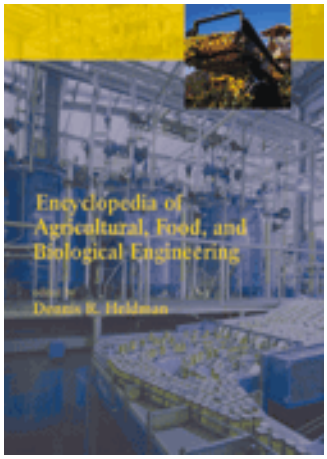
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Light Backscatter Sensor Applications in Milk Coagulation

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Light Backscatter Sensor Applications in Milk Coagulation

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Abstract
Complete automation of cheese production requires an inline method for monitoring milk coagulation. Static light backscatter measurements have been demonstrated to be one of the most effective techniques for this purpose. This study reviewed the most significant implications of recent research findings on the use of light backscatter for monitoring milk coagulation.

INTRODUCTION

Real-time estimation of curd firming and prediction of cutting time are essential for milk coaugulation control during cheese making. A plethora of devices have been developed for that purpose over the past seven decades. Most of the proposed instruments have been developed for fundamental research of coagulation and gel properties, evaluation and development of rennets and starter cultures, or development of inline cutting time sensors. Several authors have reviewed the apparatus that have been proposed for gel formation monitoring and cut-time setting.[1–4] A comprehensive classification of those techniques was published recently by Castillo.[5] Among the various techniques proposed, the group that stands out is based on optical parameters from light backscatter (LB) measurement. Optical backscatter measurement techniques using optical fibers have several inherent advantages, including the measurement of light through the equivalent of small path lengths and the miniature size of optoelectronic components. Optical techniques are also very suitable for inline measurement using optical fibers and can be continuous and nondestructive.

A fiber optic sensor technology used to measure LB has been demonstrated to be one of the most promising inline, nondestructive methods for monitoring milk coagulation. This technique has become commercially available for inline monitoring of cheese production. Since 1990, when this technique was proposed,[6] it has been extensively investigated and improved, but implications of recent research findings have not been reviewed. Our goal was to review the current state of the art of this technology.

THE LIGHT BACKSCATTER SENSOR

The LB sensor measures changes in backscatter of infrared light at 880 nm. The use of two optical fibers spaced ~0.7 mm apart (Fig. 1a) to transport the light is a unique optical configuration that yields a strong signal proportional to the changes that occur in the protein structure during coagulation. Light from a light-emitting diode (LED) is transferred to the milk through a fiber, and the light backscattered from the milk is transmitted through an adjacent fiber to an optical detector. Fig. 1b shows a picture of the LB fiber optic sensor (Model 5 CoAguLite, Reflectronics Inc., Lexington, Kentucky). The LB signal contains information about aggregation of casein micelles and gel assembly during milk coagulation. The LB profile increases sigmoidally as milk coagulation proceeds for acid-, mixed-, or rennet-induced coagulation in both cow and goat milk (raw and skim). The LB ratio is generated by dividing the voltage from the sensor, V, by the voltage V_0 obtained by averaging over the period of 1 min after adding the enzyme. The LB ratio begins with a value of 1 and represents the increase in signal during coagulation (Fig. 1c). The LB ratio profile has a latent period (Fig. 1c, I) during which enzymatic reactions predominate with no detectable change in backscatter intensity. The LB ratio increases as particle size increases during network formation due to casein micelle cross-linking. As a result, a sigmoidal period can be distinguished (Fig. 1c, II) during which aggregation reactions predominate, as well as an asymptotic period (Fig. 1c, III) during which cross-linking proceeds at an ever-decreasing rate while curd firming is developing. The LB ratio typically increases 20, 50, and 260% during coagulation for goat milk, skim cow milk, and yogurt or cottage cheese, respectively.

THE PARAMETER t_{max}

As a consequence of the sigmoidal shape of the LB ratio profile, a unique parameter is obtained: the time from

Keywords: Milk; Coagulation; Cutting time; Prediction; Monitoring; Inline; Sensor; Probe.

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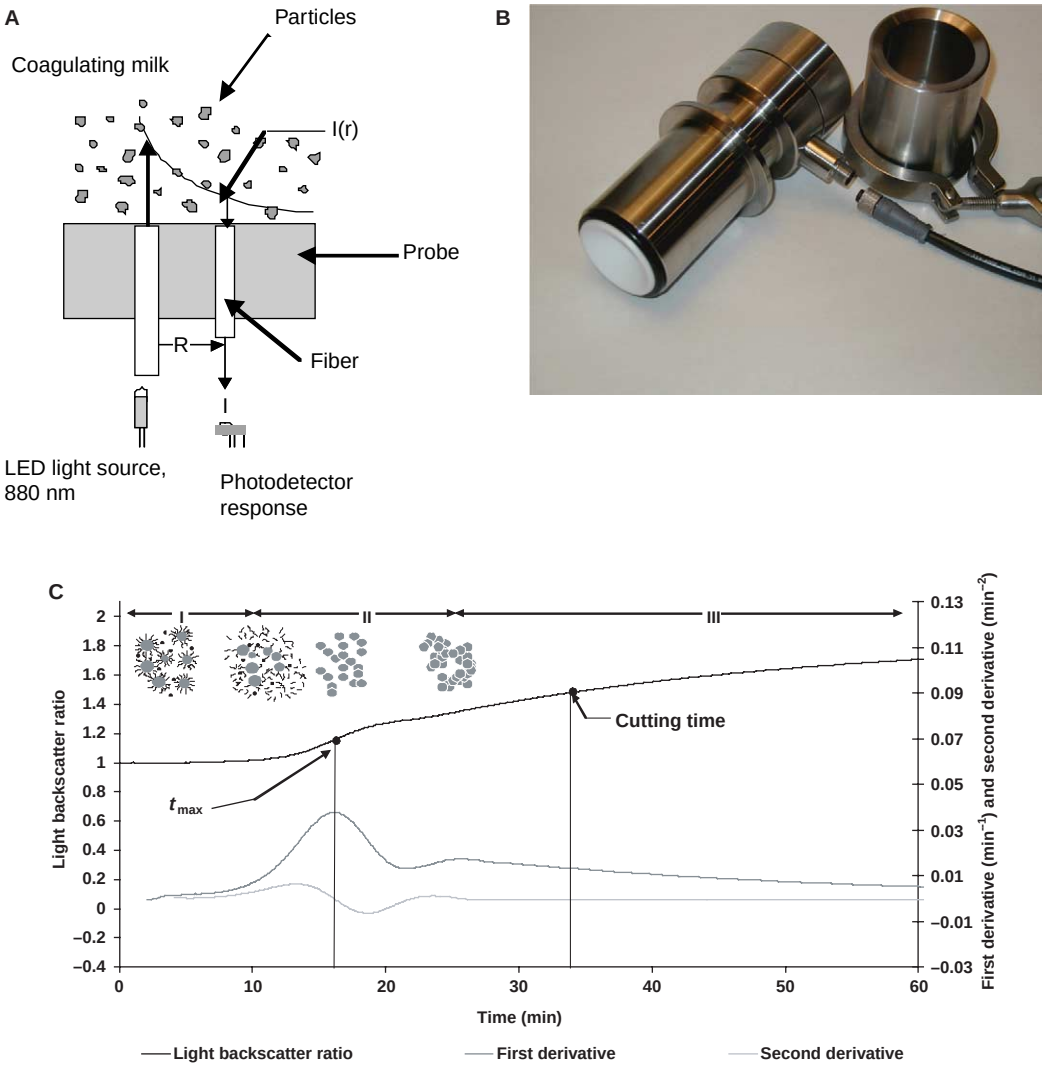


Fig. 1 (A) Schematic of LB sensor used for milk coagulation monitoring. I(r) is the light intensity as a function of radial distance. *R* is the distance between fiber centerlines. (B) Picture of the light backscatter sensor. (C) LB profile and its first and second derivatives as a function of time. (I) Latent period; (II) sigmoidal period; (III) asymptotic period.

enzyme addition to the inflection point of the LB ratio profile, t_{max} (Fig. 1c). One important discovery is the strong correlation between t_{max} and the enzymatic reaction rate, k.[7] Thus, the LB measurement indirectly measures the enzymatic reaction rate when it measures t_{max}. Another important discovery is that the onset of aggregation is located between the induction period (the time from enzyme addition to a point at which the magnitude of the first derivative of the normalized LB ratio is equal to 0.025) and t_{max}, and that the extent of hydrolysis at t_{max} is near completion.[8] Thus, it is not surprising that when temperature, pH, added calcium, or enzyme concentration change, t_{max} changes accordingly. For instance, Castillo[1] showed that t_{max} changed inversely with enzyme concentration—as Berridge clotting time—according to the Foltmann equation, and the slope of the LB ratio profile at t_{max} followed an Arrhenius-type equation as a function of temperature (in the absence of significant rennet heat inactivation).

MODEL FOR SIMULTANEOUS AGGREGATION AND CURD FIRMING

Research by Castillo et al.[8] has shown that reaction rates for aggregation, k_2, and gel firming, k_1, can be estimated from the LB ratio profile. It should be noted that at t_{max}, milk still behaves as a liquid—no signs of clotting are observed; and the storage modulus, G', is not measurable. According to Castillo et al.[8] at t_{max}, κ-casein hydrolysis is near completion ($\alpha = 0.78\%$), and micelle aggregation is in its earliest stages. Thus, the increase in LB ratio after t_{max} was attributed to the overlapping of casein micelles aggregation (the disappearance of single micelles in

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Table 1 Effect of temperature and percentage of protein on kinetic parameters for aggregation and curd firming measured by LB in coagulating goat milk

	R_∞	β_1 (%)	k_2 (s^{-1})	k_1 (s^{-1})	τ_2(s)	τ_1(s)
Temperature,[h] (°C)						
20	1.67[a]	58.6[a]	0.00237[a]	0.000263[a]	1905[a]	2942[a]
25	1.87[b]	27.6[b]	0.00810[a]	0.000324[a]	885[b]	2306[b]
30	2.08[c]	14.4[c]	0.0310[a]	0.000469[a]	379[c]	1531[c]
35	2.14[c]	9.93[d]	0.100[b]	0.000888[b]	166[d]	857[d]
40	2.13[c]	6.82[d]	0.365[c]	0.00172[c]	77[d]	492[d]
Protein,[i] (%)						
3	1.79[e]	17.3[e]	0.201[e]	0.000913[e]	378[e]	1277[e]
5	2.04[f]	25.7[f]	0.073[f]	0.000706[ef]	630[f]	1718[ef]
7	2.11[g]	27.4[f]	0.030[f]	0.000580[f]	1040[g]	1881[f]

[a–g]Least squares means with same superscript are not significantly different ($p<0.05$). Comparisons are made only within the same column. Number of replications = 3; Number of observations = 45.

[h]Least squares means for each temperature are based on average of 9 trials over a range of 3 protein levels.

[i]Least squares means for each protein level are based on average of 15 trials over a range of 5 temperatures.

R_∞, asymptotic value of the LB ratio at infinite time; β_1, increase in LB ratio, after t_{max}, resulting from aggregation reaction, expressed as a percent of the total increase in LB ratio; k_1, curd firming rate constant; k_2, aggregation rate constant; τ_1, characteristic time for curd firming process; τ_2, characteristic time for aggregation.

Source: From Cambridge University Press (see Ref. 8).

solution to form doublets, or casein micelle fusion) and curd firming reactions (the development of gel firmness as a result of the disappearance of cross-linking sites in the preliminary network by further incorporation of casein micelles or strands, or by formation of new links between micelles). A kinetic model was proposed that combines a second-order reaction model to describe aggregation reactions and a first-order reaction model to describe firming process reactions. The effect of temperature and protein concentration on the kinetics of gel assembly measured by LB ratio was studied using this model, as shown in Table 1. This model by Castillo et al.[8] showed that the LB ratio profile during coagulation contains information regarding the enzymatic reaction rate, the rate of aggregation, and the rate of gel firming.

THE PARAMETER t_{max} AS PREDICTOR

The backscatter parameter, t_{max}, is highly correlated with cutting time, as objectively determined using the Formagraph parameter k_{20}. The following prediction equation was developed by Payne et al.[9] to predict cutting time (t_{cut}):

$$t_{cut} = \beta t_{max}. \quad (1)$$

The regression coefficient β typically varies between 1.15 and 2.4, depending on the enzyme used and the type of milk and product produced. The coefficient β is selected in the plant to replicate the cheese maker's judgment of cutting time. The value of β calibrates the system and is the only variable that the cheese maker has to determine. Eq. 1 is accurate only if protein is constant. Castillo[1] showed that β decreases significantly when protein increases. This allowed the development of a prediction algorithm to correct the cutting time prediction for changes in milk protein content as follows:

$$t_{cut} = \beta_0 t_{max}(1 + \gamma P), \quad (2)$$

where the constant γ (whose value would likely depend on the type of milk used; e.g., cow milk vs goat milk) corrects the cutting time prediction for the effect of protein concentration (P). Thus, the regression coefficient β_0 is the only parameter that requires in-plant calibration.

The parameter t_{max} has also been demonstrated to predict successfully the Berridge clotting time[1] and the rheological gelation time.[10,11] The repeatability of a specific parameter such as Berridge clotting time is a concern for a laboratory measuring system. In a precision test,[12] 95% of the t_{max} measurements (average $t_{max} \pm 2$ SD) for uniform milk and constant processing conditions fell within the range 15.31 ± 0.33 min ($n = 10$), which corresponds to a relative error $\leq 2.2\%$, for 95% of the measurements. This compares with the repeatability requirements by IDF standards 110A:1987 and 157A:1997 for visual Berridge clotting time[13] and total milk clotting activity[14] determination, respectively. It should also be noted that t_{max} is an objective measurement rather than a visual subjective measurement.

Castillo et al.[15] found that the LB ratio profile parameter, t_{2min^2} (second minimum of the second derivative), was not significantly different from the gelation time determined by oscillatory rheometry

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($p=0.36$). The correlation between t_{2min^2} and gelation time was very significant ($r=0.992$, $p<0.0001$). This evidence strongly suggests that the rheologically determined gel time and the LB ratio parameter, t_{2min^2}, corresponded to the same physical phenomenon: the beginning of the gel-firming process.

DETECTION OF COLLOIDAL CALCIUM PHOSPHATE DEMINERALIZATION

In addition, a local minimum in the LB ratio profile has been observed during acid milk coagulation at high incubation temperatures in the presence of rennet.[15] The local minimum of the LB ratio profile (Fig. 2) corresponded to a weakening of the gel that was demonstrated by a simultaneous decrease of the storage modulus, G', and increase of the loss tangent, tan δ. The weakening of the gel was attributed to the progressive micelle demineralization induced by ongoing acidification of gelling or gelled casein particles. The solubilization, especially at high temperatures, of colloidal calcium phosphate (CCP) during continuous acidification in the presence of small amounts of rennet marked a transition from a rennet-type gel to an acid-type gel.

CONCLUSIONS

The LB ratio profile obtained during coagulation contains information regarding the enzymatic, aggregation, and gel-firming reaction rates. LB ratio increases for acid-, mixed-, or rennet-induced coagulation of cow and goat milk as particle size increases during network formation due to casein micelle cross-linking. A kinetic model based on this technique[8] has been proposed to monitor gel assembly that allows estimating the kinetic rate constants for casein micelle aggregation and curd firming. The LB ratio parameter, t_{max}, is measured with a relative error of 2.2%, which compares with the repeatability required by the IDF for Berridge clotting time determination. This LB ratio parameter allows the prediction of the Berridge clotting time, the rheological gelation time, the Formagraph parameter k_{20}, and the visual cutting time objectively selected by the cheese maker. The LB ratio parameter, t_{2min^2}, appears to correspond to the beginning of gel firming. Finally, the LB ratio was found to respond to the weakening of the gel network due to CCP demineralization that occurs during acid milk coagulation at high incubation temperatures in the presence of rennet. This review shows that LB represents an alternative method for studying milk coagulation and its kinetics, and is a technology that can be used commercially to improve cheese production efficiency, consistency, and quality.

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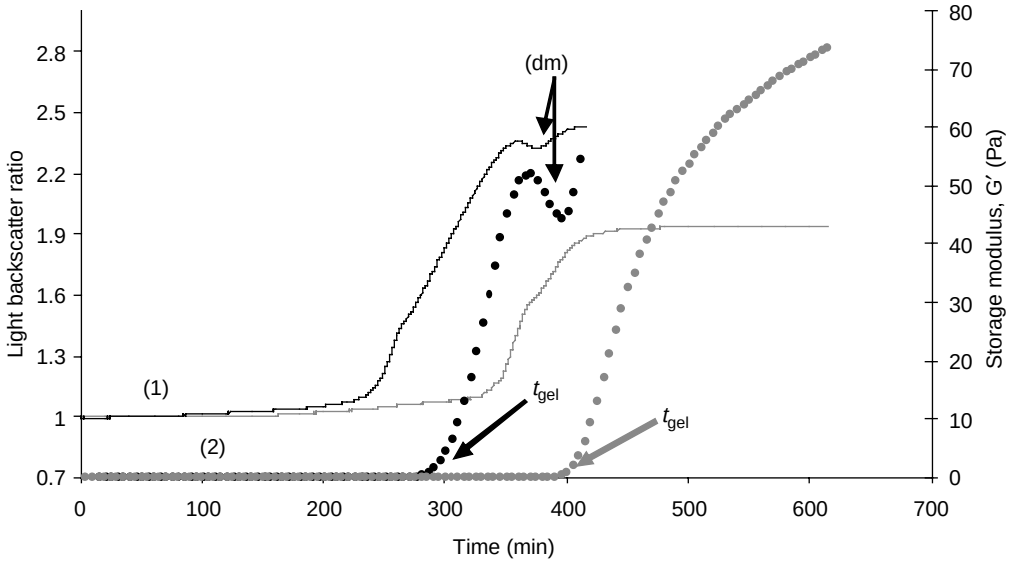


Fig. 2 Effect of rennet on formation of cottage cheese gels. Data corresponded to skim milk coagulated at 32°C using 0.5% starter culture. Black lines, gels made with 2 mg kg^{-1} of rennet; Grey lines, gels made without rennet; (1), Light backscatter profiles; (2), storage modulus; (dm), CCP demineralization; t_{gel}, rheologically-derived gelation time.
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REFERENCES

1. Castillo, M. Cutting time prediction during cheese making by near infrared light backscattering, PhD thesis, University of Murcia, Spain, 2001.
2. Thomasow, J.; Voss, E. Methods for the determination of the firmness of milk coagulum. IDF Bull. **1977**, *99*, 1–7.
3. O'Callaghan, D.J.; O'Donnell, C.P.; Payne, F.A. Review of systems for monitoring curd setting during cheesemaking. Int. J. Dairy Technol. **2002**, *55*, 65–74.
4. Lucey, J.A. Formation and physical properties of milk protein gels. J. Dairy Sci. **2002**, *85*, 281–294.
5. Castillo, M. Cutting time prediction methods in cheese making. In *Encyclopedia of Agricultural, Food, and Biological Engineering*; Heldman, D., Ed., Taylor & Francis Group: Boca Raton, FL, 2006; 1–7.
6. Payne, F.A., Madangopal, S., Hicks, C.L., Shearer, S.A., Fiber optic milk coagulation sensor for cut-time detection. Food Processing Automation Conference; American Society of Agricultural Engineers, St. Joseph, Michigan, Publication 02-90, 173, 1990.
7. Saputra, D.; Payne, F.A.; Lodder, R.A.; Shearer, S.A. Selection of near-infrared wavelengths for monitoring milk coagulation using principal component analysis. Trans. ASAE **1992**, *35*, 1597–1605.
8. Castillo, M.; Payne, F.A.; Hicks, C.L.; Laencina, J.S.; Lopez, M.B. Modeling casein aggregation and curd firming in goats' milk from backscatter of infrared light. J. Dairy Res. **2003**, *70*, 335–348.
9. Payne, F.A.; Hicks, C.L.; Shen, P.S. Predicting optimal cutting time of coagulating milk using diffuse reflectance. J. Dairy Sci. **1993**, 7648–7661.
10. O'Callaghan, D.J.; O'Donnell, C.P.; y Payne, F.A. A comparison of on-line techniques for determination of curd setting time using cheesemilks under different rates of coagulation. J. Food Eng. **1999**, *41*, 43–54.
11. Castillo, M.; Payne, F.A.; Wang, T.; Lucey, J.A. Effect of temperature and inoculum concentration on prediction of both gelation time and cutting time. Cottage cheese-type gel. Int. Dairy J. **2006**, *16*, 147–152.
12. Payne, F.A.; Gates, R.S.; Fannin, B.L. Measurement precision for a fiber optic diffuse reflectance sensor. Trans. ASAE **1996**, *39*, 2193–2198.
13. International Dairy Federation, IDF Standard 110A: Appendix A, IDF, Brussels, 1987.
14. International Dairy Federation, IDF Standard 157A, IDF, Brussels, 1997.
15. Castillo, M.; Lucey, J.A.; Payne, F.A. The effect of temperature and inoculum concentration on rheological and light scatter properties of milk coagulated by a combination of bacterial fermentation and chymosin. Cottage cheese-type gels. Int. Dairy J. **2006**, *16*, 131–146.