Prediction of malt quality on whole grain and ground malt using near infrared spectroscopy and chemometrics

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Introduction

The quality of malt used in a brewery is important for the brewing process and the end quality. Quality evaluation of malting barley and malt is expensive and time-consuming, involving both micro-malting and mashing. In breeding, as well as in quality documentation, there is a need for fast and automated instrumental analyses. Near infrared (NIR) spectroscopy is a well-established rapid method for quality determination in cereals. The objective of the current investigation is to compare NIR transmission with NIR reflectance spectroscopy. In the NIR reflectance mode, measurements on whole malt grains are compared with measurements on ground samples and full spectra partial least squares models are compared with reduced models based on interval partial least squares (iPLS).[1]

Material and methods

50 micro-malt samples, representing 25 different varieties, grown at two different locations in Denmark, were analysed. The micro-malt samples are measured in three different modes: NIR transmission, Infratec 1255 Food and Feed Analyzer on whole grains in the range of 850–1048 nm; NIR reflectance, NIRSystems 6500 on whole grains; and NIR reflectance, NIRSystems on ground samples, both in the range of 400–2500 nm.

The samples are analysed for the five parameters: β-glucan in malt, nitrogen in malt, extract, modification and β-glucan in wort, all according to Analytica-EBC.[2] The chemometric calculations are performed using Unscrambler V. 7.01 (CAMO A/S, Trondheim, Norway) and Matlab V. 5.2 (The MathWorks, Inc.).

Results and discussion

The NIR transmission spectra are limited to the range of 850–1048 nm (100 datapoints) and are compared to the NIR reflectance spectra ranging from 400 to 2500 nm (1050 datapoints), which includes the visible spectral range. The NIR reflectance spectra of the 50 whole grain micro-malt samples are shown in Figure 1.

The NIR spectra are used for prediction of the five malt quality parameters using PLS regression. The 50 samples are divided into five subgroups of ten for validation. The models are compared according to their root mean squares error of cross-validation (*RMSECV*), by the correlation between measured and predicted and by the number of PLS components required.

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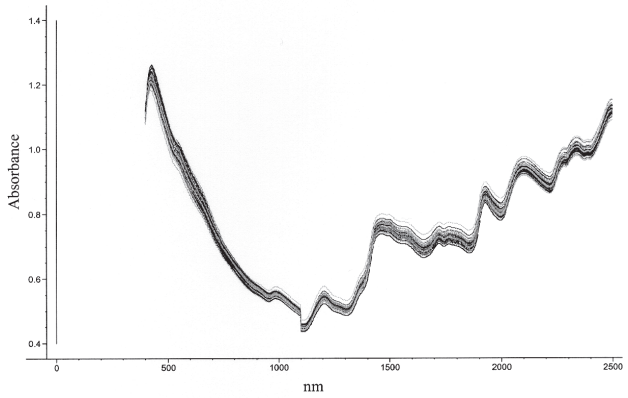

Figure 1. NIR reflectance spectra of whole malt kernels in the range of 400–2500 nm.

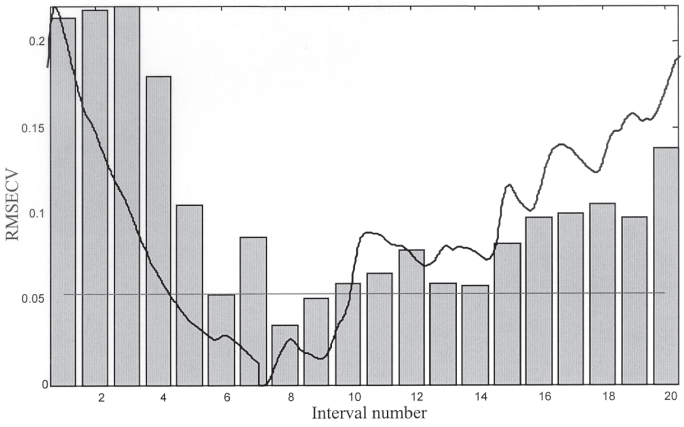

Figure 2. Interval selection using iPLS for the prediction of nitrogen in malt using the NIR reflectance spectra of whole malt kernels.

At Food Technology, The Royal Veterinary and Agricultural University, an iPLS algorithm has been developed[1] in which local PLS models of the full-spectrum are generated. In this way it is possible to focus on important spectral regions and remove interferences from other regions, thereby improving the model. After finding the region with the lowest *RMSECV*, the interval is further optimised by shifting the interval and changing the interval width.

Figure 2 shows an example in which NIR reflectance spectra of whole malt kernels are divided into 20 intervals. The *RMSECV* of the full-spectrum model predicting nitrogen in malt is shown, where the horizontal line indicates the *RMSECV* for the full-spectrum model, together with the average of the spectra. The bars represent the *RMSECV* for the different intervals and, as can be seen, interval number 8 has a considerably lower *RMSECV* than the full-spectrum model. Figure 3 shows the predicted v. measured plot, using the optimised interval ranging from 1130 to 1316 nm where the *RMSECV* is reduced from 0.05 to 0.03% N.

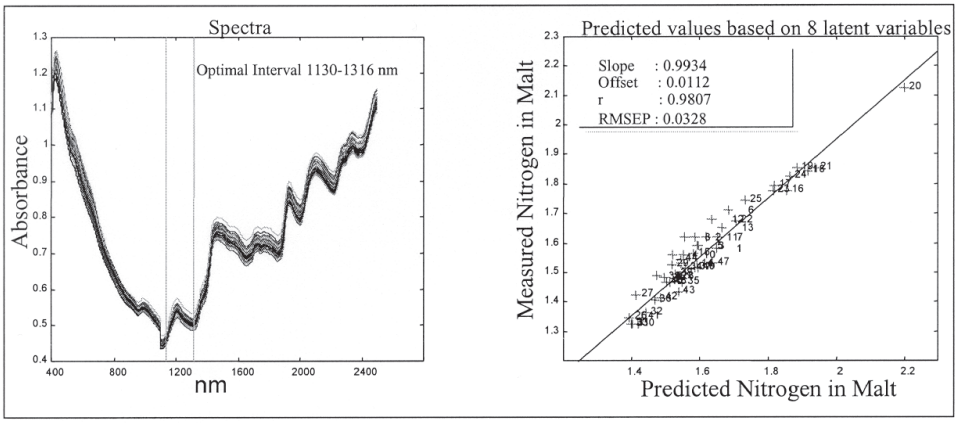

Figure 3. Prediction of nitrogen in malt using the spectral region of 1130–1316 nm.

Tables 1 and 2 summarise the performances of the NIR reflectance models, based on measurements on whole malt kernels and ground malt, with the full-spectrum models as well as with the models based on iPLS optimised models. In all ten NIR reflectance models, considerable improvements are seen, both with regard to prediction error and model complexity, i.e. the number of PLS components when using the optimised spectral region. No model improvements are seen when measuring ground sample compared to whole grain measurements. Transmission studies with the NIRSystems 6500 were unsuccessful (data not shown).

Table 1. Calibration results using NIR reflectance on whole malt grains. The full spectra models are compared with optimal iPLS interval. The table includes parameter, selected wavelength, correlation coefficient, number of PLS components (# PLS), root mean square error of cross-validation (*RMSECV*) and *RMSECV* divided by the range of the parameter (*RMSECV*/range).

Parameter	Wavelength (nm)	Correlation	# PLS	*RMSECV*	*RMSECV*/range (%)
β-glucan in malt	400–2500	0.89	10	0.20	12.6
	1330–1442	0.93	7	0.17	10.7
Nitrogen in malt	400–2500	0.95	10	0.05	6.3
	1130–1316	0.98	8	0.03	3.8
Extract	400–2500	0.92	9	0.6	8.8
	1204–1410	0.97	8	0.39	5.7
Modification	400–2500	0.87	10	5.6	12.4
	1348–1410	0.95	7	3.5	7.8
β-glucan in wort	400–2500	0.91	10	118	10.3
	1334–1436	0.97	7	75	6.5

Table 2. Calibration results using NIR reflectance on malt flour. The full spectra models are compared with optimal iPLS interval. The table includes parameter, selected wavelength, correlation coefficient, number of PLS components (#PLS), root mean square error of cross-validation (*RMSECV*) and *RMSECV* divided by the range of the parameter (*RMSECV*/range).

Parameter	Wavelength (nm)	Correlation	# PLS	*RMSECV*	RMSECV/range (%)
β-glucan in malt	400–2500	0.82	6	0.26	16.4
	1388–1598	0.87	4	0.22	13.9
Nitrogen in malt	400–2500	0.93	7	0.06	7.5
	2082–2164	0.95	2	0.05	6.3
Extract	400–2500	0.92	8	0.6	8.9
	2110–2140	0.94	3	0.48	7.0
Modification	400–2500	0.82	4	6.4	14.2
	2276–2386	0.87	2	5,4	12.0
β-glucan in wort	400–2500	0.92	10	112	9.8
	1368–1436	0.92	6	112	9.8

Table 3. Calibration results using NIR transmission on whole malt grains. The table includes parameter, selected wavelength, correlation coefficient, number of PLS components (#PLS), root mean square error of cross-validation (*RMSECV*) and *RMSECV* divided by the range of the parameter (*RMSECV*/range).

Parameter	Wavelength (nm)	Correlation	# PLS	*RMSECV*	*RMSECV*/range (%)
β-glucan in malt	850–1048	0.90	10	0.19	12.0
Nitrogen in malt	850–1048	0.97	6	0.04	5.0
Extract	850–1048	0.95	13	0.48	7.0
Modification	850–1048	0.89	10	5.3	11.7
β-glucan in wort	850–1048	0.91	10	126	11

For the NIR transmission Infratec spectra, no improvements were obtained using the iPLS algorithm, probably due to the narrow range. The performances of the full-spectrum NIR transmission models are shown in Table 3. Only minor differences in predictive performances are seen when comparing the optimised NIR reflectance models with the NIR transmission models, but the NIR reflectance models are considerably lower in model complexity.

Conclusions

NIR reflectance spectroscopy on whole malt grains can be used for determination of malt quality with accuracies comparable to near infrared transmission. The iPLS algorithm has improved NIR reflectance-based models considerably. The iPLS algorithm did not improve NIR transmission-based models.

References:

1. L. Nørgaard, A. Saudland, J. Wagner, J.P. Nielsen, L. Munck and S.B. Engelsen, submitted to *Appl. Spectrosc*. (1999).
2. *Analytica-EBC. Analysis by the European Brewery Convention*, 4. Edition. Brauerei- und Getränke-Rundschau, Zurich, Switzerland (1987).