

# Comparison of Reflectance and Interactance Modes of Visible and Near-Infrared Spectroscopy for Predicting Persimmon Fruit Quality

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#### Abstract

The principal aim of this work was to compare the ability of measurement technique among interactance and reflectance in predicting soluble solids content (SSC), ascorbic acid (ASC) and firmness of intact persimmon fruit by using visible/short-wave near-infrared (Vis/SWNIR) spectroscopy. Calibration models were developed by partial least square (PLS) regression that related near-infrared (NIR) spectra to reference values. The root mean square errors of calibration (RMSEC), the root mean square errors of prediction (RMSEP), the correlation coefficients of calibration (Rcal) and prediction (Rval) and the ratio of performance to deviation (RPD) were used to consider the model accuracy. The PLS models from interactance showed satisfactory performance, providing better prediction results than reflectance technique in all parameters. ASC and firmness presented the best calibration models. ASC, Rval = 0.92, RMSEP = 5.56 (mg kg<sup>-1</sup> FW) and RPD = 2.54 were attained when using orthogonal signal correction (OSC) pretreatment. Firmness, Rval = 0.89, RMSEP = 4.21 N and RPD = 2.14 were also achieved using OSC pretreatment. These findings highlighted the potential of Vis/SWNIR spectroscopy with the multivariate calibration technique to be applied for evaluating ASC and firmness of fresh persimmon fruit.

Keywords Ascorbic acid · Chemometrics · Diospyros kaki · Firmness · Nondestructive measurement · Spectral pretreatment

# Introduction

Persimmon (Diospyros kaki Thunb.) is a widely cultivated crop worldwide (Yonemori et al. [2000\)](#page-9-0). Over the last few decades, the production of persimmon fruit has grown

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substantially because of the growing consumption of its fruit in Southeast Asian countries (Fahmy and Kohei [2016](#page-8-0)). Persimmon fruit can be categorized by the astringent taste at harvest time, also with their response to pollination and presence of seed. These cultivars are divided into four types as pollination constant astringent (PCA), pollination constant non-astringent (PCNA), pollination variant astringent (PVA) and pollination variant non-astringent (PVNA) (Yonemori et al. [2000\)](#page-9-0). Additionally, it is an edible fruit that contains primarily natural vitamin C sources (USDA [2016](#page-9-0)). The collected data suggests that consuming about 100–150 g of fresh persimmon fruit provides the daily recommended intake of vitamin C (Giordani et al. [2011\)](#page-8-0). Furthermore, the fruit is also abundant with carbohydrates, mainly fructose, glucose and sucrose (Candir et al. [2009](#page-8-0); Del Bubba et al. [2009;](#page-8-0) Veberic et al. [2010\)](#page-9-0). However, among the Diospyros species, Diospyros kaki is the most important from a nutritional point of view (Zheng et al. [2006](#page-9-0)).

Barrett et al. [\(2010\)](#page-8-0) claimed that the characteristics that impart distinctive quality consisted of four different attributes, including (1) colour and appearance, (2) flavour, (3) texture

and (4) nutritional value. The appearance is one of the critical parameters for determining the acceptability of each product by visual inspection. Meanwhile, other attributes, as aforementioned, are hidden characteristics that have become increasingly valued by consumers in nowadays, especially for the nutritional profile that appears to provide additional benefits for people with different health concerns. Therefore, since the demand for good-quality product is increasing, using efficient and effective ways for quality assessment is desirable for postharvest handling system.

Several spectral analysis techniques are widely implemented to evaluate the quality attributes of watermelon (Jie et al. [2013\)](#page-8-0), passion fruit (Oliveira et al. [2014;](#page-8-0) Maniwara et al. [2014](#page-8-0); Maniwara et al. [2019\)](#page-8-0) and tangerine fruit (Theanjumpol et al. [2019](#page-9-0)). However, near-infrared (NIR) spectroscopy represents one of the most broadly used techniques in food chemistry, which is recognized as a rapid, non-destructive, low-cost and robust analytical method (Nicolaï et al. [2007](#page-8-0); Lopez et al. [2013](#page-8-0); Vitale et al. [2013\)](#page-9-0). Cortés et al. [\(2017](#page-8-0)) examined the feasibility of Vis/NIRS for determining the astringency of intact and the flesh of persimmons. Their results showed that using partial least square regression (PLSR) gave a high correlation ( $R =$ 0.904), demonstrating the potential of this technique for astringency assessment of persimmon fruit. Wang et al. [\(2017\)](#page-9-0) generated the regression model for predicting SSC and firmness of European pears using portable Vis/NIR spectroscopy, and the results presented a more reliable of prediction, which was obtained by using the OSC pre-processing technique coupled with PLS regression model ( $R^2 = 0.87$ ). Nordey et al. ([2017\)](#page-8-0) assessed mango internal quality using PLSR along with variable selection and pre-processing of NIRS spectral data. Results revealed the importance of pre- and postharvest factors that could lead to NIRS model development and robustness in prediction. Maniwara et al. [\(2014](#page-8-0)) conducted postharvest quality investigation for passion fruit by using Vis/SWNIR. The best PLS model for predicting SSC was obtained from interactance measurement mode, and the developed model showed 0.923 of a correlation coefficient between measured and predicted values.

The objectives of this research were (i) to determine the ability of NIRS in predicting persimmon fruit quality and (ii) to compare the measurement techniques among interactance and reflectance.

# Materials and Methods

#### Sample Preparation

For this study, 124 persimmons (Diospyros kaki Thunb.) cv. Hiratanenashi, PVA-type cultivar, were purchased immediately after harvest from Niigata prefecture, Japan. The collection time was corresponding to late September and midOctober of 2016. Removal of astringency from persimmon fruit was achieved by exposing 'Hiratanenashi' persimmon fruit to 90–100% carbon dioxide (CO<sub>2</sub>) for 24 h at  $20 \pm 2$ °C. This treatment induced the anaerobic respiration, resulting in the accumulation of acetaldehyde, which precipitates the soluble tannin during the fumigation treatment. Thus, the astringency taste is eliminated (Matsuo et al. [1991](#page-8-0)). The sorted persimmon fruit was stored at ambient air temperature overnight (20  $\pm$  2 °C, RH 60–80%) before Vis-SWNIR spectral measurements were conducted on the next day. Morphological properties of persimmon samples, including diameter, height and weight, were recorded prior to each spectroscopic measurement. The fruit shape index was carried out by the aspect ratio (height to diameter ratio) to reflect the uniformity in size and shape. The diameter and height of persimmon fruit were calculated by digital calliper (SK-calliper, SK Niigata Seiki Co., Ltd., Niigata, Japan). The weight was measured by electronic balance (AND GX-600, A & D Co., Ltd., Tokyo, Japan).

#### Visible and Near-Infrared Spectra Acquisition

All Vis-SWNIR spectra (310–1100 nm with a 3.3 nm interval wavelength) were recorded as the logarithm of reciprocal absorbance (log1/R) by using a spectrophotometer (Handy Lambda II, Spectra Co., Ltd., Tokyo, Japan). The spectra were firstly collected in reflectance mode; the lamp used was 12-V/ 100-W tungsten halogen lamps (MCR 12-150 M). Thereafter, interactance spectra of each fruit were made by a portable fibre-optic probe module containing the light source (MHAA-100 W, Moritex Co., Ltd., Saitama, Japan). The orientation angle between light source and fibre-optic detector probe was set at a 45° angle and in direct contact with the fruit surface  $(0^{\circ}$  angle) for the reflectance (Fig. [4b](#page-7-0)) and interactance (Fig. [4a](#page-7-0)) measurement procedure, respectively. Each fruit was scanned perpendicularly at four sites along the equatorial positions. Each spectrum was accumulated from ten continuous acquisitions in the marked region. Thus, a total of 40 scans were automatically averaged for each fruit. The reference spectrum was conducted by using a white ceramic plate (1.5-mm thickness) every time after examining five fruits to recalibrate the NIR apparatus. The spectra were acquired and transformed by the Wave Viewer software (Spectra Co., Ltd., Tokyo, Japan) and the Unscrambler®X version 10.4 (CAMO software, Oslo, Norway), respectively. Fig. [1](#page-3-0) shows the average spectra (absorbance) of persimmon fruit from interactance (Fig. [1](#page-3-0)a) and reflectance (Fig. [1](#page-3-0)b) measurements.

#### Determination of Fruit Quality Parameter

After non-destructive measurements using spectroscopy technique had been completed, SSC, ASC and firmness of the

tested persimmon fruit were measured by conventional destructive methods from the same location where Vis-SWNIR spectroscopy had been carried out. Firmness values were derived from a standard flat-plate compression (diameter 2 mm) using a Digital Force Gauge (model: FGJN-50, Shimpo Co. Ltd., Tokyo, Japan). Flesh firmness measurement was carried out in kilograms and converted to newtons by multiplying by 9.81.

For chemical analysis, a circular wedge (20-mm diameter) from the measurement position of each fruit was manually squeezed through a cheese cloth  $(0.25 \text{ mm}^2 \text{ porosity})$  for filtration; then, its SSC determination was performed using a digital refractometer (PR-101ɑ, Brix 0.00–45%, Palette Series, Atago Co., Ltd., Tokyo, Japan). Analysis of ASC content was processed by mixing 1 mL of extracted fruit juice with 2% metaphosphoric acid solution and then dipping appropriate test strip into the mixture and subsequently measured for its ASC by using a digital reflectometer (RQflex 10, Merck & Co., Inc., Darmstadt, Germany) (Mijowska et al. [2016\)](#page-8-0). Lastly, NIRS calibration models were developed separately based on these reference parameters. The mean and standard deviation values were determined for each quality parameter.

#### Chemometric procedure

To generate a calibration model with high robustness, 97 intact persimmon fruits were used as a calibration set. For the remaining, 27 fruits were used for an external test set. Additionally, to avoid the selection bias, all samples for calibration and validation set were conducted by random sampling in the ratio 3:1. As it is widely known, NIR spectra may comprise undesired information, such as scatter and baseline shift. It is a usual practice to apply an appropriate spectral pretreatment in suppressing and facilitating all variations of calibration model (Palou et al. [2014\)](#page-8-0). Hence, five spectral preprocessing methods and their combinations were applied with the purpose of achieving reliable and accurate calibration models. These methods included Savitzky-Golay first (1- Der) and second (2-Der) derivatives, multiplicative scatter correction (MSC), extended multiplicative scatter correction (EMSC), orthogonal signal correction (OSC) and standard normal variate (SNV). All spectral pretreatment and regression models were carried out using the Unscrambler®X version 10.4 (CAMO software, Oslo, Norway).

Thereafter, predictive models were developed for the linear relationship between spectral data and reference value using PLS regression. Each model was tested internally via fullcross validation to determine complexity using the number of factors that presented the root-mean-square error in cross validation (RMSECV). Once optimized on the calibration set, the prediction test was applied to spectra of external validation samples to test their feasibility. Afterwards, the predictability of each constituent was explained by Rcal, Rval, number of factor and RMSEP. The effective and reliable model was defined by Rcal as 0.71 or higher, as described by Williams [\(2007\)](#page-9-0). In addition, the predictive deviation or RPD was calculated using the ratio of standard deviation (S.D.) to the RMSECV or the RMSEP. Meanwhile, higher values for RPD implied the increasing reliability and precision of the models. Ideally, in an excellent model, the RPD should be greater than 2. Moreover, the important variable (wavelengths) in PLS model analysed for each parameter is presented by loading (Fig. [3](#page-5-0)).

## Results and Discussion

## Physical and Chemical Quality of Persimmon Fruit

The quality pattern of the samples showed the uniform distribution of size and shape based on the standard size available in domestic and export market, with a mean of fruit index of 0.68 (Table 1S, supplemental material). The values, mean and standard deviation for the three determined parameters (SSC, ASC and firmness) of the persimmon fruit samples are shown in Table [1.](#page-3-0)

### PLS Models for Predicting SSC

The characteristics and statistics of the models, with each spectral pretreatment and different pre-processing combinations, are shown in Table 2S and 3S (supplemental material). The best results were obtained using EMSC + OSC and OSC + 2-Der models for interactance and reflectance measurement techniques, respectively. The prediction model showed the lowest RMSEP =  $0.58\%$  and the highest Rval =  $0.82$  using 6 factors, with the EMSC and OSC processing method (interactance). However, only a 1.73 RPD value was achieved from this model, which was defined by Chang et al. [\(2001](#page-8-0)) as part of the fair model category (RPD ranges from 1.4 to 2). Meanwhile, the OSC + 2-Der model (reflectance) provided the highest correlation coefficient (0.81) with the minimum SEP (0.58), which was obtained using four factors. The optimal number of factors for the PLS model, for SSC (Fig. [2a](#page-4-0) and [2b\)](#page-4-0), was determined by the lowest RMSECV, which substantially demonstrated the predictive abilities of the smallest RMSEP.

Dominant absorption peaks of the SSC parameter were mainly located in the visible region around 450–570 nm and 660–673 nm (Fig. [3](#page-5-0)a and [3b](#page-5-0)), where carotenoids and chlorophylls are responsible for fruit colour, related to a major carotenoids present in flesh and peel tissue of persimmon fruit (Ebert and Gross [1985](#page-8-0); Yuan et al. [2006](#page-9-0)). In the NIR region, the absorption band from the interactance mode showed another significant correlation loading around 980 nm, which

<span id="page-3-0"></span>



associated primarily with 80–90% of water content in freshly fruit (Beever and Hopkirk [1990\)](#page-8-0).

Regarding analysis of the targeted regression models, applying OSC obviously reduced the number of PLS components and provided substantial improvement compared with when there was no pre-processing. Wang et al.  $(2017)$ , when studying a hybrid method for predicting SSC and firmness of European pear, obtained results showing that OSC-PLS models improved the predictability of the model, especially for the SSC parameter. Moreover, the performance of the OSC-PLS also presented higher RPD and greater coefficients of determination. Interesting results were also obtained for predicting quality parameters of straw wine; the combination between OSC and PLS regression demonstrated the improvement of the performance of regression model by reducing the model complexity and prediction error and also improvement of the accuracy of model (Croce et al. [2020\)](#page-8-0).

The validation results in Table [2](#page-5-0) showed that the developed prediction model of SSC was not completely successful for both measurement modes. Considering RPD values of PLS regression for estimation of SSC content, the display of an RPD value less than 2 generally

Table 1 Summary statistics of persimmon fruit in calibration and prediction sets



n number of samples, Min minimum, Max maximum, S.D. standard deviation, S.E. standard error, SSC soluble solids content, ASC ascorbic acid

<span id="page-4-0"></span>

Fig. 2 Changes of RMSECV and RMSEP, with the number of effective wavelengths included in PLSR analysis of SSC, ASC and firmness modelling. Results from interactance are shown in a, c and e, while b, d and f are derived from reflectance mode measurement

indicated the influence of disturbance, which caused less accurate prediction. Above this point, it could be the interference of soluble tannins, which naturally presented in the astringent persimmon variety. Noypital et al. ([2015\)](#page-8-0) observed astringency and tannin content in 'Xichu' persimmons using near-infrared spectroscopy and found that it was the accumulation of acetaldehyde under  $CO<sub>2</sub>$  treatment of this type of fruit leading to the transformation of soluble tannin into insoluble form. Meanwhile, after deastringent treatment, the flesh near the skin still presented higher content of the soluble tannin than in the flesh near the core of fruit, which could be caused by the interferences of light scattering effects, especially with the reflectance mode. From this experiment, the remaining soluble tannin also possibly generated the errors of value determination by reflectometer.

<span id="page-5-0"></span>

Fig. 3 Correlation loading of SSC, ASC and firmness modelling using PLSR analysis. Results from interactance are shown in a, c and e, while b, d and f are derived from reflectance mode measurement

## PLS Models for Predicting Vitamin C (Ascorbic Acid)

The calibration models were developed in the range of Vis-SWNIR spectra (310–1100 nm). The calibration and prediction results after applying the spectral pre-processing method are shown in Table 2S and 3S (supplemental material) for interactance and reflectance measurement mode, respectively. For interactance measurement mode, the RMSECV values





for ASC ranged between 8.30 and 9.17 (mg  $kg^{-1}FW$ ), and the RMSEP values were in the range of  $5.54-7.45$  (mg kg<sup>-1</sup>FW). Meanwhile, the correlation coefficients of prediction set ranged from 0.83–0.92 for all pretreatments. From Table [2](#page-5-0), it can be inferred that OSC decreased the number of factors needed for modelling while providing the highest precision of prediction model with  $RMSEP = 5.56$  and  $RPD = 2.54$ , which ensured the accuracy and robustness of the model for the prediction of ASC in persimmon fruit. As mentioned earlier, the OSC acts as a filter by altering the spectrum in order to remove undesirable systematic variations (Yee and George [2003\)](#page-9-0). At the same time, after observing predictive analysis of PLS-ASC by reflectance mode, the model was clearly considered less robust (Rpre =  $0.84$ , RPD = 1.84) than using interactance one  $(Rpre = 0.92, RPD = 2.54).$ 

The correlation loading plots of the best fitting model established, using interactance and reflectance measurement modes of ASC, are shown in Fig. [3c](#page-5-0) and [3d,](#page-5-0) respectively. Considering these two diagrams, high correlation loading refers to a high influence wavelength for prediction. There were four optimum wavelengths (513, 548, 673 and 970 nm) identified for the prediction by interactance mode (Fig. [3c](#page-5-0)). Wavelengths 513 nm and 548 nm are related to peel and flesh colour of persimmon fruit, since it corresponds to orange-red colour. However, the band 673 nm could be linked with the chlorophyll absorption band. Meanwhile, an absorption peak observed around 980 nm could be assigned to hydroxyl (OH) vibration absorption. At the same time, the chemical structure of ascorbic acid also contains four hydroxyl groups in its typical structural formula (Liu et al. [2006\)](#page-8-0) (Fig. [4\)](#page-7-0).

Regarding the regression model for ascorbic acid content estimation of acerola fruit using PLS regression algorithms conducted by Malegori et al. [\(2017](#page-8-0)), the study obtained less predictive accuracy despite good calibration for both analytical devices (Micro-NIR and FT-NIR). Considering these results, there were several factors that might lead to the uncertainty in the prediction results, such as variation of sample characteristics, calibrating method and spectral quality. However, it is very interesting to apply a variety of pre-processing algorithms in order to handle spectral information appropriately and to approach better predictive modelling. In the present study, the robust efficiency of spectra in diffuse reflectance mode may be affected by the light scattering phenomenon during the spectra acquisition (Frizon et al. [2015\)](#page-8-0), which could be influenced by the spherical shape and shiny skin of persimmon fruit. Munera et al. [\(2017\)](#page-8-0) described the persimmon appearance as wrinkly, with less shine in its skin during the ripening process, clearly affecting the reflectance properties. Additionally, Schaare and Frazer [\(2000\)](#page-8-0) used difference optical detection techniques to measure the chemical and physical properties (such as SSC, density and flesh colour) of kiwifruit. They claimed that the spectral acquisition by the interactance measurement technique provided the greatest prediction results, preferable to the reflectance spectra. This incident could be related to the less dominant effect of its peel colour on spectra while using interactance measurement mode.

#### PLS Models for Predicting Firmness

As seen in Table 2S and 3S (supplemental material), in general, the interactance PLS model for firmness prediction of persimmon fruit showed RMSEP values ranging between 3.97 and 5.24 N, which was lower than the result from PLS reflectance at 5.80–8.05 N. Meanwhile, in analysing the results obtained from each model of interactance technique (Table 2S, supplemental material), there were only two preprocessing methods which presented an acceptable RPD value (greater than 2.0); these were 2-Der and OSC (RPD =  $2.02$  and 2.14, respectively). Besides, raw spectrum (without spectra pre-processed) provided the highest RPD value at 2.26, with 10 factors used.

For the firmness prediction results of reflectance measurement mode (Table 3S, supplemental material), the analysis showed poorer predictions than those obtained with interactance. Regarding the RPD values, it was not possible to build a reliable calibration model. For qualitative data analysis as calibration modes, RPD values < 1.5 are considered inadequate for most application. The negative result in cases of smaller RPD values can be caused by a narrow range of reference values (small S.D.) or from a large error in prediction results (RMSECV), compared with the reference values variation (Shah et al. [2010;](#page-9-0) Cozzolino et al. [2011\)](#page-8-0). In this study, the RPD values which were achieved for reflectance were lower than 2.0 for all PLS models. Reflectance measurement also showed notably lower robustness (Rpre  $= 0.75$ , RMSEP =  $5.94$  N) than the prediction performed by interactance (Rpre =  $0.89$ , RMSEP =  $4.21$  N).

Loadings for prediction model particularly presented dominant absorption bands in the visible region of 400– 700 nm. It is well known that the absorption of carotenoid and chlorophyll take place in this region, which is mainly related to peel and flesh colour of persimmon fruit. As maturity increases, the external colour of persimmon fruit changes from yellow-orange to orange-red. However, the ripeness process of persimmon fruit was often accompanied by texture changing, which influenced the firmness parameter. A previous study (Huang et al. [2018](#page-8-0)) reported high correlation of tomato firmness with the wavelength in the visible region, which was related to chlorophyll and anthocyanin pigment. The absorption peak observed around 800–1100 nm could be related to the water absorption peak, which may be related to water content increases in the flesh as maturity increases.

However, the application of OSC produced good calibration and prediction results, where the number of factors was reduced considerably compared with the <span id="page-7-0"></span>Fig. 4 Schematic representations of interactance (a) and reflectance

(b) acquisition modes



regression model generated from the raw spectra. Palou et al. [\(2014\)](#page-8-0) discovered the same interference, as the filtered spectral data by OSC did not provide significantly better results of industrial paraffin and oil samples, according to the prediction error (RMSEP) and bias expanded to some extent. Firmness is a physical parameter that indirectly depends on several factors in persimmon fruit. Changes in physiological maturity, as well as the varying of water and sugar content, can cause fruit firmness changes and affect fruit absorption and scattering properties. Therefore, filtering spectral data had varying effects on PLS model predictions. However, an applied pre-processing technique can typically cause the loss of information for prediction; thereby, high RMSEP is obtained.

As mentioned above, there are many factors which influence the firmness parameter of persimmon fruit. The firmness parameter mainly measures the structural properties of samples, and an applied pre-processing technique may result in loss of useful information if a proper method is not selected or correctly applied. This might cause lower predictions of the firmness parameter after applying a different pre-processing method.

## Conclusion

In this research, Vis/SWNIRS in interactance and reflectance measurement techniques were studied as potential non-destructive methods to evaluate postharvest quality of persimmon fruit in terms of SSC, ASC and firmness. The results showed that PLS prediction models developed from interactance measurement mode provided the best predictive model for the ASC and firmness, but not the SSC. On the other hand, the PLS model from reflectance technique could not accurately estimate all parameters. This might have been caused by the structural characteristics of fruit, which in turn would affect light scattering. In addition, the use of spectral pre-processing can help minimize unwanted variation. Meanwhile, the other spectral pre-processing and modelling methods should also be considered in order to further improve prediction accuracy. These would be useful for the improvement of postharvest handling of persimmon fruit.

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#### <span id="page-8-0"></span>Compliance with Ethical Standards

Conflict of Interest The authors declare that they have no conflict of interest.

Ethical Approval This article does not contain any studies with human participants performed by any authors.

Informed Consent Not applicable.

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