Determination of Water Content Of Intact Sapodilla Using Near Infrared Spectroscopy

Kusumiyati\(^{1*}\), Yuda Hadiwijaya\(^{2}\), Ine Elisa Putri\(^{2}\)

\(^{1}\)Lecturer of Agronomy Studies Program, Agriculture Faculty, Padjadjaran University
\(^{2}\)Alumni of Agronomy Studies Program, Agriculture Faculty, Padjadjaran University

*Corresponding Author: kusumiyati@unpad.ac.id

Abstract. Water content is one of components that affects the fruit firmness of sapodilla. Fruit firmness is used as an indicator of physical fruit quality, which determines consumer acceptance. Generally, determination of water content is done by drying the fruit itself. However, this method damages the fruit, therefore needed a non-destructive method which able to determine the water content of sapodilla. Non-destructive method is also very useful to reduce physical damage to fruit (bruses) caused by the touch of the consumers hands to assess the fruit firmness. Near infrared spectroscopy is a non-destructive measurement that has been widely used to determine various components of fruit quality, including water content. The purpose of this study was to determine water content of intact sapodilla non-destructively using near infrared spectroscopy. This research used experimental method with multivariate data analysis by modelling, continued by data processing using ISIS software ( Integrated Spectronics Pty, Ltd, Australia and Unscrambler multivariate data software (version 9.7, CAMO, Oslo, Norway). Further research resulted value has the same accuracy as the value of non-destructive measurements. The result showed that near infrared spectroscopy was able to determine water content of intact sapodilla non-destructively.

1. Introduction
Most sapodilla fruit farmers determines the appropriate time to harvest by visual estimation such as size and skin color. Additionally, it needs laboratory analysis to obtain the internal quality of fruits. Water content of fruit is an important parameter to assess the quality of fruit, because it is related to the shelf life and also the texture of the fruit. The ripe sapodilla contains 72 to 78% of water content and total soluble solids ranged from 12 to 18 °Brix [1]. Composition of ripe sapodilla fruit is presented on Table 1.
Near infrared spectroscopy (NIR) is a non-destructive method to predict fruit quality. It has been used to predict a lot of fruits quality since 1990s [2]. This technique is time and labor reducing alternatives. Blanco and Villarroya [3] studied that near infrared spectroscopy offers a rapid and accurate measurement for fresh commodities. Spectrometer records spectra data consist of physical and chemical content such as total soluble solids, firmness and color [4]. The non-destructive method using NIR has been previously performed to measure the fruit quality of mango [5], Jujube [6;7] with high accuracy. NIR also has been used for predicting soluble solid content, moisture content and hue color of on tree and after harvesting tomato [8] firmness, color and lycopene content of tomato [8] and chlorophyll of bitter gourd [9]. Ranganna [10] studied that once separated from the plant, fruits continue to respire until senescence phase because the harvested fruit can not access water from the plant anymore for the ripening process. Respiration determines the potential storage life of fruits [11]. Hence, this research objective aimed to focus on determining of internal quality of intact sapodilla during storage using near-infrared spectrometer.

Table 1. Composition of ripe sapodilla fruit of 100 g of eatable part [12]

| Constituents     | Amount     |
|------------------|------------|
| Water content (%)| 73.37      |
| Protein          | 0.70 g     |
| Fat              | 1.10 g     |
| Mineral          | 0.05 g     |
| Fiber            | 2.60 g     |
| Carbohydrates    | 21.40 g    |
| Energy           | 98.00 cal  |
| Phosphorous      | 72.00 mg   |
| Iron             | 1.25 mg    |
| Calcium          | 28.00 mg   |
| Vitamin B1       | 0.02 mg    |
| Vitamin B2       | 0.03 mg    |
| Carotene         | 97.00 mg   |
| Vitamin C        | 0.06 mg    |

2. The Material And Method
2.1. Sample Collective
225 Sapodillas were all harvested from the orchard then stored for 0 day (H₀), 5 days (H₅), and 10 days (H₁₀). All samples were numbered before further analysis.

2.2. Spectral Data Acquirement
NirVana AG410 spectrometer with wavelength range of 312-1050 nm was used to measure the six separate measurements with three points on each side of fruits distributed along axial region (Figure 1.).
2.3. Reference Data Acquisition

Water content was calculated by the mass difference between the fresh fruit and the dried fruit. Sliced sapodilla were put into aluminum foil. Then the sample was dried using an oven until it reached a constant weight. The results were described as percentage of water content. The calculation of water content is described in the following equation:

\[
\text{Water content (\%)} = \left(1 - \frac{W_a}{W}\right) \times 100\%
\]

where:

- \( W \) : fresh fruit (g)
- \( W_a \) : dry fruit (g)

The method used in this research was multivariate data analysis. Then it continued by data processing using ISIS and The Unscrambler 9.7.

2.4. Multivariate Analysis

The calibration model was built using the partial least square (PLS) method. PLS is a chemometric technique that could analyze a large of hidden information in spectral data. It is used to evaluate a series of dependent variables (response) from immense number of independent variables (predictors).

The acceptable model is implied by \( R_{cal} \) of 0.71 or above as explained by Williams [13] that this value indicates rough screening ability, and is preferable for further prediction. Then, the acceptable calibration model was used to predict another sapodilla fruits (prediction set) in order to verify the prediction capability. The model was evaluated using coefficient of determination (\( R^2 \)) which explains the capability of independent variables explaining variations of dependent variables.

Root mean square errors of calibration (RMSEC) is the difference in values between predicted and reference value of calibration set. Root mean square errors of prediction (RMSEP) is the difference in values between predicted and reference value of prediction set.

Nicolaï et al. [14] advised that an effective model should present a high \( R^2 \) value of prediction set and low value of errors in calibration set and prediction set, either. Nevertheless both error values should not have a high difference from each other. The calculations of RMSEC and RMSEP are described in the following equations [15]:

![Figure 1. Irradiated points of fruits on each side](image-url)
\[ \text{RMSEC} = \sqrt{\frac{1}{n_c} \sum_{i=1}^{n_c} (\hat{y}_i - y_i)^2} \]

\[ \text{RMSEP} = \sqrt{\frac{1}{n_p} \sum_{i=1}^{n_p} (\hat{y}_i - y_i)^2} \]

where:
\( \hat{y}_i \) : predicted value,
\( y_i \) : measured value,
\( n_c \) : amount of samples in calibration set,
\( n_p \) : amount of samples in prediction set.

3. Result And Discussion

3.1. Destructive Data Analysis of Sapodilla

Water content analysis by destructive measurement was used as reference data for the development of calibration model. Any kind of measurement surely produce errors, as well as the destructive measurements. Hence, to diminish the errors, it was performed special handling to the samples. Sapodilla samples that have been irradiated by near infrared spectrometer, then as quickly as possible should be measured of it's water content at the laboratory. This is necessary to avoid the changes of internal chemical content of sapodilla. Reference data measurement of sapodilla samples are described on Table 2.

| Water Content | H0   | H5   | H10  |
|---------------|------|------|------|
| Minimum       | 67%  | 64%  | 63%  |
| Maximum       | 74%  | 77%  | 74%  |
| Mean          | 70%  | 72%  | 70%  |
| Standard Deviation | 1.5% | 2.8% | 2.3% |

The lowest and highest value of water content are found in H10 as 63% and H5 as 77% respectively. Highest mean value was found in H5 as 72% and the lowest mean value are found in H0 and H10 as 70%.

Standard deviation indicates the diversity of amount of data being investigated. Largest variation of data was found in H5 as 2.8% and the lowest data variation in H0 value as 1.5%.

3.2. Near-Infrared Data Analysis of Sapodilla

Data analysis of water content was measured by NIR second derivative data using multivariate data analysis as partial least square (PLS). Total samples used was 225 fruits. Each fruit was irradiated by 6 points as illustrated in Figure 1.

3.3. Water Content Assessment

In calibration set the assessment of water content used 150 samples and 75 samples for prediction set with wavelength 312-1050 nm. Lowest to highest data range in calibration set starts from 63% to 77%, while in the prediction stage ranges from 64% to 76%. The mean value of calibration was 71% and 70% for prediction. The standard deviation values of calibration was 2% and prediction was 1%,
respectively. Statistical data of sapodilla used in calibration and prediction are presented on Table 3. The calibration model of water content is showed on Figure 1. Prediction model of water content is showed on Figure 2.

### Table 3. Statistical Data of Sapodilla Samples Used In Calibration and Prediction Set.

| Set          | n  | Range      | Mean | Standard Deviation |
|--------------|----|------------|------|--------------------|
| Calibration  | 150| 63% – 77%  | 71%  | 2%                 |
| Prediction   | 75 | 64% – 76%  | 70%  | 1%                 |

Coefficient of determination of calibration set ($R^2_{cald}$) for assessment of water content of sapodilla was 0.71 means high (closed to 1). This defined the NIR prediction for water content was close to the reference measurement by using destructive analysis. RMSEC of calibration set generated was 0.01. Considering the RMSEC of calibration set, it showed the calibration model was built well due to the standard error value close to zero (0). It is required to perform the prediction set to check the accuracy of calibration model.

The prediction set of water content of sapodilla used different samples from calibration set which 75 samples from second derivate absorbance data (d2a) NIR and destructive measurement. The purpose of using a different samples was to test the capability of calibration model for predicting water content of new sapodilla fruit samples. Afterwards the calibration model would be reliable for predicting new samples. Coefficient of determination of prediction set ($R^2_{pred}$) of water content was 0.65. The accuracy level of prediction set was determined RMSEP value. It showed that the RMSEP generated was 0.01. The RMSEP was acceptable due to close to zero. Reliability of the calibration model could be examined by how close the value of coefficient of determination of the calibration set and the prediction set. The result explained the coefficient of determination and the error value in the calibration set and the prediction set were quite close. Hence calibration model that was built could be assumed to be reliable for predicting water content of sapodilla fruit.

![Figure 2. Calibration set of water content](image-url)
Figure 3. Prediction set of water content
4. Conclusion
Determination of intact sapodilla during storage using near-infrared spectrometer was able to measure water content value with good accuracy.

5. References
[1] Ganjyal G M, Hanna M A and Devadattam D S K 2005 Processing of Sapota (Sapodilla): Powdering J. Food Technol. 3 326-330
[2] Kawano S 1998 New application of nondestructive methods for quality evaluation of fruit and vegetables in Japan J. Jpn. Soc. Horticult. Sci. 67 1176–1179
[3] Blanco M and Villarroya I 2002 NIR spectroscopy: a rapid-response analytical tool Trends Anal. Chem. 21 240–250
[4] Kusumiyati, Mubarok S, Hamdani J S, Farida, Sutari W, Hadiwijaya Y, Putri I E and Mutiarawati T 2018 Evaluation of sapodilla fruit quality using near infrared spectroscopy J. Food Agric. Envi. 16 49-53
[5] Saranwong S, Sornsrivichai J and Kawano S 2004 Prediction of ripe-stage eating quality of mango fruit from its harvest quality measured nondestructively by near infrared spectroscopy Postharvest Biol. Technol. 31 137–145
[6] Wang J, Nakano K and Ohashi S 2011a Nondestructive evaluation of jujube quality by visible and near-infrared spectroscopy LWT-Food Sci. Technol. 44 1119–1125
[7] Wang J, Nakano K and Ohashi S 2011b Nondestructive detection of internal insect infestation in jujubes using visible and near-infrared spectroscopy. Postharvest Biol. Technol. 59 272–279
[8] Kusumiyati, Akinaga T, Yonemori S, Kawasaki S and Tanabe T 2007 Evaluation of Tomato Quality on Tree and after Harvesting using portable NIR Spectroscopy J. Soc. Agric Structures Japan (Nogyo Shisetsu) 38 117-126
[9] Kusumiyati, Akinaga T, Yonemori S, Shikanai T, Okamoto H, Tanabe T and Kawasaki S 2008b Internal quality sensor of fruit jagged surface: preliminary study of bitter gourd (Momordica charantia Linn) Acta Hort. 768 391-398
[10] Ranganna S 2008 Handbook of Analysis and Quality Control for Fruit and Vegetable Products McGraw-Hill Publishing Company Limited, New Delhi p 444
[11] Varoquaux P and Sani O I 2005 Packaging and produce degradation, In: Lamikanra O, Imam S, Ukuku D (Eds.), Produce Degradation: Pathways and Prevention. CRC Press pp 117–153
[12] Swaminathan M 1979 Food Science and Experimental Foods. Madras, India: Ganesh and Co pp 45-63
[13] Williams P 2007 Application to Agricultural and Marine Products, In: Ozaki Y, McClure W F, Christy A A (Eds.), Near-Infrared Spectroscopy in Food Science and Technology, John Wiley & Sons, Inc. Publication, New Jersey pp 165–218
[14] Nicolaï B M, Beullens K, Bobelyn E, Peirs A, Saeyes W, Theron K I and Lammertyn J 2007 Nondestructive measurement of fruit and vegetable quality by means of NIR spectroscopy: a review Postharvest Biol. Technol. 46 99-118
[15] Yahaya O K M, MatJafri M Z, Aziz A A and Omar A F 2015 Visible spectroscopy calibration transfer model in determining pH of Sala mangoes J. Instrum. 10 T05002