

# Visible-near infrared spectroscopy sensor for predicting curd and whey composition during cheese processing

Colette C. Fagan · Manuel Castillo ·  
Donal J. O'Callaghan · Fred A. Payne ·  
Colm P. O'Donnell

Received: 8 October 2008 / Accepted: 26 January 2009 / Published online: 13 February 2009  
© Springer Science+Business Media, LLC 2009

**Abstract** The potential of visible-near infrared spectra, obtained using a light backscatter sensor, in conjunction with chemometrics, to predict curd moisture and whey fat content in a cheese vat was examined. A three-factor (renneting temperature, calcium chloride, cutting time), central composite design was carried out in triplicate. Spectra (300–1,100 nm) of the product in the cheese vat were captured during syneresis using a prototype light backscatter sensor. Stirring followed upon cutting the gel, and samples of curd and whey were removed at 10 min intervals and analyzed for curd moisture and whey fat content. Spectral data were used to develop models for predicting curd moisture and whey fat contents using partial least squares regression. Subjecting the spectral data set to Jack-knifing improved the accuracy of the models. The whey fat models ( $R = 0.91, 0.95$ ) and curd moisture model ( $R = 0.86, 0.89$ ) provided good and approximate predictions, respectively. Visible-near infrared spectroscopy was found to have potential for the prediction of important syneresis indices in stirred cheese vats.

**Keywords** Visible-near infrared spectroscopy · Chemometrics · Syneresis · Curd moisture · Whey fat · Fibre optic · Optical sensor · On-line

## Introduction

While greater automation of cheese manufacture is desirable to the processor, there is a lack of on-line techniques available for monitoring the cheese making process. Syneresis is a critical step in cheese making with the rate and extent of syneresis having a significant effect on final cheese quality. In fact, unsuitable curd moisture content negatively impacts cheese ripening [1] and yield [2]. Emmons [3] stated that a decrease in moisture content by as little as 1% translates into an important reduction in cheese yield and profits. Although monitoring and controlling syneresis is important, this process is still empirically controlled. The development of a monitoring and control technology for syneresis is of significant importance in moving towards fuller automation of the cheese making process.

Recently, a few studies have investigated technologies with potential for syneresis monitoring and control [4–8]. Taifi et al. [5] studied the potential of ultrasonic velocity and attenuation to determine the onset of syneresis in a milk gel. They stated that velocity evolution obtained at high frequency appeared to be an indicator of the occurrence of syneresis and that syneresis was also characterized by a large increase in attenuation. Everard et al. [6] monitored syneresis in a stirred cheese vat by following colour changes occurring in the vat using a computer vision system. They found that the technique was capable of distinguishing the effects of pH and stirring speed and that, with the inclusion of known factors and calibration to a

---

C. C. Fagan (✉) · C. P. O'Donnell  
Biosystems Engineering, School of Agriculture, Food Science  
and Veterinary Medicine, University College Dublin, Belfield,  
Dublin 4, Ireland  
e-mail: colette.fagan@ucd.ie

D. J. O'Callaghan  
Moorepark Food Research Centre, Teagasc, Fermoy, Co. Cork,  
Ireland

M. Castillo · F. A. Payne  
Department of Biosystems and Agricultural Engineering,  
University of Kentucky, 128 C. E. Barnhart Building, Lexington,  
KY 40546-0276, USA

range of operating conditions, there is potential for predicting an endpoint of syneresis. However, a limitation of the technique was that images were only obtained of the surface of the curd/whey mixture, where stirring speed significantly affects the level of curd particles present.

Castillo et al. [4] and Fagan et al. [7] investigated the potential of backscatter of near-infrared light at 980 nm to monitor syneresis. They found that a sensor detecting near-infrared light backscatter in a cheese vat and with a large field of view (LFV) relative to curd particle size had potential for monitoring both milk coagulation and curd syneresis. Further, Fagan et al. [7] was able to predict whey fat content, curd yield, and curd moisture content using the LFV syneresis sensor technology. However, the preliminary predictions presented in that study were limited in their use due to the small data set used and in consequence the reduced strength of predictions found. Fagan et al. [8] expanded this research in order to determine if the LFV sensor could be used to predict important cheese making indices. They developed equations to predict curd moisture content and whey fat at the end of syneresis as well as curd moisture content as a function of processing time during syneresis. They stated that the LFV sensor technology was able to detect the rate and extent of syneresis as well as whey losses and that this technology could provide greater consistency and efficiency during cheese production over a wide range of moisture content.

Although models have been successfully developed for predicting cheese making indices using light backscatter at 980 nm, we hypothesize that a wide spectral range would improve the models as other near-infrared regions and the visible spectrum may contain additional information related to the state of the intermediate product in the cheese vat. Castillo et al. [9] utilised NIR transmission and side-scatter spectra of whey to predict whey fat content. They found that light extinction coefficients for whey fat were maximised in the region of 830 nm and minimised around 425 nm. A model using a waveband ratio, with a denominator in the range of 375–475 nm and a numerator waveband in the region of 725–1,025 nm had the highest  $R^2$ . However, an infrared spectrum frequently contains regions of overlapping information which are difficult to interpret. Chemometrics uses mathematical and statistical methods in order to resolve the issue of overlapping peaks thereby extracting the most useful information from the spectra. Loadings plots for example can be useful for separating overlapped bands and for making band assignments. Therefore applying this methodology will assist in the identification of regions of the spectra which have the greatest potential for monitoring syneresis. This method of utilising chemometrics to extract valuable quantitative information from samples through NIR spectral data has

been widely employed in both the pharmaceutical [10] and food production [11, 12] fields.

Two of the most widely used chemometric methods are partial least squares (PLS) regression and principal component analysis (PCA). PLS regression and PCA may be used to “compress” spectral data to a few linear combinations of the original data. The new smaller set of variables, called PLS loadings (L) or principal components (PC), quantifies the subtle changes of the spectra from one sample to another. Thus it is possible to describe a large proportion of the variability in the data with a much smaller number of variables. These variables can then be used to develop equations for the prediction of various attributes.

Accordingly, the objectives of this study were to (a) analyze, using chemometrics, the visible-near infrared spectra obtained during syneresis with a light backscatter sensor in order to determine which regions of the spectra may be most useful for monitoring syneresis and, (b) investigate the potential of visible-near infrared spectra in conjunction with chemometrics to predict curd moisture and whey fat contents in a stirred cheese vat. Successfully employing visible-near infrared spectroscopy in conjunction with chemometrics to predict important cheese making indices during syneresis could potentially improve overall process control during cheese making and in particular could improve the control of curd moisture content and whey losses.

## Materials and methods

### Experimental design

A three-factor, fully randomized, spherical, central composite design (CCD), was employed to provide a broad range of coagulation and syneresis rates in order to assess the strength of the syneresis index predictions. The CCD consisted of a  $2k$  factorial ( $k = 3$ ) with  $2k$  axial points and six centre points (i.e., 20 runs in total) and was carried out in triplicate. The three factors selected as independent variables were coagulation temperature (T), calcium chloride ( $\text{CaCl}_2$ ) addition level (CCAL) and cutting time ( $t_{\text{cut}}$ ). The experimental factors, their selected levels and scaled values are presented in Table 1.

Inline, continuous monitoring of milk coagulation and curd syneresis in a seven-litre cheese vat was performed using a Large Field of View (LFV) sensor developed by Castillo et al. [4]. Light backscatter response from the sensor was continuously monitored from the time of rennet addition to the end of syneresis, i.e.,  $t = 85$  min. Experimental cutting time levels were selected by a commercially

**Table 1** The experimental factors and levels employed in the central composite rotatable experimental design

Scaled value of factor	Temperature (°C)	Added CaCl <sub>2</sub> (mM)	Cutting time ( $\beta$ ) (dimensionless) <sup>a</sup>
-1.682	23.6	0.318	1.32
-1	27.0	1.00	1.80
0	32.0	2.00	2.50
1	37.0	3.00	3.20
1.682	40.4	3.68	3.68

<sup>a</sup> Experimental cutting time levels were selected as  $\beta \cdot t_{\max}$ , where  $t_{\max}$  was the time from enzyme addition to the inflection point of the light backscatter profile obtained using the CoAguLite™ sensor

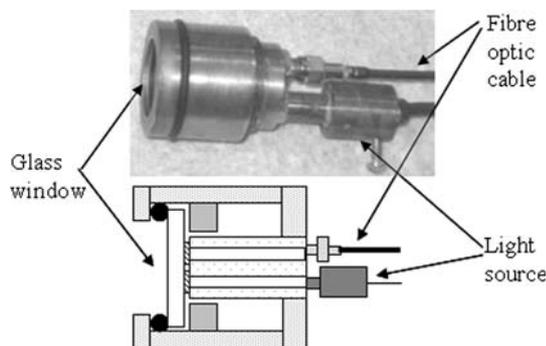
available cutting time prediction sensor technology, the CoAguLite (CL), as described below.

### Milk preparation and coagulation procedure

Unpasteurized and unhomogenized milk was obtained from a local milk processing plant (Winchester Farms Dairy, Winchester, KY, USA). Milk was prepared and coagulated according to the procedure employed by Fagan et al. [13]. Coagulation temperature was controlled using a cheese vat connected to a water bath (Lauda, RM 20, Brinkman Instrument Inc., Westbury, NY, USA). The enzyme used for milk coagulation was chymosin at a concentration of 0.06 ml kg<sup>-1</sup> (CHY-MAX® Extra; Chr. Hansen Inc., Milwaukee, WI, USA). Data acquisition for the CL and LFV sensors commenced upon addition of the enzyme.

### The large field of view sensor

The LFV sensor (Fig. 1) was proposed and designed at the University of Kentucky by Castillo et al. [4]. Light from a tungsten halogen light source (spectral range of 360 nm to 2  $\mu$ m) travels through a quartz rod, a vertical polarizer, and a glass window to the sample. Backscattered light is

**Fig. 1** Image and schematic of the large field view sensor employed for on-line monitoring milk coagulation and curd syneresis

collected from a large area through the glass window. A second polarizing plate orientated at 90° allows for the exclusive detection of horizontally polarized light. Light is conducted through another quartz rod, a SMA connector and a ~800  $\mu$ m diameter fibre optic cable (Spectran Specialty Optics, Avon, CN, USA) to the master unit of a miniature fibre optic spectrometer (model SD2000, Ocean Optics, Inc., Dunedin, FL, USA). Light emerging from the fibre optic cable is processed in the spectrometer and the data transferred to a computer through an A/D converter as described by Fagan [13]. Spectra were collected over the range 300–1,100 nm with a resolution of 0.7 nm.

### Cutting time selection and gel cutting procedure

The CoAguLite (CL) sensor (Model 5, Reflectronics Inc., Lexington, KY, USA) was employed to select the different experimental levels of cutting time as detailed by Fagan et al. [2]. When indicated by the CL data acquisition software the gel was cut into cubes of approximately 1 cm<sup>3</sup> using a manual cutting system. The start of the syneresis process was taken as time zero. The curd was left to heal for 4.5 min before stirring at 10  $\pm$  0.02 rpm was initiated (Servodyne mixer 50003-10, Cole Parmer Instrument Co., IL, USA). The stirring process continued at this speed up to 85 min after cutting.

### Curd and whey sampling procedure

Homogeneous samples of curd and whey (~150 ml) were removed from the vat for compositional analysis at 5 min after cutting and every 10 min thereafter up to 85 min after cutting (i.e., 9 samples). The samples were immediately poured into a stainless steel standard test sieve (Fisher Scientific, NH, USA) with a 75  $\mu$ m absolute pore size in order to separate the curd and whey. The sieve characteristics were selected to ensure that whey fat globules were not retained by the sieve.

### Compositional analysis of curd and whey

Approximately 3 g of curd and 5 g of whey were weighed into pre-weighed aluminium dishes using an analytical balance and then dried in a convection oven at 102°C for 15 h. Analysis was carried out in triplicate at each sampling time point. Chemical composition of whey (fat, protein and total solids content) were also determined using the MilkoScan FT120 (Foss, Hillerød, Denmark), which was calibrated using 10 certified raw bovine whey samples supplied by DQCI Services, (Mounds View, MN, USA). Filtered whey samples of 40 ml, to which two drops of preservative (Bromolab-W) were added, were stored for this purpose at 2°C for up to 7 days prior to analysis. Each

filtered whey sample was analyzed in triplicate using the MilkoScan.

### Chemometrics

In order to normalise the spectra recorded during syneresis, the spectra recorded at the cutting time were subtracted from each subsequent spectra. PCA and PLS regression were carried out using The Unscrambler software (v.9.6; Camo A/S, Oslo, Norway). Models for the prediction of syneresis indices were developed using PLS regression and confirmed by cross-validation.

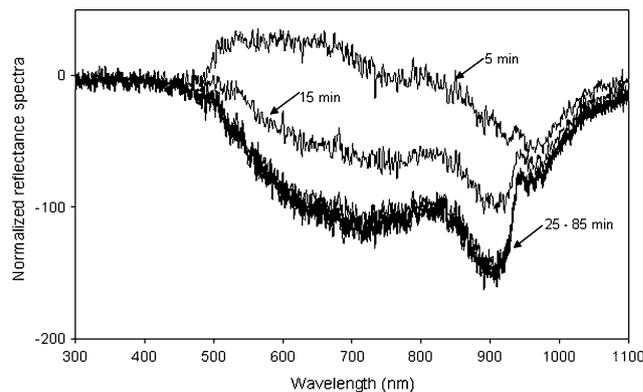
Prior to PLS regression, spectra at each sampling point ( $n = 540$ ) were pre-treated using standard normal variate (SNV) [14], smoothing, 1st and 2nd derivative using the Savitzky–Golay algorithm [15] (9 data point each side) and each derivative plus SNV. The potential of the models to predict the syneresis indices was evaluated using the root mean square error of cross-validation (RMSECV), correlation coefficient ( $R$ ) and the number of PLS loadings (#L). The range error ratio (RER), calculated by dividing the range in the reference data of a given attribute by the prediction error for that attribute, was used to determine the practical utility of the models [16].

Each of the developed PLS models was subjected to the modified Jack-knifing method developed by Martens and Martens [17]. This test was used to avoid misinterpreting spurious effects, to identify the dominating sources of instability in the modelling and to allow more or less automatic optimisation of the models [17]. It predicts the most important wavelengths for model development and once they were identified a refined model was developed using only those selected wavelengths.

## Results and discussion

### Spectral response

Figure 2, showing typical normalized (subtracted) spectra recorded during syneresis at successive sampling time points illustrates the regions of the spectra which changed most during syneresis with respect to the spectral response at the cutting time. The amount of back scattered light recorded generally decreased after cutting, although a slight increase was observed between 500 and 700 nm for the 5 min spectrum. Over all the experiments there was a high level of variability between 5 min spectra. This was attributed to the significant amount of scatter in the signal which was initiated by the cutting process and lasted for approximately 6 min. During this period the gel was cut, left to heal, and stirring blades were inserted into the vat with stirring starting at 5 min.



**Fig. 2** Typical normalized visible-near infrared spectra obtained during syneresis at 32°C at 10 min intervals between 5 and 85 min after cutting by the prototype sensor installed in the cheese vat. Spectra were normalised by subtracting the spectra recorded at the cutting time from each subsequent spectra

The observed spectral response obtained from the LFV sensor is due to a number of effects including scattering, reflection and absorption [18]. Rayleigh's theory describes scatter by particles which are much smaller than the wavelength of light, while diffuse reflectance occurs where light is reflected from a particles surface which is greater than the wavelength of light impinging on it. The particles primarily responsible for scattering in milk are fat globules and casein micelles [19]. During syneresis in a cheese vat, curd particles (aggregate of micelles) will be responsible for diffuse reflection and whey fat globules, which typically have diameters less than 10  $\mu\text{m}$ , are predominately responsible for light scattering. These properties are dependent on the difference in the refraction index of the particles and the medium in which these particles are dispersed, as well as on the ratio of the particles radius to the wavelength of the light impinging on them. During syneresis the difference in the refraction index, the number of fat globules, as well as the number and size of the curd particles vary. Hence changes in scattering and diffuse reflection will provide valuable information which could be related to the syneresis process.

The LFV spectral response may not only reflect scattered or reflected light, but also fluorescence, due to the fluorophores naturally present in milk. Such fluorophores include aromatic amino acids, e.g., tryptophan, vitamins A and B<sub>2</sub>, oxidation products, and Maillard products. Chlorophyllic compounds have also been identified in dairy products using fluorescence emission spectra [20].

Typically curd particles contract, whey volume increases, and whey fat concentration decreases during syneresis. All of these processes could be the cause of the reduction in light measurement observed in Fig. 2. It should be noted that the spectra were not observed to decrease after 5 min with experiments carried out at 40.4°C; such spectra had

the lowest reflectance values. It was shown by Fagan et al. [13] that at 40.4°C whey fat concentration increased during syneresis and that this increase in whey fat globules resulted in an increase in light backscatter. This increase in whey fat concentration during syneresis at 40.4°C is due to higher mobility of fat globules at this temperature as they have reached their general melting point of 37°C, allowing them to be expelled throughout syneresis and not just upon cutting of the gel.

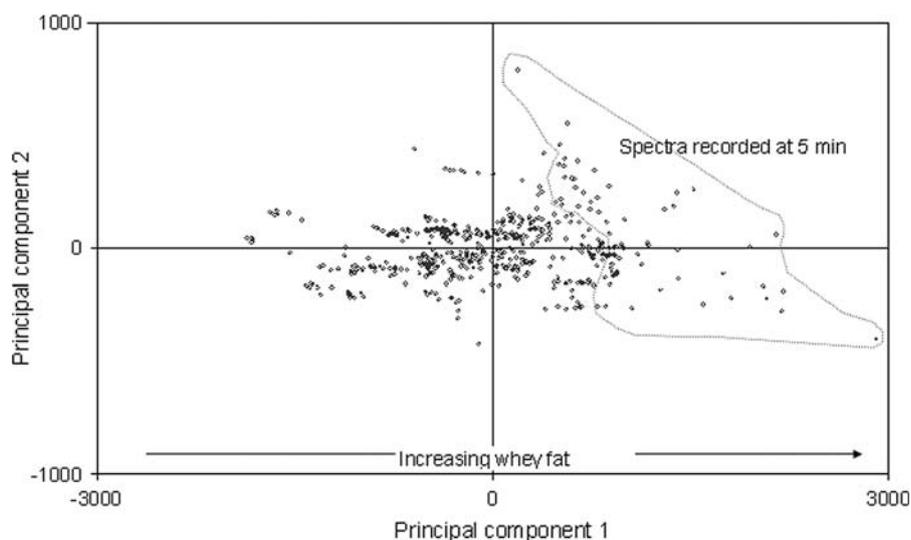
Figure 2 shows that the greatest changes in light measurement occurred between 550 and 996 nm, with wavelengths in the region of 690–766 nm and 874–931 nm showing the greatest change during syneresis. Determining why the light measured at these wavelengths diminished most during syneresis is complex as the physical state of many components in the vat change during syneresis and hence multiple factors may be influencing the sensor response at a particular wavelength. Fluorescent compounds such as tryptophan, vitamin A, vitamin B<sub>2</sub>, and chlorophyll A have maximum emission wavelengths of 357, 480, 518, and 663 nm, respectively, while absorbance at 970 nm has been previously assigned to a second overtone of the O–H stretching vibration of water [21–23]. While the space-averaged concentration of the constituents in the cheese vat cannot vary during syneresis, their spatial distribution will. Water and dissolved solutes will be expelled from the curd particle protein matrix to the surrounding environment, resulting in a concurrent reduction in particle size and increase in the fraction of free water in the system, all of which alter light scattering and absorbance in the vat. The fluorescence intensity recorded may also vary. For example vitamin B<sub>2</sub> is soluble in water and hence its concentration in whey should remain reasonably constant during syneresis. However its fluorescence intensity may vary during syneresis if the light extinction

coefficient of fluorescence light emitted by this vitamin in whey is smaller than in milk or in curd.

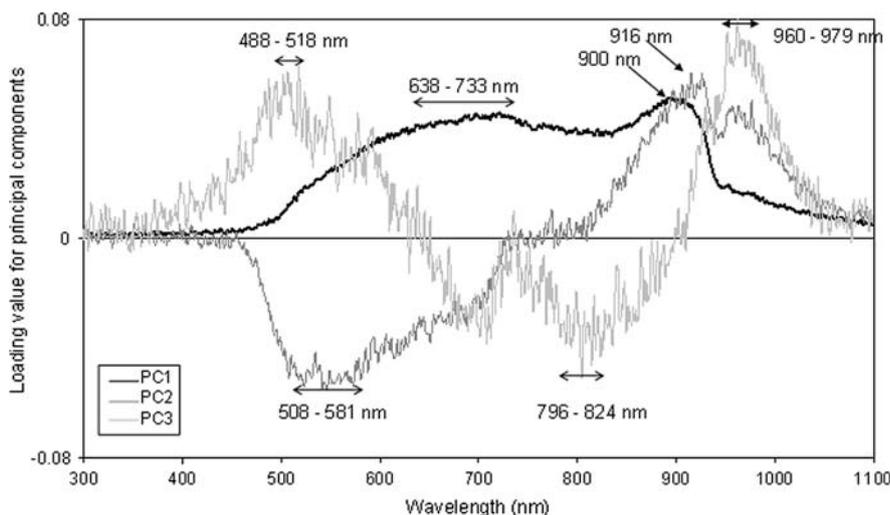
### Principal component analysis

The spectra at each sampling point were subjected to principal component analysis to detect sample patterns and groupings. The first three principal components (PC) explained 98% of the variation in the spectral database, with PC1, PC2 and PC3 accounting for 93, 4 and 1%, respectively. Figure 3 shows the score plot of PC1 and PC2 obtained from PCA. It was noted that the majority of spectra recorded at 5 min tended to be grouped towards the positive end of PC1. This further suggests that, as was indicated in Fig. 2, there are significant differences between spectra obtained in the early stages of syneresis and those obtained later in the process. Samples located at the positive extreme of PC1 also tended to have high curd moisture contents (>72% w/w). PC1 also discriminated between samples on the basis of whey fat content with a general trend of increasing whey fat content along PC1. In order to determine the regions of the spectra which were most important in discriminating between samples the loadings for the first three principle components were plotted (Fig. 4). Local maxima and minima were identified in the regions 488–581 nm, 638–733 nm, 796–824 nm, 900 nm, 916 nm and 960–979 nm, confirming that there are important regions in both the visible and near infrared spectra for discriminating between samples. Changes in the level of absorption of light by the sample during syneresis, which may impact the backscattered light, can be attributed to chemical bonds. Absorption at 840 nm is due to a combination of C–H vibrations and can be ascribed to any of the main milk components while absorption at 906 nm arises from a third overtone of C–H stretching modes of

**Fig. 3** PCA scores bi-plot of all visible-near infrared spectra recorded at all sampling time points during syneresis from all trials. PC1 and PC2 account for 93% and 4% of the variation in the data, respectively



**Fig. 4** Loadings plot for principal components 1, 2 and 3 for all subtracted visible-near infrared spectra from all trials recorded at all sampling time points during syneresis



proteins [24]. O–H stretching can account for absorption at 916 nm [25] and absorption at 966 nm probably reflects the interaction between proteins and water [24]. The absorption of light at 970 nm is due to  $2\nu_1 + \nu_3$  ( $\nu_1$ : symmetric stretching,  $\nu_3$ : antisymmetric stretching) vibration of water.

Prediction of whey fat and curd moisture content

Models were developed using spectra in a number of forms: raw, SNV, smoothed, 1st derivative, 2nd derivative and SNV plus each derivative step, giving seven models each for whey fat content and curd moisture content. However, none of the spectral pre-treatments offered an improvement in model accuracy for either parameter; hence those prediction results are not shown. The models developed using the untreated spectra were then subjected to Jack-knifing as a means of reducing the number of wavelengths to only those most significant [26]. RMSECV, *R*, and #*L* values obtained from the models developed are

**Table 2** Summary of PLS prediction results for whey fat content over the range 0.448–1.855% w/w and curd moisture content over the range 52.3–92.0% w/w using visible and near-infrared spectra collected between 5 and 85 min after cutting

Spectral treatment	<i>R</i> <sup>a</sup>	RMSECV (% w/w) <sup>b</sup>	RER <sup>c</sup>	# <i>L</i> <sup>d</sup>
Whey fat	0.88	0.106	13	6
Curd moisture	0.80	4.807	8	6
Whey fat (Jack-knifing)	0.91	0.094	15	5
Curd moisture (Jack-knifing)	0.86	4.057	10	5

<sup>a</sup> Correlation coefficient

<sup>b</sup> Root mean square error of cross validation

<sup>c</sup> Range error ratio

<sup>d</sup> Number of loadings incorporated into the model

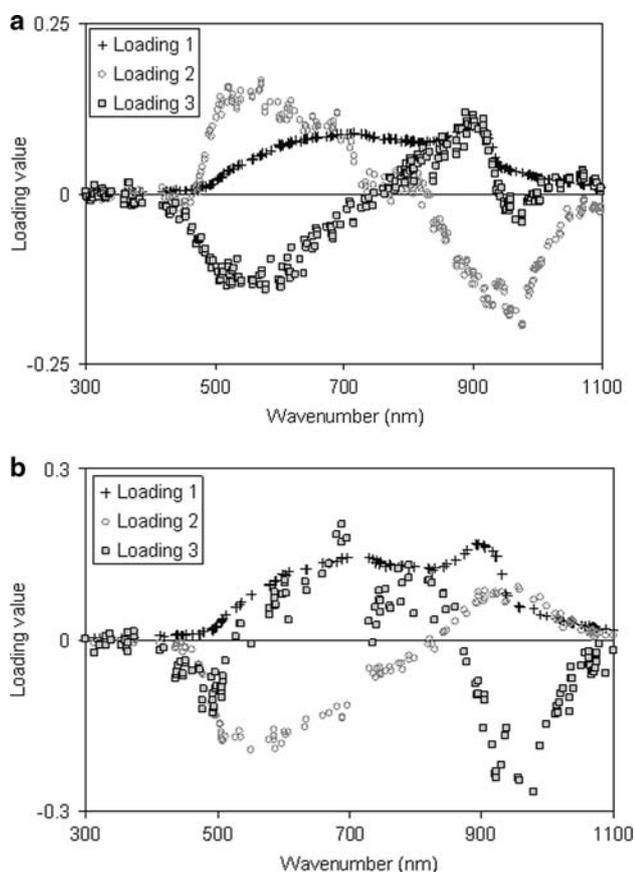
given in Table 2 for whey fat and curd moisture models. These parameters allow for assessment of model strength. The preferred predictive model for each parameter, was that which produced the lowest RMSECV and highest *R* values. It is also desirable that the preferred model incorporates the lowest possible #*L*.

The number of loadings incorporated into each model (5 and 6) can be considered relatively low suggesting that the models should be robust. The practical utility of the models was assessed using the RER, where less than 3 indicates little practical utility; between 3 and 10 indicates limited to good practical utility and above 10 indicates a high utility value [16]. The whey fat prediction models (RER = 13 and 15) were found to be of high practical utility while the curd moisture prediction models were found to have a good or high utility value (RER = 8 and 10). Jack-knifing reduced the number of wavelengths used for whey fat model development by 883 to 303, and reduced the number of wavelengths used for curd moisture model development by 1031 to 155. This was found to improve the accuracy of the developed models (Table 2). The accuracy of each model can be evaluated using the coefficients of determination (*R*<sup>2</sup>) between the predicted and measured values as stated by Williams [27]. A value for *R*<sup>2</sup> between 0.50 and 0.65 indicates that discrimination between high and low values can be made; where *R*<sup>2</sup> lies between 0.66 and 0.81 approximate predictions can be made, whereas, a value for *R*<sup>2</sup> between 0.82 and 0.90 reveals good predictions. Models having a value for *R*<sup>2</sup> above 0.91 are considered to be excellent. By reducing the number of wavelengths used in model development, *R*<sup>2</sup> improved from 0.78 to 0.83 for whey fat and 0.64 to 0.74 for curd moisture, providing good and approximate predictions for whey fat and curd moisture contents, respectively. The RER and *R*<sup>2</sup> values for the whey fat models tended to be higher than those of the curd moisture

content models, suggesting that the combined visible-near infrared spectra were more sensitive to whey fat content than to curd moisture content when analyzed using chemometric statistical techniques. In an examination of loadings 1–3 for each of the models the most important wavelengths for the prediction of whey fat were 518–578, 901 and 977 nm (Fig. 5a). In the prediction of curd moisture content the most important wavelength were 495, 512–600, 689, 790, 902 and 970 nm (Fig. 5b).

Due to the previously discussed variation in spectra obtained at 5 min due to scatter in the signal, a second set of models were developed which excluded these samples (Table 3). The removal of the 5 min samples improved both model accuracy and practical utility. It should be noted that these models are also predicted over a smaller range of reference values, as the 5 min samples tended to give extreme ranges of whey fat and curd moisture contents. However, as it is unlikely in practice that a cheese processor would require this information in the first 5 min after cutting it may be useful to develop models without such samples.

There are currently three available studies which have examined the potential of on-line technologies to predict



**Fig. 5** Loadings plot for **a** whey fat model and **b** curd moisture model developed using visible-near infrared spectra from all trials recorded during syneresis (5–85 min) and subjected to Jack-knifing

**Table 3** Summary of PLS prediction results for whey fat content over the range 0.448–1.482% w/w and curd moisture content over the range 52.3–83.2% w/w using visible and near-infrared spectra collected between 15 and 85 min after cutting

Spectral treatment	$R^a$	RMSECV (% w/w) <sup>b</sup>	RER <sup>c</sup>	#L <sup>d</sup>
Whey fat	0.94	0.067	16	6
Curd moisture	0.85	3.64	9	6
Whey fat (Jack-knifing)	0.95	0.061	17	5
Curd moisture (Jack-knifing)	0.89	3.187	10	4

<sup>a</sup> Correlation coefficient

<sup>b</sup> Root mean square error of cross validation

<sup>c</sup> Range error ratio

<sup>d</sup> Number of loadings incorporated into the model

important cheese making indices [6–8]. Everard et al. [6] proposed a method for monitoring syneresis based on changes in colour measurements occurring in a cheese vat. However, they found that moisture content of the curd could only be predicted using this method at high stirring speeds. Everard et al. [6] predicted curd moisture content ( $R = 0.78$ ), and whey total solids ( $R = 0.73$ ) at high stirring speeds.

Using light backscatter at 980 nm Fagan et al. [7] developed limited equations to predict curd yield, curd moisture and whey fat contents ( $R^2$  values were 0.75, 0.34 and 0.35, respectively). Fagan et al. [8] attempted to develop models with greater accuracy using a similar technique. They found that fat losses at the end of syneresis and curd moisture content as a function of processing time could be predicted using a combination of independent variables, milk compositional parameters and LFV light backscatter parameters with SEP of 2.65 g ( $R^2 = 0.93$ ) and 1.27% w/w ( $R^2 = 0.95$ ), respectively.

While the prediction achieved in this study using PLS regression were similar or stronger than those achieved by Everard et al. [6] and Fagan et al. [7], they were somewhat weaker than the predictions achieved by Fagan et al. [8]. However, the results of this study suggest that not only does the near-infrared region as found by Fagan et al. [8] contain important information for the prediction of syneresis indices but that a wider near-infrared region (900–977 nm) as well as regions of the visible spectrum (512–578, 600, 689, 790 nm) contain complementary information for the prediction of curd moisture and whey fat content during syneresis. Therefore an approach which combines the model development technique utilized by Fagan et al. [8], for example multiple linear regression, and information from key wavelengths as established in this study may result in the development of models with improved accuracy. Further modifications to the sensor should also improve the quality of the spectra and hence the prediction accuracy.

## Conclusions

The potential of visible-near infrared spectra, obtained using a light backscatter sensor in conjunction with chemometrics, to predict curd moisture and whey fat content in a cheese vat was examined. It was found that the use of Jack-knifing to reduce the number of insignificant wavelengths used in model development improved model accuracy. Models were developed with sufficient accuracy to demonstrate the potential of this technique ( $R = 0.86$ – $0.95$ ). Whey fat and curd moisture contents could be predicted with RMSECV of 0.094% to 0.061% (w/w) and 4.06% to 3.19% (w/w), respectively. In conclusion this technique has potential application for the prediction of important syneresis indices, such as curd moisture and whey fat contents, in a stirred cheese vat. Such a technology would be critical in improving overall process control during cheese manufacture, providing numerous benefits to both the consumer and processor.

**Acknowledgements** Funding for this research was provided by the Irish Department of Agriculture and Food through the Food Institutional Research Measure (FIRM), by the Kentucky Science and Engineering Foundation (Project KSEF-407-RDE-004), and by the US Department of Agriculture (Project NRI-USDA 2005-35503-15390).

## References

1. P.F. Fox, P.L.H. McSweeney, T.M. Cogan, T.P. Guinee, in *Fundamentals of Cheese Science* (Aspen Publishers, Gaithersburg, Editon edn., 2000), pp. 138–151
2. C.C. Fagan, M. Castillo, F.A. Payne, C.P. O'Donnell, D.J. O'Callaghan, *J. Dairy Sci.* **90**, 4499–4512 (2007). doi:10.3168/jds.2007-0329
3. D.B. Emmons, in *Cheese Yield and Factors Affecting its Control, IDF Seminar* (International Dairy Federation, Brussels, Belgium, Editon edn., 1993), pp. 293–301
4. M. Castillo, F.A. Payne, A. Shea, in *ADSA Annual Meeting*, Cincinnati, Ohio, USA, Editon edn., 2005
5. N. Taifi, F. Bakkali, B. Faiz, A. Moudden, G. Maze, D. Décultot, *Meas. Sci. Technol.* **17**, 281–287 (2006). doi:10.1088/0957-0233/17/2/008
6. C.D. Everard, C.C. Fagan, C.P. O'Donnell, D.J. O'Callaghan, M. Castillo, F.A. Payne, *J. Dairy Sci.* **90**, 3162–3170 (2007). doi:10.3168/jds.2006-872
7. C.C. Fagan, M. Leedy, M. Castillo, F.A. Payne, C.P. O'Donnell, D.J. O'Callaghan, *J. Food Eng.* **83**, 61–67 (2007). doi:10.1016/j.jfoodeng.2006.12.014
8. C.C. Fagan, M. Castillo, C.P. O'Donnell, D.J. O'Callaghan, F.A. Payne, *Int. Dairy J.* **18**, 120–128 (2008). doi:10.1016/j.idairyj.2007.09.007
9. M. Castillo, F.A. Payne, M.B. Lopez, E. Ferrandini, J. Laencina, *J. Food Eng.* **71**, 354–360 (2005). doi:10.1016/j.jfoodeng.2004.10.046
10. N. Qu, M. Zhu, H. Mi, Y. Dou, Y. Ren, *Spectrochim. Acta A Mol. Biomol. Spectrosc.* **70**, 1146–1151 (2008). doi:10.1016/j.saa.2007.10.036
11. T. Woodcock, G. Downey, C.P. O'Donnell, *J. Near Infrared Spectrosc.* **16**, 1–29 (2008)
12. T. Woodcock, C.C. Fagan, C.P. O'Donnell, G. Downey, *Food Bioprocess Technol.* **1**, 117–130 (2008). doi:10.1007/s11947-007-0033-y
13. C.C. Fagan, M. Castillo, F.A. Payne, C.P. O'Donnell, M. Leedy, D.J. O'Callaghan, *J. Agric. Food. Chem.* **55**, 8836–8844 (2007). doi:10.1021/jf070807b
14. M.S. Dhanoa, S.J. Lister, R. Sanderson, R.J. Barnes, *J. Near Infrared Spectrosc.* **2**, 43–47 (1994)
15. A. Savitzky, M.J.E. Golay, *Anal. Chem.* **36**, 1627–1639 (1964). doi:10.1021/ac60214a047
16. P. Williams, K. Norris, in *Near-infrared Technology in the Agricultural and Food Industries*, Editon edn., ed. P. Williams, K. Norris (AACC, St. Paul, 1987), pp. 143–167
17. H. Martens, M. Martens, *Food Qual. Prefer.* **11**, 5–16 (2000). doi:10.1016/S0950-3293(99)00039-7
18. J. Qin, R. Lu, *Appl. Spectrosc.* **61**, 388–396 (2007). doi:10.1366/000370207780466190
19. C.L. Crofcheck, F.A. Payne, M.P. Mengüç, *Appl. Spectrosc.* **41**, 2028–2037 (2002)
20. J.P. Wold, A. Veberg, A. Nilsen, V. Iani, P. Juzenas, J. Moan, *Int. Dairy J.* **15**, 343–353 (2005). doi:10.1016/j.idairyj.2004.08.009
21. S. Šašić, Y. Ozaki, *Appl. Spectrosc.* **55**, 163–172 (2001). doi:10.1366/0003702011951461
22. Y.A. Woo, Y. Terazawa, J.Y. Chen, C. Iyo, F. Terada, S. Kawano, *Appl. Spectrosc.* **56**, 599–604 (2002). doi:10.1366/0003702021955150
23. C. Blazquez, G. Downey, C. O'Donnell, D. O'Callaghan, V. Howard, *J. Near Infrared Spectrosc.* **12**, 149–157 (2004)
24. S. Šašić, Y. Ozaki, *Anal. Chem.* **73**, 64–71 (2001). doi:10.1021/ac000469c
25. R. Tsenkova, in *13th International Conference on Near Infrared Spectroscopy, Pre-conference Workshop No. 7*, Vaasa, Finland, Editon edn., 2007
26. H. Martens, M. Høy, F. Westad, D. Folkenberg, M. Martens, *Chemom. Intell. Lab. Syst.* **58**, 151–170 (2001). doi:10.1016/S0169-7439(01)00157-5
27. P. Williams, in *Near-infrared Technology—Getting the Best Out of Light* (Nanaimo, Canada, 1.1 edn., 2003), p. 109