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Evaluation of ultrasonic techniques for on line coagulation monitoring in cheesemaking

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ABSTRACT

Ultrasound detection techniques are interesting alternatives to monitor several industrial processes. It is known that they are nondestructive, noninvasive, robust and generally low cost when compared with other monitoring techniques. The evolution of the ultrasonic speed and the ultrasonic attenuation for compressional waves has been proposed by different authors as a tool to monitor the coagulation of milk. However, most of the studies published were performed in laboratory conditions, which not necessarily simulate the industrial process reality. In this work, the suitability of ultrasonic speed and ultrasonic attenuation for on-line milk coagulation monitoring was evaluated. Different experiments were conducted in order to study the potential of these techniques as well as possible limitations to their industrial use. The results obtained showed that both techniques could be applied in high controlled laboratory conditions, and could be used in experiments performed in parallel with the industrial process. However, limitations to their on-line application were also found. The main limitation for ultrasonic speed application was its strong dependence with temperature, whereas in the case of attenuation a deposition of an interfering element on the transducer's surface affected the results.

1. Introduction

Milk coagulation is the process in which milk is transformed into a viscoelastic semi-solid coagulum by specific enzymes called chymosins. These enzymes act upon milk's κ-caseins, fragmenting part of these molecules. This phenomenon causes micelle disruption, increasing their hydrophobicity and promoting their aggregation (Koc and Ozer, 2008). Micelle aggregation is evidenced by an increase in the medium's viscosity. The characteristics of this process are important in cheesemaking because they determine the coagulum's cutting point (end of the coagulation stage) (Fox et al., 2000).

Identifying coagulum's optimal cutting point can help to improve cheese quality significantly. If the curd is cut when the coagulum is too weak, the gel structure will not be able to retain fat and other components which are critical for cheese's quality and yield. If coagulum is cut when it is too firm, syneresis is retarded, resulting in a high humidity cheese needing a prolonged ripening stage (Benedito et al., 2002). The cutting time is generally determined based on previous batches information, making it difficult to maximize the yield for every particular batch, unless both the raw materials and the process are perfectly standardized and controlled. Therefore, a quick and reliable determination of cutting point is needed in order to standardize cheese yield as well as its physical and sensory characteristics (Koc and Ozer, 2008).

Several practical and instrumental methods are available for determination of the desired coagulum cutting time based on a wide range of mechanical, vibrational, thermal and optical techniques (Klandar et al., 2007). However, most of these methods are destructive and not applicable on-line, making it difficult to attend manufacturing standards in cheese factories.

Different authors attributed potential for coagulation monitoring to ultrasonic sensing devices because they provide nondestructive and quick measurements (O'Callaghan et al., 1999; Bamberger et al., 1999; Klandar et al., 2007). The parameters most frequently used in ultrasonic measurements are the ultrasonic velocity and attenuation (Wang et al., 2007). Several authors have successfully applied low intensity ultrasonic measurements for studying the structural changes involved in milk coagulation and for coagulation monitoring. Experiments have been carried out in casein solutions (Wang et al., 2007), reconstituted milk (Ay and

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Gunasekaran, 1994; Bakkali et al., 2001; Nassar et al., 2001, 2004) and whole milk (Montero Espinosa et al., 2002; Koc and Ozer, 2008).

Ay and Gunasekaran (1994) monitored attenuation and found it decreased during the coagulation process. They distinguished two stages, a first one with a higher decreasing rate and a second one with a lower decreasing rate. They defined the coagulation time as the time between rennet addition and the transition between these two stages. Gunasekaran and Ay (1995) proposed a methodology for cutting point determination. They measured attenuation and velocity during coagulation. They discarded velocity measurement because it showed significant fluctuations. They determined coagulation time using the same criteria as Ay and Gunasekaran (1994). The determination of the cutting point was carried out empirically, comparing the coagulation time determined by attenuation measurements with the cutting point determined by a manual method. They concluded that for their working conditions the cutting point was reached 20 min after coagulation time.

Koc and Ozer (2008) also proposed the calculus of cutting point as a function of coagulation time. They monitored attenuation at laboratory scale working in a pulse-echo mode. They defined coagulation time as Ay and Gunasekaran (1994) and concluded cutting point was reached in four times the coagulation time. They reported experiences made in a milk processing plant. These consisted in taking a sample of the coagulation vat after rennet addition and pouring it into the ultrasonic measurement cell.

Bakkali et al. (2001) concluded attenuation measurements were not an appropriate technique for coagulation monitoring as it was difficult to detect a coagulation point and the method was not sensitive to changes in temperature or rennet concentration. However, they found significant variations in ultrasonic velocity during coagulation. They found two stages, the first one with a higher variation rate than the second one. Similarly to Ay and Gunasekaran (1994), they defined coagulation time as the time when the transition between the two stages took place. They found significant differences when modifying coagulation conditions (rennet concentration and temperature), concluding ultrasonic velocity measurements were appropriate for coagulation monitoring.

Benedito et al. (2002) observed a decrease in attenuation during coagulation process. These results agreed with those reported by Gunasekaran and Ay (1995) but differed with those obtained by Benguigui et al. (1994) and Dwyer et al. (2005) who found an increase in attenuation during coagulation.

Nassar et al. (2001) used time of flight measurements to monitor coagulation process. They found different behaviors when working at different ambient temperatures. In all assays they observed a lag stage after rennet addition. For higher temperatures they found a decrease in time of flight during coagulation. While for lower temperatures, they found an increase followed by a decrease in the time of flight after the lag stage. They attributed these changes to different stages during the coagulation process and they related the changes in time of flight with changes in the medium's temperature.

Nassar et al. (2004) studied sensitivity to coagulation conditions (temperature, pH, milk powder concentration) using the same technique as Nassar et al. (2001). Passos et al., 1999 monitored the coagulation process through changes in the medium's conductivity.

Eventhough numerous authors worked in milk coagulation monitoring using ultrasound, industrial application of these techniques has not been reported. Additionally, different tendencies are reported in both the evolution of time of flight and ultrasonic attenuation during the coagulation process. In this work, we studied the suitability of velocity and attenuation of compressional waves to monitor milk coagulation in line during cheese production process. Experiments were conducted using two different systems: a laboratory cell and a submergible cell. The results were compared with classical rheological measurements.

2. Materials and methods

Coagulation was monitored using ultrasound compressional waves and classical rheology. Ultrasonic measurements were performed in two different set-ups: laboratory cell and submergible cell. The submergible cell was used at bench scale, but it could be directly used in an industrial vat. Rheological measurements were used as a reference.

2.1. Coagulation

Pasteurized whole milk was purchased from a local dairy industry. Calcium chloride (analytical quality $CaCl_2$) and rennet (*Escherichia coli* fermentation chymosin, 600 IMCU/ml) were added in concentrations of 0.02 g/L and 0.3 mL/L respectively. Milk was heated to 37 °C and maintained at this temperature within 0.1 °C using a water bath before rennet addition.

2.2. Rheological measurements

Rheological measurements were carried out in an Anton Paar Physica 301 rheometer. Oscillatory tests (1 Hz, 0.2% strain) were made using a 20 mL concentric cylinder geometry. Milk was thermostatized inside the cell before rennet addition. Rennet was added and storage (G′) and loss (G″) moduli were recorded during coagulation every 5 min.

2.3. Ultrasonic measurements

Ultrasonic velocity and attenuation were measured using two different set −ups: laboratory cell and submergible cell. Changes in ultrasonic velocity were quantified as variations in the time of flight of the ultrasonic wave travelling over a fixed distance. Time of flight (tof) refers to the time elapsed between the pulse emission and the reception of the first reflected echo.

Attenuation was measured as the variation in the energy of the signal. Both measurements were calculated relative to the initial condition.

2.3.1. Laboratory cell assays

An aluminium prismatic cell (500 cm^3) placed inside a water bath was used (Fig. 1A). Bottom and side walls were in contact with the water bath, while the top of the cell was open to ambient air. Milk was thermostatized inside the cell before the addition of rennet. A 3.5 MHz transducer was placed through one of the side

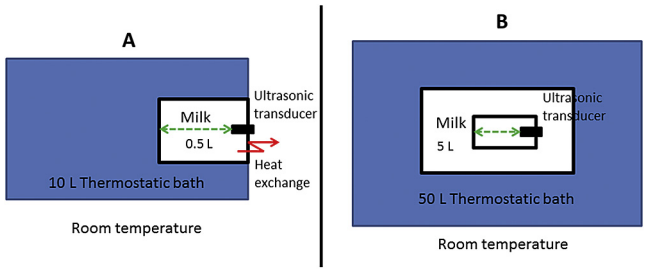

Fig. 1. Schematic view of the experimental set-ups. A) Laboratory cell B) Submergible cell. Dotted green lines indicate the propagation path for ultrasonic tests. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)