Determination of fat, protein and lactose in raw milk using shortwave NIR transmission

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Introduction

The fast and accurate measurement of raw milk composition is an important aspect of the dairy industry. Several studies have demonstrated near infrared spectroscopy (NIR) as an effective tool for analysis and quality control. Wu *et al.*[1,2] studied the determination of major compounds in milk powder using short-wave near infrared spectroscopy. Pravdova *et al.*[3] concluded that NIR spectroscopy is useful to determine somatic cell count (SCC) in milk in the wavelength region 700–1100 nm. Tsenkova *et al.*[4] showed that the short-wave NIR spectral region contains wavelengths for determination of constituents in milk. However, the spectrometers used in these studies were delicate, expensive instruments used in laboratory. In this study, a CCD based analyzer (700–1100 nm) and related chemometric software were developed at relatively low cost to explore the calibration models for fat, protein and lactose. Preliminary research also indicated that smoothing and pretreatment of milk spectral data were needed for evaluation of milk components by NIRS. The performance of models after pre-treatment methods was evaluated.

Materials and methods

Spectrometer and samples

A CCD Spectrometer (Ince, Beijing, China) was used for spectral acquisition. The NIR spectra were measured in transmission mode using a quartz sample vessel with 5mm light path in the wavelength region 700–1100 nm, with 0.2 nm interval. Raw milk samples (100) were obtained from a dairy farm in China and kept at 4 °C during the experiment. The temperature of samples at analysis was controlled by dipping sealed samples into a water-bath maintained at 40°C. Each sample was scanned three times to get an average spectrum.

Laboratory reference measurements

Reference standard values were measured with Milkoscan FT 120 (FOSS, Denmark www.foss.dk). All samples were analyzed simultaneously for fat, total protein and lactose contents.

Calibration and validation models

Data analysis was performed with a commercial software program CM2000 (Focused Photonics Inc. Hangzhou, China www.fpi-inc.com) and Matlab7.0 (The MathWorks, Natick, Ma, USA www.mathworks.com). Spectral pretreatments included a Savitzky-Golay (SG) smoothing algorithm with smoothing points of 13, multiplicative scatter correction (MSC), orthogonal signal correction (OSC), and the combination of standard normal variate (SNV) and de-trending (DT). The calibration equation was developed using partial least squares (PLS) regression. Calibration models for each combination of pretreatment methods were obtained. The ratio of calibration and validation samples was 3:1, according to the K-S (Kennard-Stone) classification.

Results and discussion

The statistical results of NIRS analysis are shown in Table 1.

For the models of fat and protein, the best correlation coefficients for prediction (R_P) and standard errors of prediction (*SEP*) were obtained after MSC pretreatment, (0.948, 0.072 and 0.859, 0.045 respectively). The purpose of MSC is to remove the multiplicative interferences of scatter and particle size among the ingredients. These results demonstrated that MSC works effectively to reduce the overlapping of the spectra, which may be due to the scattering of the fat globules that typically occurs in raw milk without homogenization. For the lactose model, the best R_P and *SEP* were 0.796 and 0.047 after OSC pretreatment. Figure 1 shows the correlations between reference values and predicted values of fat, protein and lacto.

The signal-to-noise ratio and stability needs to be improved by enhancing energy of light sources. The composition of the samples shown in Table 2 was relatively stable due to production rules from the diary factory.

To get robust models with stable performance, samples from different seasons, regions, varieties of cows and feeding methods are needed. The establishment of a database for specific dairy farms would be helpful to provide data for long term study.

Table 1. Statistics for raw milk calibrations.

Components	R_C	*SEC*	R_P	*SEP*	Factor	Pre-treatment
Fat, %	0.977	0.072	0.948	0.072	5	Savitzky-Golay + Multiplicative Scatter Correction
Protein, %	0.881	0.053	0.859	0.045	6	Savitzky-Golay + Multiplicative Scatter Correction
Lactose, %	0.982	0.021	0.796	0.047	6	Savitzky-Golay + Orthogonal Signal Correction

SEC: square error of calibration; *SEP*: square error of prediction; R_C: correlation coefficient of calibration set; R_P: correlation coefficient of prediction set.

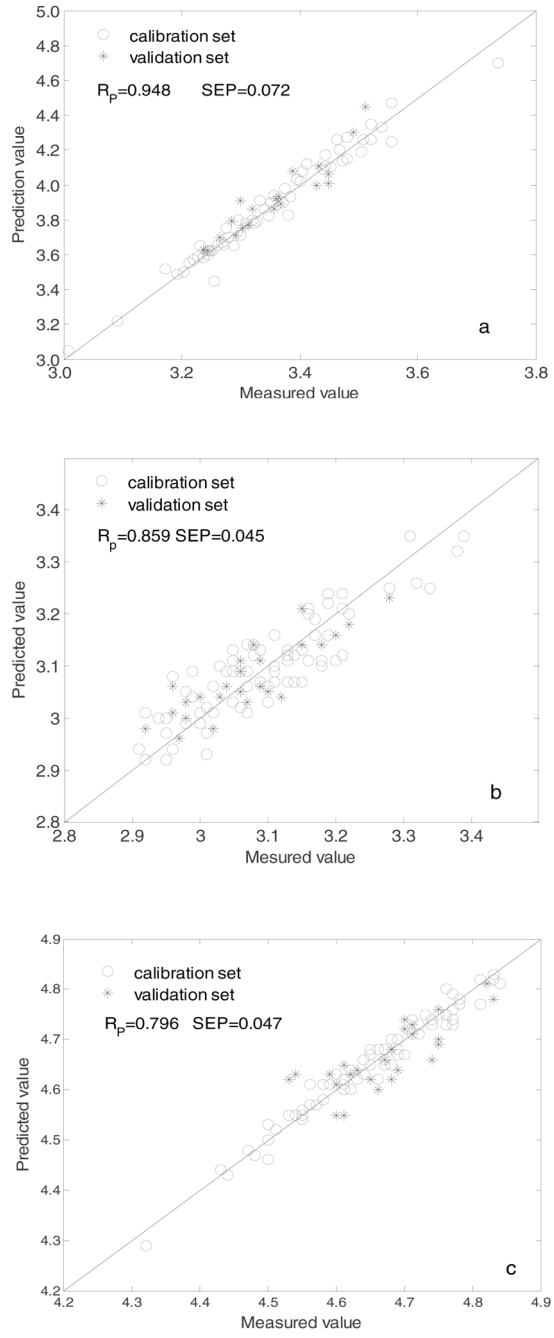

Figure 1. The correlation between measured value and predicted value of fat (a), protein (b) and lactose (c).

Table 2. Composition of milk samples.

Components	*N*	Mean	Minimum	Maximum	*SD*
Fat, %	77	3.88	3.02	4.84	0.30
Protein, %	98	3.09	2.91	3.39	0.10
Lactose, %	98	4.66	4.32	4.84	0.10

N: sample number; *SD*: standard deviation.

Conclusion

This study has provided information on a low cost CCD based short-wave near infrared spectrometer for measurement of fat, protein and lactose in raw milk. Results showed that this instrument performed sufficiently well for the determination of fat and protein, and could be used in the field for on-line measurement.

References

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