Quantification of water content in biscuit using Near-Infrared hyperspectral imaging spectroscopy and chemometrics

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Abstract
The aim of this work was to evaluate the suitability of Near-Infrared Hyperspectral Imaging Spectroscopy (NIR-HSI) for the quantification of water content in commercial biscuits; the final objective is to control on line the possible non uniformity on moisture of biscuits. Ten biscuits from two commercial brands were conditioned for one week in desiccators with different water activities controlled by different saturated solutions of salts. Biscuits were analyzed by NIR-HSI and resulting images are submitted to a PCA. A regression model is also computed using a small number of variables in order to predict moisture values for each pixel and reconstruct prediction images in false color.
These results obtained by PCA show that the water content of the two brands of biscuits can be easily monitored by NIR-HSI. R^2 obtained by regression is higher than 0.93 and the RMSEC less than 0.015. Prediction images are very relevant and can be used to study biscuits defects.

Keywords: Biscuits, Water, NIR hyperspectral imaging, Chemometrics.

Résumé
L'objectif de ces travaux est d'évaluer la pertinence de l'imagerie hyperspectrale proche-infrarouge (NIR-HSI) pour la quantification de l'humidité dans des biscuits commerciaux ; l'objectif final vise au contrôle en ligne de possible non uniformité de teneur en eau de biscuits. Dix biscuits de deux marques commerciales différentes ont été conditionnés pendant une semaine dans des dessiccateurs contenant des solutions saturées avec différents sels. Les biscuits sont analysés par NIR-HSI et les images résultantes sont traitées par analyse en composantes principales (ACP). Un modèle de régression est également calculé en utilisant un petit nombre de variables afin de prédire les valeurs d'humidité pour chaque pixel et reconstruire des images de prédiction en fausses couleurs.
Les résultats obtenus par ACP montrent que la teneur en eau des biscuits de chaque marque peut être facilement contrôlée par la technique NIR-HSI. Le coefficient R^2 calculé par régression est de 0,93 et l'erreur quadratique moyenne inférieure à 0,015. Les images de prédiction sont très pertinentes et peuvent être utilisées pour étudier les défauts dans les biscuits.

Mots-clés : Biscuits, Eau, Imagerie hyperspectrale Proche-Infrarouge, Chimiométrie.

1. Introduction

Water plays an active role in the quality of biscuits and can be responsible of some physical defects such as cracking or breaking (Ahmad et al., 2001). These defects are partially related to the glass transition phenomenon in which the water content and the temperature are critical factors. Many analytical methods (Aït Kaddour et al., 2008; Wagner et al., 2008) can be used to characterize the dough during baking and afterwards during storage. However, they all are invasive, expensive and require sophisticated laboratory procedures. As glass transition can rapidly evolve, the use of rapid and water sensitive methods for the biscuit characterization is necessary. Using the properties of high absorption of water in the Near-Infrared, Near-Infrared Spectroscopy is particularly indicated to quantify the water content in food products, including biscuits (Osborne et al., 1984). Near-Infrared Hyperspectral imaging is an alternative technique for non-destructive food analysis, enabling real-time monitoring of quality parameters (Amigo, 2013; Qin et al., 2013). The main advantage of this technique is the possibility to give spatial distribution information about the sample, compared to traditional NIR spectroscopy that provides only average spatial information. Each pixel of an hyperspectral image contains the spectrum of that specific position thus giving both spatial and spectra information of a sample (Dale, 2012).

2. Material and methods

2.1 Sample description

Ten biscuits from two commercial brands (Saint-Michel (SM), Petit Brun LU (PB)) were conditioned in 10 desiccators containing different saturated solutions of salt, having different water activities (Aw) going from 0.114 to 0.907 (Table 1). First, the biscuits were weighed and placed into the desiccators that were placed under vacuum for one week. These biscuits are coded from 01 to 10 according to increasing values of Aw. Two biscuits (one for each brand) were chosen as references and placed in plastic bag in the lab, and are numbered 11. After one week in the desiccators, cookies are weighed again and analyzed by NIR hyperspectral imaging. According to the weight difference observed between the beginning and the end of the experiment, percentage of water content were estimated for each biscuit and are presented in Table 2.

N° Desiccator and salt	*01* *LiCl*	*02* *CH_3COOK*	*03* *$MgCl_2$*	*04* *K_2CO_3*	*05* *$Mg(NO_3)_2$*	*06* *NaBr*	*07* *$CuCl_2$*	*08* *NaCl*	*09* *KCl*	*10* *$BaCl_2$*
Aw (20°C)	0.114	0.226	0.313	0.44	0.545	0.587	0.684	0.754	0.851	0.907

Table 1: Number of the desiccators and associated salts and water activities (Aw) at 20°C

Biscuit	*SM01*	*SM02*	*SM03*	*SM04*	*SM05*	*SM06*	*SM07*	*SM08*	*SM09*	*SM10*
water %	2.54	3.87	4.84	6.88	7.73	8.42	10.55	12.88	16.56	20.10
Biscuit	*PB01*	*PB02*	*PB03*	*PB04*	*PB05*	*PB06*	*PB07*	*PB08*	*PB09*	*PB10*
water %	2.63	4.25	5.24	7.48	8.72	9.85	13.19	15.95	20.46	23.18

Table 2: Percentage of water content for each biscuit

2.2 Hyperspectral NIR imaging system

Hyperspectral images were acquired with a pushbroom hyperspectral imaging system (BurgerMetrics, SIA, Riga, Latvia) consisting of a SWIR Xeva MCT camera (Xenics) combined with a Hyperspec SWIR imaging spectrometer (Headwall photonics) covering the spectral range of 950-2500nm with a spectral resolution of about 7nm. All images are acquired in absorbance and the final image size for each kernel is 300(x) × 281(y) × 212(λ), the first two values representing pixel dimensions in the x and y directions (field of view of 6.7 × 6.3mm, with a spatial resolution of 225µm) and the third value accounting for the number of spectral channels.
Near infrared hyperspectral images were then acquired for each sample and processed by chemometrics to reduce dimensionality of the data, to predict water content at each pixel of the images and to build false colors images of prediction.

2.3 Data processing

To extract the useful spectral information from the hypercubes, 1,000 pixels from each image were randomly selected and merged into a large matrix **S**. Smoothing and Standard Normal Variate (SNV) pre-processing were applied to this matrix, which was submitted to a Principal Component Analysis (PCA). Scores were gathered according to the belonging of the corresponding biscuit at a given conditioning and represented by a 95% confidence ellipse around the barycenter.
A selection of variables (wavelengths) was performed, by applying analysis of variance (ANOVA) to the matrix **S**. Ten variables were identified and introduced in the multiple linear regression algorithm to calibrate the model, including the calculations of R^2 and RMSEC. This model was applied to all the pixels and a predicted value of water content was obtained for each pixel. A new image so-called “prediction image”, based on these predicted values, can be drawn in false colors. Mean of predicted values for each image was computed and considered as the predicted water content of the product.
Multivariate image of samples stored in the lab, so-called n°11, was used as a test dataset for the model and the predicted water content estimated.

3. Results and discussion

3.1 Principal Component Analysis

A score plot of the first and second components explaining 58.7% of the variance is presented in Figure 1. Biscuits are perfectly classified along the first principal component which loading (not shown) presents the same general shape as the spectrum of pure water. The second principal component seems to be linked to the interaction between water and starch, as suggested by Delwiche et al. (1991).
These results show that it is possible to monitor the water content for the two brands of biscuits by NIR-HSI first, and thus on-line.
Hygrometry of the lab, considered as a reference value for the test of model with the sample n°11, was measured during the experiment, and evolved from 0.42 to 0.5. As water activity of sample n°04 is equal to 0.438, it is logical to find that samples coded 11 and 04 were plotted close to one another.

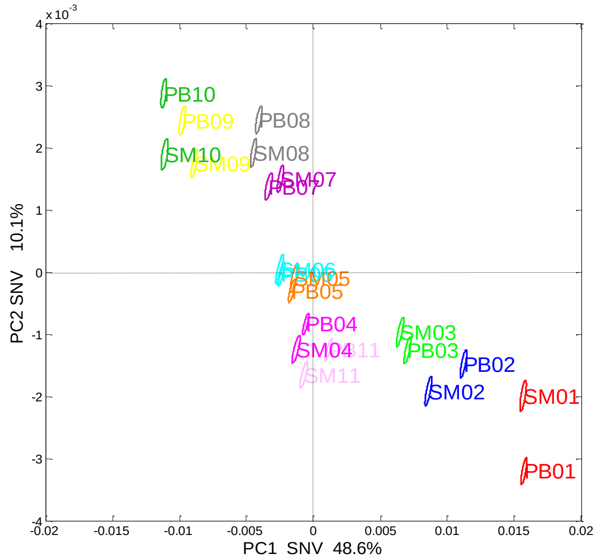

Figure 1: Score plot of the first and second components obtained using PCA

3.2 Multiple linear regression

3.2.1 Model results

The wavelengths selected by the algorithm are as follows:
1048 – 1218 – 1337 – 1411 – 1470 – 1722 – 1937 – 1967 – 2101 - 2301nm.
These variables were used for the calibration model of each brand.
Using the prediction model, false color images can be obtained for the eleven biscuits of each brand (Figure 2). A clear color gradient can be observed between the biscuits: blue is associated to low values and red to high ones. This gradient well matches with that of water activities (code: 01 to 10).
Mean of predicted values for each image was computed and considered as the predicted water content of the product (figure 2). The estimated parameter R^2 for “Petit-Brun” and “Saint-Michel” is equal to 0.9499 and 0.9388 respectively. The associated quadratic error of prediction model (RMSEC) is 0.0148 and 0.0132 for “Petit-Brun” and “Saint-Michel”, respectively.

3.2.3 Predicted water content

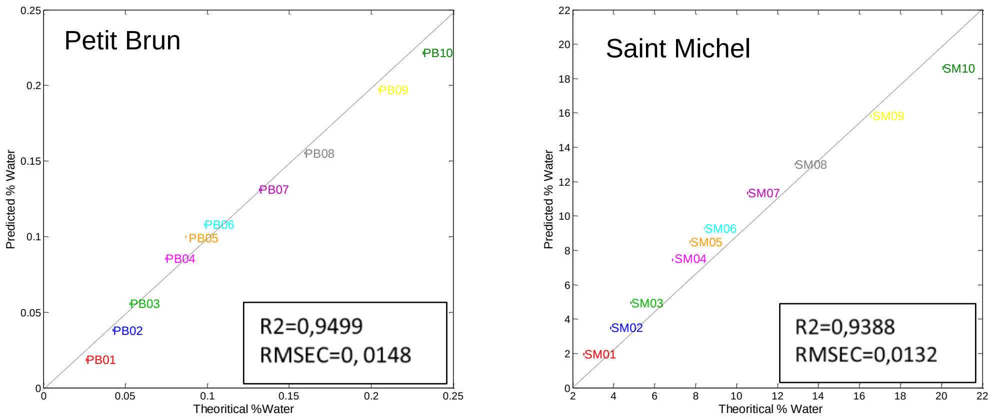

Figure 2: Predicted percentage of water versus theoretical percentage of water content for each biscuit

3.2.2 Prediction images

Prediction images are very relevant and can be used to study the homogeneity of water content in biscuits and eventually detect defects.

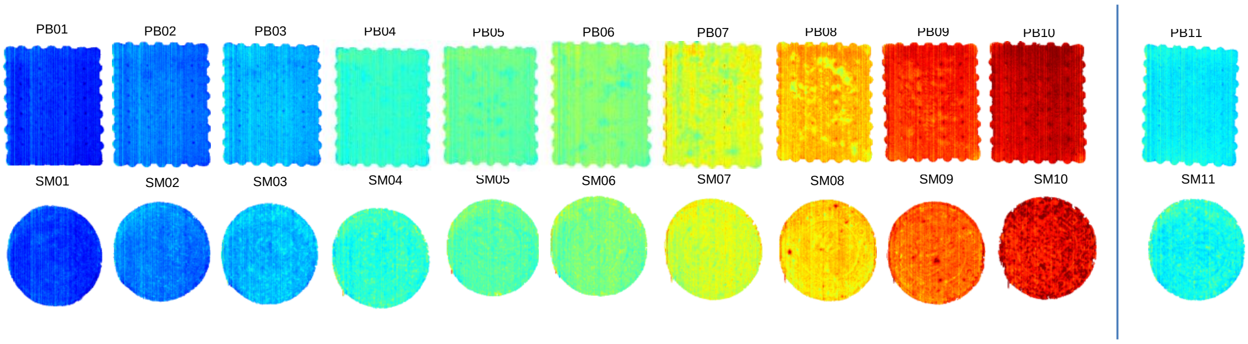

Figure 3: False color images obtained after applying the prediction model on the biscuits

The two kinds of biscuits show exactly the same behavior according to the increasing water activity. Some heterogeneities are visible in the Petit Brun biscuits for water content between 8 and 16%.
A "prediction image" has been obtained for the reference biscuit stored in the lab which confirms the result previously observed in PCA, that this biscuit has water content between the ones of the third and the fourth biscuit.

4. Conclusion

NIR-HSI appears to be a successful non-invasive method to predict the water content of food products, with high accuracy. This study shows that it is possible to create a model between NIR hyperspectral images and water content in biscuits, which were conditioned in desiccators with different water activities. Using this model, it is possible to build false color images showing the repartition of water in the biscuits.

The implementation of this technique online to measure water content along the process can be thus considered.

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