Effect of particle size on near infrared amylose determination of rice flour

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Introduction

The apparent amylose content of rice flour is an important factor influencing the processing characteristics as a food material and the quality of these processed foods. Consequently, the accurate and rapid analysis of the apparent amylose content within rice flour is important.

A number of studies have reported the use of a near infrared (NIR) spectroscopic technique as an alternative rapid and cost-effective analytical method to determine the apparent amylose content of milled rice.[1,2] However, only a few research efforts have applied the NIR technique to rice flour.[3] In addition, there have been no reports examining the influence of the rice flour particle size to the NIR data.

The objective of this study was to investigate the effect of rice flour particle sizes on the determination of amylose content using an NIR method and to characterise the combination of the NIR technique with a particle size compensation method as a routine operation for determining the apparent amylose content of rice flour.

Materials and methods

Rice flour sample preparation

Eight kinds of brown rice with different protein compositions (Shunyo, LGC soft, Akitakomachi, Menkoina, Hitomebore, Akita 39, Tatsukomochi, and Kinunohada) were purchased from the Ogata Village Country Elevator Public Corporation, Akita prefecture, Japan. Whole-grain rice

Table 1. Apparent amylose content characteristics of the calibration and validation sets used to examine the effects of rice flour particle sizes on the determination of amylose content by NIR analysis.

		N	Mean (%)	Range (%)	*SD* (%)
Original	Cal	24	19.01	3.24–30.08	8.97
	Val	24	18.77	3.31–30.30	8.91
45–63 µm	Cal	9	20.25	3.64–29.34	9.60
	Val	9	19.6	3.91–28.46	9.26
63–75 µm	Cal	16	19.11	3.51–29.49	9.36
	Val	16	18.71	3.77–28.24	9.17
75–106 µm	Cal	22	20.22	3.46–30.82	8.27
	Val	22	19.83	3.56–28.90	8.19
106–150 µm	Cal	24	19.09	3.48–30.52	9.03
	Val	24	18.73	3.54–28.97	8.89
150–250 µm	Cal	24	18.85	3.51–30.15	8.82
	Val	24	18.54	3.91–30.08	8.72

N is the number of samples; Cal. is the calibration set; Val. is the validation set; *SD* is the standard deviation.

milled to 90% and 70% was obtained using a laboratory mill (Chioda, HS-4). The whole-grain milled rice and the brown rice samples were then ground by the Awaji Flour-Milling Company. The obtained rice flour was separated by particle size as shown in Table 1. By this method 238 rice flour samples with different particle sizes were obtained.

Spectral acquisition

Spectroscopic acquisition was performed using an NIR scanning monochromator (Model 6500; Foss NIRSystems Inc., Silver Spring, MD) in the reflectance mode. All operations were performed at room temperature (25°C).

Chemical analysis

The apparent amylose content of the rice flour samples was determined using the iodine colorimetry assay of Juliano.[4] Total protein content ($N \times 5.95$) was determined using the Kjeldahl method with an analyzer unit (ACTAC, 2300).

Data analysis

A partial least squares (PLS) calibration equation was developed using the Unscrambler software (Camo, Oslo, Norway). Rice flour samples of different particle sizes were manually separated into calibration and validation sets by an odd-even method. Statistical characteristics of the samples used are provided in Table 1.

Results and discussion

The effect of particle sizes on the determination of the apparent amylose content by NIR

An apparent amylose content calibration model was developed using the spectra of the original rice flour samples. The equation with the calibration model statistics of R: 0.98, *SEC*: 1.61%, *SEP*: 2.05%, bias: –0.25% was obtained by the use of six factors constructed from the raw spectra as shown in Table 2.

From Table 2, the equation was sufficiently accurate for use with the original rice flour samples that had similar particle sizes but showed significant bias when the equation was applied to rice flour samples with different particle size. A multiplicative scatter correction (MSC) approach was used to reduce the effect of scattered light on the diffuse reflection and transmission NIR spectra. This method was found to be useful as an approach to remove varying background spectra with no scattering origins. In a similar fashion to raw spectra, an apparent amylose content calibration model was developed using the MSC spectra of the original rice flour samples. As shown in Table 2, the equation with the calibration statistics of *R*: 0.99, *SEC*: 1.29%, *SEP*: 1.79%, bias: 0.26% was obtained using the six factors constructed from the MSC spectra. The equation appeared to be more accurate for rice flours with similar particle sizes. However, the MSC approach did not remove the significant bias when applied to rice flour samples with different particle sizes.

An apparent amylose content calibration model with particle size compensation

To overcome the error due to particle sizes, an apparent amylose content calibration model with particle size compensation was developed using a self-corrective compensation method. This method follows the same approach as the temperature compensation method.[5,6] The particle sizes of the rice flour samples are therefore handled as an ingredient by the partial least squares (PLS) method. In this case, rice flour samples with various particle sizes must be used for the amylose content calibration model. Therefore, by following the temperature-compensation method, a particle-size-compensated calibration equation for determining the apparent amylose content was established based on the spectra of the rice flour samples with various particle sizes. As shown in Table 3, the equation with the calibration statistics of *R*: 0.98, *SEC*: 1.93%, *SEP*: 2.03%, bias: 0.06% based on raw spectra could be obtained using the six factors constructed from the raw spectra.

The equation is sufficiently accurate for the prediction of the validation sample set, which contains different particle sizes, and showed no apparent biases when applied to rice flour samples with different particle sizes. In addition, when the MSC spectra were used, a more accurate calibration model for the determination of the apparent amylose content with particle size compensation was obtained and is presented in Table 3. From these results, the biases produced by particle sizes could be successfully removed by the self-corrective compensation method.

To understand why the influence due to particle sizes could not be removed by MSC processing of the spectra, the regression coefficients of the PLS amylose content calibration models based on the MSC spectra without particle size compensation and with particle size compensation were investigated. Comparison of the regression coefficient plots of the equation without particle size compensation and one with particle size compensation reveals significant peaks at 1530 nm and

Table 2. Calibration and prediction of the amylose content using the NIR model developed without particle compensation based on raw and MSC spectra.

	Calibration		Validation		Prediction									
					45–63 µm		63–75 µm		75–106 µm		106–150 µm		150–250 µm	
	R	*SEC*	*SEP*	Bias	*SEP*	Bias	*SEP*	Bias	*SEP*	Bias	*SEP*	Bias	*SEP*	Bias
Raw spectra	0.98	1.61	2.05	–0.25	2.21	6.63	2.46	4.05	2.41	–1.47	5.65	–3.01	4.44	4.93
MSC spectra	0.99	1.29	1.79	–0.26	2.07	8.45	2.26	4.35	3.77	0.88	2.22	–0.35	2.52	1.85

R, correlation coefficient of calibration; *SEC*, standard error of calibration; *SEP*, bias-corrected standard error of prediction; Bias, systematic error.

Table 3. Calibration and prediction of the amylose content using the NIR model developed with particle compensation based on raw and MSC spectra.

	Calibration		Validation		Prediction									
					45–63 µm		63–75 µm		75–106 µm		106–150 µm		150–250 µm	
	R	*SEC*	*SEP*	Bias	*SEP*	Bias	*SEP*	Bias	*SEP*	Bias	*SEP*	Bias	*SEP*	Bias
Raw spectra	0.98	1.93	2.03	0.06	2.21	0.76	2.14	0.05	1.70	–0.29	2.36	–0.25	1.82	0.23
MSC spectra	0.98	1.54	1.53	0.25	1.75	–0.17	1.66	0.30	1.22	0.02	1.73	0.29	1.22	0.44

R, correlation coefficient of calibration; *SEC*, standard error of calibration; *SEP*, bias-corrected standard error of prediction; Bias, systematic error.

2000 nm only in the particle-size-compensated equation. The wavelength range around the peaks at 1530 nm and 2000 nm were correlated to the absorption band of the N–H stretching first overtone which is related to the protein content in the flour.[7] This is in agreement with the observed result that a larger particle size was associated with higher protein content. Since protein contents vary due to particle sizes of the rice flour, absorption of the light in the protein absorption band is different. Consequently, the influence of particle sizes on the NIR spectra could not be removed by MSC processing.

Conclusion

Closer inspection showed that the influence of the particle size on the NIR spectra could not be sufficiently reduced by the MSC processing approach because the compositions of the rice flour were dependent on particle size. To address this issue, the influence of the particle size on the NIR spectra was reduced by a self-corrective compensation method. In other words, the influence of particle size was automatically managed as an ingredient of the rice flour samples by a partial least squares (PLS) method.

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