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Light Backscatter of Milk Products for Transition Sensing Using Optical Fibers

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LIGHT BACKSCATTER OF MILK PRODUCTS FOR TRANSITION SENSING USING OPTICAL FIBERS

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ABSTRACT. *Transition sensors are needed, particularly in the dairy industry, for detecting transitions in pipe flow systems from product-to-water or product-to-product (such as from chocolate to vanilla ice cream mix). Transition information is used to automatically sequence valves to minimize product waste. Optical fibers were used to measure light backscatter between 400 and 950 nm as a function of milk concentration in water and milkfat concentration in milk. The normalized response (100% for product and 0% for water) as a function of product concentration in water was approximately logarithmic for skim milk between 400 and 900 nm and approximately linear for milk containing 1, 2, and 3.2% milkfat. The backscatter ratio (response relative to that for skim milk) as a function of milkfat in milk was wavelength dependent with longer wavelengths being more sensitive. The backscatter ratio at 900 nm for milk containing 3.2% homogenized fat was nearly four times that for skim milk. Backscatter ratio saturated (minimal response with increased milkfat) at 8% milkfat for homogenized cream and 16% milkfat for unhomogenized cream. Light backscatter for near infrared wavelengths around 900 nm was found ideally suited for transition sensing of dairy products and was found particularly sensitive to milkfat content. Light backscatter was found less suitable for discriminating between high milkfat products.*

Keywords. *Transition, Sensor, Fiber optic, Milk, Dairy, Clean-in-place.*

Improved inline sensors are needed in the food industry for process automation to improve product quality and consistency, decrease process control tolerances, and increase processing efficiency through reductions in materials and energy usage and waste production. One aspect of food process control in liquid piping systems is the detection of a product concentration transition from one product to another or from product to water and vice versa. Detection of the product concentration transition provides information for automatic sequencing of downstream pipe valves. An obvious benefit of automatic sequencing is the minimization of waste since diverter valves can be activated efficiently. A typical application for a "transition sensor" is in dairy plants for controlling the product stream from a pasteurizer. The pasteurizing system schematic shown in figure 1 is based on a description by Seiberling (1997) and was modified to represent the system installed at a local processing plant (Winchester Farms Dairy, Winchester, Ky.).

During startup, water is recirculated through the pasteurizing system until the pasteurization temperature upstream of the holding tube is reached. Water is fed into the balance tank and pumped through the regeneration section, homogenizer, heating section, hold tube and then diverted by the flow diverter valve back to the balance tank. When pasteurization temperature is reached the water is diverted through the flow diverter valve to the regenerator section, cooler section, and through the backpressure valve, across an optical sensor (proposed location) and to the recycle valve where it is directed to the drain. When the water level in the balance tank reaches a minimum, operators manually activate the flow of raw milk into the balance tank. The operators use stopwatches to determine the product transient time through the pasteurizer and manually divert the output stream from the drain to the pasteurized product outlet. A transition sensor is needed to determine this water-to-milk transition and

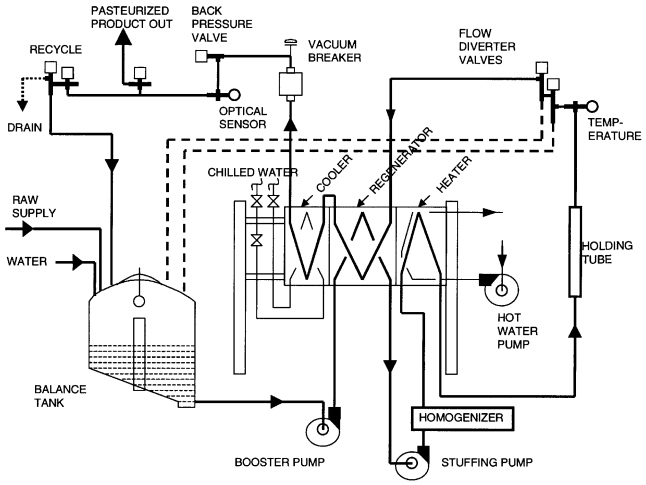

Figure 1–Process flow diagram for a milk pasteurizing system at Winchester Farms Dairy showing a typical application for a transition sensor.

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automatically close the recycle valve and send the product through the pasteurized product out valve. Likewise, at the end of a product batch, water is used to force milk through the system and a sensor is needed to determine the milk-to-water transition. A typical product-to-product transition would be the transition between chocolate and vanilla ice cream mixes being sent to a freezer.

Optical sensors have recently been introduced to detect transitions (FiberView manufactured by Reflectronics Inc., Lexington, Ky.; Optec manufactured by Papertech Inc., North Vancouver, B.C., Canada; FluidScan distributed by AW Company, Franksville, Wis.). Two different optical designs are used for measuring backscatter. One directs light from an LED through a lens at an angle and measures the light backscatter through the lens at an angle. This design reduces specular reflectance from the lens-product interface. The second is the use of optical fibers that terminate at the product surface. This design eliminates specular reflectance and measures only the backscatter from light that has penetrated into the product. All of these sensors are zeroed with water (no backscatter, dark environment) and calibrated to full scale with the product to be identified.

Difficulties arise when milk products with different composition are processed using the same equipment. Backscattered light increases as the milkfat increases (Payne et al., 1993). (Milkfat is used in this article to describe the concentration of fat in milk.) Thus, if multiple products with differing milkfat contents are processed, the optical sensor must be calibrated using the range of backscatter from all products. Correct use of optical sensors for transition detection requires knowledge of the relationship between the optical sensor's response and the concentration of milk in water as well as the concentration of fat in milk or milkfat.

Milk is a complex biological fluid composed mainly of water, fat, protein, lactose, citric acid, and inorganic compounds. Although all of these constituents play at least a minor role in the light scattering, the fats and proteins dominate. The most prevalent protein in milk is casein, which exist as a colloidal dispersion of particles known as casein micelles. The "roughly" spherical casein micelles are made up of spherical casein submicelles. The average particle size of casein micelles ranges from 130 to 160 nm and fat globules range from 100 to 1500 nm (Ruettiman and Ladisch, 1987). Fat globule size and concentration have been shown to have large effects on absorption measurements (Ben-Gera and Norris, 1968). The fat globules, as a result of their large size, are responsible for a majority of the light scattering. The scattering of light in milk therefore depends upon the number and size of casein and fat particles, the wavelength of incident radiation, and the difference in refractive index between the different kinds of particles and solvent (Walstra and Jenness, 1984).

Objectives

The shape of a backscatter response curve between milk and water is not known. Nor is the effect of light wavelength on sensor response. The goal of this work was to document the light backscatter response using optical fibers for both water-to-product and product-to-product transitions for a range of light wavelengths. This research should provide information about the response of light wavelengths for selecting sensor diodes and the response to product concentrations for guiding the selection of cutoff points. The specific objectives for this research were to measure light backscatter using optical fibers to:

1. Determine light backscatter as a function of milk product concentration in water for milk containing 0.05, 1, 2, and 3.2% milkfat, homogenized cream (half-and-half, 10% milkfat) and unhomogenized cream (40% milkfat) over light wavelengths of 400 to 950 nm.
2. Determine light backscatter as a function of milkfat in milk for milk containing 0.05, 1, 2, and 3.2% milkfat, homogenized cream, and unhomogenized cream over light wavelengths of 400 to 950 nm.

Materials and Methods

A fiber optic probe was designed and fabricated to measure light backscatter in food products. The probe as shown in figure 2 consisted of two optical fibers with one optical fiber used to transmit light to the sample and the other to return light to a photo detector. The fibers used (Product HGG-M0550T, Spectran Specialty Optics Company, Avon, Conn.) had a silica core (550 μm diameter), silica cladding (600 μm diameter) and a polymer hard coat (630 μm diameter). The fibers were epoxied into a stainless steel distal tip and polished. The fibers had a center-to-center spacing of 810 μm. A tungsten halogen light source (LS-1, Ocean Optics, Inc., Dunedin, Fla.) was connected to the probe using a 400 μm fiber cable. The fiber optic probe was connected to the spectrometer using standard SMA connectors.

Figure 3 shows the main components of the data collection system. Light backscatter was measured using a dual miniature fiber optic spectrometer composed of a master and slave unit (model SD1000 with L1 lenses, Ocean Optics, Inc., Dunedin, Fla.). Both spectrometers had a holographic grating that dispersed light across a linear array of 1024 CCD (charge-coupled device) detector elements. The master unit had a bandwidth of 350 to 850 nm, a grating of 600 lines, and reported wavelengths from 323.47 to 882.29 nm. The slave unit had a bandwidth of 550-1000 nm with a GG475 Filter, a grating of 600 lines, and reported light wavelengths from 507.83 to 1020.82 nm. The spectrometer was connected to an analog to digital conversion (ADC) board (CIO-AD16 Jr., Computer boards, Inc., Mansfield, Mass.) installed in a P5-120 MHz computer (Gateway 2000, North Sioux City, S.Dak.). The data acquisition software (SS.EXE of

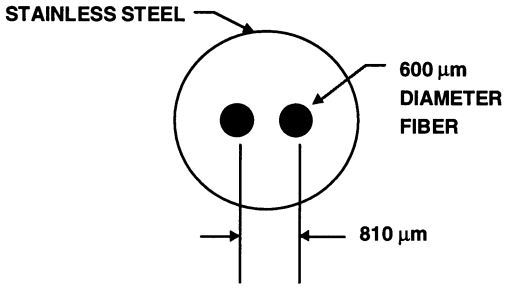

Figure 2–Schematic of the distal tip of the fiber optic backscatter sensor.

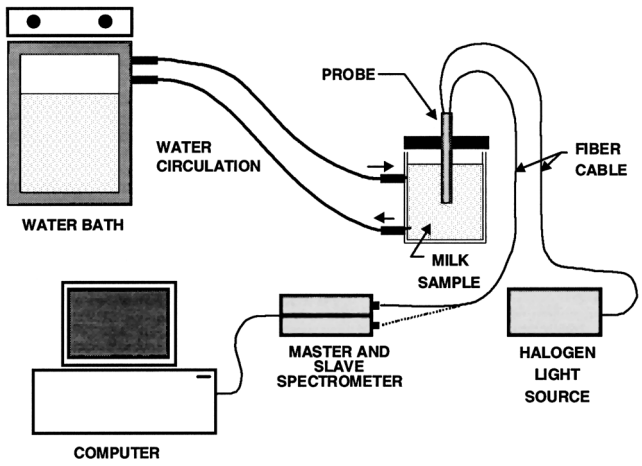

Figure 3–Schematic of the system used to measure light backscatter.

SpectraScope, Version 2.2 (8/94), Ocean Optics, Inc. Dunedin, Fla.) ran the ADC expansion board. The spectrometer sampling frequency was adjusted for each milk composition to obtain a response signal of 75% of full scale (~3000 bits of 4096 bits) for the most sensitive wavelength with an undiluted sample.

Each spectral scan, S(λ), recorded by the SpectraScope software was the average of 7 individual scans. The signal response desired, as stated in Objective 1, was best viewed by normalization to zero for water (dark environment, no backscatter) and unity for 100% product. The normalized response, $R_N(\lambda)$, was calculated from the scans using the following equation:

$$R_N(\lambda) = \frac{S(\lambda) - S_D(\lambda)}{S_{REF}(\lambda) - S_D(\lambda)} \quad (1)$$

where

$S(\lambda)$ = spectral scan taken for the respective milk concentrations

$S_D(\lambda)$ = dark spectral scan taken with the halogen light off

$S_{REF}(\lambda)$ = spectral scan of undiluted product

Skim milk (0.05% milkfat) and homogenized milk having 1, 2, and 3.2% milkfat, all with the same date code, were purchased from a local grocery store (produced by Kroger Co., Winchester Farms Dairy, Winchester, Ky.) and stored at 4°C until tested. Homogenized cream (half-and-half, 10% milkfat) and unhomogenized cream (40% milkfat) were obtained directly from the Kroger Company in one gallon containers. Samples were preheated to 25°C in a water bath (Lauda Refrigerating Circulators RMS-20, Brinkman Instruments Inc.), poured into a water-jacketed stainless steel container, and maintained at 25°C for the duration of data collection. The fiber optic probe was manually immersed into 1700 mL milk sample to a minimum vertical depth of 25 mm.

Diluted product samples were prepared by mixing the dairy product samples with deionized water (by mass) to obtain the product concentrations of 100, 64, 32, 16, 8, 4, and 2%. These increments were selected because a logarithmic response was expected. Products were tested sequentially in the above dilution sequence and reversed for each following replication. Scans were replicated at least three times for all products. All replications for a specific fat level were performed on the same day using the same milk lot. The normalized spectral scans were averaged for graphical presentation.

The effect of fat in milk on sensor response was desired (Objective 2). The effect of fat was analyzed by normalizing the signal to unity for skim milk. The spectrometer sampling frequency was adjusted to 75% full-scale response using milk with 3.2% milkfat, collecting a dark scan, and then collecting scans for milkfat concentrations of 3.2, 2, 1, and 0.05% (skim milk). Scans were replicated twice on the same day. The backscatter ratio was calculated using the following equation:

$$BR(\lambda) = \frac{S(\lambda) - S_D(\lambda)}{S_{SKIM}(\lambda) - S_D(\lambda)} \quad (2)$$

where

$S(\lambda)$ = spectral scan taken for the respective milk products

$S_D(\lambda)$ = dark spectral scan taken with the halogen light off

$S_{SKIM}(\lambda)$ = spectral scan of skim milk

Likewise for homogenized and unhomogenized cream, the spectrometer sampling frequency was adjusted to 75% full-scale response for each product and then scans were taken for the product and skim milk. All replications were performed on the same day.

The above test suggested that a more complete picture of the effect of fat on signal response would be obtained by testing over smaller increments of fat. Thus, the backscatter ratio as a function of homogenized and unhomogenized cream concentration in skim milk was determined. Samples were prepared by adding homogenized cream and unhomogenized cream by weight to skim milk and then collecting a scan. Samples were prepared for fat levels of 0.05 (skim milk), 0.1, 0.2, 0.4, 1, 2, 4, 8, and 10% for homogenized cream and at the additional concentrations of 12, 16, 20, 24, 28, 32, 36, and 40% for unhomogenized cream. The scans were replicated at least three times and averaged for graphical presentation at wavelengths of 550, 600, 650, 700, 750, 800, 850, and 900 nm.

Results and Discussion

Typical scans for various milkfat levels are presented in figure 4 for both the master and slave spectrometers. The optical response was largest between 500 and 800 nm. The optical responses above 950 nm and below 400 nm were considered of insufficient magnitude for accurate measurement and were not reported.

The normalized backscatter scans as a function of wavelength are shown in figures 5 and 6 for skim and 3.2% milk, respectively. Signal saturation was observed for 3.2% milk as shown in figure 6 at a product concentration of 64% for wavelengths at and below 450 nm. Figure 6 also shows that a 50% normalized response (an idealized point to activate a valve change) was obtained at 400 and 900 nm with a 16 and 32% product concentration, respectively.

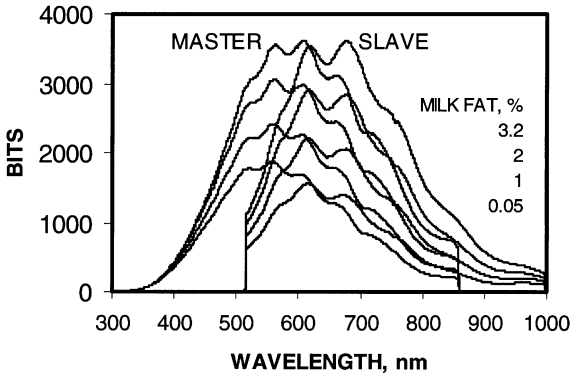

Figure 4–Spectrometer data for homogenized milk containing 0.05, 1, 2, and 3.2% milkfat.

This is nearly opposite the normalized response shown in figure 5 for skim milk (approximately 24 and 16%, respectively). Thus, a fiber optic sensor, which is activated at 50% signal response, would switch at a product concentration of 16% for skim and 32% for 3.2% milk at a wavelength of 900 nm. The normalized response at 900 nm appears logarithmic with skim milk but appears linear for 3.2% milkfat. This suggests that the scattering properties for fat are much different than casein at 900 nm wavelength.

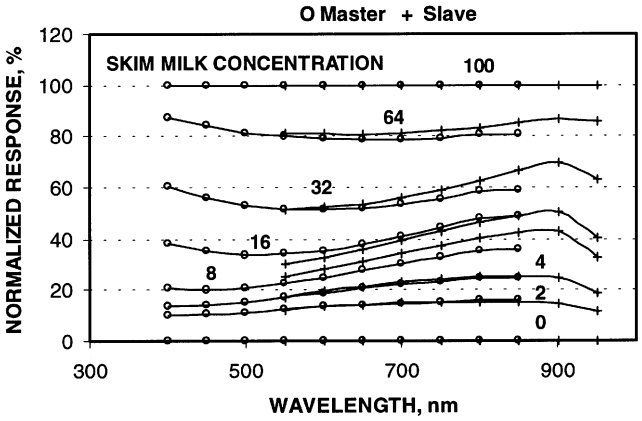

Figure 5–Normalized response for skim milk (0.05% milkfat) as a function of light wavelength.

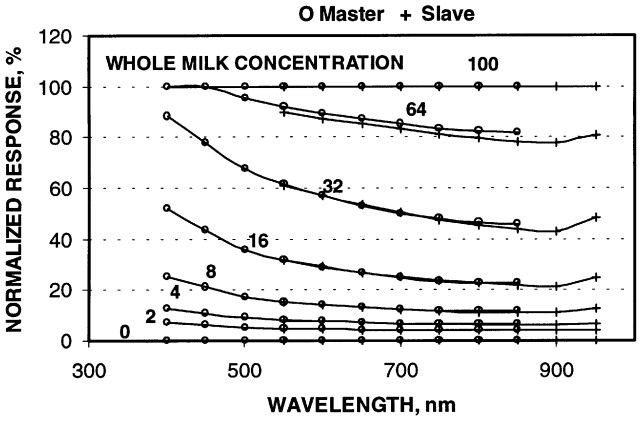

Figure 6–Normalized response for homogenized milk containing 3.2% milkfat as a function of light wavelength.

The normalized responses were plotted as a function of product concentration for wavelengths of 400, 500, 600, 700, 800, and 900 nm and are shown in figure 7a to 7f for skim, 1, 2, and 3.2% milkfat, homogenized cream, and unhomogenized cream, respectively. Figure 7e shows saturation of the normalized response above 64% homogenized cream concentration (6.4% milkfat for half-and-half) and figure 7f shows saturation above 32%

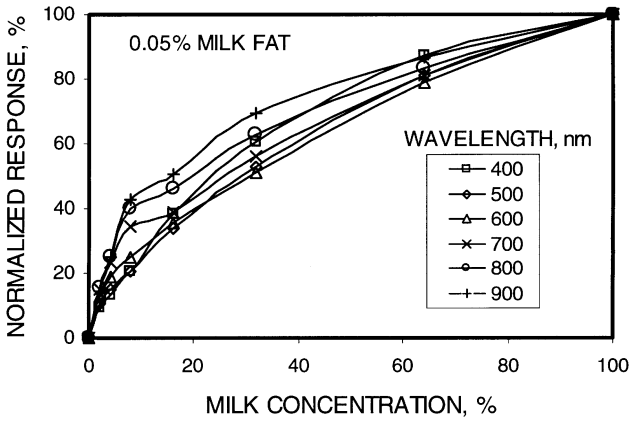

Figure 7(a)–Normalized optical response as a function of milk concentration for skim milk.

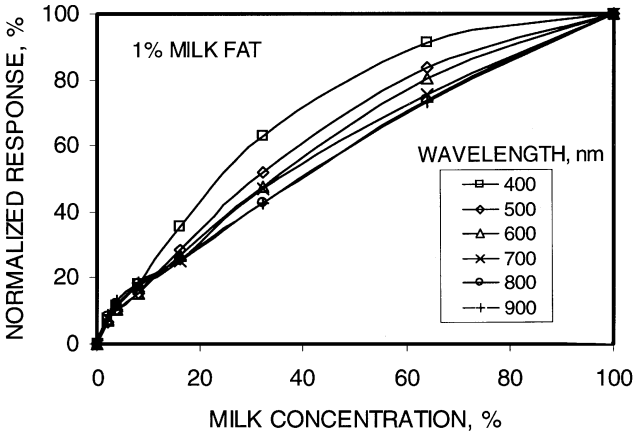

Figure 7(b)–Normalized optical response as a function of milk concentration for milk containing 1% milkfat.

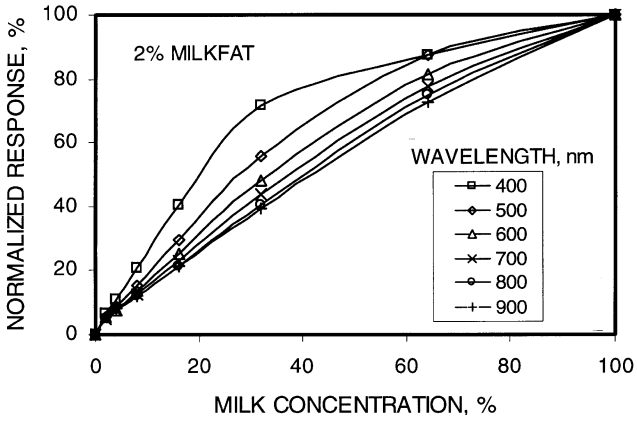

Figure 7(c)–Normalized optical response as a function of milk concentration for milk containing 2% milkfat.

unhomogenized cream concentration (12.8% milkfat). The normalized response for all products containing milkfat had a higher initial slope for the shorter light wavelengths indicating a higher sensitivity at lower wavelengths. A transition sensor activating at 50% signal response will activate at less than 20% homogenized cream and at less than 10% unhomogenized cream for all wavelengths.

For example, the normalized response at 400 nm for homogenized cream was saturated at 16% homogenized cream (1.6% fat concentration) whereas that for 900 nm was saturated at 64% homogenized cream (6.4% fat concentration). The normalized response was mixed for skim milk with the greatest increase at 900 nm and the least at 600 nm. There appears to be a region of low fat concentration where the normalized response is linear for all wavelengths tested as seen in figures 7c and 7d.

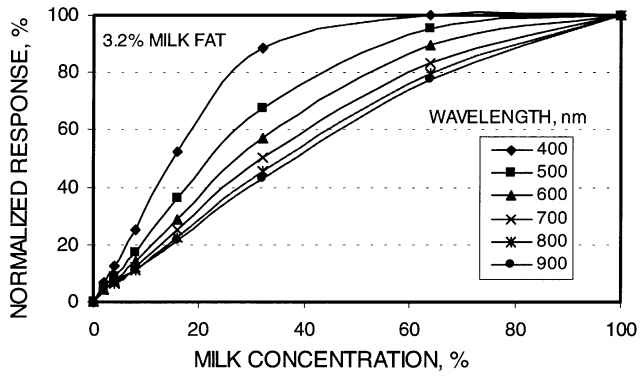

Figure 7(d)–Normalized optical response as a function of milk concentration for milk containing 3.2% milkfat.

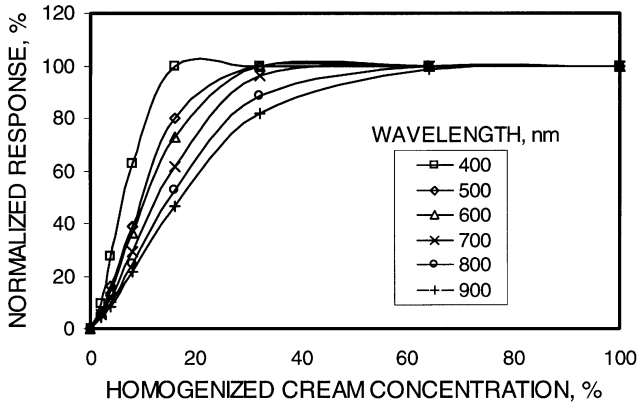

Figure 7(e)–Normalized optical response as a function of homogenized cream (half-and-half) concentration in water.

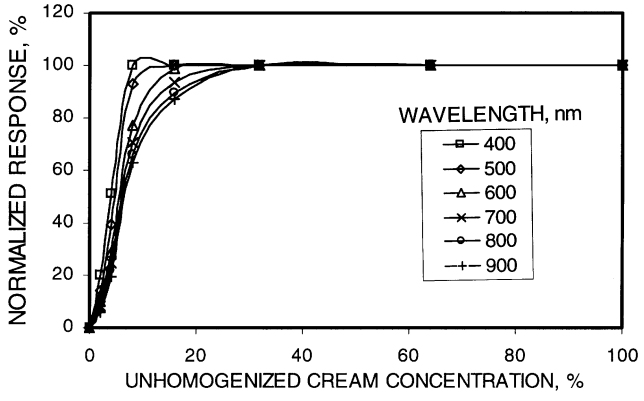

Figure 7(f)–Normalized optical response as a function of unhomogenized cream (40% milkfat) concentration in water.

The backscatter ratio (BR) at various milkfat levels is shown in figure 8. The BR ratio increased as the milkfat concentration increases from 1% to 10%. However, the BR for 10% and 40% fat are almost overlapping. As the number of milkfat particles increases, the total cross-sectional area available for scattering increases. However, at high fat concentrations (above 10%) the fat particles are so close together that any additional particles are hidden and are unable to contribute to light backscatter. Light backscatter increased with increasing wavelength until about 900 nm and then decreases above 900 nm. Part of the increase in sensitivity of the BR around 900 nm can be attributed to the fact that the average diameter of the fat particles is approximately equal to the wavelength of incident light.

The BR for milk having 3.2% milkfat at a light wavelength of 900 nm and 500 nm was approximately 4 and 1.5 times that for skim milk, respectively. Thus, scattering at the lower light wavelengths (around 400 nm) was less affected by milkfat changes. This could be a factor to consider when dairy products with different fat contents are processed. Conversely, the large change in BR at 900 nm suggests that BR at 900 nm could be used to control the blending of homogenized milkfat with skim milk for milkfat control.

The BR as a function of homogenized milkfat is shown in figure 9. The BR between 0.05 and about 0.4% milkfat shows no trend as a function of wavelength. Above 0.4% milkfat the BR first increased linearly and then asymptotically approached saturation. The BR was more responsive for the longer wavelengths tested. For instance, the BR for 10% milkfat was 4.5 at 900 nm and 1.75 at 550 nm.

The BR for unhomogenized cream in milk is shown in figure 10. The response is similar to that for homogenized cream, except that saturation occurred at approximately 16% milkfat, compared to approximately 8% for homogenized cream (fig. 9). This difference is attributed to the increase in fat particle surface area associated with

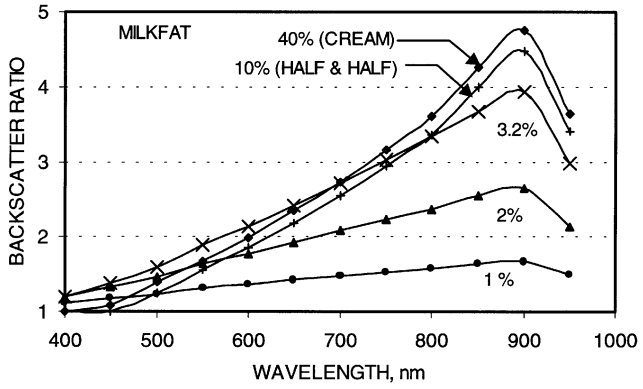

Figure 8–Backscatter ratio (relative to skim milk) for homogenized milk containing 1, 2, and 3.2% milkfat, homogenized cream (half-and-half, 10% milkfat), and unhomogenized cream (40% milkfat). Data for master and slave spectrometers were averaged.

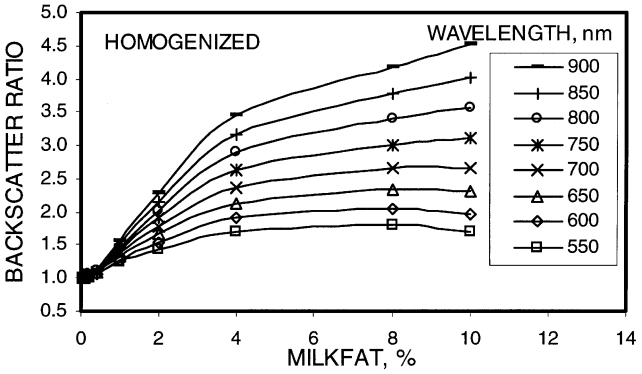

Figure 9–Backscatter ratio for different milkfat concentrations obtained by mixing homogenized cream (half-and-half) in skim milk for light wavelengths of 550 to 900 nm in 50 nm increments.

homogenization. The wavelength sensitivity appears similar for both homogenized cream and unhomogenized cream. The BR for homogenized cream and unhomogenized cream was approximately the same magnitude. The peak BR at 900 nm for homogenized cream and unhomogenized cream was 4.5 (10% milkfat) and 3.8 (28% milkfat), respectively.

Transition sensing appears feasible using light backscatter as measured using optical fibers. The normalized response is nearly linear for 900 nm light and for milks containing up to 3.2% milkfat and logarithmic for skim milk. The backscatter signal saturates for products high in milkfat such as homogenized cream and unhomogenized cream. Therefore the determination of the transition between two high fat products will be more difficult with a backscatter sensor. A proper calibration strategy will be required for process applications where multiple products with varying milkfat concentrations are processed.

Light backscatter as measured using optical fibers was found to be highly sensitive to milkfat content in milk at light wavelengths around 900 nm. This suggests that light backscatter could be used for blending homogenized cream with skim milk to control milkfat concentration.

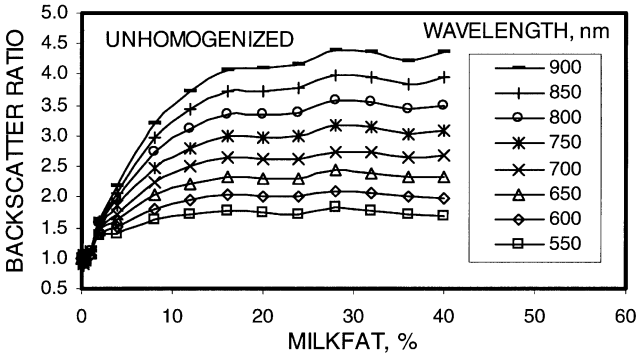

Figure 10–Backscatter ratio for different milkfat concentrations obtained by mixing unhomogenized cream (40% milkfat) in skim milk for light wavelengths of 550 to 900 nm in 50 nm increments.

CONCLUSIONS

1. Light backscatter around 900 nm as measured using optical fibers is ideally suited for detecting the transition of dairy products. The normalized backscatter response (0% for water; 100% for undiluted product) was approximately linear with respect to milk product concentration for milks containing 1, 2, and 3.2% homogenized milkfat and approximately logarithmic for skim milk. Saturation of the normalized response was observed for homogenized products having fat concentrations above 6.4% and for unhomogenized cream at a fat concentration above 12.8%. The normalized response was wavelength dependent with lower wavelengths (around 400 nm) being more sensitive to product concentration than the higher wavelengths tested.
2. The backscatter ratios for the milk products tested increased with light wavelength between 400 and 900 nm indicating that light backscatter increases with light wavelength. The backscatter ratio at 900 nm was particularly sensitive to milkfat. The backscatter ratio for milk containing 3.2% homogenized fat was nearly four times that for skim milk.
3. The use of the light backscatter to distinguish between high fat content dairy products may not be feasible because of saturation at higher milkfat concentrations. Saturation occurred for homogenized cream in milk at approximately 8% milkfat and for unhomogenized cream in milk at approximately 16% milkfat.

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