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Development of a light scatter sensor technology for on-line monitoring of milk coagulation and whey separation

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Abstract

The objective of this study was to investigate a novel light backscatter sensor, with a large field of view relative to curd size, for continuous on-line monitoring of coagulation and syneresis to improve curd moisture content control. A three-level, central composite design was employed to study the effects of temperature, cutting time, and $CaCl_2$ addition on cheese making parameters. The sensor signal was recorded and analyzed. The light backscatter ratio followed a sigmoid increase during coagulation and decreased asymptotically after gel cutting. Curd yield and curd moisture content were predicted from the time to the maximum slope of the first derivative of the light backscatter ratio during coagulation and the decrease in the sensor response during syneresis. Whey fat was affected by coagulation kinetics and cutting time, suggesting curd rheological properties at cutting are dominant factors determining fat losses. The proposed technology shows potential for on-line monitoring of coagulation and syneresis.

Keywords: Sensor; Light backscatter; Coagulation; Syneresis; Moisture content control; Yield; Whey fat

1. Introduction

Cheese manufacture is of significant economic importance to the agricultural and food processing industries. According to the Food and Agricultural Organization of the United Nations, over 18 million metric tonnes of cheese were produced worldwide in 2004 (FAOSTAT, 2005). High curd moisture content can result in many cheese defects thereby resulting in decreased cheese grade, price and shelf-life. Limited information is available on the cost impact of the production of substandard quality cheese in industry. However Smukowski and Ping (2003), using data obtained from industry through a survey, estimated that US losses in 2001 from downgraded Cheddar and Swiss cheese were $29 and $24 million, respectively. These losses highlight the importance of improving process control during cheese manufacture and in particular during unit operations which exert a significant impact on cheese quality. Two such operations are formation of the milk gel (coagulation) and the expulsion of whey from the curd (syneresis) following cutting and stirring of the gel. The rate and extent of syneresis depend on a number of factors including coagulation conditions, the resulting gel properties and cutting conditions. The control of syneresis, including complementary whey drainage obtained through mechanical and physical actions, is a crucial step in cheese technology as it determines the dry matter content and composition of drained curd and consequently those of the final product (Daviau et al., 2000). Therefore the flow of whey from the gel must be controlled to obtain the final desired cheese moisture content.

The dual impact of both coagulation conditions and subsequent applied mechanical actions on syneresis shows that adequate control of syneresis also requires excellent control of coagulation. While there are a number of techniques

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available for on-line monitoring of milk coagulation (O'Callaghan, Mulholland, Duffy, O'Donnell, & Payne, 2001; O'Callaghan, O'Donnell, & Payne, 1999), currently there are no techniques available for monitoring syneresis.

Optical sensor technologies, based on either light backscatter or transmission, have been proven as a successful tool for monitoring milk coagulation (Castillo, Payne, Hicks, & Lopez, 2000; O'Callaghan et al., 1999; Payne, Hicks, Madangopal, & Shearer, 1993). In particular the optical fibre light backscatter sensor, CoAguLite™ is a well documented on-line sensor technology to monitor milk coagulation and predict both clotting and cutting times (Castillo, 2001; Payne, Hicks, & Sheng, 1993). The CoAguLite™ sensor was initially employed to monitor coagulation and syneresis. While the preliminary data indicated the potential of the technology for monitoring syneresis, the sensor output included a high degree of scatter after cutting. It was considered that this is most likely due to the presence of two phases once the gel is cut, i.e. the curd and the whey. It was hypothesized that a light backscatter sensor having a large field of view (LFV) relative to the curd size would accurately measure syneresis and might also have the potential to monitor coagulation.

Therefore a prototype large field of view backscatter sensor was built. The objective of this study was to evaluate the potential of the LFV sensor as an innovative technique for the on-line monitoring of coagulation and syneresis. Factors affecting the properties of the milk gel at cutting (temperature, $CaCl_2$ addition level, and cutting time) were varied in order to assess the sensor response. A response surface methodology was employed to evaluate the effect of the three experimental factors on major cheese making parameters factors, whey losses, curd moisture content and curd yield.

2. Materials and method

2.1. Experimental set-up

A LFV light backscatter sensor prototype was designed and installed on the wall of a 7-l cheese vat and tested during milk coagulation and syneresis. The optical fibre light backscatter sensor CoAguLite™ (Model 5, Reflectronics, Inc., Lexington, KY, USA) was also installed in the wall of the cheese vat at a 90° angle to the LFV sensor and used as a reference to which the readings of the LFV sensor were compared.

The CoAguLite™ sensor used near infrared light at 880 nm and consisted of two 600 μm diameter fibres. One fibre transmitted infrared radiation into the milk sample while the other fibre transmitted the scattered radiation from the particles present in the milk to a silicon photodetector. Further details on the CoAguLite™ sensor and data acquisition system were presented by Castillo et al. (2000). Response data were collected every 6 s. The initial voltage response (V_0) was calculated by averaging the first ten data points. A light backscatter ratio (R') was calculated by dividing the sensor output voltage by V_0. The first derivative (R') of the light backscatter ratio profile was calculated by conducting linear least-squares regression on the most recently collected 4 min of data. The calculated slope was assigned to the midpoint of the data subset used. The time to the first maximum of R' was defined as t_{max}. The CoAguLite™ sensor gave a real time value for the cutting time (t_{cut}) based on $t_{cut} = \beta^* t_{max}$. A number of different β values were used to obtain a range of cutting times for the experiment (Table 1).

The LFV sensor prototype was connected, as shown in Fig. 1, to the master unit of a miniature fibre optic spectrometer (model SD2000, Ocean Optics, Inc., Dunedin, FL, USA) and to a tungsten halogen light source (spectral range of 360 nm–2 μm) using fibre optic cables manufactured by the University of Kentucky and Reflectronics Inc. (KY, USA). Spectra were collected over the range 360–1100 nm with a resolution of 0.7 μm. The integration time was set to 7 s by the computer software (OOIBase, Version 1.5, Ocean Optics, Inc.). Each spectral scan was automatically processed by subtracting the dark spectral scan. Each spectral scan was reduced to 36 averages by dividing them into 20 nm wavebands with mid-wavelengths of $380 + 20 \cdot n$ $(1 \leqslant n \leqslant 36)$ and averaging the optical response for the wavelengths constituting each waveband. The 36 wavebands obtained were in the range (400–1100 nm). The voltage values for the first min of data were averaged within each waveband $V(w)$ to calculated a $V_0(w)$ value. The voltage output at every waveband, $V(w)$ was divided by its corresponding $V_0(w)$ to obtain the light backscatter ratio for the LFV sensor (R_{LFV}).

2.2. Experimental design

The suitability of the LFV sensor for simultaneous monitoring of milk coagulation and gel syneresis was studied using a three-factor, central composite rotatable design with two star points ($\alpha = 1.682$) and six replicates of the central point. The experimental design used, the factors selected as independent variables, and their ranges are shown in Table 1.

2.3. Milk preparation

Unpasteurized and unhomogenized milk was obtained from the University of Kentucky dairy herd. Milk was pas-

Table 1
The three-factor central composite rotatable design used to evaluate the response of the LFV sensor during cheese manufacture

Experimental factors	$-\alpha$	-1	0	1	α
Temperature (°C)	21.6	25.0	30.0	35.0	38.4
Added $CaCl_2$ (mg kg^{-1} milk)	72	106	156	206	240
Cutting time (β value; dimensionless)[a]	1.16	1.50	2.00	2.50	2.84

[a] Experimental cutting time levels were selected as β times t_{max}, where t_{max} was the time from enzyme addition to the inflection point of the light backscatter profile obtained using the CoAguLite sensor.

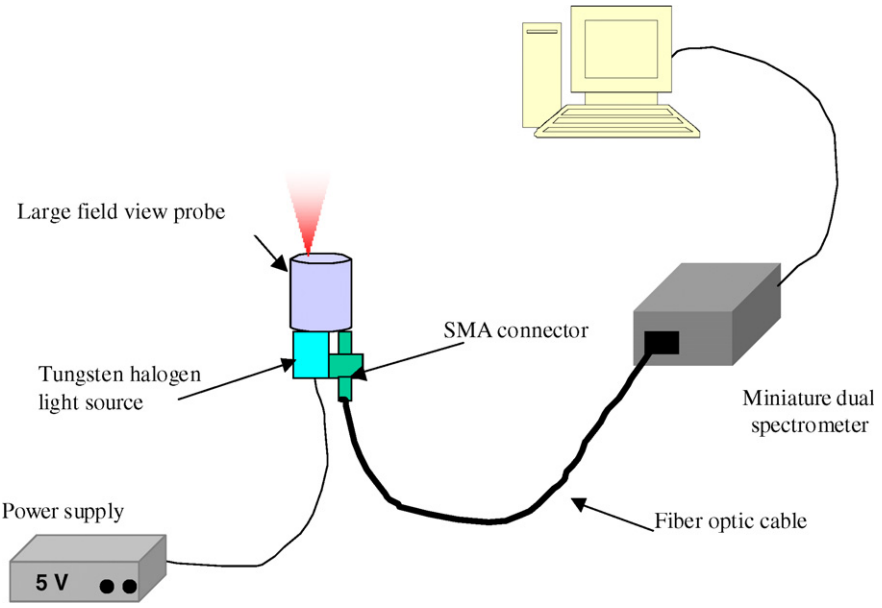

Fig. 1. Schematic of the large field view sensor configuration.

teurized at 65 °C for 30 min using a water bath (Lauda, RM 20, Brinkman Instrument Inc., Westbury, NY, USA) having a control accuracy of ±0.01 °C. Milk temperature was monitored using a precision thermistor (model 5831 A, Omega Engineering, Stanford, CT, resolution ±0.01 °C; accuracy ±0.2 °C). After pasteurization, milk was quickly cooled to 2 °C using another water bath where the water was substituted by a coolant and temperature was set several degrees below 0 °C to increase the refrigeration efficiency. For each experiment 7.2 kg of cooled pasteurized milk was weighed and $CaCl_2$ at the required level was added and the milk was stirred for 3 min. The milk was left to equilibrate for 30 min in a cold room at 2 °C. Milk was adjusted in the cold room to a pH of ~6.51 using an experimentally obtained linear regression between volume of 1.0 M HCl added and milk pH to determine the volume of acid to add. The milk was stored in the cold room overnight. Milk pH adjustment after $CaCl_2$ addition ensured that any observed effect of independent variables on dependent variables was not due to an indirect effect of $CaCl_2$ on milk pH.

2.4. Coagulation and syneresis procedure

On the day of experimentation the milk was adjusted to a final pH of 6.5 at 2 °C using 1.0 M HCl. A constant dilution rate was assured by adding de-ionised water for a total added volume of 60 ml. Milk was then slowly heated to the coagulation temperature ±0.15 °C using a Lauda, RM 20 water bath, to minimize the impact of the temperature change on casein micelle equilibrium. The temperature of the milk was monitored using a precision thermistor. Seven kg of the heated milk were added to the vat and left to equilibrate for at least 15 min until thermal equilibrium was achieved. Coagulation temperature was controlled using a single jacket cheese vat supplied with temperature controlled water through a copper-coil connected to a Lauda, RM 20 water bath having a control accuracy of ±0.01 °C. Milk temperature was measured with a precision thermistor (model 5831 A, Omega Engineering, Stanford, CT, resolution ±0.01 °C; accuracy ±0.2 °C). Chymosin (Chymax, Chr. Hansen Inc., Milwaukee, WI, USA) was added to the milk in the vat at a level of 0.06 ml kg^{-1} milk and stirred for 1 min. Upon addition of the rennet data acquisition for both sensors was started. The CoAguLite™ sensor was used to predict the cutting time as described above. When indicated by the CoAguLite™ data acquisition software the gel was cut using a manual cutting system. Cutting time was defined as $t = 0$ for the syneresis process. The coagulum cutting knife is shown in Fig. 2. The knife was pushed through the gel and rotated once ensuring all the gel was cut and then removed. As the knife was pushed vertically through the gel, the gel was cut into prismatic columns. Rotating the knife cut these columns into cubes of approximately 1 cm. The curd was left to heal for 5 min before stirring at 10 rpm was initiated (Servodyne mixer, model 50003-10, Cole Parmer Instrument Co., IL, USA). The stirring process continued at this speed for 85 min. At the end of the stirring process i.e. $t = 85$ min, homogeneous samples of curd and whey (~150 ml) were removed for compositional analysis.

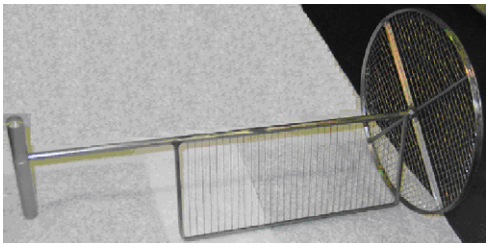

Fig. 2. The coagulum cutting knife.

2.5. Compositional analysis of milk, curd and whey and calculation of curd yield

Two drops of Bronolab-W (D&F Control Systems, Dublin, CA, USA), a preservative, was added to a 40 ml sample of milk from each experiment. Samples were stored at 2 °C for up to 7 days prior to analysis for fat, protein and total solids content using a MilkoScan FT 120 (Foss Electric, Denmark). The average and standard deviation for fat, protein and total solids content of the milk used in this study was found to be 3.6 ± 1, 3.3 ± 0.3, and 12.2 ± 1 (% w/w), respectively. The curd and whey samples removed from the vat at $t = 85$ min were first separated by passing each sample through a stainless steel sieve with a 75 μm absolute pore size. The sieve characteristics were selected to ensure that, whey fat globules were not retained by the sieve. Approximately 3 g of curd and 5 g of whey were then accurately weighed into pre-weighed aluminum dishes. The dishes were dried in a convection oven at 102 °C, until they reached a constant weight. Each sample was analyzed in triplicate. Chemical composition of whey (fat, protein and total solids content) were also determined using the MilkoScan, which was calibrated using 10 certified raw bovine whey samples supplied by DQCI Services, (Mounds View, MN, USA). Each filtered whey sample was analyzed in triplicate using the MilkoScan. Cheese yield was calculated for each experiment using a mass balance procedure. The weight of curd was expressed as percentage of the initial weight of milk used.

3. Results and discussion

3.1. Sensor response to coagulation and syneresis

Fig. 3 illustrates the typical light backscatter profiles derived from the LFV sensor at 26 wavelengths between 500 and 1000 nm during coagulation, and compares the LFV profiles with the corresponding CoAguLite™ sensor profile. As observed in Fig. 3, the LFV sensor response is less smooth. However the LFV sensor did respond in a similar manner to the CoAguLite™ sensor indicating that the LFV sensor is sensitive to chemical reactions involved in milk coagulation. The average maximum increase of the LFV signal during coagulation, which occurred at 980 nm, was $23.5 \pm 5.4\%$. This was also smaller than that of the CoAguLite™ sensor ($34.9 \pm 8.2\%$). The response of the CoAguLite™ sensor (880 nm) and the LFV sensor (940, 960 and 980 nm) during coagulation and syneresis is shown in Fig. 4.

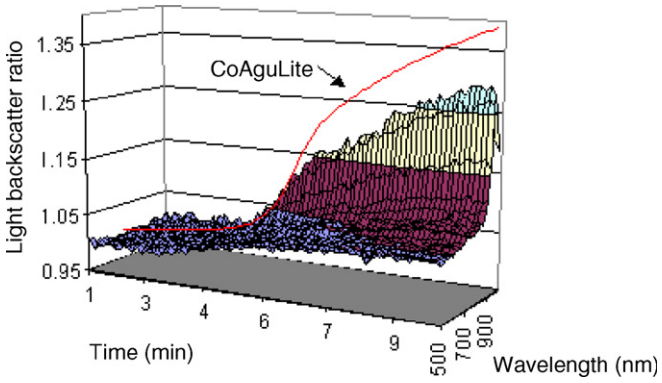

Fig. 3. Typical large field view (LFV) sensor response during coagulation (temperature = 38.4 °C; $CaCl_2 = 156$ mg kg^{-1}) at wavelengths between 500 and 1000 nm and the simultaneous CoAguLite™ response.

As was expected, the CoAguLite™ sensor displayed a high degree of scatter once the gel was cut. However, the LFV sensor displayed a significantly smoother decrease in the light backscatter ratio upon cutting, especially at 980 nm. Between cutting time and 85 min after cutting, the signal decreased by 25–61% at 980 nm. The response of the LFV sensor to varying experimental conditions during coagulation and syneresis was also investigated. No significant effect of β value on the LFV sensor response was observed during syneresis. Increasing the level of $CaCl_2$

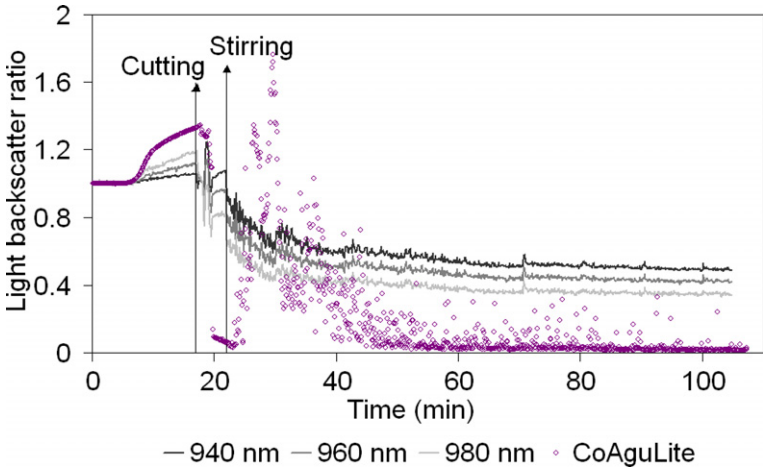

Fig. 4. Typical sensor profiles of the large field view (LFV) sensor at 940, 960 and 980 nm and the CoAguLite™ sensor during coagulation and syneresis (temperature = 30 °C; $CaCl_2 = 156$ mg kg^{-1}).

added was not observed to significantly affect the sensor response during either coagulation or syneresis. However increasing the temperature was found to significantly impact the sensor response during both steps. During coagulation, increasing the temperature resulted in an increase in the light backscatter ratio value at the cutting time (Fig. 5a–c). It was also observed that the reduction in sensor response during syneresis was amplified with increasing temperatures (Fig. 5a–c). Since temperature is one of the most important factors affecting curd syneresis, it is suggested that the LFV signal would be a function of syneresis kinetics.

3.2. Prediction of whey fat, curd moisture and yield

Fat losses in the whey, curd moisture content and curd yield are important cheese making parameters that significantly impact on final product quality. The best two-,

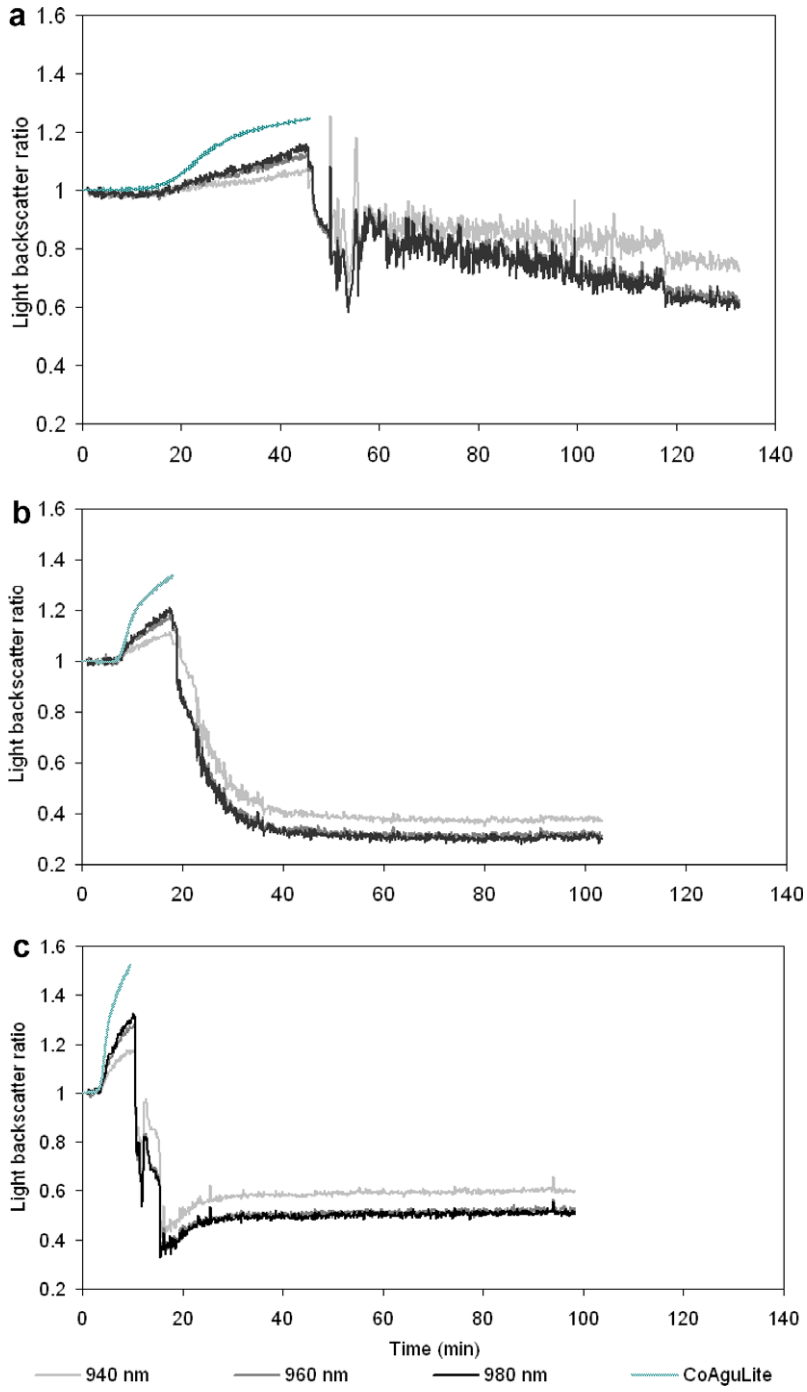

Fig. 5. CoAguLite™ and large field view sensor profiles under constant β and $CaCl_2$ levels ($\beta = 2$, $CaCl_2 = 156$ mg kg^{-1}) and increasing temperature (a) 21.6 °C (b) 30.0 °C and (c) 38.4 °C.

and three-parameter prediction models for whey fat content, curd moisture content and curd yield measured at 85 min from cutting time were obtained using the "Maximum R^2" procedure of SAS (SAS, version 8.00, 1999, SAS Institute Inc., Cary, NC), including the independent variables (β, temperature, and added $CaCl_2$ level) and several light backscatter parameters (t_{max}, and DSS; see definition below) derived from the LFV sensor signal at 980 nm.

The equations developed for predicting whey fat content (WFC), curd moisture content (CMC) and curd yield (CY) are given in Eqs. (1)–(3) as follows:

$$\mathrm{WFC} = 1.266 * \beta + 0.219 * t_{max} \quad (1)$$

$$\mathrm{CMC} = 2.757 * t_{max} + 0.539 * \mathrm{DSS} \quad (2)$$

$$\mathrm{CY} = 0.026 * C + 1.221 * t_{max} + 0.083 * \mathrm{DSS} \quad (3)$$

where t_{max} was the time from enzyme addition to the inflection point of the LFV light backscatter ratio (typically occurred before cutting), DSS was the decrease in the LFV signal during syneresis and C was the level of $CaCl_2$ added. It is of interest to note the variables, which were significant in predicting WFC, CMC, and CY. As expected, the coagulation variable t_{max} was a significant factor for the three cheese making parameters studied. This was attributed to the significant effect of milk coagulation reactions and their kinetics on the physical properties of the milk gels and, subsequently, on curd drainage. This was in agreement with Castillo, Lucey, and Payne (2006). These authors found that the impact of light backscatter parameters obtained during coagulation on the syneresis kinetics was very significant. Another important LFV parameter was DSS, which was significant for the prediction of CMC and CY (Eqs. (2) and (3)). This strongly suggested that the decrease observed in the signal after cutting was proportional to the extent and/or kinetics of curd syneresis. The third important variable identified was cutting time (β), which was significant for the prediction of WFC (Eq. (1)). Eq. (1) suggested that the rate of milk coagulation and the rheological properties of the gel at cutting are the dominant factors in determining whey fat losses during cheese manufacture. Finally, increasing the level of $CaCl_2$ added was expected to reduce t_{max} due to its direct effect on aggregation and firming rates (Castillo, Payne, Hicks, Laencina, & Lopez, 2002). This effect accounts for the inclusion of the added $CaCl_2$ term in the CY prediction equation (Eq. (3)). The prediction results are shown in Fig. 6. These results are of a preliminary nature as a consequence of the small data set. Although the predictions obtained for WFC and CMC have low R^2 values of 0.35 and 0.34 respectively the CY prediction was strong ($R^2 = 0.75$). The results demonstrate the potential viability of the LFV sensor for predicting cheese making parameters and in particular they indicate a basis for the continued development of a LFV light backscatter sensor as an innovative technique for the on-line monitoring of milk coagulation and syneresis using only one sensor.

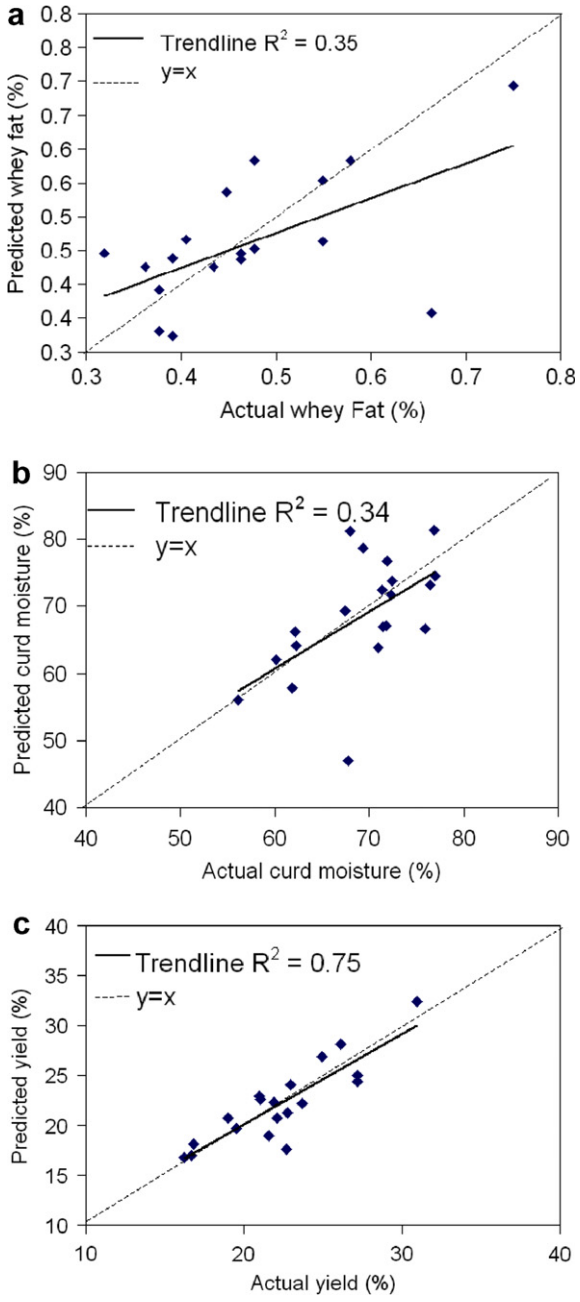

Fig. 6. Prediction results for (a) whey fat content, (b) curd moisture content and (c) curd yield using Eqs. (1)–(3), respectively.

4. Conclusions

The objective of this study was to investigate the potential application of a novel optical sensor technology to the continuous monitoring of coagulation and syneresis to improve curd moisture content control. A LFV sensor was developed and assessed using varied rates of coagulation and a range of cutting times. The LFV sensor responded in a similar manner to the CoAguLite™ sensor during milk coagulation and with a lower degree of scatter than the CoAguLite™ during syneresis. The LFV sensor signal increased sigmoidally during coagulation by an average of 23.5%. After cutting, the

signal of the LFV sensor at 980 nm was found to decrease exponentially by between 25% and 61%. It was also observed that this decreasing response during syneresis increased with increasing temperatures, suggesting that the LFV sensor might be sensitive to the changes in syneresis kinetics with temperature. Equations were developed for the prediction of the whey fat losses, the curd moisture content and yield. It was found that rheological properties of the curd at cutting are dominant factors influencing whey fat losses. Curd moisture content and yield were both predicted using one LFV parameter representing milk coagulation kinetics (t_{max}) and a second LFV parameter representing the syneresis kinetics (DSS). The results of this study suggest that with further investigation, the LFV sensor may have potential application in the on-line monitoring of both coagulation and syneresis. This could result in improved process control during cheese manufacture.

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References

Castillo, M. (2001). Cutting time prediction in cheese making by backscatter of near infrared radiation. PhD thesis. University of Murcia, Spain. Department of Food Technology.

Castillo, M., Lucey, J. A., & Payne, F. A. (2006). 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. *International Dairy Journal, 16*, 131–146.

Castillo, M., Payne, F. A., Hicks, C. L., Laencina, J., & Lopez, M. B. (2002). Effect of calcium and enzyme in cutting time prediction of coagulating goats' milk using a light scattering sensor. *International Dairy Journal, 12*, 1019–1023.

Castillo, M., Payne, F. A., Hicks, C. L., & Lopez, M. B. (2000). Predicting cutting and clotting time of coagulating goat's milk using diffuse reflectance: effect of pH, temperature and enzyme concentration. *International Dairy Journal, 10*, 551–562.

Daviau, C., Pierre, A., Famelart, M.-H., Goudedranche, H., Jacob, D., Garnier, M., et al. (2000). Characterization of whey drainage kinetics during soft cheese manufacture in relation with the physicochemical and technological factors, pH at renneting, casein concentration and ionic strength of milk. *Lait, 80*, 417–432.

FAOSTAT (2005). Food and Agricultural Organization of the United Nations Statistical Databases, Agricultural Production – Livestock Processed – World Cheese Production. <http://faostat.fao.org/faostat/> Accessed 09.01.05.

O'Callaghan, D. J., Mulholland, E. P., Duffy, A. P., O'Donnell, C. P., & Payne, F. A. (2001). Evaluation of hot wire and optical sensors for on-line monitoring of curd firmness during milk coagulation. *Irish Journal of Agricultural and Food Research, 40*, 227–238.

O'Callaghan, D. J., O'Donnell, C. P., & Payne, F. A. (1999). A comparison of on-line techniques for determination of curd setting under different rates of coagulation. *Journal of Food Engineering, 41*, 43–54.

Payne, F. A., Hicks, C. L., Madangopal, S., & Shearer, S. A. (1993). Fibre optic sensor for predicting the cutting time of coagulating milk for cheese production. *Transactions of the ASAE, 36*, 841–847.

Payne, F. A., Hicks, C. L., & Sheng, P. (1993). The near-infrared optic probe for monitoring rennet coagulation in cow's milk. *Journal of Dairy Science, 76*, 48–61.

SAS (1999). *SAS users guide: statistics. Ver. 8*. Cary, NC: SAS Institute, Inc.

Smukowski, M., & Ping, Y. (2003). Cheese defects in U.S. graded cheese. *Dairy Pipeline. A Technical Source for Dairy Manufacturers, 15*, 1.