

Optical sensor technology for measuring whey fat concentration in cheese making

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Received 9 June 2004; accepted 24 October 2004

Available online 16 December 2004

Abstract

Improving syneresis control in cheese making may require the development of an optical sensor technology to measure whey fat content during processing. Light sidescatter and transmission responses as a function of whey fat concentration (0–0.9%) were measured using a fibre optic spectrometer (300–1100 nm) to determine if an optical correlation could be developed. Normalized spectral responses versus fat concentration followed a power function. Waveband ratio combinations were calculated and tested for prediction of whey fat concentration using two prediction models. The best regressions were found to use sidescatter ratios as predictors and typically contained numerator and denominator ratio wavebands in the ranges 725–1025 and 375–475, respectively. $S_{875/425}$, the ratio calculated by dividing the normalized sidescatter intensity at waveband 875 by that at 425 nm, was found to be related to whey fat concentration ($R^2 = 0.99$) using an exponential model. These results suggest that whey fat concentration during syneresis could be measured with an inline fibre optic sensor.

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Keywords: Inline sensor; Process control; Fibre optic; Fat concentration; Whey

1. Introduction

Improving process control and automation has a large economical impact in the dairy industry and requires the development of suitable sensor technologies to characterize the properties of liquid products. Several applications using fibre optic sensor technologies have been developed for the dairy industry and cheese manufacturing during the last decade. Payne, Crofcheck, Nokes, and Kang (1999) developed an optical sensor to detect water-product transitions as they flow through

pipelines during liquid food processing. Milk coagulation induced by either acid or rennet action has been monitored using a light backscatter fibre optic sensor to predict cutting time (Payne, 1995; Payne, Hicks, & Shen, 1993), Berridge clotting time (Castillo, Payne, Hicks, & López, 2000) and the rheologically determined gelation time (Castillo, Lucey, & Payne, 2004a). An interesting application of light backscatter sensor technology has been recently proposed by Castillo, Lucey, Wang, and Payne (2004b). These authors showed a significant interaction between coagulation kinetics (light backscatter parameters) and syneresis kinetics during cheese making. A sensor technology having the ability to control curd moisture content would have a large impact on cheese manufacturing worldwide in terms of product quality, consistency and production efficiency.

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One of the approaches for the development of a syneresis sensor technology is to obtain curd shrinkage kinetics from the dilution of fat globules during syneresis. Whey fat acts as an internal tracer of whey expulsion during syneresis. A sensor technology capable of accurately measuring low concentrations of whey fat and detecting small whey fat concentration changes during syneresis is needed for this approach. In addition, syneresis control will require the correlation of the syneresis kinetic parameters to curd moisture content. This correlation could then be used to predict an optimal syneresis end-point.

Several attempts to develop a sensor to measure fat concentration in dairy products have been reported. Crofcheck, Payne, Hicks, Mengüç, and Nokes (2000) found that small quantities of milk fat (0.05–0.2%) were detectable using an optical fibre sensor in the transmission mode. A light extinction sensor has been developed by Gillette, Payne, and Crofcheck (2002) for measurement of non-homogenized fat in milk, which employed optical fibres to measure light attenuation within milk.

The goal of this work was the development of a sensor technology capable of measuring low whey fat concentration and detecting small fat concentration changes during curd syneresis. An ideal inline optical system for measuring whey fat concentration in cheese making should only respond to fat concentration changes during syneresis. The optical system should neither respond to batch-to-batch differences nor changing chemical composition during syneresis, other than fat concentration. We propose that the use of optical waveband ratios could provide normalization benefits for this application minimizing the effect of background changes during whey fat concentration measurement.

2. Materials and methods

Goat cheese whey from the industrial manufacturing of “Ripened Murcia Cheese” was collected from La Algodonera Cheese Plant (Manuel Vivancos Navarro, Alhama de Murcia, Spain). Three different batches (one per day) of raw whey were collected to obtain samples within a wide range of low fat concentrations (0.00, 0.15, 0.30, 0.45, 0.60, 0.75 and 0.90 wt.%). Three series of fat dilutions (i.e., three replications) were prepared from each batch. Light sidescatter and transmission spectra were collected from each sample and analyzed to determine if waveband ratios could be related to whey fat concentration.

2.1. Milk coagulation and whey samples collection

Goat milk was pasteurized (72–75 °C, 15 s) and then ~156 mg of anhydrous calcium chloride per kg of milk was delivered to the milk by adding a corresponding amount of a commercial CaCl₂ solution. Milk was

pre-acidified using a mesophilic lactic culture (*Streptococcus thermophilus*, *Lactococcus lactis* spp. *lactis* y *L. lactis* spp. *cremoris*) supplied by Chr. Hansen (CHN22, Chr. Hasen S.A., Madrid, Spain). Milk gelation was induced at pH ~ 6.4 by adding 0.3 ml of calf rennet (80% chymosin; 145 IMCU ml⁻¹; Caglio Star España, S.A., Murcia, Spain) per kg of milk. The gel was cut as determined by the operator. After cutting the gel, the mix of curd and whey was stirred for 40 min with a gradual temperature increase from 30 to 38 °C. When the curd cooking process was finished, the curd and whey were poured through a nylon cheese cloth (1 mm² pores) to remove the curd grains and fines from the whey. Five-litre batches of whey were collected each testing day. The drained whey was collected into an aluminium container and transported to our installations within 30 min after collecting. Whey batches were sampled and fat concentration of each batch was determined three times by the Gerber method (Standard: IDF, 152A, 1997). The average of the three measurements was reported as the batch fat concentration.

2.2. Preparation of whey sample sets having a range of fat concentrations

Each raw whey batch was split into two parts. One portion was skimmed while the other was used as whey fat source. The skim whey was obtained by centrifugation (4000 rpm for 15 min) and subsequent filtration (Whatman®, Cat No: 1006 110, pore size 2.5 µm). Fat concentration of skim whey obtained was measured in triplicates to confirm that fat concentration was ≤0.05%. Batch fat concentration was used to calculate mixture proportions of skim and raw whey to obtain 50 g samples with the desired fat level (0.00, 0.15, 0.30, 0.45, 0.60, 0.75 and 0.90 wt.%). Optical sidescatter and transmission responses were measured for each sample on the same day as collected from the cheese plant.

2.3. Optical measuring configuration

A miniature fibre optic spectrometer (model SD2000, Ocean Optics, Inc., Dunedin, FL, USA) was connected by optical fibres to a prototype cell designed for measuring light sidescatter (90°) and transmission of whey samples at different optical path lengths. The sampling cell consisted of a 40 ml cylindrical black plastic base with three 2.54 cm diameter holes for inserting the fibre optic probes, bored at 90° angle and 180°. Fig. 1 shows the configuration used. A black plastic cap was used to eliminate external light interferences. A series of calibration rod spacers were used to adjust the path length.

The fibre optic spectrometer consisted of master and slave units. The master unit had a 25 µm slit, a 300 lines mm⁻¹ diffraction grating with a range of 300–2000 nm and a detection bandwidth of 200–1100 nm. The slave

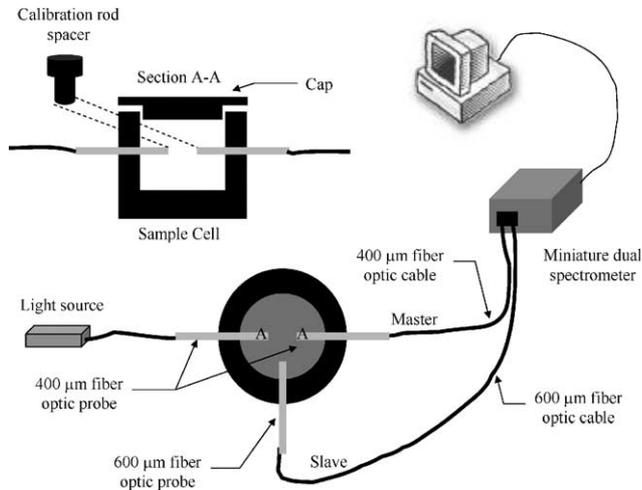


Fig. 1. Schematic diagram of the optical system configured for measuring of light transmission and sidescatter (90°).

unit had a $100 \mu\text{m}$ slit, a $300 \text{ lines mm}^{-1}$ diffraction grating with a range $300\text{--}2000 \text{ nm}$, a L2 lens and a detection bandwidth of $300\text{--}1100 \text{ nm}$. The units were equipped with a 2048-pixel linear CCD-array silicon detector (Sony ILX 511, Tokyo, Japan) with a response range of $200\text{--}1100 \text{ nm}$ and a sensitivity of 86 photons per count at 1 s integration time. The light source used was a tungsten halogen light source (LS-1, Ocean Optics, Inc.) with spectral range of $360 \text{ nm}\text{--}2 \mu\text{m}$. Light source, master and slave units were connected by using optical fibre probes and cables manufactured by the University of Kentucky and Reflectronics Inc. (KY, USA) using 400 and $600 \mu\text{m}$ diameter fibres (Spectran Specialty Optics, Avon, CN, USA). The integration time was set ($5\text{--}15 \text{ s}$) by the computer software (OOIBase, Version 1.5, Ocean Optics, Inc.) according to the light intensity.

2.4. Data analysis and calculation of normalized spectral scans

The backscatter and transmission spectral data were analyzed to determine if waveband ratios could be used to predict whey fat concentration. The transmission and sidescatter spectral scans, $S(\lambda)_T$ and $S(\lambda)_S$, were automatically processed by subtracting the respective dark spectral scans for transmission and sidescatter, $S_D(\lambda)_T$ and $S_D(\lambda)_S$, and dividing by the integration time to give the normalized spectral scans for transmission, $S_N(\lambda)_T$, and sidescatter, $S_N(\lambda)_S$, respectively (bits s^{-1}). Each normalized spectral scan, $S_N(\lambda)$, was reduced to 14 averages by dividing them into 50 nm wavebands with mid-wavelengths of $325 + 50 \cdot n$ ($1 \leq n \leq 14$) and averaging the optical response (transmission or sidescatter) for the wavelengths constituting each waveband. The 14 wavebands obtained were in the range ($375\text{--}1025 \text{ nm}$). Then, all possible transmission ratios for each test were calculated by dividing the transmission response at one

waveband by another for all waveband combinations. Sidescatter ratios were also calculated in a similar manner. A plot of the transmission and sidescatter ratios as a function of whey fat concentration suggested the following two models:

$$\text{Ratio} = \beta_0 [\text{Fat}]^{\beta_1} \quad (1)$$

$$\text{Ratio} = \beta_0 + \beta_1 e^{\beta_2 [\text{Fat}]} \quad (2)$$

where β_0 , β_1 , and β_2 were constants. Solving for whey fat concentration, these two models yielded:

$$\log[\text{Fat}] = \beta_0 + \beta_1 \log \text{Ratio} \quad (3)$$

$$[\text{Fat}] = \beta_0 \ln(\beta_1 \text{Ratio} + \beta_2). \quad (4)$$

All sidescatter and transmission waveband ratios were simultaneously tested using “NLIN” procedure of the Statistical Analysis System (SAS[®], 1999) to determine the ratios with the higher R^2 for prediction of whey fat using model I (Eq. (3)) and model II (Eq. (4)).

3. Results and discussion

3.1. Effect of fat concentration on normalized sidescatter and transmission spectral scans

Fig. 2 shows the typical normalized sidescatter (Fig. 2a) and transmission (Fig. 2b) spectral scans as a function of whey fat concentration, respectively. As expected, sidescatter response intensity was proportional to fat concentration (Fig. 2a) while transmission intensity decreased as fat concentration increased (Fig. 2b). In a liquid particulate media, light which is not absorbed by the media is either scattered or transmitted (Abdou, 1987). Light scattering intensity is not only proportional to particle concentration but also depends, among other properties, on the material of the particle (i.e., the complex index of refraction) and its size parameter ($\pi d/\lambda$, where d is the diameter of the particle) (Modest, 2003). According to Walstra and Jenness (1984), the substances responsible for light scatter in milk are mainly fat globules and casein micelles. Casein micelles scatter proportionally less light than fat globules because they are much smaller than fat particles, and the difference in index of refraction between casein and water is smaller than between fat globules and water. The casein micelle concentration in whey is small because most casein micelles are retained in the curd. Thus, light scatter in whey should be primarily attributed to fat globules. Light sidescatter should then increase with whey fat concentration, and light transmission decrease. A power law function was found to model both sidescatter and transmission profiles as shown in Fig. 3. Light transmission intensity decreased proportional to $[\text{fat}]^{-1.809}$, ($R^2 = 0.999$) while sidescatter increased proportional to $[\text{fat}]^{0.445}$, ($R^2 = 0.981$).

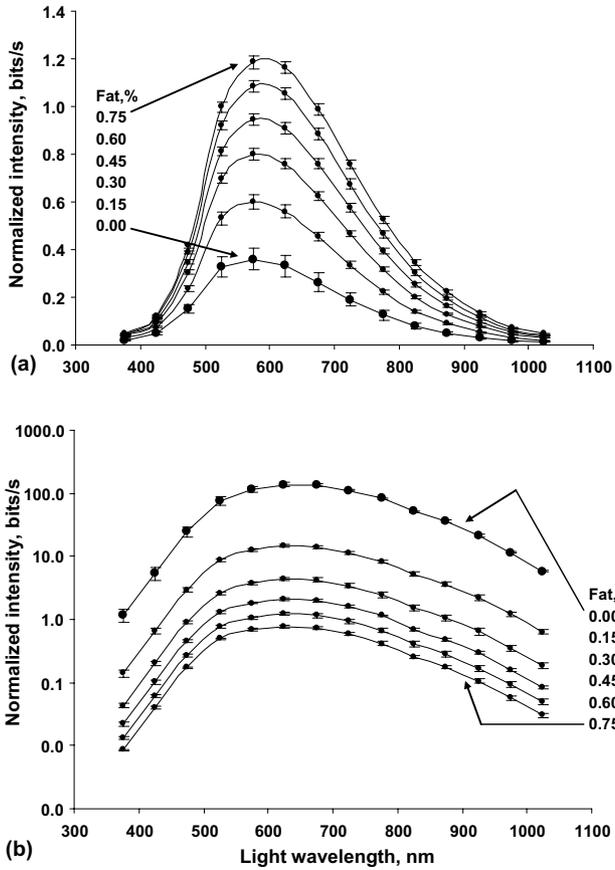


Fig. 2. Typical normalized sidescatter (a) and transmission (b) spectral scans as a function of whey fat concentration.

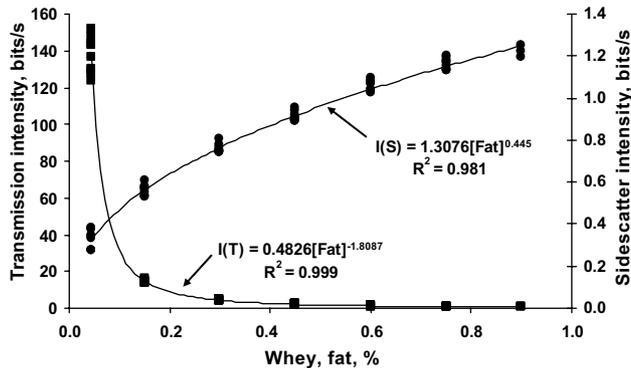


Fig. 3. Normalized optical response at 625 nm as a function of whey fat concentration [Fat]. ●, Sidescatter; I(S) ■, Transmission, I(T). Data plotted were for the entire data set (N = 57). Skim whey with measured fat concentration <0.05% was assumed to contain 0.043% whey fat which was within the measurement precision of the analytical method for measuring fat concentration.

3.2. Use of sidescatter and transmission ratios to predict whey fat concentration

All sidescatter and transmission waveband ratio combinations (182 for sidescatter and 182 for transmission) were analyzed using models I and II to compare their

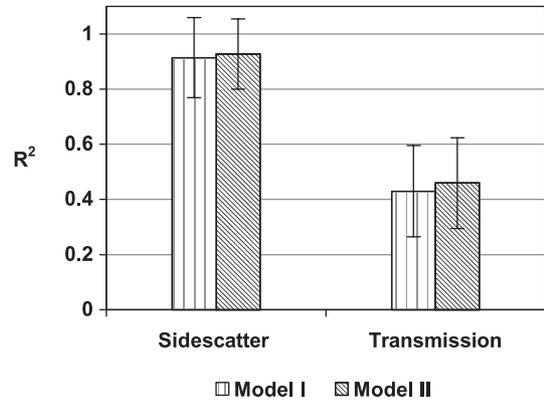


Fig. 4. Summary of the R^2 for model I and II prediction of whey fat concentration for all combinations of sidescatter or transmission ratios. For each regression, N = 57. Error bars corresponded to ± 1 SD interval of the average value of R^2 .

ability to develop a relation between whey fat concentration and waveband ratio. Fig. 4 summarizes the R^2 data for all waveband combinations for models I and II. This data suggest that sidescatter waveband ratios using either model I or model II are significantly ($P < 0.0001$) better for relating to whey fat concentration. No significant difference ($P > 0.05$) was found between predictions obtained by model I and II. Measurement of the sidescatter intensity (i.e., light scatter at an angle or “nephelometry”) is typically used for measurement of low concentrations of material because it performs better in dilute solutions where absorption and scatter are minimal (like whey samples). However, turbidity measurement, based on measurement of transmittance, performs better in concentrated solutions where difference between the intensities of the incident and transmitted beams is large.

Fig. 5 shows the distribution of R^2 values for model II using sidescatter ratios for predicting whey fat

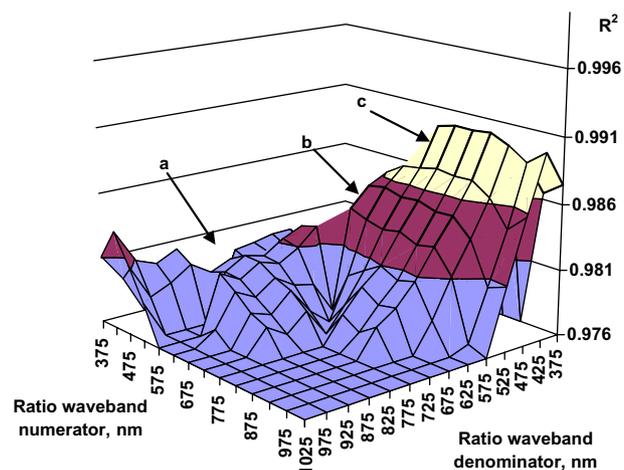


Fig. 5. R^2 values as a function of sidescatter ratios used for prediction of whey fat concentration using prediction model II. Different surface areas corresponded to predictions with R^2 values in the range: (a) 0.976–0.981; (b) 0.981–0.986; or (c) 0.986–0.991.

Table 1
The ten sidescatter ratios yielding the highest R^2 using model II and the associated regression parameters^a

Order	Waveband ratio	β_0	β_1	β_2	R^2
1	$S_{875/425}$	-0.80	-0.70	1.66	0.9906
2	$S_{775/425}$	-0.76	-0.34	1.84	0.9905
3	$S_{825/425}$	-0.79	-0.48	1.73	0.9904
4	$S_{1025/425}$	-0.70	-4.16	1.91	0.9902
5	$S_{725/425}$	-0.76	-0.27	2.02	0.9901
6	$S_{925/425}$	-0.76	-1.15	1.63	0.9900
7	$S_{975/425}$	-0.70	-2.25	1.67	0.9891
8	$S_{1025/375}$	-0.57	-1.89	2.10	0.9875
9	$S_{775/475}$	-0.72	-1.37	2.09	0.9874
10	$S_{875/475}$	-0.74	-2.78	1.84	0.9873

^a Waveband ratio subscripts represent the wavebands divided to calculate the ratios. For each regression, $N = 57$.

concentration (only R^2 values larger than 0.976 were plotted). These model II predictions were found to use higher wavelengths in the numerator and lower wavelengths in the denominator. Table 1 shows the ten prediction models which resulted in the highest R^2 . The R^2 values were within a small range (0.987–0.991) suggesting that the ten models had a similar predictive ability. These prediction models had numerator wavebands wavelengths in the range 725–1025 nm and denominator wavebands wavelengths ranging between 375 and 475 nm. The most frequent denominator waveband wavelength was 425 (seven of the ten models). Fig. 6 shows the prediction of whey fat concentration for model II using sidescatter waveband ratio, $S_{875/425}$. The sidescatter ratio $S_{875/425}$ had an R^2 of 0.99 and a coefficient of variation (CV) of 6.27%. Plot of the sidescatter waveband ratios summarized in Table 1 as a function of fat concentration showed that ratios increased with increasing fat concentration. From this, it follows that the response of infrared light (725–

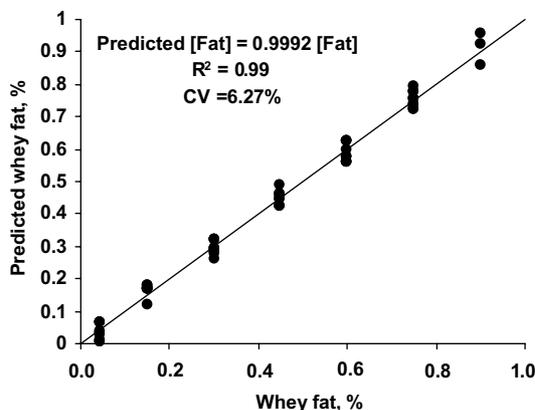


Fig. 6. Prediction of whey fat concentration as a function of the sidescatter ratio $S_{875/425}$ using model II (Eq. (4)). Regression coefficients used for the prediction were: $\beta_0 = -0.80$, $\beta_1 = -0.70$, $\beta_2 = 1.66$, $N = 57$.

1025 nm) to fat concentration was proportionally higher than the response of blue light (375–475 nm). The observed pattern is considered to be related to the aqueous nature of milk fat dispersion in whey (i.e., optical characteristic of both water and whey fat). As already mentioned, the main particles responsible for light scattering in whey are the fat globules. At low fat concentration (i.e., within the single scattering region), sidescatter intensity is linear with concentration (if particle concentration is low enough). However, as fat concentration increases, a point is reached when proportionality between concentration and intensity no longer holds. Thus, sidescatter intensity saturates and will decline even though the concentration increases. Fig. 7 shows that normalized sidescatter intensity measured at 425 nm increased with whey fat concentration until it reached a maximum (saturation) at $\sim 0.7\%$. No saturation was observed in Fig. 7 for the normalized sidescatter intensity at 875 nm. Saturation is obviously reached at lower whey fat concentration for smaller wavelength. Hale and Querry (1973) identified a water absorption minimum at 480 nm. More recently, Pope and Fry (1997) confirmed the existence of a water absorption minimum in the blue region at 418 nm. Hale and Querry (1973) reported that water absorption at 875 nm is ~ 300 times higher than at 425 nm. We hypothesized that higher water absorption of 875 nm light reduces the multiple scattering effect and as a result the maximum intensity for sidescatter light will occur at a higher whey fat concentration for 875 nm light than for 425 nm light. The sidescatter waveband ratio of, for instance, $S_{875/425}$ apparently contains this information and can be used to estimate whey fat concentration in the range of 0–0.9%.

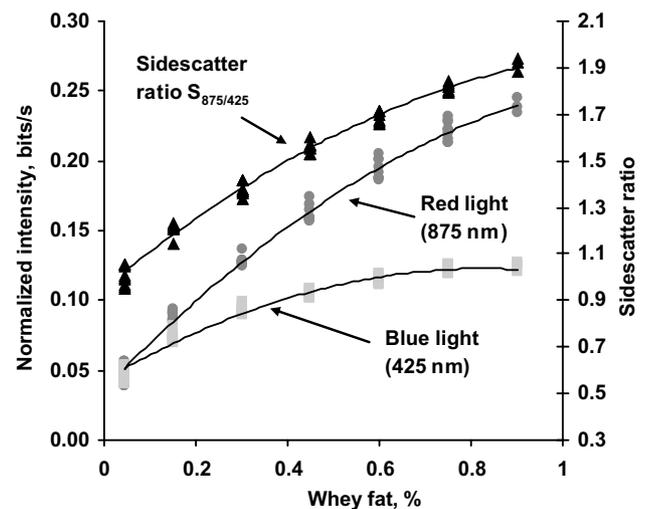


Fig. 7. Change in normalized sidescatter intensities measured at 425 and 875 nm, and in the sidescatter ratio $S_{875/425}$ versus whey fat concentration. Data plotted were for the entire data set ($N = 57$).

3.3. Light extinction coefficients

Light extinction coefficients for whey fat globules were approximated at different wavelengths using the following Beer's law expression:

$$\ln I = \ln I_0 - \alpha r[\text{Fat}] \quad (5)$$

where I is the normalized transmission intensity (bit s^{-1}), I_0 is the initial normalized intensity at zero whey fat concentration, α is the light extinction coefficient for whey fat ($\text{l g}^{-1} \text{cm}^{-1}$), r is the path length (0.6 cm) and $[\text{Fat}]$ is whey fat concentration (g l^{-1}). Fig. 8 shows in a semi log plot the variation of I values as a function of $[\text{Fat}]$. It was noted that the data were nonlinear in the semi log plot which is a result of multiple scattering effect at higher concentrations. Thus, whey fat globules light extinction coefficients were approximated by fitting I and $[\text{Fat}]$ values to Eq. (5) by linear least square regression at a low $[\text{Fat}]$ range (0–2 g l^{-1}) where Beer's law was assumed to apply. Solid straight lines in Fig. 8 show these fits corresponding to wavelengths 425 and 875 nm. Fig. 9 shows the light extinction coefficients obtained for whey fat as a function of the wavelength in the range 375–1025 nm. The light extinction coefficients were in the range $\alpha = 3.457 \pm 0.111 (\text{l g}^{-1} \text{cm}^{-1})$. A maximum ($\sim 3.571 \text{ l g}^{-1} \text{cm}^{-1}$) and a minimum ($\sim 3.251 \text{ l g}^{-1} \text{cm}^{-1}$) light extinction coefficient values were observed in the vicinity of 830 and 425 nm, respectively. The fact that *red/blue* light ratios were found to be better descriptors of fat concentration, could be a result of the existence of maximum and minimum values for light extinction coefficients in the red and blue regions, respectively, for both whey fat and water. Beer's law departs from linearity as whey fat concentration increases as a result of multiple scattering. With multiple scattering, light interacts with several fat globules and travels for longer distances in the medium. Thus, the multiple scattering effect should be less pronounced when the absorption of either fat particles or water is at a maximum. The opposite is also true. It should be noted that both water and fat absorption are

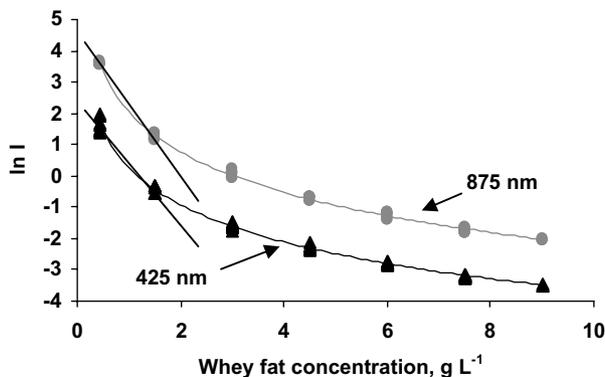


Fig. 8. Fit of experimental normalized transmission intensity and whey fat concentration to Beer's law.

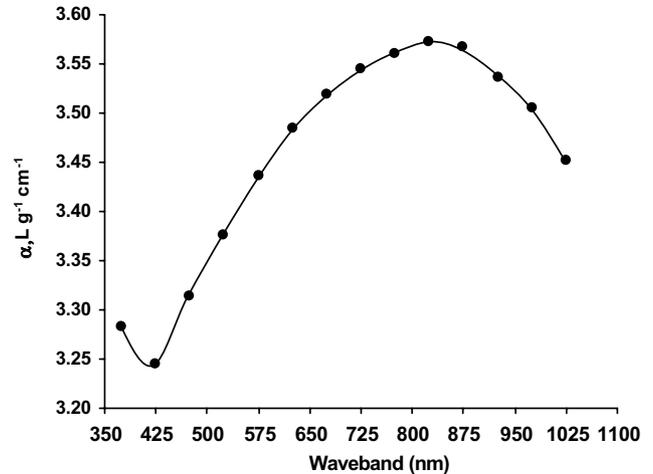


Fig. 9. Change in light extinction coefficient of fat in whey as a function of the wavelength.

at a minimum within the blue region area and maximum within the infrared region. These results show that use of optical waveband ratios for light sidescatter may have potential for measuring low concentrations of fat (0–1%) in cheese whey during syneresis and appear to result from the difference in blue and red scattered light.

4. Conclusions

Normalized spectral sidescatter and transmission response versus whey fat concentration were observed to follow a power law function. Two power law type models were tested for correlating waveband ratios with whey fat concentration. The models using sidescatter waveband ratios were significantly better than for transmission. The models which had the highest R^2 used numerator waveband wavelengths in the range of 725–1025 and denominator waveband wavelengths in the range 375–475. Light extinction coefficients for whey fat were maximum at ~ 830 nm and minimum at ~ 425 nm. Normalized sidescatter intensities saturated at low whey fat concentration for blue light (~ 425 nm) but not for infrared (~ 875 nm). Whey fat concentration was estimated with an R^2 of 0.99 and a CV of 6.27% using model II with sidescatter waveband ratio of 875 and 425 nm ($S_{875/425}$). These results suggest that whey fat in the range 0–1% can be related to the differences in light sidescatter. Thus, a light sidescatter sensor technology may have potential for development of a process control technology for the syneresis process.

Acknowledgment

This study was possible thanks to the collaboration between the Department of Biosystems and Agricultural Engineering (University of Kentucky) and the

Department of Food Technology, Human Nutrition and Bromatology (University of Murcia). We are also very grateful for financial support from the Séneca Project (PB/19/FS/99) and a Séneca Grant from the University of Murcia. During part of this research M. Castillo was supported by a post-doctoral grant from the program MECD/Fulbright from Spanish Ministry of Education Culture and Sports financed by the Spanish Secretariat of State for Education and Universities and co-financed by the European Social Fund. We deeply thank Manuel Vivancos Navarro Cheese Plant for providing the whey samples.

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