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ORIGINAL RESEARCH

Application of a Large Field-of-View sensor during coagulation and syneresis in fresh goat cheese manufacture

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A light backscatter sensor with a large field of view (LFV) was used for the online monitoring of fresh goat cheese during coagulation and syneresis. A CoAguLite™ sensor was used as a reference at 880 nm to select the appropriate wavelength and configuration details for the LFV sensor for this type of cheese and manufacturing design, which was found to be 990, 1000 and 1010 nm. The light backscatter ratio followed a sigmoid increase during coagulation, which was lower than that observed during cow milk coagulation, and decreased asymptotically after cutting at the same rate as previously found for cow-milk curd. The fat losses and curd moisture could be predicted (R^2: 0.71, SEP: 0.04 and R^2: 0.98, SEP: 0.05, respectively) from the time taken to reach the maximum of the slope of the light backscatter ratio during coagulation (t_{max}) and from the LFV backscatter ratio. The reflectance ratio was strongly influenced by the fat losses and t_{max}, which explained variations in the moisture content.

Keywords Sensor, Syneresis, Goat curd, Coagulation, Moisture.

INTRODUCTION

Goat cheese consumption continues to increase because of its versatility and adaptability to recipes, the number of varieties available and a growth in artisanal activities. Indeed, artisanal cheeses are value-added products of high quality and limited quantity, in which the yield of cheese plays an important role (Ribeiro and Ribeiro 2010). The unique qualities of these cheeses are attributed to the raw materials used, distinctive processing and the particular attention paid to natural flavour and texture profiles.

Large economic losses may result from changes in the yield, shelf life and quality of cheeses, so that an in-depth control of moisture and of all the other parameters involved is necessary (Dejmek and Walstra 2004). Moreover, the possibility of such losses highlights the importance of improving process control during cheese manufacture.

One of the most important operations, which have a significant impact on cheese quality, is syneresis (Dejmek and Walstra 2004), which can be defined as the expulsion of whey from the curd by cutting, stirring and heating. Coagulation and syneresis conditions mark the final properties of a cheese.

Fresh cheeses have the shortest shelf life of all cheese categories because of the moisture content. The dry matter content, the composition of drained curd and the characteristics of the final product were determined by monitoring syneresis and, subsequently, controlling whey drainage through mechanical and physical actions. Such control is considered a crucial step in cheese technology (Daviau *et al.* 2000). Monitoring syneresis is a useful tool for reaching the desired final cheese moisture content, although coagulation conditions must be carefully controlled too (Fagan *et al.* 2007b).

The first techniques for online monitoring during cheese manufacture were developed to control milk coagulation conditions and to predict the final cheese composition. One such technique is near infra-red spectroscopy (NIRS) alone (Gonzalez-Martín *et al.* 2008) or in conjunction with a remote reflectance fibre-optic (Rodríguez-Otero *et al.* 1995, 1997) applied in the final manufactured cheese from a mixture of three varieties of milk: ewe, goat and cow. Spectroscopy techniques are fast, relatively inexpensive, sensitive and noninvasive. Although, they cannot be used for the online monitoring of cheese manufacture, they are suitable for real-time remote control in production lines for checking or quantifying the composition of fat, protein and moisture.

Another technique depends on a fibre-optic sensor denominated CoAguLite™, which is able to

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predict cheesemaking indices, such as cutting time and cheese yield, and which can be used to develop algorithms of curd moisture and fat losses during milk coagulation (Fagan *et al.* 2007a). This sensor has been applied with good results to studying the effect of temperature, inoculum concentration and $CaCl_2$ concentration (Castillo *et al.* 2000, 2002, 2003) both in goat and cow milk, and is widely accepted as an appropriate online sensor for monitoring milk coagulation.

This optical fibre light backscatter sensor, CoAguLite™, has also been studied for possible application during curd syneresis (Fagan *et al.* 2007a, 2008). As this sensor produces a high degree of scatter after cutting, other online techniques were thought necessary to monitor syneresis. For this reason, a light backscatter sensor with a large field of view (LFV) was developed, which accurately measures syneresis and predicts curd yield and curd moisture during cheese manufacture (Fagan *et al.* 2007a). Later, the LFV online sensor was validated using different milk protein, fat, firmness, protein ratio levels and stirring speeds, and its ability to predict whey production and fat content in whey at syneresis was confirmed (Mateo *et al.* 2009a,b, 2010). However, the different compositions and stirrings considered by those authors for the implementation of the prototype differed significantly with this study conditions, because it does not resemble the physicochemical characteristics and components of goat milk and the technological stages involved in fresh cheese manufacture. These are important considerations when considering the suitability of this sensor for its subsequent application in the fresh goat cheese industry.

Goat milk differs from cow milk in its higher casein diameter, higher degree of polydispersity (Trujillo *et al.*1998), lower fat globule size and higher fat content (Attaie and Richter 2000). Such differences affect the curd protein matrix and its behaviour during syneresis. Diffuse reflection is proportional to density and inversely proportional to the ratio of distribution (Olsen 1990), and so differences in the LFV configuration for its appropriate application in this raw material should be necessary, as the ratio distribution mainly depends on the number of particles, their size and the wavelength.

No references have been found concerning application of the LFV sensor to goat milk and fresh cheese manufacture, despite the worldwide increase in goat cheese production. The aim of this research was to carry out the application of this prototype sensor in goat milk and fresh goat cheese manufacturing for its online monitoring, especially during the processes of coagulation and curd syneresis. Moreover, it is necessary to study whether this technique could be a useful tool for predicting the fat and moisture contents.

MATERIALS AND METHODS

Murciano-Granadina goat milk

Fifty litres of fresh goat milk provided by the Veterinary Faculty farm of the University of Murcia was received 24 h before each experimental day. The milk was pasteurised immediately upon reception at 78 °C for 30 s using a plate heat exchanger (100 L Alfa Laval, Lund, Sweden). The milk was later cooled and stored at 4 °C until it was used for cheesemaking. The average fat, protein and dry matter contents of the milk analysed by providers were 4.98 ± 0.01%, 3.43 ± 0.04% and 14.02 ± 0.02%, respectively.

Cheese manufacture and sampling

Pasteurised Murciano-Granadina fresh goat milk was used to manufacture cheeses at the Food Technology pilot plant of the University of Murcia. Six production days were carried out for the experimental design, for each of which two replicates were completed.

Ten litres of pasteurised cooled goat milk was tempered for 10 min until a constant temperature of 33–34 °C. Stirring slowly, 4 mL of $CaCl_2$ (Chr Hansen, France) at a concentration of 510 g/L was added. Then, 2.13 mL of liquid rennet (Caglio Star Spain S.A., Cieza, Murcia, Spain) comprising a 80/20 mixture of chymosin and pepsin was added. The cutting time (t_{cut}) was determined by multiplying t_{max} by $\beta = 2.5$, as it was previously described (Fagan *et al.* 2007a). Although this β has been applied to cow milk, it was selected here to ensure the same conditions as developed by those authors (Fagan *et al.* 2007a), although in this case, with goat milk. In this way, any differences and comparisons between models and results would be due to the milk used.

After cutting (20 s), two series of stirring (5 min) and pitching (5 min) were carried out followed by a final stirring. The curd was then moulded, pressed and salted (17° Be for 20 min). The cheesemaking procedure was repeated on alternate days. Samples of curd and whey were taken for physicochemical determinations during four different stages: cutting time, and during the first, second and third stirring steps.

OnLine light backscatter monitoring

Two sensors were coupled to a 10 L air-conditioned vat: CoAguLite™ sensor (model 5, Reflectronics Inc., Lexington, KY, USA) (Payne *et al.* 1993), which measures at 880 nm, and the LFV sensor (prototype, University Kentucky, USA) (Fagan *et al.* 2007a), which measures from 200 to 1100 nm. The light backscatter response from the two sensors was continuously monitored from the time of rennet addition (t_{c0}) to the end of syneresis, considered as the time when the vat began to be emptied. Response data were collected every 10 s. The initial voltage (V_0) was calculated by averaging the first four data points obtained before rennet addition. The light backscatter ratio (*R*) and t_{max} were calculated as previously described (Fagan *et al.* 2007a). The t_{cut} was determined using the following equation: $t_{cut} = \beta t_{max}$ (Payne *et al.* 1993).

All the LFV sensor responses in the 300–1100 range were used in this study. The sensor provided measurements every 100 ms, and each spectrum is derived from 100 sensor scans. The spectrum was automatically processed every 10 s by subtracting the dark spectrum and was reduced to 20 averages by dividing into 40-nm wavebands and averaging the optical response for the wavelengths constituting each waveband. The voltage values for the first fourteen seconds of data were averaged within each waveband V(w) to calculate $V_0(w)$. The light backscatter ratio for the LFV sensor (*R*) and t_{max} was obtained as it was previously measured (Fagan *et al.* 2008). The percentage increase was calculated as the percentage difference between the ratio of the LFV light backscatter at the inflection point and the ratio at the time the enzyme was added. The percentage decrease was measured as the percentage difference between the ratio of the LFV light backscatter at inflection point and the ratio at the final time.

Fat losses and moisture content

Curd and whey samples were separated using a dairy cloth. The fat content in whey was determined by Gerber Van Gulik method (ISO 1975). For moisture content determination, curd samples (± 3g) were dried by to constant weight (ISO 1997). Measurements for each whey sample were performed in duplicate.

Statistical analysis

Statistical treatment of the data was performed using SPSS v15.0 (2006, SPSS Ibérica S.L.U. Madrid, Spain). An average of 6 elaborations was selected for each wavelength, and an ANOVA analysis was carried out between each wavelength, to select those averages without significant differences. Multivariate linear regression analysis fitted to a number of different regression models was performed to describe the prediction models.

RESULTS AND DISCUSSION

The CoAguLite™ sensor was used as a reference for the LFV sensor to select the appropriate averages without significant difference ($P > 0.05$) for fresh goat cheese manufacture. Figure 1 shows the light backscatter ratio profiles during coagulation and syneresis obtained using the LFV sensor between 300 and 1100 nm, grouped into 20 bands of 40 nm, and the CoAguLite™ sensor (880 nm).

As can be seen in this figure, the LFV sensor–intensity response was lower than the corresponding response for CoAguLite™, as described by Fagan *et al.* (2007a) in cow milk. The signals obtained in the range 300–420 nm, whose ratio decreased during coagulation, were discarded as it has been extensively described that the signal during coagulation should increase, such as the increase observed in the reference sensor (Fagan *et al.* 2007a, 2008). Moreover, the signals corresponding to the 420–940-nm range, which remained constant until the cutting time, confirm the poor sensitivity of the LFV sensor in this range. These wavelengths, therefore, should not be considered for online monitoring, as no response was obtained during goat milk coagulation. The only profiles to show a significant response during coagulation corresponded to the wavelengths within the 940–1100 nm range, which displayed a positive slope over this process similar to that obtained with the CoAguLite™ sensor. As shown in Figure 1, the CoAguLite™ response was not sufficient to monitor syneresis, because of the high level of dispersion after the cutting time; in contrast, the LFV sensor showed a significant fall in the ratio.

Figure 2 shows the response of the CoAguLite™ sensor at 880 nm and the profiles of the wavelengths between 940 and 1090 nm obtained by the LFV sensor during coagulation and syneresis in the development of fresh goat cheese. The wavelengths represented are those that offered the best fit in relation with the reference profile obtained with CoAguLite™.

The LFV sensor response obtained during coagulation was similar to that of the CoAguLite™ sensor, which indicates that it is sensitive to the chemical changes that occur during this period. The three wavelengths showing the highest increase in value from the beginning of the process to the end of coagulation were 990, 1000 and 1010 nm, with increases of 7.73%, 7.47% and 7.58%, respectively, which are slightly lower than that obtained by the CoAguLite™ sensor (11.8%). Nevertheless, these increases in the LFV response percentages were lower than the average increase of 23.5 ± 5.4% at 980 nm obtained

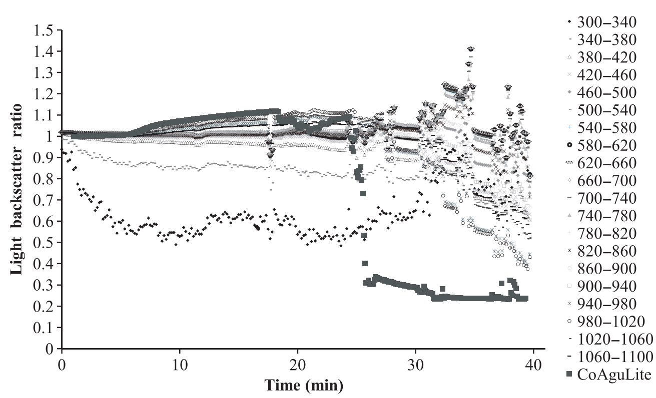

Figure 1 Sensor profiles of large field of view and the reference CoAguLite™ during coagulation and syneresis at wavelength between 300 and 1100 nm.

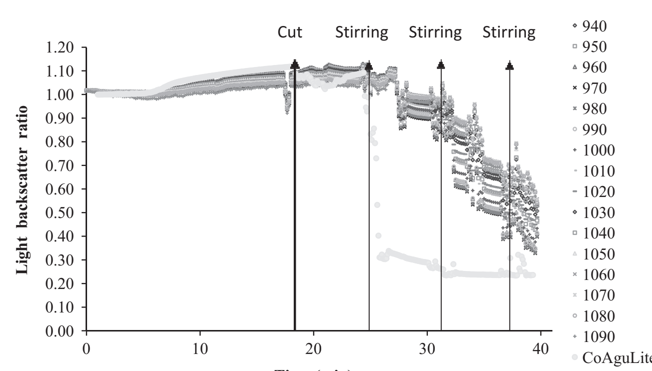

Figure 2 Sensor profiles of large field of view and the reference CoAguLite™ during coagulation and syneresis at wavelength between 960 and 1100 nm.

for cow milk curd (Fagan *et al.* 2007a). If other β values had been selected for the cutting time calculation, such as those predicted for goat milk (Castillo *et al.* 2003, 2006a,b) (1.48, 1.97 and 1.29, respectively), then the increase and decrease in coagulation percentages would not be suitable for comparing with those of other authors (Fagan *et al.* 2007a), as the cutting time would be different, so initial point of the increase and decrease would occur earlier.

The CoAguLite™ response during syneresis displayed a high degree of dispersion, while the LFV sensor response showed a downward trend, with scattering at different times, which can be attributed to stirring of the curd, a necessary step in the development of fresh goat cheese.

The percentage decrease from cutting time to the end of the process was between 45.8 and 51.91%, with the maximum decrease being observed at 990, 1000 and 1010 nm (51.91%, 51.81% and 51.84%, respectively). Wavelength 990 nm showed the least dispersion and the highest coagulation increase and decrease. Those percentage decrease observed at 990, 1000 and 1010 nm during syneresis are within the signal decrease range (25–61%) described for cow milk curd at 980 nm (Fagan *et al.* 2007a). The wavelengths chosen took into account the criteria previously described (Fagan *et al.* 2008).

Differences in polydispersity, casein diameter and fat content affect the curd protein matrix and its behaviour during syneresis and so the ratio distribution. The differences in the LFV backscatter ratio profile between cow milk and goat milk from the beginning to the end of coagulation highlight the influence of the fat content and fat and casein size in the sensor response.

When the milk fat content shows a wide range, the online visible-NIR sensor shows greater potential using a broad spectrum of wavelengths than a single wavelength (Mateo *et al.* 2009a). The fat content of goat curd and its size affect the sensor response, and so the three wavelengths finally selected (990, 1000 and 1010 nm) are considered suitable for the online monitoring of goat curd during coagulation and syneresis, all three providing similar percentages for dispersion and ratio increases and decreases. These wavelengths are slightly higher than the one selected (980) for cow milk (Fagan *et al.* 2007a; Mateo *et al.* 2009a,b, 2010). This emphasises the importance of the preliminary application of the LFV sensor in goat milk, as the milk's physicochemical features must be taken into account if the sensor is to perform properly.

Whey production can be predicted using single wavelengths, such as the 980 nm selected for cow milk and curd prediction models (Mateo *et al.* 2010). Moreover, the most appropriate wavelength for prediction models is the one showing least dispersion and highest increase and decrease in coagulation (Fagan *et al.* 2008). Therefore, taking into account both considerations, one wavelength was chosen to predict the fat content and moisture in this study (990 nm), as this wavelength showed the least dispersion and the highest coagulation increase and decrease.

Table 1 shows the models obtained by a linear multivariate regression analysis of the LFV sensor ratio at 990 nm to predict fat losses and curd moisture content. This wavelength provided significant and strong R^2 coefficients and low standard error of prediction (SEP) in all the prediction models developed.

Table 1 Prediction models for fat losses and curd moisture content

Model		*df*	β_1	β_2	R^2	*SEP*
I	$F = \beta_1 R$	11	0.88**		0.69	0.07
II	$F = \beta_1 R + \beta_2 t$ max	11	0.39^{ns}	0.02^{ns}	0.71	0.04
III	$\%W = \beta_1 R$	11	75.49***		0.84	8.85
IV	$\%W = \beta_1 R + \beta_2 t$ max	11	8.24^{ns}	2.98***	0.98	0.05
V	$\%W = \beta_1 t$ max	11	3.28***		0.98	0.23

**$P < 0.01$; *** $P < 0.001$; n.s.: not significant; df, degrees of freedom; β_1–β_2, regression coefficients; R^2, coefficient of determination; SEP, standard error of prediction; %W, curd moisture; *R*, ratio; *F*, whey fat; t_{max}, time to the first maximum of the light backscatter ratio (*R*).

The first two models (I, II) predict whey fat content. Model I recorded a low SEP (0.07) of all the models developed for whey fat content, although the corresponding R^2 coefficient (0.69) was the weakest. A similar observation was previously made by Mateo *et al.* 2010; who also developed a simple model that took into account the light backscatter reflectance ratio derived from the LFV, in their case at 980 nm and for predicting cow milk whey fat content. The model developed by these authors showed a lower R^2 value (0.42) and almost the same SEP value (0.07). This suggests that the ratio is a significant parameter for predicting whey fat content, but does have the greatest effect. Moreover, the model suggests that the differences between the signals of the LFV light backscatter obtained in cow milk (Fagan *et al.* 2007a) and those obtained in this work can be explained by differences in the fat content between cow and goat curd. This model may, then, be considered as suitable for prediction of whey fat losses at pilot scale.

With its significant R^2 coefficient (0.71) and low SEP, model II predicts the whey fat content in relation to the light backscatter ratio and t_{max}, the time elapsing from enzyme addition to the inflection point of the LFV light backscatter ratio (Fagan *et al.* 2007a). The β coefficients shown in this model indicate that the ratio is the variable exerting more impact in whey fat content prediction, as the corresponding ratio β_1 coefficient (0.39) is higher than the t_{max} β_2 coefficient (0.02). This indicates that t_{max} is not useful for predicting the whey fat content, as it has a very small β_2 coefficient ($\beta_2 < 0.05$). Moreover, this model strongly suggests that the differences between the ratio increase and decrease in our study could be related to differences in moisture content, in accordance with the results found for cow milk (Fagan *et al.* 2007a).

These first two models (I, II) shown in Table 1 indicate that the whey fat content is an important factor influencing the ratio derived from the sensor response, as seen from the R^2 coefficient, although this parameter has only a slight impact because of its lower β coefficients ($\beta < 1$).

Models III, IV and V predict curd moisture. Model III displays a significant R^2 (0.84), but has the highest SEP than models IV and V. Models IV and V show the most significant R^2 coefficients (0.98). In both equations, the moisture content is predicted by the independent variable $t_{max,}$ in model IV, including the ratio derived from the sensor response, and in model V alone.

As expected, the coagulation variable t_{max} is a significant factor in curd moisture content, which is confirmed in models IV and V. These two last predicted models have higher R^2 (0.98) and lower SEP values than other simple models developed (Mateo *et al.* 2010), with an R^2 of 0.65 and a SEP of 1.10. Milk coagulation reactions and their effect on the physical properties of the milk protein matrix suggest that t_{max} is a significant factor in the curd moisture content. Model IV suggests that the light backscatter ratio influences curd moisture measurements, but with t_{max}, its influence increases as its β coefficient is higher. Both models (IV and V) can be considered as suitable for predicting the moisture content during syneresis of fresh goat cheese using an LFV sensor with a low standard error of prediction. This demonstrates the possibilities of this sensor for predicting physicochemical cheese characteristics that are important for profitability in the industry. However, these results must be regarded as preliminary because of the limited number of data used.

The relations between measured and predicted values of the models developed are shown in Figure 3. As can be observed, all models display an appropriate upward trend line. All the R^2 coefficients from these models are the ones detailed in Table 1, showing the appropriate fit of the models.

The few points observed in each of the graphics display in Figure 3 emphasises the importance of transferring the preliminary research results to a wider experimental design at pilot scale before the LFV sensor can be confirmed as a useful tool for predicting the whey fat and moisture content in fresh goat cheese during syneresis.

Differences in the axes and number of points between measured versus predicted values from the different models shown in Figure 3 are attributed to the parameters involved in each model, as not the same number of values are available for all the parameters.

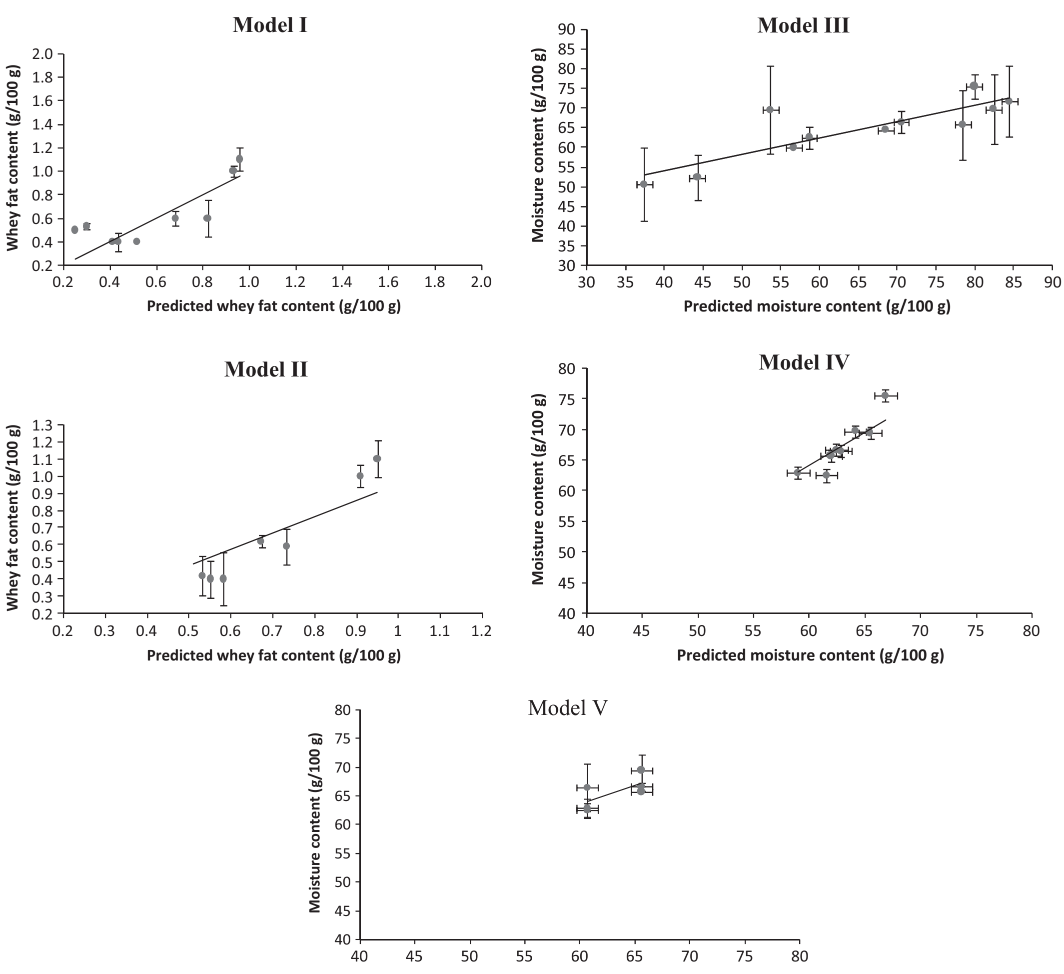

Figure 3 Measured versus predicted physicochemical indices using linear models. (Representation of the linear trend equation from the models detailed in Table 1).

CONCLUSION

This study confirms the potential of the LFV sensor for the online monitoring of the coagulation and syneresis processes in goat milk, improving the results previously reported with the use of the CoAguLite™ sensor. A higher range of wavelength (990, 1000 and 1010 nm) is necessary for goat milk than for cow milk coagulation and syneresis (980 nm), because of differences in the fat content and the casein and fat size. The LFV is a useful tool for predicting fat losses and moisture during fresh goat cheese coagulation and syneresis, as the prediction models showed high R^2 and low SEP. Further research, widening the study to include industrial-scale production, would confirm the viability of the LFV sensor for online monitoring in the cheese industry and the suitability of the models developed. This research will enable scientific–technical studies of different aspects of the industrial manufacture of goat cheese.

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