Quantitative Analysis of Raw Milk with Vis/SW-NIR Spectroscopy

A. Melenteva [1] and A. Bogomolov [1,2]

Background: Milk is a strong **light-scattering** medium due to the presence of colloidal **fat** (1-10 µm) and **protein** (80-200 nm) particles (Fig.1).

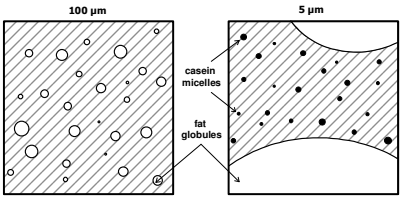
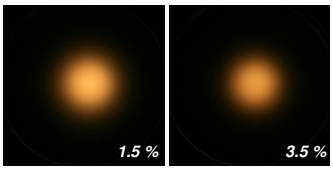


Figure 1 – Milk colloidal structure

Spectroscopic analysis of fat and protein content is usually performed in the **near infrared** (NIR) region. The **scatter is minimized** during the measurement or by applying spectral corrections. The **Visible** and short-wavelength NIR (Vis/SW-NIR) region (400-1100 nm) is typically ignored as containing **no significant absorption** of the milk components.

Problem: Can **multi-scattered** Vis light be used for **quantitative** determination of milk nutrients? Fig. 2 shows that fat content affects the light spot transmitted by homogenized milk samples.

Figure 2 – Spots of light transmitted through homogenized milk samples with different fat content



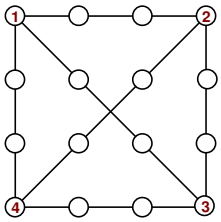
However, for the analysis of natural raw milk the method should be resistant to the **variability of particle sizes**. Can the observed scattered light intensity at different wavelengths (**spectrum**) be **correlated** to the fat and protein particle number and their size distributions?

Experimental: **Two series** of 16 samples with **varying fat and total protein** content were prepared from milk standards of exactly known composition (Table 1) by a **pair-wise mixing** design (Fig. 3). Vis/SW-NIR transmittance spectra were acquired using a 4-mm cavity. To simulate the variation of fat globule sizes each sample was analyzed 3 times: raw and after **two partial homogenizations** for 10 and 20 s with an **ultrasound** processor.

Table 1 – Composition of milk standards used for the preparation of fat and protein samples (F- and P-series respectively)

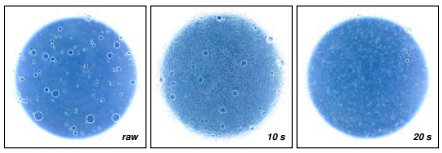
		%Fat	%Protein	%Lactose
F-series	1	2.37	3.47	4.82
	2	3.28	3.49	4.76
	3	4.24	3.57	4.79
	4	5.46	3.61	4.75
P-series	1	3.64	3.00	4.23
	2	4.00	3.37	4.70
	3	4.32	3.66	4.78
	4	4.13	4.09	5.37

Figure 3 – Pair-wise mixing design for a sample series (the numbers correspond to concentrations in Table 1)



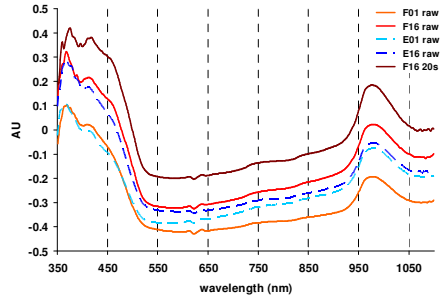
Results and Discussion: Fig. 4 shows the microscope images of a milk sample at different processing stages.

Figure 4 – Sample F4 (see Table 1) at 40x-magnification at different homogenization grades



Spectra (Fig. 5) reveal the **difference of fat and protein concentration** as well as the effect of **homogenization**.

Figure 5 – Spectra of samples with different fat and protein content at different homogenization grades



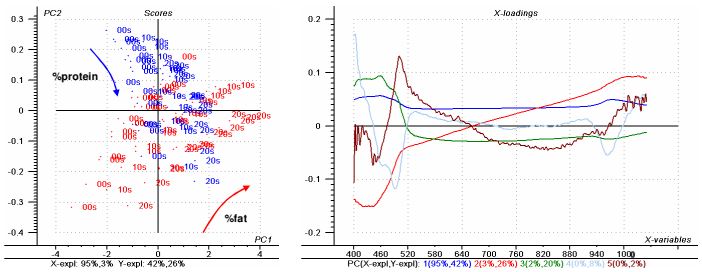
PLS-regression models for joint fat and protein data (96 measurements) exhibit high prediction performances (Table 2). Therefore, Vis/SW-NIR spectra do **capture the information on particle size** distributions that can be **quantified** by multivariate data analysis.

Table 2 – PLS modeling performance (cross-validation)

Component	RMSE	R^2	PC
%Fat	0.124	0.963	5
%Protein	0.044	0.959	5
%Lactose	0.057	0.931	5

PLS scores and loadings (Fig. 6) confirm that both fat and protein content differences as well as the homogenization effect (i.e. wide variability of particle sizes) are **clearly distinguished** in the factor space.

Figure 6 – Scores (left) and loadings (right) plots of the fat content model; F- and P- series (see Table 1) are designated by red and blue color respectively



A completely **new result** is that dissolved **lactose** can also be **accurately determined** from Vis/SW-NIR spectra. This can be explained by its absorbance or by an indirect effect on scattering conditions.

Application of **space-resolved spectroscopy** in combination with advanced data analysis is an **improvement potential**. The method is adaptable for **in-line quality monitoring** in the industry.

[1] Samara State Technical University, 244 Molodogvardeyskaya Street, 443100 Samara, Russia, www.samgtu.ru
[2] J&M Analytik AG, Willy-Messerschmitt-Strasse 8, 73457 Essingen, Germany, www.j-m.de