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# Estimation of coagulation time in cheese manufacture using an ultrasonic pulse-echo technique



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#### ABSTRACT

The essential parameter in the cheese-making industry is the coagulation time because it marks the end point of the enzymatic phase and the start point of the aggregation. Furthermore, it is used to evaluate the cutting time that characterizes the time at which curd is cut. This paper proposes a non-destructive ultrasonic pulse-echo to monitor in real-time the acoustic impedance of milk during cheese manufacturing in order to predict the coagulation time. The evolution of the acoustic impedance is going through two phases the enzymatic phase and the aggregation phase. The transition point between these two phases defines the milk coagulation time. Three rennet concentrations and three temperatures were considered in our experiments to study the effect of these factors on coagulating milk. The experimental results prove that our proposed method determines the coagulation time with efficiency, and it is more simple compared with our previous work in which the phase velocity was used to determine the milk coagulation time.

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#### 1. Introduction

The milk enzymatic coagulation is the most studied topic in cheese production which is the final product of many consecutive operations which start by adding rennet to milk, cutting the coagulum, draining the whey, shaping, and other operations. When the rennet is added to milk, it induces the kappa-casein hydrolysis that causes the casein micelles destabilization. After that comes the beginning of an aggregation reaction that leads to the formation of a net structure and a space-filling gel. These gel properties depend on different factors, like milk physico-chemical properties and conditions of coagulation process such as the concentration of enzyme, temperature, pH and  $Ca^{2+}$  concentration. (Dalgleish, 1993; Hyslop, 2003; Corredig and Salvatore, 2016).

The enzymatic phase end point and the aggregation start point are marked by the coagulation time which is used as a reference to determine the cut-time (the time at which the curd is cut), in order to drain the whey. Some cheesemakers cut the curd early rather than late as a rule, and once it is cut, the curd should be left to complete its forming process in the warmer whey which rises over it. Knives or curd breakers crush the curd rather than cutting it cleanly if the coagulum becomes too firm. Cutting when the coagulum is too soft decreases cheese yield due to increased loss of fat and curd fines. Cutting when the coagulum is too firm retards syneresis and results in high-moisture cheese. Thus, determining coagulation time is a critical parameter to find the optimal cuttime, which is important to maximize yield, increase the quality and improve homogeneity of cheese.

We can categorize the existing methods for monitoring milk coagulation in methods not supporting inline applications such as Berridge method (Berridge, 1952) or traditional practices (finger and knife tests) and methods suitable for inline applications. Some rheological methods (Steinsholt, 1973; López et al., 1999; Richardson et al., 1971: Van Hoovdonk et al., 1986: Vanderheiden, 1976; Hatfield, 1981; Scott Blair and Burnett, 1958; McMahon and Brown, 1982) have been proposed for inline applications but they are destructive. The electrical method (Dejmek, 1989) is not destructive, however, there are possibilities of interference between the original milk electrolytes and the measurement. Hori (1985) proposed a thermal method to monitor milk coagulation but this method is not well suited for measuring gel stiffness. Optical methods also were widely used for monitoring coagulation of milk (Castillo et al., 2000, 2003, 2005; Lagaude et al., 2004, 2006; Fagan et al., 2008; Nicolau et al., 2011; Lyndgaard et al., 2012;

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Tabayehnejad et al., 2012; Arango et al., 2013; De Marchi et al., 2013; Abdelgawad et al., 2014; Arango et al., 2015; Nicolau et al., 2015).

Over the years, ultrasonic sensing devices have been used for coagulation monitoring because they provide non-destructive, quick and low cost measurements. To predict the curdled milk coagulation time. Av and Gunasekaran (1994) employed a pulsetransmission method of measurement during coagulation of renneted milk. The observed changes in ultrasonic attenuation during coagulation were used to predict the coagulation time. The point at which there is a transition from more rapid to slow change in attenuation was identified as the turning point (the coagulation time). This later was determined by examining the first derivative of the attenuation versus time plots. Our research group (Bakkali et al., 2001) inspired from the work of Ay and Gunasekaran (1994) to develop a pulse-reflection technique which investigate the coagulating milk enzymatic phase by measuring the phase velocity. They reported that an efficient method is provided by ultrasonic velocity measurements to determine milk coagulation time. While Nassar et al. (2004) studied the technological factors influence (pH, temperature and milk powder concentration) on the flight time variations of an ultrasonic pulse during milk gelation. An ultrasonic spectroscopy method was proposed later to analyze rennet-induced changes in milk (Dwyer et al., 2005). The changes in ultrasonic velocity and ultrasonic attenuation over the renneting process were compared with results from dynamic rheology measurements and near infrared transmission. Koc and Ozer (2008) developed an ultrasonic measurement system which is nondestructive for controlling the coagulation of the rennet-induced whole cows milk during cheese making. They determined the coagulum optimal cutting time by monitoring the ultrasonic attenuation coefficient temporal evolution. The aim of our work is to develop a new ultrasonic measurement system providing a nondestructive determination of milk coagulation time based on acoustic impedance. This later has been used extensively in food engineering (Coupland and Saggin, 2001; Saggin and Coupland, 2001) but not in determination of milk coagulation time. Furthermore, our technique involves just one transducer and it is non-invasive, that is very important in the food industry for hygiene reasons.

#### 2. Materials and methods

#### 2.1. Materials

We prepare milk samples by dissolving 13 g of whole milk powder from Nestle brand (contains 26% of fat content i.e 3.4% of fat content after reconstitution and 24% of protein) in 90 ml of warm distilled water. After that, we stir the obtained mixture at room temperature. The milk was heated to a testing temperature during 30 min in a thermostated water bath (Model 18233, Voltage 240 V, Made in USA) to equilibrate the system. After that, we dilute a commercial rennet (Caille-lait universel, 0.22 g/l) with distilled water. Then, we mix the rennet with the milk and stir it immediately for 30s.

#### 2.2. Ultrasonic system

An ultrasonic system was designed to set-up the experimental pulse echo shown in Fig. 1. The coagulating milk is enclosed in a parallelepiped vessel of  $10 \times 10 \times 6cm^3$  dimensions, whose two sides are made from Plexiglas. An ultrasonic transducer is placed next to the Plexiglas side. The whole is in a thermostated bath of water.

We use during the experiments a broadband transducer of frequency 5 MHz and a 10 mm crystal diameter (A309S-SU Model Panametrics Olympus), the transducer played the role of an emitter/receiver. We use also an ultrasonic pulse generator (Model 5052PR Sofranel, Voltage 230 V, Bandwidth 50 MHz) to excite the transducer by an electrical short pulse. This later is transformed into an acoustic wave. After different interfaces reflections shown in Fig. 2, the received signal (Fig. 3) is amplified and digitized by a PicoScope (6403A Model, Resolution 8bits at 5GS/s, Bandwidth 350 MHz, Memory 256Ms).



Fig. 1. Experimental device.

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