ULTRASONIC PROPAGATION AND THERMAL CHANGES DURING MILK GELATION PROCESSES

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ABSTRACT

An ultrasonic measurement technique for gelation monitoring of dairy products is presented in this work. An ultrasonic pulse is emitted and received after travelling through the milk during two different processes: rennet coagulation and yoghurt formation. Changes of the ultrasonic propagation parameters containing information about the milk gelation process are reported. The temperature changes during gelation were also measured and its influence over the ultrasonic propagation was evaluated. This non-invasive and real-time measurement technique appears as an interesting method for quality control in the dairy industry.

1. INTRODUCTION

The dairy industry demands the development of simple and non destructive measuring instruments to monitor the line of different processes. The coagulation of milk is the primary stage of most of dairy products; therefore monitoring coagulation appears as a very interesting matter, specially in the cheese making where the detection of the cutting curd time is critical to improve the quality of the cheese.

The elastic wave propagation measurement constitutes a well-known non-invasive physical technique for material (solid and fluid) characterisation. The velocity and the attenuation of a propagating elastic wave depend on the physico-chemical properties of the medium. Changes in these elastic parameters can be related to changes taking place in the medium. These characteristics make ultrasonic propagation measurement a very promising method for the automatic control of the milk coagulation process.

Ultrasonic methods were used to evaluate gelation processes of dairy products some decades ago [1]. However, an increasing attention has been given recently to this topic, and attenuation measurement [2], phase velocity measurement [3] or both attenuation and velocity [4] were used to study the rennet coagulation of milk.

In this paper, a non-invasive ultrasonic technique for the evaluation of two different gelation processes: rennet coagulation reaction and yoghurt formation, is presented. From the point of view of the ultrasonic propagation, differences and similarities between both gelation processes are shown. During the coagulation process, the temperature was measured, as it is as a relevant parameter for both the sol-gel transition and the elastic wave propagation characteristics.

2. EXPERIMENTAL TECHNIQUE

A small rectangular chamber (80mmx42mmx25mm) with a capacity of 84ml was designed and constructed for the ultrasonic and temperature characterisation of the coagulation processes (Figure 1). This chamber was mainly made of an epoxy material. The walls of the chamber consist of two Plexiglas windows, 2mm width, for visual and ultrasonic evaluation. Four temperature probes (numbered from 1 to 4) were placed at different heights to have a temperature mapping along the chamber during the coagulation process. This chamber was placed inside a temperature controlled water bath at 35ºC.

The ultrasonic propagation was measured with two pairs of transducers at two different heights (T1 and T2) using a through-transmission technique. Each transducer was made of PZT 5A 20mm diameter piezoceramic bonded to a Plexiglas plate. Araldite D was used as backing. The emitting transducers were excited with a 10 cycle burst having a central frequency of 2MHz. Waves received by the transducers at the opposite site of the chamber were stored every 3 minutes. These waves run a distance of 25mm inside the milk before being detected. The store signals were digitalized and processed using a FFT algorithm. The time of flight variations were obtained from the phase of the signal spectrum.

The ultrasonic propagation changes detected were caused by both temperature fluctuations [5] and milk coagulation processes [6]. In addition, the temperature fluctuations are due to thermostatization instabilities in the water bath and physico-chemical reactions taking place during the gelation processes.

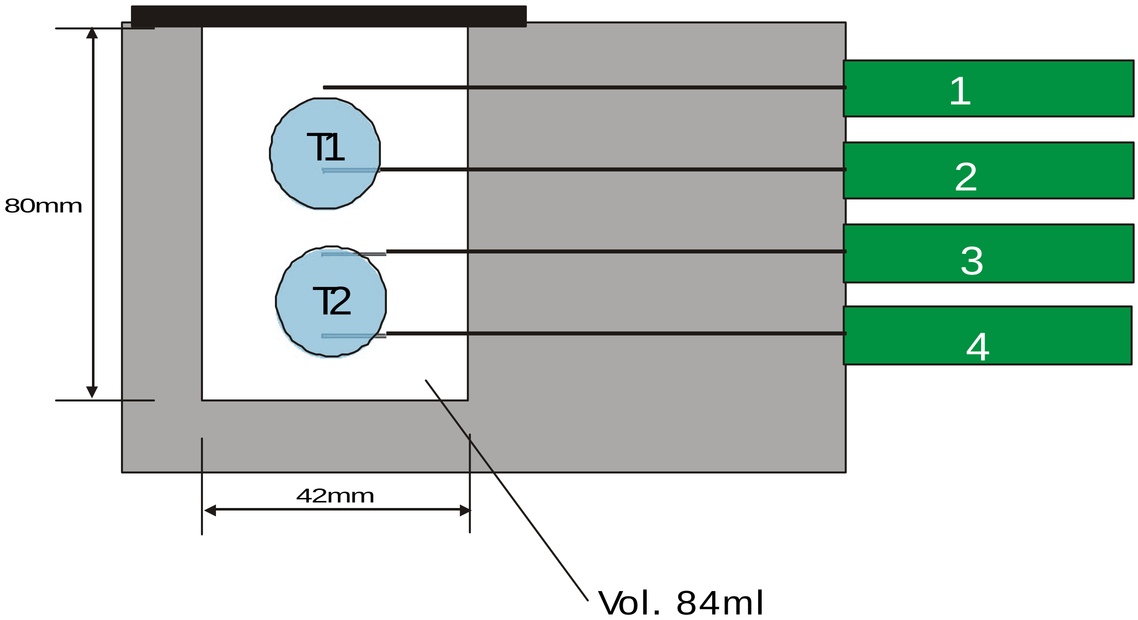

Figure 1. Ultrasonic and thermal measurement chamber for milk gelation processes. T1 and T2 refers to the upper and lower pair of transducers respectively. The items numbered from one to 4 are the temperature probes.

Two different initiators were used to promote the coagulation of milk: rennet and yoghurt. The initiator was added to the milk at ambient temperature, and was mixed carefully to prevent bubble formation. The yoghurt coagulation was started with a 10mg sample of a commercial yoghurt. The rennet gelation was started with a 30mg sample of a rennet based powder.

The reference for the measurement of the time of flight changes was taken 1hour after the milk with the starter was immersed in the water bath at 35º. This was the time the milk sample needed to reach the bath temperature from the ambient temperature (about 20ºC).

3. EXPERIMENTAL RESULTS AND DISCUSSION

Temperature Corrections on Milk Phase Velocity

Measurements of the temperature dependence of the wave velocity in milk as a function of the temperature were done to correct the temperature variations caused by the sol-gel transition during gelation processes. The temperature range studied -34-36ºC- includes the temperatures variations observed when the transition take place (Figure 2). It can be observed, that, within this range, the time of flight variation has a linear dependence with the temperature. A linear fit gives an approximate value of –7ns/ºC for this dependence. It must be noted, however, that this correction is a rough approximation because the temperature was measured at a fixed point, but the time of flight depends upon the propagation of a 20mm diameter wave front.

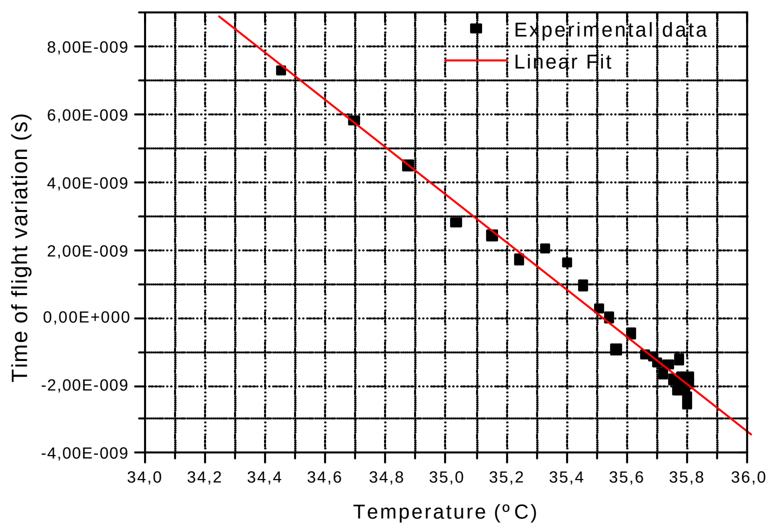

Figure 2. Time of flight variation in milk as a function of temperature. Experimental data (squares) and linear fit (red line).

Temperature Variations During Gelation

Temperature measurements during yoghurt formation are shown in Figure 3. It can be seen that, when the process starts, there is a temperature gradient in the chamber: the milk in the upper part is about 0.5ºC hotter than the milk at the bottom. Moreover, the temperature inside the chamber slowly fluctuates all along the whole test. Over these fluctuations, there is a clear temperature increment of 0.2ºC at the eighth hour. This sudden increment takes place during 20minutes and is related to the sol-gel transition [6]. It can be seen that all the temperature probes detected the same change except the upper one. This can be due to the serum separation which caused the upper probe to be situated out of the yoghurt formation region. After this transition, the temperature of the second probe went up beyond the temperature of the first probe, showing that convection vanishes when the yoghurt was formed.

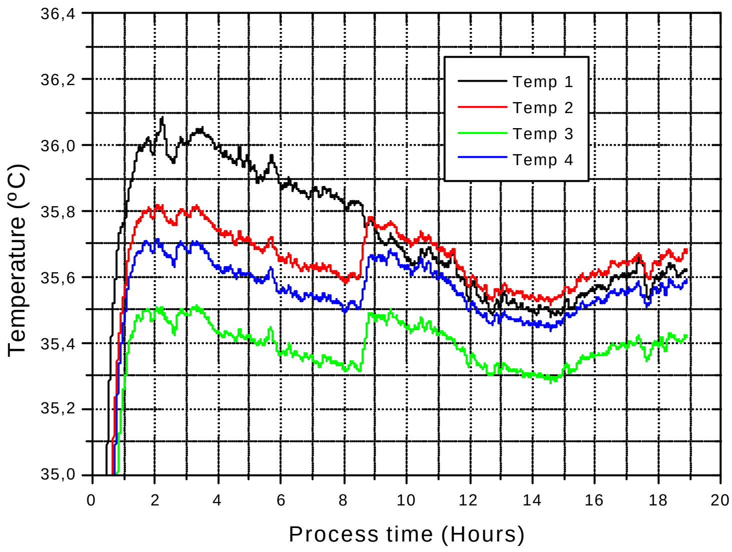

Figure 3. Temperature measurement during yoghurt milk coagulation

The temperature variation during rennet coagulation is shown in Figure 4. Although similar behaviour is shown, some differences can be noted. The temperature jump related to the sol-gel transition is bigger (about 0.4ºC) in the rennet coagulation. In addition, the temperature still increases beyond that point. In this case, the upper probe detected the gelation, but the temperature increment was smaller than the increment detected by the other probes. It can be seen that there is a small delay at the beginning of the sol-gel transition detected by the lowest probe. This retarded gelation can be related with the lower temperature at the bottom of the chamber, and was not observed in the case of yoghurt coagulation.

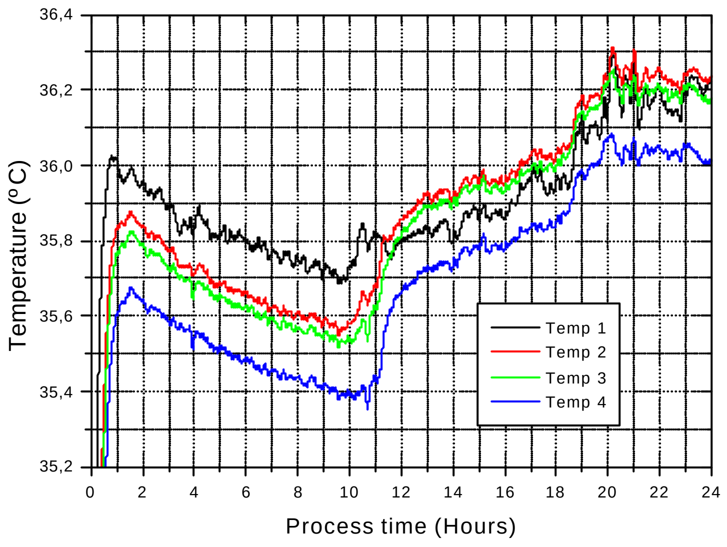

Figure 4. Temperature measurement during rennet coagulation of milk.

Phase Velocity Changes During Gelation

Figure 5 and 6 show the time of flight measurements during yoghurt and rennet coagulation processes respectively. The yoghurt coagulation stage begins at the sixth hour, when the time of flight shows a decreasing tendency which last 7 hours until the slope goes to zero. The temperature jump mentioned before occurs at the middle of this period –8.5 hours-. Comparing Figures 3 and 5, it seems that first, there is an increase of the phase velocity occurring in the milk when the structure of the protein micelles begins to change by the action of the bacteria metabolism. Nevertheless the indication that the biggest micelles are joined together would be related to the temperature jump taking place two hours later.

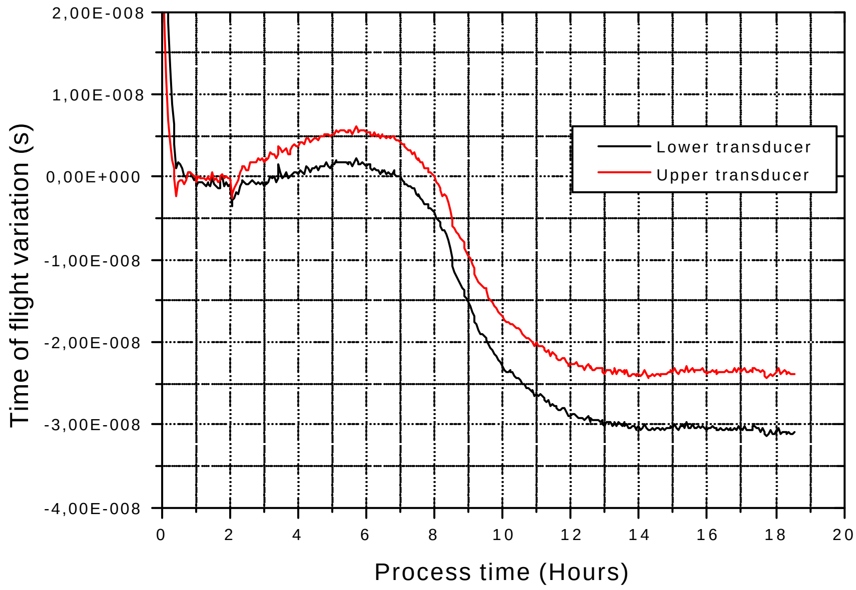

Figure 5. Time of flight variation of the ultrasonic wave received during yoghurt coagulation. Black and red lines refers to the ultrasonic propagation between the lower and the upper pair of transducers respectively –see Figure 1-.

The behaviour of rennet coagulation is somewhat different (Figure 6). This reaction occurs in two different phases. First, and enzymatic process begins to change the chemistry of the proteins. The time of flight does not give any indication of this process unlike Bakkali et al [4] who observed some delay variations in their experiments. On the other hand this time of flight plateau agrees with the results of Nassar et al [3]. After that, the second phase of the process begins and the aggregation of the micelles leads to the formation of a viscoelastic structure. This last process is related to the decreasing of the time of flight shown in Figure 6. Comparing this slope with the temperature curves in Figure 3, it can be noted that the temperature jump takes place just at the beginning of the time of flight decrease. Once the enzymatic phase of the rennet reaction has finished, the coagulation takes place very quickly with no other physical changes in the structure. Nevertheless, the consolidation of the viscoelastic structure take a longer time (about 12 hours) compared to the yoghurt coagulation (about 7 hours), and the absolute change of the propagation velocity is bigger.

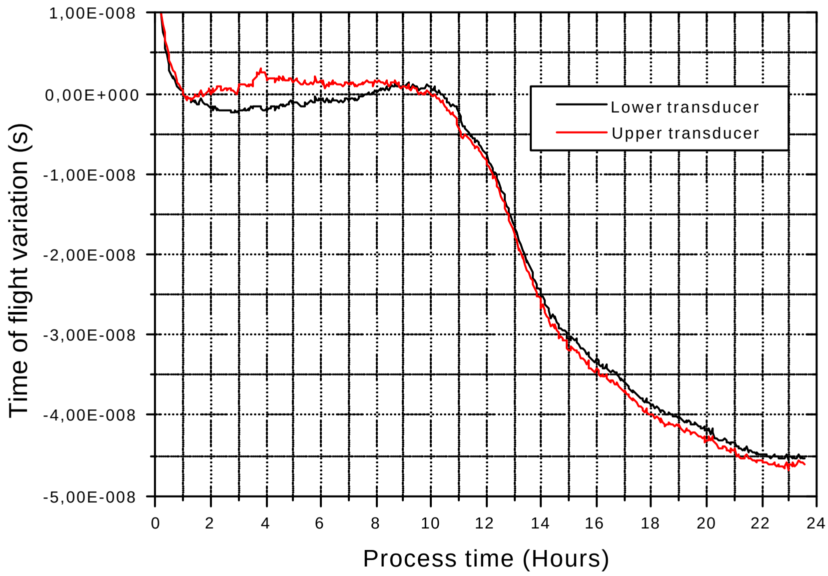

Figure 6. Time of flight variation of the ultrasonic wave during rennet coagulation of milk. Black and red lines refers to the ultrasonic propagation between the lower and upper pair of transducers respectively.

4. CONCLUSIONS

A non-invasive ultrasonic through-transmission method for the control of coagulation processes in milk was presented in this paper. The method is based on the propagation velocity changes experienced by an elastic pulse propagating through the coagulating media. The elastic properties of the coagulating milk depend on the chemical reactions that modify the structure of the protein micelles, promoting their aggregation, sol-gel transition and consolidation of the final viscoelastic structure. When the ultrasonic time of flight detection is combined with temperature measurement, different stages of this coagulation process can be identified. Yoghurt and rennet coagulation were monitored and compared. The results obtained show the suitability of this non invasive and low cost method to monitor gelation processes in the dairy industry.

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