

Determination of starch and amylose contents in various cereals using common model of near-infrared reflectance spectroscopy

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Abstract

Near-infrared reflectance spectroscopy (NIRS) was used to determine the total starch and amylose contents in various kinds of cereals namely wheat, waxy rice, non-waxy rice, millet, sorghum, waxy maize, buckwheat, barley, and hullless oat. The partial least-squares (PLS) analysis and principal component regression (PCR) were used to establish the calibration models. PLS model achieved a better effect than PCR at 1100 - 2500 nm, and the coefficient of determination (R^2) of the calibration and prediction sets were both higher than 0.9 after the best pre-treatment method, first derivative plus Savitzky-Golay. Additionally, the root mean square error (RMSE) was lower than 2.50, and the root mean square error of cross-validation (RMSECV) was less than 3.50 for starch. By comparing PLS models at different waveband regions, the optimal determination results for starch and amylose were obtained at 1923 - 1961 and 1724 - 1818 nm, respectively. NIRS was found to be a successful method to determine the starch and amylose contents in various cereals.

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Introduction

Cereals, which mainly include rice, wheat, oat, and sorghum, are a rich source of carbohydrates, vitamins, minerals, oils, and proteins. Global cereal production reached approximately 3,000 million tons, and accounted for more than 73% of the global harvest area. The outputs of maize, rice, and wheat were 10.40, 7.41, and 7.29 hundred million tons, respectively, accounting for 89% of the total cereal production (according to the Food and Agriculture Organization (FAO) statistics). Starch is the most important component of cereals as its content accounts for over 80% of rice (Souza *et al.*, 2016), at least 70% of wheat (Rahman *et al.*, 2000), 62% of maize (Longe *et al.*, 1982), and roughly 75% of millet (all on a dry weight basis).

Starch is mainly composed of two glucans, namely the linear and helical amylose, and the branched amylopectin. Native cereal starches have varied content of amylose, *e.g.*, *Indica* and *Japonica* rice have approximately 20% amylose, waxy rice only contains < 2% amylose, and waxy maize and amylo maize starch have a wide range of amylose from 1 to 70% (Buckow *et al.*, 2007). The amylose content and the multi-scale structure of starches are the major factors determining the physico-chemical

properties of rice (Wang *et al.*, 2017). Starch with higher amylose content has lower crystallinity, higher retrogradation rate, lower gel temperature, lower viscosity of gelatinisation characteristics, poorer waxy quality, and is also harder to be digested than low-amylose starch (Zhang and Hamaker, 2012; Wang and Copeland, 2015).

Starch content is usually measured by the Association of Official Agricultural Chemists (AOAC) method 996.11, by using acid hydrolysis (AOAC, 1997). Single- and double-wave colorimetric methods are used for the determination of amylose (Tian *et al.*, 2015). However, the traditional analysis process is time-consuming and non-environmentally friendly. Near infrared reflectance spectroscopy (NIRS) is a rapid, non-destructive, environmentally friendly, multi-analytical, and highly accurate determination method (Bázár *et al.*, 2016). Recently, NIRS has become a popular method in a wide range of agricultural and food industry applications, and it is even used in some online monitoring systems. Siriphollakul *et al.* (2017) analysed the eating qualities (amylose content and texture properties) of rice using NIRS. The protein and gluten contents of wheat flour (Chen *et al.*, 2017), and the protein and amylose contents of rice flour have been successfully

determined by NIRS (Xie *et al.*, 2014).

Previous literature mainly concentrated on only one kind of cereal to obtain an accurate model, but no common model suitable for all kinds of cereals disregarding the difference in chemical compounds has been established. Therefore, the present work aimed to explore the feasibility of employing NIRS in determining the starch and amylose contents in various cereals including waxy and conventional types. Pre-treatments and chemometric methods including partial least squares (PLS) and principal component regression (PCR) were applied to establish the models and perform the analysis.

Materials and methods

Plant materials

A total of 101 cereal samples were purchased from the markets in Beijing, China. The selection of cereal samples was based on their varieties and production. The samples consisted of nine kinds of cereals: wheat (Bainong AK58, 2; Jimai 22, 2), waxy rice (round waxy rice, 11; long waxy rice, 10), non-waxy rice (*Japonica* rice: Changlixiang, 5; Daohuaxiang, 5; Pearl rice, 5; Komachi rice, 2; *Indica* rice: Thai Fragrant rice, 5; Simiao rice, 5; Maoya rice, 5), millet (Qinzhou yellow millet, 5; Jingu 21, 4; Longshan millet, 3; Taohuami, 3), sorghum (Dongliang 80, 4), maize (Nonghua 101, 4; Denghai 6702, 4), waxy maize (Zhefengnuo 7, 4; Zhongnuo 301, 4), buckwheat (Yuqiao 3, 3; Tianqiao 92-1, 2), barley (Pu barley 7, 2), and hullless oat (Huabei 2, 2), which were obtained from different varieties and producing areas of China. All cereal samples were ground and passed through a 0.2 mm sieve.

The standards (amylose and amylopectin) were purchased from Sigma-Aldrich (Saint Louis, MO, USA). All other analytical grade chemicals were purchased from Sinopharm Chemical Reagent Co., Ltd., (Beijing, China).

Content analysis of starch and apparent amylose

The starch content was analysed by method 996.11 (AOAC, 1997). Apparent amylose content (AAC) was determined by forming an iodine-binding complex of soluble starch, and measuring its absorbance using a U-3010 Spectrometer (Hitachi, Tokyo, Japan) at 620 nm (Xie *et al.*, 2014). The AAC was calculated based on a standard curve plotted from different ratios of amylose and amylopectin solutions.

Near-infrared spectra acquisition

The near-infrared reflectance spectra of

cereal samples were recorded at 1100 - 2500 nm with an ANTARIS near-infrared Fourier transform spectrometer (Nicolet Instruments Corp., Thermo-Fisher Scientific, Madison, WI, USA) equipped with a halogen lamp light source and an InGaAs detector. Approximately 10 g of powder of each sample was loaded in a round cup with an internal diameter of 38 mm and a depth of 10 mm. The sample was plated with at least 5 mm of thickness to prevent light transmission. Each sample was scanned three times, and each spectrum was collected by 32 scans at a resolution of 2 cm⁻¹.

Statistical analysis

The NIR spectra were imported into the OMNIC software (Nicolet Instruments Corp., Thermo-Fisher Scientific, Waltham, MA, USA) to obtain the average spectrum of each sample. Analysis of the data was performed by PCR and PLS using the TQ Analysis V6.0 software (Thermo Fisher Scientific, Waltham, MA, USA). PCA is a statistical method that is applied to extract the main information from NIR spectra, and turn a set of possibly correlated variables into a series of linearly uncorrelated variables (PCs) (Hu *et al.*, 2017). PCR is a regression analysis technique based on PCA. PCR considers regressing the outcome on a set of covariates based on a standard linear regression model, and uses PCA to estimate the unknown regression coefficients in the model. The PLS were evaluated using the leave-one-out cross-validation. Pre-treatment methods, including smoothing (Savitzky-Golay and Norris) and derivatives (first derivative and second derivative) were used to eliminate the interference of irrelevant information before calibration stage. Savitzky-Golay filters utilise a local polynomial fit controlled by three parameters to smooth and derivatise the spectra (Savitzky and Golay, 1964). Norris derivative smoothing is used as a variable selection step, and the random noise is removed (Liu *et al.*, 2015).

Using the spectroscopic signal to set up the prediction model makes the PLS analysis as the most widely used multivariate calibration technique (Ge *et al.*, 2014). PLS analysis has no restriction in the selected number of wavelengths, and it extracts the maximum information from the spectra to obtain a suitable model (Xie *et al.*, 2009). The prediction performance was assessed by the coefficient of determination (R^2), the root mean square error of prediction (RMSEP), the root mean square error of correction (RMSEC), the root mean square error of cross-validation (RMSECV), and the ratio of standard deviation (SD) to RMSEP (RPD). The

optimal number of latent variables was determined by the lowest value of the predicted residual error sum of squares (Shao *et al.*, 2011). Models with lower RMSEC, RMSEP, RMSECV, and higher R^2 and RPD are considered better (Xie *et al.*, 2009). For the PLS model establishment, 80 samples (16 waxy rice, 28 non-waxy rice, 12 millet, 3 wheat, 6 maize, 6 waxy maize, 4 buckwheat, 3 sorghum, 1 hulless oat, and 1 barley) among 101 cereal samples were allocated at random from each cereal type to the calibration set to build the models, and the other 21 samples were used as the validation sets.

Results and discussion

Analysis of starch and AAC in cereals

The mean, range, standard deviations (SD), coefficient of variation (CV) of the starch, and AAC of all the various kinds of cereals are presented in Table 1. The starch contents of all the samples were between 67.03 - 79.57% with the mean of 72.34%. The starch contents of the various samples are consistent with previous reports. The contents of carbohydrate in non-waxy rice, waxy rice, millet, wheat, maize, barley, sorghum, and buckwheat from China are 71.8 - 77.5, 74.7 - 79.0, 73.5 - 75.1, 71.5 - 76.1, 66.6 - 69.6, 63.4 - 65.2, 70.4 - 74.7, and 66.5 - 67.8%, respectively, based on wet weight (Yang *et al.*, 2009). In previous studies, the starch contents of millet and sorghum from Nigeria were 75.0 - 85.0 and 55.6 - 75.2%, respectively, based on dry weight (Gaffa *et al.*, 2004). The starch content of waxy rice of Indian varieties was 83.48% (Pachua *et al.*, 2017), which is higher than that of the Chinese varieties assessed in the present work.

There was a significant difference in the AAC of the cereal samples, which ranged from 0.37 - 23.28%. These values indicated that the amylose

diversity of the various kinds of cereals was well represented by the selected samples. Similar results were found in previous studies, *e.g.*, 11.67 - 27.59% amylose in non-waxy rice (Yu *et al.*, 2009), 12.2 - 28.6% in rice (Azudin and Morrison, 1986), $2.2 \pm 0.27\%$ in waxy rice (Pachua *et al.*, 2017), 21.3% in millet, and 20.1 - 21.4% in sorghum (Gaffa *et al.*, 2004).

NIRS spectra analysis

The original spectra of the cereals closely resembled each other (Figure 1). The typical vibrational bands mainly corresponded to the O-H stretch, O-H combination, and H-O-H deformation combination between 1920 - 1960 nm (Lohumi *et al.*, 2014). The band at 1400 - 1600 nm is very sensitive to hydrogen bonding in the starch, which is also related to the first overtone of the hydroxyl groups (Noah *et al.*, 1997). Similar to a previous study, the band at 1580 nm corresponds to the first overtone of the O-H stretching in starch (Niu *et al.*, 2012). The intensive absorption band at approximately 2100 nm is related to the O-H bend and C-O stretch combination, the one at 2500 nm is connected with the C-H stretch, and the C-C and C-O-C stretches in starch (Aenugu *et al.*, 2011).

The absorption peaks between 1585 - 1595 nm are associated with the vibrations of intermolecular hydrogen-bonded O-H groups in amylose (Salgó and Gergely, 2012). In addition, the spectral region at 1700 - 1820 nm had a good correlation with the amylose content (Fertig *et al.*, 2004). Also, the small absorption band at 1455 - 1977 nm might be related to the C-H second overtone (Bagchi *et al.*, 2016). However, it was difficult to find a specific band linearly related to the starch or amylose content. Accordingly, chemometric methods should be used to pre-treat the original NIR spectra and establish the

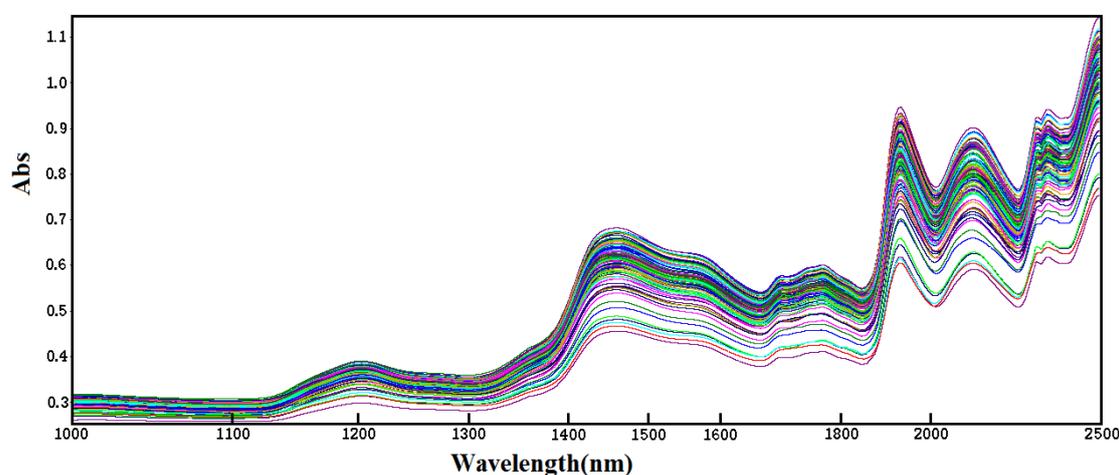


Figure 1. Near-infrared reflectance spectra of various cereal samples.

Table 1. Starch and amylose content of cereals.

	Rice	Waxy rice	Millet	Wheat	Maize	Waxy maize	Buckwheat	Hulless oat	Sorghum	Barley
Starch content										
Mean (%)	73.56	72.15	69.11	73.80	72.05	73.23	71.41	78.57	69.69	75.06
SD	2.30	2.19	2.18	1.32	0.98	1.48	1.25	1.42	1.71	3.72
Range	69.95~79.29	68.83~78.05	67.03~75.00	72.46~75.48	70.74~73.84	70.80~77.9	69.95~72.74	77.56~79.57	67.27~71.2 2	72.43~77.69
CV (%)	3.13	3.04	3.15	1.79	1.36	2.02	1.75	1.81	2.45	4.96
Total mean (%)	72.34									
Total SD	2.74									
Total range	67.03~79.57									
Amylose content										
Mean (%)	16.82	1.63	10.42	21.15	19.60	13.84	15.40	11.52	16.26	15.91
SD	2.20	0.67	2.33	2.34	2.97	0.53	2.35	0.48	1.83	0.99
Range	13.63~21.35	0.37~2.56	7.84~14.51	17.67~22.65	15.04~23.28	13.16~14.75	12.61~18.54	11.18~11.86	13.98~18.3 7	15.21~16.61
CV (%)	13.08	41.10	22.36	11.06	15.15	3.83	15.26	4.17	11.25	6.22
Total mean (%)	12.65									
Total SD	6.58									
Total range	0.37~23.28									

calibration models.

Establishment of the PCR models with full waveband

The conclusion parameters of the PCR models of starch and amylose by various pre-treatment methods are presented in Table 2. The optimised results for the detection of the starch and amylose contents were obtained using the first derivative plus Norris derivative smoothing (1 segment length and 1 gap between segment). The PC_{\max} , R^2_c , R^2_p , RMSEC, RMSEP, RMSECV, RPD, and prediction error of the starch content were 21, 0.9439, 0.8932, 0.909, 1.26, 1.26, 2.17, and -2.01~2.49, respectively; while for amylose, they were 22, 0.9473, 0.9065, 2.12, 2.59, 3.28, 2.54, and -2.04~2.40, respectively. Good prediction results were also obtained for the texture parameters of cooked cereals with R^2_c over 0.94 in the PCR models (Hu *et al.*, 2018). The accuracy of the PCR models was slightly lower than that of the PLS model, which is consistent with a previous study (Niu *et al.*, 2012). The PLS model was also better than the PCR models in the estimation of the wheat protein content (Mahesh *et al.*, 2015) and rice protein content (Xie *et al.*, 2014). The prediction error of the AAC was much higher than that of the starch in both the PLS and PCR models, probably because the chemical method for the determination of the AAC is more complex and has lower accuracy than that used for starch measurement, which also highlights the practicability of the NIRS method.

Establishment of the PLS models with full wavebands

The PLS model was established after the derivatisation and smoothing pre-processing on the NIR spectra. Four spectral pre-treatments including the first derivative, second derivative, first derivative plus Savitzky-Golay, and first derivative plus Norris smoothing were used to optimise the calibration model. Each combination of the pre-processing methods was tried, and the parameters of treatments were gradually adjusted to obtain the best calibration model (Ren and Chen, 1999). The optimal model for the starch content and AAC was that pre-treated by the first derivative plus Savitzky-Golay (five data points, three polynomial order smoothing treatments). The R^2 of the calibration and prediction sets were 0.9746 and 0.9028 for starch, and 0.9853 and 0.9334 for amylose, respectively (Table 2). The RMSEC were 0.617 and 1.13, the RMSEP were 1.21 and 2.23, the RMSECV were 1.29 and 3.31, and the RPD were 2.26 and 2.95 for starch and amylose, respectively. For the optimal PLS model of starch and amylose

content, the R^2 were higher than 0.97, and the RMSEP was lower than 2RMSEC, revealing a clear relationship between spectra and content, which means that the established models in the present work were practicable with satisfactory precision and acceptable robustness after the derivatisation and smoothing pre-processing on the NIR spectra. According to a previous report (Shao *et al.*, 2011), the PLS model of NIR region (1100 - 2500 nm) was found to be the best for the prediction of rice starch, and the R^2_c , R^2_p , and RMSEP were 0.926, 0.913, and 0.241, respectively. The prediction errors of the starch and amylose contents in all samples were -1.60~2.00 and -4.65~3.45, respectively. In the determination of different barley varieties using NIR, the total starch model showed a good R^2 of 0.96, and a RMSECV of 0.79, while the R^2 and RMSECV of the amylose model were both 0.94 (Ping *et al.*, 2013). A modified PLS model based on eight and two data points in the first and second smoothing were determined to be the best model to measure amylose content in brown and millet rice flour, and the R^2_c were 0.92 and 0.95, respectively (Xie *et al.*, 2014).

Establishment of the PLS models with selected wavebands

Some specific wavebands are more closely related to starch and amylose in cereals, *e.g.*, 1700 - 1820 nm is related to the amylose (Fertig *et al.*, 2004). Therefore, the determination results of the PLS models can be further improved if the related information of the absorbance spectra is extracted. The bands at 2494 - 2500, 2278 - 2283, 1923 - 1961, 1538 - 1587, 1724 - 1818, and 1515 - 1587 nm that are associated with the C-H/C-C/C-O-C stretch, the CH_2 deformation, the O-H stretch/O-H combination/H-O-H deformation, the vibrations of intermolecular O-H groups, and the C-H second overtone, respectively, were selected to establish separate models. The best pre-processing technique obtained from the full waveband was also applied to the selected waveband, and the PLS models were re-established.

The optimised wavelength range was 1923 - 1961 nm for the PLS model of starch content in cereals, with R^2_c of 0.9747, R^2_p of 0.9061, RMSEC of 0.616, RMSEP of 1.20, RMSECV of 1.25, RPD of 2.28, and prediction error of -1.55~2.15 (Table 3). This indicated that the O-H stretch, the O-H band combination, and the H-O-H deformation combination in the starch molecule from the various cereals produced the highest impact on modelling (Figure 2).

For the establishment of the amylose model,

Table 2. Modelling results of the starch and amylose content computed by PLS and PCR, pre-treated with derivative and smoothing at 1100 - 2500 nm.

Modelling method	Compound	Pre-processing	PLS Factors/PC _{max}	R ² _c	R ² _p	RMSEC	RMSEP	RMSECV	RPD	Prediction error
PLS	Starch	first derivative	10	0.9696	0.9009	0.674	1.22	1.28	2.25	-1.75~2.11
		second derivative	9	0.9951	0.7436	0.273	1.74	1.48	1.57	-2.13~3.40
	Amylose	first derivative + Savitzky-Golay	10	0.9746	0.9028	0.617	1.21	1.29	2.26	-1.60~2.00
		first derivative + Norris	10	0.9696	0.9009	0.674	1.22	1.28	2.25	-1.75~2.11
	Starch	first derivative	9	0.9688	0.9140	1.640	2.51	3.19	2.62	-4.98~5.22
		second derivative	10	0.9966	0.6853	0.548	4.50	4.70	1.46	-7.89~6.47
		first derivative + Savitzky-Golay	11	0.9853	0.9334	1.130	2.23	3.31	2.95	-4.65~3.45
		first derivative + Norris	9	0.9688	0.9140	1.640	2.51	3.19	2.62	-4.98~5.22
	Amylose	first derivative	8	0.7589	0.7722	1.790	1.94	1.99	1.41	-4.14~4.81
		second derivative	14	0.8749	0.7203	1.330	1.85	1.80	1.48	-2.68~5.39
first derivative + Savitzky-Golay		18	0.9368	0.9001	0.617	0.96	1.31	2.85	-2.15~2.67	
first derivative + Norris		21	0.9439	0.8932	0.909	1.26	1.26	2.17	-2.01~2.49	
PCR	first derivative	14	0.8703	0.8055	3.260	3.68	4.25	1.79	-7.54~6.69	
	second derivative	45	0.9044	0.6529	2.820	4.69	4.96	1.40	-7.70~7.41	
	first derivative + Savitzky-Golay	21	0.9442	0.9027	2.180	2.65	3.39	2.48	-6.46~4.49	
	first derivative + Norris	22	0.9473	0.9065	2.120	2.59	3.28	2.54	-2.04~2.40	

PLS Factors and PC_{max} corresponded to the parameters of PLS and PCR models, respectively.

Table 3. Modelling results of the starch and amylose content computed by PLS and pre-treated with first derivative plus Savitzky-Golay at selected wavebands.

	Waveband	PLS Factors	R^2_c	R^2_p	RMSEC	RMSEP	RMSECV	RPD	Prediction error
Starch	2494 - 2500	10	0.9745	0.9031	0.618	1.20	1.25	2.28	-1.60~2.14
	2278 - 2283	10	0.9732	0.9040	0.633	1.20	1.25	2.28	-1.62~2.18
	1923 - 1961	10	0.9747	0.9061	0.616	1.20	1.25	2.28	-1.55~2.15
	1538 - 1587	10	0.9747	0.9034	0.616	1.20	1.25	2.28	-1.61~2.14
Amylose	1724 - 1818	11	0.9862	0.9353	1.100	2.19	3.34	3.00	-4.41~3.50
	1515 - 1587	11	0.9877	0.9340	1.030	2.20	3.32	2.99	-4.61~3.67

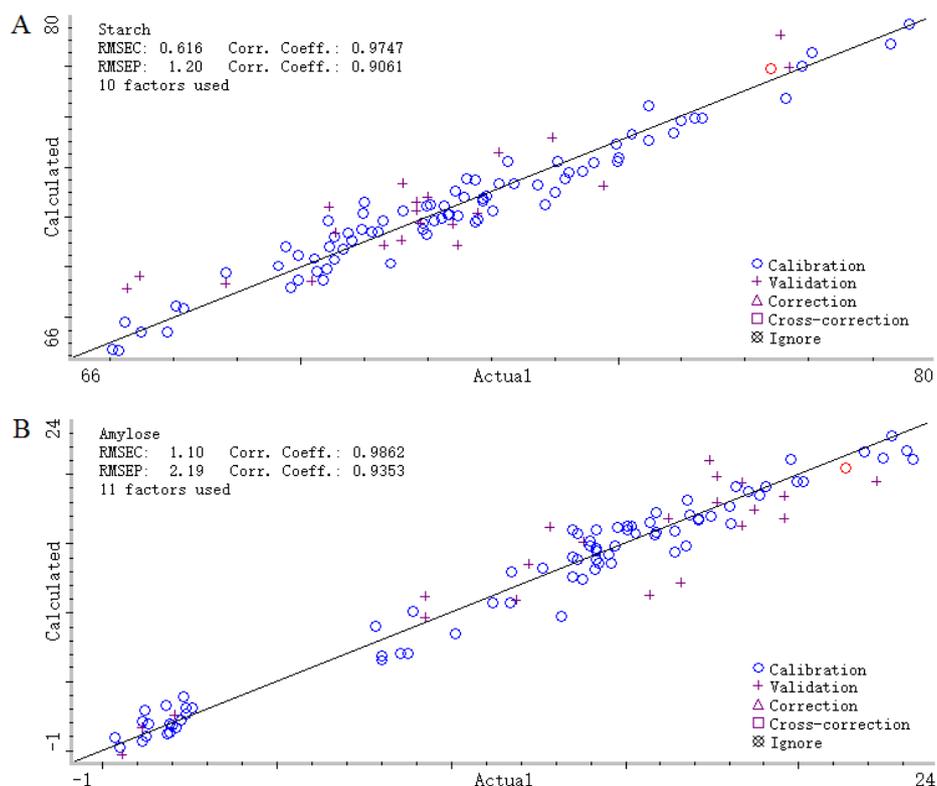


Figure 2. PLS models of starch content at 1923 - 1961 nm (A), and amylose content at 1724 - 1818 nm (B).

the characteristic waveband of amylose at 1724 - 1818 and 1515 - 1587 nm were obtained; both achieved favourable results in the establishment of the model and predictive performance. The results demonstrated that the PLS models on the selected wavebands have significant effect in improving the predictive accuracy of the models. However, this finding is different from the result of a previous study in which the PLS-VIP (the PLS models with VIP scores) models with selected wavebands had little influence on increasing the predictive accuracy of the protein content (Chen *et al.*, 2014).

Conclusions

A rapid analysis method for the quantitative

determination of starch and amylose contents in different kinds of cereals was developed based on NIRS combined with chemometrics and waveband selection. Cereal samples were effectively quantified by the PLS and PCR models, and the PLS model was found to be better than the PCR model at full waveband. The best pre-treatment for the PLS model of starch and amylose content was the first derivative plus Savitzky-Golay. The values of the R^2_c , R^2_p , RMSEC, RMSEP, RMSECV, and RPD for starch and amylose indicated good predictive accuracy and robustness of the models. The best selected waveband for modelling were 1923 - 1961 and 1724 - 1818 nm for starch and amylose, respectively. The results demonstrated that NIRS is an alternative to the chemical method for the analysis of starch and

amylose in different kinds of cereals due to its simplicity, accuracy, and environmental friendliness.

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References

- Aenugu, H. P. R., Kumar, D. S., Parthiban, S. N., Ghosh, S. S. and Banji, D. 2011. Near infra red spectroscopy - an overview. *International Journal of Chemtech Research* 3: 825-836.
- Association of Official Analytical Chemists (AOAC). 1997. Official methods of analysis of Association of Official Analytical Chemists. 16th ed. United States: AOAC.
- Azudin, M. N. and Morrison, W. R. 1986. Non-starch lipids and starch lipids in milled rice. *Journal of Cereal Science* 4: 23-31.
- Bagchi, T. B., Sharma, S. and Chattopadhyay, K. 2016. Development of NIRS models to predict protein and amylose content of brown rice and proximate compositions of rice bran. *Food Chemistry* 191: 21-27.
- Bázár, G., Romvári, R., Szabó, A., Somogyi, T., Éles, V. and Tsenkova, R. 2016. NIR detection of honey adulteration reveals differences in water spectral pattern. *Food Chemistry* 194: 873-880.
- Buckow, R., Heinz, V. and Knorr, D. 2007. High pressure phase transition kinetics of maize starch. *Journal of Food Engineering* 81: 469-475.
- Chen, H. Z., Song, Q. Q., Tang, G. Q. and Xu, L. L. 2014. An optimization strategy for waveband selection in FT-NIR quantitative analysis of corn protein. *Journal of Cereal Science* 60: 595-601.
- Chen, J., Zhu, S. P. and Zhao, G. H. 2017. Rapid determination of total protein and wet gluten in commercial wheat flour using siSVR-NIR. *Food Chemistry* 221: 1939-1946.
- Fertig, C. C., Podczek, F., Jee, R. D. and Smith, M. R. 2004. Feasibility study for the rapid determination of the amylose content in starch by near-infrared spectroscopy. *European Journal of Pharmaceutical Sciences* 21: 155-159.
- Gaffa, T., Yoshimoto, Y., Hanashiro, I., Honda, O., Kawasaki, S. and Takeda, Y. 2004. Physicochemical properties and molecular structures of starches from millet (*Pennisetum typhoides*) and sorghum (*Sorghum bicolor* L. Moench) cultivars in Nigeria. *Cereal Chemistry* 81: 255-260.
- Ge, F., Chen, C., Liu, D. and Zhao, S. 2014. Rapid quantitative determination of walnut oil adulteration with sunflower oil using fluorescence spectroscopy. *Food Analytical Methods* 7: 146-150.
- Hu, J. Q., Ma, X. C., Liu, L. L., Wu, Y. W. and Ouyang, J. 2017. Rapid evaluation of the quality of chestnuts using near-infrared reflectance spectroscopy. *Food Chemistry* 231: 141-147.
- Hu, J. Q., Wang, Z., Wu, Y. W., Liu, Y. G. and Ouyang, J. 2018. Rapid determination of the texture properties of cooked cereals using near-infrared reflectance spectroscopy. *Infrared Physics and Technology* 94: 165-172.
- Liu, X., Zhang, X., Rong, Y. Z., Wu, J. H., Yang, Y. J. and Wang, Z. W. 2015. Rapid determination of fat, protein and amino acid content in *Coix* seed using near-infrared spectroscopy technique. *Food Analytical Methods* 8: 334-342.
- Lohumi, S., Lee, S., Lee, W. H., Kim, M. S., Mo, C., Bae, H. and Cho, B. K. 2014. Detection of starch adulteration in onion powder by FT-NIR and FT-IR spectroscopy. *Journal of Agricultural and Food Chemistry* 62: 9246-9251.
- Longe, O. G., Fetuga, B. L. and Aken'Ova, M. E. 1982. Carbohydrate constituents of some Nigerian maize varieties grown at three soil nitrogen levels. *Food Chemistry* 8: 179-186.
- Mahesh, S., Jayas, D. S., Paliwal, J. and White, N. D. G. 2015. Comparison of partial least squares regression (PLSR) and principal components regression (PCR) methods for protein and hardness predictions using the near-infrared (NIR) hyperspectral images of bulk samples of Canadian wheat. *Food and Bioprocess Technology* 8: 31-40.
- Niu, X. Y., Zhao, Z. L., Jia, K. J. and Li, X. T. 2012. A feasibility study on quantitative analysis of glucose and fructose in lotus root powder by FT-NIR spectroscopy and chemometrics. *Food Chemistry* 133: 592-597.
- Noah, L., Robert, P., Millar, S. and Champ, M. 1997. Near-infrared spectroscopy as applied to starch analysis of digestive contents. *Journal of Agricultural and Food Chemistry* 45: 2593-2597.
- Pachau, L., Dutta, R. S., Roy, P. K., Kalita, P. and Lalhlenmawia, H. 2017. Physicochemical and disintegrant properties of glutinous rice starch of Mizoram, India. *International Journal of Biological Macromolecules* 95: 1298-1304.
- Ping, H., Wang, J. H. and Ren, G. X. 2013. Prediction of the total starch and amylose content

- in barley using near-infrared reflectance spectroscopy. *Intelligent Automation and Soft Computing* 19: 231-237.
- Rahman, S., Li, Z., Batey, I., Cochrane, M. P., Appels, R. and Morell, M. 2000. Genetic alteration of starch functionality in wheat. *Journal of Cereal Science* 31: 91-110.
- Ren, G. X. and Chen, F. 1999. Simultaneous quantification of ginsenosides in American ginseng (*Panax quinquefolium*) root powder by visible/near-infrared reflectance spectroscopy. *Journal of Agricultural and Food Chemistry* 47: 2771-2775.
- Salgó, A. and Gergely, S. 2012. Analysis of wheat grain development using NIR spectroscopy. *Journal of Cereal Science* 56: 31-38.
- Savitzky, A. and Golay, M. J. E. 1964. Smoothing and differentiation of data by simplified least squares procedures. *Analytical Chemistry* 36: 1627-1639.
- Shao, Y. N., Cen, Y. L., He, Y. and Liu, F. 2011. Infrared spectroscopy and chemometrics for the starch and protein prediction in irradiated rice. *Food Chemistry* 126: 1856-1861.
- Siriphollakul, P., Nakano, K., Kanlayanarat, S., Ohashi, S., Sakai, R., Rittiron, R. and Maniwaru, P. 2017. Eating quality evaluation of Khao Dawk Mali 105 rice using near-infrared spectroscopy. *LWT - Food Science and Technology* 79: 70-77.
- Souza, D., Sbardelotto, A. F., Ziegler, D. R., Marczak, L. D. F. and Tessaro, I. C. 2016. Characterization of rice starch and protein obtained by a fast alkaline extraction method. *Food Chemistry* 191: 36-44.
- Tian, B., Deng, Z. Y., Xie, Q. G. and Tian, J. C. 2015. Genetic dissection of the developmental behaviour of total starch content and its components in wheat grain. *Crop and Pasture Science* 66: 445-455.
- Wang, S. J. and Copeland, L. 2015. Effect of acid hydrolysis on starch structure and functionality: a review. *Critical Reviews in Food Science and Nutrition* 55: 1081-1097.
- Wang, S. J., Li, P. Y., Yu, J. L., Guo, P. and Wang, S. 2017. Multi-scale structures and functional properties of starches from *Indica* hybrid, *Japonica* and waxy rice. *International Journal of Biological Macromolecules* 102: 136-143.
- Xie, L. H., Tang, S. Q., Chen, N., Luo, J., Jiao, G. A., Shao, G. N., ... and Hu, P. S. 2014. Optimisation of near-infrared reflectance model in measuring protein and amylose content of rice flour. *Food Chemistry* 142: 92-100.
- Xie, L. J., Ye, X. Q., Liu, D. H. and Ying, Y. B. 2009. Quantification of glucose, fructose and sucrose in bayberry juice by NIR and PLS. *Food Chemistry* 114: 1135-1140.
- Yang, Y. X., Wang, G. Y. and Pan, X. C. 2009. China food composition. 2nd ed. Beijing: Peking University Medical Press.
- Yu, S. F., Ma, Y. and Sun, D. W. 2009. Impact of amylose content on starch retrogradation and texture of cooked milled rice during storage. *Journal of Cereal Science* 50: 139-144.
- Zhang, P. Y. and Hamaker, B. R. 2012. Banana starch structure and digestibility. *Carbohydrate Polymers* 87: 1552-1558.