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Prediction of Pasting and Thermal Properties of Mixed Hom-Mali and Glutinous Rice Flours Using Near Infrared Spectroscopy

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ABSTRACT

Starch is a major component of rice grain and plays an important role in the grain quality. The pasting properties and thermal properties are important physical qualities of rice flour. Direct measurement is time-consuming and expensive. Therefore, a rapid predictive method based on near-infrared (NIR) spectroscopy is necessary that can be applied to measure these quality parameters. Glutinous flour (RD6, Sakonnakorn and Niew Ubon varieties) and Hom-Mali rice flour (105 variety) were selected for this research. The flour samples were dry milled into a 120-mesh sieve using a turbo mill. In total, 92 mixed flour samples were prepared by blending Hom-Mali rice flour with each of the glutinous rice flour types in various ratios of which 61 mixed flour samples were used to develop calibration models for measuring pasting and thermal properties based on the NIR spectra of the mixed flour samples. The models were validated by a separate prediction set consisting of 31 mixed flour samples.

The models for determining the pasting properties using the pasting temperature, set back, final viscosity, breakdown and trough yield all had a similar level of accuracy with R^2 values of 0.99, 0.97, 0.95, 0.80 and 0.80, respectively. Furthermore, the thermal properties indicated that the models for onset temperature, peak temperature, and enthalpy produced moderate correlation values with R^2 values of 0.82, 0.75 and 0.73, respectively, whereas the conclusion temperature could not be predicted by NIR spectroscopy (R^2 = 0.06).

Keywords: Hom-Mali rice, glutinous rice, near infrared spectroscopy, pasting properties, thermal properties

INTRODUCTION

Rice is one of the most important staple foods in the world, especially in Asian countries (Wu and Shi, 2007). Glutinous rice (*Oryza sativa* L.) is one of the important economic crops of Thailand and is a staple food for Thai people in northern and northeastern Thailand. Khao Dok Mali 105 or Hom-Mali rice varieties are very popular in Southeast Asia and are widely accepted in the world due to their quality, good taste, soft texture and unique aroma. Starch from rice grains with varying amylose content is of interest for food processing because of the potential to modify the texture and quality of the finished product. Glutinous rice contains very limited amounts of

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amylose (0–4%) whereas Hom-Mali rice has an amylose content of 14–18%. Differences in the starch properties of flours contribute to their suitability for different food products and industrial use (Bao *et al*., 2007a).

Near infrared reflectance (NIR) spectroscopy is a non-destructive and chemical-free method which is easily used in continuous food quality evaluation (Cen and He, 2007). It is less expensive than other methods because it does not require any other input materials (such as chemical reagents) other than electricity. NIR spectroscopy instruments must be calibrated using standard laboratory reference methods. A calibration model is then developed by calculating the regression equation based on the NIR spectra and reference data.

NIR spectroscopy with its advantage of minimal sample preparation is being widely applied to estimate rice quality characteristics (Batten, 1998; Wu and Shi, 2003). It has been used to estimate rice nutrient characteristics, such as the protein content of milled whole-grain samples (Delwiche *et al*., 1996; Barton *et al*., 2002) and the amino acid content of milled rice flour (Barton *et al*., 1998; Wu *et al*., 2002). From previous studies, many researchers have developed NIR spectroscopy models of some cooking-related characteristics, for example, texture, gelatinization temperature, gel consistency and RVA-related characteristics from various samples (Delwiche *et al*., 1996; Windham *et al*., 1997; Bao *et al*., 2001a; Meullenet *et al*., 2007).

Pasting properties and gelatinization are the most important characteristics of starch, determining its application in food processing and other industries (Bao, 2008). Pasting properties have been used to predict the end-use quality of various products, for example cooked rice texture (Shu *et al*., 1998; Bao *et al.,* 2001b) and noodles (Bhattacharya *et al.,* 1999). The gelatinization temperature (GT) is the critical temperature at which starch granules irreversibly lose their birefringence and crystalline order during heating. The gelatinization temperature can be measured by differential scanning calorimetry (DSC; Normand and Marshall, 1989) or indirectly by the alkali spreading value test for rice grains (Little *et al*., 1958). Bao (2008) reported that the pasting temperature from RVA was 1 °C higher than the peak temperature of DSC. From previous studies, researchers have reported that cooked high-GT rice is harder in texture on accelerated staling than cooked low-GT rice (Perez *et al*., 1993; Villareal *et al*., 1993). Different rice products need unique kinds of rice with diverse starch properties; for example, a low GT is preferred in manufacturing breakfast cereals, rice breads and beer (Juliano, 1998).

The amylose content in rice grain is believed to be the most important parameter in determining the eating and cooking quality of cooked rice (Varavinit *et al*., 2003). Patindol and Wang (2002) studied the physicochemical properties of three nonwaxy, long-grain rices and found that different amylopectin structures could affect rice functionality, for example, the gelatinization, retrogradation and pasting behavior. Sodhi and Singh (2003) found that rice with an amylose content of 7.8% had the highest onset temperature (To), peak temperature (Tp), conclusion temperature (Tc) and retrogradation percentage in comparison with four other rice varieties with 15.6–18.9% amylose content. Furthermore, starch retrogradation encompassed the changes that occur in gelatinized starch from an initially amorphous to a more ordered state (Bao *et al*., 2007b). High-GT starch had higher values for enthalpy and percentage of retrogradation than low-GT starch. The popular cultivars of glutinous rice in Thailand are Sanpatong, RD6 and RD10 (Keeratipibul *et al*., 2008). RD6 and RD10 have a softer texture than the other varieties. The higher amylose content in rice grain produces a harder texture and less gloss in the cooked rice. The very low amylose content of each cultivar gave different eating quality characteristics to the processed rice products (Keeratipibul *et al*., 2008) and has been used for food ingredients, such as stabilizing sauces, gravies and puddings, to resist water separation during freeze-thaw cycles (Bean *et al*., 1984; Juliano and Hicks, 1996). Furthermore, in Asian countries, milled glutinous rice and glutinous rice flour are used as raw materials for various processed products such as sweets, desserts, rice cakes, baked rice crackers and puffed rice (Keeratipibul *et al*., 2008).

A few studies have developed an NIR spectroscopy model for the thermal properties. Delwiche *et al*. (1996) developed an NIR spectroscopy model for milled whole grain samples. Bao *et al*. (2001a) developed models for the alkali spreading value and thermal properties including the To, Tp, Tc and enthalpy of gelatinization (ΔH). Lu *et al*. (2006) established NIR spectroscopy models for the prediction of the thermal properties of potato starch.

The objective of the present study was to develop calibration equations from spectra collected from mixed flour using partial least-squares regression. The results of this research will expand the pasting and thermal properties ranges for predicting flour properties.

MATERIALS AND METHODS

Material

The commercial broken Hom-Mali rice (105 variety) was obtained from Chia Meng Rice Mill Co., Ltd. The glutinous rice samples (RD6, Sakonnakorn and Niew Ubon varieties) were obtained from the Rice Department (Bangkok, Thailand) from the 2009 harvest.

Preparation of glutinous flour, Hom-Mali rice flour and mixed Hom-Mali rice and glutinous rice flour

Glutinous flour (RD6, Sakonnakorn and Niew Ubon varieties) and Hom-Mali rice flour (105 variety) with a particle size less than 120-mesh were prepared by dry milling using a turbo mill (Sahachon Co., Ltd, Chonburi, Thailand). Flour samples were collected and packed in polyethylene bags and stored at room temperature prior to further use.

Preparation of mixed flour

Ninety two mixed flours were prepared by blending Hom-Mali rice flour with each glutinuous rice flour variety (RD6, Sakonnakorn and Niew Ubon). The sample distribution is shown in Table 1. The samples were divided into two sets—namely, a calibration set (n=61) and a prediction set (n=31). Mixed flours were blended using a mixer (Kluy Nam Tai, Bangkok, Thailand) for 15 min and packed in polyethylene bags and stored at room temperature for use in the study.

Reference analysis

Thermal properties

The thermal properties of the 92 samples of mixed flour were determined using a DSC 822[e]

Table 1 Sample distribution of mixed Hom-Mali and glutinous rice flours.

Mixed flours	Calibration	Prediction
HMRF:RD6 HMRF:Sakonnakorn HMRF:Niew Ubon	95:5, 90:10, 85:15, 80:20, 75:25 70:30, 65:35, 60:40, 55:45 50:50, 45:55, 40:60, 35:65, 30:70, 25:75, 20:80, 15:85, 10:90, 5:95, 0:100	90:10, 80:20, 70:30, 60:40 50:50, 40:60, 30:70, 20:80 10:90, 0:100
HMRF	100:0	100:0

HMRF = Hom-Mali rice flour

thermal analyzer (Mettler Toledo; Columbus, OH). Mixed flour (2.0 mg, dry basis) was weighed into an aluminum pan and 6 μL of distilled water was added. The pan was hermetically sealed, equilibrated at room temperature for 1 h and then heated at a rate of 10 °C/min from 30 °C to 110 °C. A sealed empty pan was used as a reference. All thermal properties were measured in duplicate.

Pasting properties

The pasting properties of samples were measured using the Rapid Visco Analyser (RVA; Newport Scientific Warriewood, Australia) controlled by Thermocline for Windows software version 4 (Newport Scientific, Warriewood, Australia) according to the AACC method (AACC, 2000). Each mixed rice flour sample (3 g, 12% dry basis) was mixed with 25 g of distilled water in an RVA canister. The ideal temperature was set and the following 12.5-minute test profile was run: (1) held at 50 °C for 1.0 min, (2) linearly ramped up to 95 °C for 3.8 min, (3) held at 95 °C for 2.5 min, (4) linearly ramped down to 50 °C for 3.8 min and (5) held at 50 °C for 1.4 min. The peak viscosity, trough, breakdown, final viscosity, setback from peak, peak time and pasting temperature were determined by analysis of the software output.

Spectroscopic analysis

Ninety two mixed flour samples were divided into two sets—namely, a calibration set of 61 samples and a prediction set of 31 samples. NIR reflectance spectra were obtained and converted to absorbance spectra from 1100 to 2500 nm at 2 nm intervals using an InfraAlyzer 500 spectrometer (Bran+Luebbe, Norderstedt, Germany). Two replicates of each sample were scanned using a standard cup (internal diameter 35mm, depth 8 mm). All spectral data were transferred into JCAMP.DX format and imported into the Unscrambler 9.8 software (Camo, Oslo, Norway) for data analysis. The original NIR spectra for the whole region were employed to build the models. Partial least squares regression (PLSR) was used to develop the regression equations.

RESULTS AND DISCUSSION

Reference data

The mean, range and standard deviation for the pasting properties and thermal properties of the mixed flour calibration and prediction sample sets are shown in Tables 2 and 3, respectively. The pasting properties showed high variations in these statistics while on the other hand, the thermal properties yielded few differences. The results indicated that both subsets could present the complete variation found in all the rice samples. The mixed rice flours were scanned to obtain their NIR spectra. The entire spectral region of 1100–2500 nm was applied to develop the calibration equations. The NIR spectra of mixed flours are shown in Figure 1.

Calibration validation and prediction results of PLSR models for pasting and thermal properties of the mixed flours

The statistical results for the calibration, validation and prediction of the PLSR models for the pasting and thermal properties of the mixed flour samples are summarized in Table 4. The calibration results obtained from the PLSR models were built using data from the calibration sample set; these PLSR models were validated to obtain the factor number by using the same data set (the calibration sample set). All PLSR models were tested by a separate prediction sample set to evaluate the predictive performance of the model, producing the prediction results.

The coefficient of determination is given by R^2 and indicates the closeness of fit between the NIR spectroscopy and reference data. From the calibration results, the model performances based on the original data for pasting properties

Table 2 Minimum, maximum, mean and standard deviation values of references for pasting properties of flour.

Pasting properties	Minimum	Maximum	Mean	Standard deviation
Peak (RVU)				
Calibration	210.80	311.24	252.41	23.83
Prediction	217.69	300.44	255.51	23.12
Trough (RVU)				
Calibration	131.08	185.82	155.64	13.93
Prediction	132.63	179.40	159.55	14.41
Breakdown (RVU)				
Calibration	59.72	173.28	96.77	28.02
Prediction	63.07	161.55	95.96	27.13
Final viscosity (RVU)				
Calibration	162.15	298.91	229.63	39.78
Prediction	162.15	290.60	233.90	41.91
Set back from peak (RVU)				
Calibration	-134.06	23.03	-22.22	44.97
Prediction	-134.06	21.04	-21.61	48.18
Peak time (min)				
Calibration	3.80	6.27	5.10	0.96
Prediction	3.80	6.30	5.11	0.99
Pasting temperature (°C)				
Calibration	67.98	75.63	70.79	1.97
Prediction	69.55	79.08	71.55	1.92
Moisture content (%)				
Calibration	6.87	9.74	8.61	0.72
Prediction	6.87	9.61	8.49	0.74

RVU = Rapid Visco Unit.

to determine the final viscosity, setback from peak and pasting temperature were excellent ($R^2 > 0.9$). The pasting properties of peak, trough, breakdown, peak time and thermal properties as indicated by the values for To, Tp and enthalpy produced a moderate correlation (R^2 approximately equal to 0.7–0.8) whereas Tc could not be predicted by NIR spectroscopy ($R^2 = 0.06$). The standard error of prediction (SEP) was computed from the results of the prediction sample set that had not been used in the development of the calibration. The SEP of the pasting properties ranged from 0.51 to 22.23. The SEP of the thermal properties ranged from 0.58 to 0.75.

NIR spectroscopy is a non-destructive and chemical free method which can be easily used for continuous food quality evaluation (Cen and He, 2007). The Rapid Visco Analyser (RVA) utilizes a rapid, simple rheological method for the measurement of cereal quality. The observations from this study indicated that the peak viscosity and breakdown of flour tended to decrease when the content of Hom-Mali rice increased, whereas the pasting temperature, final viscosity and set back values were substantially increased. The R^2 value from the calibration of the RVA parameters of peak, trough, breakdown, final viscosity, set back, peak time and pasting temperature were 0.74,

Table 3 Minimum, maximum, mean and standard deviation values of references for thermal properties of flour.

Thermal properties	Minimum	Maximum	Mean	Standard deviation
To (°C)				
Calibration	59.95	62.77	61.33	0.79
Prediction	60.04	62.6	62.00	0.66
Tp (°C)				
Calibration	67.04	69.57	68.24	0.61
Prediction	68.01	69.71	68.74	0.53
Tc (°C)				
Calibration	73.98	76.96	75.16	0.65
Prediction	73.83	76.91	75.10	0.74
ΔH (J/g)				
Calibration	3.46	13.81	9.15	1.94
Prediction	5.62	12.44	8.54	1.62

To = onset temperature; Tp = peak temperature; Tc = conclusion temperature; DH = enthalpy of gelatinization.

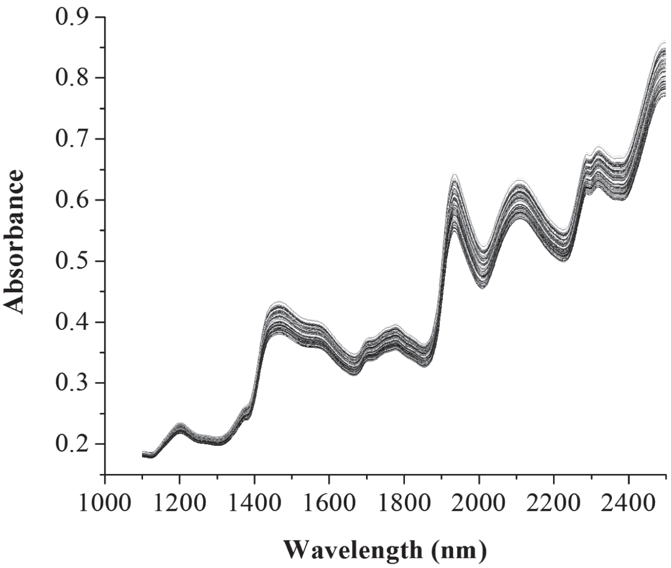

Figure 1 Original NIR spectra of mixed flour samples.

0.80, 0.80, 0.95, 0.97, 0.79 and 0.99, respectively. These high values indicated that accurate calibration equations had been developed for the mixed flour spectra.

Many researchers have examined whether the functional properties of cereals measured by RVA could be predicted by NIR methods. Juhász *et al*. (2005) reported a good relationship between NIR spectra and RVA data in germinated wheat. On the other hand, Delwiched *et al*. (1996) and Meadows and Barton (2002) reported that NIR spectra did not correlate with the traditional RVA parameters in rice.

The temperatures of the rice starch at which gelatinization begins and finishes are very important because these are related to the cooking

Table 4 Result of NIR calibration and validation sets for mixed flours.

Original	Parameter	Calibration			Validation		Prediction	
		Factor	R^2	SEC	SEV	Bias	SEP	Bias
Pasting properties	Peak (RVU)	8	0.74	11.94	21.04	0.87	20.99	-47.30
	Trough (RVU)	3	0.80	6.15	6.64	0.06	7.37	-5.66
	Breakdown (RVU)	8	0.80	12.43	21.51	0.59	21.47	-23.29
	Final viscosity (RVU)	3	0.95	9.06	9.86	0.09	13.20	-10.86
	Setback (RVU)	10	0.97	7.86	21.65	0.31	22.23	35.87
	Peak time (mins)	2	0.79	0.00	0.46	0.01	0.51	-0.18
	Pasting temp (°C)	16	0.99	0.05	1.27	0.03	1.97	4.90
Thermal properties	To (°C)	7	0.82	0.34	0.45	0.00	0.58	-0.01
	Tp (°C)	2	0.75	0.33	0.35	-0.00	0.74	-0.15
	Tc (°C)	1	0.06	1.03	1.06	0.00	0.75	0.27
	Enthalpy (J/g)	1	0.73	0.00	0.79	-0.00	0.67	-0.40

RVU = Rapid Visco Unit.
To = onset temperature; Tp = peak temperature; Tc = conclusion temperature.
R^2 = coefficient of determination.
SEC = Standard error of calibration.
SEV = Standard error of validation.
SEP = Standard error of prediction.

time. The gelatinization temperature is one of the important indicators of the cooking quality of cooked rice (Juliano, 1998). The heat energy required to completely gelatinize starch in rice or flour is critical to the food processor who must optimize the heat input, cooking time and temperature (Bao *et al.*, 2007a). Thermal properties are generally measured using DSC, which can be run at a rate of four samples per hour. Consequently, suitable calibration models by NIR spectroscopy can definitely reduce time and cost.

NIR spectroscopy instruments must be calibrated using standard laboratory reference methods. Accurate calibration equations have been developed for To, Tp and Tc with flour spectra in a previous study (Bao *et al.*, 2001a). However, the present study indicated that two parameters (To and Tp) could also be predicted with reasonable reliability. Bao *et al.* (2007a) used rice grain and rice flour to carry out NIR spectroscopy calibration for thermal properties. The results from their study found that calibration accuracy for To was higher with flour spectra than grain spectra. Furthermore, NIR spectroscopy gave a similar level of accuracy in determining the Tp and Tc with either grain or flour spectra. Enthalpy (ΔH) could only be measured with partial accuracy with the flour spectra. The present study found that calibration for thermal properties using To was excellent, while Tp and enthalpy provided a moderate correlation where Tc could not be predicted by NIR spectroscopy. The results were different because many factors affect the precision and reliability of calibration and prediction, such as sample preparation, genetic variability and the accuracy of the reference data.

CONCLUSION

Thermal properties (gelatinization) and pasting properties are the most important physicochemical properties of rice flour. The results from the present study showed that the NIR spectra of mixed flour samples could be successfully modeled for measuring the thermal properties and pasting properties. The NIR spectra of mixed flour samples were only usable for the calibration of To and Tp, whereas the conclusion temperature (Tc) could not be predicted by NIR spectroscopy. The pasting properties could be reliably modeled by spectra of mixed flour samples.

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