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

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Objectives of the study

For consumers, quality is the most important criterion for selecting a product. Although a broken product or cracks do not directly affect its taste, it is not really pleasant and even annoying to the consumer, especially if there is a large number of broken products.
Objectives of the study

The basic process of biscuit manufacturing

Commercial biscuits can often present quality fluctuations because of non-uniformity of baking conditions, and/or to further storage and handling.
Objectives of the study

The main objective of this study is to evaluate the potential of Near-Infrared Hyperspectral Imaging Spectroscopy (NIR-HSI) for the quantification of water content in commercial biscuits and determine if this technique could be implemented online.
Samples

Two commercial brands of biscuits produced in the western France.

<table>
<thead>
<tr>
<th>Composition (100 g)</th>
<th>Petit Brun</th>
<th>Saint-Michel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lipids</td>
<td>13 g</td>
<td>16 g</td>
</tr>
<tr>
<td>Saturated fat acids</td>
<td>6 g</td>
<td>11 g</td>
</tr>
<tr>
<td>Carbohydrates</td>
<td>75 g</td>
<td>74 g</td>
</tr>
<tr>
<td>Sugars</td>
<td>25 g</td>
<td>25 g</td>
</tr>
<tr>
<td>Fibers</td>
<td>2.9 g</td>
<td>2 g</td>
</tr>
<tr>
<td>Proteins</td>
<td>6.9 g</td>
<td>7 g</td>
</tr>
<tr>
<td>Salt</td>
<td>0.78 g</td>
<td>0.9 g</td>
</tr>
<tr>
<td>Sodium</td>
<td>0.307 g</td>
<td>0.300 g</td>
</tr>
</tbody>
</table>
Material and method

Ten biscuits for each commercial brand were conditioned in 10 desiccators containing different saturated solutions of salt, having different water activities (Aw).

One biscuit for each brand was kept in plastic bag in the lab (hygrometry between 0,42-0,5)

<table>
<thead>
<tr>
<th>N° Desiccator and salt</th>
<th>01 LiCl</th>
<th>02 CH₃COOK</th>
<th>03 MgCl₂</th>
<th>04 K₂CO₃</th>
<th>05 Mg(NO₃)₂</th>
<th>06 NaBr</th>
<th>07 CuCl₂</th>
<th>08 NaCl</th>
<th>09 KCl</th>
<th>10 BaCl₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aw (20°C)</td>
<td>0.114</td>
<td>0.226</td>
<td>0.313</td>
<td>0.44</td>
<td>0.545</td>
<td>0.587</td>
<td>0.684</td>
<td>0.754</td>
<td>0.851</td>
<td>0.907</td>
</tr>
</tbody>
</table>

All biscuits were weighted before and after conditioning. Dry matter was evaluated and according to the weight difference before and after conditioning, water percentages were estimated for each biscuit. Conditioning time: one week
Each biscuit is then imaged using the NIR-HSI system.
Material and method

NIR hyperspectral imaging system:

HyperPro (BurgerMetrics)

“Pushbroom” hyperspectral imaging system:

- Acquisition of an entire line (225μm spatial resolution)
- The belt advances along the y direction and a new line is acquired

Image obtained at the selected wavelength

1 min/image

\[ \lambda (212) \]
950-2500nm
Step size \sim 7nm
Data pre-treatment

ROI selection:

An automatic segmentation is performed on the averaged image for each data cube, resulting in a binary image (pixel values equal to 1 for the ROI and to 0 for the background).

Hypercube unfolding:

Smoothing+SNV
Data processing

Random sampling: 1000 pixels in each ROI

PCA analysis

Concatenation

ANOVA

Multiple linear regression

Validation model with ten variables

Application of the model to all pixels and construction of prediction images
PCA Results

Petit Brun

Saint-Michel

Increasing AW

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PCA Results

Water linked to starch
Lipid content

Free Water content

Loadings on PC1

PC1 48.8%
PC2 10.1%

Loadings on PC2 snv

Wavelength (nm)

Loadings on PC1 and PC2 snv
Anova Results

10 Wavelengths are selected for creating the regression model
## Regression Results

<table>
<thead>
<tr>
<th></th>
<th>Petit Brun</th>
<th>Saint-Michel</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Calibration model</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>R2</td>
<td>0.944</td>
<td>0.923</td>
</tr>
<tr>
<td>RMSEC</td>
<td>0.015</td>
<td>0.015</td>
</tr>
<tr>
<td><strong>Validation model</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>R2</td>
<td>0.950</td>
<td>0.927</td>
</tr>
<tr>
<td>RMSEP</td>
<td>0.015</td>
<td>0.015</td>
</tr>
</tbody>
</table>
Regression Results

Predicted percentage of water versus observed percentage of water content for each biscuit

Petit Brun

Saint-Michel
Regression Results

False color images obtained after applying the prediction model on the biscuits

Water content

0 10 20 22

PBO1 1.29  PBO2 3.59  PBO3 5.34  PBO4 8.51  PBO5 9.81  PBO6 10.6  PBO7 12.8  PBO8 15.4  PBO9 19.4  PBO10 21.8

SMO1 1.45  SMO2 3.37  SMO3 4.83  SMO4 7.39  SMO5 8.32  SMO6 9.15  SMO7 11.3  SMO8 13.1  SMO9 15.6  SMO10 18.3

SM11 6.82  PBO11 7.03

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Conclusion

✓ Two different commercial brands “Petit Brun” and “Saint-Michel” of biscuits were analyzed using NIR-HSI in order to investigate the potential of this technique to predict the relative humidity of this kind of products.

✓ These results obtained by PCA show that the water content of the two brands of biscuits can be monitored by NIR-HSI.

✓ The analysis of variance (ANOVA) help us to identify the ten most important variables.

✓ The regression model with 10 variables gives acceptable results ($R^2>0.92$), but it would be better to use one model per kind of biscuit, as they show a different behavior to water content (ex: the biscuits with less fats have a higher water content).

✓ Some heterogeneities inside the biscuits and also degradation on the borders are observable in the reconstructed false color images, especially for biscuits put in desiccators with high AW.

✓ NIR-HSI seems a very promising method to predict water content inside food products and could probably be implemented on line at different step of the process.
Perspectives

✓ Create regression models for each kind of biscuit (or product)

✓ Analyze and compare spectra where inhomogeneities are observed on the false-color images

✓ Increase the accuracy of the model on a narrow range in controlled atmosphere (around 5%)

✓ Do measurements at different steps of production (before cooking, after cooking, after cooling, etc...)
Thanks to

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Projet BRICE
Thanks for your attention
Regression methods comparison

Multiple regression
R2 = 0.984

PLS
R2 = 0.974

VIP PLS
52 variables selected
R2 = 0.927

Multiple regression
10 variables selected
R2 = 0.941
Two Way ANOVA

Water effect

Biscuit effect (fat content)

Interaction (protein, hydrocarbon?)