Rapid, easy-handling system for NIR compositional analysis of non-homogenized milk using a test tube

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Introduction

In our previous study,[1] the "MilkSpec-1" has been designed and developed to establish a simple and rapid method of quantitative analysis of raw milk without homogenization.

In this experiment, the "MilkSpec-2" and the "MilkSpec-3" as shown in Figure 1 had been designed and developed to improve the "MilkSpec-1".

Materials and methods

Samples

A total of 103 raw milk samples were used without homogenization. The samples were collected from about 26 individual milking cows at 4 times (morning and evening for 2 days) from the National Institute of Animal Industry, Tsukuba, Japan. The samples were transported to our laboratory at the National Food Research Institute in Tsukuba by a car.

Spectral acquisitions

Spectral acquisitions were done with 2 designed measuring systems, the "MilkSpec-2" and the "MilkSpec-3" (Figure 1). The "MilkSpec-2' measured NIR spectra from 700 nm to 1100 nm with the NIRSystems6500 (Foss NIRSystems, Silver Spring, U.S.A.) and a modified transmittance fiber optic (220-mm length) while the "MilkSpec-3" measured NIR spectra from 700 nm to 1000 nm with the Fruit Tester 20 (FANTEC, Kosai-city, Japan) and an attached interactance fiber optic.

(a) "MilkSpec-2" **(b) "MilkSpec-3"**

Figure 1. The NIR measuring systems of (a) "MilkSpec-2" and (b) "MilkSpec-3".

Table 1. Characteristics of calibration and validation sets of samples used.

Constituent	Calibration set			Validation set		
	Range	Mean	SD	Range	Mean	SD
Fat	2.30-6.17	4.28	0.83	2.63-5.47	4.20	0.66
Protein	2.85-4.00	3.41	0.32	2.86-3.81	3.39	0.28
Lactose	3.68-4.90	4.52	0.27	3.99-4.80	4.52	0.21
Non-fat solids	7.85-9.58	8.91	0.45	8.21-9.49	8.89	0.39
Total solids	11.00-15.48	13.16	1.06	11.27-14.63	13.10	0.91

Both systems used a ceramic as a reference material. The specification of the Fruit Tester 20 compared with the NIRSystems6500 was shown in our previous work.[2]

To measure NIR spectra, the milk sample was filled into a 10-mL simple test tube (12-mm inner diameter). A cap was used to cover the test tube during the experiment. Prior to spectral acquisition, sample temperature was controlled at 40 °C by dipping the test tube with milk sample into the water bath for 15 minutes.

Chemical analysis

Amounts of fat, protein, lactose, non-fat solids and total solids in the milk samples were determined with the Milko-scan 4300CU (Foss Electric A/S, Hillerød, Denmark).[3]

Data analysis

Partial least squares (PLS) regression was used to develop calibration equations for each constituent. Different wavelength regions (50-nm interval) were examined to produce the best calibration results. Multiplicative Scatter Correction (MSC), Savitsky-Golay Smoothing (8 nm, 16 nm averaging) and second derivative (8 nm, 16 nm averaging) pre-treatments and their combinations were applied to the NIR spectra for the best calibration results. All calculations were carried out with the Unscrambler ® program (CAMO, Oslo, Norway).

Separate sample sets were used to make calibration and validation. Characteristics of calibration and validation sets of each constituent are shown in Table 1.

Results and discussions

Original spectra [log(1/T)] of milk samples measured with the "MilkSpec-2" and "MilkSpec-3" are shown in Figure 2(a) and 2(b), respectively. The differences in log (1/*T*) values mainly caused by scatter condition of samples that depended on the amount of fat globules.

It was found that MSC treatment was needed to remove the scatter effect while second derivative pre-treatment could improve the calibration results in all calibrations except those of the lactose. The *SEP*s of the best calibration equations for each constituent of the "MilkSpec-2" and the "MilkSpec-3" are given in Table 2.

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Table 2. Standard error of prediction (*SEP*) of the calibration equations developed from NIR spectra measured with the "MilkSpec-2" and the "MilkSpec-3".

Constituent	*SEP*	
	MilkSpec-2	MilkSpec-3
Fat	0.031	0.057
Protein	0.066	0.084
Lactose	0.090	0.174
Non-fat solids	0.108	0.173
Total solids	0.118	0.214

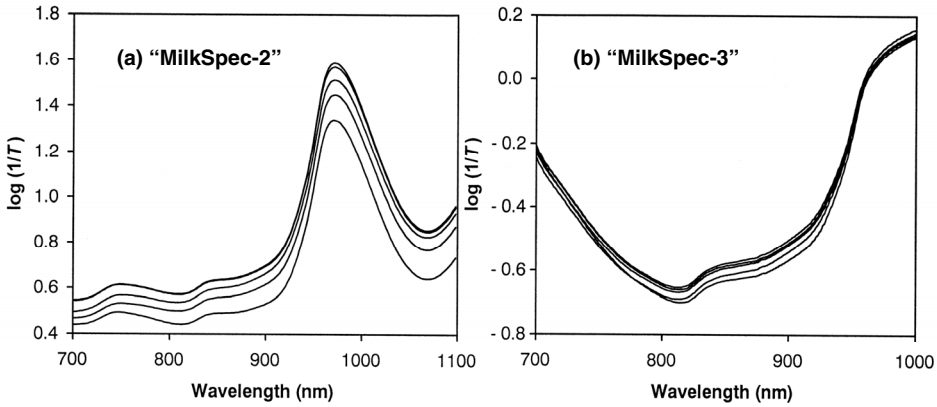

Figure 2. Original [log(1/*R*)] spectra of non-homogenized milk measured with the "MilkSpec-2" (a) and the "MilkSpec-3" (b) using a simple test tube as a sample cell.

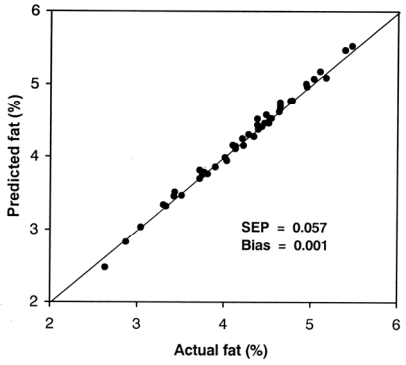

Figure 3. Scatter plots for validation set of actual fat contents vs. NIR-predicted fat contents using the "MilkSpec-3".

From Table 2, it was found that both the "MilkSpec-2" and the "MilkSpec-3" had high ability for compositional analysis of non-homegonized milk. As an example, the scatter plots for validation set of actual fat contents vs. NIR-predicted fat contents using the "MilkSpec-3" is shown in Figure 3.

Conclusion

With the use of a new measurement unit, the "MilkSpec-2" or the "MilkSpec-3", NIR rapid and easy determination of chemical compositions in raw milk without homogenization becomes possible. The accuracy for determining fat, protein and total solids of the calibration equations developed is excellent.

References

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