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Detection of low-level gluten content in flour and batter by near infrared reflectance spectroscopy (NIRS)

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ABSTRACT

Near infrared spectroscopy (NIRS) has been used as a valuable tool for quality control in the food industry. The aim of the present study was to investigate the possibility of developing a NIRS calibration for gluten determination in flour and batter, suitable for the analysis of gluten-free food products. Reflectance data was used for calibration based on modified partial least squares (MPLS) regression. Independent prediction equations were developed for flour and for batter. Spectral models using mean spectra of two scans (average spectra), were compared with those using the two individual spectral data. The best model obtained for flour was using the average spectral data ($R^2 = 0.985$; $r^2 = 0.967$) and for batter samples was using the individual spectral data ($R^2 = 0.926$; $r^2 = 0.825$). It is concluded that the application of NIRS methodology can predict accurately the concentration of gluten content in flours and batters, but it should not be considered as a reliable method for determining gluten contamination in gluten-free products.

1. Introduction

Celiac disease is an autoimmune-mediated enteropathy triggered by the ingestion of gluten-containing grains, wheat, rye, oat and barley, in genetically susceptible people. The reaction to gluten ingestion by sufferers of celiac disease is an inflammation of the small intestine leading to the malabsorption of several important nutrients including iron, folic acid, calcium, and fat soluble vitamins (Feighery, 1999). The symptoms of celiac disease vary widely and the only way to prevent them is to exclude gluten-containing cereals from the diet (gluten-free diet).

Gluten is the main structure-forming protein in wheat flour, giving to dough its elastic and extensible properties. Due to gluten functionality, the formulation of gluten-free products is a big challenge (Gallagher et al., 2004). Gluten is present in food products containing cereals mentioned before; however, it may also be present in other food products where gluten is added as a texture agent or as a vegetable source of protein. Moreover, natural gluten free products may contain gluten, due to the cross contamination occurring during the primary production, harvesting and storage of grain and/or during the manufacture of gluten-free food.

Immunochemical analytical methods are currently used to determine gluten in food products. The polymerase chain reaction (PCR), a DNA-based method of high specificity and sensitivity, has been proposed as an effective alternative for the detection of wheat or other gluten-containing cereals (Dahinden et al., 2001). These methods are both laborious and expensive, and near infrared spectroscopy (NIRS) technique could be an alternative. NIRS offers a number of important advantages over traditional chemical methods. It is a physical, non-destructive method, requiring minimal or no sample preparation, no reagents are required, no wastes are produced and several components can be determined simultaneously from a single spectrum with the help of the multivariate calibration process.

The use of NIRS for quality control of cereals is well established in the literature (Osborne, 2000) and has been introduced successfully as a rapid technique for grain (Miralbés, 2003; Scholz et al., 2007), flour (Baslar and Ertugay, 2011; Miralbés, 2004; Paulsen et al., 2003), dough (Alava et al., 2001; Kaddour and Cuq, 2011; Sinelli et al., 2008) and bread (Osborne et al., 1984;

Abbreviations: NIRS, near infrared reflectance spectroscopy; SNV, standard normal variate; DT, detrend; MSC, multiple scatter correction; MPLS, modified partial least squares; SD, standard deviation; SEC, standard error of calibration; SEP, standard error of prediction; RPD, ratio of performance to deviation; RER, range error ratio; R^2, coefficient of determination for calibration; r^2, coefficient of determination for validation.

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Sørensen, 2009). In wheat flours, protein fractions (gliadin and glutenin) have been predicted using NIRS techniques (Wesley et al., 2001).

Rapid infrared spectroscopy methods have been successfully used in adulteration detection for a wide range of complex food products, including oils, milk and wheat (Cocchi et al., 2006; Kasemsumran et al., 2007; Ozen et al., 2003) and Norris (2009) proposed a simple method using multiple calibrations on a single near infrared scan to detect adulteration in food products.

The objective of the present study was to investigate the possibility of developing a NIRS calibration, as an alternative method for the detection of low-level gluten presence in flours and in different times of batter development, using a NIRS instrument without optical fibre.

2. Materials and methods

2.1. Samples

Due to the inability to find common gluten-free bakery samples which contain natural variations of low and very low gluten levels, it was decided to contaminate commercially gluten-free flours with small amounts of wheat flour. Two commercial gluten-free flour mixes and one corn starch were mixed (adulterated) with two different commercial wheat flours, and in order to obtain more variation, each wheat flour was mixed with one gluten-free flour and with a combination of the gluten-free flours. We obtained 7 different combinations of gluten-free flours, which were mixed with different concentrations of two wheat flours. The manufacture of combinations resulted in 144 samples with a final concentration from 0 to 4.5% (w/w) of wet gluten content (Table 1).

Usually, gluten-free flours produce liquid systems, due to the lack of a gluten network. This is the reason why "batter" is the most common name to refer to gluten-free dough. It must also be mentioned that samples containing just corn starch were not possible to study during fermentation time, due to the impossibility to form a mixing structure able to develop a stable batter.

To prepare the batter ($n = 88$) on a laboratory scale, water at 20 °C (±1 °C) and yeast at 5% concentration were added to the samples. Once mixed, shortening (at 4.7%) was added at the final step of mixing. Shortening make the batters for gluten-free bread more workable and renders the final product more tender and moist. After homogenization, samples were proofed for 45 min at 37 °C. During fermentation the action of yeast results in the production of carbon dioxide and this increases batter volume.

Wet gluten content was determined according to the ICC standard method No. 137 (2001). In wheat flour, there is a plastic–elastic substance consisting of gliadin and glutelin, obtained after washing out the starch from wheat flour dough. Wet gluten was washed and separated from 10 g of flour with 2% chloride buffer using the glutomatic equipment (Perten, Stockholm, Sweden). The determinations were made in duplicate and the differences did not exceed 0.5%.

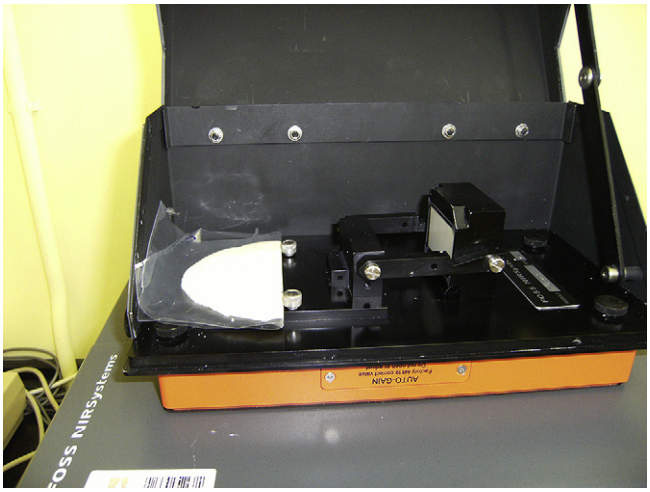

Fig. 1. Presentation of batter sample for near infrared reflectance measurements.

2.2. NIRS analysis

All samples were recorded from 1100 to 2500 nm using a NIRSystems 5000 scanning monochromator (FOSS, Hillerød, Denmark). Reflectance was recorded in 2 nm steps, which gave 692 data points for each sample, as log (1/*R*) where *R* represented reflected energy. The flour analysis was carried out in duplicate using ring cup cells. In order to make the manipulation of the batter samples easier, instead of using the ring cup cells, the batter samples were covered with a plastic layer (always the same type of plastic) and were scanned as shown in Fig. 1. All measurements were performed by the same operator.

Batter samples were scanned in duplicate at time 0 (initial time) and after 45 min (final time), when the fermentation process had been completed and the batter samples had increased their volume. The batter calibrations were developed using respectively, the initial and final sampling times (0 and 0045), and a calibration derived by combining the two sampling times.

A WinISI III (v. 1.6) software program was employed for spectra data analysis and development chemometric models. Prior to calibration, log 1/*R* spectra were corrected for the effects of scatter using the standard normal variate (SNV), detrend (DT) and

Table 1
Thirty five wet gluten concentrations (w/w) used to obtain the NIRS calibrations. Summary of wet gluten content (%) of flour and batter samples used in calibration and validation sets.

0 ppm	80 ppm	90 ppm	150 ppm	180 ppm	380 ppm	450 ppm	0.08%	0.09%
0.19%	0.23%	0.38%	0.45%	0.58%	0.68%	0.77%	0.90%	0.96%
1.13%	1.15%	1.34%	1.35%	1.53%	1.58%	1.80%	1.92%	2.25%
2.30%	2.68%	2.70%	3.07%	3.15%	3.60%	3.83%	4.50%	

	Calibration set, %				Validation set, %			
	N[a]	Range	Mean	SD[b]	*N*[c]	Range	Mean	SD[b]
Flour	108	0.0–4.5	1.15	1.222	36	0.0–4.5	1.17	1.253
Batter, general	132	0.0–4.5	1.37	1.218	44	0.0–4.5	1.37	1.200
Initial batter	66	0.0–4.5	1.41	1.250	22	0.0–4.5	1.26	1.116
Final batter	66	0.0–4.5	1.41	1.250	22	0.0–4.5	1.26	1.116

[a] Number of calibration samples.
[b] Standard deviation.
[c] Number of validation samples.