Animal welfare, etológia és tartástechnológia

Animal welfare, ethology and housing systems

Volume 5 Issue 4

Különszám

Gödöllő
2009

THE DETERMINATION OF CHEMICAL COMPOSITION OF FRESH GOAT'S CHEESES USING OF FT NIR SPECTROSCOPY

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Abstract

In our work we occupied by monitoring basic chemical composition of fresh goat's cheeses over a period of lactation from April to November. The samples were analysed every 14 days. With reference methods were defined indexes: dry matter, fat, pH, NaCl, titrable acidity (SH). The samples were measured at FT NIR spectrometer in reflectance mode with resolution 8 and number of scans 100. Calibration models were made with help partial least squares (PLS) methods. The models were checked with cross-validation. The values of correlation coefficient of calibration were as follows: dry matter 0.810; fat 0.825; pH 0.959; titrable acidity 0.953; NaCl 0.964. The values of correlation coefficient of validation on the integration sphere were as follows: dry matter 0.671; fat 0.669; pH 0.659; titrable acidity 0.901; NaCl 0.867. Results of this study indicate that FT NIR spectroscopy can be used for a rapid analysis of basic chemical composition of fresh goat's cheeses. Values of calibration coefficient of variation (CCV) and prediction coefficient of variation (PCV) in calibration models of analysis of contents of NaCl moderately exceeded boundary values (5,79; 11.15).

Keywords: near-infrared spectroscopy; goat's cheese; chemical composition; dry matter; fat; acidy

Introduction

NIR spectrometry is very often used in pharmacy, petrochemical use and medicine. Over the last years NIR spectrometry extended across to analyse of control quality of agricultural products and food (*Centner*, 1999).

In comparison with classic spectrometry and chromatography NIR spectrometry is the non-destructive method and has many advantages such as rapidity, measurement through transparent packing (*Šikola*, 2002).

In the dairy industry NIR spectrometry used to determination of basic components such as dry matter, protein, fat, and lactose. These applications were study by *Jankovská et al*, 2003 and *Laporte et al*, 1999. Papers of analyse of cheese by NIR spectroscopy were published by *Skeie et al*, 2005, *McQueen et al*, 1995.

The objective of our work was to study the possible applications of FT NIR spectrometry in the determination basic components of fresh goat's cheese.

Material and methods

Material

For the calibration of the NIR spectrometer, samples of fresh goat's cheeses were used. The cheese samples were produced every two weeks from April to November from milk during lactation. These samples were vacuum-packaged and refrigerated at 4°C until analyses and transported to the Department of Food Technology.

Reference methods

Reference data were obtained as follows: dry matter (DM) was determined gravimetrically, by oven drying at 102 ± 2 °C to constant weight (Czech State Standard No. 57 0107). Content of fat (F) in cheese was analysed method by van Gulik (ISO 1187). Content of sodium chloride (NaCl) was determined by titration with silver nitrate (Czech State Standard No. 57 0107). Titration acid expressed as SH was determined such as depletion of volumetric solution NaOH (c=0,25 mol/l) needed do neutralization of 100 g of cheese to phenolphthalein (Czech State Standard No. 57 0107). Acidity pH was determined by WTW 95 pH-meter.

FT NIR analysis

A wavelength scanning instrument FT NIR Antaris (ThermoNicolet, USA) was used with a scanning range from 4 000 to 10 000 cm-1 in reflectance mode. The sample was measured in reflectance mode, 100 spectral scans was taken for each sample with resolution 8. Each sample was analysed three times and the average spectrum was used for calibration. The whole spectrum area has been tested.

The calibration model was created by partial least squares (PLS) algorithm. The same samples were employed for full cross validation by software FT NIR Reference Analysis.

The selection of optimum number of PLS terms for the calibration was based on the standard error of prediction (SEP), which should be minimised. The statistical parameters (correlation coefficient – R and SEP) were used to determinate the final calibration equation.

All results were evaluated using the variation statistic analysis (ANOVA). Correlation matrices and regression functions were calculated according to *Snedecor and Cochran* (1967) when using the statistical package Microsoft® Excel 2000 and Unistat 5.1.

Results and discussion

To create calibration models were used PLS algorithm. Calibration models were then verified by cross validation. In Table I and II are the values of correlation coefficients of calibration and validation (R), standard error of calibration and prediction (SEC, SEP), the calibration coefficient of variation (CCV) and prediction coefficient of variation (PCV).

Reliability of the calibration model is determined by several factors. Calibration and validation rates should be as close as possible to one. The linear dependence of the reference results versus results predicted by the PLS algorithm for determination of titrable acidity (SH) and dry matter of fresh goat's cheeses is illustrated in *Figure 1 and 2* Obviously, there is a good correlation between predicted values and known chemical reference values.

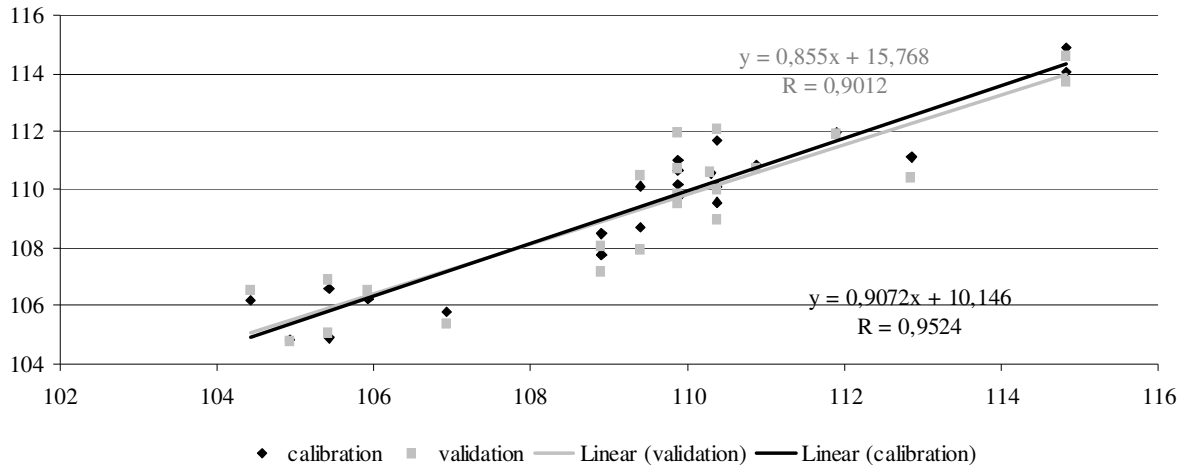

Figure 1. **Relationship of calibration and validation results of titrable acidity (SH) of fresh goat's cheeses**

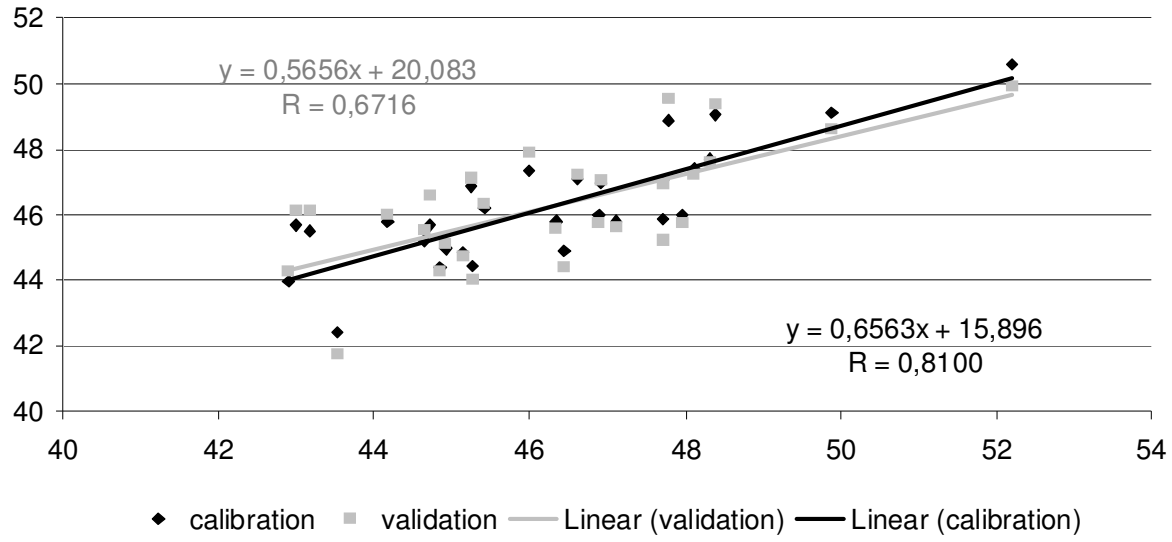

Figure 2. **Relationship of calibration and validation results of dry matter of fresh goat's cheeses**

In *Table 1 and 2*, we can note the high rates with relatively low standard deviations.

Table 1. **Parameters of the regression function $y'_i = a + bx_i$ for the calibration model**

Traits	R	SEC	CCV (%)	PLS faktory	bx + a
dry matter (%)	0.810	1.23	2.66	5	0.6563x+15.896
pH	0.959	0.02	0.48	4	0.9210x+0.3756
SH (0.25 mol/l)	0.953	0.85	0.78	6	0.9072x+10.146
NaCl (%)	0.964	0.07	5.79	10	0.9278x+0.0883
fat (%)	0.825	0.79	3.40	4	0.6800x+7.3997

SH – titrable acidity; R – correlation coefficient; SEC – standard error of calibration; CCV – calibration coefficient of variation

Table 2. **Parameters of the regression function $y'_i = a + bx_i$ for the validation model**

Traits	R	SEP	CCV (%)	PLS faktory	bx + a
dry matter (%)	0.671	1,60	3.46	5	0.5656x+20.083
pH	0.590	0,06	1.31	4	0.5366x+2.2132
SH (0.25 mol/l)	0.901	1,12	1.02	6	0.8550x+15.768
NaCl (%)	0.867	0,14	11.15	10	0.8531x+0.1830
fat (%)	0.669	1,06	4.59	4	0.5619x+10.080

SH – titrable acidity; R – correlation coefficient; SEP – standard error of prediction; PCV – prediction coefficient of variation

A well-functioning model should not have more than 15 PLS factors for determining the NIR spectrometer, which is less than a fixed component of our. *Table 1* for the determination of salt CCV value slightly exceeds 5%, also for determining the value of PCV NaCl in *Table 2* slightly exceeds the limit value of 10%. This means that the model is not reliable enough. For all other observed properties not exceeding the limit values CCV (5%) and PCV (10%). Models can therefore be considered reliable.

In *Table 3* are recorded the results of the statistical evaluation of the NIR predicted and reference values were tested by paired t-test. There was no statistically proven difference between the reference and predicted values.

***Table 3*. Parameters of basic components in fresh goat´s cheeses as estimated by NIR reference values and their mutual comparison by paired t-test, n = 30**

	xREF	xNIR	SD	t-test	$t_{1\ (krit)}$	$t_{2\ (krit)}$
dry matter (%)	46.25	46.25	0.397	0.003	1.701	2.048
pH	4.77	4.77	0.016	0.000	1.700	2.050
SH (0.25 mol/l)	109.39	109.39	0.595	0.000	1.720	2.070
NaCl	1.21	1.21	0.051	-0.050	1.710	2.060
fat (%)	23.12	23.12	0.273	0.500	1.706	2.056

SH – titrable acidity; xREF – mean of the reference values; xNIR – mean of the NIR values; SD – standard deviation of mean; t-test – values of paired T-test; $t_{1\ (krit)}$ – table´s values for $\alpha = 0.05$; $t_{2\ (krit)}$ – table´s values for $\alpha = 0.01$

Conclusions

This work has demonstrated the possibility determination of basic components (dry matter, fat, titration acid, pH) of fresh goat's cheese. Values of correlation coefficients of calibration and validation are high by low the calibration coefficient of variation (CCV) and prediction coefficient of variation (PCV). This results shows to functionality of calibration models with possibility of application in practise. Calibration model for examined NaCl was less accurate because CCV a PCV slightly exceeded recommend value.

Acknowledgments

This study was supported by the Research plan the Ministry of Education, Youth and Sports of the Czech Republic 2B08069 National plan of research - NPV II, plan 2B – Healthy and quality of life.

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