Near infrared spectroscopy applied to *pasta filata* cheese in relation to textural analysis

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Introduction

Rheological properties of cheese are important factors for the ripening process and basic parameters for quality assessment during shelf-life and trade steps to the consumer. Characteristic curd and cheese texture are associated with different coagulation processes and final products. Cheese texture also influences cutting resistance of the product, spreadability properties and consumption habits.[1]

Objective measurements of rheological properties of cheese are carried out by texturometers,[2–4] based on product deformation caused by the application of mechanical force (compression). Usually, deformation force is applied at constant speed for a defined time on samples kept at constant temperature. Sample preparation and analysis are time consuming. Therefore, a more rapid and sufficiently accurate technique for measuring firmness of cheese is advisable. Near infrared (NIR) spectroscopy is known to be a good technique for the evaluation of cheese components[5] and it has been shown to respond to changes in the state of water in foods.[6] NIR spectroscopy might give information on rheological properties of cheese during ripening and shelf-life, since these properties are related to chemical constituents and water structure. Recently, some studies have been developed to evaluate the feasibility of NIR spectroscopy in predicting physical properties of foods.[7–9]

The present study aimed to seek a correlation between rheological parameters of cheese, such as hardness, determined by a single compression test and data obtained by NIR spectroscopy.

Materials and methods

One hundred samples of *Pasta Filata* cheese, coated with paraffin and biodegradable wax to avoid mechanical damage, were analysed at room temperature during shelf-life at 90 (50 samples) and 120 days (50 samples).

Samples were cut into small cylinders (D = 3.2 cm, height = 1 cm)[10] and cheese pieces were analysed by an InfraAlyser 500 (Bran+Luebbe, Norderstedt, Germany) at 1100 to 2500 nm at 4 nm intervals (351 data points). Spectra were collected as logs R^{-1}. Data were processed by Sesame Software (Bran+Luebbe, Germany) applying the second derivative of absorbance values (gap = 0, segment = 1). Optimisation of the information provided by raw spectra was obtained by applying multivariate analysis to the whole set of processed data using the principal component analysis (PCA).

Textural characteristics were determined by an Instron Universal Testing Machine 4301 (Instron Corporation, Canton, MA, USA) on sample pieces cut into small cylinders (D = 1.7 cm, height = 2 cm), conditioned at 20°C and placed on a flat plate. Hardness was evaluated by a single compression test using a plunger having a plane circular surface (D = 5.8 cm, height = 3.7 cm). This plunger was fixed to the moving crosshead at a speed of 20 mm min^{-1}. A 10 kg load cell was used. Tests

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were performed by recording the load at 20 mm of compression (max load). Data were processed by Instron series IX Software (Instron Corporation) and expressed as a mean of eight replicates.

Total calibration was performed by applying multiple linear regression (MLR) to the first derivative of normalised absorbance values at seven wavelengths (1160, 1976, 2064, 2080, 2236, 2328 and 2336 nm) and using randomly 70 out of the 100 samples, 35 from each set. The set of 30 remaining samples, 15 at 90 days and 15 at 120 days of shelf-life, was used as a prediction set. Load values, obtained from textural analysis, were plotted against NIR prediction values. Regression coefficient (R^2), *RMSEP* in calibration, *RMSEP* in prediction and $RMSEP_{all}$ were calculated.

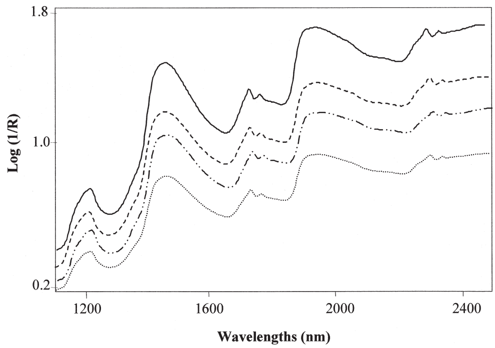

Figure 1. NIR raw spectra for *Pasta Filata* cheese coated with different waxes during shelf-life. (—) paraffin 90 days; (- - -) paraffin 120 days; (–··–) biodegradable wax 90 days; (···) biodegradable wax 120 days.

Results and discussion

Figure 1 shows an example of raw NIR spectra for *Pasta Filata* cheese during shelf-life at 90 and 120 days. Since no differences between spectra were apparently pointed out, pre-treatments were performed on data collected. Explorative analysis, carried out by a PCA on second derivative spectra, was able to gather samples according to their shelf-life period. Figure 2 shows results obtained by PCA; the first principal component (PC1) was able to discriminate cheeses according to their shelf-life.

Textural measurements, performed by the Instron, discriminated cheeses according to their hardness, which was measured as applied force.[2] An example of a typical compression curve is shown in Figure 3. Hardness corresponds to the maximum force (max load) exerted on a sample.

Both the type of wax used and the water content affected cheese hardness, in agreement with monitored weight loss data[11] (Figure 4).

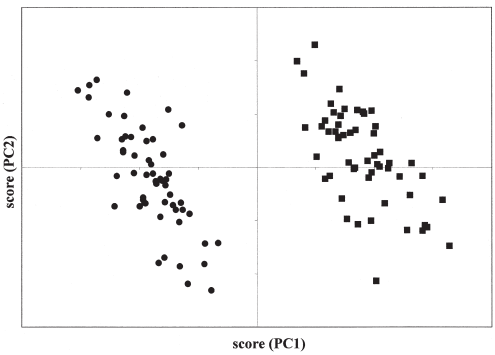

Figure 2. Explorative analysis (PCA) on 2nd derivatives of NIR spectra (gap = 0; segment = 1). (●) 90 days; (■) 120 days.

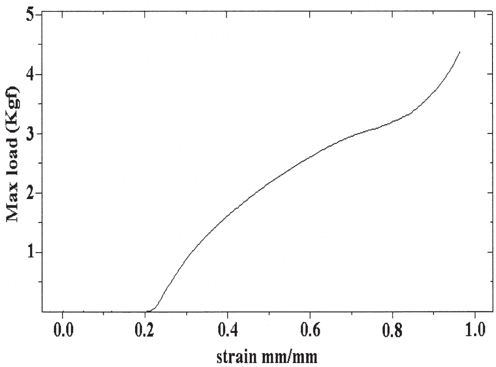

Figure 3. Example of a load-strain curve obtained from a uniaxial compression test on "Pasta Filata" cheese.

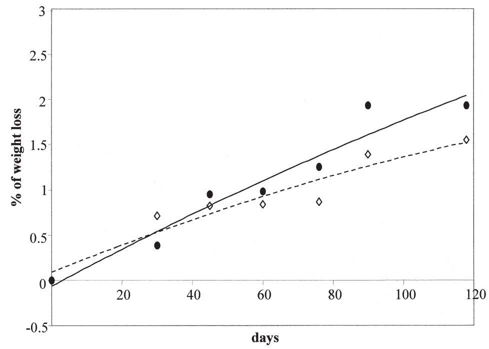

Figure 4. Weight loss rate during shelf-life of cheese. (●) biodegradable wax; (◇) paraffin.

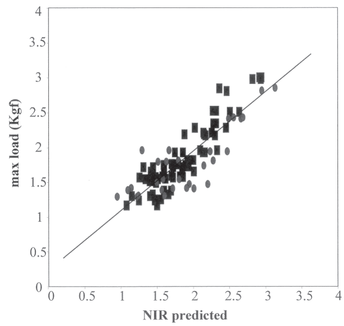

Figure 5. Plot of measured hardness against NIR predicted values for Pasta Filata cheese. (■) calibration set; (●) prediction set.

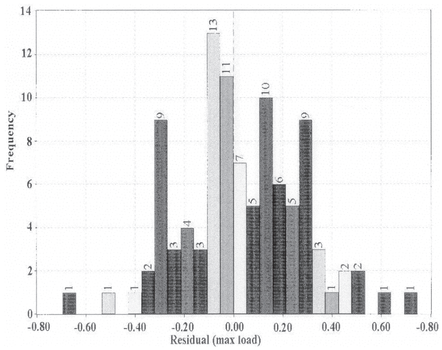

Figure 6. Analysis of residues: prediction and calibration sets.

An MRL calibration model, obtained from the seven wavelengths automatically selected by Sesame software on the first derivative of normalised NIR absorbance values, showed a good relationship between texture measurements (max load) and NIR prediction power. MRL calibration was characterised by $R^2 = 0.916$ and $RMSEP = 0.192$. The prediction set (30 samples) was also characterised by a satisfactory *RMSEP* value (0.345). $RMSEP_{all}$, associated with the whole set of samples, was 0.248. It should be pointed out that the textural analysis, carried out by Instron and used as an objective method for hardness determination, was also characterised by a coefficient of variation (*CV*) of 10% (mean value). Figure 5 shows the NIR spectroscopy prediction power as a plot of hardness values, determined by a texturometer, against the prediction values based on the first derivatives.

From the analysis of residues, calculated on MRL calibration and prediction sets, it can be observed that only 8% of the samples showed higher differences than $\pm$ 0.4 between objective hardness measurements and NIR prediction values (Figure 6). These results confirmed the accuracy of NIR prediction as 92% of residues were found to be close to zero.

Conclusions

NIR spectroscopy proved a useful tool for classifying samples according to their shelf-life period and predicting their textural characteristics, such as hardness. Both the type of wax used and the water content of samples affected cheese hardness, in agreement with monitored weight loss data. Hardness evaluation by NIR spectra was as accurate as that determined by a texturometer, thus confirming that the possible use of NIR spectroscopy for this application may facilitate the routine control of physical characteristics of cheese by a rapid and sufficiently accurate technique.

Aknowledgements

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