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Evaluation of the moisture content of tapioca starch using near-infrared spectroscopy

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The purpose of this study was to develop a calibration model to evaluate the moisture content of tapioca starch using the near-infrared (NIR) spectral data in conjunction with partial least square (PLS) regression. The prediction ability was assessed using a separate prediction data set. Three groups of tapioca starch samples were used in this study: tapioca starch cake, dried tapioca starch and combined tapioca starch. The optimum model obtained from the baseline-offset spectra of dried tapioca starch samples at the outlet of the factory drying process provided a coefficient of determination (R^2), standard error of prediction (SEP), bias and residual prediction deviation (RPD) of 0.974, 0.16%, −0.092% and 7.4, respectively. The NIR spectroscopy protocol developed in this study could be a rapid method for evaluation of the moisture content of the tapioca starch in factory laboratories. It indicated the possibility of real-time online monitoring and control of the tapioca starch cake feeder in the drying process. In addition, it was determined that there was a stronger influence of the NIR absorption of both water and starch on the prediction of moisture content of the model.

Keywords: Moisture content; tapioca starch; near-infrared spectroscopy.

1. Introduction

The tapioca starch industry is an economically important industry in Thailand. Thailand has been the top country in tapioca product export. Moreover, in 2011, 33.91% of the export value of the total tapioca products of Thailand was from tapioca starch.[1] The total tapioca starch export from Thailand in 2013 was 2,445,612.18 tons, corresponding to 1,162,652,368.50 US$ in value.[2]

The Thailand Tapioca Starch Newsletter[3] described the principle of pneumatic drying for the tapioca starch drying process, wherein the water in the tapioca starch cake was evaporated using hot

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air and the dried tapioca starch was conveyed to a cyclone separator, where the dried tapioca starch and saturated air were separated. At present, most tapioca starch factories use the outlet air temperature in the drying process, measured by a thermocouple, as a parameter for controlling the starch cake feed rate. When the outlet air temperature is high, indicating that the cake feed rate is too low, the controller will increase the speed of the feeder. The set point is 60°C. If the outlet temperature reaches 60°C, the controller will stop the feeder. However, the moisture content of dried tapioca starch is checked regularly during the drying process using an infrared moisture analyzer, which requires approximately 10 min per sample. When the moisture content is found to exceed the acceptable standard value (13%), the dried starch lot must be returned to the process. Thus, the starch production time is approximately 30 min (20 min of drying tube retention and 10 min of moisture content measurement). For the production capacity factory of 200 tons per 12 h, 8 tons of unacceptable dried starch must be returned to the process to be dried again. This is very expensive, with costs including the drying energy, product unpacking and repacking, material handling, and labor costs. This does not include the time required to readjust the system to the proper conditions, which must be performed by experienced workers. Based on discussions with the factory production engineers, the use of the moisture content of the dried starch as a direct control parameter of the starch cake feeder would solve this problem.

Near-infrared (NIR) spectroscopy is a rapid method for chemical component analysis. Its advantages include its high precision and accuracy, non-destructive measurements, chemical-free procedure and environmental-friendliness as well as no or minimal sample preparation. However, this technique needs a calibration model to be developed for prediction, which is calibrated with a standard reference method. If the validation of its accuracy is proved, analysis takes only 2–3 s.

Some studies use NIR spectroscopy to measure the moisture content of powders of agricultural products. Vesela *et al.*[4] reported a calibration model developed from the NIR absorbance spectra (1100–2500 nm) of cocoa powder samples, which was used for the determination of the moisture content. The results showed that the relative root mean square error of cross-validation (RMSECV) was 5.2% and the determination correlation (R^2) was 0.94. Camps *et al.*[5] developed a NIR spectroscopy method to determine the moisture in flour of dry *Artemisia annua* leaves, and the model accurately predicted moisture with R^2, RMSECV and root mean square error of prediction (RMSEP) of 0.99, 0.8% and 1.4%, respectively. Gillon *et al.*[6] found close relationships between the foliage moisture content and ground foliage absorbance in the 400–2500 nm spectral range ($R^2 = 0.93$–0.99, standard error of cross-validation (SECV) = 2–7%). Ren and Chen[7] used NIR reflectance spectroscopy to measure the moisture content in hot-air-dried Asian ginseng (*Panax ginseng*) roots, freeze-dried Asian ginseng roots, red Asian ginseng roots and hot-air-dried American ginseng (*Panax quinquefolium*) roots. The calibration equation showed a high correlation between the NIR and reference analytical methods ($R^2 = 0.998$), with a standard error of prediction (SEP) of 0.14% and a bias of only 0.12%. The NIR spectroscopy in diffuse reflectance mode has been used for determination of starch content, total sugar, sucrose, cellulose, total nitrogen and ash of tuber crop flour, e.g., cassava,[8] taro,[8,9] yam[8,10] and sweet potato.[8,11] However, there are few papers that are using NIR spectroscopy for determination of moisture content or dry matter of flour or starch of tuber crop. Fernandez *et al.*[12] used the Fourier transformation near-infrared (FT-NIR) spectrometer in reflectance mode with 4000–10,000 cm^{-1} wavenumber region for determination of dry matter content of mashed potatoes. The calibration model showed the R^2 of 0.92 and SECV of 4.5. Hartmann and Büning-Pfau[13] reported the NIR model developed from the NIR absorbance spectra (1100–2500 nm) to measure the dry matter content of mashed potatoes. The model provided the R^2 and SEP of 0.97 and 0.19%, respectively. Based on these previous studies, NIR spectroscopy is promising for use in tapioca starch factories for the evaluation of the moisture content of the starch. In factories, the inconsistent moisture contents of starch cake and dried starch affect the process control to ensure acceptable products. Measurement using NIR spectroscopy may facilitate rapid measurement of the starch moisture content and response to the process control. It is worth researching the performance of NIR spectroscopic models using samples obtained from the real conditions of the factory production process. Therefore, this research aimed to evaluate the moisture content of both tapioca starch

cake and dried starch in the drying process of a tapioca starch factory using NIR spectroscopy. This information is useful for quality assurance and process control.

2. Materials and Methods

2.1. *Samples*

The tapioca starch samples were collected at the Sangpetch Tapioca Flour Co., Ltd., factory in Nongbua-rawe district at Chaiyaphum, Thailand, in two forms: tapioca starch cake at the inlet of the drying process and dried tapioca starch at the outlet. The 210 samples (105 for each form) were kept in plastic cups with covers and immediately brought to the factory laboratory, where the room temperature was $25 \pm 1^{\circ}$C. The samples were collected over two periods: 64 samples on 24–26 August 2013 and 146 samples on 19–23 December 2013.

2.2. *Near-infrared spectroscopy experiment*

The spectra of tapioca starch samples were measured with a diode-array NIR spectrometer (DA7200, Perten, Sweden) in reflection mode at 950–1650 nm with a resolution of 2 nm. The NIR exposure time was 0.164 s per scan. The sample was presented to the spectrometer in a 75-mm-diameter sample dish (72.05.04, Perten, Sweden). The NIR illumination was over the dish area. The scanning was done while the sample dish was rotated consecutively for 3 rounds and a reference material (polystyrene) was automatically scanned before each sample scan. Therefore, there were 3 spectra for each sample and they were averaged, 210 spectra in total.

2.3. *Moisture measurement*

After scanning, the moisture content of a 5 g tapioca starch sample was immediately analyzed by the factory reference method using an infrared moisture analyzer (HB43-S Halogen, Mettler Toledo, Switzerland) at 130°C. Duplicate tests were run for each sample. Therefore, the repeatability, which was the standard deviation of the difference between duplicates, was calculated to indicate the precision of the test.

2.4. *Spectrum pre-treatment and NIR spectroscopy model establishment*

The NIR spectroscopic models for predicting the moisture content of tapioca starch were established using partial least squares (PLS) regression using the Unscrambler X 10.3 (Camo, Norway). Three groups of sample spectra were used for model development: those of tapioca starch cake samples, dried tapioca starch samples and combined samples (cake and dried samples). The data set of the first two groups was divided into a calibration data set (two-thirds of the data set, 70 samples) and a prediction data set (one-third of the data set, 35 samples) after the data were arranged in ascending order. Similarly, the data for the third group were 140 samples for the calibration set and 70 samples for the prediction set. This was done by assigning the first two samples to be in the calibration set and the third one to be in prediction set until all samples had been allocated. The calibration set was used for model development. The NIR spectra used for model development were either not pre-treated or pre-treated. The following pretreatments were used: mean normalization, maximum normalization, range normalization, first derivatives (5, 11 and 21 points), second derivatives (5, 11 and 21 points), baseline offset, standard normal variate (SNV), detrending, SNV + detrending or multiplicative scatter correction (MSC). The model accuracy was determined using full cross-validation, as indicated by the coefficient of determination (R^2) and SECV.

In external validation using the prediction set, the optimum model was selected based on the high coefficient of determination (R^2), low SEP and low prediction bias.

3. Results and Discussion

The repeatability of the traditional moisture content evaluation method was 0.45% and 0.15% for starch cake samples and dried starch samples, respectively. These values were accepted by the factory. The maximum (Max), minimum (Min), mean and standard deviation (SD) of the moisture content in tapioca starch of different groups are shown in Table 1.

Figures 1(a) and 1(b) show the average absorbance of the raw and second-derivative spectra of cake and dried tapioca starch samples, respectively. There were 3 dominant absorption regions in the

Table 1. Moisture content (% wet basis) of tapioca starch measured by the standard method used to develop the prediction model and validate the test set.

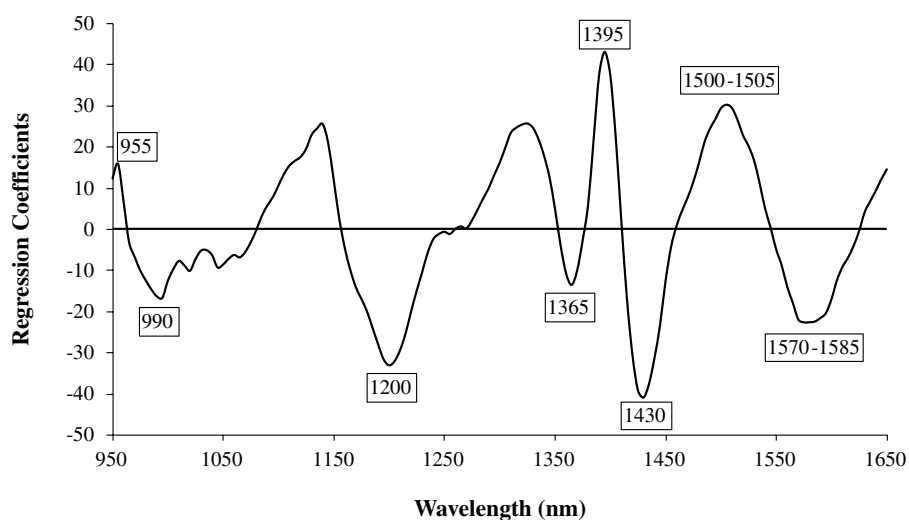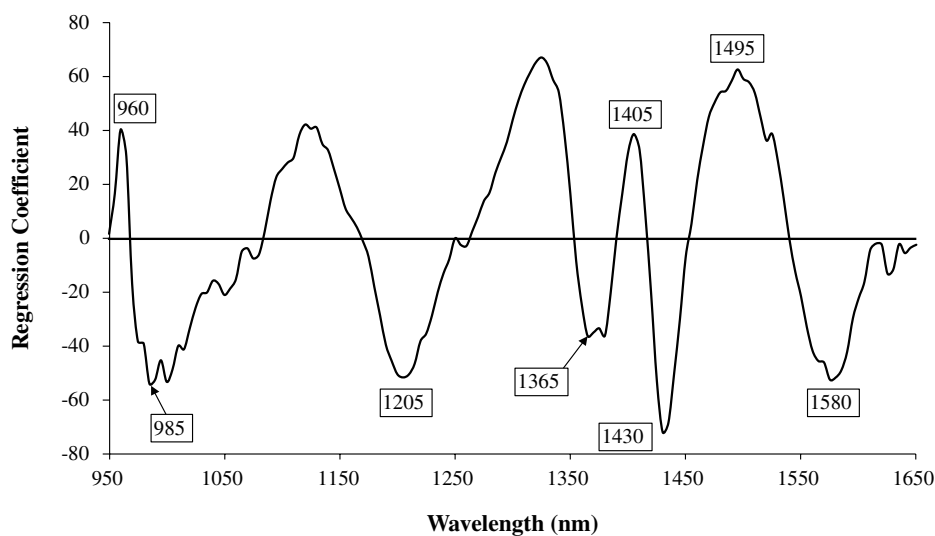| Group	Calibration					Prediction				
	N	Max	Min	Mean	SD	N	Max	Min	Mean	SD
Tapioca starch cake	70	39.42	29.32	33.41	1.84	35	41.13	29.93	33.60	2.09
Dried tapioca starch	70	14.53	10.91	12.81	0.66	35	18.82	11.39	12.98	1.18
Combined tapioca starch	140	39.42	10.91	23.12	10.43	70	41.13	11.39	23.29	10.52
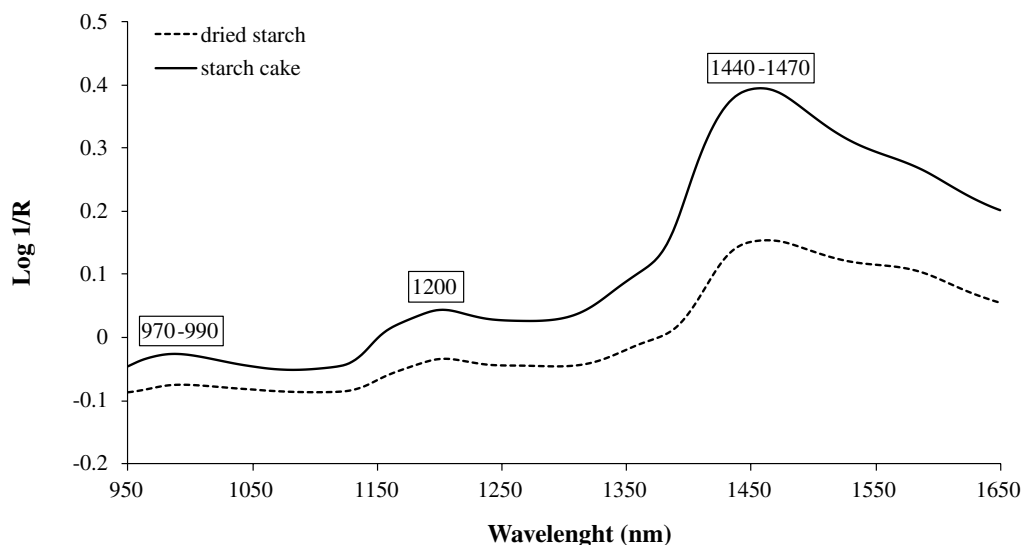
(a)
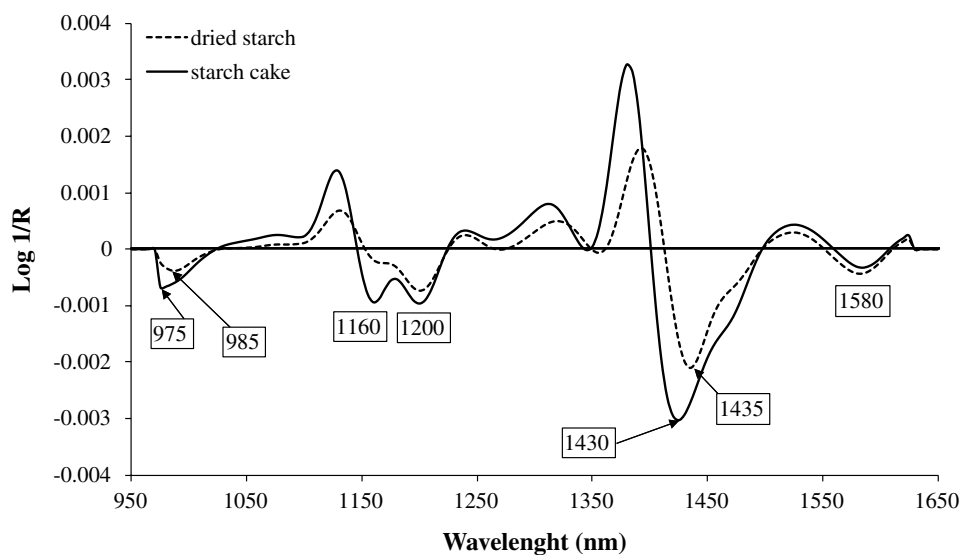
(b)

Fig. 1. Averaged NIR spectra of tapioca starch cake and dried tapioca starch. (a) Average raw absorbance spectra. (b) Average second derivative spectra (11 points).

Table 2.   X (NIR spectra) and Y (Moisture) explained variance for tapioca starch moisture content models.

| Model	PLS factor	X-explained variance (%)	Y-explained variance (%)
Tapioca starch cake samples	1	88	17
(Max normalize)	2	11	55
	3	0	9
	4	0	15
Dried tapioca starch samples	1	99	37
(Baseline)	2	1	32
	3	0	23
Combined samples	1	99	99
(Second derivative, 5 points)	2	0	1

(a)

(b)

Fig. 2.   Regression coefficient plots of optimum models for moisture content in tapioca starch. (a) Tapioca starch cake samples. (b) Dried tapioca starch samples. (c) Combined tapioca starch samples.
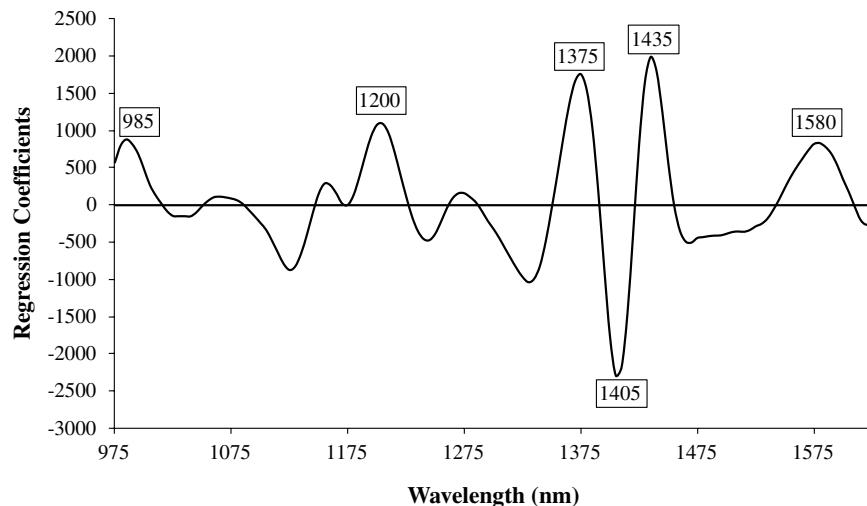
(c)

Fig. 2. (*Continued*)

raw spectra (Fig. 1(a)) i.e., 970–990, 1200 and 1440–1470 nm. In the measured range 950–1650 nm, water absorption due to the vibration of O–H bonds can be found at approximately 970, 1200 and 1450 nm.[14,15] However, according to Williams, the peak in the 1400 nm region was associated with the glucose molecules in the starch constituents.[16] In our case, the second-derivative spectra (Fig. 1(b)) shows that the tapioca starch cake has higher absorption than that of the dried tapioca starch which indicated that the peak in 1400 nm region is more

due to the absorption of water in the samples. Bands at 1200 and 1580 nm were also observed in both the cake and the dried starch spectra, corresponding to the starch band (1202 nm) and the first overtone associated with the O–H stretching of starch (1580 nm).[17] This was confirmed by Fig. 1(b) where there was very slightly different of absorption intensity between starch cake and dried starch indicated pure starch absorption. From second-derivative spectra (Fig. 1(b)), there is a peak at 1160 nm which is the shifted peak of 1190 nm of
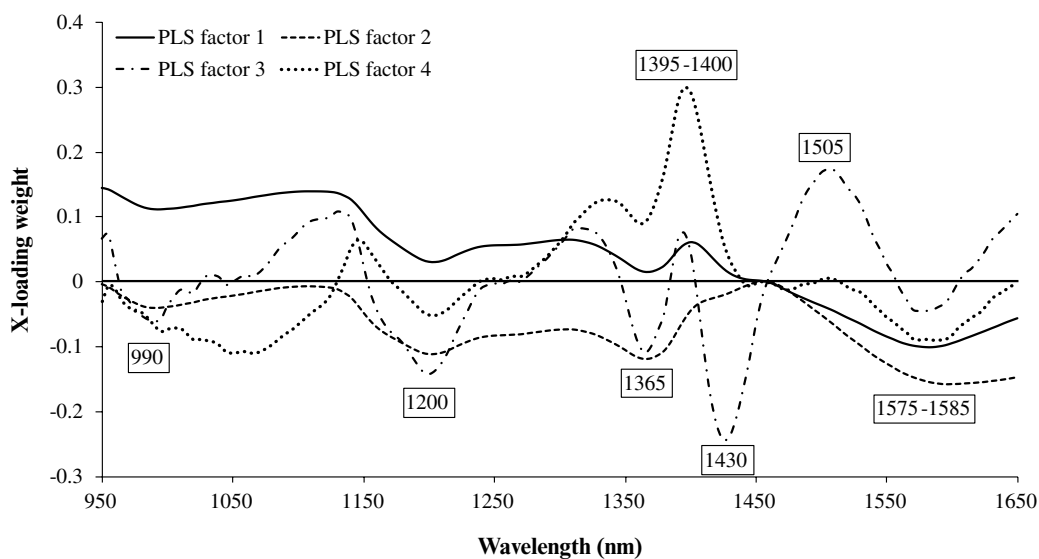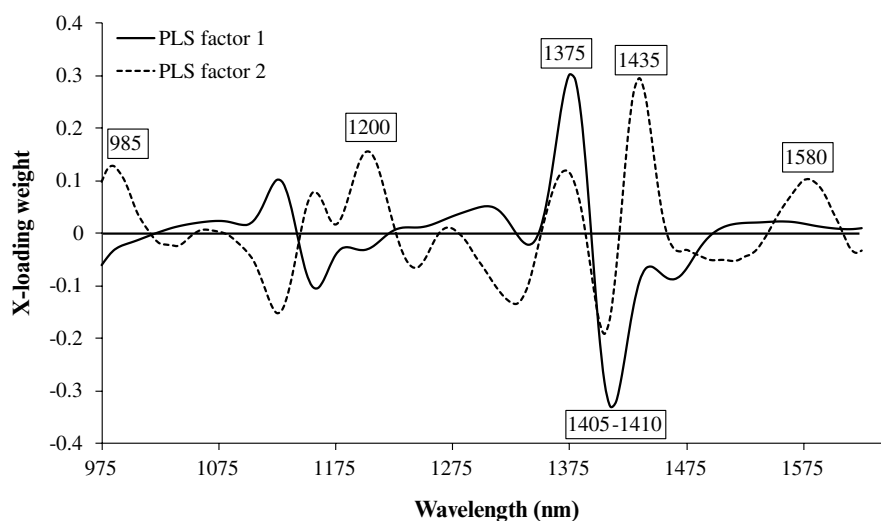

(a)

Fig. 3.   X-loading weight plots of optimum models for moisture content in tapioca starch. (a) Tapioca starch cake samples. (b) Dried tapioca starch samples. (c) Combined tapioca starch samples.
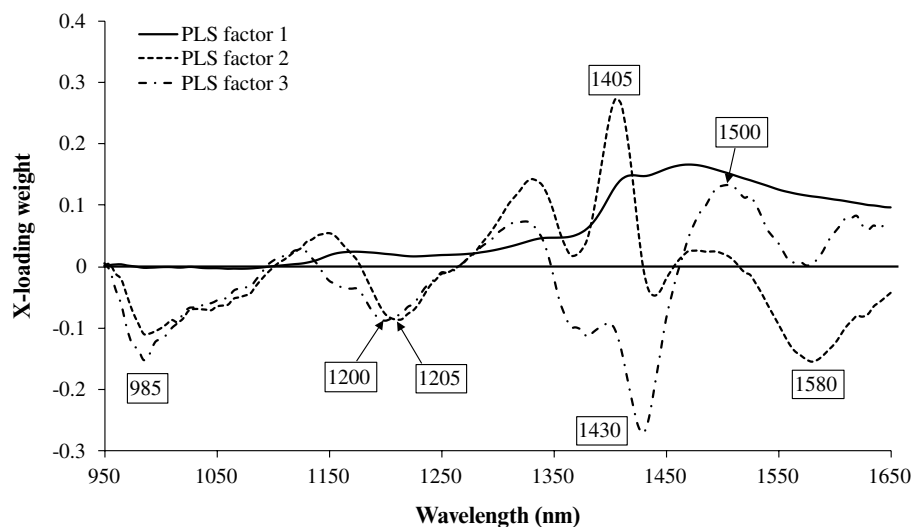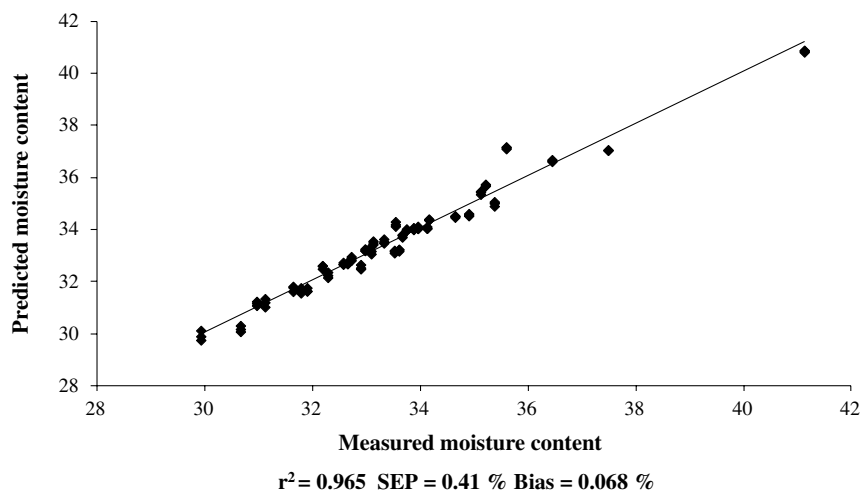
(b)

(c)

Fig. 3.   (*Continued*)

absorption band of water[18] where the absorbance intensity of starch cake is higher than that of dried starch. However, a band at 975 nm in the cake spectrum and 985 nm in dried starch spectrum could be observed, which were the second overtone associated with the O–H stretching of water (970 nm) and the second overtone associated with starch (990 nm).[17] The shift in the position of the band at 975 nm in the cake spectrum to 985 nm in the dried starch spectrum may have been a result of the removal of water during drying.
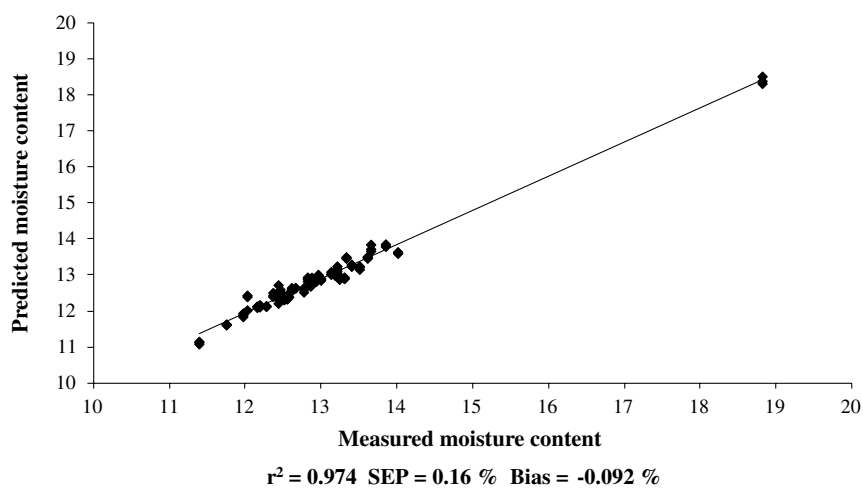
The optimum models for the three different groups were developed from the maximum-normalization, baseline-offset and second-derivative

(11 points) spectra, respectively, using 950–1650 nm wavelength range. The appropriate number of PLS factors were selected based on the minimum value of SECV. The optimum models for starch cake, dried starch and combined starch samples were developed from four, three and two PLS factors, respectively. The first PLS factor usually accounts for the highest proportion of the total variance (i.e., the combined spectra and reference data) of the system.[19] The explained variance in the X variables (NIR spectra) and Y variable (moisture content) by the PLS factors are shown in Table 2.

Figures 2(a)–2(c) show the regression coefficient plots of the optimum models for the moisture

r² = 0.965 SEP = 0.41 % Bias = 0.068 %

(a)

r² = 0.974 SEP = 0.16 % Bias = -0.092 %

(b)

r² = 0.995 SEP = 0.76 % Bias = 0.081 %
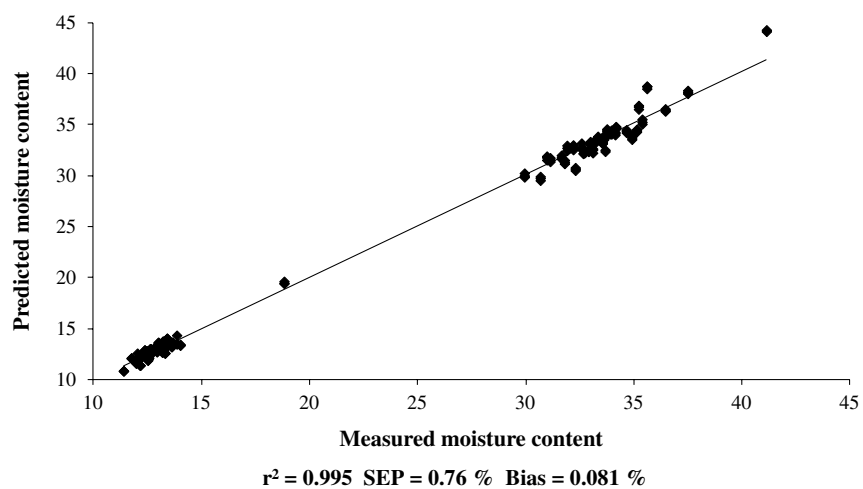
(c)

Fig. 4.   Validation plots of measured moisture content (X) with predicted moisture content (Y) in tapioca starch samples. (a) Tapioca starch cake samples. (b) Dried tapioca starch samples. (c) Combined tapioca starch samples.

content of starch cake, dried starch and the combined samples, respectively. The common bands relevant to the moisture prediction of the three different groups of starch models were those at approximately 985–990, 1200–1205, 1365–1375 and 1580 nm, which were, respectively, the second overtone associated with the O–H stretching of starch (990 nm), the starch bands (1202 nm) and (1360 nm) and the first overtone associated with the O–H stretching of starch (1580 nm), as indicated by Osborne *et al.*[17] Moreover, there was also an obvious band of the highest regression coefficient between 1430–1435 nm, where it was confirmed by second-derivative spectra (Fig. 1(b)) that they were the band of water. Therefore, these confirmed the high influence of O–H bands of water on the prediction of moisture content of tapioca starch.

The X-loading weights for the prediction models of the moisture content of tapioca starch are shown in Fig. 3. The X-loading spectra of various PLS factors show that vibration bands relevant to the moisture prediction of the three different groups starch models were located at 985–990, 1200–1205, 1395–1405, 1430–1435 and 1575–1585 nm. These bands were associated with starch and water.[14,15,17] It was again observed that the highest peak was at the 1430–1435 nm region showed the high influence of absorption of water in prediction of moisture

content. These confirmed that O–H bands of starch and the O–H bands of water influenced the prediction of the moisture content of tapioca starch.

Figures 4(a)–4(c) show the validation plots of the NIR spectroscopic models for the moisture content of tapioca starch cake, dried starch and combined samples, respectively, which are useful for illustrating the precision of each calibration model. The optimum models provided a coefficient of determination (R^2) of 0.965–0.995, SEP of 0.16–0.76%, bias of (0.068)–(−0.092)% and residual prediction deviation (RPD) of 5.1–13.8. The model of dried tapioca starch samples using baseline-offset spectra (Fig. 4(b)) had the highest prediction ability, i.e., the lowest SEP (0.16%), while the R^2, bias and RPD were 0.974, −0.092% and 7.4, respectively. Williams[19] has indicated that an R^2 of between 0.66–0.81, 0.83–0.90 and 0.92–0.96 implies that a model can be used for screening, usable with caution for most applications, including research, and in most applications, including quality assurance, respectively. In addition, he also indicated that an RPD of 3.1 to 4.9, 5.0 to 6.4 and 6.5 to 8.0 implies that the model can be used for screening, for quality control, and for process control, respectively. This confirm that both values are higher two level than screening application. The results of PLS regression models for predicting the moisture content of tapioca

Table 3. Results of the PLS calibration models.

Spectrum pretreatment	Calibration		Prediction			
	R^2	SECV	r^2	SEP	Bias	RPD
For tapioca starch cake samples prediction						
No pretreatment	0.973	0.32	0.902	0.71	0.164	2.9
Normalized						
Mean	0.955	0.41	0.958	0.42	−0.007	5.0
Max*	0.967	0.35	0.965	0.41	0.068	5.1
Range	0.967	0.35	0.965	0.41	0.074	5.1
Derivatives						
1st derivative (5 points)	0.962	0.37	0.910	0.66	0.125	3.2
1st derivative (11 points)	0.967	0.34	0.911	0.66	0.130	3.2
1st derivative (21 points)	0.973	0.31	0.903	0.71	0.167	2.9
2nd derivative (5 points)	0.937	0.49	0.909	0.65	0.103	3.2
2nd derivative (11 points)	0.968	0.35	0.918	0.62	0.105	3.4
2nd derivative (21 points)	0.965	0.35	0.917	0.63	0.119	3.3
Baseline	0.971	0.33	0.905	0.69	0.154	3.0
SNV	0.964	0.36	0.952	0.49	0.108	4.3
SNV + Detrending	0.968	0.34	0.954	0.48	0.107	4.4
Detrending	0.967	0.35	0.910	0.66	0.126	3.2
MSC	0.964	0.36	0.952	0.49	0.108	4.3

Table 3. (*Continued*)

Spectrum pretreatment	Calibration		Prediction			
	R^2	SECV	r^2	SEP	Bias	RPD
For dried tapioca starch samples prediction						
No pretreatment	0.942	0.16	0.963	0.21	−0.086	5.6
Normalized						
Mean	0.384	0.54	0.047	0.14	−0.050	1.0
Max	0.947	0.16	0.914	0.32	−0.123	3.7
Range	0.949	0.15	0.940	0.26	−0.122	4.5
Derivatives						
1st derivative (5 points)	0.939	0.17	0.961	0.21	−0.086	5.6
1st derivative (11 points)	0.941	0.16	0.961	0.21	−0.089	5.6
1st derivative (21 points)	0.947	0.16	0.968	0.18	−0.102	6.6
2nd derivative (5 points)	0.939	0.19	0.959	0.22	−0.076	5.4
2nd derivative (11 points)	0.939	0.17	0.967	0.20	−0.075	5.9
2nd derivative (21 points)	0.941	0.16	0.963	0.21	−0.088	5.6
Baseline*	0.949	0.16	0.974	0.16	−0.092	7.4
SNV	0.941	0.16	0.931	0.29	−0.105	4.1
SNV + Detrending	0.944	0.16	0.949	0.24	−0.098	4.9
Detrending	0.939	0.17	0.964	0.20	−0.095	5.9
MSC	0.941	0.16	0.931	0.29	−0.105	4.1
For combined tapioca starch samples (starch cake and dried starch) prediction						
No pretreatment	0.982	1.42	0.955	2.28	0.293	4.6
Normalized						
Mean	0.033	11.95	0.015	10.39	0.076	1.0
Max	0.992	0.96	0.991	0.97	−0.032	10.8
Range	0.994	0.85	0.994	0.83	0.023	12.7
Derivatives						
1st derivative (5 points)	0.995	0.78	0.992	0.96	0.127	11
1st derivative (11 points)	0.993	0.92	0.988	1.17	0.157	9
1st derivative (21 points)	0.981	1.45	0.952	2.36	0.356	4.5
2nd derivative (5 points)	0.988	1.17	0.972	1.85	0.321	5.7
2nd derivative (11 points)*	0.997	0.63	0.995	0.76	0.082	13.8
2nd derivative (21 points)	0.995	0.79	0.992	0.98	0.155	10.7
Baseline	0.977	1.58	0.947	2.55	0.456	4.1
SNV	0.992	0.92	0.992	0.97	0.102	10.8
SNV + Detrending	0.993	0.90	0.992	0.96	0.105	11
Detrending	0.981	1.44	0.953	2.34	0.343	4.5
MSC	0.992	0.93	0.992	0.97	0.103	10.8

*Selected optimum model, coefficient of determination for calibration model (R^2), coefficient of determination for prediction (r^2), SECV, SEP, SNV, MSC and RPD.

starch of starch cake, dried starch and combined sample groups are shown in Table 3. It could be used as guidance for process control and further application in a tapioca starch factory. This result was comparable to the results of FT-NIR spectroscopy for the prediction of moisture content of whole-wheat flour, where SEP, root mean standard error of prediction (RMSEP) and correlation coefficient (r) values of 0.15%, 0.38% and 0.85, respectively, were obtained.[20]

4. Conclusion

From the results presented in this study, NIR spectroscopy could be used as a rapid and powerful alternative to evaluate the moisture content of tapioca starch in factory quality control laboratories. The model developed from dried tapioca starch samples using baseline-offset spectra performed best. The effectiveness of the model leads to the possibility of using the moisture content of the starch at the end of the drying process as an alternative control

parameter for the starch cake feeder instead of the outlet temperature of the process. The NIR-based model, which was obtained from NIR off-line spectrometer (DA7200, Perten, Sweden), developed in the study can be installed into the NIR on-line spectrometer (DA7300, Perten, Sweden) for the on-line measurement of the moisture content of tapioca starch at the end of drying process in a tapioca starch factory. In addition, it was found that the influence of the NIR absorption of starch was stronger than that of water in the prediction of the moisture content of the model.

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