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Rapid in-process analysis of fat and dry matter in cheese

Thermo Scientific™ Antaris™ II FT-NIR Analyzer

Abstract

This study shows FT-NIR spectroscopy as an ideal tool for in-process quantitative analysis of processed cheeses. Two components were analyzed—fat and dry matter. Partial Least Squares methods were constructed to allow the prediction of these two components simultaneously from the same sample spectrum.

Introduction

The international cheese market produces billions of pounds of cheese annually, making it one of the largest food industries worldwide. Cheese and other dairy products are regularly tested for components like fat, moisture, protein, and dry matter content. These factors affect the taste, price, and manufacturability of the cheese, making their prediction critical. In the current study, a cheese producer needed to be able to quickly examine and accurately predict the amounts of fat and dry matter in cheese products.

Current protocols for the prediction of components like fat involve the Babcock method. This method uses concentrated sulfuric acid to break dairy components down before analysis. In addition, the Babcock procedure requires a skilled operator running multiple samples destructively in order to give an average number for each component. Procedures like the Babcock method are not only time consuming, but require hazardous materials. In addition, they destroy the sample being analyzed, so the sampling methodology cannot be designated as truly representative of the whole.

Dry matter analyses use infra-balances or microwave driers which are rapid, but their accuracy is far worse than even classical gravimetric determination. Also, the price of appropriate equipment is almost prohibitive and would result in a poor return-on-investment.

The amount of time, materials, and labor involved with testing cheese on a large scale can be greatly reduced by incorporating analytical QC solutions like FT-NIR into the testing protocol. The Thermo Scientific™ Antaris™ II FT-NIR Analyzer provides a fast, easy, and accurate alternative to current testing protocols for cheese. In addition, FT-NIR testing can be extended to other components like moisture or protein, which involves another complicated testing protocol, the Kjeldahl method. The Antaris II FT-NIR Analyzer allows for the collection of a single spectrum in less than 60 seconds without damaging the sample. In addition, multivariate analysis techniques allow the user to predict multiple components (properties) of the cheese from the same single spectrum.

It is necessary to stress that this study used different kinds of processed cheeses—non-fat, middle fatty, fatty, all with different flavors and additives. Differences in temperature of the samples were also intentionally ignored because any pre-collection treatment of the samples eliminates the greatest advantage of FT-NIR—the speed of determination.

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Experimental

Blends of 50 – 200 kg of raw materials (water, butter, cottage cheese, and additives) were tested for fat content and dry matter during agitation in melting pots. Hot aliquots of processed cheese were scanned using the Antaris FT-NIR Analyzer from 4000 to 10,000 cm^{-1} at a resolution of 8 cm^{-1}. Eighty scans were taken per sample resulting in an acquisition time of approximately one minute. Each sample was placed on the integrating sphere sapphire window and collected three times. The window was cleaned in between measurements using 96% ethanol and dried before each sample. Spectral averaging was then used for the three replicate measurements for both standard and challenge samples to achieve an average component value.

Calibration models for the data were developed using the Thermo Scientific™ TQ Analyst™ Chemometric Analysis Software. The model used a constant pathlength and a first derivative treatment of the spectral data without smoothing. The wavelength range from 4450 to 8700 cm^{-1} was used with a one-point baseline. The number of standards used in the calibrations was 477 for fat and 479 for dry matter.
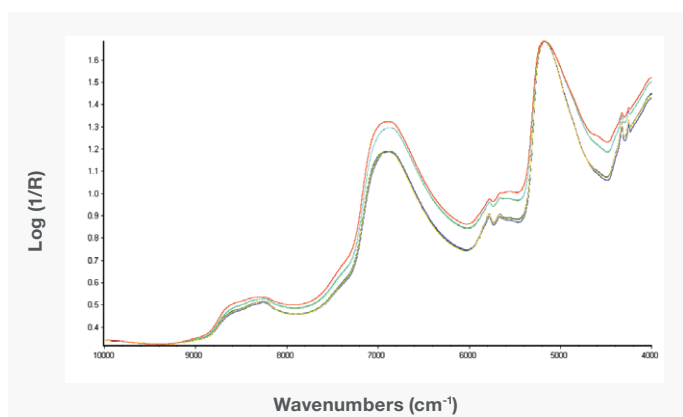

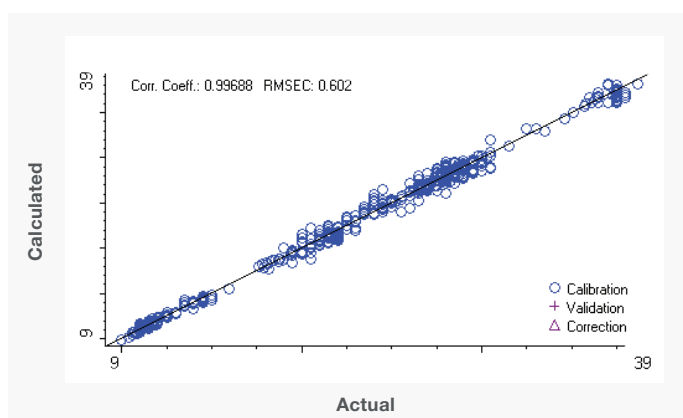
Figure 1. Cheese standards

Figure 2. Calibration curve for fat

Results and discussion

Figure 1 shows NIR spectra of some of the standards used in the calibration. There are broad, highly-absorbing water peaks around 5000 cm^{-1} (combination band) and 7000 cm^{-1} (overtone). There are also relatively sharp C-H combination bands from 4230 to 4490 cm^{-1} and second overtones of C=O groups from 5620 to 5950 cm^{-1}. In addition, there is a considerable amount of scattering from these samples, evidenced by the baseline offsets.

The first method quantifies fat in the blend. 477 standards between 10 and 38.7% wt. were used. The TQ Analyst region wizard suggested the region between 4235 and 5836 cm^{-1}. The calibration was first done with 6 PLS factors yielding a Root Mean Square Error of Calibration (RMSEC) of 0.996 and a Root Mean Square Error of Cross Validation (RMSECV) of 1.07. These two parameters are good indicators of how well a calibration is performing. The RMSEC shows how close your standards are to the calibration curve—the closer the better. RMSECV shows how robust your calibration is. If only a few standards represent the most important variability in your method, then this will show up as a large RMSECV value. High RMSECV values imply a weak calibration which can be remedied by adding standards or changing method parameters. The method was improved when the region was changed to 4449 – 8709 cm^{-1} using the first derivative of the spectra and a one-point baseline. This yielded a calibration using 8 PLS factors with an RMSEC of only 0.602 and an RMSECV of 0.779. Figure 2 is a plot of the calibration curve for the analysis of fat in cheese by FT-NIR spectroscopy. The Predicted Error Sum of Squares (PRESS) plot shows an optimum pattern with high PRESS at low factors and low PRESS at higher numbers of factors (Figure 3).
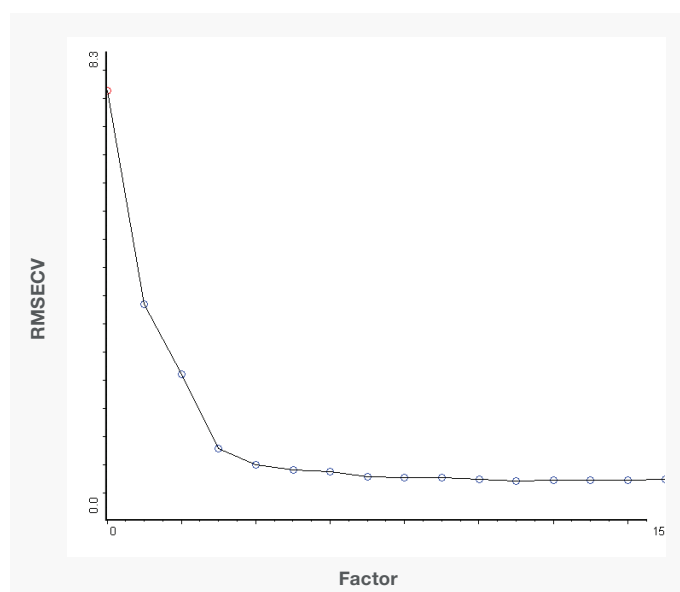

Figure 3. PRESS plot for fat

The second method quantifies dry matter in the blend. 479 standards with values between 31.36 and 53.65% wt. were used. The optimized method included the region from 4075 to 8909 cm^{-1} with a first derivative spectral pretreatment and a one-point baseline. The calibration yielded an RMSEC value of 0.645 and an RMSECV of 0.776. Figure 4 is a plot of correlation and a part of the calibration results. Again, with a calibration set of over 400 samples, the dry matter calibration shows an excellent correlation with the reference data. The PRESS plot for the dry matter was also very reasonable.

FT-NIR offers an excellent alternative to traditional methods for the determination of fat and dry matter in processed cheeses during their manufacture. The user is able to obtain the data quick enough for control of (feedback to) the process of raw materials and additives blending in melting pots resulting in saving raw materials and sustaining a constant quality of products. In addition, FT-NIR is a non-destructive technique that allows the collection of a single spectrum for the prediction of multiple chemical or physical properties in a sample. This methodology could easily be extended to predict other critical factors in cheese processing, such as total protein or moisture content, obviating the need for more involved testing protocols.
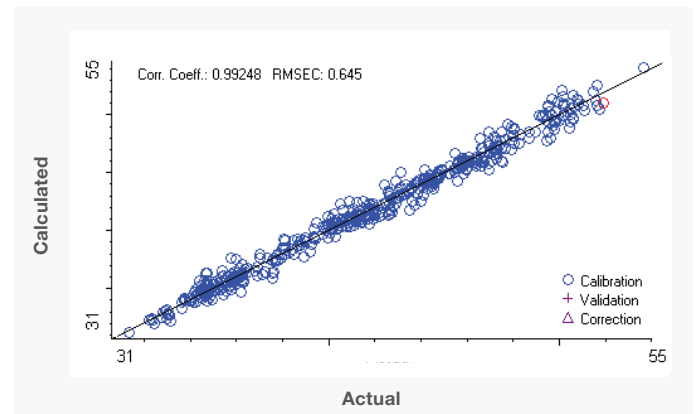

Figure 4. Calibration curve for dry matter

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