best@buchi No. 19
Nutrition: Analysis of cheese by FT-NIR Technology

This study was conducted to develop a rapid and accurate NIR analysis method for determining fat, moisture, protein and NaCl in cheese. Commercial cheeses (thousand samples) were used to create calibration models. Conventional analysis of cheese ingredients is both time-consuming and expensive. Near infrared (NIR) spectroscopy is fast, inexpensive and can provide useful information on the ingredients of cheese.

Determination of the composition of cheese

Near infrared (NIR) spectroscopy was used to determine the fat, moisture,protein and salt content of hard, semihard and young cheese. NIR spectroscopy is a rapid and precise analysis method and can be used to replace wet chemistry to simultaneously quantify different parameters of cheese. In comparison to traditional wet chemistry, which requires hours for the analysis of each component, the time requirement of this method is slashed to 16 seconds. A set of thousand samples from diverse types of cheese (Emmental, Gruyeres, St. Paulin, Raclette, Appenzell, Tisler etc) were used to calculate a calibration for an FT-NIR spectrometer of type BUCHI NIRFlex N-500.
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Near infrared spectroscopy

Chemical analysis provides a listing of the components in a particular food or ingredient. Given that most of these are organic molecules which absorb light in the near infrared (NIR) spectral region (750-2500nm), the NIR spectrum of a particular food or feedstuff provides information about its chemical composition. Additional knowledge about its physical makeup may also be present. Analytical laboratories have looked to the ability of spectroscopy techniques to characterize the quality and/or authenticity of foods.

Advantages and limits of NIR spectroscopy

The major advantages offered by NIR spectroscopy include the speed of analysis, the fact that no or only little sample preparation is needed (such as mixing or grinding), and the simultaneous measurement of different parameters. On the other hand, NIR is an indirect method which requires calibration. The cost of developing calibrations for complex products is substantial. However, tools for calibration development have been improved over the past five years, and many of the routine problems can now be kept under control (for example Calibration Wizard for automatic calibrations in BUCHI NIRCal). Nevertheless, to obtain a useful and reliable calibration is not a simple task. It is usually a multi-step procedure in which it is unlikely that all variables will be covered by the initial calibration. BUCHI is therefore offering pre-calibrated applications to be used with NIRFlex. The quality analysis in a typical laboratory procedure is limited to a handful of samples a day when classical methods are used. When an error occurs in the production line, the delay may mean that a significant amount of product will be wasted before the results are known and the process

can be corrected. Fast availability of measurement results is therefore highly valuable.

Materials and methodology

1. Properties of interest

Fat, salt, protein and moisture content are essential for determining hard cheese quality in the cheese industry as well as for cheese research. Fat is a very important parameter for the richness of flavor. Moisture, on the other hand, relates to the profitability of the final product, protein and the salt content are essential for the taste and shelf life of the cheese. Therefore, the relevant contents of these ingredients are important quality and monetary parameters.

2. Type of samples

A set of thousand samples from diverse types of cheese (Emmentaler, Tisler, Appenzeller, Cheddar, Gruyère, etc.) were used to calculate a quantitative calibration for moisture, fat, protein and salt.

3. BUCHI NIRFlex N-500 FT-NIR spectrometer

This instrument provides quantitative measurements of food and animal feed samples using light in the 1000 to 2500 nm range. The NIRFlex N-500 is characterized by its excellent wavelength reproducibility, which is crucial for ensuring calibration portability and stability. The instrument is equipped with an extended range in gaas detector. It yields spectra up to 1500 data points for each spectrum.

4. Measurement cell NIRFlex Solids

The measurement cell used is the NIRFlex Solids, which measures samples in diffuse reflectance. A rotating sample desk enhances the repeatability of measurement results. Samples are measured in standard glass petri dishes. The sample may also be measured in monouse plastic petri dishes.

5. Software

The software is divided into two separate packages:
For calculation of the calibration the BUCHI Chemometric software NIRCal is used. NIRCal optimizes the calibration with wizards, 2D and 3D representations and all appropriate algorithms. For cheese calibrations, the PLS (Partial Least Squares) method was

used. Routine measurements are performed with NIRWare, a modular and easy-to-use software package with SOPs for sample analysis, giving step-by-step instructions.

Data collection & development of the calibration
Sample presentation
Samples of about 100 g were grated using a commercial cheese grater and without applying too much mechanical pressure. The samples were transferred to glass petri dishes and measured by means of the NIRFlex Solids measuring cell in diffuse reflectance. A measurement consisted of 64 scans. Each sample was automatically measured three times. The reference values only had to be allocated once per sample.
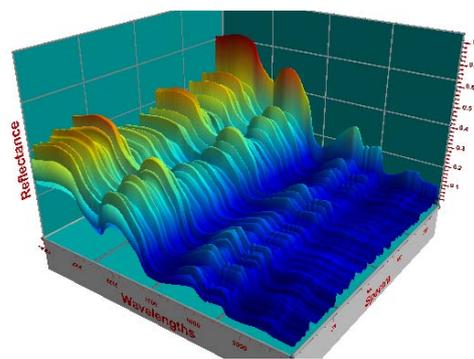
Calibrations
To establish the calibrations, several different cheese varieties (Tête de Moîne, Raccard, Appenzeller, Parmesan, Chester, Gauda, Emmentaler, Edamer, Assiago) were analyzed on the basis of 10-13 different samples each, which had different production dates. The spectra were recorded consecutively using several NIRFlex N-500 units to ensure maximum robustness of the calibration. A common library was created of all the spectra which served as a basis for calculating the calibration. It was found that one calibration is sufficient for each individual parameter, i.e. there is no need to switch between calibrations with high or low content. The samples were measured at room temperature.

Composition of test material

Fat	6 - 37 %
Moisture	27 - 68 %
Protein	4 - 51 %
NaCl	0.1 - 4.0%

Reference methods:
The reference values required for calibration were determined by the following methods: Moisture: drying oven; salt content: titration with silver-nitrate solution; fat: Gerber or Soxhlet method, Protein: Kjeldahl method. The diagram below shows all spectra used for calibration. To achieve a successful calibration calculation, the division into calibration and validation spectra is crucial, beside the correct data pretreatment

The calibrations were calculated using the NIRCal software. Different data pretreatment methods were used in doing so:

Parameter	Method	Range of calibration wavenumber [cm-1]
Fat	PLS	4598 - 10000
Moisture	PLS	4400 - 9000
Fat in dry matter	PLS	4397 - 10000
NaCl	PLS	4230 - 9250

Diagram 1 compares the predicted values with the reference values of the "Fat in cheese" calibration. The strength of the calibration with a good coverage in the range of 12-37% fat and the rather weaker coverage in the lower range are apparent.
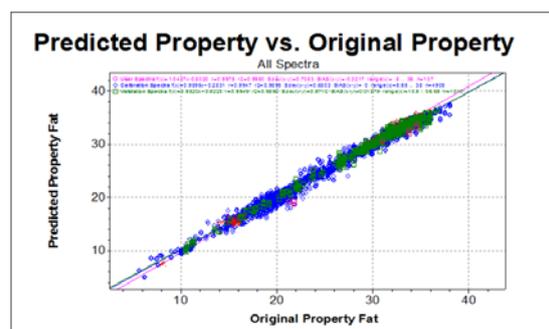

Diagram 1: Fat in hard cheese calibration

Diagrams 2 and 3 show the relevant straight calibration lines for the parameters protein and moisture. Both calibrations convince by their good coverage across the entire range.

Results and discussion
The accuracy of the calibrations can be assessed on the basis of the standard error of prediction (SEP) and the regression coefficient of the straight calibration line. The closer this Rcoefficient is to 1, the more accurate is the prediction of new samples or matching with the values of conventional analysis.
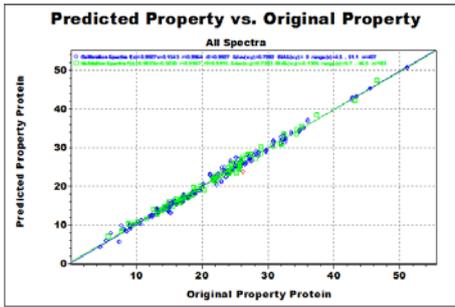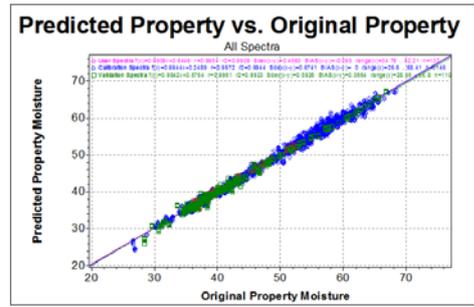
Diagram 2: Protein in cheese calibration

Diagram 3: Moisture in cheese calibration

Properties	Number of samples spectra for calibration	Calibration range (%)	SEP	Standard-deviation of reference method	R-Co efficient
Mosisture	6253	27 - 68	0.59	0.31	0.996
Fat	5915	6 - 37	0.57	0.28	0.995
Protein	590	4 - 51	0.73	0.35	0.997
Salt (NaCl)	2078	0.1 - 4.0	0.16	0.06	0.973

Table 1: Calibration performance

In addition to the precision presented here, FT-NIR technology also offers numerous practical benefits in routine analytics in the field of quality inspections. They include:

Versatility: Simultaneous measurements of different parameters
Rapidity: Results available in a matter of seconds
Economy: Low operating costs
Ecology: No chemical waste
Safety: No chemical reaction
Simplicity: No specialist training required for operation
Calibration portability: Fourier Transform Spectrometers allow calibration data to be transferred from one instrument to another

Quality in your hands

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