

DETERMINATION OF MOISTURE CONTENT AND SUGAR CONTENT IN FLESH OF DRIED WHOLE LONGAN USING NEAR-INFRARED SPECTROSCOPY AND HYPERSPECTRAL IMAGING TECHNIQUES



A Thesis Submitted in Partial Fulfillment of the Requirements for the Degree Master of Science Program in Food Technology Department of Food Technology Graduate School, Silpakorn University Academic Year 2015 Copyright of Graduate School, Silpakorn University DETERMINATION OF MOISTURE CONTENT AND SUGAR CONTENT IN FLESH OF DRIED WHOLE LONGAN USING NEAR-INFRARED SPECTROSCOPY AND HYPERSPECTRAL IMAGING TECHNIQUES



A Thesis Submitted in Partial Fulfillment of the Requirements for the Degree Master of Science Program in Food Technology Department of Food Technology Graduate School, Silpakorn University Academic Year 2015 Copyright of Graduate School, Silpakorn University การใช้เทคนิคสเปกโทรสโกปีอินฟราเรดย่านใกล้และการถ่ายภาพสเปกตรัมในการติดตาม ปริมาณความชื้นและปริมาณน้ำตาลในเนื้อลำไยอบแห้งทั้งผล



วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต สาขาวิชาเทคโนโลยีอาหาร ภาควิชาเทคโนโลยีอาหาร บัณฑิตวิทยาลัย มหาวิทยาลัยศิลปากร ปีการศึกษา 2558 ลิขสิทธิ์ของบัณฑิตวิทยาลัย มหาวิทยาลัยศิลปากร The Graduate School, Silpakorn University has approved and accredited the Thesis title of "Determination of moisture content and sugar content in flesh of dried whole longan using near-infrared spectroscopy and hyperspectral imaging techniques" submitted by Miss Aleeya Lerdkuson as a partial fulfillment of the requirements for the degree of Master of Science in Food Technology

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The objective of this study was to use near-infrared spectroscopy (NIRS) and hyperspectral imaging (HSI) as rapid and non-destructive techniques to analyze moisture and sugar contents in flesh of dried whole longan fruits. Total of 496 whole longans for moisture content analysis and the total of 186 samples for sugar content analysis with different moisture content of flesh ranged between 9.99-88.62% were taken for spectrum acquisition using an FT-NIR spectrometer (800 - 2500 nm) in reflectance mode with a nominal resolution of 16 cm⁻¹ and scan time of 32 at four different positions (calyx, bottom and the both cheeks of fruit). After that, the samples were imaged by reflectance HSI at the wavelength of 400 - 1000 nm. The image was captured 1 position with the equator position facing toward the lens. Then sample was examined for moisture contents of peel, flesh, seed and whole fruit, total soluble solids (TSS) and sucrose, glucose, fructose and total sugar contents. Calibration and validation models of both techniques were built using a partial least square regression analysis. It was found that averaging spectra of all positions of dried whole longan by NIRS was suitable for determining the moisture content of flesh, TSS, sucrose, fructose and total sugar content that provided the coefficient of determination (R²) of 0.9513, 0.9219, 0.8343, 0.8158 and 0.9082, respectively and gave the root square error of prediction (RMSEP) of 5.53%, 6.52 ^oBrix, 53.8 mg/g fresh weight, 13.9 mg/g fresh weight and 51.9 mg/g fresh weight, respectively. While, the HSI could not be used for determining moisture content of flesh, TSS, sucrose, glucose, fructose and total sugar content due to low R^2 of 0.7071, 0.6676, 0.5247, 0.2699, 0.4944 and 0.5333, respectively and high RMSEP of 13.60%, 13.36 ^oBrix, 90.93 mg/g fresh weight, 24.03 mg/g fresh weight, 23.86 mg/g fresh weight and 115.42 mg/g fresh weight, respectively.

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คำสำคัญ : ปริมาณความชื้น/ปริมาณน้ำตาล/ลำไยอบแห้งทั้งผล/เทคนิคสเปกโทรสโกปีอินฟราเรดย่าน ใกล้/เทคนิคการถ่ายภาพสเปกตรัม

เอลียาห์ เลิศกุศล : การใช้เทคนิคสเปกโทรสโกปีอินฟราเรคย่านใกล้และการถ่ายภาพ สเปกตรัมในการติดตามปริมาณความชื้นและปริมาณน้ำตาลในเนื้อลำไยอบแห้งทั้งผล. อาจารย์ที่ปรึกษาวิทยานิพนธ์ : ผศ.ดร.บุศรากรณ์ มหาโยธี. 164 หน้า.

้งานวิจัยนี้มีวัตถุประสงค์เพื่อนำเทคนิคสเปกโทรสโกปีอินฟราเรคย่านใกล้ (NIRS) และ การถ่ายภาพสเปกตรัม (HSI) ซึ่งเป็นวิธีการที่รวดเร็ว และไม่ทำลายตัวอย่างมาประยกต์ใช้ในการ ตรวจสอบปริมาณความชื้นและปริมาณน้ำตาลในเนื้อลำไขอบแห้งทั้งผล โดยจะทำการวัดสเปกตรัม ของตัวอย่างถำไยสดและอบแห้งสำหรับการวิเคราะห์ปริมาณความชื้น 496 ผล และการวิเคราะห์ ้ปริมาณน้ำตาล 186 ผล ในตำแหน่งที่ต่างกัน ได้แก่ ขั้ว กัน และด้านข้างทั้งสองข้างของลำไย ด้วย เครื่อง FT-NIR (800-2500 นาโนเมตร) จากนั้นทำการถ่ายภาพสเปกตรัมด้วยกล้องถ่ายภาพสเปกตรัม (400-1000 นาโนเมตร) 1 ภาพต่อผล โดยให้ด้านกว้างของลำไยตรงกับเลนส์กล้อง สำหรับการสร้าง สมการทำนายปริมาณความชื้นในส่วนเปลือก เนื้อ เมล็ค และทั้งผล ปริมาณของแข็งทั้งหมดที่ละลาย ใด้ (TSS) และปริมาณซูโครส กลูโคส และฟรุกโตสของเนื้อลำไยอบแห้ง สร้างสมการทำนายโคยใช้ วิธีการถคถอยกำลังสองน้อยที่สุดบางส่วน (partial least squares, PLS) จากการศึกษาพบว่าการเฉลี่ย เส้นสเปกตรัมที่วัดจากทกตำแหน่งของลำไยอบแห้งทั้งผลด้วยเทคนิค NIRS มีความเหมาะสมในการ นำมาตรวจสอบปริมาณความชื้น TSS ปริมาณซูโครส ฟรุกโตส และปริมาณน้ำตาลทั้งหมดได้ โดยให้ ค่าสัมประสิทธ์การตัดสินใจ (coefficient of determination, R²) เท่ากับ 0.9513, 0.9219, 0.8343, 0.8158 และ 0.9082 ตามลำคับ และมีค่าความคลาดเคลื่อนในการทำนาย (root square error of prediction, RMSEP) เท่ากับ 5.53% 6.52 องศาบริกซ์ 53.8 มิลลิกรัมต่อกรัมน้ำหนักสด 13.9 มิลลิกรัมต่อกรัม น้ำหนักสด และ 51.9 มิลลิกรัมต่อกรัมน้ำหนักสด ตามลำดับ และจากผลการศึกษาด้วยเทคนิค HSI พบว่าเทคนิค HSI ยังไม่สามารถใช้ในการตรวจสอบปริมาณความชื้นในเนื้อถำไย TSS ปริมาณซูโครส กลูโคส ฟรุกโตส และปริมาณน้ำตาลทั้งหมุดลำไยอบแห้งทั้งผลได้ เนื่องจากให้ค่า R² และ RMSEP ที่ ต่ำ โดยให้ก่า R²เท่ากับ 0.7071, 0.6676, 0.5247, 0.2699, 0.4944 และ 0.5333 ตามลำคับ และให้ก่า RMSEP ที่สูงเท่ากับ 13.60% 13.36 องศาบริกซ์ 90.93 มิลลิกรัมต่อกรัมน้ำหนักสด 24.03 มิลลิกรัมต่อ ้กรัมน้ำหนักสด 23.86 มิลลิกรัมต่อกรัมน้ำหนักสด และ 115.42 มิลลิกรัมต่อกรัมน้ำหนักสด ตามลำคับ

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CHAPTER 1 INTRODUCTION

1.1 Background

Longan (*Dimocarpus longan* Lour.) is one of the economically important crops in Thailand especially in Lamphun and Chiang Mai provinces. In 2015, around 553 thousand tons of longan was exported as fresh, frozen, dried and canned longan that earned the income approximately 15.8 billion Baht (Office of Agricultural Economics, 2016). Twenty five percent of the total production volume was processed by drying. More than 80% of fruits for drying were dried as whole longan which was then exporting to China and Hong Kong. Dried whole longan fruit has a high export volume about 110,729 tons with value around 5.4 billion Baht (Office of Agricultural Economics, 2016).

According to the Thai Agricultural Standard for exporting dried whole longan, the qualities required are size (19-25 mm), peel color (brown or reddish dark brown color), moisture content for the whole fruit not exceeding 13.5% or not exceeding 17% for the flesh only, water activity of the flesh not exceeding 0.6, total soluble solids of the flesh not lower than 76 °Brix and pH of the flesh not lower than 5.0 (Thai Agricultural Standard, 2006).

Major of drying method is used a batch dryer cabinet which has LPG burner for heat providing and the drying temperature is almost unstable (Tippayawong et al., 2008). Normally, non-homogeneity of drying is obtained and leads to higher moisture content of flesh which risks for mold growth during storage (Nagle et al., 2010). Thus, the moisture content of product at the end of acceptance point is absolutely important in drying. In addition, most of the carbohydrates in longan are in the form of sucrose, glucose and fructose which provide longan's characteristic taste that is directly related to flavor and acceptability of consumers (Rangkadilok et al., 2005). Recently, there has been the production of longan syrup and longan sugar, new and high commercial product market. These products are studied to have functional properties such as the ability to act as a sugar replacer and use for fructo-oligosaccharides production (Surin et al., 2012).

In general, internal quality of dried longan is determined by chemical composition such as moisture content which has commonly been used a conventional oven-drying method and the sweetness has generally been assessed in term of total soluble solids (TSS) with refractometer and individual sugars by HPLC (high performance liquid chromatography) which provide acceptable accuracy for industry. However, standard methods for determining these qualities are destructive, time consuming and not suitable for rapid monitoring or on-line sorting applications (Rady and Guyer, 2015). Hence, rapid and nondestructive method is needed.

Nowadays, several studies have been successfully used the near-infrared spectroscopy (NIRS), which is a method to predict the quality of food due to the speed of analysis, nondestructive and chemical-free technique. In 2013, this technique was applied to study about olive and the result found that NIRS had a possibility to assess moisture content of olive (Morales-Sillero et al., 2011). According to Rady and Guyer (2015), the use of NIR diffuse reflection for quantitative analysis was effectively evaluated glucose and sucrose content of potatoes. Moreover, Bai et al. (2014) found that the different positions of measurement of NIRS on the blueberry fruits (the calyx or a position not on the calyx) gave different characteristics of spectra which related to the various prediction results. Furthermore, there is another technique that integrates imaging and spectroscopy technologies for attaining spatial and spectral information of product. The main advantage of this technique is to select any region of interest (ROI) in the HSI image, although NIRS is only to collect an individual spectrum at one point on the

sample (Elmasry and Sun, 2010). Several researches have been using this technique for the quality assessment in agricultural products for example; Huang et al. (2014) had successfully applied HSI for determining moisture content of soybean and the prediction of TSS of table grapes using HSI was operated by Baiano et al. (2012). However, nobody has used this technique for determining quality of dried whole longan of Thailand. Thus, the goal of this study is to examine the feasibility of two nondestructive techniques (NIRS and HSI) to apply for determining moisture content and sugar content in flesh of dried whole longan fruit which can be further using in routine analysis in industry.

1.2 Aim of the study

To study the possibility of NIRS and HSI for determining moisture and sugar contents of dried whole longan.

1.3 Hypothesis

Different conditions of drying (temperature and time) affect the changing of moisture and sugar content in longan. NIRS and HSI techniques can vibrate the chemical bonds of molecules of water and sugar that includes C, H, O and N atoms. Thus, there may be possible for determining moisture and sugar content in dried whole longan.

1.4 Scope

1.4.1 Fresh longan fruits cultivar Edor (AA grade, diameter 24-27 mm) were dried at different condition (60, 70 and 80 °C) using a tray dryer with a through flow mode or until the dried flesh had moisture content less than 18%. Samples were taken for analyses at different times of drying.

1.4.2 Dried longans (at different condition) were measured NIRS (800-2500 nm) and imaged by HSI (400-1000 nm).

1.4.3 To analyze moisture content by using hot air oven 105 °C until the weight was constant (AOAC, 2000) and sugar content by HPLC (Liu et al., 2013).

1.4.4 To build suitable model for predicting quality values of dried longan (from NIRS and HSI) and validate the model.



CHAPTER 2 LITERATURE REVIEWS

2.1 Longan

Longan (*Dimocarpus longan* Lour.) is a subtropical fruit widely grown in China and Asia, including Thailand, Vietnam, and the Philippines (Angkasith et al., 1999). Longan has many scientific names such as *Euphoria longana* Lam., *Euphoria longan* Strend., *Nephelium longana* Camb. and *Dimocarpus longan* Lour. (Pawin et al., 2000) and common names are longan, lungan, dragon's eye or eyeball. Longan is one of the economically important crops in the northern part of Thailand. Moreover, the cultivars planted longans in the varied regions in Thailand, they are cv. Edor, Chompoo, Haew and Biew Khiew. Thus, the most popular cultivar is Edor about 66 % of the total longan growing area in the country. Second, cv. Chompoo about 10% and then, cv. Haew about 8%, cv. Biew Khiew about 7% and other cultivars about 9% (Pawin et al., 2000).

Longan is a non-climacteric fruit and accordingly, they do not ripen until they are harvested. Consequently, the fruit must be picked at an optimal eating quality (Huang, 1995). Longan fruit consists of an almost large black or dark brown seed at maturity. The fruit is heart-shaped or round with a thin and leathery pericarp. The pericarp has yellowish to light brown color, and the skin is smooth. The tissue of flesh is fleshy, soft and has translucent white color. In addition, the longan tree has the height approximately 10-20 feet. The tree is similar to lychee tree. Normally, longan tree flowers at the end of winter and the mature longan fruit was harvested over July to September (Jiang et al., 2002).

Harvest indexes of longan are skin color, weight of fruits and total soluble solids of flesh. The concentration of total sugar increases in longan fruit during ripening with total sugar content varying with stage of maturity and cultivar (Menzel and Waite, 2005). In addition, Longan is a fruit which gives high energy for costumer as a result of the presence of sugars. The major sugars are sucrose, fructose and glucose (Li et al., 2009). The fresh pulp of longan has several nutritional components, and investigated to have health benefits (Huang et al., 2012). The nutritional composition of fresh flesh and dried flesh of longan are shown in Table 1. Moreover, longan involves high amount of secondary metabolites such as phenolic acids, flavonoids and polysaccharide. The major phenolic compounds of longan are gallic acids, ellagic acid and coliragin. These compounds were found in all parts of the fruit such as peel, flesh and seed (Prasad et al., 2009).

Nutritional composition	Fresh flesh longan	Dried flesh longan
Moisture content (%)	81.10	7.80
Fat (%)	0.11	0.40
Fiber (%)	0.28	1.60
Protein (%)	0.97	4.60
Ash (%)	0.56	2.86
Total carbohydrate (%)	16.98	72.70
Energy (kcal/kg)	305.70	1,310.00
Calcium (mg. 100^{-1} g)	5.70	27.70
Iron (mg. 100 ⁻¹ g)	7770.35	2.39
Phosphorus (mg. 100 ⁻¹ g)	35.30	59.50
Ascorbic acid (mg. 100 ⁻¹ g)	69.20	137.80
Sodium (mg. 100 ⁻¹ g)	-	4.50
Potassium (mg. 100 ⁻¹ g)	-	2,012.00
Nicotinic acid (mg. 100 ⁻¹ g)	-	3.03
Vitamin B2 (mg. 100 ⁻¹ g)	-	0.37

Table 1 The nutritional	composition of flesh longan
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Source: Tongdee (1997)

2.2 Drying whole longan

Dried whole longan fruit is an important processed agricultural product and a significant export item of Thailand. The volume of dried whole longan exports increased slightly around 10 times during 2006-2015. In 2011 had the highest volume of exporting as shown in Figure 1 (Office of Agriculturel Economic, 2016). Longan drying process can extend a period of time for preservation and can reduce loss arising from seasonal glut (Thai Agricultural Standard, 2006). There are many longan cultivars for drying which includes Edor, Chompoo, Haew and Biew Khiew. However, longan cv. Edor is the most popular cultivar for drying, because of its large size and good color of peel. Longan cv. Edor which is sweet and has good flavor, is rather tough and not as crispy as compared to the flesh of cv. Biew Khiew. About cv. Haew, it gives dark peel color when it was dried (Maitree and Wijit, 2004).

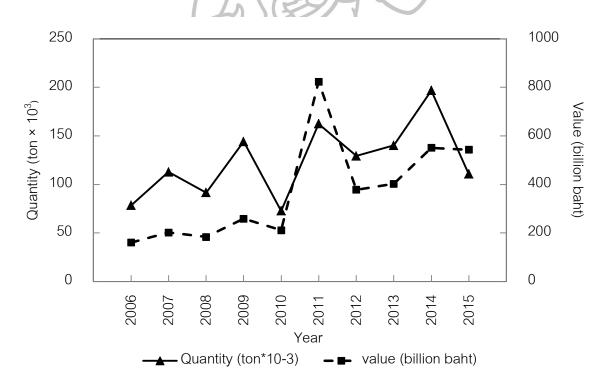


Figure 1 Export volume and value of dried whole longan fruit between 2006-2015.

Source: Office of Agricultural Economics (2016)

2.2.1 Standard for exporting dried whole longan fruit

According to the Thai Agricultural Standard for exporting dried whole longan, the fruit will has either brown or dark brown color. The skin will be free from burnt and mycelium. The flesh will be dry, non-sticky and its flavor will be sweet and free from sour or bitter taste (Thai Agricultural Standard, 2006).

Dried whole longan fruit according to this standard is classified into 4 sizes as follows: size code 1 (>2.5 cm diameter), 2 (>2.2-2.5 cm diameter), 3 (>1.9-2.2 cm diameter) and 4 (1.5-1.9 cm diameter)

For the chemical requirement, the moisture content for the whole fruit (including flesh, pit and skin) must not exceeding 13.5% or not exceeding 17% for the flesh only, water activity of the flesh must not exceeding 0.6, total soluble solids of the flesh not lower than 76 °Brix and pH of the flesh must not lower than 5.0 (Thai Agricultural Standard, 2006).

2.2.2 Whole longan dryer

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There are many whole longan dryers in Thailand. Most dryers are manually used batch dryer cabinet. Generally, the drying cabinet has the dimensions of 2.35 m × 2.35 m × 1.0 m with a loading capacity of around 1,500- 2,000 kg of fresh longan per batch. The batch dryer consists walls of galvanized steel sheet with a perforated metal tray which is higher from the floor of bed for hot air transition as shown in Figure 2. Fresh unpeeled longan is laid on top of a stationary thick bed on one large tray. Heat is supplied to the fruit layer by a diesel or liquefied petroleum gas (LPG) burner. The hot air is induced by a fan, which is driven by a motor on the top. Hot air heats up the longans and also carries moisture out. The moist air leaves at the top of the bed to the ambient environment (Tippayawong et al., 2008). Normally, it uses time about 48 hr. for drying and the price of the dryer was about 30,000-40,000 Baht (Tippayawong et al., 2008).

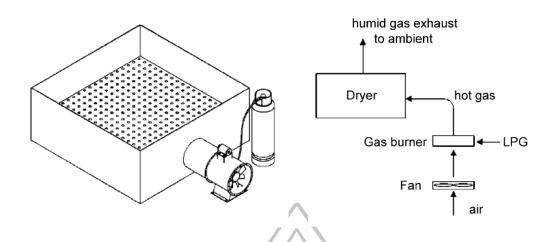


Figure 2 Structure and operation process of a batch dryer cabinet.

Source: Tippayawong et al. (2008)

2.2.3 Method of processing of dried whole longan

Presently, drying the whole longan in Thailand has many methods and processes. However, majority of drying methods is the same. Maitree and Wijit (2004) reported about the method of drying the whole longan as follows:

Firstly, fresh longan fruits cultivar Edor were graded into AA grade, A grade, B grade, C grade and broken longan. Due to the difference of sizes, the small fruit used at least 40 hr. for drying while the bigger one used at least 48 hr. After that, the fresh longan fruits were dried by batch dryer cabinet at temperature 70-80°C for 40-48 hr. The big fruits should be placed below the dryer for faster drying. The difference of fruit sizes were separated each other by perforated metal trays. During drying, operators usually turn the fruit layer upside down and inside out completely in 12-15 h intervals (Tippayawong et al., 2008). In drying process, the temperature should not be too high. It might lead to dark color or burn on longan peel. After finishing, dried longans were left for 1-2 hr. and then graded again by sorting machine. Finally, 10 kg plastic bags were packed inside the paper boxes for distributing (Maitree and Wijit, 2004).

According to drying the whole longan by batch dryer cabinet, non-uniform drying in term air flow and temperature is obtained. Drying operator usually solves this

problem by transposition layers of longans in the dryer. This method causes bruises and cracks of dried longans (Janjai et al., 2006). Thus, there were some researchers who studied about this problem for improving the drying.

Janjai et al. (2006) has studied on air velocity distribution in the plenum of the dryer for improving drying uniformity. It was found that the distribution of air temperature and air speed in the dryer were very non-regular that affected longans in the lower layer were dried faster than the fruits in other layers. This problem was solved by placing air guides and enlarging the space for mixing air under the perforated tray.

Nagle et al. (2010) has focused on improving quality and energy performance of a fixed-bed longan dryer by thermodynamic modification that included air deflector installations in the plenum and insulation. The study found that air velocity distribution was improved. Modifications decreased non-uniform drying and there were more homogenous product color. However, non-homogeneity of drying is still found.

2.2.4 Changes of longan after drying

Phupaichitkun et al. (2005) reported that the moisture content of dried whole longan decreased when the drying time was increased. Air temperature and size of fruit affected the whole longan drying. Same as other agricultural products the drying time decreases when the air temperature is increased as shown in Figure 3. The larger fruit took longer time to dry whereas the increased diffusion path within the fruit (Figure 4). The result also found that the rate of water transfer from the surface of the fruit to the air was faster than the rate of water diffusion from the inside of the fruit to the surface of the fruit. However, the mass transfer from the inside to the surface was the main affecting factor during the drying process for the whole longan. About the water activity, often the water activity in the seed will be greater than 0.6 that still remains a storage risk. The higher water activity in comparison with the fruit flesh showed that the seed has a specific water sorption.

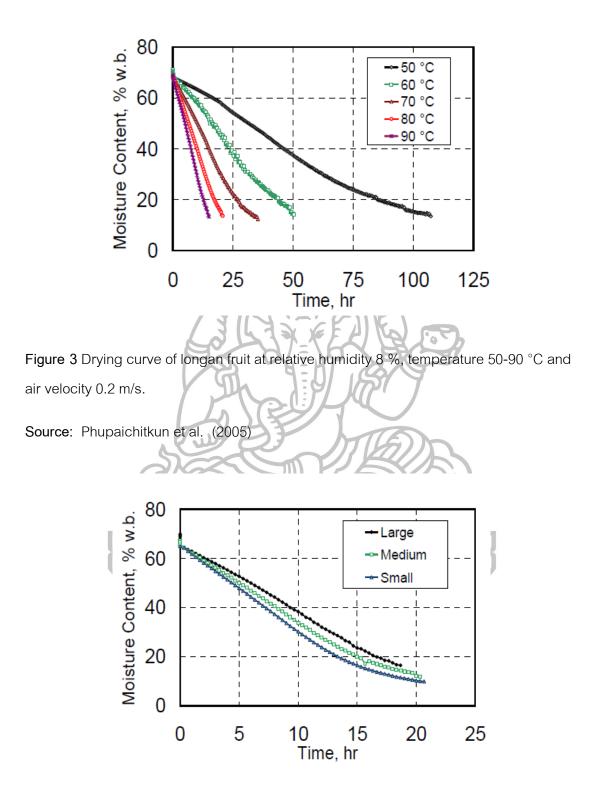


Figure 4 Drying curve of longan fruit at different size, relative humidity 8 %, temperature 80 °C and air velocity 0.2 m/s.

Source: Phupaichitkun et al. (2005)

Phupaichitkun et al. (2006) studied about the shrinkage inside Thai unpeeled longan fruit. Each part contains a different structure and substance. The shrinkage characteristic of longan depends on the structure of dried solid and interaction between liquid and solid in each part. According to the structure of peel, the external layer contains thick wall and high porosity cells that is why there are no shrinkage during drying. The aril comprises high concentration of sugar solution in soft tissue. Thus, the interaction between liquid and solid part is very high. Seed has white meat with black seed coat which is low water permeability and the shrinkage of seed has a specific characteristic because of non-deformation in seed coat during drying. The kinetic of seed, aril and pericarp are shown in Figure 5. It was found that the structure of fruit had an effect on water transportation. The water of the seed only diffused through the seed stalk to the drying air. Hence, the drying rate of the seed was very slow. About the aril, the moisture content can diffuse through the pericarp.

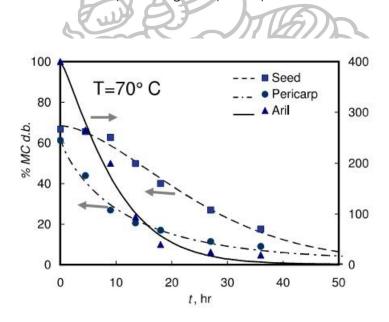


Figure 5 Moisture content from experiment of seed, pericarp and aril during drying at 70°C.

Source: Phupaichitkun et al. (2006)

2.3 Near- infrared spectroscopy (NIRS)

Near- infrared spectroscopy (NIRS) has been used as a method to predict the quality of food due to the speed of analysis, nondestructive and chemical-free techniques. In addition this technique can determine both of quantity and quality of products. Thus, several studies have been successfully used the NIRS technology for determining total soluble solids, acidity, texture and moisture content (Nicolaï et al., 2007). NIR radiation covers the range of the electromagnetic spectrum between 780-2500 nm or 12,500-4,000 cm⁻¹. In NIRS spectroscopy, the product which has O-H, C-H, N-H and C=H bonds is irradiated with NIR radiation, and the reflected or transmitted radiation is measured. Although, the radiation passes through the sample, its spectral characteristics change by scattering and absorption processes. This change depends on the chemical composition of the product which is related to the microstructure in the sample. Moreover, advanced statistical techniques, such as partial least squares regression is then applied to extract the information from the spectra (Sun, 2009). An NIRS system consists of a light source (usually a tungsten halogen light), sample presentation (sample holder or sample cell), interferometer, detector, optical components, such as lenses, collimators, beam splitters, integrating spheres and optical fibers and computer. The example of NIRS system is shown in Figure 6.

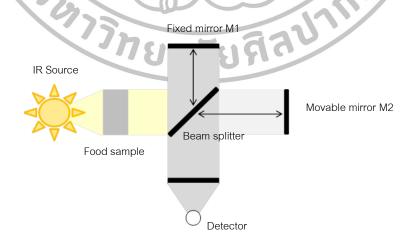


Figure 6 NIRS system Source: Sun (2009).

NIR radiation has a penetration depth of up to 10 mm in the 800-1100 nm range and between 1 and 3mm in the 1100-2500 nm range (Nicolaï et al., 2007). Furthermore, the appropriate mode of NIRS measurement will be controlled by the optical properties of the samples (Figure 7). Translucent materials are commonly measured in transmittance mode (Figure 7A). Impure liquids or semi-solids or solids may be measured in diffuse transmittance mode (Figure 7B), diffuse reflectance mode (Figure 7C) or transflectance mode (Figure 7D/E), depending on their absorption and scattering characteristics (Reich, 2005).

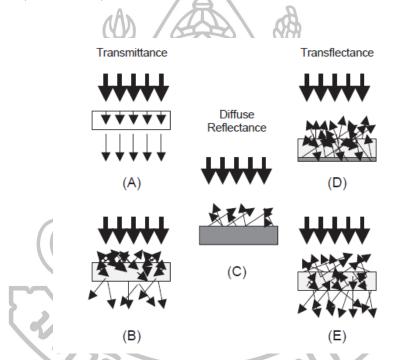


Figure 7 NIRS measuring modes (A/B) transmittance, (C) diffuse reflectance and (D/E) transflectance.

Source: Reich (2005)

NIRS technique can analyze both of quality and quantity information using relationship between spectral data and reference data. However, the spectra, received from this technique, were constructed from overtone and combination of function groups. Thus, NIRS spectra are often complex and normally acquire broad bands and overlapping peaks. Moreover, chemical, physical, and structural properties of sample influence the measured spectra. Thus, there are some techniques for reducing this problem. Mathematical treatments used to develop NIRS spectra such as multiplicative scatter correction (MSC) and standard normal variate (SNV). Both methods have basically been used to process reflectance and transmittance spectra for reducing scatter-induced baseline offsets. Normalization algorithms used to reduce baseline shifts and intensity difference occurring from variable positioning or path length variations. Moreover, the resolution of overlapping bands and baseline offsets may be reduced by derivatives and smoothing algorithms (Reich, 2005).

After mathematical preprocessing, the multivariate regression methods most usually used in quantitative NIRS analysis are principal component regression (PCR) and partial least-squares (PLS) regression. Spectra acquisition and reference values are built the multivariate model to relate the spectral variations to the reference values of the analytical target. And then, cross validation or external validation performed to validate model (Reich, 2005).

2.3.1 Near- infrared spectroscopy for food analysis

NIRS technique is a promising technique for the non-destructive analysis and quality checks of many kinds of food. The capabilities of near- infrared spectroscopy for quantitative analysis of food (especially, moisture content and sugar) are shown in Table 2.

Sample	Parameter	Range	No. of	Wavelength (nm)	Measurement mode	Data analysis	Accuracy	Reference
			sample		A			
Ginseng	Moisture content	0-9%	92	1100-2500	Reflectance	PLS	$R^2 = 0.998$	Ren and Chen (1996)
powder			A				RMSEP=0.140%	
otato chip	Moisture content	1.2-4.0%	15	1000-2500	Reflectance	PLS	$R^2 = 0.990$	Shiroma and Rodriguez-Saona
			5	17=16			RMSECV=1.93%	(2009)
live	Moisture content	54.7-75.9%	698	400-2498	Reflectance	PLS	$R^2 = 0.880$	Morales-Sillero et al. (2011)
			V	Dh $1:9/$	TIM		RMSECV=1.35%	
Cabbage	Moisture content	93.54-95.82%	120	500-1100	Reflectance	PLS	$R^2 = 0.740$	Kramchote et al. (2014)
				AT			RMSEP=2.50%	
Potato	Sugar content	Glucose: 0.0028-1.1355%	540	900-1685	Reflectance	PLS	$R^2 = 0.77$	Rady and Guyer (2015)
			212				RMSEP=0.0665%	
		Sucrose: 0.0045-2.1842%)	$R^2 = 0.75$	
				J LL Y			RMSEP=0.1324%	
<i>l</i> lango	TSS	7.7–26.3%	592	700-1100	Reflectance	PLS	$R^2 = 0.9$	Rungpichayapichet et al.
			$\langle (C$		API/	٠Į	SEP= 1.2%	(2016)
Curry soup	TSS	16.75-35.02%	113	800-2500	Reflectance	PLS	$R^2 = 0.92$	Sirisomboon and Nawayon
		1473		111		RMSEP= 0.95%	(2016)	
					22V/			

 Table 2 The capabilities of near- infrared spectroscopy for quantitative analysis of food.

Ren and Chen (1996) used NIRS to measure the moisture content in the ginseng powder in the segment 1100-2500 nm. The spectra were treated by first derivative and scatter correction. A high correlation of determination was found to be 0.998, with a standard error of prediction (SEP) of 0.14%. Bias was only 0.12%. The results indicated that the equation obtained from this study would be used for the moisture content monitoring of the processed ginseng roots.

Shiroma and Rodriguez-Saona (2009) applied NIRS (1000-2500 nm) for rapid monitoring of potato chip quality including moisture content and fat content. The spectral data were analyzed by partial least squares regression (PLSR). Fat content ranged from 18 to 45% and moisture content ranged from 1.2 to 4%. Correlation coefficient (R) for moisture was >0.97 with standard error of cross validation (SECV) < 0.3% and the prediction models for fat had r > 0.96 and SECV < 1.60.

Morales-Sillero et al. (2011) adopted NIRS in the range of 400-2498 nm to assess the properties of intact olives. Chemical parameters, such as oil and moisture contents were investigated. Coefficient of determination (R^2) was 0.880 for moisture, with RMSECV of 1.35%. The results show that NIRS is a useful, rapid and non-destructive technique for determining moisture content of table olive industry.

de Oliveira et al. (2014) investigated the potential of NIRS to predict passion fruit ripening parameters as sugars, organic acids and carotenoids. Spectra of 56 samples of the lyophilized passion fruit were collected using in NIR range (800-2500 nm). Partial least square regression (PLSR) was used to build calibration models. R² were found 0.862 for glucose, 0.756 for fructose, 0.691 for sucrose and 0.833 for total sugars. For glucose and fructose contents both methods were unsatisfactory due to low concentrations of these components in the passion fruits.

Kramchote et al. (2014) applied NIRS (500-1100 nm) to determine the quality of cabbage (moisture, SSC and ascorbic acid contents) and compare the prediction ability of interactance and reflectance measurements. The calibration model were built by partial least squares (PLS) regression. In case of moisture content, coefficient of determination (R^2) of 0.67 and root mean square error of prediction (RMSEP) of 2.34

g/kg for interactance, with R^2 of 0.74 and RMSEP of 2.50 g/kg for reflectance. Finally, there was possible to use the Vis/NIR spectroscopy as a rapid tool.

Rady and Guyer (2015) has been extensively and successfully applied NIRS (900-1685 nm) on sugar content in potatoes. This study aimed to study the prediction of glucose and sucrose for potato tubers that were important to the frying industry. Partial least squares regression (PLSR) was applied for building prediction models. Prediction models for glucose showed R^2 was 0.77 with RMSEP of 0.0665% and for sucrose, the R^2 was found 0.75 with RMSEP of 0.1324%. This study presented NIR reflectance spectroscopy to effectively evaluate the sugar content of potatoes.

Rungpichayapichet et al. (2016) used NIRS (700-1100 nm) to determine postharvest quality of mango such as firmness, total soluble solids (TSS), titratable acidity (TA) and ripening index (RPI) using partial least squares (PLS) regression analysis. For TSS, the results showed R² of 0.9 and SEP of 1.2%. The results indicated that NIRS can be used as a good non-destructive technique for mango quality assessment.

Sirisomboon and Nawayon (2016) studied about the potential of NIRS (12,500- 3600 cm^{-1}) to assess the total solids content of instant curry soups containing coconut milk. R², RMSEP, bias and RPD of 0.92, 095%, -0.20% and 3.71, respectively. The results showed that NIRS could be applied in an instant curry soup production line for production control and quality assurance.

From the results, it can be seen that NIRS gave high accuracy of moisture content and sugar content prediction. Thus, this technique might be used for determining moisture content and sugar content in flesh of dried whole longan in my study.

2.4 Hyperspectral imaging (HSI)

Hyperspectral imaging is a technique that integrates spectroscopy and imaging techniques to permit identification of different components and their spatial distribution in sample. This technique contributes a two-dimensional spatial array of vectors which represents the spectrum at each pixel location. The resulting three-dimensional dataset involving the two spatial dimensions and one spectral dimension is known as the datacube or hypercube (ElMasry and Sun, 2010) as illustrated in Figure 8. Every pixel in HSI image has an individual spectrum that containing information about chemical composition. Thus, the spectra can be collected at any points or areas of sample.

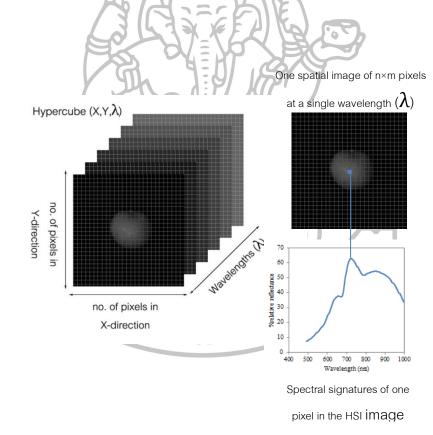


Figure 8 Hyperspectral image cube (hypercube).

The basic components of the system consist of illumination units (in the region 400-1000 nm), HSI camera with a CCD detector (charge-coupled device), spectrograph, translation stage and a computer system as shown in Figure 9.

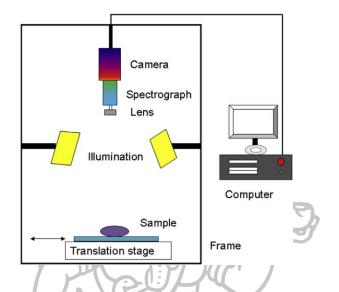


Figure 9 Components of a typical hyperspectral imaging system.

Source: Iqbal et al. (2014)

The theory of this technique is similar to NIRS, the light from light source (radiation in the wavelength range of 400-1000 nm) strikes the sample and then the reflected light beam from sample will enter the lens and passes into the spectrograph that has grating which can disperse the light into single wavelength. And then each wavelength will be captured by CCD detector. Hence, it can conduct the data in form 3 dimension of hypercube which x and y are rows and column of two dimensional spatial information and λ is spectral information.

There are four applications to acquire 3-D hypercube (x, y, λ), which are point scanning, line scanning, area scanning, and single shot method as shown in Figure 10 First, point scanning method as the whiskbroom method (Figure 10a) a single point is scanned at one pixel to contribute the spectrum of this point. However, other points are scanned by moving sample or detector along spatial dimension. The weaknesses of

this application are time-delayed for positioning the object and need advanced hardware to support. Second method is line scanning or pushbroom method (Figure 10b), a hypercube can be obtained as the line is scanned along x dimension. Line scanning is especially suitable in conveyor belt systems that are commonly used in food process lines. Thus, this method is the most popular method for food quality and safety inspection. While the area or plane scanning (Figure 10c) is a method which obtains with full spatial information at a single wavelength at a time. Lastly, the single shot method archives both of spatial and spectral information in one exposure to capture the images (Figure 10d). This method is very suitable when fast imaging is required (Wu and Sun, 2013).

Moreover, there are three common modes of measurement for hyperspectral imaging i.e. reflectance, transmittance and interactance (ElMasry and Sun, 2010) as shown in Figure 10e-g.

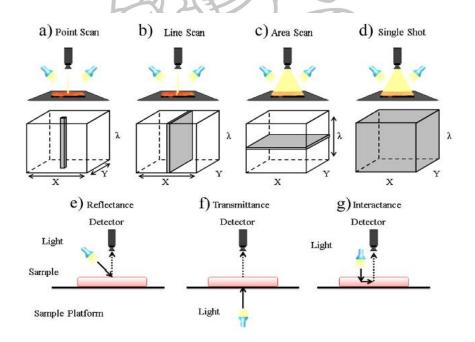


Figure 10 Acquisition of hyperspectral images and modes of measurement.

Source: Wu and Sun. (2013)

The advantages of hyperspectral imaging over the traditional methods include less sample preparation, nondestructive, fast acquisition times and visualizing whole spatial distribution of chemical compositions concurrently. The difference of HSI comparison between imaging and spectroscopy is shown in Table 3.

 Table 3 Main differences among imaging, spectroscopy and hyperspectral imaging techniques.

Features	Imaging Spectroscopy	Hyperspectral
	maging opectroscopy	imaging
Spatial information	A second	\checkmark
Spectral information	La Perfection	\checkmark
Muti-constituent information	x	\checkmark
Building chemical images	x	\checkmark
Flexibility of spectral	x x	\checkmark
Information extraction		2
		<i>µ</i>)
Source: ElMasry and Sun. (2010)	MALL A)
$\nabla \mathcal{A}$	K (GEOD)	
2.3.2 Hyperspectral image	ging for food analysis	\sim
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There are many researches that determine the potential of HSI in the application to assess of quantitative food. The capabilities of HSI for quantitative analysis of food (especially, moisture content and sugar) are shown in Table 4.

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Sample	Parameter	Range (%)	No. of	Wavelength (nm)	Measurement mode	Data analysis	Accuracy	Reference
			sample		B			
Pre-sliced turkey	Moisture content	43.03-80.40%	158	900-1700	Reflectance	PLS	$R^2 = 0.88$	lqbal et al. (2013)
ham					218		RMSEP= 2.51%	
Atlantic salmon	Moisture content	54.19-76.52%	266	400-1700	Reflectance	PLS	$R^2 = 0.85$	He et al. (2013)
				0 17=			RMSEP= 1.80%	
Soybean	Moisture content	49.0-67.7%	270	400-1000	Reflectance	PLS	R = 0.971	Huang et al. (2014)
			Y	Jun 1	ו יתגוץ		RMSEP= 9.2%	
Mango slices	Moisture content	10.35-90.12%	162	400-1000	Reflectance	PLS	$R^2 = 0.972$	Pu and Son (2016)
			E.	WOF	MAD	6	RMSEP= 4.61%	
Lychee	Moisture content	8.36-70.26%	360	400-1000	Reflectance	PLS	$R^2 = 0.948$	Yang et al. (2015)
			(d)		PAV.	5)))	RMSEP= 0.83%	
Table grape	TSS	11.92-21.12%	140	400-1000	Reflectance	PLS	$R^2 = 0.94$	Baiano et al. (2012
					1		RMSEP= 0.06%	
Whole Port wine	TSS	9.1-25.0%	240	380-1028	Reflectance	PLS	$R^2 = 0.89$	Fernandes et al.
grape berry				ス U F		51	RMSEP= 1.1%	(2015)
			95					
				mar	ัยสิลป			
				ברטי)	51510			

Table 4 The capabilities of HSI for quantitative analysis of food

Baiano et al. (2012) applied the HSI technique (400-1000 nm) for prediction of some physico-chemical and sensory indices of table grapes. A partial least squares regression (PLSR) model was applied. In case of soluble solid contents, the result was found $R^2 = 0.94$ with RMSEP 0.06%. However, the model of sensory gave a bad prediction because the spectra information did not correspond to the sensory data.

He et al. (2013) studied about the potential of HSI technique (400-1700 nm) for determining the distribution map of moisture content in farmed Atlantic salmon. The relationship between spectral data and the moisture content values was effectively conducted by partial least squares regression (PLSR). Three spectral ranges of 400–1000 nm, 900–1700 nm and 400–1700 nm were analyzed for selection of the best spectral range. The R² of 0.893, 0.902 and 0.849, and RMSEP of 1.513%, 1.450% and 1.800% for three spectral ranges, respectively. The best model was built the image for visualizing moisture content of salmon fillets. The results showed that hyperspectral imaging technique has a great potential to predict the moisture content distribution of salmon fillets (Figure 11)

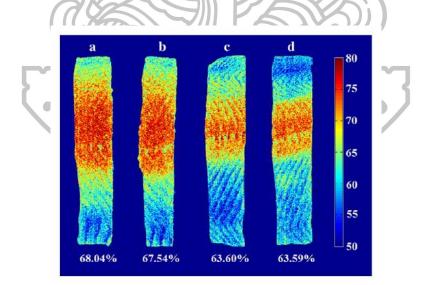


Figure 11 Moisture distribution maps in salmon fillets. The values in the bottom of figures showed the average concentration of moisture in the whole fillet. Fillets (a) and (b) had high moisture content (MC) while fillets (c) and (d) had low MC.

Source: He et al. (2013)

Iqbal et al. (2013) developed a HSI system in the NIR region (900–1700 nm) to predict the moisture content, pH and color in cooked, pre-sliced turkey hams. Spectral data were extracted and analyzed by using partial least-squares (PLS) regression. Nine important wavelengths for moisture prediction were selected (927, 944, 1004, 1058, 1108, 1212, 1259, 1362 and 1406 nm). With the identified reduced number wavelengths, good coefficient of determination (R²) of 0.88 with RMSECV of 2.51% for moisture content.

Huang et al. (2014) developed regression models for predicting the color and moisture content of soybeans concurrently during the drying process using a HSI technique in the range of 400-1000 nm. Predicting models were built using the partial least squares regression method. The prediction results of moisture content were found correlation coefficient (R) = 0.971 and root-mean-square errors of prediction or RMSEP = 9.2. The results showed significant potential in measuring moisture content of soybeans simultaneously during the drying process.

Fernandes et al. (2015) measured pH, sugars, and anthocyanin content of whole grape berries using HSI technigue (380-1028 nm) The R² of sugar content was found 0.89 with RMSEP of 1.1%. The results presented HSI had a potential to predict sugar content of whole grape berries.

Yang et al. (2015) focused on analyzing the relationship between browning levels of lychee and moisture contents of pericarp, and establishing calibration models for determining browning degree of lychee based on the MC prediction of pericarp using HSI technique (400-1000 nm). The results indicated that coefficients of determination (R^2) of 0.948, and root mean square error (RMSEP) of 0.83% for moisture content. At last, the visualization map of lychee with different browning levels was build and distribution of browning degree in a pericarp was detected by considering color variation of pixels in the map as shown in Figure 12. According to the results, browning level 1(no browning) had 55-70% moisture content, level 2 (slight browning) had 35-55% moisture content, level 3 (moderate browning) had 15-35% moisture content and level 4 (serious browning) had 0-15% moisture content.

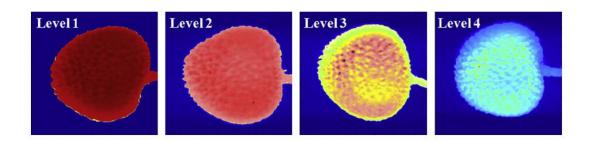
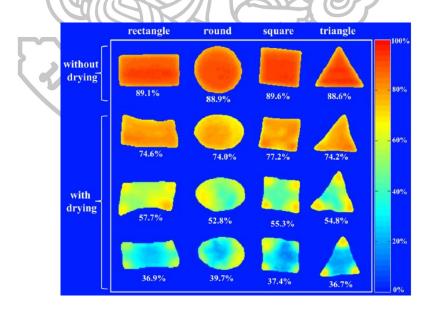
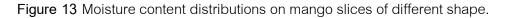


Figure 12 Visualization map of lychee with different browning levels.

Source: Yang et al. (2015)

Pu and Sun. (2016) used HSI system (400-1000 nm) for moisture prediction of mango slices. Partial least square (PLS) was applied to build calibration and validation models. The best model was found $R^2 = 0.972$ and RMSEP = 4.611%. The model was adapted into the moisture visualization. Moisture distribution map showed that the moisture content of the center of the mango slices was lower than the other parts (Figure 13). From this study indicated that hyperspectral imaging had a potential for measuring and visualizing the moisture content during drying process.





Source: Pu and Sun. (2016)

Moreover, there was a research that studied the effects of the light source of HSI system on spherical objects. Gómez-Sanchis et al. (2008) said that the curvature of spherical objects as citrus fruits in acquiring HSI images affected some problems. The images of the fruits which were near the edge were darker than in the center. This problem made the images more difficult to analyze as the calibration which were achieved to correct the spatial and spectral inhomogeneity would have mistaken. A Lambertian ellipsoidal surface was applied to build 3D model of the fruit for calculating the part of the radiation that can reach the camera and to make the intensity of the radiation homogeneous over the whole of the fruit surface captured by the camera. The proposed correction methodology has demonstrated to be effective for minimizing the difference from the central area to edge areas. Figure 14 shows that this method can solve the problem. Four regions obtained from the hyperspectral image after being corrected with the proposed model become more uniform, to the point where they actually overlap.

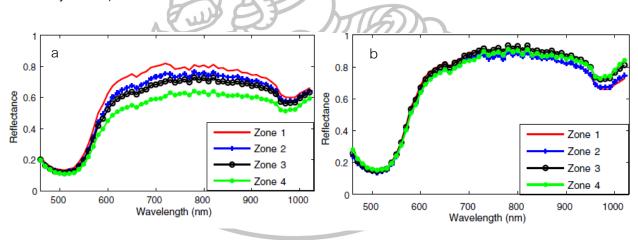


Figure 14 Averaged (a) uncorrected spectra of healthy zones of clemenules mandarinand (b) corrected spectra of healthy zones of clemenules mandarin of four different regions of the image.

Source: Gómez-Sanchis et al. (2008)

Obviously, the result of HSI technique found that the model gave high accuracy of moisture content and sugar content prediction. Thus, this technique also might be applied in moisture content and sugar content prediction in flesh of dried whole longan.



CHAPTER 3 MATERIALS AND METHODS

3.1 Materials

3.1.1 Longan samples

Fresh longan fruits cultivar Edor grade AA (Figure 15) with a diameter of 24-27 mm at a physiological maturity stage were purchased from the wholesale fruit market in Nakhon Pathom, Thailand. Fruits were transported from the orchards in Chiang Mai province. They were sorted for a uniform in size and shape with no visual damages (Figure 15). Then, they were trimmed to remove stalk and kept in a refrigerator $(5.1\pm0.8^{\circ}C, 79.9\pm8.7\%RH)$ until drying experiments. For this study, three batches of longan were purchased during August and September, 2014.



Figure 15 Fresh longan fruits cultivar Edor grade AA.

3.1.2 Chemicals

Standard sucrose, glucose and fructose were purchased from the Sigma-Aldrich (USA). Distilled water (HPLC-grade) was obtained from the Vunique (Thailand).

3.2 Methods

3.2.1 Physical and chemical properties of fresh longan

3.2.1.1 Physical properties

- Size and weight

Every longan fruit was measured for height, diameter and width (Thai Agricultural Standard, 2006) with verneir caliper (11205-200, INSIZE 1205 series, England) as shown in Figure 16. In addition weight per fruit was also measured using a digital scale (BP 211S, Sartorius AG, Germany).

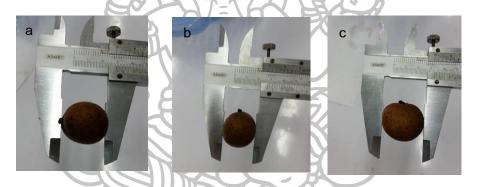


Figure 16 Measuring size of longan by using vernier caliper. (a) height, (b) diameter and ปสิลปาร์ (c) width.

3.2.1.2 Chemical properties

- Total soluble solids

For each batch, ten fruits were peeled. Total soluble solids (TSS) of flesh longan from each fruit was examined in duplicate by dropping the juice through a muslin cloth onto the digital refractometer (Pal-1, Atago, Japan) with a scale 0-53 °Brix (AOAC, 2000).

- Moisture content

Sixteen fruits of each batch were separated into peel, flesh and seed and then they were blended by using a blender (A 11 Basic, IKA, Germany). Samples were weighted and put into a moisture can. The weight was recorded. The moisture content (MC) was measured using hot air oven at 105 °C until the weight was constant (AOAC, 2000). MC (wet basis) was calculated corresponding by Eq. 1.

$MC (\%) = \frac{\text{weight of wet sample (g)-weight of dry sample (g)}}{\text{weight of wet sample (g)}} \times 100$ (1)

Moreover, the moisture contents of the whole fruit and flesh+seed were obtained by calculated from %weight of each fruit part (Appendix G).

- Sugar content

For each batch, six longans were peeled and the flesh was extracted for analysis of individual sugars using the method from Liu et al. (2013). The sample was homogenized. And then, 1 g of sample was mixed with 9 ml distilled water and then centrifuged at 8000 rpm for 10 min at 4 °C. The supernatant was filtrated by 0.45 nylon syringe filter. The filtered supernatant was kept at -18° C for HPLC analysis. Individual sugars were separated using Rezex RCM-Monosaccharide Ca⁺²column (300 × 7.8 mm i.d., 5 µm particle size) and the column was kept in a column heating box at 80 °C. Deionized water was used as a mobile phase at a flow rate of 0.6 ml/min. Individual sugar was detected by using a refractive index detector (RID-10A, Shimadzu, Japan). For quantification and calibration, a standard solution mixture was prepared by dissolving sucrose, glucose and fructose in distilled water (HPLC grade). Final values were expressed in mg/g fresh weight. Standard curves are shown in Appendix A.

3.2.2 Sample preparation

The total of 616 whole fresh longans were dried in a single layer by using a through flow mode laboratory tray dryer (the stem part was turned down). The placement of longans in the tray is shown in Figure 17.



Figure 17 The placement of longans for drying.

The samples were dried at temperature of 60, 70, and 80 °C with an air velocity of 0.2 m/s. Drying had been done until the dried flesh of longan has water activity about 0.55-0.60. Samples were taken in every 6 h. for 60°C and 5 h for 70°C and 80°C. The samples were separated into 2 groups for analysis moisture content and individual sugars. Moreover, drying was repeated in duplicate of each temperature. Drying condition and sampling are shown in Table 5.

			Otoreare	Total		A inc a suit	Amount of tak	ken at each time	Amount of sar	mples for
Drying		Temperature	Storage	drying	Take	Amount	and cond	dition (fruit)	the study	(fruit)
batch	Replication	(°C)	time	time	sample in	of -	Moisture	Sugar	Moisture	Sugar
			(day)	(h.)	every (h.)	sampling	content	content	content	content
Batch 1	1	60	0	60	6	11	8	3	88	33
	2	60	3	60	6		8	3	88	33
Batch 2	1	70	0	50	5	11	8	3	88	33
	2	70	3	50	5	//11/7/	8	3	88	33
Batch 3	1	80	0	40	5	9	8	3	72	27
	2	80	2	40	5	9	8	3	72	27
Total			\sim			50			496	186
		(4,	A	SUDE	gr,	(5)			
			19	13.	ยาลัย	201				
				1	ยาวัย	99				

 Table 5 Drying conditions and sampling of longan for the study.

3.2.3 Nondestructive measurement for determination of moisture content and sugar content in dried whole longan

3.2.3.1 Near-infrared spectroscopy (NIRS) measurement

- Spectra Acquisition

Whole fresh and dried longans were measured spectra by NIRS measurement. The reflectance spectra of a total of 682 samples were measured using a FT-NIR spectrometer model MPA with OPUS software (Bruker optics, Ettlingen, Germany), as shown in Figure 18, in the wavenumber ranging from 12,500 cm⁻¹ to 4,000 cm⁻¹ (800-2500 nm) at a resolution of 16 cm⁻¹ and scan time of 32 scans. The sample temperature was controlled at 25 °C by keeping in cooled incubator (KB, Binder Ltd., Germany) for 1 hr. before spectra measurement. The spectrum of each sample was collected at four positions; calyx (P1), bottom (P2) and the both cheeks of fruit (P3 and P4). The measured positions are shown in Figure 19.

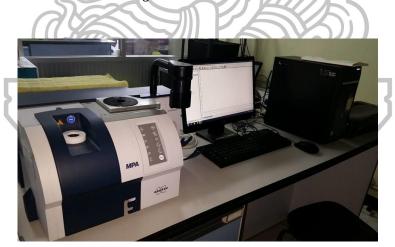


Figure 18 Near-infrared spectroscopy system.

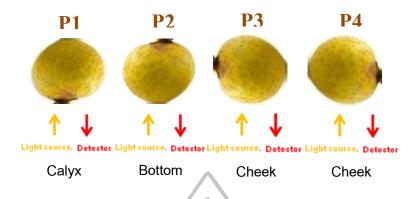


Figure 19 The four measured positions for NIR spectrum acquisition.

- Reference analysis for NIRS

The moisture content of peel flesh and seed as well as the whole fruit, TSS and sugar content of flesh were analyzed after spectra measurement. The samples were separated into peel, flesh and seed then measured moisture content of each part as well as the whole fruit by using a hot air oven at 105°C (AOAC, 2000), TSS (AOAC, 2000) and sugar content of flesh (Liu et al., 2013) were analyzed as described in 3.2.1.2.

- Spectral data analysis for NIRS

The calibration models were developed by partial least squares analysis (PLS) with an external validations (calibration: validation; 60:40) for considering observed spectra and results of chemical analyses. The OPUS chemometric software (Bruker optics, Ettlingen, Germany) version 7.2.139.1294 was applied. To improve calibration accuracy by reducing noises and scattering effects, spectral data were conducted to a variety of preprocessing such as first derivative, second derivative, min-max normalization, vector normalization or standard normal variate correction (SNV) and multiplicative scattering correction (MSC) together with a wavelength region selection were performed according to the optimization function of the OPUS software. The

performance of developed models was presented in terms of R² (the coefficient of determination), RMSEP (root mean square error of prediction), PLS factor (or number of latent variables), bias and RPD (the ratio of the standard deviation of validation set to SEP). The RPD was calculated using Eq.2

$$RPD = \frac{SD_{val}}{SEP}$$
(2)

- Accuracy of calibration models for NIRS

The accuracy of prediction models which had RPD value more than 2.0 were tested by using bias checking (paired *t*-test) method, SEP checking (F-test, ratio of 2 variances) method and slope checking method from International standard ISO 12099 (2010). All statistic formulas are shown in Appendix D.

3.2.3.2 Hyperspectral imaging (HSI) measurement

- Image acquisition for HSI

The laboratory hyperspectral imaging system was used to acquire hyperspectral images of longans in a reflectance mode. The main components of system are displayed in Figure 20. HSI system is mainly consisted of a hyperspectral frame camera with a spectrograph and CCD detector (Cubert GmbH, Germany) covering the spectral range of 400-1000 nm, a light source; 50 W tungsten halogen lamp (Pro Lamp, ASD, USA), two mirrors positioned at an angle of 120° and a computer with Cubert_Gui software (Cubert GmbH, Germany). The distance between the lens and longan was fixed at 40 cm. Longan sample was placed on the sample holding. A total of 682 samples were measured. Each fruit was imaged for 1 position with the equator position facing vertically toward the imaging system. The hyperspectral images were recorded as spatial dimension of 910×900 pixels for every 4 nm and resulted in a total of 128 spectral bands.

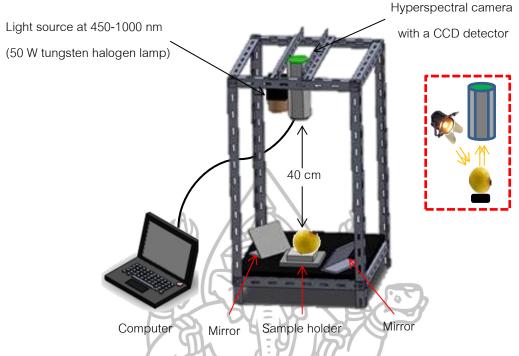


Figure 20 Schematic diagram of hyperspectral image system.

To improve the raw acquired hyperspectral imgages (I), two images for black (B) and white (W) references were applied to reduce the effects of illumination and detector sensitivity. The light source was turned on for 15 min before hyperspectral image acquisition. And then the black image (~0% reflectance) was acquired by recording a spectral image after closed the camera lens completely with a black board. The white reference image was acquired by taking a spectral image from a white canvas (~99% reflectance). The corrected hyperspectral image in a unit of %relative reflectance (R) was calculated using the following equations:

$$R_{i} = \frac{I_{i} - B_{i}}{W_{i} - B_{i}} \times 100 \tag{3}$$

Where I was the raw image, W was the white reference image, B was the dark reference image. The *i* is the pixel index, i.e. i = 1,2,3,...,n and n is the total number of pixels. The final improved spectral images were applied as the basis for spectral

extraction and data analysis (Iqbal et al., 2014). The final hyperspectral images are shown in Figure 21.

Fresh60°C for 15 hr.60°C for 30 hr.60°C for 50 hr.Figure 21 Example of hyperspectral images of fresh and dried whole longan obtainedfrom drying at 60°C for 15, 30 and 50 hr.

- ROI identification and spectra extraction for HSI

After image acquisition, the images were exported form Cubert_Gui software (Cubert GmbH, Germany) in term .hdr and .cue. Ten regions of interests (ROIs) with circle shape (10*10 pixels) within hyperspectral images were collected as displayed in Figure 22. The ROI identification process was developed and the spectral data within ROIs for the fruit were extracted and averaged by using R version 3.1.1 (R Foundation for Statistical Computing, Vienna, Austria) (Appendix E). The reflectance spectrum was obtained by averaging spectral data from 1000 pixels.

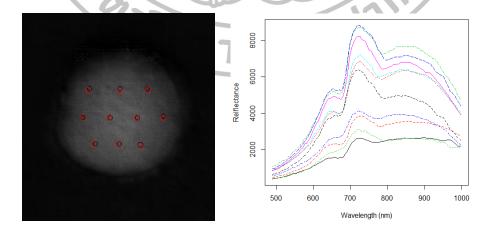


Figure 22 10 points selected of sample in R program.

- Reference analysis for HSI

The moisture content of peel flesh and seed as well as the whole fruit, TSS and sugar content of flesh were analyzed after image acquisition. The samples were separated into peel, flesh and seed then measured moisture content of each part as well as the whole fruit by using a hot air oven at 105°C (AOAC, 2000), TSS (AOAC, 2000) and sugar content of flesh (Liu et al., 2013) were analyzed as described in 3.2.1.2.

- Spectral data analysis for HSI

The Unscrambler software (CAMO Software AS, Oslo, Norway) was used to pretreat the spectral data such as first derivative, second derivative, standard normal variate correction (SNV) and multiplicative scattering correction (MSC), as well as produce calibration and validation model using partial least squares analysis (PLS). The data about 52.5% was set for calibration set and 47.5% was set for validation set. The predictive ability and accuracy of the models were presented in terms of R² (the coefficient of determination), RMSEP (root mean square error of prediction), PLS factor (or number of latent variables), bias and RPD (the ratio of the standard deviation of validation set to SEP). The optimum model was selected if the model provided best prediction performance on a validation set which showed high R² and RPD while low RMSEP (Kaewsorn and Sirisomboon, 2014).

Moreover, the distribution maps were carried out by using R version 3.1.1 (R Foundation for Statistical Computing, Vienna, Austria) (Appendix E). The result distribution map was presented in different colors, where each color shows specific concentration of the predicted chemical value in whole fruit.

- Accuracy of calibration models for HSI

The methods were the same as the verification of calibration models for NIRS.

CHAPTER 4 RESULTS AND DISCUSSION

4.1 Physical and chemical properties of fresh longan

The physical properties of fresh longan are shown in Table 6. The width of fruits was not significantly different between each batch. It revealed that all sample had similar size which is important for NIRS measurement. Because of the size of object affect to the scattering and of light inside (Nicolaï et al., 2007). According to Thai agricultural standard. (2004), the diameter of fruits in this research are classified into AA grade (>2.5 cm) which is suitable for dried whole longan production.

Batch	N	Height (mm)	Diameter	Width	Weight/fruit
Baton			(mm)	(mm) ^{ns}	(g)
1	242	27.53 <u>+</u> 0.89 ^ª	26.88±1.05 ^ª	31.37 <u>+</u> 1.93	14.35 <u>+</u> 0.72 ^ª
2	242	26.54 <u>±</u> 0.94 ^b	26.16±1.05 ^b	30.78 <u>+</u> 1.21	13.48 <u>+</u> 0.96 ^b
3	198	27.27 <u>±</u> 0.62 ^a	26.85±1.14 ^ª	30.93 <u>+</u> 1.23	13.58 <u>+</u> 1.31 ^b
Average		27.11±0.93	26.63±1.12	31.03 <u>+</u> 1.50	13.80 <u>+</u> 1.09

MILE)

Table 6 Height, diameter, width and weight per fruit of fresh longan

J

^{a-b} Mean values in the same column with different letters are significantly different (P \leq 0.05).

^{ns} Non-significant different (P>0.05).

The chemical quality of the fresh longan can be seen from Table 7. The quality of the fruit in term moisture content (peel, flesh and seed as well as the whole fruit of fresh longan) for all the samples was the same. With regard to Phupaichitkun et al. (2005), initial moisture content of fruits was similar to the results of this study.

longan								_			
						6					
			NO (L L ^{ns}		ns	2	送 (x)		Sucrose	Glucose	Fructose
Batch	Ν	MC peel (%)	MC flesh ^{ns}	MC seed (%)	MC whole ^{ns}	N	TSS (°Brix)	N	(mg/g fresh	(mg/g fresh	(mg/g fresh
			(%)	A					weight)	weight)	weight)
1	16	28.91 <u>+</u> 4.86 ^ª	80.91 <u>+</u> 1.47	39.55 <u>+</u> 1.75°	70.29±1.31	10	19.78 <u>+</u> 1.16 ^{ab}	6	106.44±17.25 ^ª	25.64±4.68 ^{ab}	24.50±4.29 ^{ab}
2	16	33.37±7.15 ^{ab}	81.05±1.13	37.17 <u>±</u> 1.29 ^b	70.75 ±1.37	10	18.60±1.69 ^ª	6	99.66±11.32 ^ª	29.11 <u>±</u> 6.12 ^ª	27.04±6.87 ^ª
3	16	36.29 <u>+</u> 9.65 ^b	80.83 <u>+</u> 1.45	38.76 <u>+</u> 1.72 ^ª	70.86 <u>+</u> 1.50	10	20.65 <u>+</u> 0.92 ^b	6	121.51 <u>+</u> 3.10 ^b	20.36±2.26 ^b	20.08±1.68 ^b
Average		32.86±7.94	80.93 <u>±</u> 1.33	38.49±1.86	70.63 <u>±</u> 1.39		19.67 <u>±</u> 1.52	P	109.20 <u>+</u> 14.71	25.04 <u>±</u> 5.71	23.88±5.37

Table 7 Moisture content (MC) of peel, flesh and seed as well as the whole fruit, TSS, sucrose, glucose and fructose content of fresh flesh

 $^{\rm a-b}$ Mean values in the same column with different letters are significantly different (P \leq 0.05).

^{ns} Non-significant different (P>0.05).



4.2 Near-infrared spectroscopy for determination of moisture content and sugar content in dried whole longan

4.2.1 Diffuse reflectance spectra of fresh and dried whole longan by NIRS

Figure 23 shows the example of original spectra of fresh and dried whole longans obtained from different temperatures for 40 h at the measurement position 1 (P1) in the spectral region of 800-2500 nm. All temperatures of drying showed similar spectrum that has baseline shift from scattering effect due to water and fiber in fruits (Bai et al., 2014). These characteristic bands also found in blueberry fruit (Bai et al., 2014) and apple (Fan et al., 2016). Moreover, there were absorption peaks around 1100-1200 nm which related to C-H stretching of second overtone. These peaks are associated with carbohydrate (e.g. sucrose) or lipids and amino acids (proteins) (Kaewsorn and Sirisomboon, 2014). The region near 1400-1500 nm contains the first overtones O-H stretching that could be refer to water of the fruit (Osborne et al., 1993; Magwaza et al., 2012; Fan et al, 2016). The bands situated between 1700-1800 nm occur to due to the first overtone associated with C-H stretching that represent the structure of CH_2 , CH_3 and cellulose (Osborne et al., 1993). And in the region 1900–2100 nm important bands related to water (Rungpichayapichet et al., 2016),

The example of original spectra of dried whole longans obtained from different positions and drying at 60°C for 40 hours and the average spectrum from all positions are shown in Figure 24. For the spectra observed at the calyx (P1) were different from those observed at other positions. These differences were caused by surface feature at the calyx which was different from the other positions and directly affect its diffuse reflectance (Bai et al., 2014). In addition, it was also due to amount of moisture content at the calyx that has higher moisture content than other positions. The higher moisture content at the calyx might be due to the sample which was turned down to the tray affected the moisture content was released from this part.

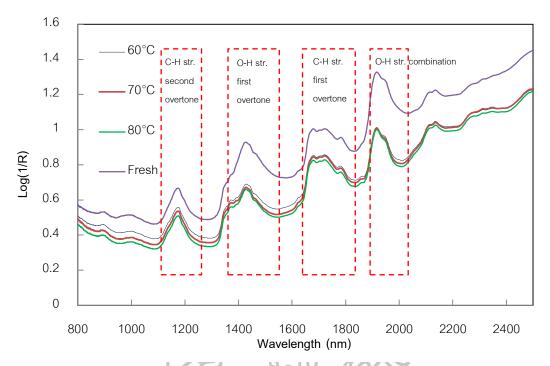


Figure 23 Example of original spectra of fresh and dried whole longans dried at different temperatures for 40 hours at measurement position 1 (P1).

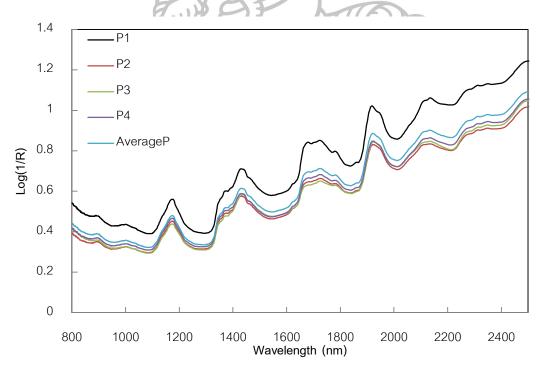
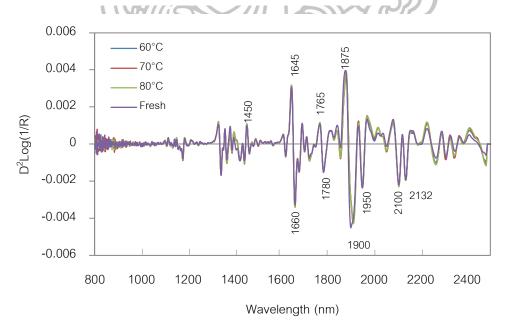
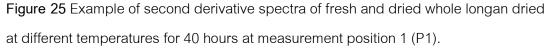


Figure 24 Example of original spectra of dried whole longans obtained from different positions and drying at 60°C for 40 hours.

However, many overlaps in the vibrations of chemical bonds result in large bands and poor peak resolution in original NIR spectra. Thus, using the second derivative techniques, which is one of spectral preprocessing, solved these problems and created a spectrum with specified peaks (Bai et al., 2014). The Savizky-Golay second derivative spectra obtained from fresh and dried whole longans dried at different temperatures for 40 hours at measurement position 1 (P1) are shown in Figure 25. The figure shows eight similar peaks at 1450 nm, 1645 nm, 1660 nm, 1765 nm, 1780 nm, 1875 nm, 1900 nm and 2100 nm. The peak at 1450 nm occurs to be due to the first overtone associated with O-H stretching that represents the structure of water (Osborne et al., 1993; Magwaza et al., 2012; Rungpichayapichet et al., 2016). And the peaks at 1645 nm, 1660 nm, 1765 nm and 1780 nm occur to be due to the first overtone associated with C-H stretching that represent the structure of CH2, CH3 and cellulose (Osborne, 1993). The peaks at 1875 nm and 1900 nm occur to be due to the second overtone associated with C=O stretching and the peak at 2100 nm corresponds to the combination of O-H stretching and 2×C-O stretching that represent the structure of starch (Osborne et al., 1993; Kaewsorn and Sirisomboon, 2014)





4.2.2 Calibration models for moisture content by NIRS

A total of 496 spectra were divided into calibration set (60% of all samples) and validation set (40% of all samples) for building the models. Mean, standard deviation (SD), minimum and maximum of moisture content (%) of longan fruit samples from different fruit parts and the whole fruit showed in Table 8. The range in moisture content of peel was between 3.35-51.74%, the range in flesh was between 9.99-88.62%, the range in seed was between 7.61-49.90%. The range of moisture content in whole fruit was between 8.15-73.36%. The result showed that the flesh part had the widest range of moisture content. During drying, the moisture content of 51.73%, 76.66%, 37.16% and 69.44% for MC peel, MC flesh, MC seed and MC whole, respectively. At the end point of drying, the moisture content of each part were found 3.35%, 13.17%, 8.95% and 9.40% for MC peel, MC flesh, MC seed and MC whole, respectively. Obviously, flesh had the highest moisture content in dried longan while, peel had the lowest value.

				KU I	m	XX))					
Fruit part	N	C	Calibratio	n set (60)%)		Valio	Validation set (40%)				
		Min	Max	Mean	SD		Min	Max	Mean	SD		
Peel	298	3.35	51.74	15.81	9.51	198	3.73	46.51	15.75	9.34		
Flesh	298	9.99	88.62	45.05	25.35	198	11.04	83.21	45.03	25.29		
Seed	298	7.61	49.90	26.60	11.53	198	7.98	47.23	26.58	11.49		
Flesh+seed	298	7.27	71.26	35.06	21.14	198	7.92	70.48	35.07	21.11		
Whole fruit	298	8.15	73.36	37.34	21.87	198	8.77	73.03	37.32	21.85		

Table 8 Mean, standard deviation (SD), minimum and maximum of moisture content (%)for longan fruit samples from different fruit parts and the whole fruit.

N = number of samples

Figure 26(a) and (b) show the distribution of moisture content of peel in longan samples in calibration set and validation set, respectively. It can be seen that there were more samples at low moisture content (10%) than other values because of decreasing of water content in peel during drying. Figure 26(c) and (d) show the distribution of moisture content of flesh in longan samples in calibration set and validation set, respectively. The results also found that there were more samples at low moisture content (20%) than other values because most of fresh flesh of longans became to dried flesh. Figure 26(e) and (f) show the distribution of moisture content of seed in longan samples in calibration set and validation set, respectively. Obviously, most of samples had high moisture content of seed (40%) because the water in seed can only diffuse through the seed stalk that affect the drying rate of this part is very slow (Phupaichitkun et al., 2006). Thus, more samples still had high moisture content. Figure 26 (g) and (h) show the distribution of moisture content of flesh and seed in calibration set and validation set, respectively. And Figure 26 (i) and (j) show the distribution of moisture content of the whole fruit in calibration set and validation set, respectively. The results of these 2 part found that there were more sample at low moisture content (20%) because of decreasing of water content in sample during drying.



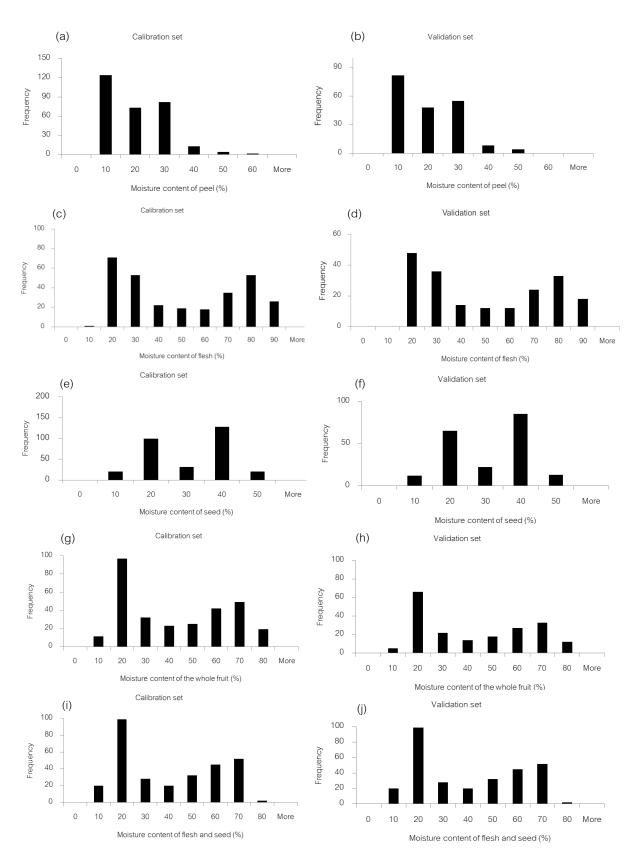


Figure 26 Histogram of distribution of moisture content of peel, flesh, seed, flesh and seed and the whole fruit of samples in calibration set and validation set.

The calibration and validation results associated with the PLS models for moisture content of peel by NIRS are shown in Table 9. The optimum model was selected if the model provided best prediction performance on a validation set which showed high R² and RPD. In addition, Williams (2007) has indicated that an R² of 0.98+ showed that a model is excellent and usable in any application and R² between 0.92 and 0.96 showed that a model is usable in most applications, including that of quality assurance. Furthermore, RPD, which is the ratio of the standard error of performance to the standard deviation of the reference data (Huang and Lu, 2010), is a necessary parameter in assessing the performance of prediction models. An RPD between 1.5 and 2 demonstrates that the model can separate low from high values of the response variable. A value between 2 and 2.5 demonstrates that coarse quantitative predictions are possible, whereas a value between 2.5 and 3 or above corresponds to good and excellent prediction accuracy, respectively (Nicolaï et al., 2007).

In this study, the result showed that the optimum models for moisture content of peel generated from averaging spectra at all positions of measurement by using 5 PLS factors in the range of 1182.7-1333.6 nm 1638.8-2175.0 nm that were pre-treated by the first derivative+SNV methods. This model showed a coefficient of determination (R^2) , root mean squared error of prediction (RMSEP), ratio of standard error of validation to the standard deviation (RPD) and a bias of 0.9850, 1.06%, 8.22 and -0.128% , respectively. Bias is the average difference between actual and NIRS predicted values. According to the best model of peel, It can be seen that the bias value had minus (-0.128%) which means the average actual value from reference method had higher value than the average NIRS predicted value (Nicolaï et al., 2007). Moreover, R² showed that a model is excellent and usable in any application. And RPD values was larger than 3 corresponds to excellent prediction accuracy. Therefore, R² and RPD of these models can be used. The reason might be due to the peel which is external part was able to absorb the NIRS radiation directly. Good prediction results of moisture content of fruit peel also found in Ahmad et al. (2014) that used NIRS for predicting moisture content of mangosteen peel during storage (R = 0.882).

Fruit			Wavelength	PLS	Calibrat	ion set (60%)	Va	alidation set (40)%)	
part	Position	Pre processing	(nm)	factor	R ²	RMSEC(%)	R^2	RMSEP(%)	Bias(%)	RPD
Peel	P1	MSC	1063.4-1836.1	8	0.9232	2.67	0.9601	1.76	-0.1	5.01
			2171.3-2356.9	(73)	TXF)	Ka m				
	P2	First derivative	1063.4-1640.9	10	0.9364	2.44	0.9502	2.04	-0.301	4.53
		(17pts.)	2171.3-2262.3			ד זק				
	P3	First derivative+MSC	1063.4-2175.0	6	0.9272	2.59	0.9560	1.84	-0.0519	4.77
		(17pts.)	Rung .	RI	$\sim M$					
	P4	First derivative+SNV	1063.4-1836.1	6	0.9414	2.33	0.9680	1.60	-0.209	5.64
		(17pts.)	1833.5-2356.9			H C				
	P1P2P3P4	First derivative+MSC	1063.4-1333.6	_10	0.9202	2.69	0.9548	1.94	-0.192	4.73
		(17pts.)	2171.3-2356.9	C D	jee		5)			
	Average P	First derivative+SNV	1182.7-1333.6	5	0.9636	1.83	0.9850	1.06	-0.128	8.22
		(17pts.)	1638.8-2175.0	817	3419	120				

Table 9 Calibration and validation results for moisture content of peel by NIRS.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, SNV: standard normal variate, 17 pts: 17 smoothing points, P1: calyx, P2: bottom, P3 and P4: 2 side of cheek, P1P2P3P4: all positions and Average P: average all positions.

The scatter plot demonstrating the measured value of moisture content and the prediction value for the peel model is shown in Figure 27. It can be seen that the points close to target line that mean the NIRS predicted values were similar to the measured values. However, most points were quite under the target line that affected to this result had negative bias value (Nicolaï et al., 2007).

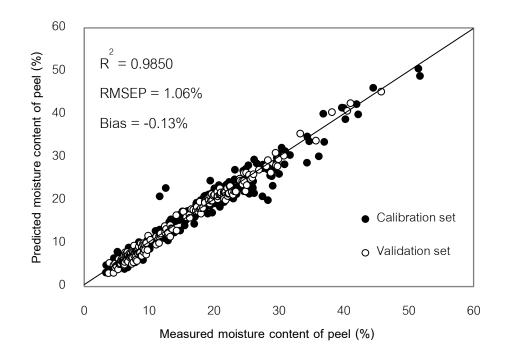


Figure 27 Scatter plot of moisture content of peel from the model built from averaging spectra.

The calibration and validation results associated with the PLS models for moisture content of flesh by NIRS are shown in Table 10. The result showed that the optimum models generated from averaging spectra at all positions of measurement were by using 10 PLS factors in the range of 1063.4-1640.9 nm and 2171.3-2356.9 nm that were pre-treated by the first derivative+SNV methods. This model shows R^2 , RMSEP, RPD and a bias of 0.9513%, 5.53, 4.53 and 0.085%, respectively. The result of R^2 showed that this model is usable in most applications, including that of quality assurance (Williams, 2007). And RPD values was larger than 3 corresponds to excellent prediction accuracy (Nicolaï et al., 2007). Previously, NIRS was also applied to determine moisture content in frozen guava and yellow passion fruit pulps (Alamar et al., 2016) that reported the good prediction and R^2 of guava and passion fruit were found 0.94 and 0.93, respectively. While RMSEP of guava and passion fruit were found 0.24 and 0.41, respectively that were less than longan model in this study. The reason might be due to guava and passion had narrower range of moisture content.



Fruit			Wavelength	PLS	Calibra	ation set (60%)	V	′alidation set (4	40%)	
part	Position	Pre processing	(nm)	factor	R^2	RMSEC(%)	R^2	RMSEP(%)	Bias(%)	- RPD
Flesh	P1	First derivative+MSC	1063.4-1640.9	8	0.9169	7.41	0.9277	6.76	0.401	3.73
		(17pts.)	1833.5-2175.0	43=						
	P2	SNV	1063.4-1183.8	10	0.9355	6.55	0.9341	6.43	0.232	3.90
			1638.8-1733.0	Y I	JI 19	3/ 4				
			2171.3-2356.9	P	J.J.	\bigcirc				
	P3	First derivative+SNV	1063.4-2175.0	6	0.9278	6.88	0.9163	7.25	0.162	3.46
		(17pts.)	2025	Sm	Y P	5				
	P4	First derivative+MSC	1332.2-1640.9	10	0.9423	6.2	0.9407	6.09	0.57	4.13
		(17pts.)	2258.4-2356.9		EC					
	P1P2P3P4	First derivative+MSC	1063.4-1640.9	10	0.9137	7.45	0.9367	6.37	-0.195	3.98
		(17pts.)	2171.3-2356.9	30						
	Average P	First derivative+SNV	1063.4-1640.9	10	0.9596	5.18	0.9513	5.53	0.085	4.53
		(17pts.)	2171.3-2356.9	570	194					

Table 10 Calibration and validation results for moisture content of flesh by NIRS.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, SNV: standard normal variate, 17 pts: 17 smoothing points, P1: calyx, P2: bottom, P3 and P4: 2 side of cheek, P1P2P3P4: all positions and Average P: average all positions

The scatter plot for the moisture content of flesh model is shown in Figure 28. The result found that most points close to the target line that means the NIRS predicted values were similar to the measured values (Nicolaï et al., 2007). While, some points were spread out from the target line which related to rather high error from this model.

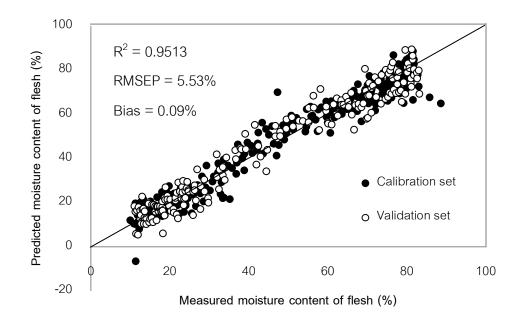


Figure 28 Scatter plot of moisture content of flesh from the model built from averaging spectra.

However, RMSEP as an error of this model was rather high when applied for determining moisture content of flesh of dried whole longan fruit in exporting routines (<18%). The reason might be due to the wide range of the moisture content of flesh in fresh to dried longan (9.99-88.62%) and the penetration of light which had to pass through the peel and air gap between peel and dried flesh of longan affected to some errors of prediction model. To reduce the errors, the lower moisture content (such as 9-40% moisture content of flesh) could be used for developing new model. Table 11 showed that the errors were reduced but the performance of the model was also reduced. Thus, the old model was chosen for predicting the unknown samples.

Fruit			Wavelength	PLS	Calibration set (60%)		Va	%)			
position Pre part		Pre processing	(nm)	factor	R ²	RMSEC(%)	R^2	RMSEP(%)	Bias(%)	- RPD	
Flesh	Average	SNV	1063.4-1333.6	8 10	0.8934	2.15	0.799	2.47	-0.403	2.26	
(9-40%)			2171.3-2356.9	3K	XX	ELICA					

Table 11 Calibration and validation results for moisture content of flesh (9-40%) by NIRS.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, SNV: standard normal variate, P1: calyx, P2: bottom, P3 and P4: 2 side of cheek, P1P2P3P4: all positions and Average P: average all positions



The calibration and validation results associated with the PLS models for moisture content of seed by NIRS are shown in Table 12. The result showed that the optimum models generated from averaging spectra at all positions of measurement were by using 7 PLS factors in the range of 1332.3-1640.9 nm that were pre-treated by the first derivative+SNV methods. This model showed R², RMSEP, RPD and a bias of 0.9577, 2.32%, 4.93 and -0.37%, respectively. The result of R² showed that this model is usable in most applications, including that of quality assurance (Williams, 2007). And RPD values was larger than 3 corresponds to excellent prediction accuracy (Nicolaï et al., 2007). Moreover, the negative bias was found in this result which means the average actual value from reference method had higher value than the average NIRS predicted value (Nicolaï et al., 2007).

Good prediction result was found in Posom et al. (2016) that used NIRS for determining moisture content of Leucaena leucocephala pellets ($R^2 = 0.995$ and RPD = 13.9). Nevertheless, the error of longan seed model might be occurred by the penetration of light from peel to the seed of longan and the characteristic of seed which has glossy seed coat (Phupaichitkun et al., 2006) can reflect the light related to the error in spectrum acquisition (Osborne, 1993).



Fruit	Deeitien		Wavelength	PLS	Calibra	ation set (60%)	Va	alidation set (40	0%)	
part	Position	Pre processing	(nm)	factor	R ²	RMSEC(%)	R^2	RMSEP(%)	Bias(%)	RPD
Seed	P1	MSC	1332.3-1640.9	10	0.9231	3.29	0.9441	2.67	-0.411	4.28
	P2	SNV	1332.3-1640.9 1833.5-2175	6	0.9041	3.61	0.9342	2.90	-0.678	4.01
	P3	First derivative+MSC	1833.5-2356.9	9	0.9250	3.21	0.9551	2.39	-0.497	4.83
	P4	(17pts.) MSC	1063.4-1836.1	10	0.9282	3.14	0.9428	2.72	-0.259	4.20
	P1P2P3P4	First derivative+MSC	1332.3-1640.9	10	0.9182	3.31	0.9355	2.88	-0.304	3.96
		(17pts.)	2171.3-2356.9	「「 広		P	\mathbf{S}			
	Average P	First derivative+SNV	1332.3-1640.9	7	0.9336	3.01	0.9577	2.32	-0.37	4.93
		(17pts.)	Vn	גרנ	519	3.01				

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Table 12 Calibration and validation results for moisture content of seed by NIRS.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, SNV: standard normal variate, 17 pts: 17 smoothing points, P1: calyx, P2: bottom, P3 and P4: 2 side of cheek, P1P2P3P4: all positions and Average P: average all positions

The scatter plot for the moisture content of seed model is shown in Figure 29. It can be seen that most points close to the target line that means good prediction of model (Nicolaï et al., 2007). Obviously, some points were under the target line that means the average actual value from reference method had higher value than the average NIRS predicted value and correspond to negative bias from the model result (Nicolaï et al., 2007).

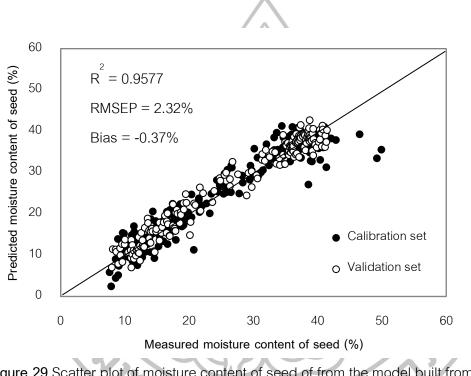


Figure 29 Scatter plot of moisture content of seed of from the model built from averaging spectra.

For flesh and seed part, the calibration and validation results associated with the PLS models are shown in Table 13. The result showed that the optimum models generated from averaging spectra at all positions of measurement were by using 9 PLS factors in the range of 1332.3-1471.4 nm and 1833.5-2064.2 nm that were pre-treated by MSC methods. This model shows R^2 , RMSEP, RPD and a bias of 0.9604, 4.13%, 5.10 and -0.731%, respectively. The result of R^2 showed that this model is usable in most applications, including that of quality assurance (Williams, 2007). RPD values was

larger than 3 corresponds to excellent prediction accuracy (Nicolaï et al., 2007). Moreover, the fairly high error also found same as the results of flesh model and seed model in previous table (Table 11 and 12).

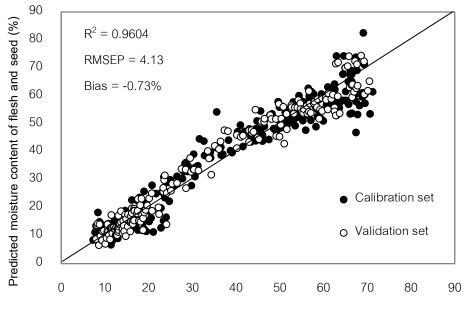


Fruit			Wavelength	PLS	Calibra	tion set (60%)	V	alidation set (4	0%)	
part	Position	Pre processing	(nm)	factor	R^2	RMSEC(%)	R^2	RMSEP(%)	Bias(%)	RPD
Flesh	P1	SNV	1063.4-1836.1	A	0.9168	6.17	0.9386	5.19	-1.06	4.12
and			2171.3-2356.9	33		E				
seed	P2	Second derivative	1332.3-2356.9	9	0.9102	6.43	0.9295	5.55	-0.292	3.77
		(17pts.)	A A	9	JI IA	317				
	P3	First derivative+SNV	1063.4-1640.9	10	0.9102	6.38	0.9237	5.82	-0.438	3.63
		(17pts.)	1833.5-2356.9	3F	M	ADD				
	P4	First derivative+SNV	1063.4-1640.9	9	0.9236	5.93	0.9484	4.74	-0.627	4.44
		(17pts.)	1833.5-2356.9	\mathbb{Z}		YY,				
	P1P2P3P4	First derivative+SNV	1063.4-1640.9	10	0.8919	6.97	0.9310	5.51	-0.540	3.83
		(17pts.)	1833.5-2175	ΥG	20	P/	5			
	Average P	MSC	1332.3-1471.4	9	0.9451	5.03	0.9604	4.13	-0.731	5.10
			1833.5-2064.2		E.a	20%	7			

Table 13 Calibration and validation results for moisture content of flesh and seed by NIRS.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, SNV: standard normal variate, 17 pts: 17 smoothing points, P1: calyx, P2: bottom, P3 and P4: 2 side of cheek, P1P2P3P4: all positions and Average P: average all positions

According to the result from Table 13, the scatter plot for the moisture content of flesh and seed model is shown in Figure 30. Most points close to the target line that means good prediction of model (Nicolaï et al., 2007).



Measured moisture content of flesh and seed (%)

Figure 30 Scatter plot of moisture content of flesh and seed from the model built from averaging spectra.

The calibration and validation results associated with the PLS models for moisture content of the whole fruit by NIRS are shown in Table 14. The result represented that the optimum models for moisture content of whole fruit generated from averaging spectra at all positions of measurement were by using 10 PLS factors in the range of 1289.8-1640.9 nm and 2060.9-2356.9 nm that were pre-treated by the first derivative+SNV methods. This model showed R^2 , RMSEP, RPD and a bias of 0.9673, 3.89%, 5.53 and -0.138%, respectively. According to Williams (2007) and Nicolaï et al. (2007), R^2 and RPD of this model can be used in most applications, including that of quality assurance. Moreover, good prediction of moisture content (R^2 was 0.880 and

RMSEP was 1.35%) also found in Morales-Sillero et al. (2011) that applied NIRS for assessing moisture content of table olive.



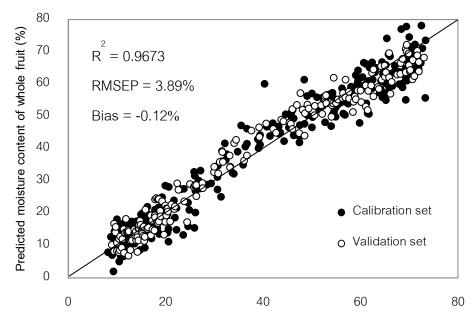
Fruit	Position		Wavelength	PLS	Calibra	ation set (60%)	Va	alidation set (4	0%)	RPD
part	Position	Pre processing	(nm)	factor	R^2	RMSEC(%)	R^2	RMSEP(%)	Bias(%)	- RPD
Whole	P1	MSC	1332.3-1471.4	10	0.9459	5.18	0.9412	5.25	-0.334	4.13
fruit			1833.5-2064.2		FA	6				
	P2	MSC	1063.4-1333.6	3	0.8933	3 7.18	0.9586	4.34	-0.355	4.93
			1638.8-2175.0			ך זק				
			2171.3-2356.9	Ð						
	P3	SNV	1638.8-2175.0	9	0.9129	6.56	0.9354	5.46	-0.775	3.97
				Z]])	PA)			
	P4	First derivative+MSC	1063.4-1640.8	10	0.9280) 5.97	0.9626	4.20	-0.24	5.18
		(17pts.)	2171.3-2356.9	ぼ	32	ST/	7			
	P1P2P3P4	SNV	1063.4-1836.1	10	0.9097		0.9419	5.19	-0.313	4.15
			2171.3-2356.8			111				
	Average P	First derivative+SNV	1289.8-1640.9	10	0.9538	5 4.80	0.9673	3.89	-0.138	5.53
		(17pts.)	2060.9-2356.9							

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Table 14 Calibration and validation results for moisture content of whole fruit by NIRS.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, SNV: standard normal variate, 17 pts: 17 smoothing points, P1: calyx, P2: bottom, P3 and P4: 2 side of cheek, P1P2P3P4: all positions and Average P: average all positions

The scatter plot for the moisture content of the whole fruit model is shown in Figure 31. Most points also close to the target line that means good prediction of model (Nicolaï et al., 2007). However, the error points also found that related to high RMSEP in the prediction model.

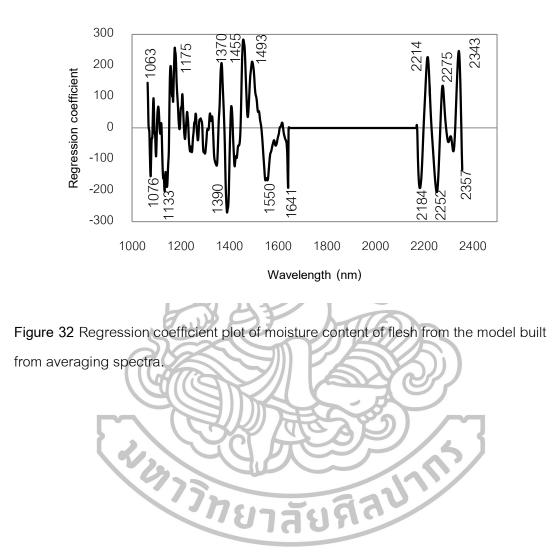


Measured moisture content of whole fruit (%)

Figure 31 Scatter plot of moisture content of whole fruit from the model built from averaging spectra.

Figure 32 shows the regression coefficient plots for moisture content of flesh from the model built from averaging spectra. The strong effects of the bond vibration on the predictions of moisture content are displayed by the high peaks of the regression coefficient plots. This plot had wavelengths in the range of 1063.4-1640.9 nm and 2171.3-2356.9 nm. As mentioned by Osborne et al. (1993), the apparent peaks, their bond vibration and the structure in the regression coefficient plot are shown in Table 15 which displays the intensity of the peaks in descending order. It was observed that the vibration band of O-H which appeared as high peak (1455 nm) indicated that water had an important influence on the prediction of moisture content of longan (Osborne et al.,

1993; Magwaza et al., 2012; Rungpichayapichet et al., 2016). There were also high absorption peaks owing to the C-H which related to the structure of starch and sugar in sample (1175 nm and 1390 nm) (Rungpichayapichet et al., 2016).



Wavelength	Wavelength (nm)	Bond vibration	Structure
(nm)	from references		
1063	1060	N-H str. second	RNH ₂
		overtone	
1076	1080	2×C-H str.+2×C-C str.	benzene
1133	1143	C-H str. second	aromatic
		overtone	
1175	1170	C-H str. second	HC=CH
	Gor 14	overtone	
1370	- 787	HELT STY	
1390	1395	2×C-H str.+ C-H def.	CH ₂
1455	1450	O-H str. first overtone	starch, H ₂ 0
1493	1490	O-H str. first overtone	cellulose
		(intramol. H-bond)	
1550			2
1641	1645	C-H str. first overtone	aromatic
2184	2180	2×amide I + amide III	protein
2214	130	5.220	
2252	2252	O-H str. + O-H def.	starch
2275	2276	O-H str. + C-C def.	starch
2343	2347	CH_2 sym. Str. += CH_2	HC=CHCH ₂
		def.	
2357	-	-	-

Table 15 The absorption bands with high regression coefficients of model for moisturecontent of flesh of the longan fruits.

Source: Osborne et al. (1993)

Figure 33 shows the regression coefficient plot of for moisture content of the whole fruit from the model built from averaging spectra. This plot had wavelengths in the range of 1289.8-1640.9 nm and 2060.9-2356.9 nm. The obvious peaks and their bond vibration in the regression coefficient plot are shown in Table 16 which indicates the intensity of the peaks in descending order. It was observed that the vibration of O-H also appeared as high peak (1449 nm), indicating an implicit high influence on the prediction of the moisture content of the whole longan fruit (Osborne et al., 1993; Magwaza et al., 2012; Rungpichayapichet et al., 2016). Moreover, there is also a high peak in the absorption band for cellulose and starch at 1494 nm and 1526 nm, respectively (Onsawai and Sirisomboon, 2015).

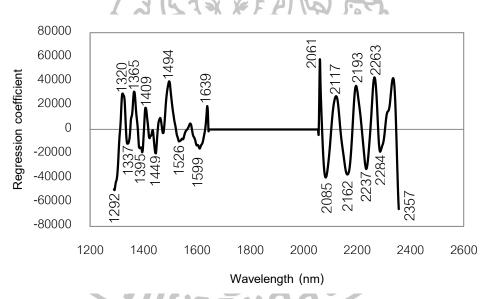


Figure 33 Regression coefficient plot of moisture content of the whole fruit from the model built from averaging spectra.

Wavelength	Wavelength (nm)	Bond vibration	Structure
(nm)	from references		
1292	-	-	-
1320	-	-	-
1337	-	-	-
1365	1360	2×C-H str.+ C-H def.	CH ₃
1395	1395	2×C-H str.+ C-H def.	CH ₂
1409	1410	O-H str. first overtone	ROH
1449	1450	O-H str. first overtone	starch, H_20
1494	1490	O-H str. first overtone	cellulose
		(intramol. H-bond)	
1526	1528	O-H str. first overtone	starch
	Sac	(intramol. H-bond)	2
1578	1580	O-H str. first overtone	starch,glucose
_		(intramol. H-bond)	2
1599		(GEZE)	
1639			\sim
2016	2080	- 220	-
2080	2080	O-H str.+ O-H def.	ROH, sucrose,
			starch
2117	2110	N-H sym. Str. + amide	CONH ₂ , CONHR
		III	
2162	2160	2×amide I + amide III	CONHR
2193	2190	CH ₂ asym. Str. + C=str.	HC=CH
2237	2242	N-H str. + NH_3^+ def.	amino acid
2284	2280	C-H str. + C-H def.	CH ₃

Table 16 The absorption bands with high regression coefficients of the PLS model formoisture content of the whole longan fruits.

Wavelength	Wavelength (nm)	Bond vibration	Structure
(nm)	from references		
2357	2352	C-H def. second	cellulose
		overtone	

Source: Osborne et al. (1993)

For checking accuracy of the model, the average spectra of all position of 120 dried whole longan fruits from this study which had moisture content of flesh in the range of 14-24% were predicted by the best fit model for moisture content of flesh. This moisture range could be in the range of the final moisture content of flesh when the drying stopped at the drying manufacturing. Table 17 represents the comparison of measured and predicted moisture content of flesh. To compare the methods between destructive and NIRS measurement, the paired *t* test method was applied. The result showed that the p- value was 0.513 which was higher than 0.05 (data not shown). The paired *t* test result demonstrated that the predicted values obtained from the NIR spectroscopy and the measured values from reference method did not show significant difference at 95% confidence interval (p>0.05).

N	Measured (%)	Prediction (%)	% error	Ν	Measured (%)	Prediction (%)	% error
1	14.21	17.17	20.86	31	18.42	23.40	27.07
2	14.42	18.32	27.07	32	18.54	16.49	11.06
3	14.50	14.29	1.42	33	18.58	18.72	0.75
4	14.60	13.25	9.27	34	18.69	16.94	9.38
5	14.61	13.13	10.14	35	18.77	14.38	23.38
6	14.66	19.48	32.91	36	18.81	18.19	3.31
7	15.09	13.87	8.08	37	19.24	17.58	8.62
8	15.28	14.32	6.27	38	19.27	24.67	28.06
9	15.62	18.43	18.01	39	19.28	21.74	12.77
10	15.72	19.06	21.28	40	19.50	22.71	16.46
11	15.75	17.06	8.30	41	19.52	20.59	5.51
12	16.05	15.62	2.70	42	19.58	23.81	21.62
13	16.07	19.05	18.56	43	20.04	16.75	16.42
14	16.27	14.60	10.25	44	20.06	18.68	6.86
15	16.48	15.26	7.43	45	20.44	21.87	6.99
16	16.62	17.82	7.25	46	20.68	22.18	7.23
17	16.63	18.43	10.82	47	20.80	23.78	14.34
18	16.79	14.92	11.16	48	21.16	22.58	6.71
19	16.88	16.15	4.33	49	21.16	24.22	14.45
20	17.31	18.11	4.65	50	21.24	19.22	9.52
21	17.32	16.42	5.19	51	21.57	21.74	0.81
22	17.50	17.82	1.83	52	21.62	23.35	7.98
23	17.63	16.52	6.30	53	21.68	22.53	3.92
24	17.64	17.42	1.26	54	21.90	18.93	13.55
25	17.72	15.62	11.87	55	22.01	23.29	5.82
26	17.90	19.79	10.55	56	22.02	21.48	2.47
27	18.04	21.19	17.46	57	22.19	23.04	3.81
28	18.11	19.16	5.83	58	22.32	21.39	4.15
29	18.11	16.50	8.87	59	22.50	15.07	33.03
30	18.11	13.96	22.93	60	22.70	26.18	15.33

Table 17 The comparison of measured and predicted moisture content of flesh of 120dried whole longan fruits from this study by the best fit model.

Ν	Measured (%)	Prediction (%)	% error	Ν	Measured (%)	Prediction (%)	% erro
61	22.82	27.86	22.10	91	17.52	18.45	5.32
62	22.85	28.76	25.86	92	17.84	15.68	12.09
63	23.12	21.22	8.21	93	17.84	16.11	9.70
64	23.20	25.66	10.62	94	18.06	18.06	0.01
65	23.23	28.96	24.68	95	18.07	20.63	14.16
66	23.48	22.60	3.74	96	18.15	16.41	9.60
67	23.48	21.03	10.45	97	18.63	22.26	19.48
68	23.50	25.20	7.24	98	18.69	14.26	23.70
69	23.64	15.87	32.88	99	18.83	21.38	13.56
70	23.87	23.80	0.28	100	19.39	23.05	18.87
71	23.92	26.41	10.41	101	19.41	21.48	10.66
72	23.99	19.14	20.22	102	19.61	19.31	1.53
73	24.05	18.75	22.04	103	19.77	18.67	5.55
74	24.07	24.72	2.71	104	20.12	21.49	6.84
75	14.05	13.03	7.29	105	20.24	16.29	19.5
76	14.06	10.28	26.90	106	20.95	17.19	17.94
77	14.60	19.65	34.60	107	20.98	15.54	25.9
78	14.81	11.12	24.90	108	21.41	21.08	1.52
79	15.00	12.63	15.80	109	21.43	22.24	3.77
80	15.45	13.13	15.03	110	21.74	24.50	12.7
81	15.49	19.13	23.50	111	22.56	25.13	11.3
82	15.79	11.22	28.89	112	22.57	25.02	10.8
83	15.92	19.04	19.57	113	22.86	22.18	2.98
84	16.28	15.91	2.24	114	23.11	27.18	17.6
85	16.33	18.36	12.41	115	23.24	24.59	5.81
86	16.65	12.11	27.26	116	23.33	29.88	28.0
87	16.68	18.31	9.74	117	23.57	18.87	19.9
88	16.96	18.30	7.91	118	23.60	21.75	7.82
89	17.10	16.04	6.19	119	23.93	22.22	7.16
90	17.51	17.29	1.26	120	23.95	25.32	5.72
				Average	e 19.24	19.42	12.4

Table 17 The comparison of measured and predicted moisture content of flesh of 120samples by PLS models (cont.).

Moreover, the best fit model was applied to predict moisture content of flesh of 20 unknown dried longan samples from the market. Table 18 shows the comparison of measured and predicted moisture content of flesh of the unknown samples by the best fit model of flesh part. To compare the methods between destructive and NIRS measurement, the paired *t* test method was applied. The result showed that the p-value was 0.135 which was higher than 0.05 (data not shown). It can be concluded that the predicted values obtained from the NIR spectroscopy and the measured values from reference method did not show significant difference at 95% confidence interval (p>0.05).



No	Measured (%)	Prediction (%)	% error	
1	27.10	24.73	8.76	
2	25.93	22.62	12.78	
3	28.23	29.17	3.33	
4	27.49	27.99	1.80	
5	26.62	23.88	10.31	
6	27.54	26.71	3.01	
7	25.69	22.84	11.11	
8	29.26	23.61	19.31	
9	28.87	33.21	15.04	
10	27.41	28.81	5.09	
11	28.14	30.54	8.52	
12	26.27	31.57	20.18	
13	28.73	34.35	19.58	
14	26.76	32.18	20.26	
15	26.28	28.43	8.21	
16	26.28 28.15	34.74	23.44	
17	25.68	27.88	8.57	
18	25.32	25.09	0.90	
19	28.17	28.96	2.78	
20	25.79	30.25	17.28	
Average	27.17	28.38	11.01	

Table 18 The comparison of measured and predicted moisture content of flesh of 20unknown samples by the best fit models.

To ensure the accuracy of prediction PLS model of flesh and other parts, bias checking, SEP checking and slope checking methods from International standard ISO 12099 were used. These methods can demonstrate that predicted value obtained from the instrument were not different from actual values significantly at 95% confidence interval. Therefore, the models that pass all tests are usable in any application.

It can be seen in Table 19, the results showed that NIRS moisture content of peel, flesh and the whole fruit models passed bias checking, SEP checking and slope checking method. However, NIRS moisture content of seed and flesh+seed models did not pass bias checking that mean the results show significant difference between NIRS predicted and measured values. The reason might be due to the gloss of black seed coat of the longan fruit which can reflect the light and make some errors in NIRS measurement. The details of verification of each method were illustrated in Appendix D.

 Table 19 Verifying the accuracy by bias checking, SEP checking and slope checking

 method for NIRS moisture content models.

Model	Bias checking	SEP checking	Slope checking
NIRS moisture content of peel	资产		} √
NIRS moisture content of flesh	ไยาลัย	82121	\checkmark
NIRS moisture content of seed	×	\checkmark	\checkmark
NIRS moisture content of flesh and seed	×	\checkmark	\checkmark
NIRS moisture content of the whole	\checkmark	\checkmark	\checkmark
fruit			

4.2.3 Calibration models for TSS of flesh by NIRS

A

A total of 186 spectra were divided into calibration set (60% of all samples) and validation set (40% of all samples) for building the TSS models. Mean, standard deviation (SD), minimum and maximum of TSS of flesh of longan fruit samples are shown in Table 20. The range of TSS in flesh of samples were 13-98 °Brix in calibration set and 18-90 °Brix in validation set.

Table 20 Mean, standard deviation (SD), minimum and maximum of TSS of flesh in

longan fruit sam	ples.					
Parameter	Data set		Min	Max	Mean	SD
TSS (°Brix)	Calibration (60%)	112	13	98	56.71	24.00
	Validation (40%)	74	18	90	55.64	23.43
N = number of samples	4	R	MY			

R

Figure 34 (a) and (b) show the distribution of TSS of flesh in longan samples in calibration set and validation set, respectively. It can be seen that there were more samples at a low TSS (30-40 °Brix) and high TSS (80-90 °Brix) than other values. According to Thai Agricultural Standard (2006), TSS of the flesh must not lower than 80 °Brix for flesh dried longan and 76 °Brix for flesh of dried whole longan for exporting.

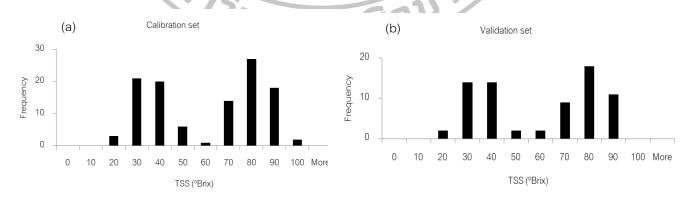


Figure 34 Histrogram of the distribution of TSS of flesh of samples in calibration and validation set.

The calibration and validation results with the PLS models for TSS of flesh in longan fruit samples are shown in Table 21. The best model was obtained by averaging spectra at all positions of measurement which was pretreated by SNV preprocessing technique and using 4 PLS factors in the range of 1833.5-2175.0 nm. This model shows R2, RMSEP, RPD and a bias of 0.9219, 6.52 °Brix, 3.59 and -0.454 °Brix, respectively. This model showed the RPD value above 3 which corresponds to excellent prediction accuracy (Nicolaï et al., 2007). However, rather high RMSEP in TSS of flesh might be due to the penetration of light which had to pass through the peel and air gap between peel and dried flesh of longan affected to some errors of prediction model as mentioned in previous result about moisture content of flesh part.

Recently, NIRS was also used to assess TSS in Japanese plums, navel oranges and mangoes (Louw and Theron, 2010; Liu et al., 2013; Rungpichayapichet et al., 2016) that reported the good prediction and R^2 were shown above 0.80.

Figure 35 shows the relationship between measured and predicted TSS of flesh from the model built from averaging spectra. It can be seen that the points close to the target line that mean this model had good prediction. Some points were found under the target line that related to negative bias of the model (Nicolaï et al., 2007).



			PLS	Calibr	ation set (60%)		Validation cot (4	00/)	
Position	Pre processing	Wavelength	PLS	Calibra	ation set (60%)		Validation set (4	076)	RPD
		(nm)	factor	R ²	RMSEC(°Brix)	R^2	RMSEP(°Brix)	Bias(°Brix)	
P1	First derivative+SNV (17pts.)	1332.3-1640.9		0.9111	7.39	0.8751	8.22	-0.434	2.83
P2	Second derivative (17pts.)	1730.7-1836.1	6	0.8659	9.04	0.8810	8.03	-0.589	2.91
		2171.3-2356.9	hol		()				
P3	Second derivative (17pts.)	1833.5-2175.0	2	0.8386	9.73	0.8941	7.55	0.289	3.08
		AC			RA				
P4	MSC	1332.3-1640.9	10	0.9142	7.37	0.9131	6.88	-0.724	3.41
		2171.3-2356.9							
P1P2P3P4	MSC	1063.4-1640.9	10	0.8854	8.11	0.9049	7.29	-0.558	3.25
		2171.3-2356.9	U.D		5/5				
Average P	SNV	1833.5-2175.0	4	0.8947	7.93	0.9219	6.52	-0.454	3.59

 $\mathbf{\Lambda}$

 Table 21 Calibration and validation results for TSS in flesh of dried whole longan by NIRS.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, SNV: standard normal variate, 17 pts: 17 smoothing points, P1: calyx, P2: bottom, P3 and P4: 2 side of cheek, P1P2P3P4: all positions and Average P: average all positions

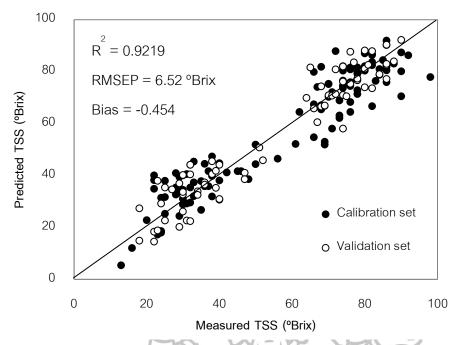


Figure 35 Scatter plot of TSS of flesh from the model built from averaging spectra.

The regression coefficient plot of the best fit TSS model is shown in Figure 36. This plot had wavelengths in the range of 1833.5-2175.0 nm. The important wavelengths for TSS were 1873, 1898, 1926, 1947, 1981, 2003, 2064, 2110 and 2160 nm. These wavelengths represented -H and -OH functional groups which are related to carbohydrates (starches and sugars), water and organic acids (Rungpichayapichet et al., 2016).

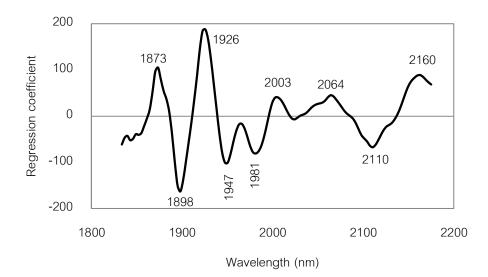


Figure 36 Regression coefficient plot of TSS of flesh from the model built from averaging spectra.

Table 22 shows the verification of the accuracy of TSS model by bias checking, SEP checking and slope checking method. As demonstrated, the model passed all verification methods. NIRS predicted values were not different from actual values from standard method significantly at 95% confidence interval. The details of verification of each method were illustrated in Appendix D.

 Table 22 Verifying the accuracy by bias checking, SEP checking and slope checking

 method for TSS model

Model	Bias checking	SEP checking	Slope checking
NIRS TSS in flesh	\checkmark	\checkmark	\checkmark

4.2.4 Calibration models for sugar content of flesh by NIRS

A total of 181 spectra were divided into calibration set (60% of all samples) and validation set (40% of all samples) for building the models. Mean, standard deviation (SD), minimum and maximum of sugar content of longan fruit samples are showed in Table 23. The range of sucrose was 12.47-593.92 mg/g fresh weight, the range of glucose was 11.05-148.96 mg/g fresh weight, the range of fructose was 17.84-157.70 mg/g fresh weight and the range of total sugar was 116.94-655.84 mg/g fresh weight. Compared in 3 type of sugar, it can be seen that sucrose had the widest range of sugar content in flesh of longan. During drying, each sugar was increased. For example, fresh longan had the sugar content of 125.51 mg/g fresh weight, 21.46 mg/g fresh weight, 21.23 mg/g fresh weight and 168.20 mg/g fresh weight for sucrose, glucose, fructose and total sugar content, respectively. At the end point of drying, each sugar content were found 388.93 mg/g fresh weight, 76.21 mg/g fresh weight, 91.53 mg/g fresh weight and 556.66 mg/g fresh weight for sucrose, glucose, fructose and total sugar content, respectively. Obviously, sucrose had the hightest content in flesh of dried longan while, glucose had the lowest content. The lowest value of glucose might be due to glucose was used as a substrate for generating Maillard reaction in dried longan (Rungtip, 2006). *ระหว่าท*ยาลัยศิลปาก

Sugar		Calibration set (60%)						Validatio	n set (40%)	
(mg/g fresh	N.	Min	Max	Mean	SD	N	Min	Max	Mean	SD
weight)		IVIII I	WIGA	woon		SIE	IVIII I	IVIAA	Wiedh	00
Sucrose	109	12.47	593.92	288.78	138.80	72	79.28	543.18	288.32	133.24
Glucose	109	11.05	148.96	58.21	30.13	72	15.65	133.51	57.94	29.43
Fructose	109	17.84	157.70	73.18	35.10	72	19.25	147.45	72.99	34.38
Total sugars	109	116.94	655.84	419.36	172.91	72	145.20	647.75	420.38	171.47

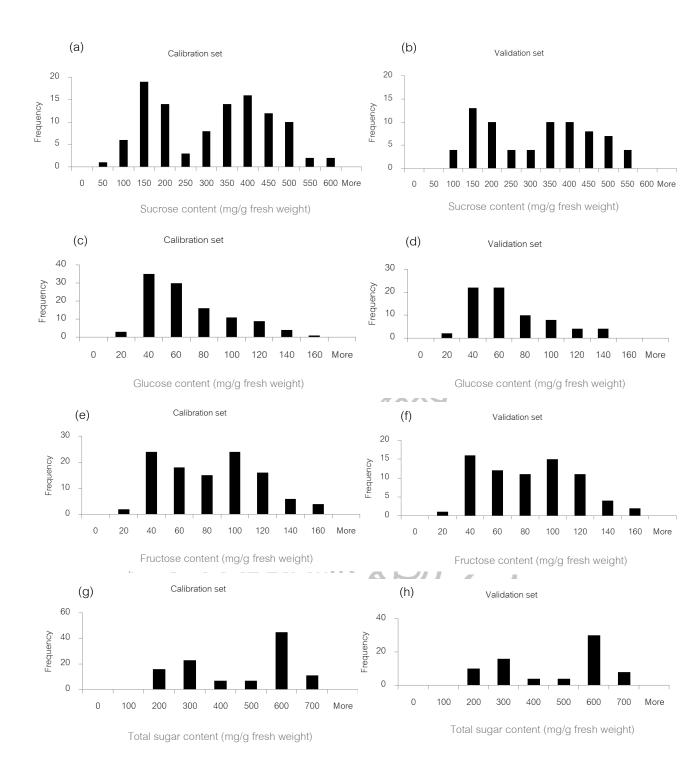
 Table 23 Mean, standard deviation (SD), minimum and maximum of sugar content of flesh in longan fruit samples.

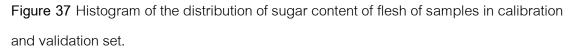
N = number of samples



Figure 37 (a) and (b) show the distribution of sucrose content of flesh in longan samples in calibration set and validation set, respectively. Obviously, there were more samples in the range of 150-200 mg/g fresh weight and 350-450 mg/g fresh weight than other values. Figure 37 (c) and (d) show the distribution of glucose content of flesh in calibration set and validation set, respectively. It can be seen that there were more sample at low content (40-60 mg/g fresh weight) than other values. Figure 37 (e) and (f) show the distribution for fructose in calibration set and validation set, respectively. These histograms represent that the data were evenly distributed. Figure 37 (g) and (h) show the distribution for total sugar in calibration set and validation set, respectively. They show more samples in high total sugar content (600 mg/g fresh weight) than other values.







4.2.4.1 Sucrose

The calibration and validation results with the PLS models for sucrose content of flesh in longan fruit samples are shown in Table 24. Averaging spectra at all positions of measurement and SNV preprocessing technique by using 6 PLS factors in the range of 1063.4-1836.1 and 2171.3-2356.9 nm were provided the best prediction which gave R^2 , RMSEP, RPD and a bias of 0.8343, 53.8 mg/g fresh weight, 2.48 and -7.26 mg/g fresh weight, respectively. Obviously, the result of the model showed negative bias value which means the average actual value from reference method had higher value than the average NIRS predicted value (Nicolaï et al., 2007). It is worth noting that Williams (2007) had demonstrated that R^2 of 0.83-0.90 indicates that a model was usable with caution for most applications, including research and RPD of 2.5 corresponds that the model had good prediction accuracy. In addition, good prediction model was also found in Liu et al. (2006) that applied NIRS for determination of sucrose in apple and R was shown 0.969 and RMSEP of 0.335%.

Figure 38 shows the relationship between measured sucrose content value and predicted sucrose content value from the best model. It clearly showed that the data slightly scattered and some data were under the target line.



		Wavelength	PLS	Calibrat	ion set (60%)	Validation set (40%)			
Position	Pre processing	(nm)	(Λ)	R^2	RMSEC	R^2	RMSEP	Bias	RPD
		(1111)	factor		(mg/g fresh weight)	ĸ	(mg/g fresh weight)	(mg/g fresh weight)	
P1	SNV	1063.4-2175.0	6	0.7247	74.9	0.8281	54.8	-6.24	2.43
					P JEX				
P2	First derivative+MSC	2171.3-2356.9	54	0.7118	75.9	0.7708	64.5	-10	2.11
	(17pts.)	L	4)		1				
P3	MSC	1063.4-1640.9	1	0.7035	75.9	0.8049	58.7	-8.52	2.29
		1833.5-2175.0	\sim		SV.	()			
P4	MSC	1063.4-1640.9	MP	0.6957	76.9	0.7945	59.9	-10.1	2.24
		1833.5-2175.0		デデ	50)		7		
P1P2P3P4	First derivative+SNV	1063.4-1836.1	80	0.7514	69.4	0.7946	61.4	-2.66	2.21
	(17pts.)	2171.3-2262.3							
Average P	SNV	1063.4-1836.1	6	0.7969	62.9	0.8343	53.8	-7.26	2.48
		2171.3-2356.9							

Table 24 Calibration and validation results for sucrose content of flesh by NIRS.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, SNV: standard normal variate, 17 pts: 17 smoothing points, P1: calyx, P2: bottom, P3 and P4: 2 side of cheek, P1P2P3P4: all positions and Average P: average all positions

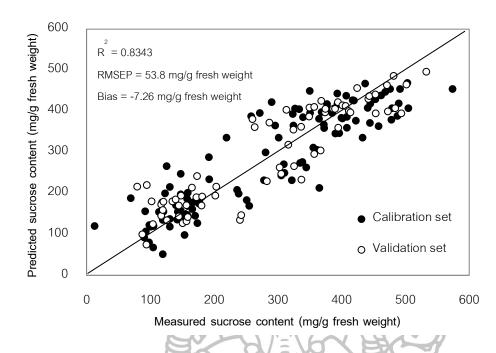


Figure 38 Scatter plot of sucrose of flesh from the model built from averaging spectra.

4.2.4.2. Glucose

The calibration and validation results with the PLS models for glucose content of flesh in longan fruit samples are shown in Table 25. Measurement at average all positions and first derivative+MSC preprocessing technique by using 6 PLS factors in the range of 1332.3-1640.9 and 1833.5-2356.9 nm provided the highest prediction model for glucose content prediction with showed R^2 , RMSEP, RPD and a bias of 0.5146, 17.3 mg/g fresh weight, 1.51 and -5.39 mg/g fresh weight, respectively. As mentioned by Willams (2007), R^2 value of 0.50-0.64 can be used just for rough screening. Nevertheless, RPD from this model was 1.51 which indicates that the model can discriminate low from high values of the response variable but cannot use in research and quality assurance (Nicolaï et al., 2007).

Low performance of prediction may be explained by histogram of glucose content in samples (Figure 37) which showed the narrowest and less uniform sugar distribution obtained in longan samples when compared to other sugars. This result also found in Rady and Guyer (2015) that used NIRS for evaluation of sugar content in potatoes. The prediction result showed low R and RPD (0.73 and 1.44, respectively) because of narrow and less uniform glucose distribution in potato samples.

Figure 39 shows the relationship between measured glucose content value and predicted glucose content value from the best model. This plot clearly showed that the points scattered and almost being under the target line related to negative bias of the model.



		Wavelength	PLS	PLSCalibration set (60%)			Validation set (40%)			
Position	Pre processing	(nm)	factor	R ²	RMSEC	R^2	RMSEP	Bias	RPD	
		(1111)		(A)	(mg/g fresh weight)		(mg/g fresh weight)	(mg/g fresh weight)		
P1	First derivative+MSC	1063.4-2175.0	7	0.4035	24.1	0.4622	19.9	-3.57	1.39	
	(17pts.)	P	The second	AVE)	なく風					
P2	SNV	1063.4-1333.6	7	0.4408	23.3	0.5386	16.6	-1.07	1.48	
		1638.8-2175.0	5	R	40					
P3	First derivative+SNV	1063.4-1640.9	WA E	0.4266	23.2	0.3294	23.2	1.18	1.18	
	(17pts.)	1833.5-2356.9	37		PER	5)				
P4	SNV	1063.4-1640.9	3	0.3255	25.1	0.2917	24.6	-4.51	1.21	
		1833.5-2356.9		》注	Sign					
P1P2P3P4	First derivative+MSC	1063.4-2356.9	9	0.4218	23.1	0.4962	19.7	-1.09	1.41	
	(17pts.)	14								
Average P	First derivative+MSC	1332.3-1640.9	6	0.5282	21.3	0.5146	17.3	-5.39	1.51	
	(17pts.)	1833.5-2356.9	~	Jd	UTIC					

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Table 25 Calibration and validation results for glucose content of flesh by NIRS.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, SNV: standard normal variate, 17 pts: 17 smoothing points, P1: calyx, P2: bottom, P3 and P4: 2 side of cheek, P1P2P3P4: all positions and Average P: average all positions

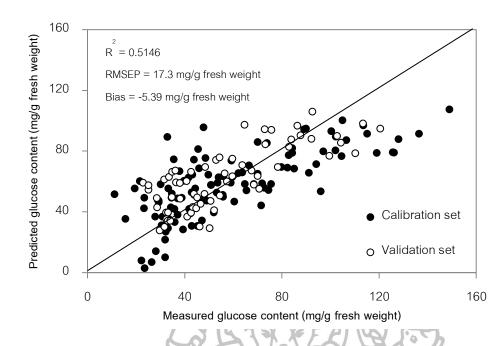


Figure 39 Scatter plot of glucose of flesh from the model built from averaging spectra.

<u>4.2.4.3. Fructose</u>

The calibration and validation results with the PLS models for fructose content of flesh in longan fruit samples are shown in Table 26. The best prediction model for fructose was obtained by averaging spectra at all positions of measurement which was pretreated by combination of first derivative and MSC preprocessing techniques and using 7 PLS factors in the range of 1332.3-1640.9 and 1833.5-2175.0 nm. R², RMSEP, RPD and bias of this model were 0.8158, 13.9 mg/g fresh weight, 2.33 and -0.321 mg/g fresh weight, respectively. The R² value of 0.82-0.90 implies that a model was usable with caution for most applications, including research (Williams, 2007) and the RPD of 2-2.5 demonstrates that coarse quantitative predictions are possible (Nicolaï et al., 2007). The relationship between measured and prediction results of fructose content are illustrated in Figure 40.

In the case of intact apple fruit, NIRS model for predicting fructose content provided R value of 0.968 and RMSEP value of 0.298%. This result showed that NIRS can be used for sugar content analysis (Liu et al., 2006).

		Wovelength		Calibra	tion set (60%)		Validation set (40%)			
Position	Pre processing		PLS factor	R ²	RMSEC (mg/g fresh weight)	R^2	RMSEP (mg/g fresh weight)	Bias (mg/g fresh weight)	RPD	
P1	Second derivative (17pts.)	2171.3-2356.9	2	0.5456	23.9	0.7140	17.5	1.62	1.88	
P2	First derivative+MSC (17pts.)	1063.4-1640.9	8	0.7507	18.2	0.7696	16.3	0.824	2.09	
		1833.5-2356.9	h	LIST.						
P3	Second derivative (17pts.)	1332.3-1836.1	1	0.6007	22.0	0.6944	17.7	-1.12	1.88	
P4	SNV	1063.4-1333.6	8	0.7280	19.0	0.7576	16.4	-3.5	2.08	
		1638.8-1836.1	\sim	ヲ))) E	ays.)j)				
		2171.3-2356.9		20		5				
P1P2P3P4	First derivative+MSC (17pts.)	1063.4-1640.9	8	0.7017	19.3	0.7411	16.6	-0.926	1.97	
		1833.5-2356.9	Q.	灰	97/	5)				
Average P	First derivative+MSC (17pts.)	1332.3-1640.9	7	0.7635	17.7	0.8158	13.9	-0.321	2.33	
		1833.5-2175.0	181	าลีย	ลลบ					

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 Table 26 Calibration and validation results for fructose of flesh by NIRS.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP,

MSC: multiplicative scatter correction, SNV: standard normal variate, 17 pts: 17 smoothing points, P1: calyx, P2: bottom, P3 and P4: 2 side of cheek, P1P2P3P4: all positions and Average P: average all positions

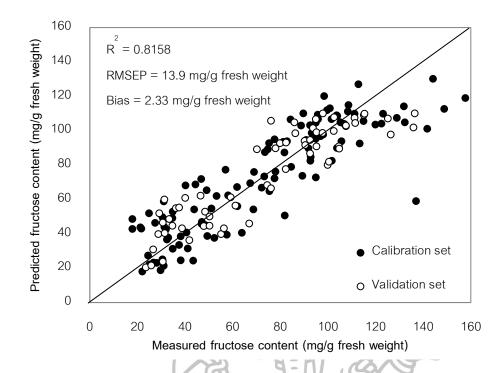


Figure 40 Scatter plot of fructose of flesh from the model built from averaging spectra.

Total sugar 4.2.4.4.

The calibration and validation results with the PLS models for total sugar content of flesh in longan fruit samples are shown in Table 27. The best model was achieved by averaging spectra at all positions of measurement which was pretreated by MSC preprocessing technique and using 7 PLS factors in the range of 1332.3-1471.4 nm and 1833.5-2064.2 nm. R², RMSEP, RPD and bias of this model were 0.9082, 51.9 mg/g fresh weight, 3.30 and -1.05 mg/g fresh weight, respectively. According to Williams (2007), An R² of 0.83-0.90 indicates that a model was usable with caution for most applications, including research. And RPD of 2.5-3 indicated that the model has excellent prediction accuracy (Nicolaï et al., 2007). The scatter plot of measured and prediction result of total sugar is shown in Figure 41.

		Wavelength	PLS	Calibra	ition set (60%)				
Position	Pre processing	(nm)	factor	R ²	RMSEC	R^2	RMSEP	Bias	RPD
		(1111)			(mg/g fresh weight)	IX.	(mg/g fresh weight)	(mg/g fresh weight)	
P1	SNV	1063.4-1836.1	5	0.8180	75.5	0.8400	68.1	6.51	2.51
P2	MSC	1063.4-1333.6	8	0.8046	79.4	0.8099	74.2	-5.12	2.30
		1638.8-2175.0	Th	7-6					
P3	SNV	1638.8-2175.0	2	0.7757	82.7	0.8358	68.4	-2.02	2.47
P4	SNV	1332.3-2356.9	J C	0.8315	73.4	0.8721	60.9	5.85	2.81
P1P2P3P4	First derivative+SNV	1063.4-1836.1	8	0.8202	73.5	0.8503	66.0	2.39	2.59
	(17pts.)	2171.3-2356.9	HD.	2//					
Average P	MSC	1332.3-1471.4		0.8590	67.1	0.9082	51.9	-1.05	3.30
		1833.5-2064.2	36	下采		15	5		

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 Table 27 Calibration and validation results for total sugar of flesh by NIRS.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP,

MSC: multiplicative scatter correction, SNV: standard normal variate, 17 pts: 17 smoothing points, P1: calyx, P2: bottom, P3 and P4: 2 side of cheek, P1P2P3P4: all positions and Average P: average all position

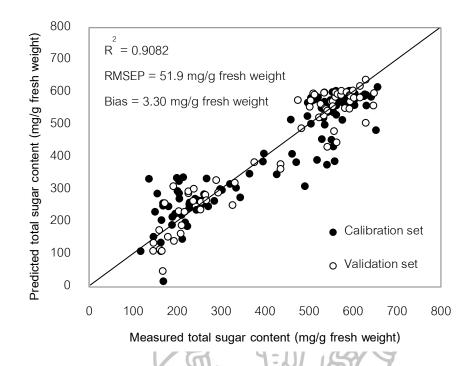


Figure 41 Scatter plot of total sugar of flesh from the model built from averaging spectra.

Figure 42 (a) shows the regression coefficient plot of the best fit of sucrose content model. These wavelengths (1369 nm and 1409 nm) own to -H and -OH of carbohydrates (Rungpichayapichet et al., 2016). Figure 42 (b) shows the regression coefficient plot of the best fit of glucose content model. There were high peaks at 1921 nm, 2178 nm and 2294 nm owning to the second overtone of CONH group in amide, combination of protein and combination of amino acid, respectively. Figure 42 (c) show the regression coefficient plot of the best fit of fluctose content model. The peaks at 1426, 1451, 1893, 2005 and 2074 nm related to -OH functional groups of carbohydrates such as sugar and starch (Osborne et al., 1993; Magwaza et al., 2012; Rungpichayapichet et al., 2016). And Figure 42 (d) shows the regression coefficient plot of total sugar. There were high peak at 1396 nm and 1919 nm which represented first overtone of CH₂ and second overtone of CO₂H, respectively. Moreover, there were high peak at 1903, 1941 and 1992 nm which related to carbohydrates (starches and

sugars), water and organic acids (Osborne et al., 1993; Rungpichayapichet et al., 2016).

The regression coefficient plots of sucrose, fructose and total sugar also found the wavelengths which corresponded to sugar indicated sugar had an important influence on the prediction of sucrose, fructose and total sugar content of longan. However, the wavelengths which related to sugar were not found in the regression plot of glucose affected to poor prediction of glucose model.

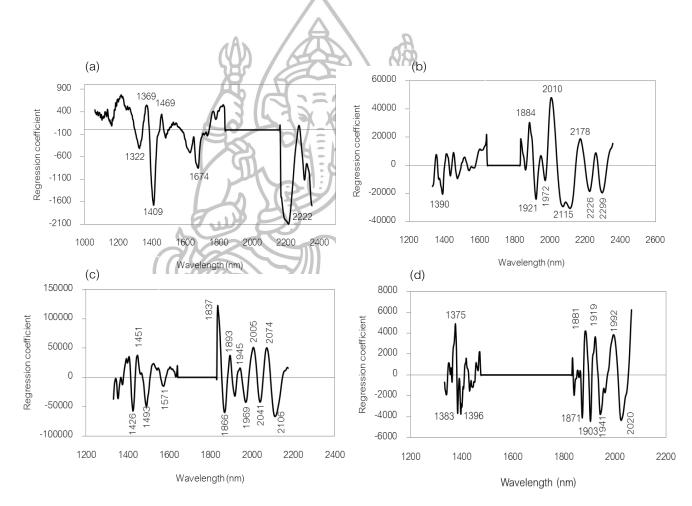
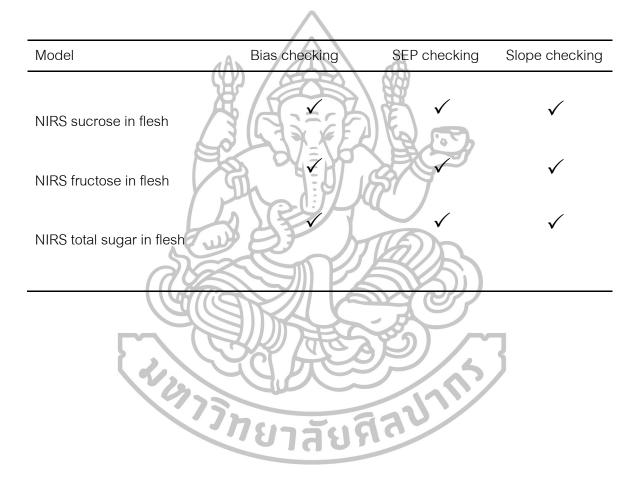


Figure 42 Regression coefficient plot of PLS models validated for sugar content of flesh of the longan fruits: (a) sucrose; (b) glucose; (c) fructose and (d) total sugars.

The models that had RPD value more than 2.0 were tested for verifying the accuracy by bias checking, SEP checking and slope checking method. Table 28 represents that sucrose, fructose and total sugars model passed the verification.

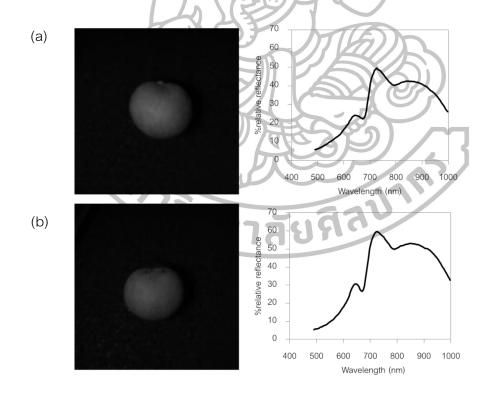
Table 28 Verifying the accuracy by bias checking, SEP checking and slope checkingmethod for sugar content models.



4.3 Hyperspectral imaging for determination of moisture content and sugar content in dried whole longan

4.3.1 HSI images and reflectance spectra of fresh and dried whole longans

HSI images acquisition and extracted spectra were obtained from fresh and dried whole longan at 60°C for 40 hours which were are shown in Figure 43. 10 points selected from R program were averaged and represent the spectrum of the fruit. These spectra of fresh and dried whole longan can observe that the spectrum of dried had higher %relative reflectance that the spectrum of fresh. The original HSI spectra of fresh and dried whole longan at different temperatures for 40 hours are shown in Figure 44. From this Figure, there were 2 absorption peaks around 690 and 780 nm related to the presence of chlorophyll pigment (Rajkumar et al., 2012) and O-H third stretching overtone was assigned to moisture content (Wu et al., 2012).





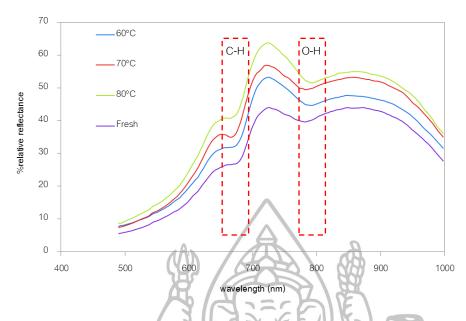


Figure 44 Example of original spectra of fresh and dried whole longan dried at different temperatures for 40 hour.

However, the board band and overlap peak of HSI spectra were observe. And same as the NIR spectra, they need preprocessing techniques to remove these problems such as second derivative which performs resolves peak overlapping and removing baseline shifts (Nicolaï et al., 2007). Second derivative HSI spectrum of dried whole longan obtained from drying at 60°C for 40 hours is shown in Figure 45. There was a strong adsorption peak at around 690 nm which was associated with the presence of chlorophyll (Rajkumar et al., 2012). Moreover, absorption bands at around 780 nm and 980 nm were the results from the absorption behavior of sugar and water corresponding to its stretching third and second overtone, respectively (Osborne et al., 1993).

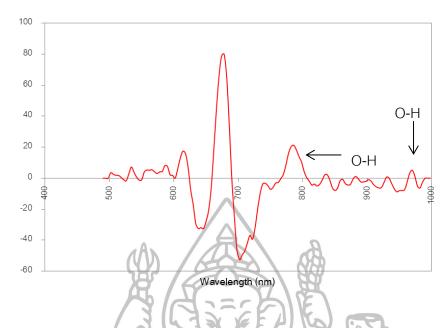


Figure 45 Example of second derivative HSI spectrum of dried whole longan obtained from drying at 60°C for 40 hours.

4.3.2 Calibration models for moisture content by HSI

A total of 496 spectra were divided into calibration set (52.5% of all samples) and validation set (47.5% of all samples) for building the models. Mean, standard deviation (SD), minimum and maximum of moisture content (%) of longan fruit samples from different fruit parts and the whole fruit showed in Table 29. The results were similar as the statistics of NIRS prediction which had the range of peel between 3.35-51.74%, the range of flesh part was between 9.99-88.62%, the range of seed part was between 7.61-49.90%, the range of whole was between 8.15-73.36% and the range of flesh and seed was between 7.27-71.26%. Same as NIRS, the result showed that the flesh part had the widest range of moisture content.

Fruit part	Ν	Calibration set (52.5%)				N	Validation set (47.5%)				
		Min	Max	Mean	SD	IN	Min	Max	Mean	SD	
Peel	261	3.35	51.74	15.75	9.73	235	3.72	45.76	15.45	9.05	
Flesh	261	9.99	88.62	44.65	25.47	235	11.04	83.07	44.44	25.17	
Seed	261	7.61	49.90	26.45	11.66	235	7.98	46.98	26.33	11.41	
Flesh+seed	261	7.27	71.26	34.74	21.23	235	7.92	70.12	34.53	20.98	
Whole fruit	261	8.15	73.36	36.99	21.97	235	8.77	72.93	36.79	21.72	

Table 29 Mean, standard deviation (SD), minimum and maximum of moisture content(%) for longan fruit samples from different fruit parts and the whole fruit (HSI).

N = number of samples

The calibration and validation results associated with the PLS models for moisture content by HSI are shown in Table 39-43 (Appendix F). As a result of one position of imaging, the results were compared the preprocessing method such as smoothing, normalize, MSC, first derivative, second derivative, SNV and combined methods to find the optimum model. Table 30 shows the best models for moisture content from different fruit parts and the whole fruit.

For moisture content of peel, the result found that the optimum model was from raw spectra by using 10 PLS factors. This model showed R², RMSEP, RPD and a bias of 0.6396, 5.43%, 1.69 and 1.01%, respectively. For flesh part, the result showed that the optimum models generated from smoothing (7) method by using 10 PLS factors. This model showed R², RMSEP, RPD and a bias of 0.7071, 13.60%, 1.85 and -0.19%, respectively. For seed part, the result showed that the optimum models generated from MSC+ first derivative method by using 10 PLS factors. This model showed R², RMSEP, RPD and a bias of 0.76%, respectively. For flesh and seed, the result showed that the optimum models generated from second derivative (5) method by using 10 PLS factors. This model showed R², RMSEP, RPD and a bias of 0.7175, 11.1%, 1.88 and 0.61%, respectively. The optimum models for moisture content

of whole fruit generated from smoothing (7) method by using 14 PLS factors. This model showed R^2 , RMSEP, RPD and a bias of 0.7458, 10.93%, 1.98 and 0.31%, respectively.

Obviously, the model of peel and seed showed the lowest performance when compared with other parts. It could be expected that the chemical data of these 2 parts had narrower range than the others. Moreover, low performance of seed model might be due to the presence of seed of longan which is deepest part and the characteristic of seed that has glossy seed coat. As the result of these problems, the light can reflect and make an error in HSI measurement.

Williams (2007) had indicated that R² of 0.50-0.64 implies that a model usable for rough screening and R² of 0.66-0.81 implies that a model usable for screening and some other approximate calibrations. For RPD evaluation of these moisture content models, all models had RPD between 1.5 and 2 indicates that the model can discriminate low from high values of the response variable (Nicolaï et al., 2007). In case of vegetable soybean (Huang et al., 2014), HSI (400-1000 nm) was applied for prediction of moisture content during drying. The moisture content was in the range of 4.9-67.7% that has narrower range than longan in this study. However, the results showed high R and RPD of 0.971 and 4.3, respectively while low RMSEP (4.7%) was found.

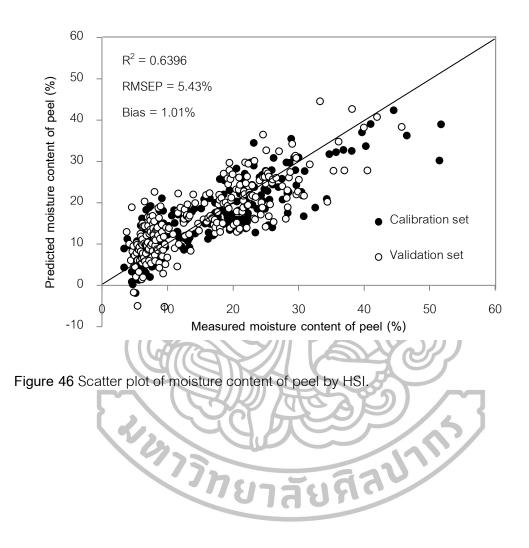
Comparing to NIRS result, the results from HSI were not as good as the result from NIRS technique. The poorer correlation might be due to the difference of the range of wavelength which relate to the difference of the presence of absorption peaks and the conditions of hyperspectral imaging system which might not suitable with small and round shape of sample such as longan. Each point of their surface can reflect the light from light source differently that affect to dark area in the HSI image. Furthermore, the dark image (Figure 41) from HSI technique affect to the uncorrected spectra which relate to poorer correlation when built the models between measured and predicted value (Go'mez-Sanchis et al., 2008).

Fruit part	Pre processing	PLS	Calibratio	n set (52.5%)	Val	Validation set (47.5%)		
		factor	R ²	RMSEC(%)	R^2	RMSEP(%)	Bias(%)	RPD
Peel	Raw	10	0.7390	4.96	0.6396	5.43	1.01	1.69
Flesh	Smoothing (7)	614	0.7637	12.35	0.7071	13.60	-0.19	1.85
Seed	MSC+ first derivative	7	0.6479	6.90	0.6663	6.58	-0.76	1.74
Flesh and seed	Second derivative (5)	10	0.7860	9.80	0.7175	11.13	0.61	1.88
Whole fruit	Smoothing (7)	14	0.7455	11.06	0.7458	10.93	0.31	1.98
	G			$\gamma = 0$	511			

Table 30 Calibration and validation results for moisture content from different fruit parts and the whole longan fruit samples by HSI.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, (5,7): number of left and right average point.

The scatter plots demonstrating the measured value of moisture content (X) and the prediction value (Y) for the peel model, flesh model, seed model, flesh and seed model and the whole fruit model are shown in Figure 46-50. However, the points rather dispersed from the target line that showed low performance of prediction.



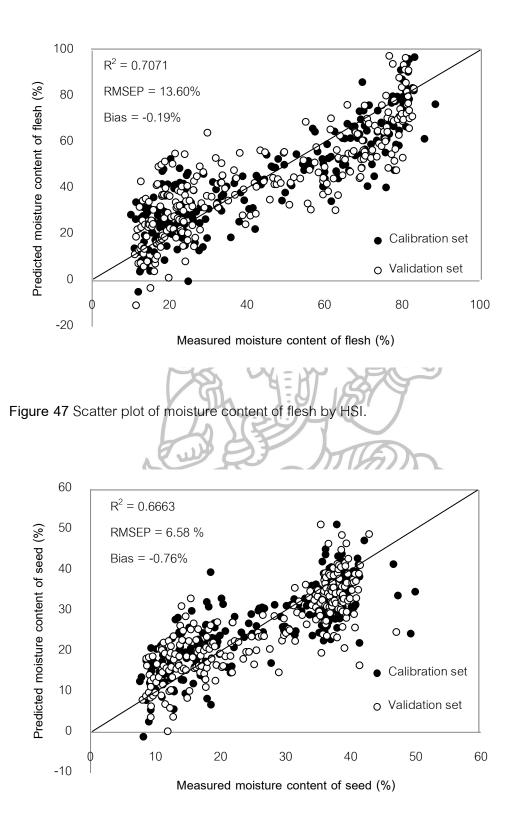


Figure 48 Scatter plot of moisture content of seed by HSI.

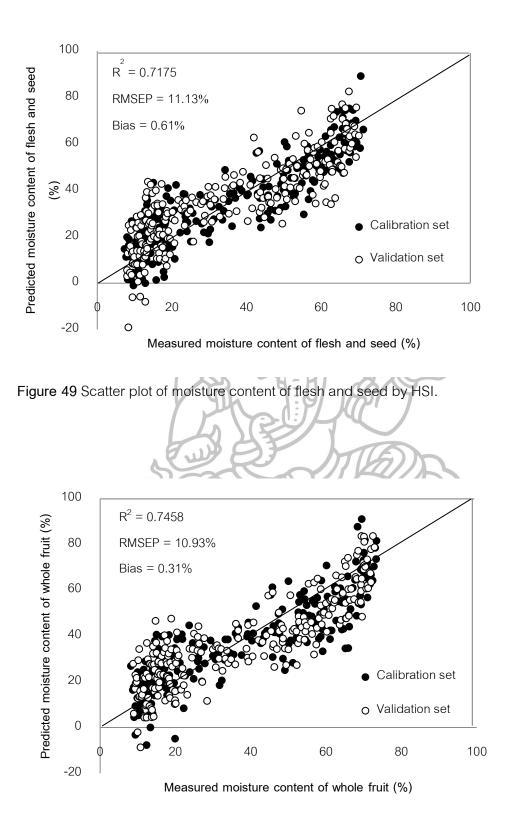


Figure 50 Scatter plot of moisture content of whole fruit by HSI.

Figure 51 (a) shows the regression coefficient plot of moisture content of flesh. There were high peaks at 658, 678, 706, 734, 750, 798, 950 and 994 nm. Wavelengths at 678 nm could be associated with chlorophyll pigment (Rajkumar et al., 2012) and 734 and 750 nm could be associated with the O-H stretching third overtone vibration in ROH and ArOH and the wavelength at 994 nm could be associated with O-H stretching second overtone vibration of starch (Osborne, 1993). Figure 51 (b) shows the regression coefficient plot of moisture content of the whole fruit. The wavelengths at 682, 802 and 994 nm could be associated with the vibration of chlorophyll pigment, amino group and starch, respectively. However, the wavelengths which associated with water (760 and 970 nm) were not found in both regression coefficient plots.

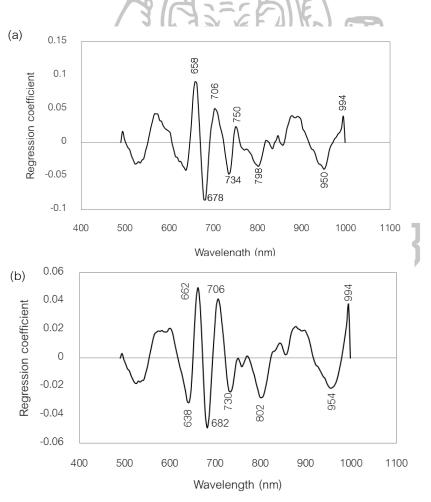


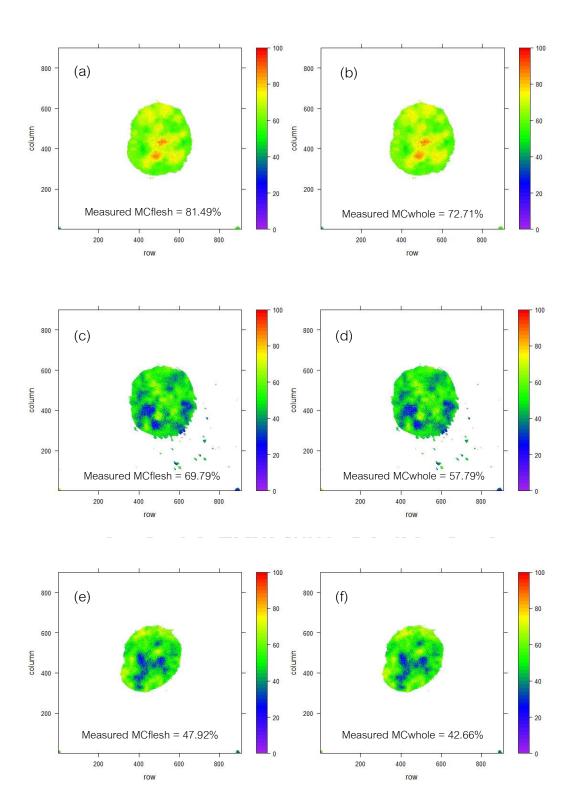
Figure 51 Regression coefficient plots of (a) moisture content of flesh and (b) moisture content of the whole fruit by HSI.

Due to HSI technique can collect the spectra in term of areas. Thus, this technique can provide the chemical properties distribution in fruit. With regard to the distribution of water in dried whole longan is absolutely important determining the quality of dried whole longan products. Beuchat (1981) said that water activity is an important factor for inducing the growth of microorganism especially, pathogenic bacteria in food. Furthermore, high moisture content would influence the high water activity that affected the microorganism growth. Thus, water content affects the quality and shelf life of dried whole longan and the determination of moisture content distribution in dried whole longan by HSI technique is very useful.

In this research, the observed maps was generated based on the PLS models of moisture content of flesh and the whole fruits which build from R program to exhibit the distribution of moisture content within flesh and the whole fruits. The pixels that had homologous spectral characteristics in the distribution map produced the same predicted values of moisture, which were observed in a same color in the acquired image. Figure 52 shows the example of distribution maps of the whole longan fruits with different moisture content of flesh and the whole fruit levels. The changes in moisture content were defined with a linear colour scale which was used to present different moisture concentrations in different area of the whole fruit with different colors (purple, blue, green, yellow and red). Figure 52 (a) and (b) show the fresh whole longan which was opened from the PLS models of moisture content of flesh and the whole full and the whole fruit with different colors (purple, blue, green, yellow and red). Figure 52 (a) and (b) show the fresh whole longan which was opened from the PLS models of moisture content of flesh and the whole full are dried whole longan at 60°C for 12 hr, (e) and (f) are dried whole longan at 60°C for 30 hr; (g) and (h) are dried whole longan at 60°C for 36 hr. and (i) and (j) are dried whole longan at 60°C for 60 hr.

The color in maps explained the distribution of moisture in the whole longan fruit which could not be distinguished by normal color image. The variation of moisture content in fresh longan is subject to longan species, farming condition, natural variations due to climatic and seasonal factors, harvesting time and storage condition. Whereas, the variation of moisture content in dried whole longan might be depend on the drying condition. The fresh fruit showed a more uniform yellow and red, indicating that their moisture content of flesh and whole fruit were relatively high (Figure 52 (a) and (b)). While, the dried fruits which contained lower moisture content had the color tended to blue (Figure 52 (c) - (j)). However, the poor correlation from the models gave some errors of prediction in the distribution maps. As can be seen in the figures, the image from PLS models of moisture content of flesh and the whole fruit of each fruit had similar the variation of color while the sample had different moisture content between flesh and the whole fruit. Moreover, the uncorrected colors on distribution map are observed at the low moisture content (Figure 52 (i) and (j))





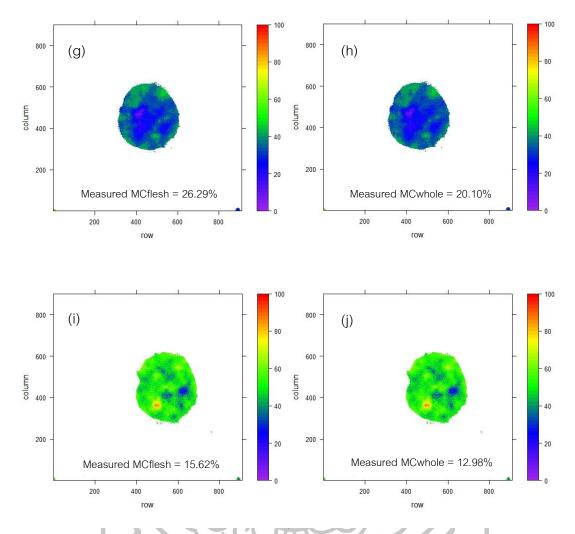


Figure 52 The example of moisture content distribution maps of whole longan generated by PLS model of moisture content for flesh and the whole fruit (a) and (b) fresh whole longan; (c) and (d) dried whole longan at 60°C for 12 hr; (e) and (f) dried whole longan at 60°C for 30 hr; (g) and (h) dried whole longan at 60°C for 36 hr and (i) and (j) dried whole longan at 60°C for 60 hr.

4.3.3 Calibration models for TSS of flesh by HSI

A total of 186 spectra were divided into calibration set (52.5% of all samples) and validation set (47.5% of all samples) for building the models. Mean, standard deviation (SD), minimum and maximum of TSS of flesh are showed in Table 31. The range of TSS between 13-98 °Brix was found same as the statistic of NIRS.

Table 31 Mean, standard deviation (SD), minimum and maximum of TSS of flesh inlongan fruit samples for predicting TSS (HSI).

				}		
Parameter	Data set	Ν	Min	Max	Mean	SD
TSS (°Brix)	Calibration (52.7%)	98	13	98	56.72	24.15
	Validation (47.5%)	88	18	90	56.75	23.30
N = number of samples			THE			

The calibration and validation results associated with the PLS models for TSS by HSI are shown in Table 44 (Appendix F). Table 32 shows the best models for moisture content from different fruit parts and the whole fruit. The best model was obtained from MSC+ smoothing preprocessing techniques by using 11 PLS factors. The results showed R^2 , RMSEP, RPD and a bias of 0.6676, 13.36 °Brix, 1.78 and 3.05 °Brix. According to Williams (2007), R^2 of 0.66-0.81 indicates that a model usable for screening and some other approximate calibrations. About RPD of this model, the value was found between 1.5 and 2 indicates that the model can discriminate low from high values of the response variable (Nicolaï et al., 2007).

Moreover, there was using HSI (500-1000 nm) for predicting TSS in blueberries (Leiva-Valenzuela et al., 2013) which had fairly a similar shape to longan. Lower correlations ($R_p = 0.69-0.79$) and RPD (1.3-1.6) were found, compared with that reported for large fruits such as apple (Lu, 2004).

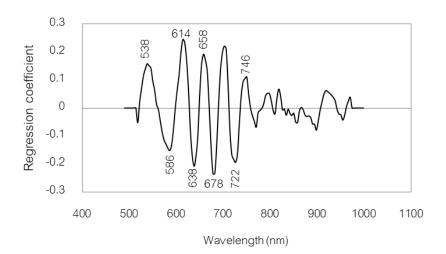
	PLS	Calibrati	on set (52.5%)	Valida					
Pre processing	factor	R^2	RMSEC	R^2	RMSEP	Bias	RPD		
	lactor	Γ	(°Brix)	Γ	(°Brix)	(°Brix)			
MSC+	11	0.7729	11.45	0.6676	13.36	3.05	1.78		
smoothing									
Abbreviations: R ² : coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of									
prediction, RPD: ratio of s	tandard dev	iation of reference	e data in validation set to	SEP MSC: mult	inlicative scatter	correction			
prodiction, ra privato ere					phoduro ooddor	00110001011			
		XX /	$\langle \cdot \cdot \cdot \rangle$	/ 斑					
Figure 53	3 showe	d the relation	onship between	measured	and predi	cted TSS t	from		
		131	JJ GE	415					
the best calibrati	ion mode	el. It can b	e seen that the	points disp	ersed from	the target	line		
that showed low	perform	ance of pre	diction.	Ð					

Table 32 Calibration and validation results for TSS in flesh of longan fruit samples by HSI

 $R^2 = 0.6676$ RMSEP = 13.36 °Brix Bias = 3.05 °Brix Predicted TSS (°Brix) Calibration Set o Validation set Measured TSS (°Brix)

Figure 53 Scatter plot of TSS of flesh by HSI

Figure 54 shows the regression coefficient plot of the best TSS model. There were high peaks at 538, 586, 614, 638, 658, 678, 708, 722, 746 and 770 nm. Wavelengths at 678 nm could be associated with chlorophyll pigment (Rajkumar et al., 2012) and Wavelengths at 746 nm could be associated with the C-H stretching fourth overtone vibration in CH_2 (Osborne, 1993).





4.3.4 Calibration models for sugar content of flesh by HSI

The statistics of HSI prediction for sugar content are shown in Table 33. The results were similar as the statistics of NIRS prediction which had the range of sucrose content between 12.47-593.92 mg/g fresh weight, the range of glucose content between 11.05-148.96 mg/g fresh weight, the range of fructose content between 17.84-157.70 mg/g fresh weight and the range of total sugar content between 116.94-655.84 mg/g fresh weight. Same as NIRS, the result showed that glucose had the lowest value and narrowest range.

Sugar (mg/g fresh weight)	Ν	Calibration set (52.5%)				B	Validation set (47.5%)			
		Min	Max	Mean	SD		Min	Max	Mean	SD
Sucrose	95	12.47	593.92	292.73	142.08	86	79.28	532.84	289.00	132.60
Glucose	95	11.05	148.96	59.67	31.35	86	15.65	130.06	57.43	28.23
Fructose	95	17.84	157.70	74.04	36.23	86	19.25	144.46	72.67	33.72
Total sugars	95	116.94	655.84	423.23	174.76	86	145.20	646.68	421.83	169.93
N = number of samples					าลีย	33		3		

 Table 33 Mean, standard deviation (SD), minimum and maximum of sugar content for longan fruit samples (HSI).

The calibration and validation results associated with the PLS models for sugar content by HSI are shown in Table 45-48 (Appendix F). Table 34 shows the best models for sugar content of flesh. For sucrose, the result found that the optimum model was organized from MSC+ smoothing preprocessing techniques by using 10 PLS factors. This model showed R², RMSEP, RPD and a bias of 0.5247, 5.43 mg/g fresh weight, 1.69 and 1.01 mg/g fresh weight, respectively. For glucose, the optimum model was conducted from SNV+ second derivative preprocessing techniques by using 3 PLS factors. R², RMSEP, RPD and bias of this model were 0.2699, 24.03 mg/g fresh weight, 1.17 and -2.25 mg/g fresh weight, respectively. For fructose, first derivative (4) was given the optimum model by using 6 PLS factors. This model shows R², RMSEP, RPD and a bias of 0.4944, 23.86 mg/g fresh weight, 1.41 and 2.54 mg/g fresh weight. And for total sugar, the optimum model was organized from MSC+smoothing preprocessing techniques by using 7 PLS factors. This model shows R², RMSEP, RPD and a bias of 0.5333, 115.42 mg/g fresh weight, 1.47 and 12.98 mg/g fresh weight.

It is worth noting that prediction model of sucrose and total sugar had R^2 of 0.26-0.49 implies that the models have poor correlation. While prediction model of glucose and fructose had R^2 of 0.50-0.64 implies that the models are OK for rough screening (Williams, 2007). However, the RPD values of all models were found less than 1.5 that means the models are poor and not recommended for research and most applications (Nicolaï et al., 2007).

In the case of potatoes (Rady et al., 2014), HSI (400-1000 nm) was used for predicting glucose and sucrose content. Poor correlation models were found with low R of 0.38 and 0.14, and low RPD of 0.93 and 1.02 for glucose and sucrose, respectively.

Same as sugar content results by NIRS, the glucose content model gave the lowest accuracy predicted. One reason might be due to low concentration of glucose in longan.

			Calibration	set (52.5%)		Validation set (47.5%)			
Sugar	Pre processing	PLS factor	R2	RMSEC mg/g fresh weight)	R ²	RMSEP (mg/g fresh weight)	Bias (mg/g fresh weight)	RPD	
Sucrose	MSC+ smoothing	10	0.6704	81.12	0.5247	90.93	19.32	1.48	
Glucose	SNV+ second derivative	3	0.2221	27.50	0.2699	24.03	-2.25	1.17	
Fructose	First derivative (4)	6	0.4796	25.99	0.4944	23.86	2.54	1.41	
Total sugars	MSC+ smoothing	AVE	0.6883	97.03	0.5333	115.42	12.98	1.47	

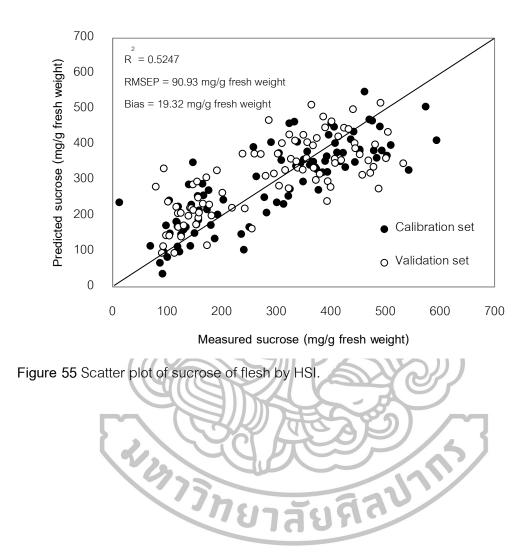
 Table 34 Calibration and validation results for sugar content in flesh of longan fruit samples by HSI

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP,

MSC: multiplicative scatter correction, SNV: standard normal variate, (4): number of left and right average point



Figure 55-58 show the scatter plots of sucrose, glucose, fructose and total sugar model. According to poor performance of these models, the points scattered out from the target line.



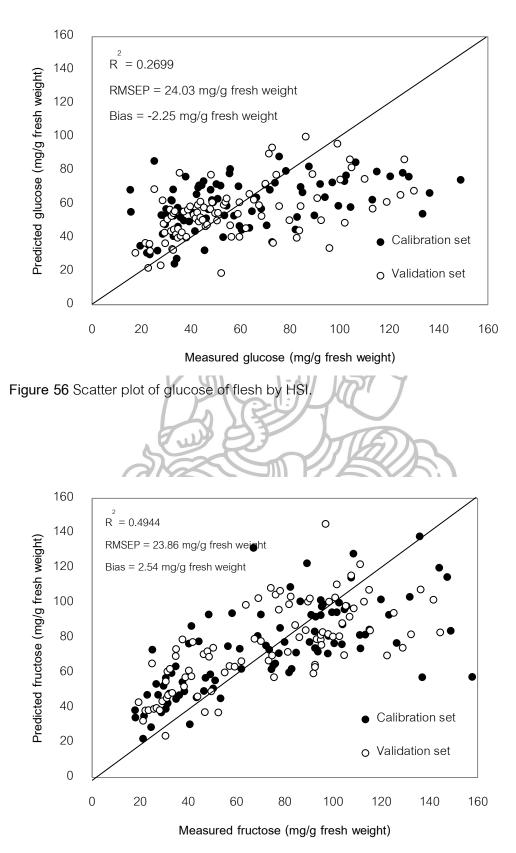


Figure 57 Scatter plot of fructose of flesh by HSI.

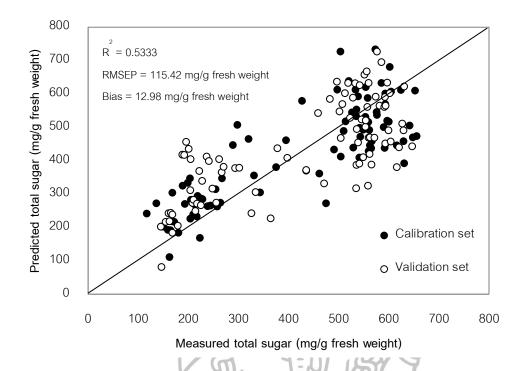


Figure 58 Scatter plot of total sugar of flesh by HSI.

Figure 59 (a) shows the regression coefficient plot of sucrose. The wavelengths at 678, 882, and 938 nm could be associated with chlorophyll pigment, chloroform and methylene compound, respectively (Osborne, 1993; Rajkumar et al., 2012). Figure 57 (b) shows the regression coefficient plot of sucrose. The wavelengths at 690 and 714 nm could be associated with chlorophyll pigment and benzene (Osborne, 1993; Rajkumar et al., 2012). Figure 59 (c) and (b) also show the peaks of chlorophyll in the plots (Rajkumar et al., 2012).

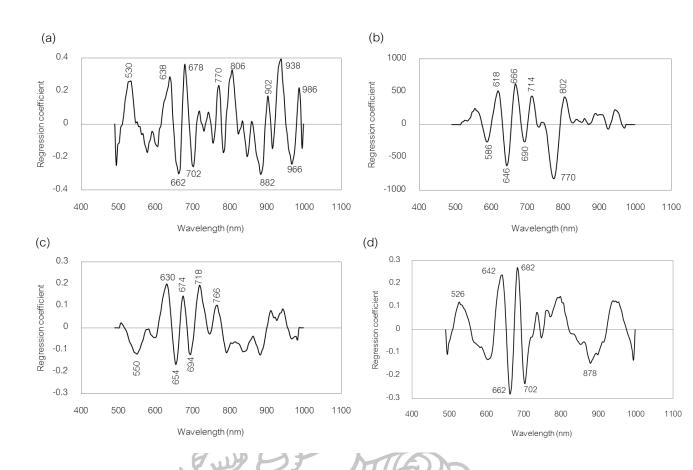


Figure 59 Regression coefficient plot of PLS models validated for sugar content of flesh of the longan fruits by HSI: (a) sucrose; (b) glucose; (c) fructose and (d) total sugars



CHAPTER 5 CONCLUSION

The results from this study have been shown that the NIRS technique had better prediction performance than the HSI technique in all parameters (moisture content, TSS, sucrose, glucose, fructose and total sugar). HSI technique showed poor accuracy model for prediction because the small and round shape of longan provided the dark acquired image that affected to the uncorrected spectral acquirement and related to poor correlation when built the prediction models.

High effective of all prediction models were obtained using average spectra of all position from NIRS technique. For moisture content, the best fit model of peel, flesh and the whole fruit showed acceptable prediction accuracy and precision. The prediction statistics recommend that these models are usable in most applications, including quality assurance. In addition, the predictive values for new dried longan samples obtained with developed NIRS model for flesh part did not show significant difference with the reference values.

For TSS and sucrose, fructose and total sugar, the results of best fit model showed high accuracy of prediction while, NIRS could not be predicted glucose in any positions. The reason might be due to low concentration and low distribution of glucose content in flesh of dried whole longan.

Thus, NIRS which is a rapid non-destructive method could be used as an alternative technique to determine quality of whole dried longan especially, moisture content of peel, flesh, whole fruit, TSS, sucrose, fructose and total sugar parameters.

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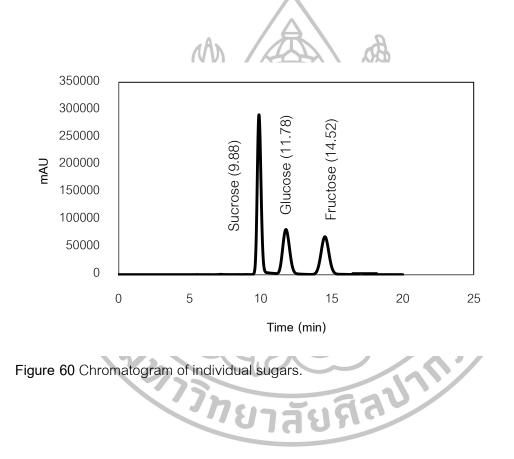
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Standard preparation

Standard solutions of sugars (sucrose, glucose and fructose) were prepared 7 levels by dilution with distilled water. The concentrations of sucrose were 500, 2500, 5000, 10000, 15000, 20000 and 30000 ppm and the concentrations of glucose and fructose were 250, 1250, 2500, 5000, 7500, 10000 and 15000 ppm. The standard solution was filtered with a 0.45 μ m membrane filter and keep in light brown bottle.



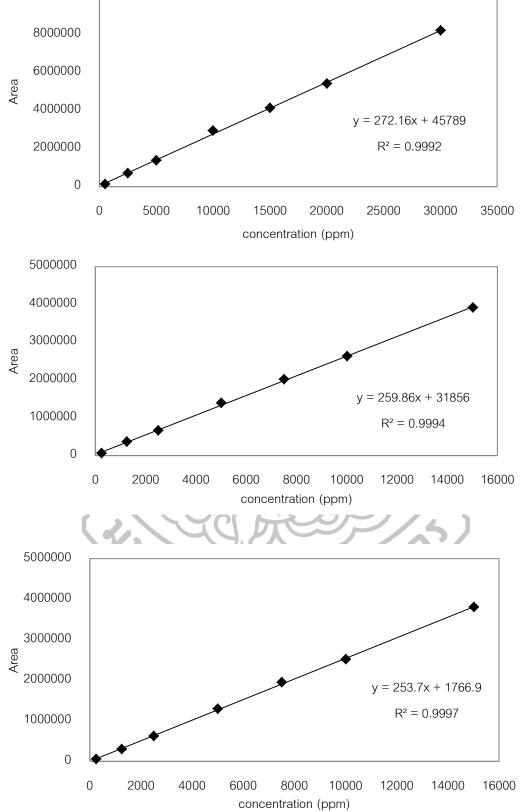
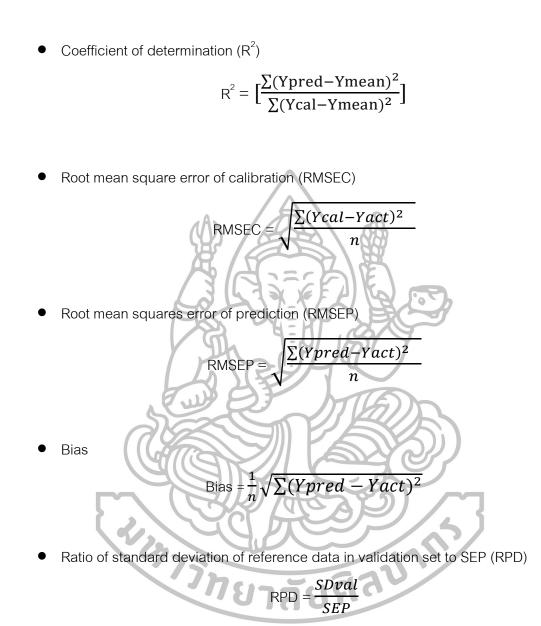


Figure 61 Standard curve for sucrose (a) glucose (b) and fructose (c).





NIRS

Where:

n	The number of spectra
Yact	The actual value
Ymean	The mean value
Ycal	The calculated value
Ypred	The predicted value of the fruit attributes.



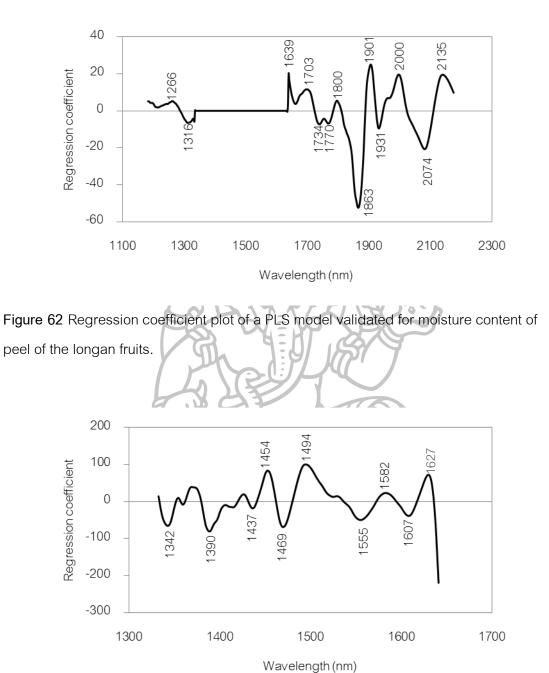
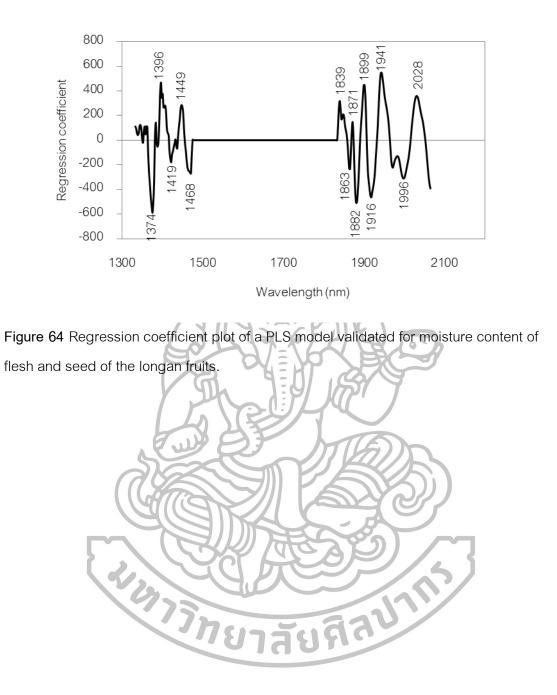


Figure 63 Regression coefficient plot of a PLS model validated for moisture content of seed of the longan fruits.





Verify accuracy of calibration models

The accuracy of prediction models were tested by using bias checking (paired *t*-test) method, SEP checking (F-test, ratio of 2 variances) method and slope checking method from International standard ISO 12099. These methods can demonstrate that predicted value obtained from the instrument were not different from actual values significantly at 95% confidence interval. Moreover, the models that pass all tests are usable in any application.

First bias checking, this method is conducted by paired t-test for calculating the bias confidence limits (BCLs, T_p) which is the acceptance or denial limit of efficiency of a predictive model. The bias value form the model must less than T_b value. It means the bias is accepted (at 95% confidence) and it can conclude that no significant mean difference between NIRS and reference methods. Second SEP checking, this method use F-test for estimating the unexplained error confidence limit (UECLs, T_{UE}). The SEP value from the model must less than T_{UE} value. It means SEP is accepted (at 95% confidence). And slope checking, *t*-test can be calculated to check the hypothesis that slope of scatter plot between actual and NIRS predicted value = 1. This method has to calculate observed *t*-value (t_{obs}) and t_{obs} value must less than *t*-value for a probability of $\alpha = 0.05$ (t(1- $\alpha/2$)). It means the slope is not significantly difference from 1.

Bias checking (paired t-test)

$$T_{\rm b} = \pm \frac{t_{(1-\alpha/2)} \times SEF}{\sqrt{n}}$$

$$\mathsf{SEP} = \sqrt{\frac{\sum (e_i - \bar{e})^2}{n - 1}}$$

 $T_{\rm b}$ is bias confidence limits

n = no. of samples in validation set

Bias< $T_{\rm b}$; Bias is accepted (at 95% confidence)

• SEP checking (F-test, ratio of 2 variances)

$$T_{\text{UE}} = \text{SEC}\sqrt{F_{(\alpha:\nu,M)}}$$
$$\text{SEC} = \sqrt{\frac{\sum e_i^2}{n_c - p - 1}}$$

 $T_{\rm UE}$ = The unexplained error confidence limit (UECLs)

lpha = probability of making a type I error, 5%

V = n-1 (degree of freedom associated with SEP)

 $M = n_c$ -p-1 (degree of freedom associated with SEC)

 $n_c = no.$ of samples in calibration set

p = no. of terms (wavelengths or factors)

SEP< $T_{\rm UE}$; SEP is accepted (at 95% confidence)

b

 $\frac{(y_i - a + b\hat{y}_i)}{n-2}$

Slope checking

n = number of independent samples

 ${S_{\gamma}}^2$ = variance of the n predicted values

 S_{res} = residual standard deviation

a = intercept

b = slope

 y_i = the reference value

 \hat{y}_i = the predicted value

 $t_{\scriptscriptstyle obs} \geq t_{(1-lpha/2)}$, slope b is different from 1

Model	Bias checking	SEP checking	Slope checking	Usable in any
				application
NIRS moisture	\checkmark	\checkmark	\checkmark	\checkmark
content of peel				
NIRS moisture	\checkmark	\bigwedge	\checkmark	\checkmark
content of flesh	AN /		B	
NIRS moisture			\checkmark	×
content of seed	王	SEZENE		
NIRS moisture				\checkmark
content of whole fruit		4.11.18		
NIRS moisture	L'		\checkmark	×
content of flesh and		FM	3Dr	
seed	NACC			,
NIRS TSS in flesh	YIG X			\checkmark
NIRS sucrose in flesh		ДЭ),	253	\checkmark
NIRS fructose in flesh	177718	าลัยสิจ	J	\checkmark
NIRS total sugar in	\checkmark	\checkmark	\checkmark	\checkmark
flesh				

Table 35 Verifying the accuracy by bias checking, SEP checking and slope checkingmethod.

Table 36 Bias checking

Model	t-value	T _b	Bias	Result	Bias
MOGEI	t-value	/ b	Dido	Result	checking
NIRS moisture content	1.97	<u>+</u> 0.16	-0.128	bias< $T_{\rm b}$	\checkmark
of peel					
NIRS moisture content	1.97	<u>+</u> 0.78	0.085	bias< $T_{\rm b}$	\checkmark
of flesh		\wedge			
NIRS moisture content	1.97	±0.31	-0.37	bias> $T_{\rm b}$	x
of seed		2031			
NIRS moisture content	1.97	±0.54	-0.138	bias< $T_{\rm b}$	\checkmark
of whole fruit	Lo L	VELER			
NIRS moisture content	1.97	±0.60	-0.731	bias> $T_{\rm b}$	x
of flesh and seed					,
NIRS TSS in flesh	1.99	±1.54	-0.454	bias< $T_{\rm b}$	\checkmark
a	AC		500		,
NIRS sucrose in flesh	1.99	±13.17	-7.26	bias< $T_{\rm b}$	\checkmark
G		ALL A	5	-	/
NIRS fructose in flesh	2.00	<u>+</u> 3.53	2.33	$<$ bias< $T_{\rm b}$	\checkmark
20	AA				/
NIRS total sugar in	2.00	±12.47	-1.05	bias< $T_{\rm b}$	\checkmark
flesh	10	1284			

Model	F value	Т	SEP	Result	SEP
Model	F value	T _{UE}	3EP	Result	checking
NIRS moisture content	1.24	2.04	1.09	SEP< $T_{\rm UE}$	\checkmark
of peel					
NIRS moisture content	1.24	5.77	5.54	SEP< $T_{\rm UE}$	\checkmark
of flesh		\wedge			
NIRS moisture content	1.24	3.35	2.19	$SEP < T_{UE}$	\checkmark
of seed			88		
NIRS moisture content	1.24	5.35	3.77	SEP< $T_{\rm UE}$	\checkmark
of whole fruit	60143	V=KEK			
NIRS moisture content	1.24	4.23	4.20	SEP< $T_{\rm UE}$	\checkmark
of flesh and seed					
NIRS TSS in flesh	1.42	8.85	6.44	SEP< $T_{\rm UE}$	\checkmark
a	17 A				
NIRS sucrose in flesh	1.43	72.67	55.22	SEP< $T_{\rm UE}$	\checkmark
6		ALL .		_	,
NIRS fructose in flesh	1.44	20.14	14.01	$<$ SEP $< T_{UE}$	\checkmark
2	LA A				,
NIRS total sugar in	1.43	77.26	51.93	SEP< $T_{\rm UE}$	\checkmark
flesh	19.	าลยห			

Table 38 Slope checking

Madal	4	tuoluo	Result	Slope
Model	$t_{\rm obs}$	<i>t</i> -value	Result	checking
NIRS moisture content of	0.0137	1.97	$t_{obs} < t$ -value	\checkmark
peel				
NIRS moisture content of	0.1248	1.97	$t_{\rm obs} < t$ -value	\checkmark
flesh				
NIRS moisture content of	0.1073	1.97	t _{obs} < t-value	\checkmark
seed		3318		
NIRS moisture content of	0.1960	= 1.97	t _{obs} < <i>t</i> -value	\checkmark
whole fruit	0			
NIRS moisture content of	0.0840	1.97	$t_{\rm obs}$ < t-value	\checkmark
flesh and seed	1 P	UJ-Y-C	<u> </u>	
NIRS TSS in flesh	0.0626	1.99	t _{obs} < t-value	\checkmark
a	XC	NE		,
NIRS sucrose in flesh	0.3127	1.99	t _{obs} < <i>t</i> -value	\checkmark
G		HEX-		
NIRS fructose in flesh	0.2754	2.00	t _{obs} < <i>t</i> -value	↓
15	AL			
NIRS total sugar in flesh	0.0441	2.00	t _{obs} < <i>t</i> -value	V
	רטיי	יזטה		



ROI identification

Warning messages:

- 1: package 'hyperSpec' was built under R version 3.1.2
- 2: package 'mvtnorm' was built under R version 3.1.2
- > source("G:\\Yean master degree\\longanselect.r")
- > fresh<-select("LFB160W001000.cue")</pre>
- .read.ENVI.header: Guessing header file name (./LFB160W001000.hdr)
- num [1:10, 1:128] 457 585 478 452 844 ...
- attr(*, "dimnames")=List of 2
- ..\$: chr [1:10] "spec1" "spec2" "spec3" "spec4"
- ..\$: NULL
- > save(fresh,file="fresh.Rdata")
- > fresh.dataframe<-data.frame(fresh)
- > str(fresh)
- num [1:10, 1:128] 457 585 478 452 844 ...
- attr(*, "dimnames")=List of 2
- ..\$: chr [1:10] "spec1" "spec2" "spec3" "spec4" .
- ..\$: NULL
- > write.csv(fresh.dataframe,file="fresh.csv")
- > save.image("D:\\projectปอโท\\โปรเจคลำไย\\เล่ม thesis\\freshhhh")

PLS distribution map

load("D:\\projectปอโท\\โปรเจคลำไย\\data MC\\HSI result\\distibution map\\mixR.Rdata") load("D:\\projectปอโท\\โปรเจคลำใย\\data MC\\HSI result\\distibution map\\mixS.Rdata") local({pkg <- select.list(sort(.packages(all.available = TRUE)),graphics=TRUE) if(nchar(pkg)) library(pkg, character.only=TRUE)}) mixS.SG<-savitzkyGolay(mixS,p=2,w=15,m=0) str(mixS.SG) MCflesh<-data.frame(mixR,mixS.SG) str(MCflesh) local({pkg <- select.list(sort(.packages(all.available = TRUE)),graphics=TRUE) if(nchar(pkg)) library(pkg, character.only=TRUE)}) MCflesh.pls<-plsr(V1~.,data=MCflesh,validation="LOO",ncomp=30) summary(MCflesh.pls) R2(MCflesh.pls) plot(MCflesh.pls,"prediction",ncomp=29,asp=1,line=TRUE) source("G:\\Yean master degree\\longanselect.r") dried6060<-bgremove("LDB160W170000.cue") NETAURAUMANA predict<-predict(MCflesh.pls,ncomp=29,newdata=dried6060) nr<-910 nc<-900 nw<-128 wavelenght<-seq(490,998,4) predict.mean<-matrix(rowMeans(predict),nr,nc) colpal<-colorRampPalette(c("blue","green","yellow","red"))

levelplot(predict.mean,colorkey=list(at=seq(0,100,by=5)),col.regions=colpal(20))

dried6060<-bgremove("LDB160W168000.cue")

predict<-predict(MCflesh.pls,ncomp=29,newdata=dried6060)

levelplot(predict.mean,colorkey=list(at=seq(0,100,by=5)),col.regions=colpal(20))

dried<-bgremove("LDB160W165000.cue")

predict<-predict(MCflesh.pls,ncomp=29,newdata=dried)

nr<-910

nc<-900

nw<-128

wavelenght <- seq(490,998,4)

predict.mean<-matrix(rowMeans(predict),nr,nc)

colpal<-colorRampPalette(c("blue","green","yellow","red"))

levelplot(predict.mean,colorkey=list(at=seq(0,100,by=5)),col.regions=colpal(20))

q()





Pre processing	PLS	Са	Calibration set Validation set			RPD	
	factor	R^2	RMSEC(%)	R^2	RMSEP(%)	bias	RFD
Raw	10	0.7390	4.96	0.6396	5.43	1.01	1.69
Smoothing	10	0.7383	4.97	0.6360	5.45	0.96	1.68
Normalize	9	0.7129	5.20	0.6205	5.57	1.12	1.66
MSC	7	0.6920	5.39	0.6110	5.64	1.09	1.63
First derivative (2)		0.7237	5.10	0.6203	5.57	1.23	1.66
Second derivative (9)	10	0.7519	4.84	0.6068	5.67	1.04	1.62
SNV	30	0.7013	5.31	0.6175	5.59	0.96	1.64
SNV+ smoothing	507	0.6974	5.34	0.6159	5.34	0.97	1.63
SNV+ first derivative	5	0.6722	5.56	0.6100	5.64	1.14	1.63
SNV+ second derivative	7	0.7066	5.26	0.5811	5.85	1.26	1.58
MSC+ smoothing	9	0.7281	5.06	0.6236	5.55	1.07	1.66
MSC+ first derivative	8	0.7695	4.66	0.6388	5.43	1.02	1.69
MSC+ second derivative	7	0.7101	5.23	0.5877	5.80	1.19	1.59

Table 39 Calibration and validation results for moisture content of peel of whole longanfruit samples by HSI.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC; multiplicative scatter correction, SNV:

standard normal variate, (2 and 9); number of left and right average point

Pre processing	PLS	Calibration set		Validation set			RPD
	factor	R^2	RMSEC(%)	R^2	RMSEP(%)	bias	RPD
Raw	9	0.7400	13.00	0.7026	13.70	-0.30	1.83
Smoothing (7)	14	0.7637	12.35	0.7071	13.60	-0.19	1.85
Normalize	9	0.7314	13.17	0.6756	14.31	-1.07	1.76
MSC	7	0.6856	14.25	0.6655	14.53	-1.33	1.74
First derivative (4)	8()	0.7311	13.18	0.6951	13.87	0.22	1.81
Second derivative (7)	8	0.7752	12.05	0.7004	13.75	0.01	1.83
SNV	8	0.7024	13.86	0.6595	14.66	-1.31	1.72
SNV+ smoothing	12	0.7428	12.90	0.6849	14.10	-0.75	1.78
SNV+ first derivative	72	0.7167	13.53	0.6687	14.46	-0.68	1.74
SNV+ second derivative	9	0.7780	11.97	0.6845	14.11	-0.75	1.78
MSC+ smoothing	11	0.7360	13.06	0.6794	14.22	-0.80	1.77
MSC+ first derivative	T	0.7153	13.56	0.6668	14.50	0.70	1.73
MSC+ second derivative	9	0.7771	12.00	0.6835	14.13	-0.81	1.78

Table 40 Calibration and validation results for moisture content of flesh of whole longan fruit samples by HSI.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, SNV:

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standard normal variate, (4 and 7): number of left and right average point ั้งกับกลัยศิลปาก

Pre processing	PLS	Calibration set		Validation set			RPD
	factor	R^2	RMSEC(%)	R^2	RMSEP(%)	bias	RPD
Raw	10	0.6276	7.10	0.6486	6.75	-0.73	1.70
Smoothing (7)	13	0.6221	7.15	0.6559	6.68	-0.50	1.71
Normalize	9	0.5956	7.40	0.6528	6.71	-0.70	1.71
MSC	9	0.6027	7.33	0.6496	6.74	-0.74	1.70
First derivative (1)	8	0.6809	6.57	0.6599	6.64	-0.71	1.72
Second derivative (7)	78	0.6178	7.19	0.6450	6.79	-0.74	1.69
SNV	10	0.6154	7.21	0.6474	6.76	-0.56	1.69
SNV+ smoothing	12	0.5942	7.41	0.6434	6.80	-0.23	1.68
SNV+ first derivative	72	0.6491	6.89	0.6654	6.59	-0.71	1.74
SNV+ second derivative	7	0.6093	7.27	0.6370	6.86	-0.86	1.67
MSC+ smoothing	11	0.5846	7.50	0.6443	6.79	-0.76	1.68
MSC+ first derivative	4	0.6479	6.90	0.6663	6.58	-0.76	1.74
MSC+ second derivative	8	0.6154	7.21	0.6454	6.78	-0.96	1.70

Table 41 Calibration and validation results for moisture content of seed of whole longanfruit samples by HSI.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, SNV: standard normal variate, (1 and 7): number of left and right average point

Pre processing	PLS	Са	Calibration set		Validation set		RPD
	factor	R^2	RMSEC(%)	R^2	RMSEP(%)	bias	KPD
Raw	10	0.7183	11.64	0.7396	11.06	0.10	1.96
Smoothing (7)	14	0.7455	11.06	0.7458	10.93	0.31	1.98
Normalize	9	0.7011	11.99	0.7279	11.31	0.19	1.92
MSC	9	0.7093	11.82	0.7255	11.36	0.20	1.91
First derivative (4)	10	0.7409	11.16	0.7400	11.06	0.60	1.96
Second derivative (7)	8	0.7420	11.14	0.7368	11.12	1.01	1.96
SNV	10	0.7193	11.62	0.7262	11.34	0.25	1.91
SNV+ smoothing	13	0.7215	= 11.57	0.7315	11.23	0.42	1.93
SNV+ first derivative	10	0.7193	11.62	0.7262	11.34	0.25	1.91
SNV+ second derivative	7	0.7154	11.70	0.7176	11.52	0.97	1.89
MSC+ smoothing	12	0.7187	11.63	0.7294	11.27	0.36	1.92
MSC+ first derivative	Y	0.6758	12.48	0.7076	11.72	0.36	1.85
MSC+ second derivative	7	0.7139	11.73	0.7177	11.52	0.98	1.89

Table 42 Calibration and validation results for moisture content of whole longan fruitsamples by HSI.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, SNV: standard normal variate, (4 and 7): number of left and right average point

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Pre processing	PLS	Ca	libration set	Validation set			RPD
	factor	R^2	RMSEC(%)	R^2	RMSEP(%)	bias	KFD
Raw	10	0.7509	10.57	0.6961	11.54	0.03	1.81
Smoothing (4)	14	0.7698	10.16	0.7065	11.34	0.15	1.85
Normalize	9	0.7302	11.00	0.6834	11.78	0.31	1.78
MSC	10	0.7596	10.39	0.6967	11.53	-0.02	1.82
First derivative (1)	10	0.8191	9.01	0.7113	11.25	0.64	1.86
Second derivative (5)	10	0.7860	9.80	0.7175	11.13	0.61	1.88
SNV	11	0.7659	10.25	0.7198	11.49	0.22	1.82
SNV+ smoothing	12	0.7430	10.74	0.6911	11.64	0.12	1.80
SNV+ first derivative	9	0.7933	9.63	0.7124	11.23	0.89	1.87
SNV+ second derivative	9	0.7629	10.32	0.7157	11.16	0.69	1.88
MSC+ smoothing	11	0.7420	10.76	0.6910	11.64	0.07	1.80
MSC+ first derivative	9	0.7947	9.60	0.7167	11.15	0.74	1.88
MSC+ second derivative	9	0.7624	10.33	0.7166	11.15	0.61	1.88

 Table 43 Calibration and validation results for moisture content of flesh and seed of whole longan fruit samples by HSI.

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Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, SNV: standard normal variate, (1 and 4): number of left and right average point

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Pre processing	PLS	Cali	bration set		Validation se	et	
	factor	R^2	RMSEC	R^2	RMSEP	bios	RPD
		ĸ	(°Brix)	ĸ	(°Brix)	bias	
Raw	8	0.7308	12.46	0.6209	14.27	2.17	1.64
Smoothing (7)	13	0.7968	10.83	0.6170	14.34	3.93	1.68
Normalize	11	0.8200	10.19	0.6328	14.04	3.27	1.70
MSC	7 Ar	0.7109	12.91	0.6156	14.36	1.64	1.62
First derivative (1)	6	0.7724	11.46	0.6053	14.56	3.29	1.63
Second derivative (9)	6	0.7355	12.35	0.5754	15.10	3.52	1.58
SNV	20	0.7288	12.51	0.6285	14.12	2.06	1.66
SNV+ smoothing	9	0.7580	11.81	0.6162	14.35	3.00	1.65
SNV+ first derivative	5	0.7536	11.92	0.6117	14.44	2.13	1.62
SNV+ second derivative	12	0.7702	11.51	0.6134	14.41	3.20	1.65
MSC+ smoothing	11	0.7729	11.45	0.6676	13.36	3.05	1.78
MSC+ first derivative	5	0.7533	11.93	0.6114	14.44	2.06	1.62
MSC+ second derivative		0.7720	11.47	0.6188	14.31	3.13	1.66

Table 44 Calibration and validation results for TSS in flesh of dried whole longan fruitsamples by HSI.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, SNV: standard normal variate, (1,7 and 9): number of left and right average point

	PLS	Cali	bration set		Validation se	et	
Pre processing	factor	R^2	RMSEC	R^2	RMSEP	bias	RPD
	laotor		(mg/g)		(mg/g)	5105	
Raw	8	0.5957	89.84	0.5260	90.80	3.63	1.45
Smoothing (1)	8	0.5859	90.93	0.5280	90.61	3.58	1.43
Normalize	7	0.5798	91.60	0.4988	93.37	8.01	1.42
MSC	9(A)	0.6619	82.15	0.5072	92.59	18.01	1.45
First derivative (4)	5	0.5491	94.88	0.5147	91.87	0.53	1.43
Second derivative (3)	2	0.3800	111.25	0.3717	104.54	11.12	1.27
SNV	90	0.6068	82.03	0.4817	94.95	20.32	1.42
SNV+ smoothing	11	0.6821	79.66	0.5108	92.25	20.43	1.47
SNV+ first derivative	5	0.5802	91.55	0.4913	94.06	3.21	1.40
SNV+ second derivative	13	0.3309	115.58	0.3468	106.59	10.51	1.24
MSC+ smoothing	10	0.6704	81.12	0.5247	90.93	19.32	1.48
MSC+ first derivative	5	0.5800	91.57	0.5000	93.30	2.95	1.41
MSC+ second derivative		0.3271	115.91	0.3482	106.48	10.51	1.24

Table 45 Calibration and validation results for sucrose in flesh of dried whole longan fruitsamples by HSI.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, SNV: standard normal variate, (1,3 and 4): number of left and right average point

	PLS	Cali	bration set	Validation set			
Pre processing	factor	R^2	RMSEC	R^2	RMSEP	bias	RPD
			(mg/g)	R	(mg/g)	5103	
Raw	6	0.2268	27.41	0.1938	25.25	-1.17	1.11
Smoothing (4)	10	0.3110	25.88	0.2042	25.08	-0.59	1.12
Normalize	5	0.2455	27.08	0.1833	25.41	-1.84	1.11
MSC	5 A)	0.2732	26.58	0.2190	24.85	-0.87	1.13
First derivative (5)	5	0.2574	26.87	0.2369	24.56	-0.27	1.14
Second derivative (7)	3	0.2199	27.54	0.2868	23.75	-1.42	1.18
SNV	50	0.2143	27.64	0.1781	25.49	0.83	1.10
SNV+ smoothing	8	0.3069	25.96	0.2513	24.33	-1.88	1.16
SNV+ first derivative	4	0.2790	26.47	0.2252	24.75	-1.31	1.14
SNV+ second derivative	3~)	0.2221	27.50	0.2699	24.03	-2.25	1.17
MSC+ smoothing	9	0.3177	25.75	0.2408	24.50	-1.82	1.15
MSC+ first derivative	4	0.2797	26.46	0.2225	24.80	-1.32	1.13
MSC+ second derivative	3	0.2217	27.51	0.2698	24.03	-2.26	1.17

Table 46 Calibration and validation results for glucose in flesh of dried whole longan fruitsamples by HSI.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, SNV: standard normal variate, (4, 5 and 7): number of left and right average point

	PLS	Calil	oration set	Validation set			
Pre processing	factor	R^2	RMSEC	R^2	RMSEP	bias	RPD
			(mg/g)		(mg/g)	bido	
Raw	8	0.5036	25.39	0.4684	24.46	0.98	1.37
Smoothing (1)	8	0.4966	25.56	0.4676	24.48	1.04	1.37
Normalize	6	0.4467	26.80	0.4540	24.79	2.08	1.36
MSC	7A)	0.4791	26.01	0.4824	24.14	2.48	1.40
First derivative (4)	6	0.4796	25.99	0.4944	23.86	2.54	1.41
Second derivative (4)	2	0.3355	29.37	0.4022	25.94	3.02	1.30
SNV	<u>JO</u>	0.4959	25.58	0.4580	24.70	1.17	1.36
SNV+ smoothing	72	0.4854	25.85	0.4603	24.65	1.08	1.36
SNV+ first derivative	6	0.4911	25.70	0.4785	24.23	3.10	1.40
SNV+ second derivative	12	0.3231	29.64	0.3947	26.10	3.37	1.30
MSC+ smoothing	C T	0.4688	26.26	0.4832	24.12	2.36	1.40
MSC+ first derivative	5	0.4747	26.12	0.4841	24.10	3.11	1.40
MSC+ second derivative		0.3190	29.73	0.3956	26.09	3.43	1.30

Table 47 Calibration and validation results for fructose in flesh of dried whole longan fruitsamples by HSI.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, SNV: standard normal variate, (1 and 4): number of left and right average point

	PLS	Cali	bration set	Validation set			
Pre processing	factor	R ²	RMSEC	R^2	RMSEP	bias	RPD
			(mg/g)		(mg/g)	bidb	
Raw	7	0.6459	103.41	0.5226	116.73	12.65	1.46
Smoothing (1)	7	0.6442	103.68	0.5204	117.00	12.58	1.45
Normalize	7	0.6822	97.98	0.5093	118.36	9.68	1.43
MSC	7(A)	0.6981	95.49	0.5289	115.96	13.25	1.47
First derivative (5)	8	0.6985	95.42	0.5264	116.27	7.74	1.46
Second derivative (7)	6	0.6824	97.94	0.5135	117.84	3.92	1.43
SNV	ZÒ	0.7113	93.38	0.5129	117.92	14.22	1.44
SNV+ smoothing	78	0.7078	95.07	0.5075	118.57	13.39	1.43
SNV+ first derivative	8	0.7419	88.30	0.5227	116.73	10.47	1.45
SNV+ second derivative	4	0.5912	111.12	0.4491	125.40	9.16	1.35
MSC+ smoothing	A LAND	0.6883	97.03	0.5333	115.42	12.98	1.47
MSC+ first derivative	8	0.7324	89.91	0.5286	116.00	9.42	1.46
MSC+ second derivative	4	0.5901	111.28	0.4630	123.81	9.31	1.37

Table 48 Calibration and validation results for total sugars in flesh of dried whole longanfruit samples by HSI.

Abbreviations: R²: coefficients of determination, RMSEC: root mean square error of calibration, RMSEP: root mean square error of prediction, RPD: ratio of standard deviation of reference data in validation set to SEP, MSC: multiplicative scatter correction, SNV: standard normal variate, (1, 5 and 7): number of left and right average point



Calculation of moisture content (MC) for the whole fruit

1. Calculate %weight of each part $\frac{\text{Fresh weight of peel}}{\text{Fresh weight of peel+flesh+seed}} \times 100$ - % Weight of peel = Fresh weight of flesh Fresh weight of peel+flesh+seed × 100 - % Weight of flesh = Fresh weight of seed Fresh weight of peel+flesh+seed × 100 - % Weight of seed 2. Calculate %MC of the whole fruit by %weight %weight of peel ×%MC peel (calculate from Eq.1) - % MC peel by %weight 100 %weight of flesh ×%MC flesh (calculate from Eq.1) - % MC flesh by %weight 100 %weight of seed ×%MC seed (calculate from Eq.1) - % MC seed by %weight 100 ... %MC of the whole fruit = % MC peel by %weight + % MC flesh by

%weight + % MC seed by %weight

Calculation of moisture content (MC) for flesh and seed part

- 1. Calculate %weight of each part
 - Fresh weight of flesh - % Weight of flesh × 100 = Fresh weight of peel+flesh+seed
 - Fresh weight of seed - % Weight of seed × 100 = Fresh weight of peel+flesh+seed

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- 2. Calculate %MC of flesh and seed by %weight
 - %weight of flesh ×%MC flesh (calculate from Eq.1) - % MC flesh by %weight 100
 - %weight of seed ×%MC seed (calculate from Eq.1) - % MC seed by %weight = 100
 - ✤ %MC of flesh and seed = % MC flesh by %weight + % MC seed by

%weight

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Nomenclature

1, 7	number of left and right averaging point			
°Brix	degree brix			
°C	degree Celsius			
CCD	charge-couple device			
cm	centimeter			
CV.	cultivar			
g	gram			
HSI	hyperspectral imaging			
LV	latent variable			
MC	moisture content			
mg	milligram			
ml	millimeter			
MLR	multiple-linear regression			
mm	millimeter			
MSC	multiplicative scatter correction			
NIR	near infrared			
NIRS	near infrared spectroscopy			
nm	nanometer			
No.				
PLS	partial least square			
R	correlation coefficient			
R^2	coefficient of determination			
RMSEProot mean square error of prediction				
ROI	region of interest			
RPD	ratio of standard deviation of reference data in validation set to SEP			
SNV	standard normal variate			

TSS total soluble solids

- W width
- µm micrometer



Curriculum Vitae

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Conference

Aleeya Lerdkuson, Wikarnda suwannaporn, Sarawut Phupaichitkun and Busarakorn Mahayothee. 2013. Applying Hyperspectral Imaging to Detect Chemical Properties in Pineapple. In the Engineering and Technology Conference during 2-3 Dec 2013 at faculty of Engineering and Industrial Technology, Silpakorn University, Nakhon Pathom, Thailand. (Poster presentation)

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