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Using a fiber optic sensor for cutting time prediction in cheese manufacture from a mixture of cow, sheep and goat milk

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ABSTRACT

NIR light backscatter technology has been proven successful for monitoring cow milk coagulation and predicting cutting time but has never been tested with milk mixtures. In Spain ~40% of the cheese produced is made from cow, sheep and goat milk mixtures. The aim of this study was to evaluate if the proposed optical technology could be used to monitor milk coagulation and predict cutting time in milk mixtures. A randomized factorial design with three factors and three replicates was employed. Cow, goat and sheep milk was mixed in two different proportions. Milk mixtures were coagulated at constant calcium chloride addition level, pH and fat concentrations using two different enzyme concentrations and three coagulation temperatures ($N = 36$ tests). Milk coagulation was monitored using small amplitude oscillatory rheometry and a NIR fiber optic light backscatter sensor. Simultaneously, clotting time was visually evaluated. Optical parameter t_{max} was highly correlated ($0.80 < r < 0.99$, $P < 0.0001$) with the rheological and visual parameters studied. Enzyme concentration and temperature had a significant effect ($P < 0.05$) on optically-, rheologically-, and visually-derived parameters. Milk mixture proportion was not significant for optical parameters related to clotting time but was significant for the aggregation rate and rheological parameters related to curd firming and syneresis. Models for predicting cutting time were developed successfully with $R^2 = 0.93$. Results strongly suggest that milk mixture proportion exerts an effect on gel assembly (i.e., on both aggregation and curd firming) and syneresis. This finding has important implications for inline process control when goat and sheep milk are used.

1. Introduction

Milk coagulation is typically induced by acidification or enzymatic action to form a continuous, solid curd that entraps fat globules and some water (Castillo et al., 2006a,b,c). The most common method of milk coagulation is enzymatic coagulation. Once a sufficiently firm gel has been obtained, the gel needs to be cut into small pieces (curd grains) to induce syneresis (i.e., the expulsion of whey as a result of curd grains shrinkage). This operation increases the gel surface/volume ratio allowing the whey to escape while the gel network is rearranging and contracting (Castillo, 2006). Traditionally, the curd is cut after a predetermined time from the enzyme addition or upon the operator's judgment based on empirical evaluation of firmness and visual appearance of the gel properties. Cutting the coagulum after a pre-fixed time is questionable, since variations in milk properties and processing conditions affect curd firmness and gel microstructure, modifying the optimum cutting time. A number of authors have noted the disadvantages of an inappropriate cutting time selection (Hori, 1985; Payne et al., 1993a; Passos et al., 1999). Real-time estimation of curd firming and cutting time is essential for cheese making as those two factors exert a substantial impact in both cheese yield and quality (Bakkali et al., 2001). A plethora of devices have been developed for milk coagulation and gel firming monitoring over the past seven decades. A comprehensive classification of those devices was published by Castillo (2006). In general, those systems studying rheological properties are destructive and not practical for inline application. To date, an objective and effective method to determine optimum cutting time is not available, although some existing methods can consistently reproduce the cutting time subjectively selected by the cheesemaker. An inline optical sensor designed to measure changes in light backscatter of infrared light at 880 nm was proposed by Payne et al. (1993b) to predict cutting time. This technology has been specifically developed for cow milk, and adapted successfully to goat milk (Castillo, 2001). However, it has never been tested on milk mixtures having different proportions of cow, goat and sheep milk. Previous studies have shown that light backscatter does not only depend on milk composition

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(fat and protein percentages) but also on casein micelle and fat globule size. Milk from different animal species has both different milk composition and particle size distribution (Payne et al., 1993b; Castillo, 2001). Almost half of the cheese produced in Spain is made with different mixtures of milk from cow, sheep and goat (ICEX, 2004). The production of cheese using mixtures of milk (hereafter "*mixed milk cheese* –**MMC**–") has achieved widespread acceptance in Spain. A large variety of MMCs are typically manufactured in Spain such as "*Cabrales*", "*Picón*", "*Gamonedo*" and "*Ibérico*". Proportions of the different milk types were established by the Ministry of Agriculture in 1987, in compliance with cheese producers ("Orden de 9 de julio de 1987"), but this law has been recently repealed ("Real Decreto 262/2011, de 28 de febrero"). MMCs are very important for the Spanish cheese sector, not only for the proportion of sales it represents, but also because of technology differences required for appropriate processing of the different milk mixtures encountered. Sheep, goat and cow milk show marked differences in their colloidal structure and chemical composition, which introduces additional difficulties, compared with cheeses made with one type of milk, as regards the control of coagulation and the selection of cutting time.

The general objective of this paper was to evaluate if near infrared light backscatter could be used to monitor milk coagulation and predict cutting time in cheese made from different proportions of cow, goat and sheep milk. This general objective was divided into two specific objectives: (a) evaluate the effect of milk mixture proportions, enzyme concentration, and coagulation temperature on the light backscatter profile, and (b) obtain the best prediction models for several milk coagulation indicators and cutting time.

2. Materials and methods

2.1. Experimental design

A randomized factorial design with three factors (a, b and c) and three replications ($n = 3$) was used to determine the effect of different milk coagulation temperatures, enzyme concentration levels and milk mixture proportions on the light backscatter profile during milk coagulation and on the prediction of clotting and cutting time during cheese manufacturing. Different levels of the experimental factors were selected to obtain light backscatter profiles under a wide range of milk coagulation conditions. Two levels of "*milk mixture*" were prepared using cow, goat and sheep milk as detailed in Table 1. Those two levels of milk mixture were established according to typical industrial practice for MMCs typically manufactured in Spain in compliance with current Spanish regulations (Real Decreto 262/2011, de 28 de febrero). Milk mixtures were coagulated using two "*enzyme concentrations*" (200 and 400 mg L^{-1}) at three "*temperatures*" (27, 32 and 37 °C) at constant fat concentration, calcium chloride ($CaCl_2$) addition level and pH (4.5%, 174 mg L^{-1}, and 6.5, respectively). A total of 36 tests (N = nabc = 3 2 2 3 = 36) were conducted under this design. A fresh batch of each type of milk was obtained for each replication to reconstitute mixtures 1 and 2 (i.e., mixtures for each replication were made using a new batch of the three milk types). The different treatments of each replication were conducted in random order. Fig. 1 shows the flow chart of the experimental design. Milk coagulation was monitored using small amplitude oscillatory rheometry (**SAOR**) and near infrared (**NIR**) light backscatter. Simultaneously, clotting time was visually determined.

Table 1
Types of milk mixtures used in the experiment.

	Cow (%)	Goat (%)	Sheep (%)
Mixture 1	60.0	30.0	10.0
Mixture 2	75.0	12.5	12.5

2.2. Materials

Milk: Goat and sheep milk were obtained from the Universitat Autònoma de Barcelona (**UAB**) farm while the cow milk was obtained from Can Badó farm (S.A.T. Can Badó, La Roca Del Vallès, Spain). Immediately after milk was received in the Centre Especial de Recerca Planta de Tecnologia dels Aliments (**CERPTA**) at UAB, milk was stored at ~4 °C until it was used (typically within the first 24 h from milk reception) to prepare the corresponding milk mixtures. *Calcium chloride*: A calcium chloride solution 40% *w*/*v* was prepared using dehydrate calcium chloride ($CaCl_2$ $2H_2O$; Panreac Química S.A., Montcada i Reixac, Barcelona, Spain). A constant amount of 0.74 mL of the prepared calcium solution was added per liter of milk to each milk mixture after pasteurization. This calcium solution aliquot was calculated to deliver 174 mg of anhydrous calcium chloride ($CaCl_2$) per liter of milk. *Enzyme*: Bovine rennet (~70% chymosin, ~30% pepsine) with strength of 1:10,000 was obtained from Laboratories' Arroyo (Santander, Spain) and used to induce milk coagulation. *Chemical reagents*: Fat concentration was determined by Gerber method using sulfuric acid (H_2SO_4) solution (90–91% *w*/*w*; $M = 98.08$, $\delta = 1.82$ g cm^{-3}) from Scharlab (Mas d'en Cisa, Spain) and using 3-methyl-1 butanol ($C_5H_{11}OH$, $M = 88.15$) from Panreac Química S.A. (Montcada i Reixac, Barcelona, Spain). For pH measurements, standard buffer solutions were used (pH buffers 7.00 and 4.01; Crison Instruments, S.A., Alella, Barcelona, Spain). For pH adjustment, sodium hydroxide solution 1 M (Scharlab) and hydrochloric acid 1 M (Panreac Química) was used.

2.3. Milk mixtures preparation

Unpasteurized and unhomogenized cow, goat and sheep milk was stored at 4 °C right after collection and until it was used to prepare the two milk mixture types established by the experimental design. Gerber (IDF Standard 83, 1987) and Dumas methods (Dumas, JAOAC 59, 141 (1976)) were used to determine the fat and protein content of milk, respectively. Milk total solids were determined using a convention oven (IDF Standard 86, 1981). Ash content of milk was determined by dry ashing (AOAC 954.46, 2000) using a high temperature muffle furnace. Lactose was calculated by difference. Table 2 shows the average composition of the milk used in this study.

On the day after collection and for each replication, a batch of each one of the two milk mixtures was prepared in compliance with the experimental design (Fig. 1), by mixing the corresponding proportions of cow, goat and sheep milk. Milk mixtures were skimmed at ~45 °C using a small cream separator (Elecrem1-125L/h, Elecream, Vanves, France). Fat content of resulting skimmed milk mixtures and corresponding cream batches were determined using the Gerber method to calculate the adequate proportions of skimmed milk mixture and cream for adjustment of the fat concentration of the final milk mixtures to 4.5%. Mixtures proportions were calculated based on mass balance. The final milk mixtures were vat pasteurized at 65 °C for 30 min. Then a constant amount of calcium chloride solution (0.74 mL per liter of milk) was added to each milk mixture. The milk was stirred for 3 min and stored in a cooler until the temperature reached 23 °C. A linear regression between mL of 1 M HCl dilution and pH was conducted at 23 °C and used to predict the amount of acid needed to adjust the pH of milk to 6.5. The day before the experiment, milk samples were tested for pH and the amount of acid required for pH adjustment was calculated. Milk samples were placed into glass containers and the acid was added slowly and with continuous stirring. A constant sample dilution