Non-destructive measurement of fracturability and chewiness of apple by FT-NIRS

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Abstract In order to assess quickly and non-destructively the fracturability and chewiness of apple fruit by FT-NIR spectra in the wavelength range of 4000 cm^{-1} – 12000 cm^{-1} , multivariate models were built using multiple linear regression (MLR), partial least squares regression (PLSR), and principal component regression (PCR). Fracturability and chewiness reference data were instrumentally measured using a Texture Profile Analysis (TPA) test. The effects of various pre-processing methods of the spectroscopic data on the performance of the multivariate models were analyzed. Standard normal variate transformation (SNV), multiplicative scatter correction (MSC), Min-Max normalization (MMN) and first derivative (FD) were tested. The performance of the fracturability prediction models was better for the PLSR model ($R^2=0.91$, RMSEP=101.90) than for the PCR and MLR models. With regard to chewiness, the performance of the PCR model ($R^2=0.88$, RMSEP=13.46) was similar to the one of the PLSR model but better than the one of the MLR model. The results demonstrated that NIR spectra together with stoichiometry could determine precisely fracturability and chewiness of apple, and the predictive ability of the models developed by other methods may be improved in the future.

Keywords Apple · NIR · Texture · TPA test · Nondestructive

Introduction

Texture attributes such as crispness, crunchiness, hardness and juiciness are the main quality characteristic of apples because they are associated with freshness (Bechar et al. 2005; Mehinagic et al. 2004; Zdunek et al. 2010). Texture quality is often estimated by sensory analysis and instrumental measurements, and previous studies (Harker et al. 2002; Harker et al. 1997; Mehinagic et al. 2003; Mehinagic et al. 2004; Brookfield et al. 2011) have paid particular attention to correlations between sensory texture attributes and objective instrumental measurements; but those techniques are subjective, destructive and time-consuming (Mehinagic et al. 2003). In recent years, there is an increasing interest in the purchase of the rapid and non-destructive technology to evaluate internal quality attributes in fruit. Those technology included acoustic (Zdunek et al. 2010), near infrared spectroscopy (Liu et al. 2010a, b), magnetic resonance imaging (Blasco et al. 2009) and electricity (Sosa-Morales et al. 2009).

Near infrared spectroscopy (NIR) provides a rapid, non-destructive, little sample preparation, and good reproducibility method to measure the quality of food and agriculture products (Liu et al. 2010a; Liu et al. 2010b). Infrared spectroscopy has been widely used to evaluate the internal quality (SSC, pH, acidity and firmness et al.) of fruits such as apple (Bobelyn et al. 2010; Fan et al. 2009), pear (Paz et al. 2009; Sun et al. 2009), orange (Liu et al. 2010a; Liu et al. 2010b), grape (Cao et al. 2010), peach (Kurz et al. 2010; Ziosi et al. 2008), and kiwifruit (Clark et al. 2004; Moghimi et al. 2010) et al.. The first attempts to establish the

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relationship between sensory texture and NIRS measurements were made by Mehinagic et al. (2003) who reported good correlations between penetrometry and sensory texture but poor correlations between NIR and sensory data were found.

The purpose of the research was to evaluate the potential of FT-NIR spectrometric technique to measure Fuji apple, by investigating the connection between FT-NIR measurements, and fracturability and chewiness obtained by TPA test. Furthermore, the prediction ability of different spectral analysis models such as multiple linear regression (MLR), partial least squares regression (PLSR), principal component regression (PCR) was investigated. In addition, several different spectral pre-treatment types such as Min/Max normalization (MMN), Standard normal variate correction (SNV), Multiplicative scatter correction (MSC) and First derivative (FD) were also tested.

Materials and methods

Sample preparation Fuji apples were purchased in October, 2010 in Yongshou County, Baishui County, Yangling District of Shaanxi Province, China. Three hundred fruits free of mechanical damages, diseases and insect pest were stored in a cold storage chamber at 0 °C (± 1 °C) and 90–95 % RH.

FT-NIR spectroscopy measurements For six different storage periods (0d, 30d, 60d, 90d, 120d, 150d), 40 apples were kept in a room (temperature was 20 °C ± 1 °C) for 24 h, then three locations were marked every 120° around equatorial position. 240 samples were divided into the calibration set (204) and prediction set (36). The sample number of six storage periods was equivalent in the calibration and prediction sets. The selection function in OPUS software Version 5.5 which was provided by Bruker Optics was that

following the sequence of numbers, one was chosen as prediction sample at an interval of five numbers. For each period, 34 samples were put randomly in calibration set by the selection function of OPUS 5.5 and others were chosen in prediction set. FT-NIR spectra were acquired by a multi-purpose analyser (MPA) spectrometer (Bruker Optics, Wissembourg, France) equipped with a solid fiber of spectrum. The MPA was software-controlled by the OPUS 5.5. Each spectrum was obtained between 4000 cm^{-1} and 12000 cm^{-1} at 8 cm^{-1} spectral resolution with scanner velocity of 5 V and the average of 64 scans. For each apple, an average spectrum was calculated using three marked locations spectra.

Texture measurements Three cylindrical specimens were obtained from each apple using a stainless steel borer, nominally 0.5 cm in diameter. 0.5 cm away from the marked locations skin, each cylinder was trimmed to a length of 1 cm by a mechanically guided razor blade. For each apple, cylindrical specimens were taken from each marked location to the core of the fruit (Alvarez et al. 2002; Varela et al. 2005). TPA tests were performed using a Texture Analyser (TA.XT-plus Stable Micro System Ltd.Co.UK), equipped with a 5 cm diameter cylindrical stainless steel probe and a 5 kg load cell. TPA tests were done at 1 mm/s and were axially compressed to 40 % of their original height. Figure 1 shows TPA plot of cylindrical specimen of Fuji apple.

Texture attributes were calculated by the software of Texture Exponent 32 which was provided by Stable Micro System as follows: hardness is the second peak value if there are three peaks found on the TPA curve. Cohesiveness is computed as the ratio of the positive force area during the second compression portion to that during the first compression. Springiness is calculated as the ratio of the time from the start of the second area

Fig. 1 TPA plot for flesh of Fuji apples

Table 1 Summary statistics of Fuji apple fruit in calibration and prediction sets

Sample sets	Total samples	Parameter	Min	Max	Mean	Standard Deviation
Total samples	240	Fracturability	738.13	2546.53	1505.36	365.97
		Chewiness	55.18	277.40	142.65	46.94
Calibration set	205	Fracturability	738.13	2546.53	1535.08	326.95
		Chewiness	55.18	277.39	148.48	45.61
Prediction set	35	Fracturability	762.34	2331.27	1404.07	341.83
		Chewiness	56.52	237.04	121.94	44.90

up to the second probe reversal over the time between the start of the first area and the first probe reversal. Adhesiveness is the negative area between the point at which the first curve reaches a zero force value after the first compression and the start of the second curve. Fracturability is given as the first peak force only when there are three peaks. Resilience is taken as the ratio of areas from the first probe reversal point to the crossing of the x-axis and the area produced from the first compression cycle. Chewiness is estimated as hardness $\times$ cohesiveness $\times$ springiness (Alvarez et al. 2002). The average of texture attributes were computed for each apple.

Data analysis Spectra were pretreated using different pre-treatments in the OPUS 5.5. The higher R^2 of calibration models, the pre-treatment we chose. So MMN, SNV, MSC and FD were chosen to build different models respectively in the OPUS 5.5. Then, the pretreated spectrums were analyzed with MLR, PCR and PLSR for multivariate calibration in order to obtain linear correlations between the NIR values and the texture attributes. Models of MLR and PCR were developed in the Unscrambler v9.7 software (CAMO AS, Trondheim, Norway) and PLSR models were built in the OPUS 5.5. The quality of the calibration model was characterized by the coefficients of determination (R^2) between the predicted and measured parameters, the root mean square error of calibration (RMSEC), the root mean square error of prediction (RMSEP) and the root mean square error of cross-validation (RMSECV).

Results and discussion

Texture distributions An overview of fracturability and chewiness distributions of Fuji apple samples in the calibration and prediction sets was shown in Table 1. The fracturability and chewiness measurements of 240 samples were evenly distributed around the mean value (1505.36 g, 142.65 g), with standard deviation (SD) of 365.97 g and 46.94 g. In the calibration set, the range of the fracturability

was from 738.13 g to 2546.53 g and the range of the chewiness was from 55.18 g to 277.39 g. In the prediction set, the range of the fracturability was from 762.34 g to 2331.27 g and the range of the chewiness was from 56.53 g to 237.04 g. For each parameter, the range of prediction set was between the range of the calibration set, so calibration models were reliable and stable.

Spectra investigation Figure 2 shows the absorbance spectra of some Fuji apple fruit of different storage periods. The trends of spectra were quite similar for all storage periods. The wavelength range of 12000 cm^{-1} –4000 cm^{-1} was used to develop the calibration models. NIR method was sensitive to the concentrations of organic material, involving the response of molecular bonds (C-H, O-H and N-H), so it should be possible to relate fracturability and chewiness, which was related to firmness. According to Tu et al. (2009), firmness was associated with pectin, and pectin was an organic molecule and contained the bonds C-H and O-H. Thus, it was possible to use NIR method for determination of fracturability and chewiness in Fuji apples. From the fig. 2, there were absorption peaks at 4545 cm^{-1} , 7145 cm^{-1} , 8333 cm^{-1} and 8500 cm^{-1} et al., those peaks were associated with a combination of second overtone of bond C-H, first overtone of bond O-H and overtones of bond O-H in H_2O , respectively. While spectra had a very similar shape, absorbance had a large variability.

Spectral pre-processing analyses The NIR spectra are usually affected by irrelevant information such as baseline drift and noise. A mathematical pre-treatment such as MMN, MSC, second derivative (SD) and SNV is often required to remove them. Table 2 summarizes the prediction results of different calibration models which were built by PLSR in terms of R^2 , RMSEC and RMSECV for fracturability and chewiness. PLSR models were built using the automatic optimization function in the OPUS 5.5. The RMSEC, RMSECV and R^2 had large differences with different pretreatment methods and wavelengths. The better model of fracturability was developed using the SNV pre-treatment with a

Fig. 2 FT-NIR spectra of Fuji apple fruits: absorbance vs. wave number (cm^{-1})

wavelength range of $7502\text{ cm}^{-1} \sim 5446\text{ cm}^{-1}$, the coefficient of prediction (R^2), RMSEC and RMSECV were 0.91, 101.57 g and 111.47 g, respectively; the better model of chewiness with high R^2 of prediction (0.82), low RMSEC and RMSECV (15.16; 16.98) was obtained by PLSR+FD model based on wavelength from 6102 cm^{-1} to 5446 cm^{-1} .

MLR, PCR and PLS models The wavelength range of $7502\text{ cm}^{-1} \sim 5446\text{ cm}^{-1}$ was used to develop the fracturability models by PLSR, MLR and PCR with SNV preprocessing, separately. And the chewiness models were obtained by PLSR, MLR and PCR with FD pretreatment and the wavelength range of $6102\text{ cm}^{-1} \sim 5446\text{ cm}^{-1}$.

PLSR is one of the most popular methods to develop linear model at present. Owing to its simplicity and small volume of calculations, it is used to analyze data. After eliminating the unusual spectra and the abnormal value, the performance of the fracturability models was better with R^2 , RMSEC and RMSECV; the better model of chewiness

with high R^2 of correlation and prediction and low RMSEC and RMSECV was obtained by PLSR+FD.

MLR is the simplest method for model creation. Before developing the MLR model, the wavelengths were selected by regression coefficient. Considering that wavelength having higher absolute value contributed more to the model, the wavelengths which were at the valleys or peaks of the curve and had higher absolute value were selected. Figure 3 shows the regression coefficient plots obtained by PLSR. According to A(1, 2, 3) in Fig. 3, the wavelengths of $7502\text{ cm}^{-1} \sim 5446\text{ cm}^{-1}$ preprocessed by SNV were divided into 3 sections, $7502\text{ cm}^{-1} \sim 6734\text{ cm}^{-1}$, $6735\text{ cm}^{-1} \sim 5962\text{ cm}^{-1}$ and $5963\text{ cm}^{-1} \sim 5446\text{ cm}^{-1}$, respectively, and the following wavelengths were selected as effective wavelengths (EWs) for fracturability, 7371 cm^{-1} , 7367 cm^{-1} , 6704 cm^{-1} , 6700 cm^{-1} , 6696 cm^{-1} , 6511 cm^{-1} , 6507 cm^{-1} , 6503 cm^{-1} , 6102 cm^{-1} , 6098 cm^{-1} , 6075 cm^{-1} and 5982 cm^{-1} . After FD pretreatment, the wavelengths of $6102\text{ cm}^{-1} \sim 5446\text{ cm}^{-1}$ were divided into 2 sections,

Table 2 Performance of the calibration PLS models obtained for fracturability and chewiness

Parameter	Pretreatment method of Spectral	Wavelength (cm^{-1})	R^2	RMSEC/g	RMSECV/g
Fracturability	None	6102~5446	0.66	189.00	214.00
	MSC	7502~5446 4602~4247	0.75	172.00	195.00
	SNV	7502~5446	0.91	101.60	111.50
	FD	6102~4247	0.82	151.60	169.80
	MMN	6102~5446	0.76	166.00	205.00
Chewiness	None	6102~5446	0.64	26.90	27.20
	MSC	6102~5446	0.49	32.00	27.70
	SNV	6102~5446	0.52	31.00	27.10
	FD	6102~5446	0.82	15.20	17.00
	MMN	6476~5446	0.67	23.60	26.00

None Original spectrum; *MSC* Multiplicative Scatter Correction; *SNV* Standard Normal Variate Transformation; *FD* First Derivative; *MMN* Maximum Minimum Normalization

Fig. 3 Regression coefficients vs. wave number for fracturability (A1, A2, A3) and chewiness (B1, B2)

Fig. 3 (continued)

6102 cm^{-1} ~5666 cm^{-1} and 5666 cm^{-1} ~5446 cm^{-1} (Fig. 2 B(1 and 2)), and effective wavelengths of chewiness were selected as 5882 cm^{-1} , 5473 cm^{-1} , 5469 cm^{-1} , 5465 cm^{-1} , 5462 cm^{-1} , 5454 cm^{-1} , 5450 cm^{-1} and 5446 cm^{-1} . Then the MLR model was developed by the EWs. For comparison,

PCR models were developed too. Effect of models summarized in Table 3.

RMSEC, RMSECV, RMSEP and R^2 are often used for evaluating a NIR calibration model; a better model usually has higher R^2 , lower RMSEC, RMSECV and RMSEP, and the

Table 3 Results of calibration and test-validation of different models

Parameter	Methods	Calibration ($n=204$)		Test-validation ($n=36$)		RMSEC/RMSECV
		R^2	RMSEC/g	R^2	RMSECV/g	
Fracturability	MLR	0.89	116.61	0.83	144.15	0.81
	PCR	0.87	129.43	0.84	142.10	0.91
	PLS	0.92	101.57	0.90	111.47	0.91
Chewiness	MLR	0.94	10.03	0.64	24.27	0.41
	PCR	0.84	15.93	0.83	16.92	0.94
	PLS	0.86	15.16	0.82	16.98	0.89

MLR Multiple Linear Regression; PCR Principal Components Regression; PLS Partial Least Squares Regression

Fig. 4 Correlation between predicted values of optimized model and actual values of fracturability (a) and chewiness (b) in apple

values of RMSEC, RMSECV and RMSEP close to each other. Table 3 lists the R^2 , RMSEC, and RMSECV for all the calibration models of fracturability and chewiness. For the fracturability models, the PLS model was better than PCR and MLR models, and the result of MLR was superior to PCR. Better calibration model of fracturability was developed with respect to SNV in wavelength range of $7502\text{ cm}^{-1} \sim 5446\text{ cm}^{-1}$; As to the calibration models of chewiness, due to the fact that RMSEC/RMSECV of PCR model was higher than that of MLR and PLS, and the RMSEC/RMSECV of PCR model was close to 1, the PCR model of chewiness had satisfactory robustness; so the result of PCR was inferior to PLSR, and the models of PLSR and PCR were slightly

superior to MLR, the PCR calibration model of chewiness with high R^2 of correlation and prediction (0.84; 0.83) and low RMSEC and RMSECV (15.93; 16.92) was obtained by PCR+FD based on wavelength from 11995 cm^{-1} to 4247 cm^{-1} .

Predictive ability of the models The better models of fracturability and chewiness were obtained by PLS and PCR, and were used to predict the unknown samples. Figure 4 shows the correlation between predicted values using the better model and actual values of apples' fracturability and chewiness. Values between measured and predicted of fracturability or chewiness in Fig. 4 implies that all the points were distributed on the bisector, The R^2 and RMSEP of the model of fracturability were 0.91 and 101.97 g; R^2 and RMSEP of the model of chewiness were 0.88 and 13.46 g. The predictive ability of fracturability model using PLSR and chewiness model of applying PCR method was better than other methods. Nevertheless, Guifeng et al. (2008) had built the model of fracturability with higher R^2 (0.92) and lower RRMSEP (0.304 kg/cm^2), the prediction accuracy was slightly poorer in this study in comparison to Guifeng et al. (2008). Indeed, the prediction accuracy of texture models which were obtained by NIR may be influenced by the parameter of TPA, the ambient environment and measurement models (reflectance, interactance and transmission). The predictive ability of models of texture of apple will be studied in the future and improvements may be made with better control of influential factors and more precise reference methods.

Models of other textural parameters Table 4 shows correlation coefficients between textural parameters of apple tissue. Hardness and fracturability were positively correlated; cohesiveness, resilience and chewiness were significantly correlated, the highest correlation was found between cohesiveness and resilience, those findings could be due to the definition of the parameters; but adhesiveness and springiness were poor correlated with other parameters. According to the results predicted by models of fracturability and chewiness, values of hardness, cohesiveness and resilience can be obtained, and R^2 of calibration models of hardness, cohesiveness

Table 4 Correlation matrix between textural parameters of apple tissues

	Hardness	Fracturability	Adhesiveness	Springiness	Cohesiveness	Chewiness	Resilience
Hardness	1						
Fracturability	0.90	1					
Adhesiveness	-0.441	-0.40	1				
Springiness	-0.04	0.00	0.17	1			
Cohesiveness	0.42	0.30	-0.21	0.06	1		
Chewiness	0.78	0.68	-0.31	0.29	0.81	1	
Resilience	0.45	0.33	-0.24	-0.01	0.94	0.77	1

and resilience were between 0.83 and 0.88 developed by PLSR. However the effect of models of adhesiveness and springiness built by PLSR, PCR and MLR was very poor, so the unlinear regression methods such as ANN, spline and SVM and other preprocessed methods may be studied in future. Stability of NIR models of fruits is a hot topic in the lossless research field. In order to improve the stability, factors such as different varieties, plucking times, growth years, and fields could be considered to build models. In this study, samples, obtained in single year in Shaanxi Province, were only come from six different periods without enough number. So models may be poor in stabilization and improved could be made using more samples in different growth periods and different producing area.

Conclusions

The feasibility of FT-NIRS combining with the linear regression methods used to nondestructively measure texture of Fuji apple was investigated. The performance of the fracturability prediction models was better for the PLSR model ($R^2=0.91$, RMSEP=101.90) than for the PCR and MLR models. With regard to chewiness, the performance of the PCR model ($R^2=0.88$, RMSEP=13.46) was similar to the one of the PLSR model but better than the one of the MLR model. R^2 of calibration models of hardness, cohesiveness and resilience were between 0.83 and 0.88 developed by PLSR. However, the effect of models of adhesiveness and springiness built by PLSR, PCR and MLR was very poor. From these results it can be concluded that NIR spectroscopy combining with stoichiometry was a feasible method technique for nondestructively measuring texture of apple; and with better control of influential factors and more precise reference methods, the models of texture of Fuji apple would be improved.

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Conflict of interest None.

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