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### NON-DESTRUCTIVE MEASUREMENT OF SSC, PH, FIRMNESS AND DENSITY OF 'DANGSHAN' PEAR USING FT-NIR SPECTROMETRY

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**ABSTRACT** This research aims to investigate the feasibility to rapidly determine the quality characteristics of Chinese 'Dangshan' pear by using near infrared (NIR) spectroscopy. A total of 151 'Dangshan' pears with various ripen stages were used in this study. The NIR spectrum from each fresh intact sample was calibrated against four major physiological properties: soluble solid content (SSC), pH, firmness and density. The calibration models for these parameters were developed using the partial least square (PLS) based on the original and the four preprocessed spectra, respectively. The optimum spectral regions, the best pretreatment method and the fewest number of regression factors in the PLS-models were decided according to the minimum standard error in cross-validation (SECV). Finally, the proposed calibration models were validated using an independent sample set to evaluate their predicting ability. The best models for SSC, pH and firmness showed good predictability with the lowest standard error of prediction (SEP) of 0.349<sup>0</sup>Brix, 0.081 and 0.310N, and corresponding correlation coefficients of 0.985, 0.910 and 0.905, respectively, indicating that it is possible to measure SSC, pH and firmness of Chinese 'Dangshan' pear by using NIR spectroscopy. However, validation results also showed that no satisfactory NIR calibration equation for the density could be obtained.

**Keywords:** NIR spectroscopy, Pear, Soluble solid content, pH, Firmness, Density.

**INTRODUCTION** The Chinese pear 'Dangshan' is a famous variety in the 'white pear system' and is very popular among Chinese consumers. The customer purchase behavior is mostly determined by the fruit quality. Thus it is very important for producers to evaluate fruit quality and to sort them accordingly to satisfy the customer taste preferences. Unfortunately, little research work concerning the quality evaluation and grading of 'Dangshan' pear has been reported. The 'Dangshan' pears usually are sold without quality evaluation and grading in china due to lacking rapidly responding technologies. Fruits usually are sorted manually or automatically according to their size, color and surface defect. However, internal quality attributes such as SSC and pH, which are considered to be closely related to the taste of ready-to-eat fruits, are important factors which are most likely to match the consumer's perception and favor. These parameters

require measurement techniques which are traditionally destructive, complex and time-consuming. To sort intact fruits in terms of their internal quality characteristics, the need for a rapid and nondestructive measurement technique is imperative.

NIRS has been widely adopted as a laboratory technique to estimate useful chemical compositions of fruit due to its outstanding advantages: rapid and simultaneous measurement of multiple attributes and repeated measurement on the same sample (Dull et al., 1989; Dull et al., 1992; Slaughter, 1995; Lu, 2001; Manuela et al., 2002; Nicolai et al., 2006). The potential of NIR spectroscopy for measuring internal quality attributes has been evaluated extensively in the recent ten years (Lammertyn and Nicolay, 1998; McGlone et al., 2002; Liu and Ying, 2005; Gómez et al., 2006; Liu et al., 2006; Zude et al., 2006). In addition, many researches currently focus on the development of NIR methods for the nondestructive detection of defects, for example, brownheart and bruise in apples, grading of peaches etc. (Clark et al., 2003; Carlomagno et al., 2004; Kleynen et al., 2005; McGlone et al., 2005; Lu & Peng, 2006). Kojima, Inoue & Tanaka (1994) investigated the feasibility of NIR spectroscopy for the prediction of SSC in Japanese pear. Sirisomboon et al. (2007) reported that NIR spectroscopy has potential to measure the pectin constitutes of Japanese pear. Tanaka and Kojima (1996) presented that the growth period of Japanese pear fruit could be monitored by the near-infrared spectroscopy on basis of sugar concentration constitutes. As described by Han et al. (2006), the Chinese pear ‘Yali’ with brown core could be nondestructively identified and be graded into three classes by using the transmission visible/NIR spectroscopy.

Most research work about quality detection of freshly harvested and stored fruit focus on SSC and acidity of fruits. Actually, firmness and density are closely correlated with texture properties and eating quality of fruit and also are important quality attributes influencing on perception of customers (Zhang and Wang, 1991). However, little has been reported in the literatures about the NIR spectroscopy to simultaneously determine these parameters.

The objective of this study was to examine the potential of NIR spectrometry to simultaneously measure SSC, pH, firmness and density of the Chinese ‘Dangshan’ pear.

## MATERIALS AND METHODS

**Samples** A total of 151 ‘Dangshan’ pears were purchased in local auctions and used in this study. Table 1 describes the morphological characteristics of the samples. Specially, the intact pears of various ripen levels were selected so that their ranges of variability in SSC, pH, firmness and density were as wide as possible. All samples were stored for 24 hours at 20<sup>0</sup>C to equilibrate the temperature to room condition (19- 21<sup>0</sup>C) before being examined.

Table 1. Morphological properties and descriptive statistic data of pear samples

	Maximum	Minimum	Mean	SD*	CV** (%)
Radial diameter (cm)	11.42	7.23	8.82	0.69	7.83
Lengthways diameter (cm)	13.28	6.61	8.51	0.84	9.81
Mass (g)	709.40	211.17	357.00	88.99	24.88
Volume (cm <sup>3</sup> )	700.00	192.00	354.76	94.82	26.73

\*SD=Standard deviation; \*\*CV=Coefficient of variation

**Reference methods** For each pear, three tissue samples were cut from the marked regions close to the positions in which the NIRS reading had been taken. Then these tissue samples were macerated with a manual fruit squeezer. The filtered juice samples were finally used for SSC measurement by a hand-held sugar refractometer (Model WAY, Instrument Co. Ltd, Shanghai, China), with range 0–80 °Brix and accuracy  $\pm 0.1\%$ . The average of three measurements was used for calibration analysis. The pH was measured using a SJ-4A pH meter (Instrument Co. Ltd, Shanghai, China). Results are given as the average of triplicate measurements. A universal testing machine (TA.XTplus, Stable Micro System CO., UK) was used to determine the firmness. A cylindrical probe 5 mm in diameter was pressed in the peeled pear to a depth of 10 mm. The drive forward speed of testing machine was set at 2 mm/s. The firmness is defined as the maximum force attained in MT penetrometer test. The density was determined by measuring pear's weights in air and water and volume based on Archimedes' principle by full pear immersion in two litres of clean tap water, respectively. This measurement was repeated three times and the average of triplicate measurements were used to correlate to the spectral data.

**Near infrared spectroscopy** Each sample was scanned at 2 nm intervals in the spectral range 850 – 2500 nm using the Antaris II FT-NIR spectroscopy instrument (Thermo Nicolet CO., USA). Three separate spectral measurements were carried out on each sample on three equatorial positions ( $120^\circ$  turn) around the equator of the fruit. Three reflective spectra were averaged to provide a mean spectrum for calibration equations.

**Data treatment** The samples were split into a calibration set (115 fruits) for development of calibration and a validation set (36 fruits) for validation of the proposed best prediction model. The reflectance spectra were calibrated against the four parameters individually using partial least squares (PLS). In order to enhance the relevant spectral information, four pre-processing of spectra: Multiplicative Scatter Correction (MSC), Standard Normal Variance Transformation (SNV), First Derivation (FD) and Second Derivation (SD), available in the TQ Analyst 7.2.0.161 software, were investigated. The minimum standard error in cross-validation (SECV) with the leave-one-out option was used to choose the number of regression factors in the PLS-models. The optimal spectral region and pretreatment method also was decided based on the lowest SECV. The best calibration model for each parameter was individually selected according to the lowest SEVC and the highest correlation coefficient ( $R_{cal}$ ). After calibration, the resulting models were validated using validation set. Correlation coefficient ( $R$ ) between the predicted and measured values, standard error of prediction (SEP) and bias i.e. the mean difference between the predicted and actual values in the validation set were used to evaluate the performance of the best models.

The software TQ Analyst 7.2.0.161 was used for near infrared spectral data collection, spectral processing and calibration model development.

## RESULTS AND DISCUSSION

**Reference measurements** The descriptive statistics for SCC, pH, firmness and density of all samples were listed in Table 2. This table shows that the samples span greater parameter ranges in SSC and firmness, while the variations in pH and density are minor. Some characteristics of fruit internal quality are known to be inter-dependent. For most

fruits, the SSC usually increased and firmness decreased with the increased in maturity. Table 3 lists the cross correlation values (R) of these parameters. It can be observed that the dependency exists between SSC and pH. However, there are not dependencies between SSC and firmness, SSC and density, firmness and pH, firmness and density, and pH and density.

Table 2 Statistic data of Soluble solid content, PH, Firmness and Density in calibration and prediction sets

Samples	Calibration set				Prediction set			
	SCC (°Brix)	PH (pH)	Firmness (N)	Density (g/cm <sup>3</sup> )	SCC (°Brix)	PH (pH)	Firmness (N)	Density (g/cm <sup>3</sup> )
No. of samples	107	105	105	107	35	33	35	35
Mean	11.00	5.20	2.83	1.01	10.99	5.20	2.68	1.02
Maximum	13.08	5.42	4.34	1.08	12.13	5.34	3.62	1.05
Minimum	8.58	4.88	2.09	0.93	9.90	5.02	2.18	0.95
SD*	0.86	0.09	0.38	0.03	0.55	0.09	0.37	0.03
CV**(%)	7.82	1.79	13.55	3.36	5.00	1.71	13.63	3.16

Table 3 Cross correlation values for reference data

	SCC	PH	Firmness	Density
SCC	1	-0.496	-0.366	0.092
PH	-0.496	1	0.215	-0.046
Firmness	-0.366	0.215	1	0.090
Density	0.092	-0.046	0.090	1

**NIR predictions** Fig.1 shows that the spectral absorption curves display several peak bands in 1100 – 2450nm. Two prominent peaks appear at around 1449 and 1934 nm, which are related to the strong water absorbance bands at 1440 and 1940 nm. Other two light broad peaks can be seen at around 1210 and 1780 nm, respectively, which are associated to the C-H second and third overtones. It should be noted that the absorbance curves for pear ‘Dangshan’ are similar to that for apple (Liu and Ying, 2004), peach (Kawano et al. 1992) and mandarin (Gómez et al., 2006). Table 4 summarize the efficiency of the NIRS calibrations for SSC, pH, firmness and density developed using the raw spectra in the optimal spectral ranges proposed by the analysis software TQ Analyst 7.2.0.161. For SSC, pH and firmness, their best calibration models were obtained in spectral region 1075 – 2298nm, with the highest coefficient of determination ( $R^2$ ) and the lowest SECV. For the density, the calibration model developed in spectral region 1653 – 2298nm yielded the highest  $R^2$  and the lowest SECV. Therefore, the spectral regions 1075 – 2298nm and 1653 – 2298 nm were used in the model optimizing in following sections, respectively.

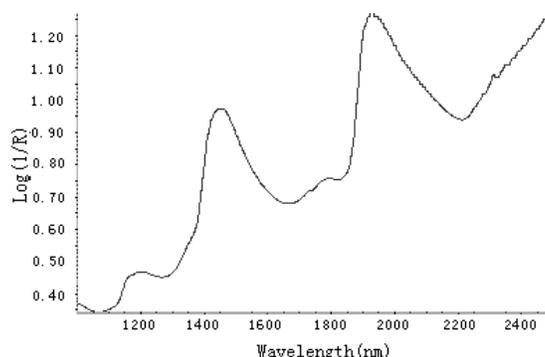


Figure 1. Average spectra of one pear sample in the spectral region 850 – 2500 nm

Table 4 Calibration and cross-validation results for SSC, pH, firmness and density based on original spectra in the optimal wavelength ranges

Parameter	Wavelength Range(nm)	No.of factors	$R_{cal}$	SECV ( $^{\circ}$ Brix)
SCC	1075 - 2298	8	0.935	0.462
pH	1075 - 2298	7	0.857	0.068
Firmness	1075 - 2298	9	0.859	0.325
Density	1653 - 2298	6	0.779	0.029

**SSC** After 8 outlier samples were eliminated, calibration equations for SSC were developed using 107 samples in the spectral region 1075 – 2298nm based on four pre-processed spectra. Calibration and cross validation statistics for the SSC equations were listed in Table 5. Present calibration statistics show that the calibration equation based on MSC treated spectra has the highest  $R_{cal}$  and the lowest SECV compared to the other equations. Moreover, in comparison to the calibration model developed based on original spectral data, a slightly improvement can be observed in  $R_{cal}$  and SECV after the MSC treatment, indicating the effect of pre-treatment on the prediction ability of the equations. After removing one outlier sample, the independent validation set consisting of 35 samples was used to test the predictive ability of the best equation. The 7-component model gave a standard error of prediction (SEP) 0.349  $^{\circ}$ Brix, bias -0.010  $^{\circ}$ Brix and a simple correlation coefficient ( $r$ ) 0.980, indicating the excellent calibration model. The results are superior to those reported by Nicolai et al. (2008), who obtained a reasonable PLS model for SSC of pear with RMSEP 0.44  $^{\circ}$ Brix and  $R^2$  0.60 based on raw reflectance spectra in the range 780 – 1200nm. And Liu et al. (2006) in pear with  $r = 0.915$  and  $RMSEP = 0.683^{\circ}$ Brix. Fu et al. (2006) proposed the best SSC PLS-model for three cultivars of pears with  $R^2$  of 0.93 and RMSEP of 0.57  $^{\circ}$ Brix, respectively, being comparable to the present results. In addition, the present SECV is lower than those reported for apple (Moons et al., 1997; Ventura et al., 1998), for mango (Saranwong, et al., 2004) and for peaches (Kawano, 1994; Kawano et al., 1992). Thus, it could be concluded that the calibration model for the prediction of SSC was successfully developed.

Table 5 Calibration and cross-validation results of SSC-PLS models based on processed spectra with four math treatments in the optimal wavelength range

Wavelength region (nm)	Pretreatments	No. of factors	Rcal	SECV
1075 – 2298	SNV	7	0.939	0.472
	MSC	7	0.940	0.460
	FD	4	0.938	0.673
	SD	6	0.934	0.674

**pH** The calibration equations for pH were developed based on four treated spectra in the spectral regions 1075 – 2298nm. After eliminating 10 outliers, the equation incorporated 105 of the total calibration set. Table 6 shows the results of the multiple regression analysis. The highest  $R_{cal}$  of 0.862 was achieved with the corresponding lowest of SECV 0.067 when applying the treatment SNV to the spectral data. The relatively high determination coefficient indicates that there is a good correlation between the measured and NIRS predicted pH for the calibration set. The best calibration model was validated using the validation set including 33 samples (three samples were outliers). The validation results presented a standard error of prediction (SEP) 0.081, bias 0.005 and a simple correlation coefficient ( $r$ ) 0.910. These results are remarkably superior to the results reported by Gómez et al. (2006), who calibrated NIR reflectance spectra of Satsuma mandarin against pH values, obtaining a correlation coefficient ( $r$ ) 0.84 and SEP 0.18 using 7-components PLS model. Our results are comparable to those reported by Liu and Ying (2005), who presented that the lowest SEP 0.068 with corresponding correlation coefficient ( $r$ ) 0.831 could be obtained using a proposed calibration model for pH of ‘Fuji’ apple. Their reported SEP is lower than our proposed, but their reported correlation coefficient ( $r$ ) 0.830 is significant less than ours ( $r$ ). In addition, present obtained in this study inferior to the results reported by Lammertyn and Nicolay (1998) in apples, with a regression coefficient ( $r$ ) 0.93 and SEP 0.068.

Table 6 Calibration and cross-validation results of pH-PLS models based on processed spectra with four math treatments in the optimal wavelength range

Wavelength region (nm)	Pretreatments	No. of factors	Rcal	SECV
1075 - 2298	SNV	6	0.862	0.067
	MSC	6	0.859	0.067
	FD	5	0.828	0.071
	SD	9	0.821	0.085

**Firmness** The wavelength regions 1075 – 2298nm and four math treatments were used in construction of calibration equations for the firmness. Ten samples were found as outlier samples. Table 7 summarizes the calibration and cross validation statistics for the firmness equations. The lowest resultant SECV and the highest  $R_{cal}$  were obtained based on the treated spectra by SNV method. In comparison with the calibration results shown in Table 4, an improvement in calibration statistics is observed when treatment SNV was adopted. Equation validation was carried out to test the performance of the best equation after eliminating one sample in validation set. The result of validation statistics presents the standard error of prediction (SEP) 0.310 N and a correlation coefficient ( $r$ ) 0.905. The relatively high correlation coefficient ( $r$ ) indicates good performance of the present model.

Table 7 Calibration and cross-validation results of firmness-PLS models based on treated spectrum with four math treatments in the optimum wavelength range

Wavelength regions (nm)	Pretreatments	No. of factors	R <sub>cal</sub>	SECV (N)
1075 - 2298	SNV	7	0.865	0.329
	MSC	6	0.832	0.331
	FD	1	0.581	0.727
	SD	1	0.699	0.733

These results are comparable to that reported by Fu et al. (2006), who found that the best prediction model for firmness in all three cultivars of pear gave  $R_{cal}$ , RMSEC and RMSEP 0.92, 2.29 N, 2.95 N, respectively. Liu, Chen, Ouyang & Ying (2007) presented that the combination of PLS regression and first derivation of spectra resulted in a correlation coefficient ( $r$ ) of 0.8125 and RMSEP of 1.3778N for firmness of pear, respectively.

In comparison with previously published data for other fruits, the present calibration equations show comparable precision with relatively intermediate SEC. For instance, even if the best equation gave slight lower  $R_{cal}$  than those reported for apple by Gómez et al. (2006) ( $R_{cal} = 0.87$ ), their reported SEC value corresponds to 17.88% of the range of firmness in the calibration set, while our SEP is 8.49%. In another study, Schmilovitch et al. (2000) found that the best prediction for firmness of mango with MLR models had a SEP of 17.14 N and a coefficient of determination of 0.8226, respectively.

**Density** Table 8 summarizes the calibration and cross-validation results based on the treated spectra in the optimal wavelength range 1653 – 2298nm. The best calibration equation was found based on preprocessed spectra with FD treatment, where a 1-component model gave the highest correlation coefficient ( $R_{cal}$ ), the lowest SEC and SECV, respectively. These results are poorer than those given by the calibration model based on raw spectra in the same wavelength range shown in Table 4, showing that no better performance of the equation could be obtained when four pre-processing methods were applied to calibration. As a result, the calibration equation established based on

untreated spectra in wavelength range 1653 – 2298nm was the best one. The calibration and cross validation on the best model seems to display slightly successful results. However, the validation of the best equation with the independent data set resulted in poor results with a relative high SEP 0.033 g/cm<sup>3</sup>, bias 0.028 g/cm<sup>3</sup> and low correlation coefficient (*r*) 0.218. From these results, we can conclude that the satisfactory calibration model for the density of pear could not be established by using NIR spectroscopy.

Table 8 Calibration and cross-validation results of density-PLS models based on treated spectrum with four math treatments in the optimum wavelength range

Wavelength regions (nm)	Pretreatments	No. of factors	Rcal	SECV (g/cm <sup>3</sup> )
1653 - 2298	SNV	2	0.587	0.029
	MSC	2	0.587	0.029
	FD	1	0.630	0.030
	SD	1	0.631	0.030

Our failure to construct an acceptable NIR prediction model for the pear density might be due to the too narrow range in density. The considerable large range in size of pear samples might be another factor to affect the construction of a reasonable calibration model. Further research with respect to size and density range of pear fruit is necessary. Additionally, the absorption and tissue properties of pear fruit also need to be investigated. If the noisy levels could be reduced, the calibration model could be refined and NIR technique might be acceptable to predicting this parameter.

**CONCLUSION** This study demonstrates that NIR had good reliability in the prediction of SSC and pH of Chinese pear ‘Dangshan’. The proposed model can be used for routine predicting for the SSC and PH of Chinese pear ‘Dangshan’. Data from this study also shows that NIR is a viable technique for predicting the physical property of Chinese pear ‘Dangshan’. Good prediction performance by the proposed calibration model for the firmness indicates that NIR technique has potential to estimate the firmness of Chinese ‘Dangshan’ pear. However, the performance of density equation is poor and will be subjected to continue expansion and refinement. Further research work is needed to improve the prediction accuracy of the density equation. To sum up, this study has confirmed the potential of the NIR for predicting SSC, PH and firmness of Chinese pear ‘Dangshan’. Both calibration and validation results have indicated that the presented NIR models are useful tools for nondestructive measuring internal quality attributes of Chinese pear ‘Dangshan’ with acceptable accuracy.

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