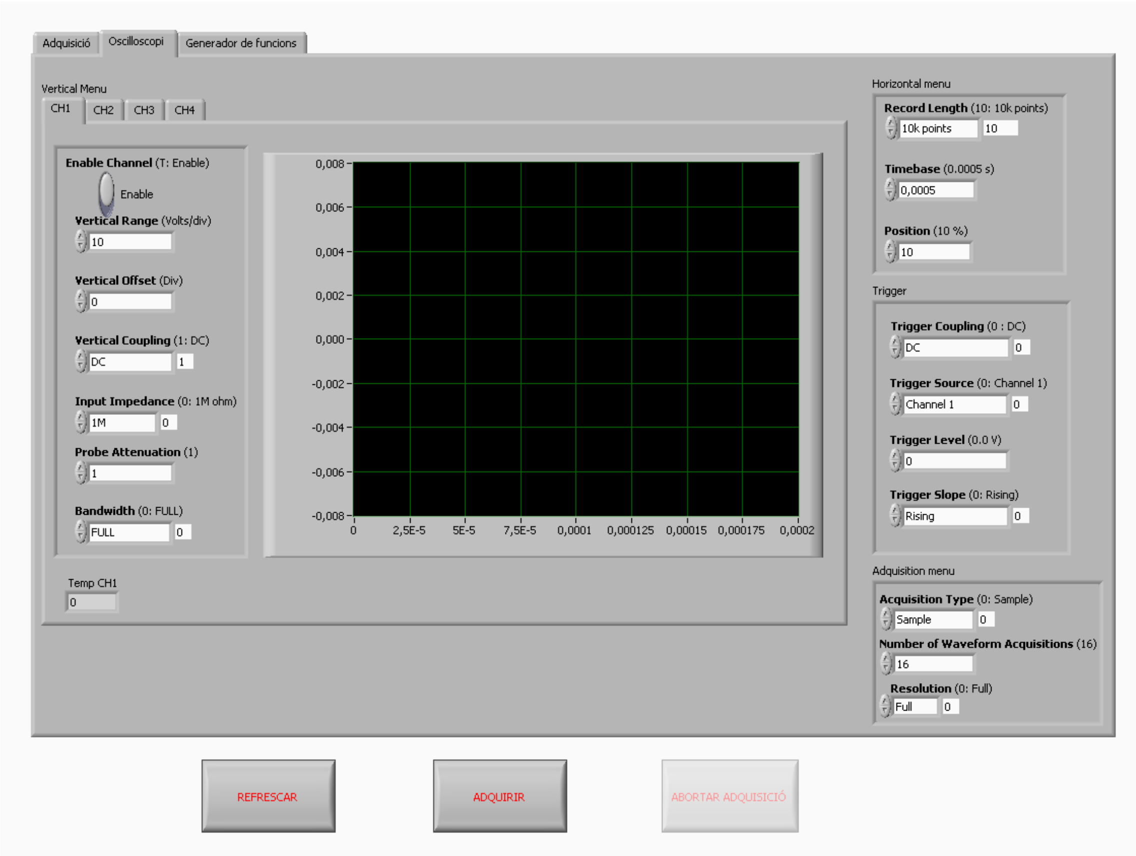

Figure 7. Capture of the program interface developed for acquiring signals from instruments.

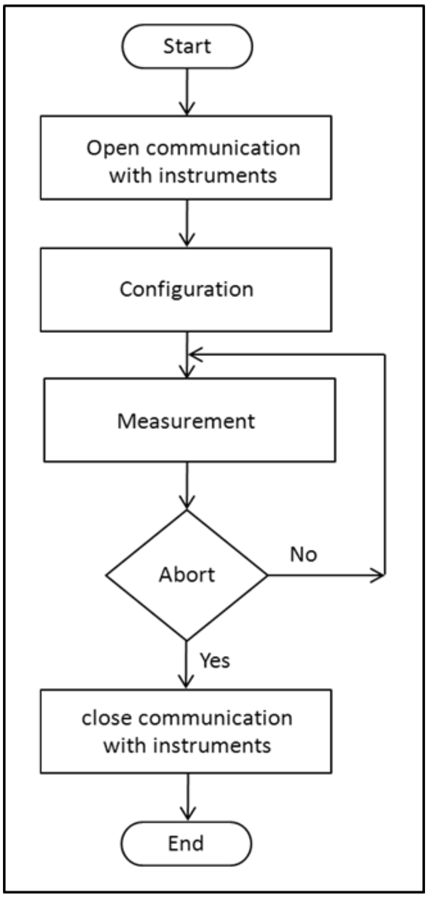

Figure 8. Program structure.

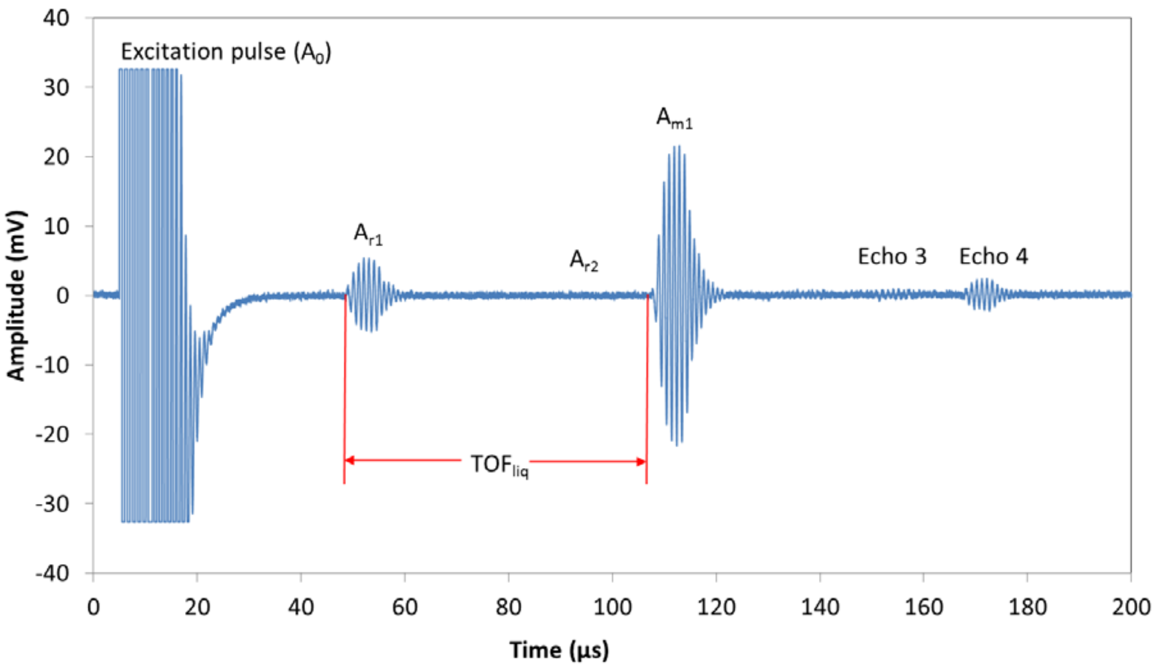

Figure 9. Waveform recorded by a digital oscilloscope and stored each time the ultrasonic system performs a measurement on the selected channel. A_0 refers to the incident ultrasonic excitation pulse that propagates across the buffer rod, A_{r1} refers to the part of the incident wave that is echoed back onto the ultrasonic transducer, A_{r2} refers to the second echo from the liquid-buffer interface, Echo 3 and Echo 4 refers to other echoes of the ultrasonic wave, and TOF_{liquid} refers to the time of flight of the ultrasonic signal through the liquid.

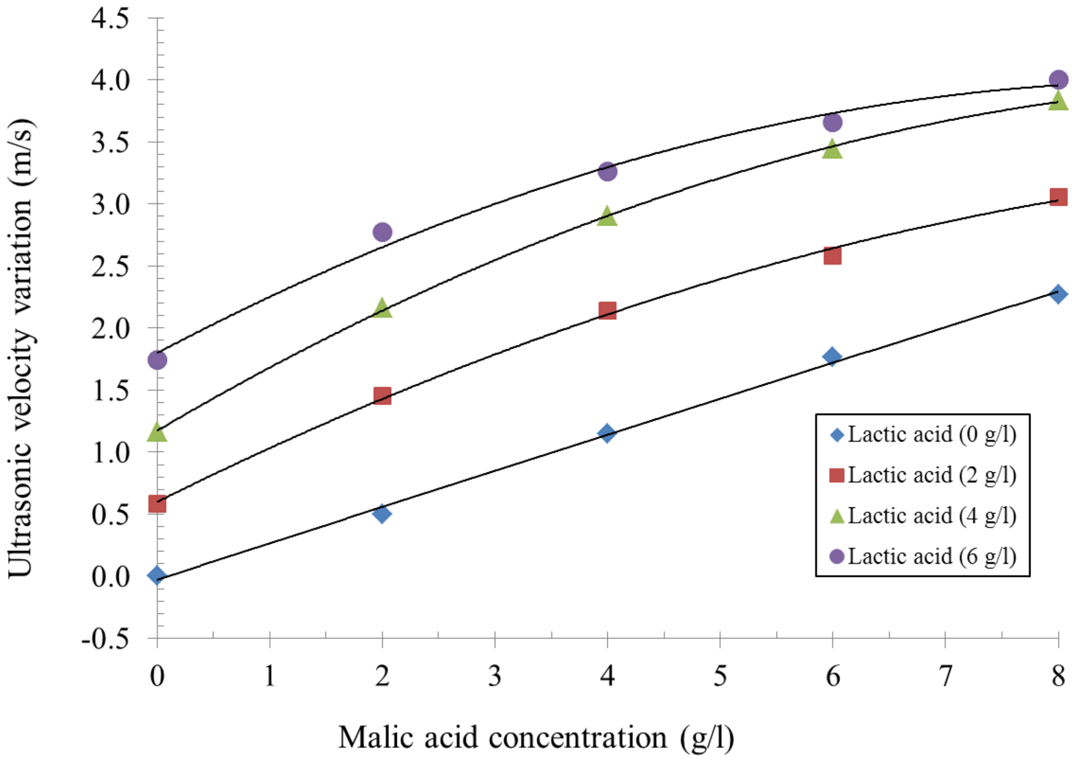

Figure 10. Variations of ultrasonic propagation velocity in ternary mixtures of water-lactic acid-malic acid thermostated at 22.20 ± 0.05 °C.

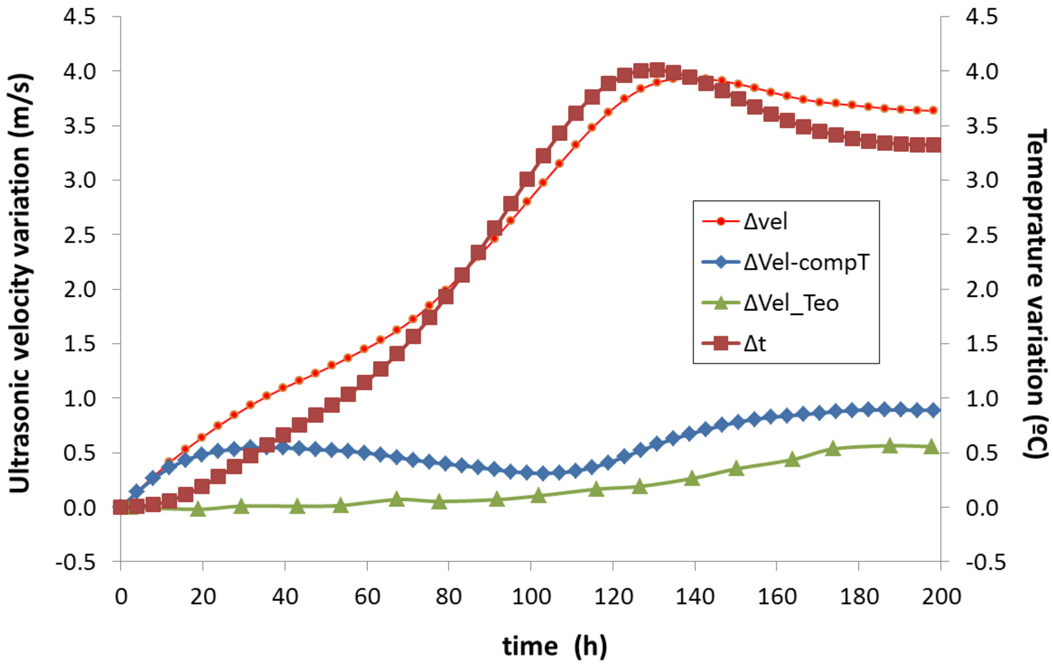

Figure 11. Ultrasonic velocity and temperature variation during a malolactic fermentation process. ΔVel refers to the ultrasonic velocity variation during the process. ΔT refers to the temperature variation during the process. ΔVel-compT refers to the ultrasonic velocity variation after applying the algorithm to compensate for the temperature effect. ΔVel_Teo refers to the theoretical ultrasonic velocity variation, estimated from the measured concentrations of malic and lactic acids.

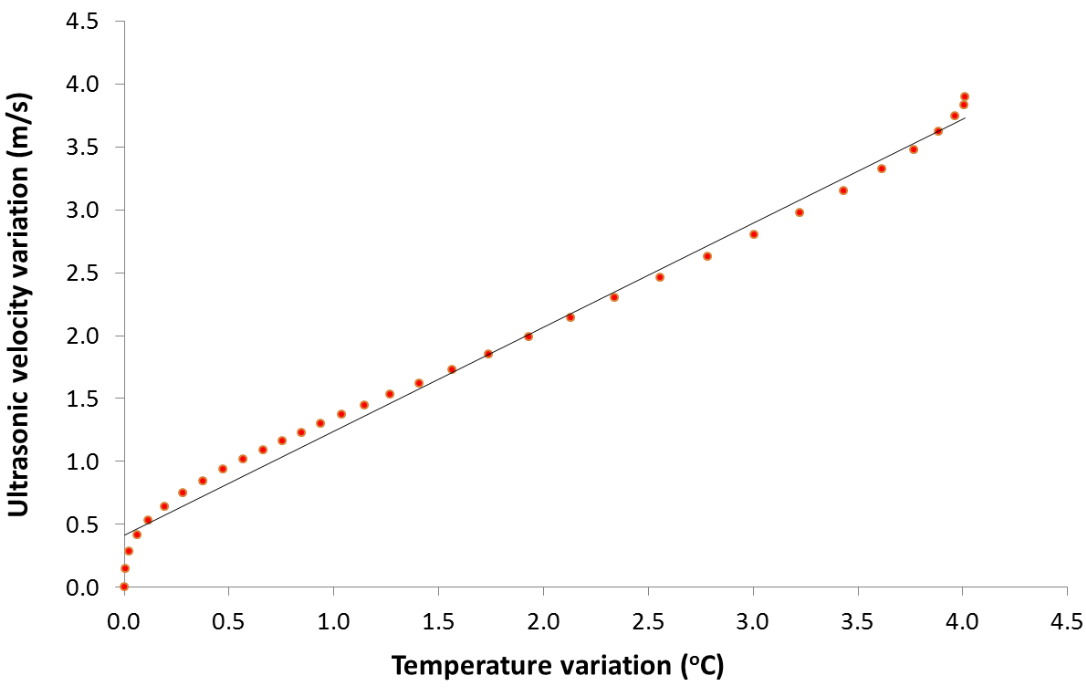

Figure 12. Obtained correlation between ultrasonic velocity of propagation and temperature (Palencia wine sample).

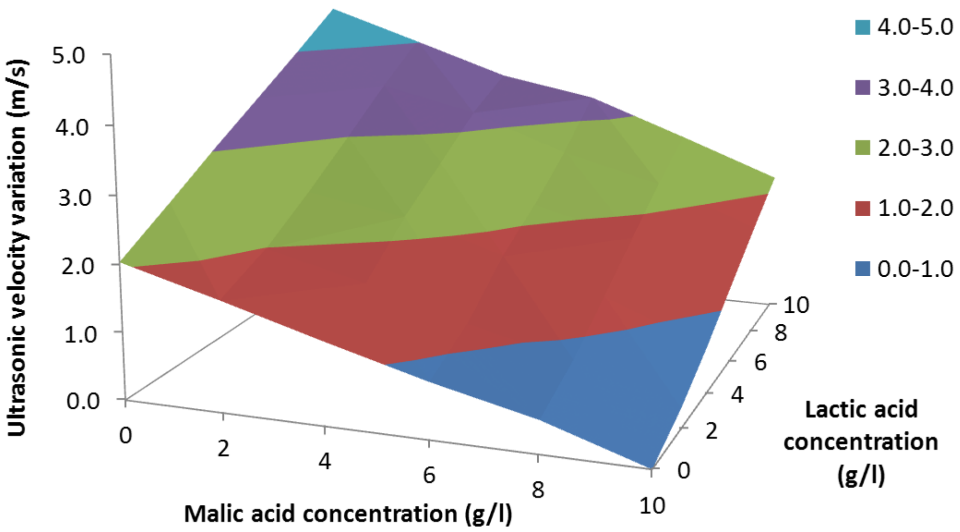

Figure 13. 3D graph representation of the ultrasonic velocity in quaternary mixtures of water-ethanol 11.5% v/v-lactic acid-malic acid, thermostated at 22.20 ± 0.05 ºC.