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Nondestructive measurement of internal quality of Nanfeng mandarin fruit by charge coupled device near infrared spectroscopy

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ABSTRACT

The soluble solids content (SSC) and total acidity (TA) are the major characteristics for assessing quality and maturity of Nanfeng mandarin fruits. The feasibility of charge coupled device near infrared spectroscopy (CCD-NIRS) combining with effective wavelengths selection algorithm used to measure SSC and TA nondestructively was investigated. The effective wavelengths to SSC and TA were chosen by interval partial least squares (iPLS) in the wavelength range of 600–980 nm. The predictive ability of SSC model used PLS regression was improved with *r* of 0.92 and RMSEP of 0.65 °Brix using effective wavelengths of 681.36–740.51 nm, 798.60–836.19 nm and 945.52–962.75 nm. The TA model was simplified with *r* of 0.64 and RMSEP of 0.09% using effective wavelengths of 817.57–836.19 nm, 909.85–927.60 nm and 945.52–962.75 nm. The experimental results demonstrated that the CCD-NIRS technique combining with iPLS algorithm was a feasible method to measure SSC and TA of Nanfeng mandarin fruits nondestructively.

1. Introduction

Fruit quality is an important factor affecting its market value, transportation and storage requirements. Fruit quality indices consist of internal quality, such as soluble solids content (SSC) and total acidity (TA), and external quality, such as size and weight. To measure the external quality of fruit, many researches have been conducted leading to promising advancements. As a result, there are many types of fruit sorters based on size or weight of the fruit (Blasco et al., 2003; Leemans et al., 2002; Kondo et al., 2000). However, determining the internal quality of fruit is not as straightforward as external quality measurement. Internal quality of fruit is usually measured by destructive or invasive approaches which involved a considerable amount of manual work.

Quantitative spectroscopy has been greatly improved by use of a variety of multivariate statistical methods of partial least squares (PLS) and principal component regression (PCR) (Haaland, 1988; Haaland and Thomas, 1988a,b). PLS or PCR can easily treat very large data matrices, extracting the relevant part of the information and producing reliable but very complex models (Geladi and Kowalshi, 1986; Chu et al., 2004). Recently PLS was considered to be almost unaffected by noise, and therefore it was commonly stated that no feature selection at all was required (Thomas and Haaland, 1990). This attitude has changed, and therefore it has been widely recognized that a feature selection can be highly beneficial, since a double goal can be reached: improve the predictive ability of the model and highly simplify it (Leardi and Gonzólez, 1998; Borin and Poppi, 2005). The interval partial least squares (iPLS) is developed by Nørgaard et al., which searches for a spectral interval that is particularly informative with respect to the parameter under consideration (Pereira et al., 2008). The SSC and TA are two most important internal quality parameters of Nanfeng mandarin fruit. But SSC and TA determination are normally performed destructively on juice. Charge coupled device near infrared spectroscopy technique (CCD-NIRS) combining with chemometrics algorithms is a powerful tool, because of its fast detection and simple operation in sampling. NIRS technology has been used to determine SSC and TA in mandarin fruit (Gómez et al., 2006; McGlone et al., 2003; Liu et al., 2009), apple (Liu et al., 2007; Liu and Ying, 2005; Sánchez et al., 2003; Lu and Ariana, 2002; Lammertyn et al., 1998), tomato (Shao et al., 2007; André et al., 2005; Slaughter et al., 1996) and peach (Slaughter and Crisosto, 2006; Ying et al., 2005). Even if these instruments are highly accurate, their application to field research, such as monitoring chemical changes of developing fruits on trees, are limited by their large sizes and weights. CCD-NIRS sensor supplies the chance to develop portable or online operation, because its advantages of low cost, robust performance and small size. The charge coupled device (CCD) micro-spectrometers sometimes suffer from poor performance compared to conventional spectrometers but are perfectly suited for use with fiber optics (Davies et al., 2001). And some recent studies have illustrated the use of CCD micro-spectrometers for portable NIR applications.

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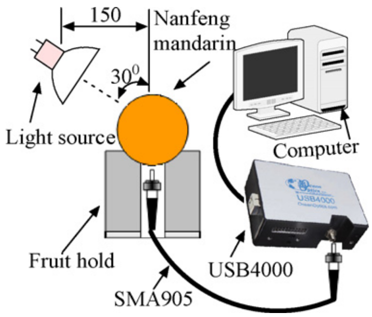

Fig. 1. Schematic of Nanfeng mandarin fruit CCD-NIRS acquisition device.

Saranwong et al. (2003) reported that the accuracy of the Kubota NIR instrument "Fruit Selector" was 0.40 °Birx in SEP for SSC value determination of intact mango. Temma et al. (2002) showed that the portable NIR instrument developed by their research center had excellent potential in determining SSC value of intact apples. Reita et al. (2008) evaluated variety compatibility of fruit to a NIR Brix calibration system of NIR-GUN (FANTEC, Kosar-city, Japan). Camps and Christen (2009) developed a portable NIR instrument for determining SSC, TA and firmness of apricots with a miniature optical component (S-2000).

The objectives of this study are to evaluate the feasibility of CCD-NIRS in nondestructive measurement of the SSC and TA in Nanfeng mandarin fruit. The prediction of calibration models were also compared in the manuscript.

2. Materials and methods

2.1. Nanfeng mandarin fruit

One hundred and fifty three Nanfeng mandarin fruit samples were harvested in a local orchard of Shishan, Nanfeng, China, in 10 October 2007 (latitude: 116.50; longitude: 27.23). After arriving laboratory, they were placed in airtight polyethylene bags and stored in an ice filled refrigerator to keep at cold temperature (4 ± 1 °C). All fruit samples were allowed to equilibrate to room temperature (20 °C) before NIRS measurement.

The sample composed of two varieties of Mandarin namely Daguoxi (77 fruits) and Xiaoguoxi (76 fruits). For increasing the application scope of models, Daguoxi and Xiaoguoxi samples were put together. Thus 153 fruit samples were used to establish the calibration models. However, because of chemical measurement errors, 4 and 13 samples were deleted as outliers for the SSC and TA calibration, respectively. The residual samples were divided into calibration and prediction sets, the statistical results were presented in Table 1.

2.2. System set up and transmission measurements

Fig. 1 showed the schematic of Nanfeng mandarin fruit CCD-NIRS acquisition system. The system consisted of a tungsten halogen lamp (24 V/50 W), CCD spectrometer (USB4000), an optical fiber (SMA905), USB data line and PC. The CCD spectrometer was 3648 pixel photodiode array. The wavelength range of the spectrometer was 350–1040 nm with a 0.2 sampling interval. Reflectance, interactance and transmittance modes compared by Nicolaï et al. (2007). The transmittance mode was applied, because the peel of mandarin was interference with determination internal quality precisely (Krivoshiev et al., 2000). The transmitted light carried information about the skin and core of the mandarin when the arrangement of 180° was applied, which might not be relevant to internal quality. Therefore an arrangement of about 30° was adopted between the lamp axis and the vertical line. The horizontal distance was about 150 mm between the center of lamp and fruit hold. And a similar system had been applied to determine SSC of intact citrus with RMSEP of 0.54% by Lu et al. (2007). Fu et al. (2007) detected brown heart of pear and Sun et al. (2009) investigated the effect of fruit moving speed on predicting SSC of 'Cuiguan' pear by the same similar arrangement.

NIR spectra were collected and transformed by OOIBase32 software (Oceanoptics Inc., Dunedin, USA), from three positions which were marked with a circle beforehand on each mandarin fruit around equatorial position. The averaged transmission reflectance spectrum of every mandarin fruit was analyzed with the statistical program for multivariate calibration of Unscrambler v9.5 software (CAMO AS, Trondheim, Norway).

2.3. SSC and TA reference analysis

SSC and TA were determined by traditional destructive tests. Each fruit unit was juiced, then SSC was obtained using a digital refractometer PR-101α (Atago Co. Ltd., Tokyo, Japan), TA was assessed by potentiometric titration of a 2 mL sample of juice and expressed as percentage of citric aid.

3. Results and discussion

3.1. Evaluation index of model

The performances of the PLS calibration model were evaluated in terms of the root mean square errors of cross validation (RMSECV), the root mean square errors of prediction (RMSEP) and the correlation coefficient (r). The RMSECV was calculated as follows:

$$E_c = \sqrt{\frac{1}{n_c - 1}\sum_{i=1}^{n_c}(\hat{y}_i - y_i)^2} \quad (1)$$

where E_c is the RMSECV, $\hat{y}_i$ is the prediction value of the ith observation, y_i is the measured value of ith observation and n_c is the number of observation in calibration set.

For the prediction set, the root mean square error of prediction (RMSEP) is calculated as follows:

$$E_p = \sqrt{\frac{1}{n_p - 1}\sum_{i=1}^{n_p}(\hat{y}_i - y_i)^2} \quad (2)$$

where E_p is the RMSEP, $\hat{y}_i$ is the prediction value of prediction set sample, y_i is the measured value of prediction set sample, and n_p is the number of observation in prediction set.

Table 1
Statistics of SSC and TA measured by the standard destructive methods for the calibration and prediction sets of Nanfeng mandarin fruit.

	Calibration set					Prediction set				
	Number	Average	Max.	Min.	S.D.	Number	Average	Max.	Min.	S.D.
SSC (°Brix)	107	14.00	18.00	11.20	1.68	43	14.00	10.50	18.00	1.64
TA (%)	104	0.44	0.81	0.23	0.08	42	0.45	0.75	0.22	0.07

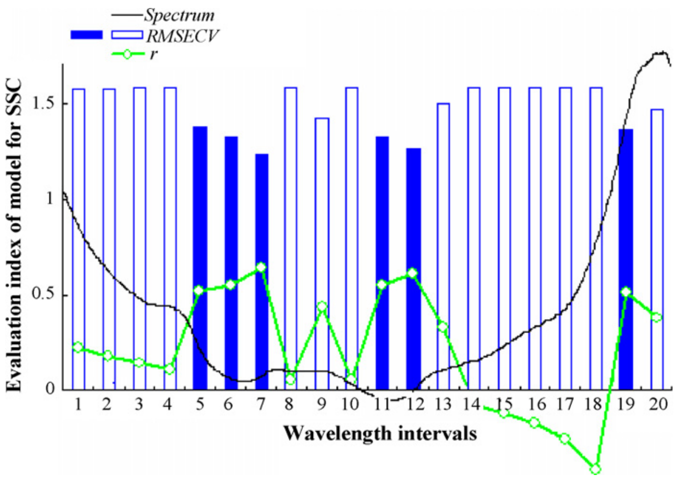

Fig. 2. Result of effective wavelength bands selection for SSC prediction.

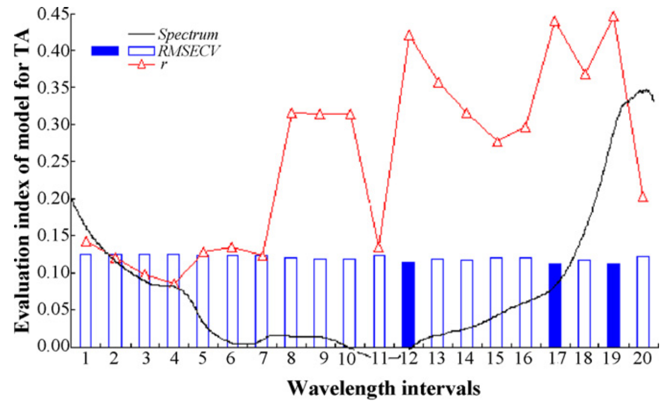

Fig. 3. Result of effective wavelength bands selection for TA prediction.

Correlation coefficients between the predicted and the measured value are calculated for both calibration and prediction set, which are calculated as follows:

$$r = \sqrt{\frac{\sum_{i=1}^{n}(\hat{y}_i - y_i)^2}{\sum_{i=1}^{n}(\hat{y}_i - y_m)^2}} \tag{3}$$

where r is the correlation coefficient, $\hat{y}_i$ is the measurement or prediction value of samples in calibration and prediction sets, y_m is the mean of the reference measurement results for all samples in calibration and prediction sets, and y_i is the number of observation in calibration and prediction set.

3.2. Effective wavelengths selection by iPLS

The iPLS algorithms used in the research were developed by Nørgaard et al. (2000). The principle of the algorithms are to split the spectra into smaller equidistant regions, and the regression models are developed in every subinterval. Therefore, the RMSECV of every subinterval will be calculated. The interval is chosen when the RMSECV is lower than the averaged RMSECV of all the subintervals. The main advantage of the iPLS algorithms is to represent a local regression model in a graphical display, to choose better intervals and permit a comparison between interval and the full-spectrum models. The algorithms are intended to give an overview of the data and can be helpful in interpretation.

Wavelength selection not only enhances the stability of the calibration model resulting from the collinearity in multivariate spectra but also helps to interpret the relationship between the model and the sample compositions (Fearn, 1997). Xie et al. (2009) selected the effective wavelength to ethylene content in tomatoes in the visible and shortwave NIR region. Piron et al. (2008) selected the most efficient wavelength bands for discriminating weeds from crop.

In order ot improve the predictive ability for SSC and TA determination of Nanfeng mandarin fruit in the range of 600–980 nm, the selection of effective wavelengths for Nanfeng mandarin fruit was accomplished by the iPLS regression. The spectrum was divided into 20 equidistant subintervals, for more than 20 intervals did not improve the predictive ability, and a calibration model was developed in each interval based on PLS regression. The ideal model of all subintervals should have higher r value and lower RMSECV, so the r and RMSECV value are used to evaluate the models in 20 intervals. Result of effective wavelength bands selection for SSC prediction were presented in Fig. 2. It showed that the intervals of 5, 6, 7, 11, 12 and 19 were effective intervals for the SSC calibration model by the higher r and the lower RMSECV. The effective wavelengths of the SSC calibration were about 681.36–740.51 nm, 798.60–836.19 nm and 945.52–962.75 nm. The intervals of 12, 17 and 19 were effective intervals for the TA calibration model. The effective wavelengths for the TA calibration model were 817.57–836.19 nm, 909.85–927.60 nm and 945.52–962.75 nm. Result of effective wavelength bands selection for TA prediction were presented in Fig. 3.

3.3. Nondestructive determination of SSC and TA

The relationships between the full-spectrum, effective wavelengths and the reference values of determination the SSC and TA were built, respectively. The performance of different calibration models used to predict the SSC and TA were showed in Table 2. The calibration model of the SSC was developed using effective wavelengths, which got much better results than full-spectrum. The range of 681.36–740.51 nm and 945.52–962.75 nm were the effective wavelengths, because the strong water absorbance bands were presented around 960 nm and 760 nm for the second and third harmonics of the fundamental O–H stretching vibration. The effective wavelengths were selected around 680 nm, because the absorbance band of chlorophyll was about 680 nm. And the effective wavelengths of 798.60–836.19 nm were selected, for 840 nm was an inflexion point because of the second harmonic of a combinational O–H stretching and bending vibration. The results of calibration and prediction models for the SSC were presented in Fig. 4(a) and (b), respectively. For the SSC calibration model, the correlation coefficients and root mean square errors of calibration

Table 2
Statistic results of calibration models for SSC and TA.

	LVs	Wavelength bands	r	RMSECV
SSC (°Brix)	6	681.36–701.10 nm	0.52	1.38
	4	710.30–720.89 nm	0.54	1.33
	5	721.08–740.51 nm	0.64	1.23
	5	798.60–817.57 nm	0.55	1.33
	4	817.57–836.19 nm	0.61	1.26
	4	945.52–962.75 nm	0.51	1.36
	14	681.36–740.51 nm 798.60–836.19 nm 945.52–962.75 nm	0.94	0.53
	16	600–980 nm	0.93	0.58
TA (%)	4	817.57–836.19 nm	0.42	0.11
	5	909.85–927.60 nm	0.44	0.11
	4	945.52–962.75 nm	0.45	0.11
	5	817.57–836.19 nm 909.85–927.60 nm 945.52–962.75 nm	0.65	0.10
	5	600–980 nm	0.65	0.10

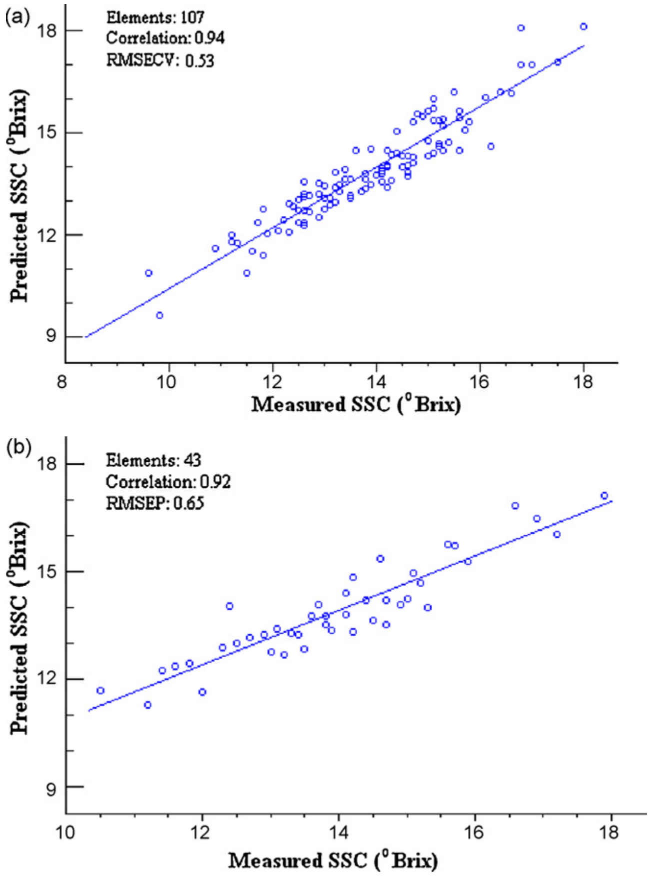

Fig. 4. (a) Results of SSC calibration model using effective wavelengths. (b) Results of SSC prediction mode using effective wavelengths.

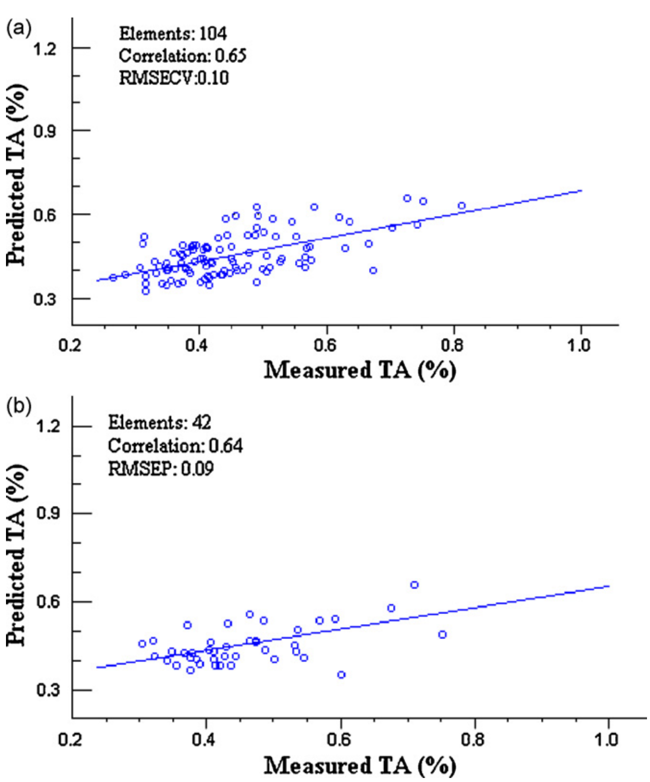

Fig. 5. (a) Results of TA calibration model using effective wavelengths. (b) Results of TA prediction mode using effective wavelengths.

were 0.94 and 0.53 °Brix, and the SSC prediction model were 0.92 and 0.65 °Brix.

The RMSEP of the SSC obtained in this research is lightly superior to those obtained by Camps and Christen (2009) with the RMSEP of 0.67–1.10 °Brix in apricot by a portable visible-near infrared spectroscopy and Liu et al. (2008) with the RMSEP of 0.66 °Brix in pear by a research instrument. But better results were reported in mango with the SEP of 0.40 °Brix by “Fruit Tester 20” (FANTEC, Kosai-city, Japan) and “Model 6500” (Foss NIRSystems, Silver Sping, USA) (Saranwong et al., 2003), in apple with standard errors of prediction (SEP) of 0.46 °Brix by FT-NIR spectrometer (Thermo Nicolet Corporation, USA) (Liu and Ying, 2005) and in mandarin fruit with the RMSEP of 0.33 °Brix by a research instrument (Gómez et al., 2006).

The effective wavelengths for prediction the TA were 817.57–836.19 nm, 909.85–927.60 nm and 945.52–962.75 nm by the iPLS regression (Fig. 3). It was slight superior to Gómez et al. (2006) in mandarin fruits and Cayuela (2008) in oranges. For the TA calibration model, the correlation coefficients and root mean square errors of calibration were 0.65 and 0.10%, and of prediction were 0.64 and 0.09%, respectively. The relatively poor results of the TA prediction by CCD-NIRS might well be due to the very narrow distribution of the quantitative data set (S.D. ~0.07%) compared with the reported NIR measurement precision of the RMSEP about 0.05% in apples (McGlone et al., 2002). The results of calibration and prediction models for TA were presented in Fig. 5(a) and (b) using effective wavelengths, respectively.

4. Conclusions

The feasibility of charge coupled device near infrared spectroscopy (CCD-NIRS) combining with effective wavelengths selection algorithm used to measure SSC and TA nondestructively was investigated. The effective wavelengths to SSC and TA were chosen by interval partial least squares (iPLS) in the wavelength range of 600–980 nm. The predictive ability of SSC model used PLS regression was improved with r of 0.92 and RMSEP of 0.65 °Brix using effective wavelengths of 681.36–740.51 nm, 798.60–836.19 nm and 945.52–962.75 nm. The TA model was simplified with r of 0.64 and RMSEP of 0.09% using effective wavelengths of 817.57–836.19 nm, 909.85–927.60 nm and 945.52–962.75 nm. The experimental results demonstrated that the CCD-NIRS technique combining with iPLS algorithm was a feasible method to measure SSC and TA of Nanfeng mandarin fruits nondestructively.

Conflict of interest

No conflict of interest.

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