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Czarena L. Crofcheck
University of Kentucky, crofcheck@uky.edu

Frederick A. Payne
University of Kentucky, fred.payne@uky.edu

Clair L. Hicks
University of Kentucky, clhicks@uky.edu

M. Pinar Mengüç
University of Kentucky, menguc@engr.uky.edu

Sue E. Nokes
University of Kentucky, sue.nokes@uky.edu

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C. L. Crofcheck, F. A. Payne, C. L. Hicks, M. P. Mengüç, S. E. Nokes

Abstract. *A light backscatter technique using optical fibers to deliver and receive light was investigated for measuring the milkfat content of unhomogenized cream. Light backscatter through cream at wavelengths of 450 to 900 nm was measured for fiber separation distances from 2 to 6.5 mm and for cream containing 10 to ~40 weight percent (wt%) milkfat. Unhomogenized cream (~40 wt% milkfat) was mixed with skim milk (~0.05 wt% milkfat) to yield samples with five different milkfat levels. Three optical response models were tested for correlation with milkfat content: one using the light intensity measurement at a single separation distance, the second using the ratio of the light intensity at two distances, and a third using the light intensity as a function of separation distance based on the backscatter of light in a particulate solution. The calibration equations from all three methods were used to predict milkfat content in the evaluation samples with root mean square errors (RMSEs) of 1.5 to 2.0 wt%. Statistical analysis did not find a significant difference between the three methods. For simplicity, using the ratio of the intensities measured and two different separation distances is attractive for further sensor design.*

Keywords. *Fiber Optic Sensors, Cream, Fat, Light Scattering.*

Improved automation is needed to ensure that U.S. food processing facilities, including dairies, remain competitive in the world economy and to improve product consistency, quality, and safety. Process control allows for tighter production tolerances, increased consistency of food properties, process optimization, improved quality, and savings in raw materials, energy, and waste disposal. Lack of suitable sensors for characterizing the properties of liquid particulate food materials is hindering the implementation of modern process control technologies.

Specifically, there is need for an inline sensor capable of measuring the milkfat content of creams (35 to 45 wt% milkfat) so that the cream separation process can be automated. When raw milk is separated, the two resulting streams are milkfat–rich cream and milkfat–depleted skim milk. After separation, these two streams are then processed or simply mixed to yield various dairy products ranging from butter to freeze–dried skim milk. This study focuses on the high–milkfat products, including heavy cream (40 wt%) and lighter creams (10 or 18 wt%). The process control objective for cream separation is to provide a consistent cream milkfat content by automatically adjusting the stream flow from the separator in response to process changes (e.g., a change in the milkfat content of the incoming milk or in the plant air pressure controlling the valves). Improved process control will minimize milkfat loss in the skim milk stream of the separator. A cream sensor that provides separator control will give better consistency of the product, minimizing heating and cooling costs while improving downstream pumping and cooling operations (personal communications with Tony Suda, ESE Inc., Marshfield, Wisc.). Separator control requires a measurement of the milkfat content in the cream stream and/or the milkfat content in the skim stream.

Milkfat can currently be measured with existing commercial systems, primarily based on Fourier transform infrared spectrometry (Luinge et al., 1993). The required investment of $60,000 to $90,000 for a triple separator system is considered prohibitively expensive for this application. However, it is projected that an automated milkfat control system would have a one–year cost savings of $36,000 to $45,000, depending on production and number of separators (personal communications with Tony Suda, ESE Inc., Marshfield, Wisc.). Existing optical reflectance sensors tend to saturate at high milkfat levels (typically above 6% to 15% milkfat, Payne et al., 1999). The strong light attenuation of 35% to 45% milkfat creams limits transmission sensors. A new technology is needed to provide a technically feasible sensor for this application. Some work has been done using electrical conductivity and capacitive reactance (Lawton and Pethig, 1993) to measure milkfat contents of 0.15 to 51 wt%, but this work has not yet found commercial acceptance.

Fiber optic sensors take advantage of the miniature size characteristic of optical fibers (typically less than 1 mm in diameter) and the small light penetration depths of liquid particulate food materials. They have been shown to provide robust and inexpensive methods for measuring the diffuse reflectance of multiple–scattering fluids (Meeten and Wood, 1993). Fiber optic sensors have been developed for monitor-

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The authors are **Czarena L. Crofcheck, ASAE Member Engineer,** Assistant Professor, **Fred A. Payne, ASAE Member Engineer,** Professor, and **Sue E. Nokes, ASAE Member Engineer,** Associate Professor, Biosystems and Agricultural Engineering Department; **Clair L. Hicks**, Professor, Animal Science Department; and **M. Pinar Mengüç**, Professor, Mechanical Engineering Department, University of Kentucky, Lexington, Kentucky. **Corresponding author**: Czarena L. Crofcheck, University of Kentucky, 128 CE Barnhart Building, Lexington, KY 40546–0276; phone: 859–257–3000, ext. 212; fax: 859–257–5671; ccrofche@bae.uky.edu.

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ing changes in backscatter (diffuse reflectance) during enzymatic coagulation of milk (Payne et al., 1993; Payne, 1995) and the culture of cottage cheese (Payne et al., 1997; Crofcheck et al., 1999). In addition, similar sensors may be used for transition sensing of dairy products, as product lines are switched from water to milk (Payne et al., 1999) and for determining the milkfat content in skim milk (Crofcheck et al., 2000). In this study, we focus on the use of light backscattering, combining the reliability of optical systems with the required ability to measure at high milkfat concentrations.

General Model Development

In cream, light is scattered due to the presence of milkfat globules. When light is directed into a sample and diffused by scattering in all directions, the backscattered intensity decreases as the distance between the emitting fiber and the detecting fiber increases, according to absorption and scattering principles. If scattering is dominant, then the widely used diffusion approximation is valid, and the scattered light distribution in the particle–laden media is described by the following equation (Bolt and ten Bosch, 1993):

$$I(r) = I_0 \frac{\exp(-\bar{\beta} r)}{r^m} = I_0 \frac{\exp(-C_1 \text{ fat } r)}{r^m} \qquad (1)$$

where

- I_0 = apparent intensity at radial centerline of emitting fiber.
- $I(r)$ = light intensity as a function of radial distance from the emitting fiber.
- r = separation distance between the emitting and the detecting fiber.
- $\bar{\beta}$ = backscatter light coefficient ($\bar{\beta}$ = C_1 fat).
- C_1 = fitting parameter.
- fat = wt% of milkfat.
- m = exponent relating light diffusion in the radial direction.

The backscatter light coefficient ($\bar{\beta}$) is based on the ability of the sample to scatter light and depends on the number and properties of the particles in the sample. As the number of particles increases, the value of $\bar{\beta}$ increases. Hence, it may be assumed that $\bar{\beta}$ is directly related to the milkfat level ($\bar{\beta}$ = C_1 fat).

The value of *m* depends on whether the detector is placed in the intermediate area (*m* = 2) or the diffusion area (*m* = 2) (Bolt and ten Bosch, 1993). The diffusion area is defined as the area in which sufficient multiple scatterings have taken place so that the diffusion approximation is valid. The intermediate area makes up the area between the source and the beginning of the diffusion area, where the multiple light scattering approximations are not yet valid. Within the intermediate area, short–path backscattered photons are dominant; within the diffusion area, long–path diffusely scattered photons dominate (Schmitt and Kumar, 1996; Crofcheck, 2001). The distance between the source and the diffusion area depends on the scattering properties of the medium. As the scattering in the medium increases, the extent of the intermediate area decreases. Therefore, the appropriate value of *m* and the distance between source and detector will depend on the scattering properties of the medium.

Figure 1 shows the fiber optic measurement of two intensities at two radial distances (r_1 and r_2) from the emitting fiber. The intensity measured at a single distance (r_1) reduces equation 1 to:

$$I(r_1) = C_2 \exp(C_3 \text{ fat}) \qquad (2)$$

While the ratio of the intensities at two radial distances (r_1 and r_2) reduces equation 1 to the following equations:

$$\begin{aligned} \frac{I(r_1)}{I(r_2)} &= \left(\frac{r_2}{r_1}\right)^m \exp[C_1 \text{ fat}(r_2 - r_1)] \\ &= C_4 \exp(C_5 \text{ fat}) \\ &\cong C_6 + C_7 \text{ fat} \end{aligned} \qquad (3)$$

Here, the exponential can be approximated using a linear equation since r_1 and r_2 are close together and C_5 is much smaller than C_4. A linear response for milkfat content would be advantageous to implement electronically. Finally, by measuring the intensity at several radial distances (r_n), the resulting intensities can be fit using equation 1:

$$I(r) = C_8 \exp(C_1 \text{ fat } r) r^{C_9} \qquad (4)$$

Objectives

The current study was undertaken to study the backscatter of light in cream based on these three models: the intensity measured at a single distance (eq. 2), the ratio of two intensities measured at two distances (eq. 3), and the intensities at several distances fit to the backscatter equation (eq. 1). By investigating these models, the feasibility of using a fiber optic technique to determine the milkfat content in cream can be determined. Before further sensor development can take place, it would be helpful to know what separation distances and wavelengths are most appropriate. Once the appropriate wavelength is determined, a light–emitting diode (LED) with a similar wavelength can be tested, and hopefully the resulting sensor performance will be a further improvement. The specific objectives of the study were to:

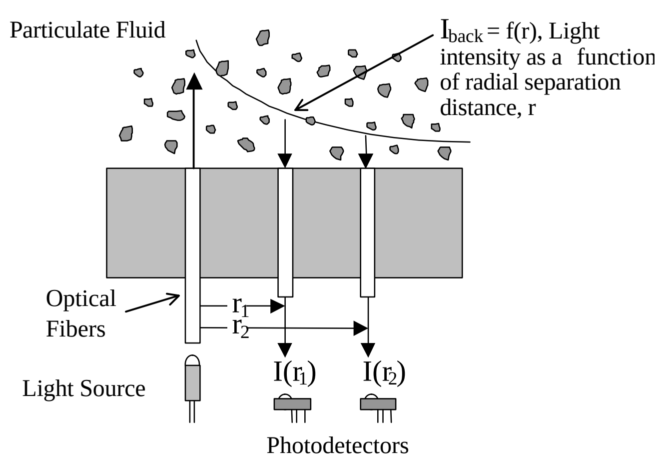

Figure 1. Schematic of probe concept for measuring the backscatter coefficient.

1. Determine the relationship between the milkfat content in cream and light backscattering using three models described above (calibration).
2. Evaluate the calibrated model for predicting the milkfat content in cream.

Materials and Methods

Experimental Equipment

A fiber optic backscatter system was assembled to accurately adjust the distance between a light delivery fiber and a light receiving fiber with submicron resolution. The system consisted of a stainless steel base that supports two translation stages such that fiber optics can be mounted on the stages and immersed into the sample of interest. The optical fibers (HCG–M0365T, Spectran Specialty Optics Company, Avon, Ct.) had a numerical aperture of 0.22 ±0.02 and a core–plus–cladding diameter of 430 +5/–10 μm. The terminal ends of the fibers were bonded into small stainless steel blocks using epoxy (BIPAX, TRA–Bond, TraCon Inc., Bedford, Mass.), and the distal ends were polished using 2 μm polishing paper. The small steel blocks were then mounted onto the translation stages so that the two fibers were axially aligned.

The light delivery fiber was mounted onto a multi–axis stage (461–XYZ–M, Newport Corp., Irvine, Cal.) with fine adjustment screws (AJS–0.5, Newport Corp.), allowing for fiber alignment in three directions with a resolution of less that 1 μm. The light detecting fiber optic was mounted on a single–axis stage (462–X–M, Newport Corp.) with a long–travel, high–resolution micrometer (HR–1, Newport Corp.) so that the second fiber could be moved away from the first fiber in accurate measurable increments. The fibers were aligned by sight with the help of a digital camera (Javelin SmartCam, Javelin Technologies, Torrance, Cal.) and image capturing software (DT–Acquire image capture program, Data Translations, Marlboro, Mass.), which was also used to measure the resulting fiber separation distance.

The light source was a tungsten halogen light source (LS–1, Ocean Optics, Inc., Dunedin, Fla.). Light reaching the second fiber was detected using a spectrophotometer (S2000, Ocean Optics, Inc.). The incoming light was transmitted first to the spectrometer housing, where it was broken into separate wavelengths before hitting the charge–coupled device (CCD, 2048–pixel Sony ILX511 linear sensor). The CCD collected photons at each pixel location during an integration time set by the computer software (OOIBase32, Version 1.0, Ocean Optics, Inc.). After the integration time had elapsed, the number of photons collected in the CCD wells, represented by a voltage, was transmitted from the CCD to an analog input board in the computer, where the voltage was then converted to a 12–bit digital value. Intensities (in terms of bit counts) for wavelengths from 400.15 to 1105.25 nm (2.27 pixels/wavelength) were recorded for each scan. Integration times ranged from 5,000 to 20,000 ms depending upon the light level (lower light levels, longer integration times). Intensities readings from the spectrometer were corrected with a dark reading, divided by the integration time, and referred to as backscatter intensities (I_{back}).

Sample Preparation

Homogenized and pasteurized skim milk was obtained from a local grocery store (Kroger Company) and unhomogenized cream (~40 wt% milkfat, the actual wt% milkfat was dependent on the separator efficiency at the time of separation) was obtained from Winchester Farms Dairy (Winchester, Ky.; also a Kroger Company). Cream samples with milkfat contents of 10, 18, 26, 34, and ~40 wt% milkfat were prepared. The milk and cream were stored at 4°C, and all tests were performed within four days of the purchase date. The milkfat content of the skim milk and cream samples were determined using a Milkoscan FT 120 (Foss Electric, Denmark) with a specified accuracy of ≤1.0 % (Cv% = standard deviation divided by the average of 100 samples).

A sample of reconstituted skim milk (8.7 wt% total solids) was prepared before each experiment and used as a standard (the same freeze–dried skim milk was used for the duration of the study). By measuring a sample with essentially the same size and number of particles, the effect of time and sensor drift could be quantified. Freeze–dried skim milk was prepared by freezing skim milk at –23°C for at least 3 hours and freeze drying (Vitris Repp, Detroit, Mich.) for at least 24 hours with a condenser temperature of –55°C. Care was taken to ensure that the sample temperature remained below 37°C to prevent the denaturiztion of the milk protein. Freeze–dried skim milk was double bagged in plastic and stored at 0°C until use.

Experimental Procedure

Three tests were performed for calibration purposes and three for evaluation purposes, each with a different batch of milk. The intensity of light backscatter for wavelengths between 400 and 1000 nm in cream samples with milkfat levels of 10, 18, 26, 34, and ~40 wt% was measured at radial distances of 2, 2.5, 3, 3.5, 4, 5, and 6 mm. The order in which the various milkfat level samples were tested was completely randomized, as well as the order in which the distances were tested. The light backscatter profile for a reconstituted skim milk standard was measured before and after each milk test to track changes and drifts in the fiber optic system.

Cream samples were placed in a 150–ml glass beaker, and the beaker was placed in a water–jacketed container (fig. 2). Samples were continually stirred using a magnetic stirring plate and rod. A sample temperature of 4°C was maintained by the water–jacketed stainless steel container (8.25 × 8.25 × 10 cm) connected to a recirculating cooling–bath (Brinkman Instruments, RM20, Westbury, N.Y.). A thermometer was placed in the sample to monitor temperature. The appropriate integration time (resulting in a maximum peak of about 2000 counts, the detector limit was 4095) and scan averaging were chosen (S2000 Quick Start Manual). Once the spectrophotometer signal had equilibrated, the scan was collected and saved to a file.

Data Analysis

Intensities (in terms of counts) of light at specific wavelengths of interest (450 to 900 nm by 10 nm increments) backscattered through the milk samples at various distances were obtained from the saved data file. Using the first three sets of data, the ratio calibration equations (eq. 3) were obtained by utilizing a least squares method, and the single distance (eq. 2) and the backscatter (eq. 1) calibration

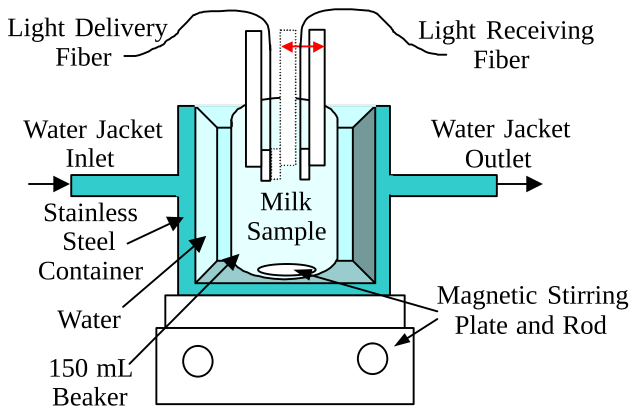

Figure 2. Schematic of the sample container in the fiber optic backscatter measurement system.

equations were determined using a non–linear regression. For each approach, the best wavelength and separation distance combination was chosen based on the minimum of the summation of the standard error of the individual coefficient divided by the value of the coefficient. The resulting three calibration equations were then applied to the evaluation data (the last three sets of data), and the predicted milkfat values were compared with the observed milkfat values using the root mean squared error (RMSE). Evaluation data were not used to calibrate the models.

Results and Discussion

Typical backscatter intensity responses versus wavelength for different milkfat levels at a constant separation distance of 2.5 mm are shown in figure 3. Table 1 summarizes the three models, including the sensor design parameters (wavelength and separation distance) and the calibration and evaluation statistics.

For a single separation distance, the best fit of the data and predictive power of the model was found using the intensities at a wavelength of 480 nm and a separation distance of 1.5 mm. The calibration and evaluation data are shown in figure 4 with the single–distance calibration curve (table 1). Although the fit is adequate, using an exponential model in sensor development can be electronically difficult.

The possibility of using a linear equation was pursued by testing the ratio of two intensities measured at different distances (eq. 3). This approach is adaptable to self–calibration, which is not inherent in the single–intensity approach. The best–fit prediction equation was found to be a linear relationship between the ratio of the backscatter intensities at 600 nm measured at separation distances of 2 and 5.5 mm. The calibration and evaluation data are shown in figure 5 with the linear calibration curve (table 1). The R^2 values for several wavelengths and intensity ratios are shown in table 2. Notice that the R^2 values for a wavelength of 520 nm remained relatively high, reaching a maximum with an intermediate separation distance of 2 mm (the ratio of the intensity at 2 mm divided by the intensity at 4 mm), while the R^2 values for the shorter wavelength of 470 nm reached a maximum with a shorter separation distance of 0.5 mm, and the R^2 values for the longer wavelengths (600 and 700 nm) reached a maximum with a longer separation distance (3.5 and 4.5 mm, respectively). These results indicate that the appropriate separation distance for the sensor depends on the choice of wavelength, illustrating that as the wavelength changes the separation distance required for equation 2 to be valid changes as well.

In order to test the predictive power of the backscatter equation for each wavelength, data from the first three experiments were fit to equation 1. The best fit was obtained at a wavelength of 520 nm, as shown in figure 6, where m = 2.16 and $\overline{\beta}$ = –0.02 fat. The value of m for the remaining wavelengths remained above 2, indicating that for these milkfat levels and operating distances the diffusion approximation is valid (m = 2). Data from the last three experiments were used to predict the amount of milkfat based on the backscatter using the above calibration with an RMSE of 1.5 wt%.

To compare the predictive power of the three models described above, an analysis was performed to determine if there was a significant difference between the predicted and observed milkfat contents for the validation samples. Data were fit to a line and a standard F test was used to test the null hypothesis, Ho: intercept = 0 and slope = 1 (Teng, 1981). With a resulting F statistic of 0.550 for the single–distance model, 0.709 for the ratio model, and 0.190 for the

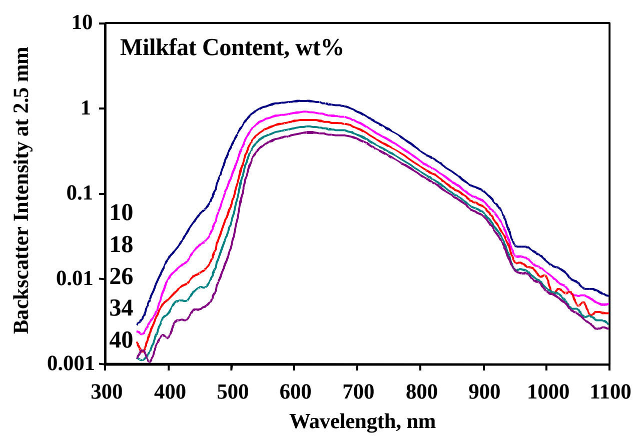

Figure 3. Typical backscatter intensity response as a function of wavelength at a separation distance of 2.5 mm and for milkfat levels of 10, 18, 26, 34, and 40 wt%.

Table 1. Summary of the three backscatter models with statistics.

		Fitting parameters						
Model	Equation	a ±SE	b ±SE	c ±SE	Wavelength (nm)	Distance (mm)	F	RMSE (wt%)
Single distance	$I_{back} = a\ e^{-b\ fat}$	0.229 ±0.0124	0.109 ±0.0044		480	1.5	0.550	1.5
Ratio	$I_{back,ratio} = a\ fat + b$	1.10 ±0.018	6.72 ±0.491		600	2 / 5.5	0.0709	2.0
Backscatter equation	$I_{back} = a\ e^{-b\ fat}\ r^{-c}$	7.92 ±0.406	0.0200 ±0.00037	2.16 ±0.0507	520	2.5 to 6.5	0.190	1.5

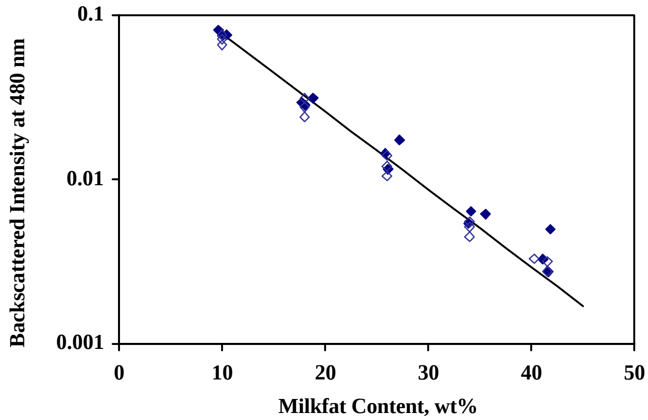

Figure 4. Backscatter intensities at a separation distance of 1.5 mm and a wavelength of 480 nm plotted versus fat content. Calibration data shown as closed symbols and evaluation data shown as open symbols.

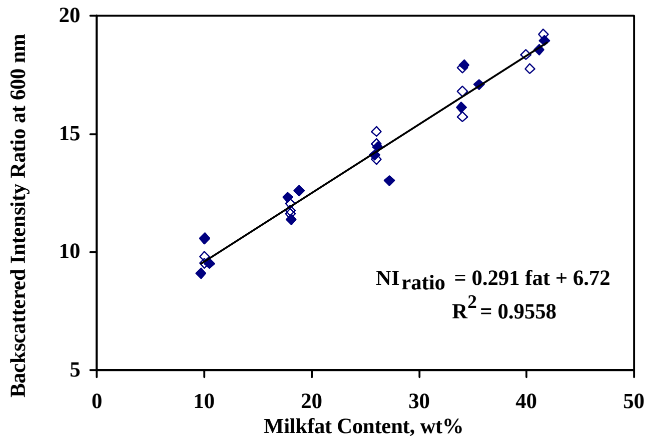

Figure 5. Calibration plot of fat content versus the ratio of the backscatter intensities at 600 nm and two different separation distances (2 and 5.5 mm). Calibration data shown as closed symbols and evaluation data shown as open symbols.

Table 2. R^2 values for the ratio model at various separation distances and wavelengths.

Ratio of distances	Wavelength			
	470 nm	520 nm	600 nm	700 nm
2/2.5	0.9597	0.9169	0.6644	0.5814
2/3.0	0.9499	0.9302	0.7012	0.5836
2/3.5	0.9176	0.9304	0.8492	0.7878
2/4.0	0.5573	0.9432	0.8087	0.6960
2/4.5	0.5240	0.9161	0.8689	0.7535
2/5.5	0.1874	0.8643	0.9558	0.8645
2/6.5	0.1512	0.9130	0.9355	0.8759

backscatter model, and F_{crit} ($\alpha = 0.05$) values of 3.81, 3.81, and 3.9 (Snedecor and Cochran, 1989), respectively, the null hypothesis cannot be rejected in all three cases. Based on this evidence, it was concluded that the predicted and observed milkfat content values are not significantly different and the prediction equations can all be considered a good fit. The prediction equation based on the backscatter ratio resulted in the lowest F value, yet the RMSE found by comparing the predicted and observed values is the highest of the three cases.

From these results, it appeared that all three models provide adequate prediction of milkfat level. However, using

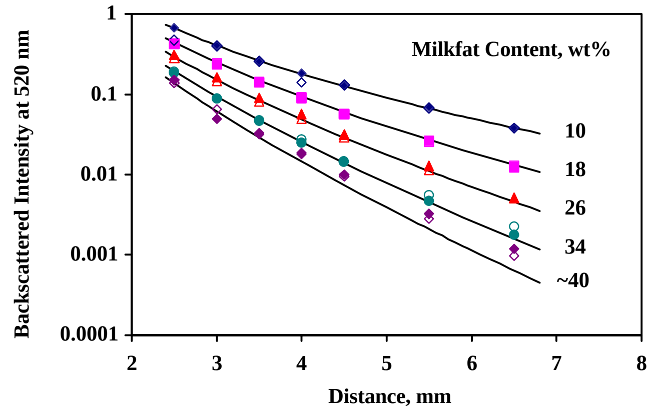

Figure 6. The calibration data and the resulting backscatter model, where the backscatter intensity at 520 nm is a function of fat content and radial distance, as shown in equation 1.

the ratio model would have advantages. It is less complicated than the backscatter equation model and possibly more self–calibrating than the single–distance model. It is believed that the use of a dedicated sensor will further improve the prediction power of these models.

There was very little drift in the backscatter of light from the fiber optic source through the standard 8.7% total solids skim milk sample. Only on the last day of experiments did the intensity need to be adjusted because the fiber cables were inadvertently shifted. The coefficient of variation of the standard measurements were <7.5 % prior to correcting the last experiment and <5.5 % after correction.

Conclusions

There was a significant relationship between the amount of milkfat in cream and the amount of light backscatter. Three different models relating backscatter measurements at various radial distances to milkfat level were developed. These models were based on a single radial distance intensity (eq. 2), the ratio of intensities at two different radial distances (eq. 3), and the intensity as a function of radial distance (eq. 1). For the model based on intensity at a single distance, the intensities at 480 nm with a separation distance of approximately 1.5 mm resulted in an exponential relation with RMSE values of 1.5 wt%. For the model based on the ratio of two intensities, the best wavelength was at 600 nm, and the best ratio was obtained with distances of 2 and 5.5 mm with an RMSE of 2.0 wt%. The best wavelength for the backscatter model was 520 nm with an RMSE of 1.5 wt%.

The results from the ratio portion of the study indicated that the exponential equation could be approximated linearly if the appropriate separation distance was chosen for the wavelength of interest. The shorter wavelengths required shorter emitter/detector separation distances (<1.5 mm for 470 nm). The intermediate wavelength of 520 nm worked well with the separation distances in this study (0.5 to 4.5 mm). The longer wavelengths required longer separation distances (>3 mm for 600 and above). Unfortunately, the low signals at the longer separation distances were difficult to detect. Hence, the best wavelengths for further investigations are in the 470 to 600 nm range, and the best separation distances depend on the choice of wavelength.

The value of *m* for 520 nm was found to be 2.16, indicating that with these milkfat levels and separation distances the amount of scattering does allow for the application of the diffusion approximation. The fit of the backscatter model to the data indicated that such a model could be used as a tool for sensor design, since it may be used to simulate intensities at a large number of distances. However, this model may also be too complicated to be used directly in the sensor because several intensities need to be measured for a 3–parameter model (as opposed to two intensities with a 2–parameter model).

Using the ratio model for further sensor testing seems most appropriate. The error associated with these measurements was similar to the other measurement techniques, yet the linear nature of the technique would make sensor design easier than with the other approaches. Considering the errors associated with these experiments, further improvement of the sensor is necessary before implementation in a commercial facility (prediction within 1 wt% would be preferable). Based on these results, a prototype sensor can be fabricated with a fixed fiber separation distance and a light source (LED) with a fixed wavelength range. With this prototype sensor, work can be done to characterize sensor performance with different fat types (such as homogenized) and temperatures. These studies are currently underway in our laboratory.

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