

EVALUATION OF SENSORY QUALITY OF RICE IN RICE QUALITY IMPROVEMENT  
PLANT USING NEAR INFRARED SPECTROSCOPY

RAVIPAT LAPCHAROENSUK

A THESIS SUBMITTED IN PARTIAL FULFILLMENT  
OF THE REQUIREMENT FOR THE DEGREE OF  
DOCTOR OF ENGINEERING IN AGRICULTURAL ENGINEERING  
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### บทคัดย่อ

งานวิจัยนี้มีวัตถุประสงค์เพื่อพัฒนาเทคนิคเนียร์อินฟราเรดสเปกโทรสโกปีเพื่อการตรวจสอบเชิงปริมาณและเชิงคุณภาพของคุณภาพทางประสาทสัมผัสของข้าว นอกจากนี้การศึกษาค้นคว้าแตกต่างของคุณภาพทางประสาทสัมผัสของข้าวตามชนิดของข้าวและการศึกษาความสัมพันธ์ระหว่างเนื้อสัมผัสทางประสาทสัมผัสกับเนื้อสัมผัสที่วัดได้จากเครื่องได้ถูกศึกษาด้วย ในการศึกษาครั้งนี้ใช้ตัวอย่างข้าวสาร 4 ชนิด ได้แก่ ข้าวเหนียว ข้าวขาว ข้าวหอมมะลิใหม่และข้าวหอมมะลิเก่า คุณภาพทางประสาทสัมผัสของข้าวประกอบด้วย ความร่วน ความแข็ง ความเหนียว ความแห้ง ความขาว และความหอม ถูกประเมินโดยผู้ทดสอบทางประสาทสัมผัสที่ถูกฝึกฝน ข้าวสารและข้าวสอยถูกสแกนด้วยเครื่อง FT-NIR Spectrometer ที่ความยาวคลื่น 350 – 2,500 นาโนเมตร เครื่อง short wavelength diode array ที่ความยาวคลื่น 700 – 2,500 นาโนเมตร แบบจำลองเนียร์อินฟราเรดสเปกโทรสโกปีเชิงปริมาณถูกสร้างโดยใช้วิธี partial least squares regression แบบจำลองเนียร์อินฟราเรดสเปกโทรสโกปีเชิงคุณภาพถูกสร้างโดยใช้วิธี soft independent modeling of class analogy (SIMCA) and partial least squares discriminant analysis (PLS-DA) คุณภาพทางประสาทสัมผัสของข้าวทุกค่าแสดงความแตกต่างอย่างมีนัยสำคัญทางสถิติตามชนิดของข้าว สมการเชิงเส้นถูกสร้างจากความสัมพันธ์ระหว่างเนื้อสัมผัสทางประสาทสัมผัสกับเนื้อสัมผัสที่วัดได้จากเครื่อง แสดงค่า  $r$  เท่ากับ 0.54 – 0.94 สำหรับผลของการทำนายคุณภาพทางประสาทสัมผัสของข้าวสารโดยการใช้สเปกตรัมของข้าวสารที่ได้จากเครื่อง FT-NIR Spectrometer แสดงค่า  $R^2_{val}$  ในช่วงระหว่าง 0.876 ถึง 0.920 ในขณะที่ค่า  $R^2_{val}$  ของแบบจำลองที่สร้างขึ้นจากสเปกตรัมของข้าวสอยเท่ากับ 0.650 ถึง 0.690 แบบจำลองที่สร้างจากสเปกตรัมของข้าวสารที่ได้จากเครื่อง FT-NIR spectrometer สามารถใช้ประเมินคุณภาพทางประสาทสัมผัสของข้าวในโรงงานปรับปรุงคุณภาพข้าวแบบจำลองเนียร์อินฟราเรดสเปกโทรสโกปีสำหรับคุณภาพทางประสาทสัมผัสของข้าวซึ่งใช้สเปกตรัมของข้าวสารและข้าวสอยที่ได้จากเครื่อง short wavelength diode array ให้ผลค่า  $R^2_{val}$  อยู่ในช่วง

0.823 ถึง 0.918 แบบจำลองที่ได้นำเสนอนี้สามารถใช้ในการประเมินคุณภาพในประสาทสัมผัสของข้าวได้ ผลการทำนายคุณภาพทางประสาทสัมผัสของข้าวโดยใช้แบบจำลองเนียร์อินฟราเรดสเปกโทรสโกปีที่สร้างจากสเปกตรัมของข้าวสารและข้าวสอยจากเครื่อง long wavelength diode array แสดงค่า  $R^2_{val}$  ในช่วง 0.194 ถึง 0.544 แบบจำลองเนียร์อินฟราเรดสเปกโทรสโกปีที่สร้างจากสเปกตรัมของข้าวสารและข้าวสอยจากเครื่อง long wavelength diode array ไม่สามารถใช้ในการประเมินคุณภาพในประสาทสัมผัสของข้าวได้ นอกจากนั้นผลการทดลอง จากกราฟสัมประสิทธิ์ความสัมพันธ์ของแบบจำลองเหล่านั้นจากเครื่อง NIR spectrometer ทั้งสามเครื่องระบุได้ว่าน้ำไม่มีความสัมพันธ์กับคุณภาพทางประสาทสัมผัสของข้าว อย่างไรก็ตามผลการทดลองแสดงให้เห็นว่าแป้งเป็นองค์ประกอบทางเคมีหลักที่มีผลต่อคุณภาพทางประสาทสัมผัสอย่างชัดเจน ความผิดพลาดโดยรวมของแบบจำลองในการแบ่งกลุ่มด้วยวิธี SIMCA ซึ่งสร้างจากสเปกตรัมของข้าวสารและข้าวสอยจากเครื่อง FT-NIR spectrometer short wavelength diode array และ long wavelength diode array มีค่ามากกว่า 10 เปอร์เซ็นต์ สิ่งนี้บ่งบอกได้ว่าแบบจำลองจากวิธี SIMCA เหล่านี้ไม่สามารถใช้ในการแบ่งกลุ่มคุณภาพทางประสาทสัมผัสของข้าวได้ ค่าความผิดพลาดทั้งหมดจากแบบจำลองการแบ่งกลุ่มทั้งหมดด้วยวิธี PLS-DA จากเครื่อง FT-NIR spectrometer และ short wavelength diode array มีค่ามากกว่า 10 เปอร์เซ็นต์ สิ่งนี้บ่งบอกได้ว่าแบบจำลองจากวิธี PLS-DA จากเครื่อง FT-NIR spectrometer และ short wavelength diode array ไม่สามารถใช้ในการแบ่งกลุ่มคุณภาพทางประสาทสัมผัสของข้าวได้ สำหรับเครื่อง short wavelength diode array ค่าความผิดพลาดทั้งหมดจากแบบจำลองของ ความร้อน ความแห้ง และความเหนียวซึ่งสร้างจากสเปกตรัมของข้าวสารและความร้อนและความหอมซึ่งสร้างจากสเปกตรัมข้าวสอยแสดงน้อยกว่า 10 เปอร์เซ็นต์ ดังนั้นแสดงว่าแบบจำลอง PLS-DA เหล่านี้สามารถนำไปใช้งานได้ ค่าความผิดพลาดทั้งหมดของแบบจำลองจากวิธี PLS-DA โดยใช้สเปกตรัมของข้าวสารจากเครื่อง long wavelength diode array ของค่าความร้อน ความแข็งแรง และความหอมมีค่ามากกว่า 10 เปอร์เซ็นต์ ดังนั้นจึงกล่าวได้ว่าแบบจำลองเหล่านี้ไม่สามารถใช้งานได้

<b>Thesis</b>	Evaluation of Sensory Quality of Rice in Rice Quality Improvement Plant Using Near Infrared Spectroscopy
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## ABSTRACT

This research aimed to develop near infrared spectroscopy for quantitative and qualitative evaluation of the sensory qualities of cooked rice using milled rice or cooked rice spectra. In addition, the sensory quality of rice of different types and relationship between sensory texture attributes and instrumental texture properties of rice were studied. Four different types of milled rice were used: parboiled, white, new Jasmine, and aged Jasmine. The sensory qualities of cooked rice (adhesiveness, hardness, stickiness, dryness, whiteness, and aroma) were evaluated by trained sensory panelist method. Milled rice and cooked samples were scanned by FT-NIR spectrometer at 12,500-4,000  $\text{cm}^{-1}$  (800-2,500 nm), short wavelength diode array at 600 – 1,100 nm and long wavelength diode array at 1,150-2,150 nm. Near infrared spectroscopy quantitative models were established using partial least squares regression. The near infrared spectroscopy qualitative models were created by soft independent modeling of class analogy (SIMCA) and partial least squares discriminant analysis (PLS-DA). All sensory qualities of rice were significantly different among types of rice. The linear equations relating to the texture properties by instrument and by sensory test were calculated where the  $r$  were between 0.54 – 0.94. For FT-NIR Spectrometer, predicted results of sensory qualities using milled rice spectra showed the  $R^2_{\text{val}}$  values between 0.876 and 0.920 while the  $R^2_{\text{val}}$  values of models developed from cooked rice spectra were 0.650 to 0.690. The models created from milled rice spectra of FT-NIR spectrometer can be used to evaluate sensory quality of rice in rice quality improvement plant. The NIR spectroscopy models for sensory quality of

cooked rice using milled rice and cooked rice spectra scanning scanned by short wavelength diode array yielded  $R^2_{\text{val}}$  between 0.823 and 0.918. The proposed models can be utilized for evaluation of sensory quality of rice. Prediction results of sensory quality of rice using NIR spectroscopy models created by milled rice and cooked rice spectra from long wavelength diode array showed the  $R^2_{\text{val}}$  between 0.194 and 0.544. The NIR spectroscopy models created from spectra of long wavelength diode array were not applicable to evaluate sensory quality of rice. The regression coefficient plots of these models from three NIR spectrometer indicated that the water content of the rice samples was not related to the sensory quality of the cooked rice. However, it was clearly apparent that starch as the main chemical component of rice, was the dominant factor. The total error of all SIMCA classification models created from milled rice and cooked spectra of FT-NIR spectrometer, short wavelength diode array and long wavelength diode array were more than 10%. This indicated that these SIMCA models could not be used for classification of sensory quality of rice. The total error of all PLS-DA classification models by spectra of milled and cooked rice from FT-NIR spectrometer were more than 10%. This indicated that these PLS-DA classification models could not be used for classification of sensory quality of rice. For short wavelength diode array, the total error of adhesiveness, dryness and stickiness (for milled rice) and adhesiveness and aroma (for cooked rice) were less than 10% (8.2 – 9.7% and 9.1 and 8.1%, respectively). Therefore, these models were fairly models while other models were not applicable. The total error of the PLS-DA models using milled rice spectra scanned by long wavelength diode array for sensory quality of rice was more than 10%. Therefore these PLS-DA models were not applicable.

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Ravipat Lapcharoensuk

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# Chapter 1

## INTRODUCTION

### 1.1 BACKGROUND OF THE RESEARCH

Rice (*Oryza sativa* L) is one of the principal foods consumed by humans around the world especially in Asia. Important rice products include milled rice and parboiled rice. Milled rice is produced by removing the husk, bran layer and the germ. In contrast, parboiled rice is unprocessed rice that has undergone partial steaming.

Consumer's appreciation of rice depends upon the quality of the cooked rice product. Determination of the sensory quality of cooked rice is closely monitored in the process of rice processing and milling industry. During the rice production process, sampling is conducted to determine the six key sensory quality attributes: adhesiveness, hardness, dryness, stickiness, aroma and whiteness. At present, the sensory quality of samples is determined using a descriptive analytical sensory analysis by trained human panelists. Unfortunately, sensory based analysis methods are slow, complex and lead to a destruction of the test objects. Therefore, a novel analytical method that is fast, easy and nondestructive for evaluating sensory quality of rice is highly sought after within the rice processing industry. Near infrared (NIR) spectroscopy is a nondestructive method that has been applied to evaluate a variety of properties of agricultural products and foods. NIR spectroscopy is efficient because it dramatically reduces the time needed for experimental analysis, thereby leading to lower overall costs. Researchers have studied the application of NIR spectroscopy to analyze the amylose content, protein content, and lipid content of rice, with satisfactory outcomes (Shimizu et al., 1999; Shu et al., 1999; Bao et al., 2001 and Chang et al., 2010). Researchers also reported the use of NIR spectroscopy to predict the sensory characteristics of foods such as virgin olive oil (Inarejos-García et al., 2013), apple (Mehinagic et al., 2003), table grape (Parpinello et al., 2013), lamb meat (Kamruzzaman et al., 2013) and beef steaks (Liu et al., 2003). These studies show coefficient of determination ( $R^2$  value) between 0.17 and 0.72.

The goal of this research was to investigate the relationship between the sensory quality of cooked rice and its near infrared spectral data measured using an FT-NIR spectrometer, FQA-NIR gun and MICRONIR spectrometer. The results from this study could prove useful in real world applications associated with the rice processing and marketing.

## **1.2 OBJECTIVE**

1.2.1 The objective of this research was to study sensory quality of rice according to different types of rice i.e. parboiled rice (processed from white rice), white rice (from varieties other than Jasmine), new Jasmine rice (Khao Dawk Mali 105 variety; harvested in 2012 and sold to the plant where it was evaluated that year) and aged Jasmine rice (harvested either in 2006 or 2007–2011; “aged” means the rice was kept for at least 1 year postharvest before processing and evaluation) and relationship between sensory texture attributes and instrumental texture properties of rice, including adhesiveness, hardness, and stickiness.

1.2.2 This research aimed to establish the near infrared spectroscopy models for prediction and classification of sensory quality of rice, including adhesiveness, hardness, dryness, stickiness, aroma, and whiteness which could be applied to rice industry.

## **1.3 HYPOTHESIS**

The sensory quality of cooked rice including adhesiveness, hardness, dryness, stickiness, aroma, and whiteness has the correlation with the near infrared spectral characteristic of milled rice and cooked rice.

## 1.4 SCOPE

This research was limited to using near infrared spectroscopy method in the wavelength range of 700-2,500 nm for prediction adhesiveness, hardness, dryness, stickiness, aroma, and whiteness of cooked rice that is indicated of sensory quality of rice. About 250 rice samples were collected from the rice improvement factory (C.P. Rice Co., Ltd.). Rice samples consisted of parboiled rice, white rice, new Jasmine rice and aged Jasmine rice.

## 1.5 PROCEDURE

1. The literatures about using near infrared spectroscopy for evaluating rice quality were reviewed.
2. The sensory quality evaluation method used in rice industry was investigated.
3. Sensory panelists consisted of 10 people (5 males and 5 females) who were trained for reference sensory evaluation.
4. The 250 samples of milled rice were collected from rice improvement factory (C.P. Rice Co., Ltd.). The rice samples were collected from 3 stations in the factory including raw material receiving station, in front of color sorter and under the storage bin. Milled rice samples consisted of parboiled rice, white rice, new Jasmine rice and aged Jasmine rice which are routinely received as raw material in processes of the plant.
5. Milled rice and cooked sample was scanned with FT-NIR Spectrometer (MPA, Bruker, Germany) at  $12,500-4,000\text{ cm}^{-1}$  (800-2,500 nm), FQA-NIR GUN at 600–1,100 nm and MicroNIR spectrometer at 1,150-2,150 nm.
6. Then the milled rice was cooked with rice:water ratio following rice improvement factory and the cooked rice was evaluated for sensory quality

(adhesiveness, hardness, dryness, stickiness, aroma and whiteness) by sensory panelists.

7. The cooked rice samples were subjected to the texture analyzer (TA HD Plus, Stable Micro System, London, UK) using Back Extrusion test platform. The texture parameters were determined from force-deformation curve. The determined texture parameters were stickiness (maximum negative force, N) hardness (maximum force, N), toughness (area under curve above zero force, Nmm) and adhesiveness (area under curve below zero force, Nmm). The relationships between sensory texture of cooked rice and instrumental texture of cooked rice were studied.

8. The near infrared spectra of rice may have been interfered by external factor such as non-uniform of temperature. Therefore, the spectra had to be preprocessing by the chemometric such as first derivative, second derivative, multiplicative scatter correction (MSC), normalization, baseline offset, standard normal variate (SNV) and detrending.

9. Next, the NIRS model was developed using the spectral data and the sensory quality reference data by partial least square regression (PLS) for predicted sensory quality. Models accuracy was determined by statistics which were coefficient of determination ( $R^2$ ), standard error of calibration (SEC), standard error of prediction (SEP) and bias. Classification models were developed by soft independent modeling of class analogy (SIMCA) and partial least squares discriminant analysis (PLS-DA) method.

10. The results of thesis were summarized.

## Chapter 2

# LITERATURE REVIEW

### 2.1 RICE QUALITY IMPROVEMENT PLANT

The rice quality improvement plants affect the economy of Thailand as they produced a large volume of rice for export and domestic consumption. The wholesale prices of milled rice vary by the moisture contents of the rice, while the retail prices i.e. prices paid by consumers depend upon the rice sensory quality, a practice that has been adopted by many including the rice quality improvement plant. The raw material of the plants is milled rice with different varieties including Jasmine rice and white rice (other varieties except Jasmine rice) and parboiled rice which is produced from white rice.

The improvement process for milled rice raw materials processed in quality improvement plants includes cleaning, destoning and polishing, grading by size, sorting by color, winnowing, silo storage, and packaging. Quality-improved rice products are developed according to formulas that are a combination of different categories of raw materials designed by the plant's research and development section. The various quality attributes of raw materials such as moisture, instrument texture parameters, color, and tested sensory quality were previously measured by traditional methods before the rice formulas were developed.

During these processes the rice is sampling for quality evaluation on milled and cooked rice. The quality index of milled rice included moisture content, whiteness, transparency and color while the quality index of cooked rice is evaluated for texture for quality assurance and control at the raw material receiving station, behind color sorter and under storage bin.

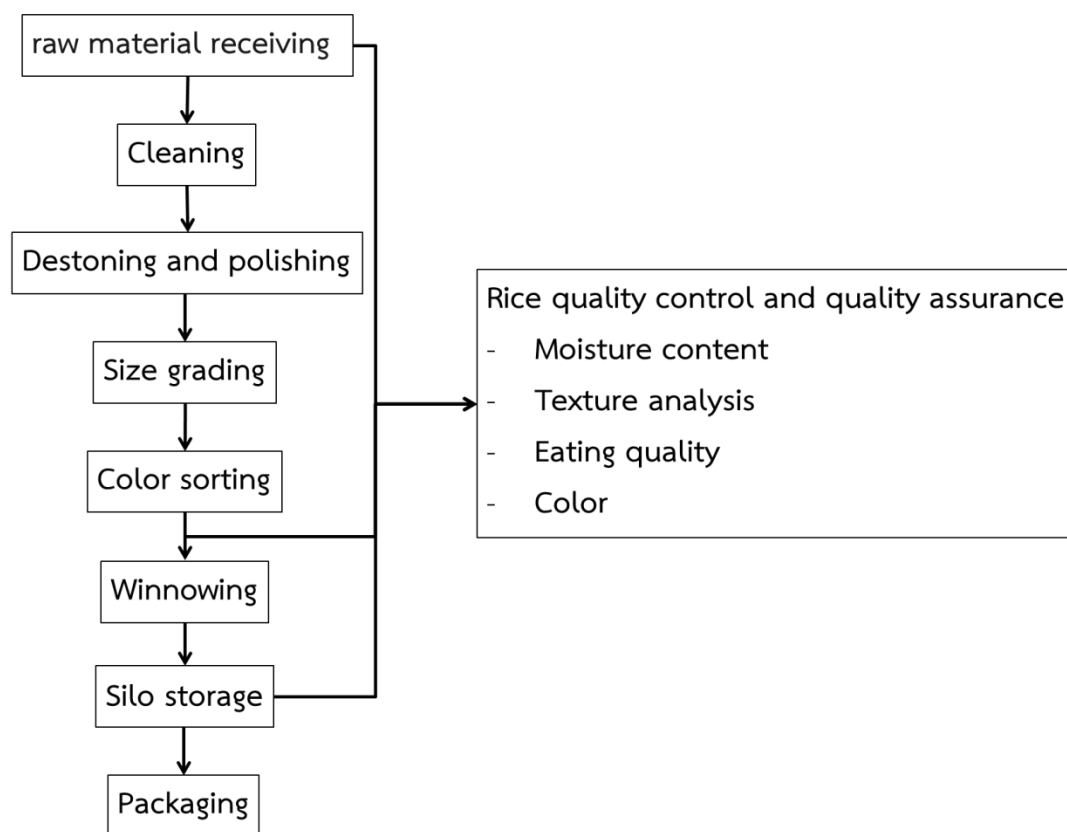


Figure 2.1 Rice quality improvement processing

## 2.2 EVALUATION OF SENSORY QUALITY OF RICE

Sensory quality evaluation of rice is important in deciding some quality of rice through the sensory assessed by human. The rice industries have used sensory quality evaluation for assessing flavor, aroma, visual and texture properties of rice that were obvious attributes that could not be measured easily by instruments and to indicate the human perception. Typically descriptive analysis by trained panelist is a popular method to evaluate sensory quality of rice in rice industries.

In the 1940's, the first trained panels were developed in an effort to make measurements of food more objective, given the inherent subjectivity and variability of human evaluators (Gengler, 2009). Viriyajaree. (2002) describe the following *“Trained panelists method is a method of measuring the sensory characteristics of a product. A small group of panelists is selected and trained to describe the characteristics they perceive in a product. The numbers of panelists were 8-20*

persons who were selected from different gender, ages and regions. The panelists provide quantitative information about product differences that can be used to guide new product development and maintain product quality. The evaluation of sensory attribute was measured by score levels which were rated in 5, 7 or 9 levels. Therefore, the panel can be used regularly as an analytical tool". Trained panelists method has been used to assess quality of rice in many studies. Sensory attributes of rice evaluated by several researchers are shown in table 2.1.

**Table 2.1** Definitions of sensory attribute of rice.

Sensory attribute	Definitions	Reference
<b>Visual attribute</b>		
Appearance	- The comprehensive attribute including brightness, head rice yield and shape of cooked rice.	Qingyun et al. (2007)
Brightness	- Brightness showed by the surface of cooked rice often generated by oil.	Qingyun et al. (2007)
Whiteness	- The surface characteristics of cooked rice.	Srisawas et al. (2007)
Glossiness	- The surface characteristics of cooked rice.	Srisawas et al. (2007)

Table 2.1 (Continued)

Sensory attribute	Definitions	Reference
<b>Aroma attribute</b>		
Aroma	- The odor intensity of cooked rice.	Qingyun et al. (2007)
Fragrance	- The aroma of cooked rice.	Srisawas et al. (2007)
<b>Flavor attribute</b>		
Taste	- The sense of sweetness when chewed and passed through the throat.	Qingyun et al. (2007)
<b>Texture attribute</b>		
Adhesiveness to lips	- Degree to which the sample adheres to the lips.	Meullenet et al. (2002)
Manual and visual adhesiveness	- A teaspoon of rice was placed on plate and manipulated with the back of the spoon.	Windham et al. (1997)
Chewiness	- Amount of work to chew the sample. - Rice was evaluated during chew	Champagne et al. (2001) Windham et al. (1997)
Cohesiveness	- Degree to which the grains deform rather than crumble, crack, or break when bitten with molars. One-half teaspoon of rice was evaluated at first bite.	Champagne et al. (2001) Windham et al. (1997)
Cohesiveness of mass	- Maximum degree to which the sample holds together in a mass while being chewed. - Degree to which chewed sample holds together. - Rice was evaluated during chew.	Champagne et al. (2001) Meullenet et al. (2002) Windham et al. (1997)
Hardness	- The force needed to bite through cooked rice. - The sample between molars and evaluating the force required to bite through the sample.	Qingyun et al. (2007) Srisawas et al. (2007) Yang et al. (2013)

Table 2.1 (Continued)

Sensory attribute	Definitions	Reference
<b>Texture attribute</b>		
Hardness	- Force required to bite through the sample with the molars.	Champagne et al. (2001)
	- Force required to compress the sample.	Meullenet et al. (2002)
Loose particle	One-half teaspoon of rice was evaluated at first bite.	Windham et al. (1997)
	- Particles remaining in and on the surface of mouth after swallowing.	Meullenet et al. (2002)
Initial starchy coating	- Amount of pastelike thickness perceived on the product before mixing with saliva (three passes).	Champagne et al. (2001)
	- The surface of rice placed behind front teeth.	Windham et al. (1997)
Moisture absorption	- Amount of saliva absorbed by sample during chewing.	Champagne et al. (2001)
Particle size	- Amount of space the particle fills in mouth.	Meullenet et al. (2002)
Residual loose particle	- Amount of loose particles in mouth.	Champagne et al. (2001)
	- Swallowed characteristics of rice.	Windham et al. (1997)
Roughness	- Amount of irregularity in the surface of the product.	Champagne et al. (2001)
	- The surface of rice placed behind front teeth.	Windham et al. (1997)
Roughness of mass	- Amount of roughness perceived in chewed sample.	Meullenet et al. (2002)
Self-adhesiveness	One-half teaspoon of rice was evaluated at first bite.	Windham et al. (1997)
Slickness	- Maximum ease of passing tongue over the rice surface when saliva starts to mix with sample	Champagne et al. (2001)

Table 2.1 (Continued)

Sensory attribute	Definitions	References
<b>Texture attribute</b>		
Springiness	- The degree of grains returning to original shape after partial compression.	Yang et al. (2013)
	- Degree to which grains return to original shape after partial compression.	Champagne et al. (2001)
	One-half teaspoon of rice was evaluated at first bite.	Windham et al. (1997)
Starchy mouth coating	- Swallowed characteristics of rice.	Windham et al. (1997)
Stickiness	- The chewing stage as the degree to which the sample adhered to the teeth or palate.	Yang et al. (2013)
	- Maximum ease of passing tongue over the rice surface when saliva starts to mix with sample.	Champagne et al. (2001)
	- The degree of adhesion of cooked rice on the teeth.	Qingyun et al. (2007)
	- The sample adhered to the teeth or palate.	Srisawas et al. (2007)
Stickiness to lips	- Rice was compressed lightly between lips.	Windham et al. (1997)
Surface slickness	- The surface of rice placed behind front teeth.	Windham et al. (1997)
Toothpack	- Amount of product adhering in or on teeth.	Champagne et al. (2001)
	- Amount of product packed into the crowns of teeth after mastication.	Meullenet et al. (2002)
	- Swallowed characteristics of rice.	Windham et al. (1997)
Toothpull	- Force required to separate jaws during mastication.	Meullenet et al. (2002)
Uniformity of bite	- Rice was evaluated during chew	Windham et al. (1997)

**Table 2.1** (Continued)

Sensory attribute	Definitions	Reference
<b>Overall liking</b>		
Eating quality	The comprehensive evaluation for cooked rice.	Qingyun et al. (2007)
Overall acceptability	The rate of overall liking on sensory properties.	Yang et al. (2013)

### 2.3 FACTORS AFFECTING ON SENSORY QUALITY OF RICE

Sensory quality of rice is influenced by many factors such as variety, chemical component, rice processing method and cooking method. Rice varieties relate to chemical component and grain quality characteristics. Furthermore, rice processing, such as handing, drying, storage and rice milling; also affect sensory quality of rice. Finally, different cooking methods affect sensory quality of rice.

**Table 2.2** Chemical constituents of parboiled rice, white rice and Jasmine rice.

Chemical component	Parboiled rice (Chanklang, 2004)	White rice (Chanklang, 2004)	Jasmine rice (Sunghanghwa, 2010)
Moisture content (%)	11.2-12.2	10.33-11.57	14.0
Protein content (%)	6.13-9.11	7.15-7.94	6.20
Lipid content (%)	1.86-2.08	1.40-1.50	1.10
Total carbohydrate (%)	76.16-80.24	74.69-74.75	80.40
Fiber content (%)	0.12-0.23	2.88-2.95	0.60
Ash content (%)	0.80-1.10	2.88-2.95	-

Chemical component of milled rice mainly affected sensory quality of rice. The table 2.2 show Chemical component of parboiled rice, white rice and Jasmine rice. Typically, rice consisted of about 76.16 – 80.40% of carbohydrates, 6.13 - 7.94% of protein and about 15% of the remainder. Amylose and protein was formerly considered as the most important predictor of texture properties for cooked rice

(Champagne et al., 2001 modified from Juliano et al., 1965; Sowbhagya et al., 1987; Mundo et al., 1989). Protein is the major nutrient of rice, but the rice with high protein content will become light yellow (Chang et al., 2010). Lipid may compound with higher molecular weight amylose and the longer amylopectin chains during cooking and also influence cooked rice texture (hardness, less stickiness) (Champagne et al., 2001 modified from Priestley, 1976; Biliaderis et al., 1993; Ong and Blanshard, 1995)

**Table 2.3** Correlation coefficient ( $r$ )<sup>a</sup> values between chemical component and sensory texture attribute of cooked rice.

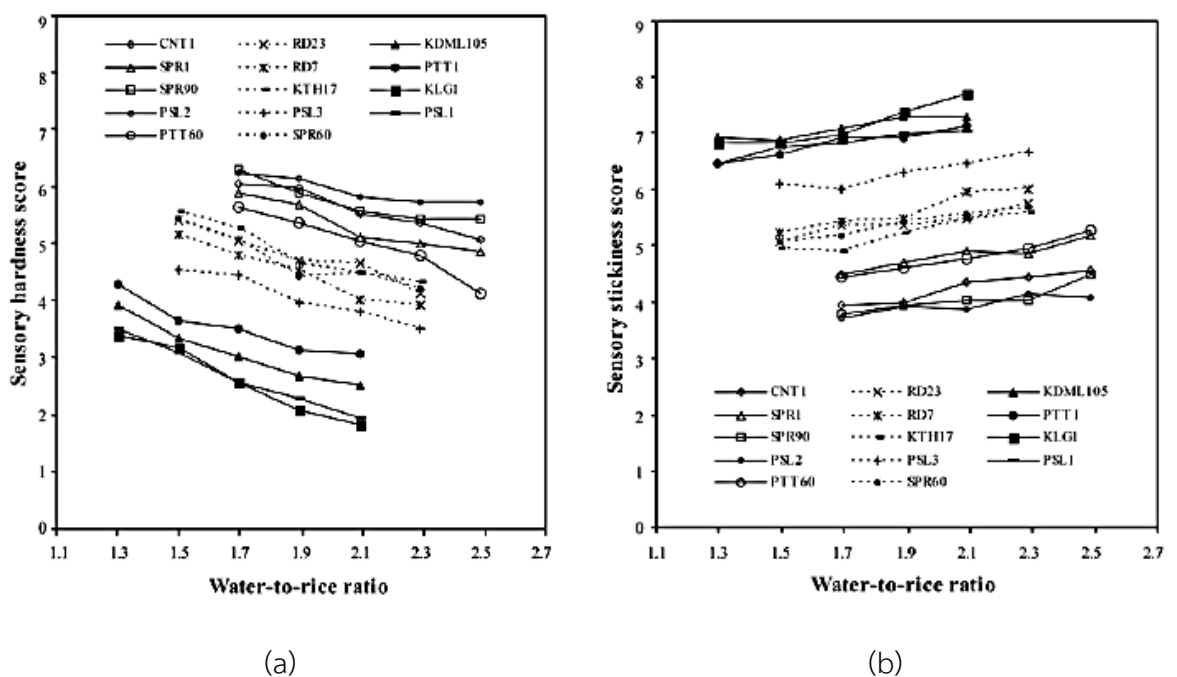
Texture attribute	Amylose	Protein	Lipid
Initial starchy coating	-0.60	-0.38	0.21
Slickness	-0.29	-0.51	0.22
Roughness	0.01	0.36	-0.23
Stickiness	-0.64	-0.38	0.05
Springiness	0.27	0.19	-0.04
Cohesiveness	-0.35	-0.15	0.08
Hardness	0.46	0.30	-0.12
Cohesiveness of mass	-0.77	-0.29	0.14
Chewiness	-0.10	0.01	-0.14
Uniformity of bite	-0.32	-0.10	0.06
Moisture absorption	-0.34	-0.26	0.21
Residual loose particles	-0.11	0.23	-0.17
Toothpack	-0.43	-0.05	0.08

<sup>a</sup>Correlation coefficient ( $r$ ) values at  $P < 0.05$

**Source** Champagne et al. (2001)

Champagne et al. (2001) studied relationship between chemical component and texture attribute of cooked rice. The results are shown in table 2.3. The cohesiveness of mass, initial starchy coating, and stickiness showed negative correlation (more than 0.60) with amylose.

The other factor, the ratio of water to rice and time of cooking, showed important effects on sensory quality of rice. Srisawas et al. (2007) reported effects of varieties and water to rice ratio for cooking on sensory texture attribute of rice. The sensory texture attribute of rice consisted of hardness and stickiness. Figure 2.2 showed sensory evaluation scores of cooked rice as functions of water-to-rice ratio for 14 Thai milled rice varieties. All texture attributes significantly differed according water to rice ratio and varieties. Figure 2.2(a) displayed hardness score decreased with increasing of the amount of water used in cooking. Stickiness score exhibited an increase with the water-to-rice ratio as shown in Figure 2.2(b). Both results revealed that the rice variety was a major effect on sensory texture attributes.



**Figure 2.2** Sensory evaluation scores of cooked rice as a function of water-to-rice ratio for 14 Thai milled rice varieties: (a) hardness and (b) stickiness.

**Source** Srisawas et al. (2007)

## 2.4 NEAR INFRARED SPECTROSCOPY AND ITS APPLICATION FOR SENSORY QUALITY OF RICE

Near infrared reflectance spectroscopy (NIRS) is a nondestructive and rapid technique which has been applied to evaluate food quality. NIRS is an integration of electromagnetic science, computer science and chemometrics for evaluating quality of food and agricultural products. Cen and He, (2007) said “the chemical component with 0.1% concentration could be detected and evaluated by NIRS”. NIR spectroscopy was first used in agricultural applications by Norris (1964) to evaluate moisture in grain. The increasing importance of NIR spectroscopy for quality evaluation is obvious from the recent increase in numbers of publication. This part is a review of the recent literatures, discussion of a fundamental theory, chemometrics and application of NIR in sensory quality evaluation of rice.

### 2.4.1 Theory and features of NIR spectroscopy

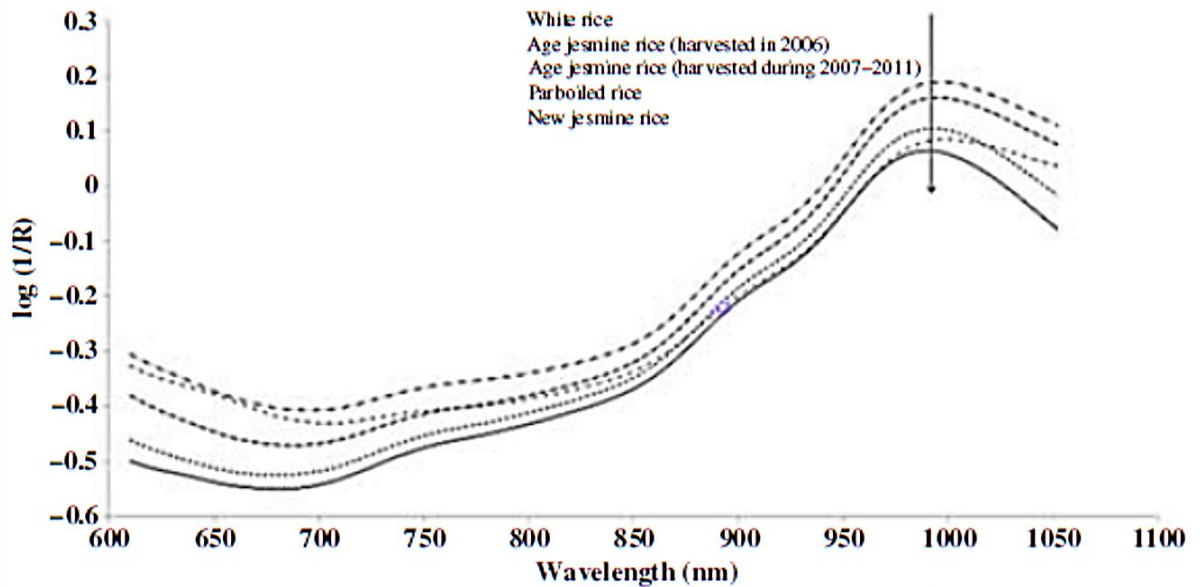
Near infrared (NIR) radiation is electromagnetic wave which range from 780 to 2,500 nm (12,500 - 4,000  $\text{cm}^{-1}$ ). When near infrared radiation emit on sample, the incident radiation may be reflected, absorbed or transmitted in which each phenomenon depends on the chemical constitution and physical parameters of the sample. The intensity of reflection or transmission radiation ( $I_R$  or  $I_T$ ) is related to the intensity incident on this sample ( $I_0$ ) to the path length in the sample ( $b$ ) and to the concentration of the desired component ( $c$ ) by the equation of Beer-Lambert (Imsil, 2012 modified from Osborne et al., 1993a). The absorbance of each wavelength often plotted as  $\log (1/I_R)$  versus wavelength (Shown in Figure 2.3).

$$\log \left( \frac{I_0}{I_{R,T}} \right) = abc \quad (2.1)$$

Where:

$a$  = absorptivity of the component at a particular wavelength

$\log \left( \frac{I_0}{I_{R,T}} \right) =$  absorbance (A)



**Figure 2.3** Visible and Shortwave NIR (600-1100 nm) spectra of rice.

**Source** Lapcharoensuk and Sirisomboon (2015)

Near infrared spectra is from overtone or combination bands which originated from the fundamental vibrations. The fundamental vibrations modes are stretching (i.e., symmetric and asymmetric) and bending (i.e., rocking, scissoring, wagging and twisting). Vibration in which there is a continuous change in the interatomic distance along the axis of the bond between the two atoms is known as stretching (Osborne & Fearn, 1986). In the case of a triatomic group of atoms  $AX_2$ , stretching may occur symmetrically whereby the two A-X bonds vibrate in and out together or, asymmetrically when they vibrate in opposite directions (Osborne & Fearn, 1986). Vibration involving a change in bond angle is known as bending and may be further classified into four types, scissoring, rocking, wagging and twisting (Osborne & Fearn, 1986). In Figure 2.4, the fundamental vibrations modes are shown. Overtone bands are multiple frequencies of fundamental vibrations: 2 times ( $1^{st}$  Overtone), 3 times ( $2^{nd}$  Overtones) and etc. Intensities of overtones are 1/10- 1/100 of fundamental vibrations in in the spectral range between 780 and 2000 nm (Cho, 2013). Combination bands are combination of 2 or more fundamental vibrations that give new absorption bands in spectral range of between 1900 and 2500 nm (Cho, 2013). Typically the NIR bands are limited on hydrogen bond such as O-H, C-H, S-H, N-H and etc. Another functional group relative to NIR spectroscopy can include carbon to

oxygen stretch, carbon to nitrogen stretch, carbon to carbon stretch, and metal halides (Imsil, 2012). The specific molecular bonds active are shown in the Table 2.4.

The NIR region is divided into short-wave NIR (SW-NIR) and common NIR at 1300 nm where the range of short-wave NIR (SW-NIR) and common NIR are 780-1300 and 1300-2500 nm respectively. The SW-NIR region is considered as the absorption band of high overtones, while the latter belongs to first or second overtone. The absorption intensity will decrease when the overtone increases. Thus, SW-NIR is usually applied in the transmission analysis with long path length, and common NIR is used in diffuse reflection analysis (Cen and He, (2007).

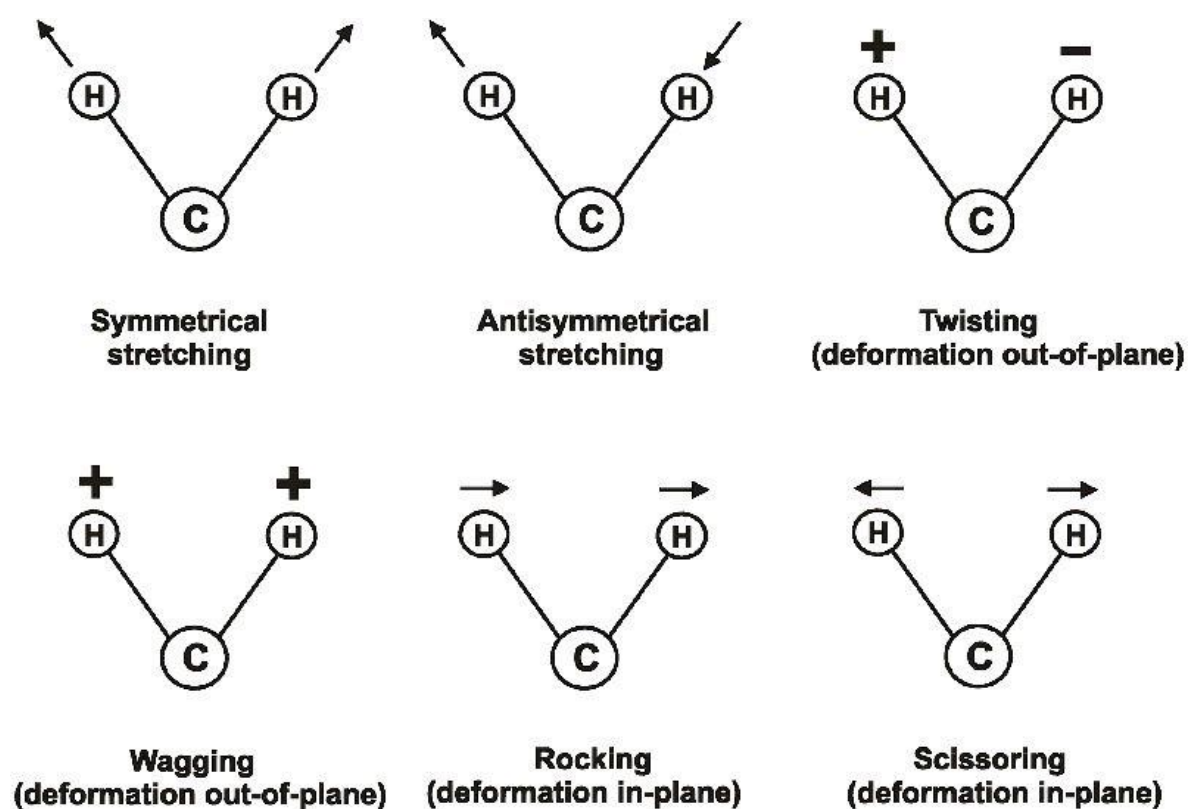


Figure 2.4 The fundamental vibrations modes.

Source Nawrocka and Lamorska (2013)

**Table 2.4** The specific molecular bonds active.

Molecular bonds	Molecular bonds
C=O from aldehydes	C-N from amines, alkyl
C=O from amides	C-N from amines, aromatic
C=O from carboxylic acids	C-O from alcohols, ethers and esters
C=O from esters	N-H from amides
C=O from ketones	N-H from amines
C-H from aldehydes	NO <sub>2</sub> from nitro groups
C-H from alkanes	O-H from alcohols (no hydrogen bonding)
C-H from alkenes	O-H from alcohols (with hydrogen bonding)
C-H from alkynes	O-H from carboxylic acids
C-H from aromatic compounds	

**Source** Imsil (2012) modified from Workmand and Weyer (2008)

Cen and He, (2007) said *“The whole measurement processing of NIR spectroscopy generally consists of the following several steps: (1) spectral data acquisitions; (2) data pre-processing to eliminate noises and baseline shift from the instrument and background; (3) to build calibration models using a set of samples with known analyzed concentration obtained by suitable reference methods; and (4) to validate the models using another set of samples without the calibration set”*

#### 2.4.2 Spectra pre-processing

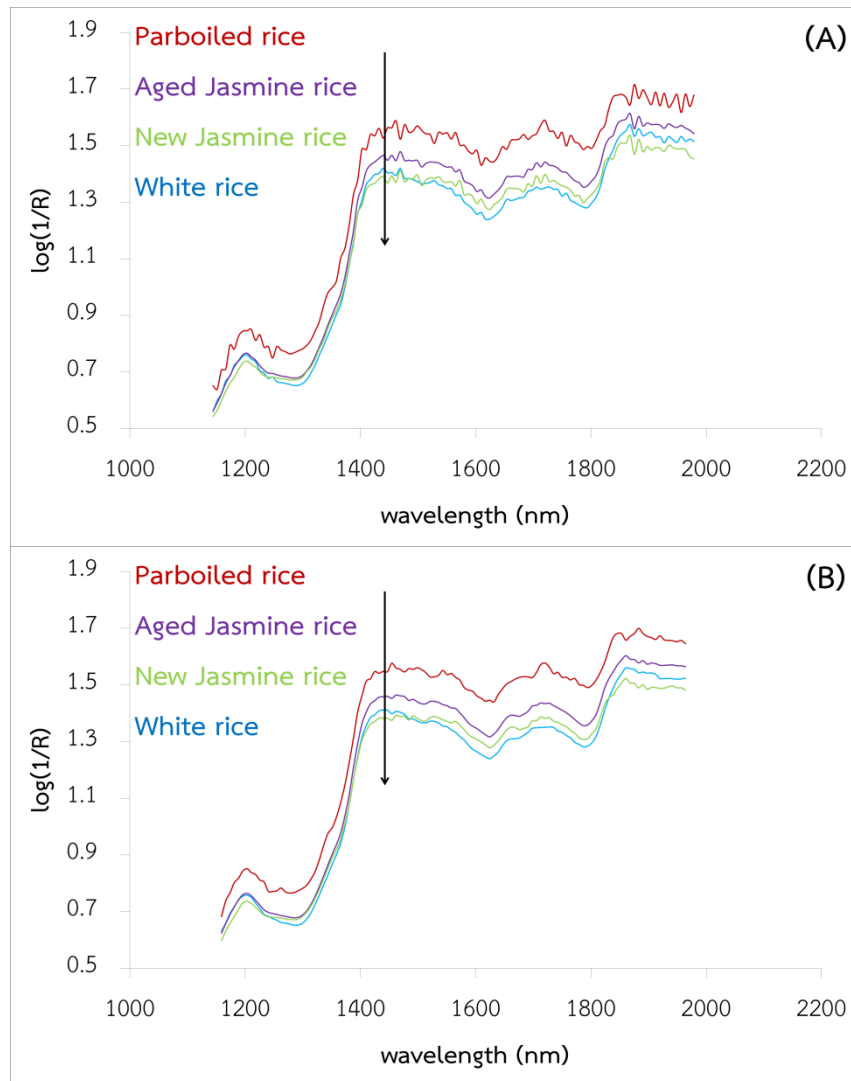
Raw NIR spectral data can cause an offset error or multiplicative errors due to many effects such as instrument sources, sample sources and operational sources. Spectral pre-processing techniques are used to remove any irrelevant information which cannot be handled properly by the regression techniques (Nicolai et al., 2007). Several pre-processing methods are utilized to modify the spectral data. Methods often used which benefit to correct NIR spectra data are listed in Table 2.5. Pre-processing methods are explained in this literature review.

**Table 2.5** Spectra Pre-processing method.

<b>Spectra Pre-processing</b>	<b>Benefit</b>
Smoothing	Smoothing is a kind method for de-noising.
Multiplicative scatter correction (MSC)	MSC is used to correct multiplicative scattering effects in spectral data.
Frist and second derivative	Derivation is employed to increase spectral resolution.
Normalization	Normalization is used to get all data in approximately the same scaling, or to get a more even distribution of the variances and the average values.
Baseline offset	The method is used to correct the baseline of samples
Standard normal variate (SNV)	SNV is normalized to zero mean and unit variance of each individual spectrum.
Detrending	Detrending is a transformation which seeks to remove nonlinear trends in spectroscopic data.
SNV+Detrending	SNV+Detrending is combined to reduce multicollinearity, baseline shift and curvature.

#### 2.4.2.1 Smoothing

The smoothing techniques have been proposed to remove random noise from NIR spectra. While smoothing obviously improves the visual aspect of NIR spectra, it does remove information at stage where it is not clear yet whether this information is useful (Nicolai et al., 2007). Popular smoothing methods are moving average filter and Savitzky-Golay algorithm (Nicolai et al., 2007; Neas et al., 2004).



**Figure 2.5** Spectra of milled rice (A) original milled rice spectra (B) milled spectra pre-processing by the Savitzky-Golay smoothing at  $N = 5$  and polynomial order 2.

The moving-average method is the simplest type of smoothing. The absorbance of each variable is replaced with an average of the adjacent observations (including itself). The number of observations on which to average is the user-chosen “segment size” parameter. The moving-average absorbance value of each variable is calculated by equation 2.2 (Ozaki et al., 2007).

$$X_{\text{Moving-average}} = \sum_{k=-n}^n w_k X_{k+n} \quad (2.2)$$

Where:

$X_{\text{Moving-average}}$	=	moving-average absorbance value of each variable
$w_k$	=	defining the smoothing, is called the convolution weight.
$n$	=	number of points beside the center point ( $N = (2n-1)$ ; $N =$ number of points to be average)
$k$	=	the center point

The Savitzky-Golay algorithm fits a polynomial to each successive curve segment, thus replacing the original values with more regular variations. The idea of Savitzky-Golay method is the vicinity of measurement point a spectrum can be fitted by polynomials (Ozaki et al., 2007). In practice, the convolution weight ( $w_k$ ) is calculated by fitting the spectrum with polynomials using least squares regression. The  $w_k$  value is calculated from the different order of polynomials and  $N$  ( $N = 2n+1$ ). The original milled rice spectra and milled spectra pre-processing by the Savitzky-Golay smoothing at  $N = 5$  and polynomial order 2 are shown in Figure 2.5.

#### 2.4.2.2 Multiplicative scatter correction (MSC)

NIR spectral data may include additive (baseline shift) and multiplicative effects which are induced by physical effects, such as the non-uniform scattering through-out the spectrum as the degree of scattering is dependent on the wavelength of the radiation, the particle size and the refractive index (Nicolai et al., 2007). Those effects often happen on grain samples in which particle size of each grain is different.

The basis of MSC lies in the fact that light scattering has wavelength dependence different from that of chemically based light absorbance (Ozaki et al., 2007; Geladi et al., 1985). MSC corrects spectra which rotates each spectrum with the average spectra over a set of samples (Imsil, 2012; Rinnan et al., 2009). Thus, the first state of MSC is calculation average spectra. Then, each spectrum ( $x_i$ ) is transformed according equation 2.3 (Conzen, 2006).

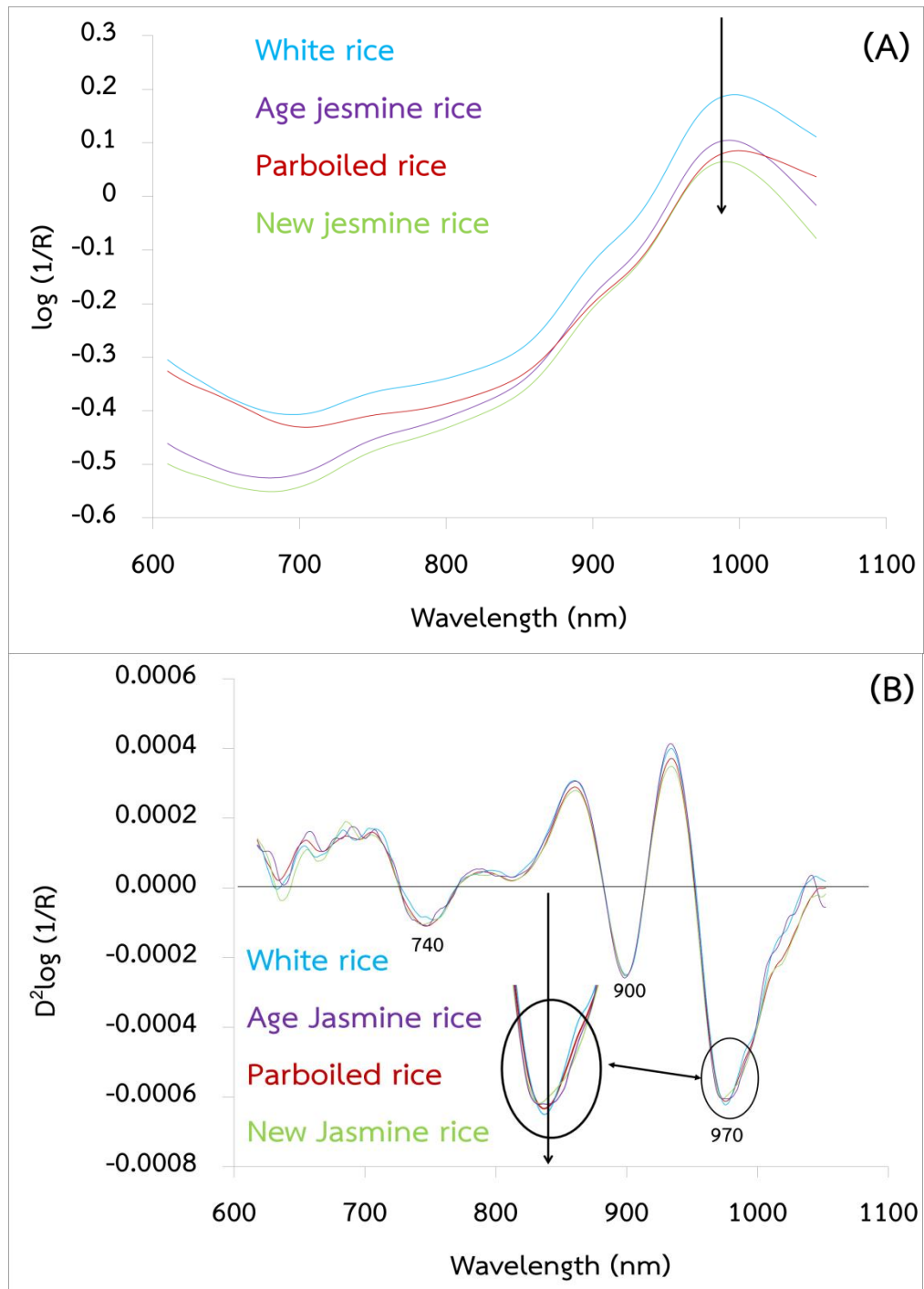
$$x_{MSC} = u + v(x_i) \quad (2.3)$$

Where:

- $x_{MSC}$  = MSC absorbance value of each variable
- $x_i$  = absorbance value of each variable
- $u$  and  $v$  = the coefficients value are chosen such that the difference between the transformed spectrum and the mean spectrum has a minimum.

#### 2.4.2.3 Derivative

Derivative is used often to remove baseline shift and superposed peaks which have long been used in NIR spectroscopy. The second order of derivative is most popularly used for clearing additive and multiplicative effects (Nicolai et al., 2007; Ozaki et al., 2007). They are usually calculated according to Savitzky-Golay algorithm (Nicolai et al., 2007; Naes et al., 2004). The superimposed peaks of second derivative spectral invent graphically from original spectral. Figure 2.6 shows the original and second derivative spectra of milled rice. The original spectra of milled rice showed the one obvious peak at 970 nm (Figure 2.5A). After spectra were pre-processing with Savitzky-Golay second derivative method, the spectral displays obvious peaks at 744, 900, and 970 nm. In addition, spectral data are eliminated baseline shift and/or baseline offset.



**Figure 2.6** The original and second derivative spectra of rice obtained with the Savitzky-Golay method.

#### 2.4.2.4 Normalization

Normalization methods are used to suppress unwanted source of variability by making a group of spectra have more features in common. Normalization is used

to eliminate the influence of different optical path lengths in case of transmission (Conzen, 2006). Many techniques have been invented for normalization on spectral data which are vectors normalization, max normalization, range normalization and mean normalization. The formula of those techniques is listed in the table 2.6.

**Table 2.6** The formula of normalization techniques.

Normalization technique	Formula	Parameter
Vector normalization	$X_{\text{vector nor}} = \frac{x_i}{\sqrt{\sum_k x_{i,k}^2}}$	$X_{\text{vector nor}}$ = Vector normalization absorbance value of each variable $x_i$ = Absorbance value of each variable $k$ = number of variable
Mean normalization	$X_{\text{mean nor}} = \frac{x_i}{x_{\text{mean}}}$	$X_{\text{mean nor}}$ = Mean normalization absorbance value of each variable $x_i$ = Absorbance value of each variable $x_{\text{mean}}$ = Mean of spectral data
Max normalization	$X_{\text{max nor}} = \frac{x_i}{x_{\text{max}}}$	$X_{\text{max nor}}$ = Maximum normalization absorbance value of each variable $x_i$ = Absorbance value of each variable $x_{\text{max}}$ = Maximum of spectral data
Range normalization	$x'_{\text{range nor}} = \frac{x_i}{(x_{i,\text{max}} - x_{i,\text{min}})}$	$X_{\text{range nor}}$ = Maximum normalization absorbance value of each variable $x_i$ = Absorbance value of each variable $x_{\text{min}}$ = Minimum of spectral data

**Source** Cho (2013)

### 2.4.2.5 Baseline offset

Baseline offset method is used to correct the baseline of samples. Baseline shifts are also induced by the influence of optical fiber cables (Ozaki et al., 2007). The formula for the baseline offset correction can be written as follows equation 2.5 (CAMO Software AS., 2007).

$$X_{\text{baseline}} = X - X_{\text{min}} \quad (2.5)$$

Where:

$X_{\text{baseline}}$	=	baseline offset absorbance value of each variable
$X$	=	absorbance value of each variable
$X_{\text{min}}$	=	Minimum of spectral data

### 2.4.2.6 Standard normal variate (SNV) and Detrending

Standard normal variate (SNV) is a technique usually applied to pre-process spectral data. This method can be used to remove scatter effects by centering and scaling each individual spectrum (i.e. a sample-oriented standardization). The results of SNV are similar to multiplicative scatter correction (MSC) which removes multiplicative interferences of scatter and particle size effects from spectral data. Each absorbance value ( $X_i$ ) in a spectrum of data is transformed according to the equation 2.6 (CAMO Software AS., 2007).

$$X_{\text{SNV}} = \frac{X_i - X_{\text{mean}}}{\text{SD}} \quad (2.6)$$

Where:

$X_{\text{SNV}}$	=	SNV absorbance value of each variable
$X_i$	=	absorbance value of each variable
$X_{\text{mean}}$	=	Mean of spectral data
SD	=	standard deviation

Detrending is a transformation which seeks to remove nonlinear trends in spectroscopic data. This pre-processing method calculates a baseline function as a least squares fit of a polynomial to the sample spectrum.

SNV is often used with Detrending for pre-processing spectral data. This combination can reduce multicollinearity which has baseline shift and curvature in spectral data. The SNV-corrected data may still be affected by baseline curvature. DT can remove this effect by using a second-order (or higher degree) polynomial in regression analysis (CAMO Software AS., 2007). Thus combination of SNV and detrending is interested to pre-process spectral data.

### **2.4.3 NIR spectroscopy calibration models for qualitative and quantitative analysis**

Calibration models creating are very important procedure for qualitative and quantitative analysis in using NIR spectroscopy, which involves the prediction of discrimination and property for unknown samples. Many mathematical techniques have been used to develop for quantitative analysis for example multiple linear regression (MLR), principle component regression (PCR), partial least square regression (PLSR), artificial neural network (ANN) and etc. For qualitative analysis, principle component analysis (PCA), soft independent modeling of class analogy (SIMCA), partial least square discriminant analysis (PLS-DA), artificial neural network (ANN) are most developed methods in agricultural product and food quality evaluation. However, in this thesis, some techniques were used for qualitative and quantitative analysis which were principle component analysis (PCA), soft independent modeling of class analogy (SIMCA), partial least square discriminant analysis (PLS-DA), and partial least square regression (PLSR). Thus, in literature review only those techniques will be explained.

### 2.4.3.1 Principle component analysis (PCA)

Principal component analysis (PCA) is multivariate analysis technique used to reduce the number of variable and solve the multicollinearity problem (Maitra and Yan, 2008; Bair et al., 2006). The aim of PCA is explaining most of the variance in the data while reducing the number of variable to few uncorrelated component (Imsil, 2012). The information carried by the original variables is projected onto a smaller number of underlying (latent) variables called principal components (PCs). The first principal component ( $PC_1$ ) covers as much of the variation in the data as possible. The second principal component ( $PC_2$ ) is orthogonal to the first and covers as much of the remaining variation as possible and so on (CAMO Software AS., 2007). By plotting the principal components, one can view inter-relationships between different variable, and detect and interpret sample patterns (Imsil, 2012; Anderson, 2003).

Benefits of PCA are identification groups of variables. Identification groups of variables by PCA based on the loading that is correlation between the variables and the principal component; and group of individual based on the principle scores. The result of PCA is generally called “score” (equivalent to the variable) which is concentrated onto a few underlying variables. Each sample has a score along each model component. The score shows the locations of the samples along each model component, and can be used to detect sample patterns, grouping, similarities and differences (Imsil, 2012; Anderson, 2003).

### 2.4.3.2 Partial least square regression (PLSR)

Partial Least Square regression (PLSR) is mathematical technique which has been used in chemometric analysis. The PLS is the most popular multivariate techniques which have been used for creating NIR spectroscopy calibration model. The PLSR is a technique that generalizes and combines features from principle component analysis (PCA) and multiple linear regressions (MLR). The Figure 2.7 shows concept of PLS. The PLS technique is particularly useful to predict a dependent variable data (quality of agricultural product and food) by large set of independent

variable data (NIR spectral). Formula of PLSR shows in equation 2.7 (Ozaki et al., 2007).

$$Y=Xb \quad (2.7)$$

Where:

- X = a matrix containing the predictor variable.  
 Y = a column matrix containing the dependent variable.  
 b = a parameter that is determined by least-squares regression.

In practice, PLSR can be applied to create calibration model on 2 modes (i.e., PLS1 and PLS2). The PLS1 is developed for prediction one dependent variable by one model. While, the PLS2 is used to predict many dependent variables by one model.

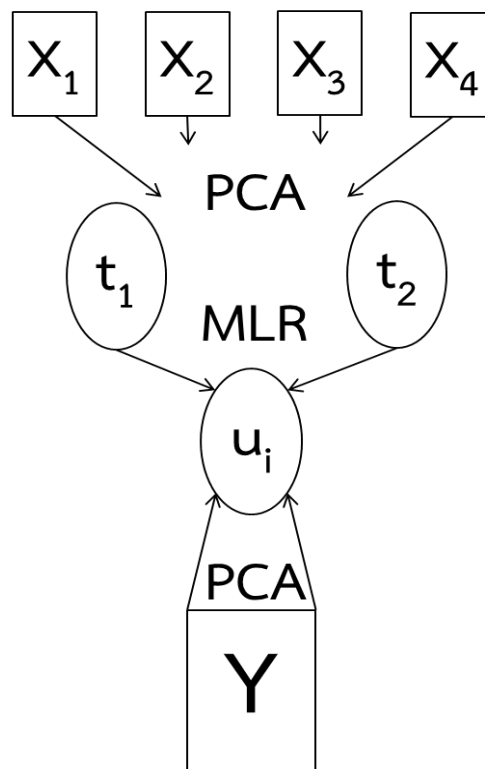


Figure 2.7 Concept of PLS

Source Cho (2013)

### 2.4.3.3 Soft independent modeling of class analogies (SIMCA)

Generally PCA is not useful for classification because the class information is not used in the construction of the PCA model. Thus, the PCA model just describes the overall variation in the data. Anywise PCA can be worked together with the class information by soft independent modeling of class analogies (SIMCA). SIMCA is the first and one of the best-known modeling classification methods (Ballabio and Todeschini, 2009).

SIMCA models each class independently by PCA, for each class, an acceptance boundary defined by the maximum residuals of the samples from that group is created. Each class is expected to be modeled by a different number of principal components (PCs) (Shiqiang et al., 2015). Ballabio and Todeschini, (2009) explained SIMCA algorithm that “the PCA is separately calculated on each class; since the number of significant component can be different for each category, cross-validation has been proposed as a way of choosing the number of retained components of each class model. IN this way, SIMCA defines the number of classes subspaces (class models); then, a new object is projected in each subspace and compared to it in order to assess its distance from the class. Finally, the object assignation is obtained by comparing the distance of the object from the class models.”

### 2.4.3.4 Partial least squares discriminant analysis (PLS-DA)

Primarily, the class models in SIMCA are calculated with the aim of describing variation within each class: when PCA is applied on each category, it finds the directions of maximum variance in the classes, on the opposite of, for example, Partial least squares discriminant analysis (PLS-DA), which directly models the classes on the basis of the descriptors.

Partial least squares discriminant analysis (PLS-DA) is classification method based on modeling the differences between several classes with PLSR. Nowadays, the PLS-DA is one of the most popular techniques used in NIR spectroscopy. The PLS-DA bases on the PLS2 algorithm that searches for latent variable with a

maximum covariance with Y variables. The main difference is related to the dependent variables, since these represent qualitative values, when dealing with classification. In PLS-DA the Y-block describes which objects are in the classes of interest. In binary classification problem, the Y variable can be easily defined by setting its values to 1 if the objects are in the class and 0 if not. Then, the model will give a calculated Y, in the same way as for a regression approach; the calculated Y will not have either 1 or 0 values perfectly, so a threshold (equal to 0.5, for example) can be defined to decide if an object is assigned to the class (calculated Y greater than 0.5) or not (calculated Y lower than 0.5). When dealing with multiclass problems, the same approach cannot be used: if Y is defined with the class numbers (1, 2, 3, ..., n) this would mean that a mathematical relationship between the class exists (for example, that class g is somehow in-between class g - 1 and class g + 1). The solution to this is to unfold the class vector and apply the PLS2 algorithm for multivariate qualitative responses (PLS-DA). For each object, PLS-DA will return the prediction as a vector of classes, with values in-between 0 and 1. Since predicted vectors will not have the form but real values in the range between 0 and 1, a classification rule must be applied; the object can be assigned to the class with maximum value in the Y vector or, alternatively, a threshold between zero and one can be determined for each class (Ballabio and Todeschini, 2009).

#### **2.4.4 Statistical evaluation of accuracy and precision**

Performances of NIR spectroscopy are checked statistically. Typically, statistics are usually used for evaluation performances of NIR spectroscopy which are coefficient of determination  $R^2$ , standard error of calibration (SEC), standard error of prediction (SEP), bias, and ratio of standard error of prediction to the standard deviation (RPD). The  $R^2$  value, the bias and the RPD are the most meaningful statistics for “instant” appraisal of analytical efficiency by NIR spectroscopy (Williams, 2007). Therefore, in this thesis, those values were used and will be explained.

### 2.4.4.1 Coefficient of determination ( $R^2$ )

The coefficient of determination is shown with  $R^2$ . It is calculated for explaining the proportion of the variance in x data that can be described by the variance in the y data (Williams, 2007). Guidelines for the Interpretation of  $R^2$  are listed in the table 2.7. The formula of Coefficient of determination ( $R^2$ ) shows in equation 2.8 (Cenzen, 2006).

$$R^2 = \left[ 1 - \frac{\sum_{i=1}^M (Y_i - Y_i^{\text{pred}})^2}{\sum_{i=1}^M (Y_i - Y_m)^2} \right] \quad (2.8)$$

Where:

$Y_i$	=	Concentration value of sample i
$Y_i^{\text{pred}}$	=	Prediction concentration value of sample i
$Y_m$	=	Mean concentration value
M	=	Number of samples

**Table 2.7** Guidelines for the Interpretation of  $R^2$

$R^2$	Interpretation
Up to 0.25	Not usable in NIR spectroscopy calibration
0.26-0.49	Poor correlation, reason should be researched
0.50-0.64	OK for rough screening
0.66-0.81	OK for screening and some other “approximate” calibrations
0.83-0.90	Usable with caution for most applications, including research
0.92-0.96	Usable in most applications, including quality assurance
0.98+	Excellent, usable in any application

**Source** Williams (2007)

#### 2.4.4.2 Standard error of calibration (SEC) and Standard error of prediction (SEP)

The standard error of calibration (SEC) is computed to check result of prediction of the sample in establishing of the calibration model. SEC is the SD of differences between NIR spectroscopy and reference samples in calibration set (Williams, 2007). Computing of standard error of prediction (SEP) resembles the SEC but the SEP is calculated from standard deviation of differences between NIR spectroscopy and reference in validation set. The formula for those calculation displays in equation 2.9 (Williams, 2007).

$$\text{SEC, SEP} = \sqrt{\frac{1}{M} \sum_{i=1}^M (Y_i^{\text{meas}} - Y_i^{\text{pred}})^2} \quad (2.9)$$

Where:

$Y_i^{\text{meas}}$	=	Measurement concentration value of sample i
$Y_i^{\text{pred}}$	=	Prediction concentration value of sample i
M	=	Number of samples

#### 2.4.4.3 Bias

The bias is the mean of difference between reference and NIR spectroscopy data. It is used to measure of the overall accuracy of the calibration. In the practices, the bias is most important statistical evaluation? because it indicates differences of predicted result by NIR spectroscopy and reference data. The formula for computing bias is presented in equation 2.10 (Williams, 2007).

$$\text{Bias} = \left( \frac{\sum X}{N} \right) - \left( \frac{\sum Y}{N} \right) \quad (2.10)$$

Where:

X	=	reference data
Y	=	NIR spectroscopy data

N = Number of samples

#### 2.4.4.4 Ratio of standard error of prediction to the standard deviation (RPD)

The ratio of standard error of prediction to the standard deviation (RPD) is the relative evaluation of SEP in terms of the standard deviation of the reference data. It is calculated by dividing the SD of the reference values used in the validation set by SEP (Williams, 2007). The table 2.8 illustrates the impact of some values of the RPD.

$$RPD = \frac{SD_{\text{validation set}}}{SEP} \quad (2.11)$$

$$RPD = \frac{1}{(1-R^2)^{\frac{1}{2}}} \quad (2.12)$$

Where:

$SD_{\text{validation set}}$  = standard deviation of the reference values  
 SEP = standard error of prediction (SEP)  
 $R^2$  = coefficient of determination

**Table 2.8** The RPD statistical

RPD value	Classification	Application
0.0-2.3	Very poor	Not recommended
2.4-3.0	Poor	Very rough screening
3.1-4.9	Fair	Screening
5.0-6.4	Good	Quality control
6.5-8.0	Very good	Process control
8.1+	Excellent	Any application

**Source** Williams (2007)

#### 2.4.4.5 Ratio of standard error of prediction to standard deviation (SEP/SD)

The ratio of standard error of prediction to standard deviation (SEP/SD) of the reproducibility test (SEP/SD) was computed to determine the evaluation capability of all the calibration models, where SEP/SD < 1.0 represents an excellent NIR model, 1.0–2.0 a good model, 2.1–2.4 a fair model, 2.5–3.0 a poor model, and >3.0 an unreliable model (Tony Pietrountonio, Caltest, USA, personal communication).

#### 2.4.5 Application of NIR spectroscopy for evaluation of sensory quality of rice

A considerable amount of research has been studied on evaluation of sensory quality of rice using NIR spectroscopy. Applications of NIR spectroscopy for evaluation of sensory quality of rice are listed in the table 2.9

##### 2.4.5.1 Visual sensory attributes

Some researchers investigated the visual sensory attributes, i.e. appearance, brightness and glossiness, using NIR spectroscopy. Qingyun et al. (2007) used multiple linear regression (MLR) method to compare the reflectance and transmission data at wavelength of 540, 640 and 970 nm with appearance and brightness of cooked rice. Three rays were selected for developing model because it could predict some traits of cooked rice (Mikami et al., 2000). The models for these visual sensory attributes were shown in equation 2.13 and 2.14. The coefficient of correlation of validation ( $r_{val}$ ) of appearance is 0.88 and standard error of prediction (SEP) is 0.55. The prediction results of brightness showed  $r_{val} = 0.84$  and SEP = 0.94.

$$\text{Appearance} = -11.517 + 3.219R_1 - 1.330R_2 - 0.978T_1 + 2.374T_2 \quad (2.13)$$

$$\text{Brightness} = -6.324 + 2.820R_1 - 2.200R_2 - 0.0270T_1 + 1.349T_2 \quad (2.14)$$

Where:

$R_1$  and  $R_2$  = reflectance data at 540 and 970 respectively.

$T_1$  and  $T_2$  = transmission data at 540 and 640 respectively.

In 2007, Srisawas et al. studied that PLSR calibration model strongly correlated with glossiness of cooked rice. The calibration model was created from NIR reflectance spectral range of 1100 - 2500 nm. The result showed high ability of prediction ( $R_{cal}^2 = 0.948$ ;  $R_{val}^2 = 0.890$ ; SECV = 0.23; SEP = 0.31; Bias = -0.003; RPD = 2.99).

#### 2.4.5.2 Aroma sensory attributes

Aroma of cooked rice was researched by Qingyun et al. (2007). They developed NIR spectroscopy model by MLR method. The MLR model for evaluation of aroma of cooked rice was shown in equation 2.15. The  $r_{val}$  value of aroma was 0.78 and the SEP was 0.43.

$$\text{Aroma} = -8.686 + 1.461R_1 - 0.255R_2 + 0.313T_1 + 0.609T_2 \quad (2.15)$$

#### 2.4.5.3 Flavor sensory attributes

Qingyun et al. (2007) created the MLR model for evaluation of taste of cooked rice. They used NIR spectral at wavelength length of 540, 640 and 970 nm in reflectance and transmission mode. The equation 2.16 is the MLR model for prediction of taste of cooked rice. The prediction results of taste showed  $r_{val} = 0.84$  and SEP = 0.52.

$$\text{Taste} = -0.482 + 2.048R_1 - 2.252R_2 - 0.036T_1 + 0.708T_2 \quad (2.16)$$

#### 2.4.5.4 Texture sensory attributes

Evaluation of texture attributes of cooked rice by NIR spectroscopy were studied by many researchers. Texture attribute is important indicator for deciding quality of rice in rice industries. Among textural attributes, sensory hardness and stickiness are reported to be the most critical characteristics governing the palatability of cooked rice (Srisawas et al., 2007; Okabe, 1979). Therefore, a study on evaluation of sensory texture attributes using NIR spectroscopy is an interesting research topic.

**Table 2.9** Lists of application of NIR spectroscopy for evaluation of sensory quality of rice

Sensory attributes	References	Acquisition mode	Spectral range (nm)	Pre-processing method	Modelling method	Prediction results
<b>Visual attribute</b>						
Appearance	Qingyun et al. (2007)	Reflectance	540 and 970		MLR	$r_{\text{cat}} = 0.71$ ; $\text{SEC} = 0.89$ ; $r_{\text{val}} = 0.88$ and $\text{SEP} = 0.55$
		Transmission	540 and 640			
Brightness	Qingyun et al. (2007)	Reflectance	540 and 970		MLR	$r_{\text{cat}} = 0.84$ ; $\text{SEC} = 0.72$ ; $r_{\text{val}} = 0.75$ and $\text{SEP} = 0.94$
		Transmission	540 and 640			
Glossiness	Srisawas et al. (2007)	Reflectance	1100-2500	Original spectral	PLS	$R^2_{\text{cat}} = 0.948$ ; $R^2_{\text{val}} = 0.890$ ; $\text{SECV} = 0.23$ ; $\text{SEP} = 0.31$ ; $\text{Bias} = -0.003$ ; $\text{RPD} = 2.99$
<b>Aroma attribute</b>						
Aroma	Qingyun et al. (2007)	Reflectance	540 and 970		MLR	$r_{\text{cat}} = 0.73$ ; $\text{SEC} = 0.49$ ; $r_{\text{val}} = 0.78$ and $\text{SEP} = 0.43$
		Transmission	540 and 640			
<b>Flavor attribute</b>						
Taste	Qingyun et al. (2007)	Reflectance	540 and 970		MLR	$r_{\text{cat}} = 0.81$ ; $\text{SEC} = 0.59$ ; $r_{\text{val}} = 0.84$ and $\text{SEP} = 0.52$
		Transmission	540 and 640			

Table 2.9 (Continued)

Sensory attribute	Author (Year)	Acquisition mode	Spectral range (nm)	Pre-processing method	Models created method	Prediction results
<b>Texture attribute</b>						
Adhesiveness to lips	Meullenet et al. (2002)	Reflectance	400-2500	Second derivative	PLS	$R^2_{\text{cal}} = 0.88$ ; $R^2_{\text{val}} = 0.83$ ; RMSEC = 0.54; RMSEP = 0.51 RPD = 2.37
Manual adhesiveness	Windham et al. (1997)	Reflectance	424-1800	MSC and second derivative	PLS	RAP = 0.57
Visual adhesiveness	Windham et al. (1997)	Reflectance	424-1800	MSC and second derivative	PLS	RAP = 0.54
Cohesiveness	Windham et al. (1997)	Reflectance	424-1800	MSC and second derivative	PLS	See in Figure 2.7
Cohesiveness of mass	Windham et al. (1997)	Reflectance	424-1800	MSC and second derivative	PLS	See in Figure 2.7
	Champagne et al. (2001)	Reflectance	700-1078; 1118-2018	MSC and second derivative	PLS	$R^2_{\text{cal}} = 0.96$ ; $R^2_{\text{val}} = 0.83$ ; RMSEC = 0.12; RMSEP = 0.22 DI = 2.3
	Meullenet et al. (2002)	Reflectance	400-2500	Second derivative	PLS	$R^2_{\text{cal}} = 0.79$ ; $R^2_{\text{val}} = 0.72$ ; RMSEC = 0.61; RMSEP = 0.64 RPD = 1.90

Table 2.9 (Continued)

Sensory attribute	Author (Year)	Acquisition mode	Spectral range (nm)	Pre-processing method	Models created method	Prediction results
<b>Texture attribute</b>						
Hardness	Champagne et al. (2001)	Reflectance	700-1078; 1118-2018	MSC and second derivative	PLS	$R^2_{\text{cal}} = 0.85$ ; $R^2_{\text{val}} = 0.67$ ; RMSEC = 0.22; RMSEP = 0.32 DI = 1.9
	Meullenet et al. (2002)	Reflectance	400-2500	Second derivative	PLS	$R^2_{\text{cal}} = 0.79$ ; $R^2_{\text{val}} = 0.67$ ; RMSEC = 0.29; RMSEP = 0.29 RPD = 1.73
	Qingyun et al. (2007)	Reflectance	540 and 970		MLR	$r_{\text{cal}} = 0.67$ ; SEC = 0.66;
		Transmission	540 and 640			$r_{\text{val}} = 0.56$ and SEP = 0.69
	Srisawas et al. (2007)	Reflectance	1100-2500	Original spectral	PLS	$R^2_{\text{cal}} = 0.950$ ; $R^2_{\text{val}} = 0.915$ ; SECV = 0.27; SEP = 0.31; Bias = -0.111; RPD = 3.42
Initial starchy coating	Windham et al. (1997)	Reflectance	424-1800	MSC and second derivative	PLS	See in Figure 2.7

Table 2.9 (Continued)

Sensory attribute	Author (Year)	Acquisition mode	Spectral range (nm)	Pre-processing method	Models created method	Prediction results
<b>Texture attribute</b>						
Initial starchy coating	Champagne et al. (2001)	Reflectance	700-1078; 1118-2018	MSC and second derivative	PLS	$R^2_{\text{cal}} = 0.88$ ; $R^2_{\text{val}} = 0.76$ ; RMSEC = 0.13; RMSEP = 0.20 DI = 2.0
Roughness	Windham et al. (1997)	Reflectance	424-1800	MSC and second derivative	PLS	See in Figure 2.7
Roughness of mass	Meullenet et al. (2002)	Reflectance	400-2500	Frist derivative	PLS	$R^2_{\text{cal}} = 0.49$ ; $R^2_{\text{val}} = 0.35$ ; RMSEC = 0.28; RMSEP = 0.28 RPD = 1.23
Self-adhesiveness	Windham et al. (1997)	Reflectance	424-1800	MSC and second derivative	PLS	RAP =
Slickness	Champagne et al. (2001)	Reflectance	700-1078; 1118-2018	MSC and second derivative	PLS	$R^2_{\text{cal}} = 0.81$ ; $R^2_{\text{val}} = 0.53$ ; RMSEC = 0.25; RMSEP = 0.38 DI = 1.6
Starchy mouth coating	Windham et al. (1997)	Reflectance	424-1800	MSC and second derivative	PLS	See in Figure 2.7

Table 2.9 (Continued)

Sensory attribute	Author (Year)	Acquisition mode	Spectral range (nm)	Pre-processing method	Models created method	Prediction results
<b>Texture attribute</b>						
Stickiness	Qingyun et al. (2007)	Reflectance	540 and 970		MLR	$r_{\text{cal}} = 0.88$ ; $\text{SEC} = 0.50$ ;
		Transmission	540 and 640			$r_{\text{val}} = 0.76$ and $\text{SEP} = 0.67$
	Srisawas et al. (2007)	Reflectance	1100-2500	Frist derivative	PLS	$R^2_{\text{cal}} = 0.935$ ; $R^2_{\text{val}} = 0.914$ ;
	Champagne et al. (2001)	Reflectance	700-1078; 1118-2018	MSC and second derivative	PLS	$\text{SECV} = 0.30$ ; $\text{SEP} = 0.32$ ;
						$\text{Bias} = -0.020$ ; $\text{RPD} = 3.38$
						$R^2_{\text{cal}} = 0.71$ ; $R^2_{\text{val}} = 0.58$ ;
						$\text{RMSEC} = 0.28$ ; $\text{RMSEP} = 0.33$
						$\text{DI} = 1.8$
Stickiness to lips	Windham et al. (1997)	Reflectance	424-1800	MSC and second derivative	PLS	$\text{RAP} = 0.56$
Surface slickness	Windham et al. (1997)	Reflectance	424-1800	MSC and second derivative	PLS	See in Figure 2.7
Toothpack	Meullenet et al. (2002)	Reflectance	400-2500	Second derivative	PLS	$R^2_{\text{cal}} = 0.85$ ; $R^2_{\text{val}} = 0.77$ ;
						$\text{RMSEC} = 0.18$ ; $\text{RMSEP} = 0.18$
						$\text{RPD} = 2.19$

Table 2.9 (Continued)

Sensory attribute	Author (Year)	Acquisition mode	Spectral range (nm)	Pre-processing method	Models created method	Prediction results
<b>Texture attribute</b>						
Toothpull	Meullenet et al. (2002)	Reflectance	400-2500	Second derivative	PLS	$R^2_{\text{cal}} = 0.76$ ; $R^2_{\text{val}} = 0.71$ ; RMSEC = 0.28; RMSEP = 0.31 RPD = 1.88
Uniformity of bite	Windham et al. (1997)	Reflectance	424-1800	MSC and second derivative	PLS	See in Figure 2.7
<b>Overall liking</b>						
Eating quality	Qingyun et al. (2007)	Reflectance	540 and 970		MLR	$r_{\text{cal}} = 0.81$ ; SEC = 0.73; $r_{\text{val}} = 0.84$ and SEP = 0.65
		Transmission	540 and 640			

In 1997, Windham et al. reported the potential of NIR spectroscopy for determination of 11 texture attributes of cooked rice. The PLSR was used to reveal the relationships between the 11 textures attribute of cooked rice and NIR spectral of milled rice. The wavelength region was selected on 424-1800 nm for developing PLS Models. The relative ability of prediction (RAP) was used to compare the predictive ability of calibration models. This is a background compensation number where a value of 1.0 represents the best possible prediction (Windham et al., 1997; Martens and Martens, 1986). The RAP value is calculated as:

$$\text{RAP} = \frac{(S_{\text{tot}}^2 - \text{RMSEP}^2)}{(S_{\text{tot}}^2 - S_{\text{ref}}^2)} \quad (2.17)$$

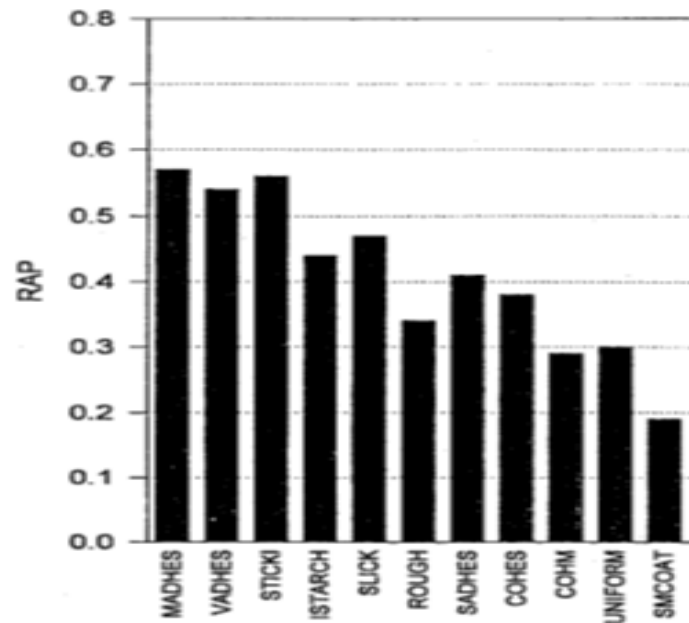
Where:

- $S_{\text{tot}}$  = the standard deviation of a sensory attribute.
- $S_{\text{ref}}$  = the standard error of the reference method.
- RMSEP = the root mean square error of prediction.

The maximum values for RAP of the 11 sensory attributes, when using NIR spectroscopy for the prediction, are shown in the Figure 2.8.

Champagne et al. (2001) examined the ability of NIR spectroscopy to predict texture attribute of diverse rice cultivars. The relationships between each of the sensory texture attributes and NIR spectral data of milled rice were examined by PLSR. The PLS models were generated using spectral in 700-1078 and 1118-2018 nm range. Thirteen sensory attributes were measured in this research but only five of the 13 attributes were successfully predicted by NIR calibration models ( $R_{\text{cal}}^2 = 0.71-0.96$ ;  $R_{\text{val}}^2 = 0.53-0.83$ ; RMSEC = 0.12-0.28; RMSEP = 0.20-0.38). In addition, discrimination index (DI) was calculated for evaluation ability of NIR spectroscopy models which formula of DI showed in equation 2.18.

$$\text{DI} = \frac{\text{standard deviation of texture attribute}}{\text{RMSEP}} \quad (2.18)$$



**Figure 2.8** Relative ability of prediction (RAP) of NIR spectroscopy instrument for 11 sensory texture attributes (MADHES = manual adhesiveness; VADHES = visual adhesiveness; STICKI = stickiness to lips; ISTARCH = initial starchy coating; SLICK = surface slickness; ROUGH = roughness; SADHES = self-adhesiveness; COHES = cohesiveness; COHM = cohesiveness of mass; UNIFORM = uniformity of bite; SMCOAT = starchy mouth coating).

**Source** Windham et al. (1997)

Meullenet et al. (2002) studied on NIR spectroscopy for predicting cooked rice texture as affected by postharvest history. NIR reflectance spectra of milled rice were recorded on 400-2500 nm. Sensory texture attributes models were developed using PLSR technique. Seven sensory texture attributes were evaluated by NIR spectroscopy models. Five of seven sensory texture attribute were successfully predicted that showed  $R^2_{val}$  between 0.67 and 0.83.

Multiple linear regression (MLR) was used to determine the relationships between sensory texture attribute and NIR spectral data (Qingyun et al., 2007). Texture attribute of cooked rice included hardness and stickiness. MLR models were developed from three rays of NIR spectral (i.e. 540, 640, and 970 nm.) in reflectance and transmission mode. Equation 2.19 and 2.20 are MLR models for prediction hardness and stickiness of cooked rice. The prediction results of hardness and stickiness showed  $r_{val} = 0.56$  and  $0.76$ , respectively.

$$\text{Hardness} = -3.204 - 3.207R_1 + 3.822R_2 - 2.070T_1 + 2.300T_2 \quad (2.19)$$

$$\text{Stickiness} = 0.516 + 1.467R_1 - 2.215R_2 + 1.247T_1 - 0.681T_2 \quad (2.20)$$

Srisawas et al. (2007) investigated the relationship between sensory texture attributes of cooked rice and NIR spectroscopy. NIR spectral of cooked rice were obtained in reflectance mode over the wavelength range of 1100-2500 nm. The NIR spectra were used to develop predictive models for each sensory texture attributes of cooked rice using PLSR. The prediction results presented good prediction on hardness ( $R^2_{\text{cal}} = 0.950$ ;  $R^2_{\text{val}} = 0.915$ ; SECV = 0.27; SEP = 0.31; Bias = -0.111; RPD = 3.42) and stickiness ( $R^2_{\text{cal}} = 0.935$ ;  $R^2_{\text{val}} = 0.914$ ; SECV = 0.30; SEP = 0.32; Bias = -0.020; RPD = 3.38).

#### 2.4.5.5 Overall liking

Overall liking is summary attribute of sensory evaluation. Determination of eating quality of cooked rice using NIR spectroscopy was studied by Qingyun et al. (2007). They used NIR spectral at wavelength length of 540, 640 and 970 nm in reflectance and transmission mode for creating MLR model. The equation 2.21 is the MLR model for prediction of eating quality of cooked rice. The prediction results of taste showed  $r_{\text{val}} = 0.84$  and SEP = 0.65.

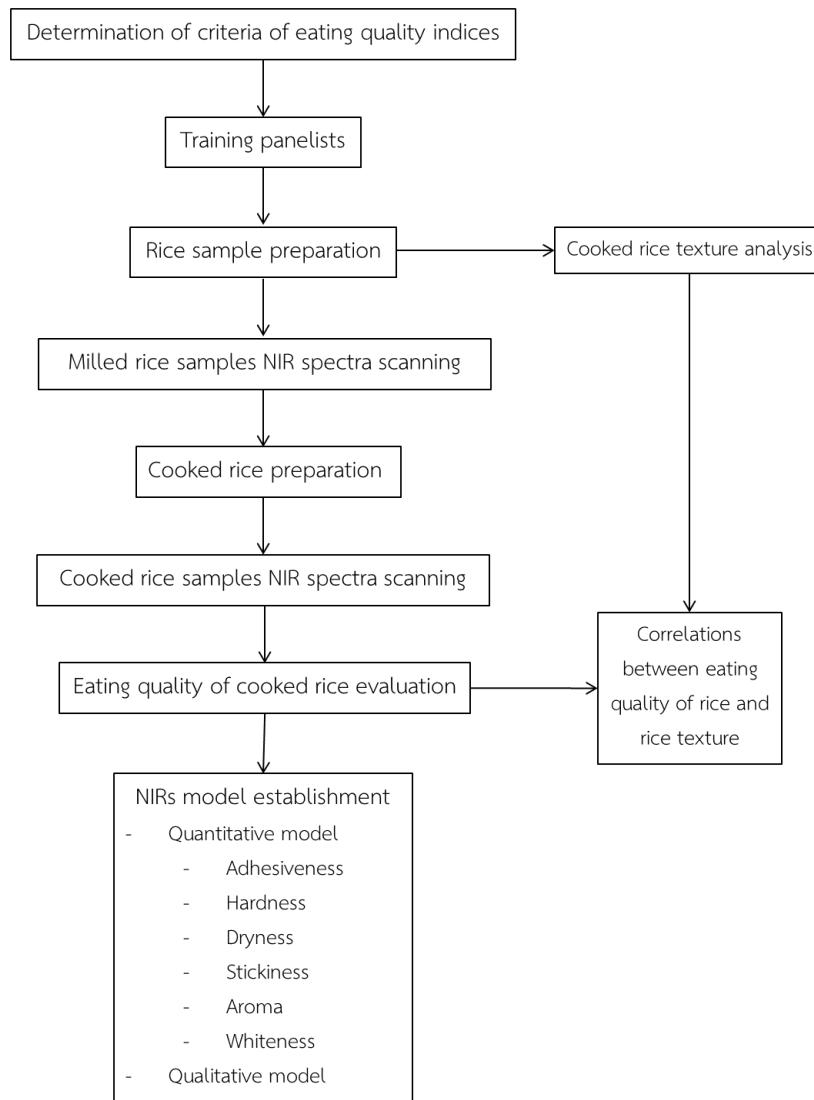
$$\text{Eating Quality} = -2.199 + 2.931R_1 - 2.767R_2 - 0.539T_1 + 1.417T_2 \quad (2.21)$$

# Chapter 3

## METHODOLOGY

### 3.1 PROCESS OF EXPERIMENT

Processes of experiment consisted of determination of criteria of sensory quality indices, training panelists, rice sample preparation, milled rice samples NIR spectra scanning, cooked rice preparation, cooked rice NIR spectra scanning, sensory quality evaluation, and NIRs model Establishment. Flow Diagram of experiment is shown in Figure 3.1.



**Figure 3.1** Flow Diagram of experiment

### 3.2 DETERMINATION OF CRITERIA OF SENSORY QUALITY INDICES

Samples of five types of milled rice were delivered to the Near Infrared Spectroscopy Research Center for Agricultural Product and Food at King Mongkut's Institute of Technology Ladkrabang, Bangkok, Thailand. These five types (i.e. Jasmine rice 2 samples, white rice 1 sample and parboiled rice 2 samples) are the most representative of the range of sensory qualities as recommended by the quality control section of the plant because they cover the products quality range routinely processed by the plant. In this research, the sensory quality indices of rice encompassed adhesiveness, hardness, stickiness, dryness, whiteness, and aroma, all of which are used by the rice quality improvement plant for quality evaluation.

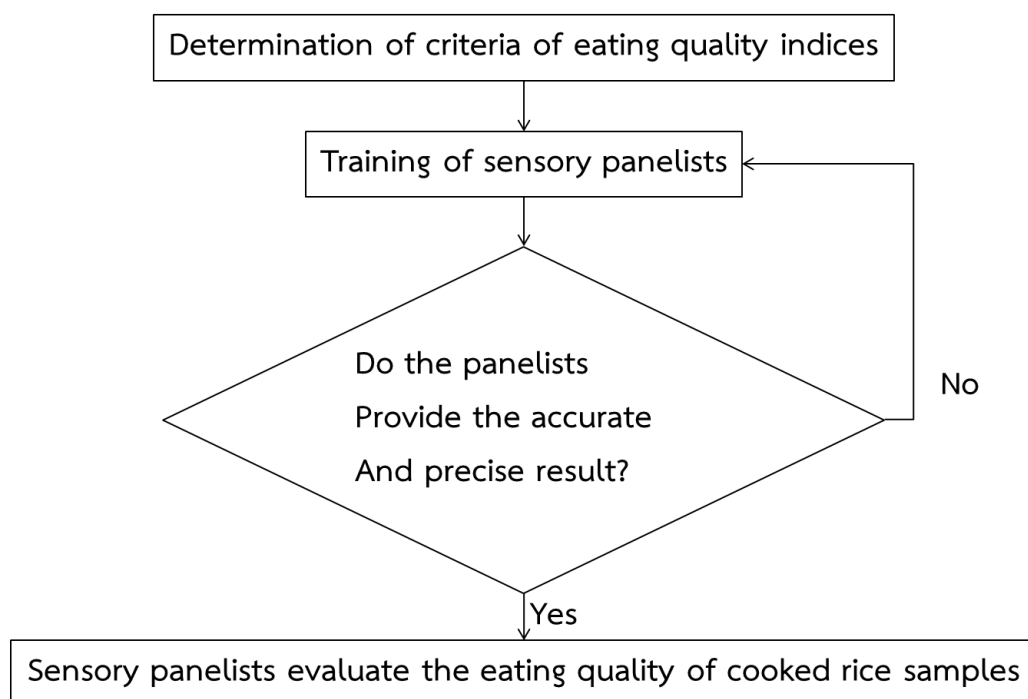
The rice samples were cooked according to the water:rice ratios suggested by the manufacture. A team of 10 panelists (5 males and 5 females) were each served with the five types of cooked rice. The panelists conferred amongst each other before collectively deciding on the score allocated to each of the sensory quality indices. Scoring was on a scale of 1–5 (whole integers only), where 1 denoted maximum level and 5 minimum, except in the case of whiteness and aroma where 1 denoted maximum and 5 minimum.



**Figure 3.2** The panelists would confer for deciding on the score of sensory quality indices.

### 3.3 PANELIST TRAINING

All panelists were given multiple training sessions to acquaint them with the scoring criteria. Each panelist was served with one scoop each of all five cooked rice types, with aromatic assessment performed first. They were trained to assess adhesiveness by shaking small, covered plastic containers containing cooked rice and/or gently scraping the surface of the cooked samples with a small plastic spoon. In regard to the assessment of hardness and stickiness, the panelists were asked to chew the samples and then to explain their perceptions in simple words (e.g., for hardness, extremely hard, hard; for stickiness, extremely sticky, sticky). The panelists were trained to determine dryness and whiteness by visual observation. The overall sensory quality criteria were then used in the subsequent scoring at the experimental stage.



**Figure 3.3** Flow diagram of sensory method of cooked rice.

Two replicates of each type of cooked rice were used in each sensory evaluation training session, and these sessions continued until all participating panelists had become skilled at quality assessment. It should be noted that before

each subsequent assessment, the panelists were served with drinking water to eliminate the aftertaste and rice residues from the previous assessment; and the 5 cooked rice samples in the training sessions were of different types to those (i.e., 250 samples) used in the experimental stage. A flow diagram illustrating the sensory method used for evaluation of cooked rice is shown in Figure 3.3.



**Figure 3.4** Training panelists.

### **3.4 RICE SAMPLE PREPARATION**

All samples ( $n = 250$ ) of milled rice were received from the rice quality improvement plant (C.P. Rice Co., Ltd., Thailand). The rice samples were randomly garnered from three locations within the plant premises (the raw material receiving station, behind the color sorter machine, and under the storage bin). These three locations were selected for sample collection because they are the sites at which rice samples are typically collected for internal quality inspection. The samples of milled rice tested were parboiled (processed from white rice;  $n = 51$ ), rice (from

varieties other than Jasmine; n = 23), new Jasmine (Khao Dawk Mali 105 variety; n = 12; harvested in 2012 and sold to the plant where it was evaluated that year), and aged Jasmine (n = 164; harvested either in 2006 or 2007–2011; “aged” means the rice was kept for at least 1 year post-harvest before processing and evaluation). Types of milled rice are listed in table 3.1. The weight of each milled rice sample was approximately 200 g, and samples were retained in a plastic zipper bag until experimentation.

**Table 3.1** Number of milled rice sample.

Type of milled rice	Numbers of sample
parboiled rice	51
white rice	23
new Jasmine rice (harvested in 2012)	12
aged Jasmine rice (harvested in 2006 or during 2007-2011)	164

### 3.5 NEAR INFRARED SCANNING OF MILLED RICE

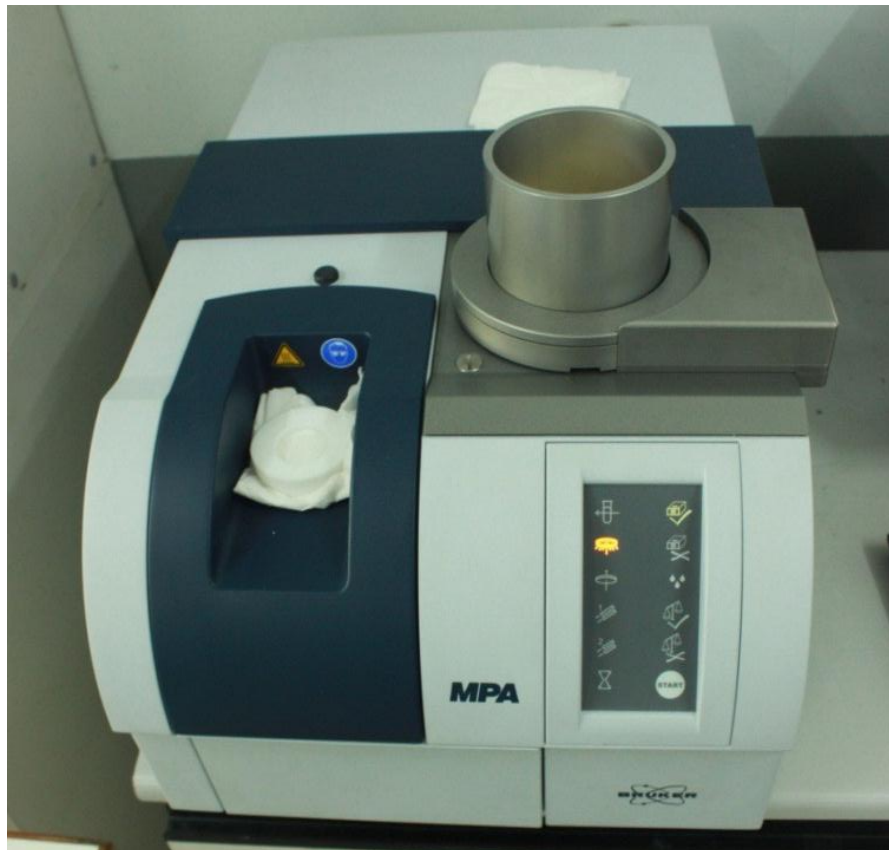
#### 3.5.1 Milled rice sample scanning by FT-NIR spectrometer

Each milled rice sample was poured from the zipper bag into the quartz-sampling cup of a rotary diffuse reflectance holder. NIR spectrum was measured with FT-NIR spectrometer (Bruker Ltd., Germany) in reflection mode on 12,500-4,000  $\text{cm}^{-1}$  (800-2,500 nm). Each rice sample was scanned 64 times at resolution of 8  $\text{cm}^{-1}$ . The scan results were averaged and recorded in absorption mode ( $\log 1/R$ ) for each sample. Before milled rice spectrum recording, reference spectrum of gold was scanned. The quartz-sampling cup was cleaned with a vacuum cleaner prior to subsequent use.

#### 3.5.2 Milled rice sample scanning by short wavelength diode array

Milled rice samples were transferred from zipper bags to glass beakers (250 ml). The NIR spectrum was measured with a portable short wavelength diode array

unit (FANTEC, Japan; reflection mode on 600–1,100 nm with resolution of 2 nm and integration time of 10 ms; Figure 2). Scan results were recorded in absorption mode ( $\log 1/R$ ) for all samples. For every scan, the NIR GUN and sample were covered with black cloth to minimize interference by light.



**Figure 3.5** Collection of spectrum on milled rice by the FT-NIR Spectrometer (MPA, Bruker, Germany).

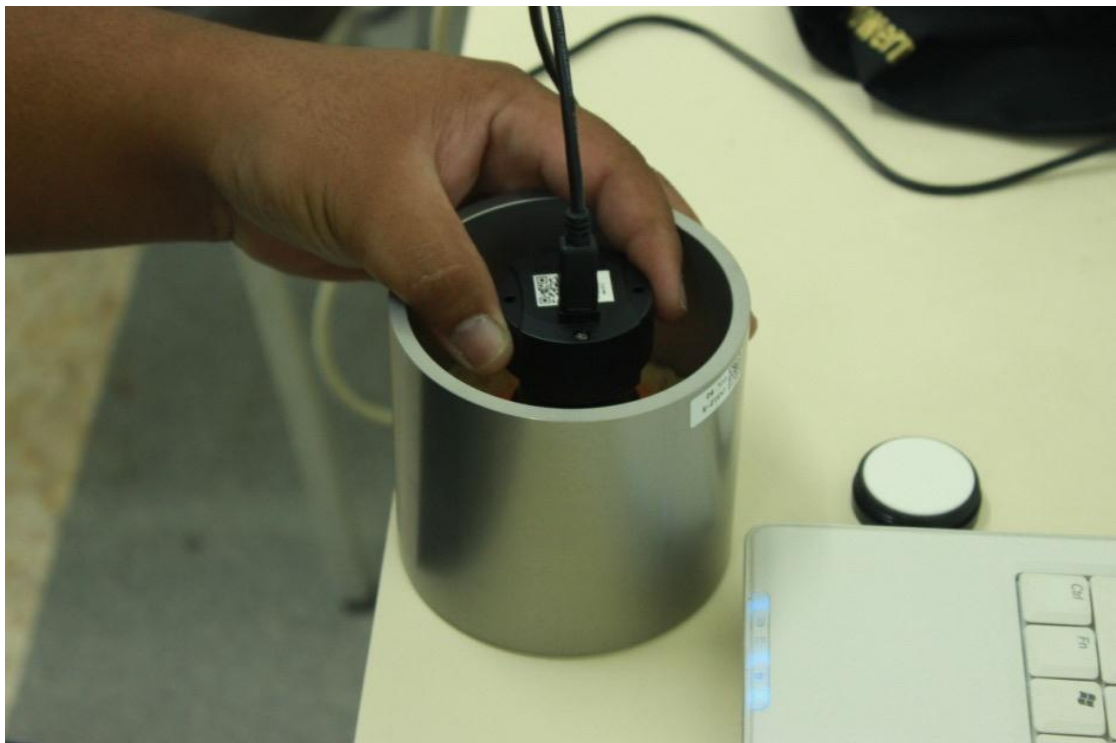
### 3.5.3 Milled rice sample scanning by long wavelength diode array

Each milled rice sample was poured from the zipper bag into the quartz-sampling cup. NIR spectrum was measured with long wavelength diode array (JDSU Ltd., USA) in Interactance mode on 1,150-2,150 nm. Each rice sample was scanned for 5000 times with integration time of 100  $\mu$ s at the resolution of 7 nm. Reference spectrum of spectralon was recorded on InGaAs array before scanning of milled rice. Each sample was scanned twice and average. Spectra were recorded in absorption

mode ( $\log 1/R$ ) for each sample. The quartz-sampling cup was cleaned with a vacuum cleaner prior to subsequent use.



**Figure 3.6** Collection of spectrum on milled rice by the short wavelength diode array (Fantec, Japan).



**Figure 3.7** Collection of spectrum on milled rice by the long wavelength diode array.

### 3.6 COOKED RICE PEPARATION

Home electronic rice cookers (RC-10 MM; Toshiba, Thailand) were used to cook the 250 rice samples, and cooking was performed according to the water:rice ratios recommended by the plant (i.e., 2.5:1 for parboiled rice, 1.6:1 for white rice, 1:1 for new Jasmine rice harvested in 2012, and 1.2:1 and 1.4:1, respectively for aged Jasmine rice harvested in 2007–2011 and in 2006. After the rice was fully cooked and the rice cooker automatically switched to warm mode, the cooked rice was gently and thoroughly mixed using a plastic ladle before being transferred to small, covered plastic containers for sensory evaluation. The cooked rice samples were then presented to the sensory panelists for evaluation.

**Table 3.2** Water to rice ratios for cooking.

Type of milled rice	Water to rice ratios
Parboiled rice	2.5:1
White rice	1.6:1
New Jasmine rice (harvested in 2012)	1:1
Aged Jasmine rice (harvested in 2007-2011)	1.2:1
Aged Jasmine rice (harvested in 2006)	1.4:1

### 3.7 NEAR INFRARED SCANNING OF COOKED RICE

Before NIR scanning, three sub-samples from one rice cooker was weighted about 12 g by digital weighing balance (Adventurer, Ohaus Instruments (Shanghai) Co., Ltd., China). Each sub-sample was scooped into a Petri dish (53.5 mm diameter and 15 mm height).



**Figure 3.8** Weighting of cooked rice about 12 g on Petri dish by digital weighing machine.

### 3.7.1 Cooked rice sample scanning by FT-NIR spectrometer

The NIR spectra of the sub-sample of cooked rice were measured using an FT-NIR spectrometer (MPA, Bruker Ltd., Germany) in reflection mode between 12,500–4,000  $\text{cm}^{-1}$  (800–2,500 nm) at a resolution of 8  $\text{cm}^{-1}$ . Before cooked rice spectrum recording, reference spectrum was internally scanned on reference material (gold). An averaged spectrum for each sample was obtained from 64 separate scans recorded in absorption mode ( $\log 1/R$ ).

### 3.7.2 Cooked rice sample scanning by short wavelength diode array

The NIR spectra of the sub-sample of cooked rice were measured with a portable short wavelength diode array unit (FANTEC, Japan; reflection mode, 600–1100 nm). The probe of instrument was just touched the sample surface. Scan results were recorded in absorption mode ( $\log 1/R$ ) for all samples. For every scan, the Instrument and sample were covered with black cloth to minimize interference by light.



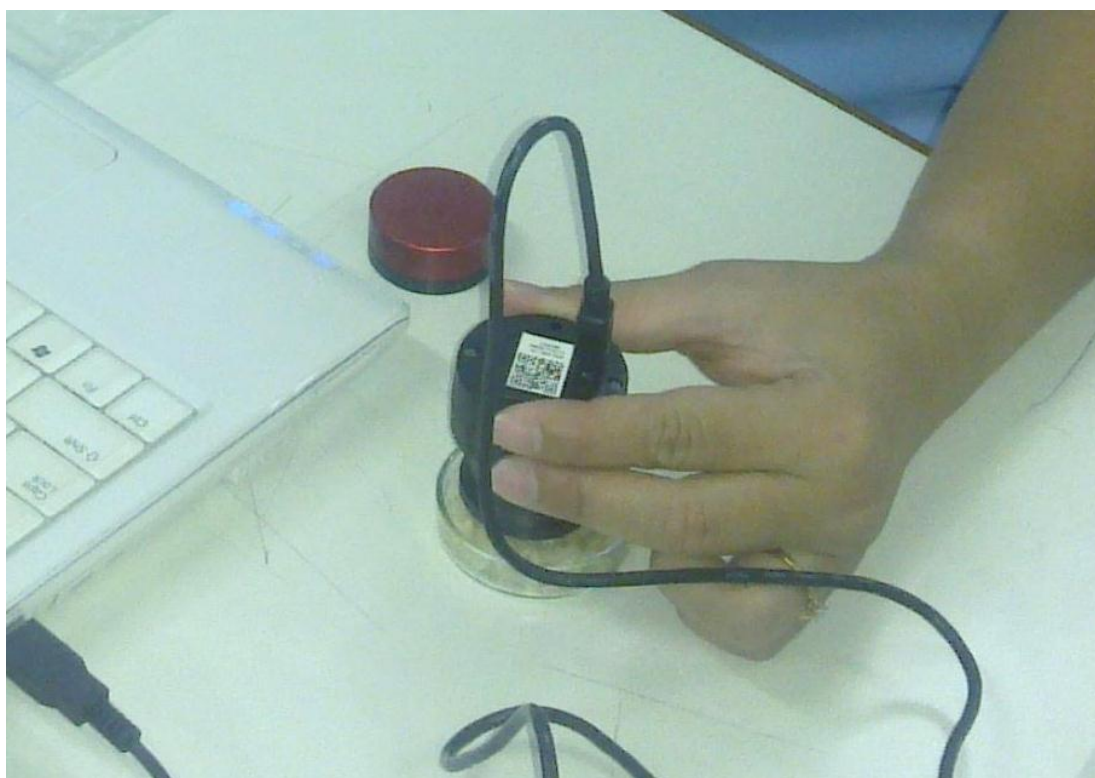
Figure 3.9 Collection of spectrum on cooked rice by FT-NIR Spectrometer (MPA, Bruker, Germany).



Figure 3.10 Collection of spectrum on cooked rice by short wavelength diode array (Fantec, Japan).

### 3.7.3 Cooked rice sample scanning by long wavelength diode array

Spectrum of the sub-sample of cooked rice was measured with MICRO NIR spectrometer (JDSU Ltd., USA) in Interactance mode on 1,150-2,150 nm. The probe of instrument was just touched the sample surface. Each cooked rice part of sample was scanned 5000 times with integration time 100  $\mu$ s at resolution of 7 nm. Reference spectrum was recorded on InGaAs array before cooked rice spectra were scanned. The 2 replicated measurements were scanned and were averaged. Spectra were recorded in absorption mode ( $\log 1/R$ ) for each sample.



**Figure 3.11** Collection of spectrum on milled rice by long wavelength diode array.

## 3.8 SENSORY QUALITY OF COOKED RICE EVALUATION

Scoring by panelists was as described above. The scores of the sensory quality indices allocated by the panelists contained only one decimal point. Evaluation of the 250 cooked rice samples followed the same procedures as those at the training stage, with 8–10 samples being served to each panelist simultaneously. Prior to tasting by the panelists, each sample was assigned a random

3-digit number. Prior to the commencement of sensory evaluation each day, the panelists received retraining on the sensory evaluation criteria.



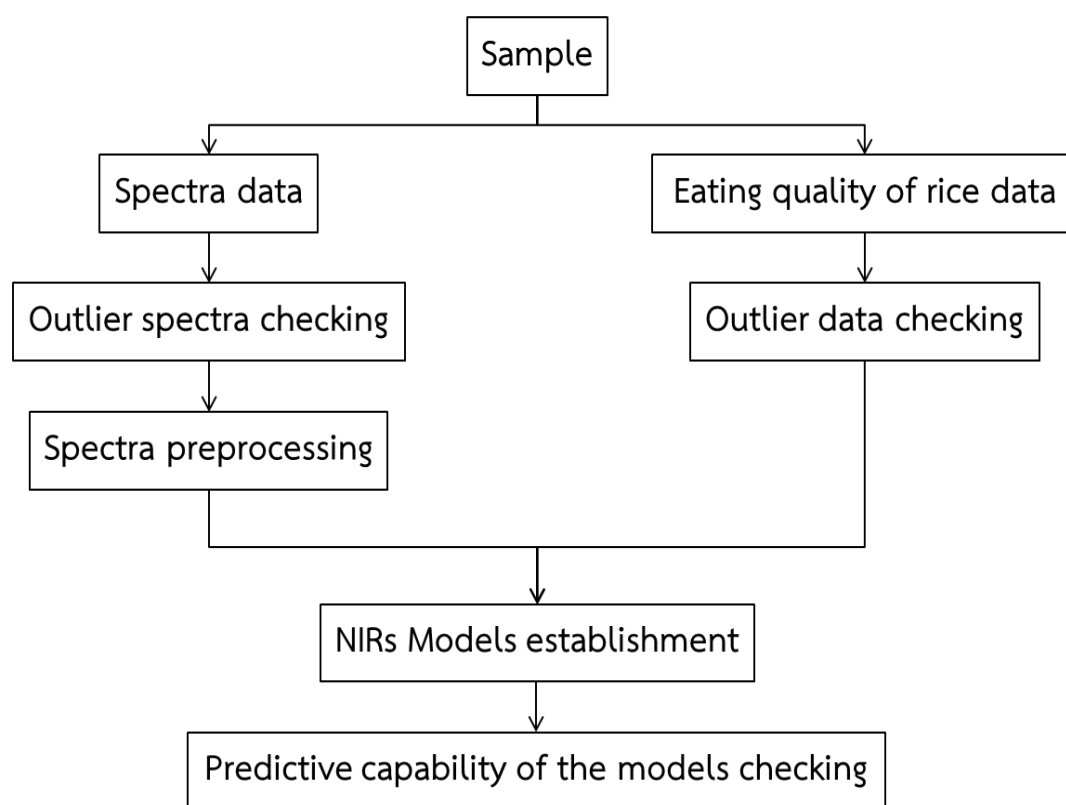
Figure 3.12 The sensory quality indices allocated by the panelists

Panelist 1					Date				
Sensory quality evaluation of cooked rice									
Faculty of engineering, King Mongkut's Institute of Technology Ladkrabang									
Sensory quality evaluation of cooked rice form									
Name _____		Sex _____		Age _____		Year _____			
Recommended	1. Please tast each sample and get the score of sample in code box.								
	2. The score can get only one decimal point								
	3. Drink water between change sample								
Code of sample									
Adhesiveness									
very high=1 high=2 midium=3 low=4 very low=5									
Hardness									
very high=1 high=2 midium=3 low=4 very low=5									
Dryness									
very high=1 high=2 midium=3 low=4 very low=5									
Stickiness									
very high=1 high=2 midium=3 low=4 very low=5									
Aroma									
very high=5 high=4 midium=3 low=2 very low=1									
Whiteness									
very high=5 high=4 midium=3 low=2 very low=1									

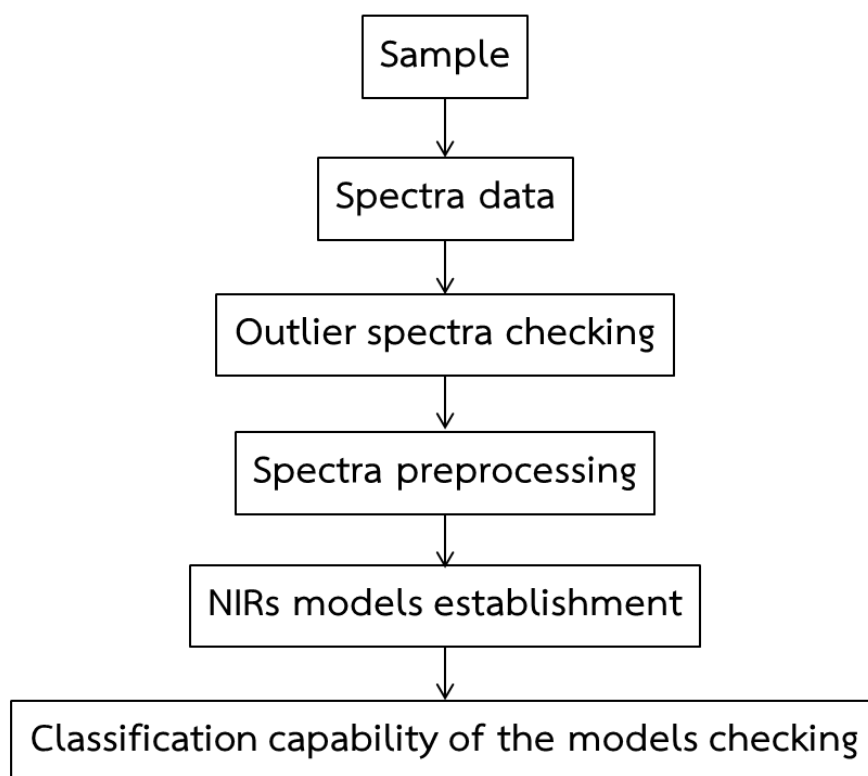
Figure 3.13 Sensory quality evaluation form.

### 3.9 NIR SPECTROSCOPY MODEL ESTABLISHMENT

The NIR spectroscopy (NIRs) models were created for quantitative and qualitative analysis. The NIRs quantitative models were established using partial least squares regression (PLS) for prediction the sensory qualities of cooked rice. The NIRs qualitative models were created by principle component analysis (PCA), soft independent modeling of class analogy (SIMCA) and partial least squares discriminant analysis (PLS-DA) for classification of rice according to level of sensory quality of cooked rice. The flow diagram of NIRs models for quantitative and qualitative analysis were shown in Figure 3.14 and Figure 3.15 respectively.



**Figure 3.14** The flow diagram of NIRs models for quantitative analysis.



**Figure 3.15** The flow diagram of NIRs models for qualitative analysis.

### 3.9.1 NIR spectroscopy quantitative model establishment from rice spectral scanning by FT-NIR spectrometer

The NIRs models for predicting the sensory quality of cooked rice were established using the partial least squares regression (PLS). The models were created from milled and cooked rice. The multivariate analysis software (OPUS, v. 7.0, Bruker, Germany) was used in both spectrum pre-processing and model creation. About 80% of the experiment samples (i.e. 201 samples from 250 were analyzed for milled rice models and 601 samples from 750 for cooked rice models) were used in the calibration group while the remainder (i.e. were 49 samples for milled rice models and 149 samples for cooked rice models) was placed in the validation group. The calibration group was used for the model creation. The model error was then calculated using the full cross-validation method. The same model was then used to predict the samples in the validation group. The optimum wavenumber range and pre-processing method were selected using the default optimization procedures of the software. The predictive capability of the model was assessed using coefficients

of determination of the calibration and validation groups ( $R^2_{cal}$  and  $R^2_{val}$ , respectively), root mean square error of estimation (RMSEE), root mean square error of prediction (RMSEP), bias, and ratio of standard deviation of validation data to RMSEP (RPD).

### **3.9.2 NIR spectroscopy quantitative model establishment from rice spectral scanning by short wavelength diode array and long wavelength diode array**

The NIR spectra were pre-processed by mean normalization, maximum normalization, range normalization, multiplicative scatter correction (MSC), first and second derivatives each with 2 smoothing points on both left and right sides and 5 smoothing points on both sides, baseline offset, detrending (D), and standard normal variate (SNV) and detrending (SNV+D). Savitzky golay smoothing method was used for spectral pre-processing of NIR spectra from long wavelength diode array. For eighty percent of the experimental samples (201 samples for milled rice; 601 samples for cooked rice) were used in the calibration group and the remainder (49 samples for milled rice; 149 samples for cooked rice) in the validation group. The calibration group was used for model creation and the model error was calculated by the full cross-validation method. The same model was then used to predict the sensory qualities of rice in the validation group. The optimum model was selected by coefficients of determination of the calibration and validation groups ( $R^2_{cal}$  and  $R^2_{val}$ ), standard error of calibration (SEC), standard error of prediction (SEP), bias, and ratio of standard deviation of validation data to SEP (RPD). The software for multivariate analysis (Unscrambler, v. 9.8; CAMO, Norway) was used in both spectrum pre-processing and model creation.

### **3.9.3 NIR spectroscopy qualitative model establishment**

The NIRs qualitative models were created by soft independent modeling of class analogy (SIMCA) and partial least squares discriminant analysis (PLS-DA) for classification groups of rice according to level of sensory quality of cooked rice.

Levels of sensory quality of cooked rice were divided to 4 levels according to the range of each sensory quality index score (level 1 score was 1-2; level 2 score was 2-3; level 3 score was 3-4 and level 4 score was 4-5). Level 1, 2, 3 and 4 implied the very high, high, low and very low of the attributes or vice versa. For eighty percent of the experimental samples (201 samples for milled rice; 601 samples for cooked rice) were used in the calibration group and the remainder (49 samples for milled rice; 149 samples for cooked rice) in the validation group. Classification models of sensory quality level of cooked rice are based on spectra measured by FT-NIR spectrometer, FQA-NIR spectrometer and long wavelength diode array. Original spectra from FT-NIR spectrometer and FQA-NIR spectrometer were used to create SIMCA and PLS-DA classification models. The NIR spectra obtained from long wavelength diode array were pretreated with Savitzky Golay smoothing average (polynomial order 2 with number of smoothing point of 5) to reduce noise. Multiplicative scatter correction (MSC) was used to reduce base line shift. Classification results for each discriminant model were determined as the percent of false positive, false negative, and total error (incorrectly classified samples). The percentage of false positive is the error rate of incorrectly self-classification and the percentage of false negative is the error rate of incorrectly other group classification. The software Unscrambler, v. 9.8 (CAMO, Norway) was used for pre-processing, model creation and validation.

#### **3.9.4 Overall precision test**

The overall precision test is important for the precision of reference test, in this case, sensory quality were evaluated by the panelists. The NIR spectroscopy model cannot evaluate properties more accurate than those by the reference test. The ratio of SEP to the SD of the reproducibility test (SEP/SD) was computed to determine the evaluation capability of all the calibration models, where  $SEP/SD < 1.0$  represents an excellent NIRs model, 1.0–2.0 a good model, 2.1–2.4 a fair model, 2.5–3.0 a poor model, and  $> 3.0$  an unreliable model (Tony Pietroutonio, Caltest, USA, personal communication).

For milled rice models, the overall accuracy or reproducibility of reference tests was determined by conducting tests on 9 pairs of blind duplicates alongside

the regular experiment. The duplicates were selected from 18 out of the 250 rice samples, and reproducibility is the standard deviation (SD) of the differences between the values of the blind duplicates. In addition, another 18 rice samples were selected as a new set of duplicates (9 pairs) which were not blind to determine the repeatability of the reference tests, which is the SD of the differences between the values of these duplicates

For cooked rice models, the overall precision of reference tests was determined by conducting tests on 27 pairs of blind duplicates along with the normal experiment. The duplicates were from 54 out of the 750 cooked rice samples. The reproducibility is the standard deviation (SD) of the differences between the values of the blind duplicates. In addition, another twenty seven rice samples were selected as a new set of duplicates (27 pairs) to determine the repeatability of the reference tests, which is the standard deviation of the differences between the values of these duplicates.

### **3.10 COOKED RICE TEXTURE ANALYSIS**

#### **3.10.1 Cooked rice preparation**

As reported by Kladsuk and Sirisomboon (2012), the rice cooking method by cooking rice in beakers in a pot of rice cooker was the better method than normal cooking by directly cooking in the pot in terms of less variation (lower coefficient of variation) in texture property of cooked rice. Each rice type was cooked at different water to rice ratio recommended by the factory: 2.5:1; 1.6:1; 1:1 (harvested in 2012); and 1.2:1 (harvested in 2007-2011) and 1.4:1 (harvested in 2006) for parboiled rice; white rice; new Jasmine rice and aged Jasmine rice, respectively. Weighed 25 g milled rice and water ratio by electronic balance (ARC120, Adventurer, OHAUS) and immediately put in a 100 ml beakers (spread evenly). Put 5 beakers of rice sample in a pot of rice cooker (RC-10 MM (WT) A, Toshiba) and poured 400 g water in the pot. Inserted a thermocouple pair (FLUKE 52-2) into the rice cooker to measure the temperature while cooking rice. When the temperature reached about 100 °C or water vapor came up, waited for another 20 min and then press the Cancel button

and then left it in the rice cooker for another 10 min. Then put the beakers upside down on the screen and covered with plastic lid and left it for one hour. Then knocked out the cooked rice from the beaker and put only the middle portion into plastic cup for 3 g. There were 3 cups per sample.



**Figure 3.16** Cooking rice in beakers in a pot of rice cooker.

### 3.10.2 Texture analysis

As reported by Sonsanguan, N. et al. (2012), the back extrusion test was the best objective method compared to other methods such as texture profile analysis and Ottawa test, to measure the texture of cooked rice to be used in research and industry. The cooked rice samples were subjected to the texture analyzer (TA HD Plus, Stable Micro System, London, UK) using Back Extrusion test platform as shown in Figure 3.17 The Back Extrusion test used 3 g cooked rice, compression speed of 1mm/s and moving distance of 99 mm of the 100 mm starting point from the surface of platform. The texture parameters were determined from force-deformation curve. The determined texture parameters were stickiness (maximum negative force, N) hardness (maximum force, N), toughness (area under curve above zero force, Nmm)

and adhesiveness (area under curve below zero force, Nmm). There were 3 replicates per sample.



**Figure 3.17** Back Extrusion test platform.

### **3.11 CORRELATION BETWEEN SENSORY TEXTURE OF COOKED RICE AND INSTRUMENTAL TEXTURE OF COOKED RICE**

The relationships between sensory texture of cooked rice and instrumental texture of cooked rice, what were hardness, stickiness and adhesiveness, were computed using the linear regression function of the Microsoft Excel application program. The correlation coefficient ( $r$ ) was used in determination of the correlations with the confident level of 95%.

## Chapter 4

# RESULTS AND DISCUSSION

### 4.1 SENSORY QUALITY OF RICE

Table 4.1 presents the average value of sensory quality of different types of rice. The sensory qualities of rice were significantly different among types of rice including all sensory texture properties of cooked rice i.e. adhesiveness, hardness and stickiness. The whiteness of new Jasmine rice was the highest followed by aged jasmine rice, white rice and parboiled rice, respectively. Aroma and whiteness of parboiled rice was the lowest because natural parboiled rice color is amber. The amber color on parboiled rice was affected from the diffusion of husk color into endosperm (Lamberts et al., 2006a; Lamberts et al., 2006b) and chemical reaction (Lamberts et al., 2008).

### 4.2 TEXTURE ATTRIBUTE OF COOKED RICE

Table 4.2 shows the texture attributes of different cooked rice, including parboiled rice, new Jasmine rice, aged Jasmine rice and white rice. The stickiness is the maximum negative force (N) obtained from the force-deformation curve which occurred when the probe was moving back but the sample was stuck to it. The higher the stickiness, the more difficult (more force needed) to separate the rice from the probe. The stickiness of new Jasmine rice was higher than aged Jasmine rice, white rice and parboiled rice, respectively. The Jasmine rice has low amylose content and other categories of rice mostly have high amylose content (Bureau of Rice Research and Development, 2015). As reported by Yu et al., (2009) the rice cultivars with higher amylose content showed the lower stickiness. Parboiled rice samples were made from varieties of white rice which were not Jasmine rice. Because the rice stickiness decreased with storage time (Yu et al., 2009). The stickiness of new Jasmine

rice was higher than that of aged Jasmine rice. The adhesiveness is the area under curve below zero force (Ns) obtained from the force-time curve which occurred when the probe was moving back but the sample was stuck to it. The higher the adhesiveness, the more energy needed for separating the rice from the probe. The adhesiveness of new Jasmine rice was higher than aged Jasmine rice, white rice and parboiled rice, respectively. The hardness is the maximum force (N) of the compression to 99% strain. The higher the hardness, the more difficult to be compressed the rice. The white rice was the hardest while the parboiled rice was the softest. The hardness of white rice was higher than aged Jasmine rice, new Jasmine rice and parboiled rice, respectively. The white rice, mostly had high amylose content and Jasmine rice had low amylose content (Bureau of Rice Research and Development, 2015). Hardness of cooked milled rice positively correlated with amylose content ( $0.706 \leq r \leq 0.866$ ,  $P < 0.01$ ) (Yu et al., 2009). Since the hardness of cooked rice increased as a consequence of storage (Yu et al., 2009), the aged Jasmine rice was harder than new Jasmine rice. In this study, the parboiled rice was the softest because the water to rice ratio recommended by the factory was very high (2.5:1), though in general if the ratio was the same, the parboil rice should be the hardest. The toughness (Ns) is the area under force-deformation curve during the cooked rice compression to 99% strain. The higher the toughness, the more energy needed for deforming the rice. The white rice was the toughest while the parboiled rice was the least tough. Like in case of hardness, the toughness of white rice was higher than aged Jasmine rice, new Jasmine rice and parboiled rice, respectively (Sonsanguan and Sirisomboon, 2013; Kladsuk and Sirisomboon, 2013).

**Table 4.1** Average value of sensory quality of different types of rice

Type of rice	Sensory quality of rice					
	Adhesiveness	Hardness	Dryness	Stickiness	Aroma	Whiteness
Parboiled rice	4.4±0.3 <sup>d</sup>	1.8±0.4 <sup>a</sup>	1.8±0.3 <sup>a</sup>	4.4±0.2 <sup>d</sup>	1.7±0.3 <sup>a</sup>	1.8±0.4 <sup>a</sup>
White rice	3.8±0.2 <sup>c</sup>	2.2±0.3 <sup>b</sup>	2.3±0.3 <sup>b</sup>	3.8±0.3 <sup>c</sup>	3.1±0.3 <sup>b</sup>	3.0±0.2 <sup>b</sup>
Aged Jasmine rice	1.9±0.4 <sup>b</sup>	4.0±0.4 <sup>c</sup>	4.0±0.4 <sup>c</sup>	2.0±0.4 <sup>b</sup>	4.2±0.4 <sup>c</sup>	4.2±0.4 <sup>c</sup>
New Jasmine rice	1.1±0.1 <sup>a</sup>	4.3±0.1 <sup>d</sup>	4.5±0.2 <sup>d</sup>	1.1±0.1 <sup>a</sup>	4.2±0.3 <sup>c</sup>	4.4±0.2 <sup>d</sup>

Values are given as mean ± SD. In the same column, values not sharing a common superscript letter (a–d) differ significantly at  $p < 0.05$  (DMRT).

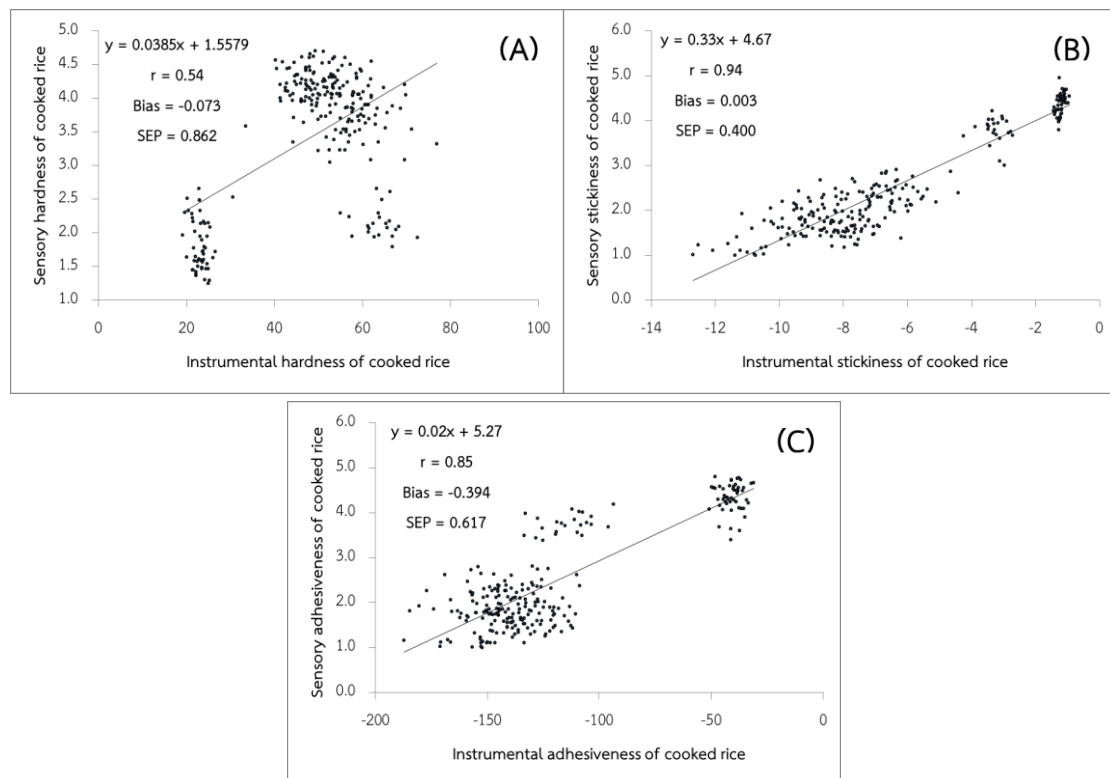
**Table 4.2** Average value of texture attributes of different types of rice

Type of rice	Texture attributes of rice			
	Adhesiveness	Hardness	Toughness	Stickiness
Parboiled rice	-40.73 <sup>d</sup>	23.19 <sup>a</sup>	257.26 <sup>a</sup>	-1.20 <sup>d</sup>
White rice	-116.13 <sup>c</sup>	63.36 <sup>c</sup>	596.08 <sup>c</sup>	-3.46 <sup>c</sup>
Aged Jasmine rice	-139.77 <sup>b</sup>	53.22 <sup>b</sup>	470.05 <sup>b</sup>	-7.87 <sup>b</sup>
New Jasmine rice	-157.79 <sup>a</sup>	52.30 <sup>b</sup>	451.42 <sup>b</sup>	-11.37 <sup>a</sup>

Values are given as mean ± SD. In the same column, values not sharing a common superscript letter (a–d) differ significantly at  $p < 0.05$  (DMRT).

### 4.3 CORRELATION BETWEEN SENSORY TEXTURE ATTRIBUTES AND INSTRUMENTAL TEXTURE ATTRIBUTES OF COOKED RICE

The linear regressions of the correlation between sensory texture of cooked rice and instrumental texture of cooked rice are shown in figure 2.1. The correlation coefficient ( $r$ ) between sensory texture of cooked rice and instrumental texture of cooked rice were higher than 0.54. The high  $r$  values point to the fact that sensory method used was corresponded to the objective method using texture analyser. The linear equations of all texture attributes show a positive slope, indicating positive correlations of the sensory texture of cooked rice and instrumental texture of cooked rice. The results revealed that only stickiness of cooked rice could be substituted in the linear equations to determine sensory texture of cooked rice.



**Figure 4.1** The linear regressions related sensory texture of cooked rice and instrumental texture of cooked rice ((A) hardness, (B) stickiness and (C) adhesiveness).

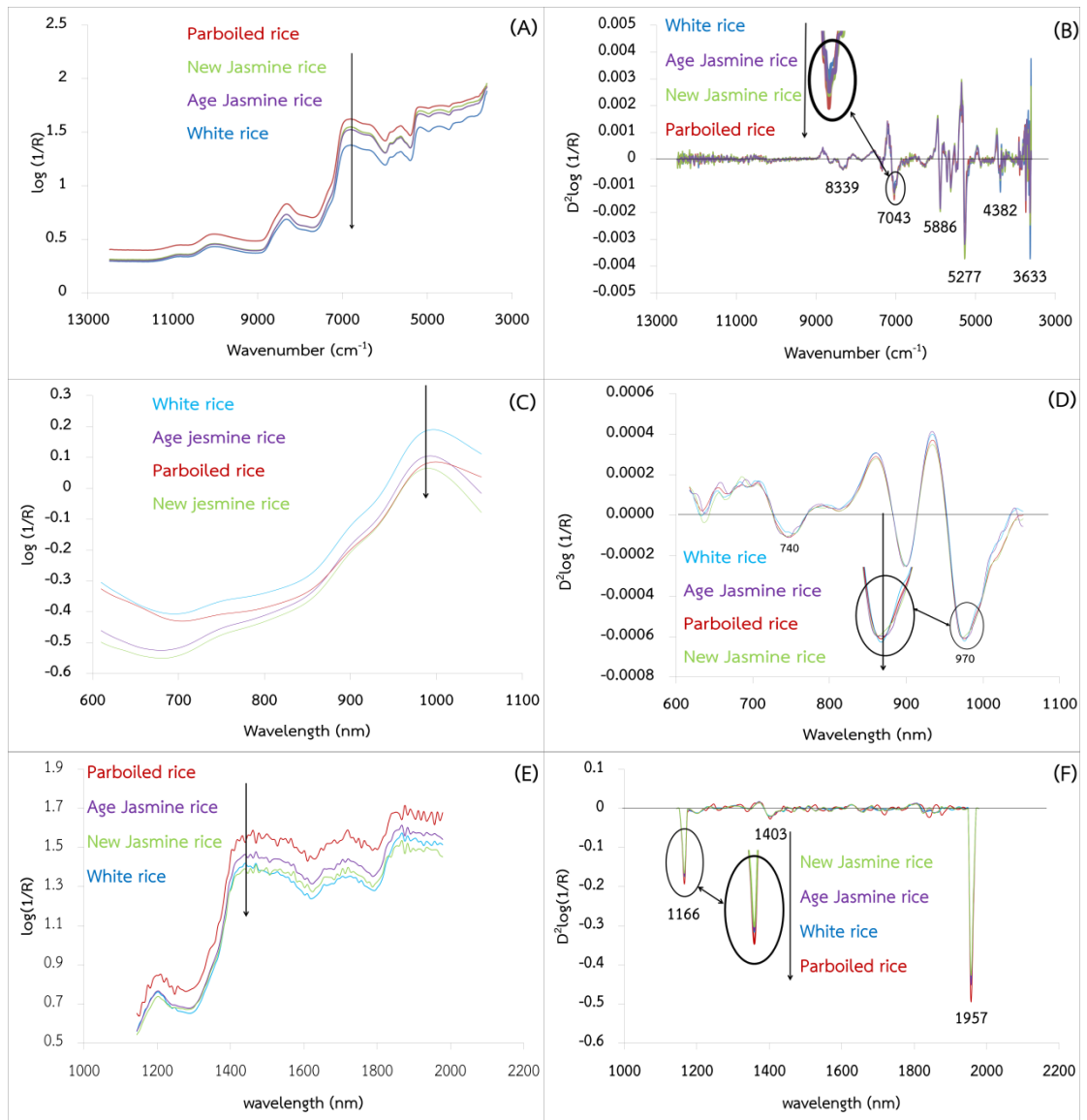
#### 4.4 NIR SPECTRA OF RICE SAMPLES

The spectra of milled rice and cooked rice of different types obtained from FT-NIR spectrometer are shown in Figure 4.2(A) and Figure 4.3(A) respectively. All four types of rice showed a high absorbance peak ( $\log 1/R$ ) of 10370, 8525, 6896 and 5245  $\text{cm}^{-1}$  (964, 1173, 1450 and 1906 nm). However, spectral data may have baseline shift that led to dislocation of absorbance. These problems can be resolved by use of the second derivative method. Second derivative ( $d^2 \log (1/R)$ ) spectra of milled rice and cooked rice are shown in Figure 4.2(B) and Figure 4.3(B), respectively. The spectral data showed prominent peaks at 8339, 7043, 5886, 5277, 4382 and 3633  $\text{cm}^{-1}$  (1199, 1420, 1698, 1895, 2282 and 2752 nm). The peak at 1420 might be the shifted peak of 1450 nm which was the absorbance of O-H stretching second overtone (ArOH) (Osborne & Fearn, 1986). The sharp peak at 2280 nm belonging to starch is also prominent (Rittiron et al., 2005).

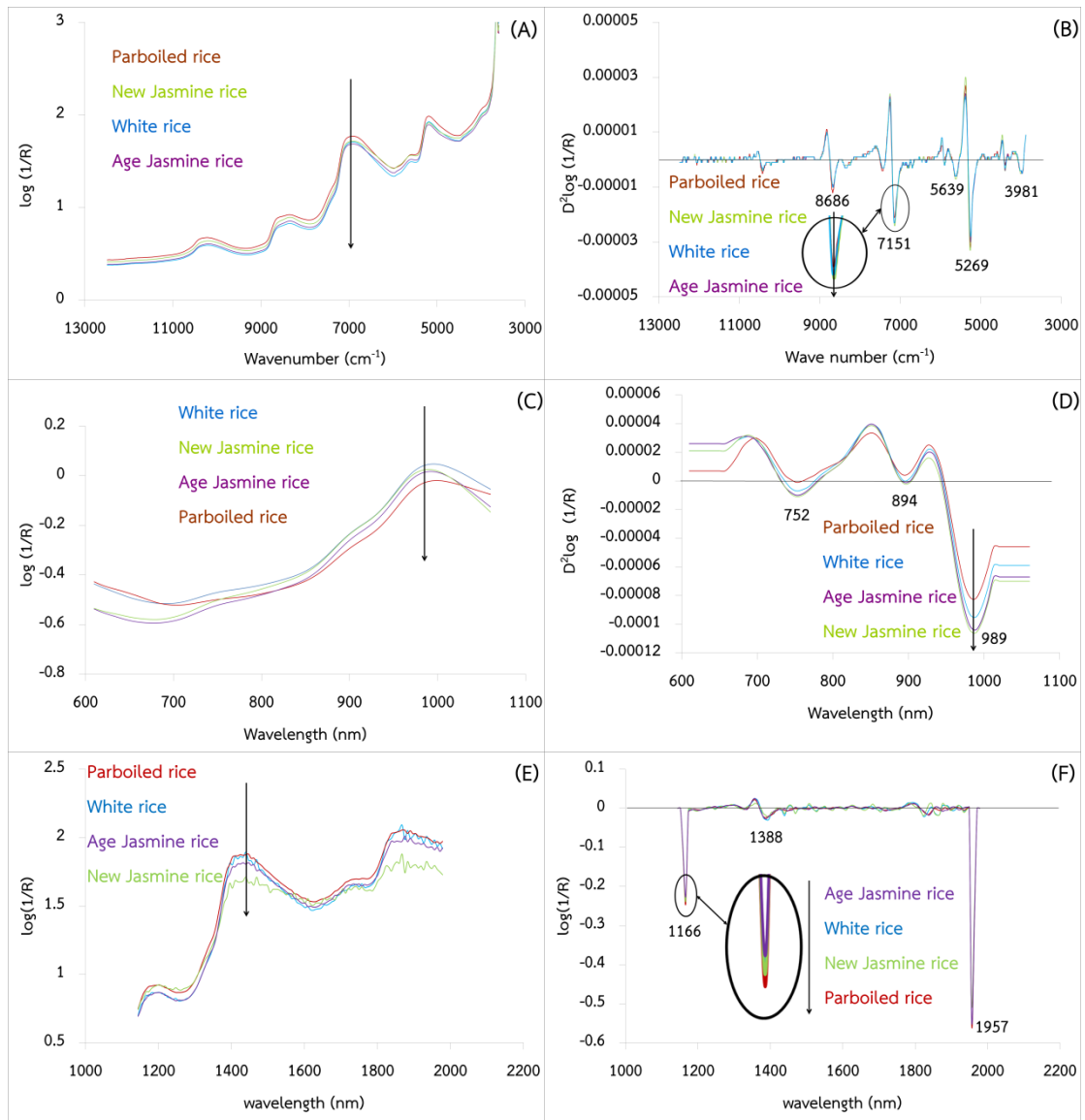
In Figure 4.2(C) and Figure 4.3(C), spectra of all four types of rice from short wavelength diode array showed a high absorbance peak ( $\log 1/R$ ) of 950–1000 nm. The second derivative spectra of milled rice and cooked rice are shown in Figure 4.2(D) and Figure 4.3(D), respectively. The spectral plots showed obvious peaks at 744, 900, and 970 nm. The peak at 900 nm is absorption band of C-H stretching of third overtone and the absorbance peak at 970 nm is absorption band of water (Osborne & Fearn, 1986). In Figure 4.2(D) and Figure 4.3(D), different types of rice affected the spectral level of the absorbance peak, especially at 970 nm, which is the absorbance band of water. The intensity of the peak was highest for new Jasmine rice.

The spectra of milled rice and cooked rice of different types obtained from long wavelength diode array are shown in Figure 4.2(E) and Figure 4.3(E), respectively. The spectra of all four types of rice from MICRONIR gun were interfered by noise and light scattering which the problems were resolved by use of the Savitzky Golay

smoothing and second derivative method (Figure 4.2(F) and Figure 4.3(F)) where the obvious peaks at 1166, 1403 and 1957 nm were shown.



**FIGURE 4.2** Spectra of milled rice. Raw spectra ( $\log 1/R$ ) of different types of milled rice from FT-NIR spectrometer (A); short wavelength diode array (C) and long wavelength diode array (E). Second derivative spectra of different types of milled rice from FT-NIR spectrometer (B); short wavelength diode array (D) and long wavelength diode array after Savitzky Golay smoothing (F).



**FIGURE 4.3** Spectra of cooked rice. Raw spectra ( $\log(1/R)$ ) of different types of cooked rice from FT-NIR spectrometer (A); short wavelength diode array (C) and long wavelength diode array (E). Second derivative spectra of different types of cooked rice from FT-NIR spectrometer (B); short wavelength diode array (D) and long wavelength diode array after Savitzky Golay smoothing (F).

## 4.5 QUANTITATIVE RESULTS OF NIR SPECTROSCOPY

### 4.5.1 Prediction results of sensory quality of cooked rice using milled rice spectra obtained from FT-NIR spectrometer

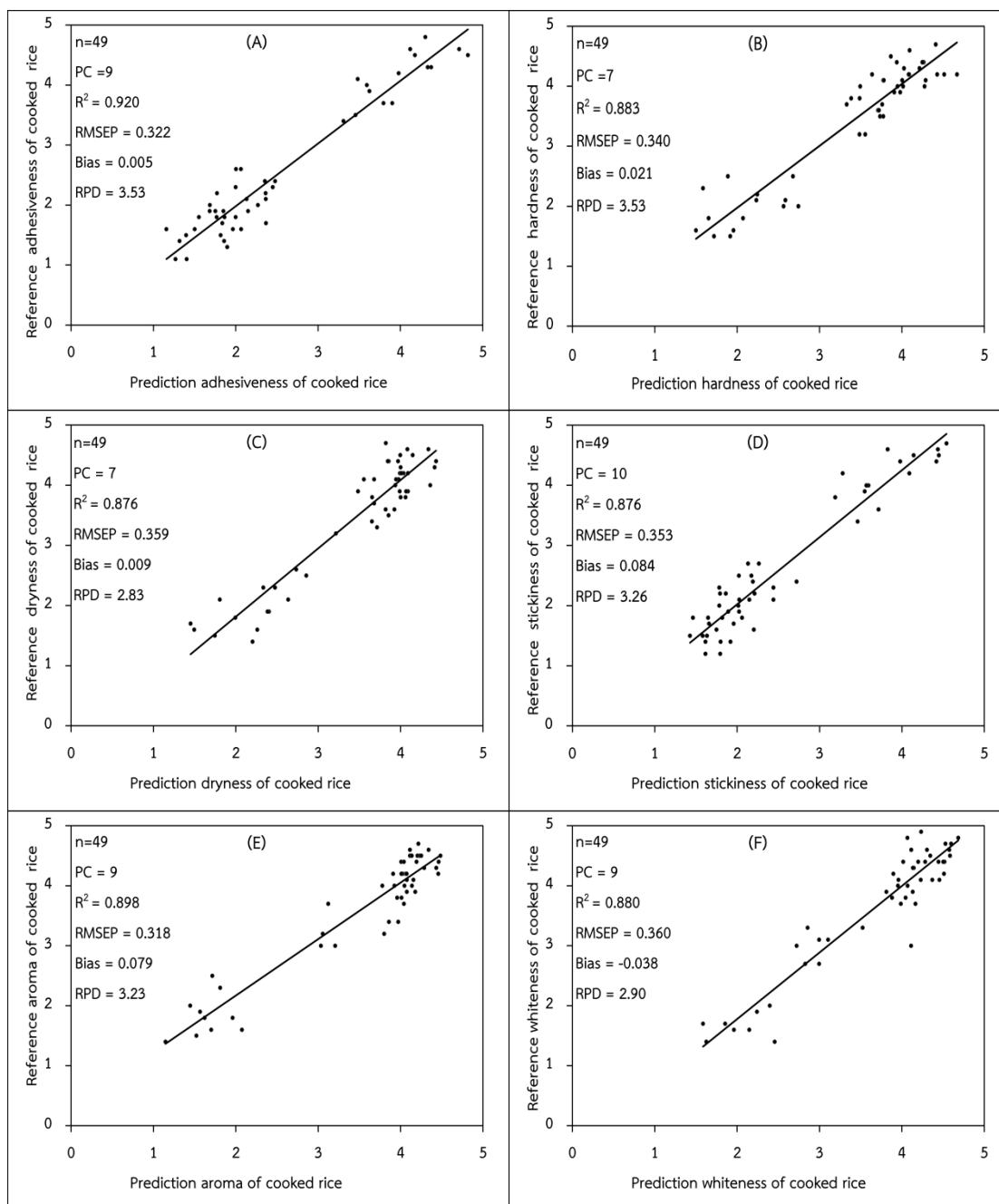
The mean, standard deviation (SD), maximum (Max), and minimum (Min) values of the sensory quality of cooked rice for calibration and validation groups are shown in Table 4.3. The range of stickiness was widest, i.e. 1.0 to 5.0., whereas the hardness range was narrowest, 1.3 to 4.7. The range of adhesiveness was equal to those of aroma and whiteness (1.0 to 4.8), while the dryness range was from 1.2 to 4.7.

The statistics of prediction of the sensory quality of cooked rice by PLS models are shown in Table 4.13. The optimum models for stickiness evaluation were derived from the spectra that were preprocessed by the vector normalization method on wavenumber of 9403-4597  $\text{cm}^{-1}$ . The 1<sup>st</sup> derivative method was the spectra preprocessed method that gave the optimum model for prediction of adhesiveness. The optimum model for prediction of dryness and whiteness were established from spectra that pre-processed by straight line subtraction method. The models for prediction of aroma created by the original spectra of milled rice and hardness were preprocessed with MSC method. The prediction results of all sensory quality indices showed  $R^2_{\text{val}}$  between 0.837 and 0.918. The results on adhesiveness indicted its highest prediction ability of all the sensory quality indices (i.e.  $R^2_{\text{val}} = 0.920$ ; RMSEP = 0.322; Bias = 0.005; RPD = 3.53).  $R^2_{\text{val}}$  for hardness and stickiness, the texture properties of cooked rice, ranged from 0.883 to 0.900. The dryness and whiteness, which were evaluated visually, showed  $R^2_{\text{val}}$  of 0.876 and 0.880, respectively. The aroma of cooked rice exhibited  $R^2_{\text{val}}$  of 0.898. These results are better than those reported in Qingyun et al. (2007) which the  $R^2$  value of all sensory quality less than 0.71. This might be because the multiple linear regression, where the absorption at only some wavelengths were included, was applied for developing the evaluation sensory quality models in their work. In our work, the partial least square regression was applied, where the data of whole spectrum was used. The

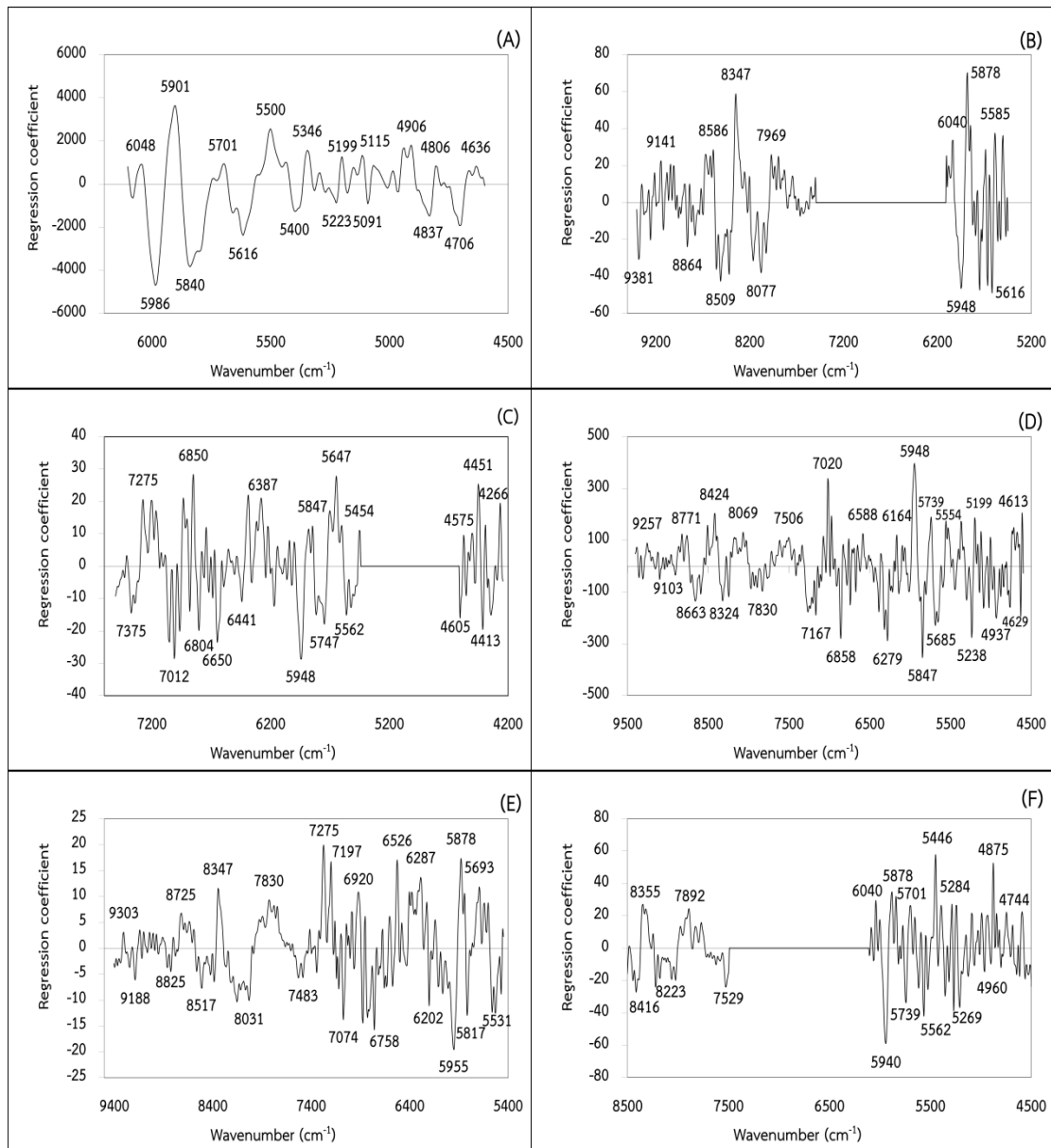
scatter plots (reference data (X) with prediction data (Y)) of the sensory quality are shown in Figure 4.4.

The regression coefficient plots of optimum models for evaluation of the sensory quality of cooked rice are shown in Figure 4.5. The adhesiveness, stickiness and whiteness showed peak regression coefficient at  $5200\text{ cm}^{-1}$ . The peak possesses the frequency characteristics of O-H stretching and O-H deformation combination (O-H molecular of water) (Workman and Weyer, 2008).

The mean and standard deviation (SD) of the differences between the duplicates and ratios of standard error of prediction (SEP) to standard deviation (i.e., SEP/SD) of repeatability and reproducibility tests are shown in Table 4.5. The SEP/SD values of moisture content of milled rice and whiteness of cooked rice were 0.7 and 0.9, respectively. This indicated the robustness of both models. Dryness, stickiness, aroma and satisfaction showed SEP/SD values between 1.0 to 1.4, thereby indicating that the NIR spectroscopy of milled rice could satisfactorily be applied to evaluate moisture content of milled rice, whiteness, dryness, stickiness, aroma, and eater's satisfaction of cooked rice. For adhesiveness and hardness, SEP/SD values were 2.5 and 2.6, respectively.



**Figure 4.4** The scatter plots of reference data with prediction data of sensory quality of cooked rice of prediction sensory quality of rice using NIR spectral of milled rice scanning by FT-NIR spectrometer. (A) Adhesiveness; (B) Hardness; (C) Dryness; (D) Stickiness; (E) Aroma; (F) Whiteness.



**Figure 4.5** Regression coefficient plots of optimum models for evaluation of sensory quality of cooked rice developed by using NIR spectra of milled rice scanned by FT-NIR spectrometer. (A) Adhesiveness; (B) Hardness; (C) Dryness; (D) Stickiness; (E) Aroma; (F) Whiteness.

**Table 4.3** Minimum (Min), maximum (Max), mean, and standard deviation (SD) of sensory quality indices of rice of calibration and validation groups used for model development where the NIR spectra of milled rice scanned by FT-NIR spectrometer

	Calibration					Validation				
	Number of Samples	Min	Max	Mean	SD	Number of Samples	Min	Max	Mean	SD
Adhesiveness	201	1.0	4.8	2.5	1.16	49	1.1	4.8	2.5	1.15
Hardness	201	1.3	4.7	3.4	1.03	49	1.5	4.7	3.4	1.01
Dryness	201	1.2	4.7	3.4	1.04	49	1.4	4.7	3.4	1.03
Stickiness	201	1.0	5.0	2.6	1.14	49	1.2	4.7	2.6	1.13
Aroma	201	1.0	4.8	3.6	1.05	49	1.4	4.7	3.6	1.00
Whiteness	201	1.2	5.0	3.6	1.09	49	1.4	4.9	3.7	1.05

**Table 4.4** Statistics of prediction of sensory quality of cooked rice by PLS models using NIR spectra of milled rice scanned by FT-NIR spectrometer.

Parameter	Pre-treatment	Wavenumber range (cm <sup>-1</sup> )	PC	R <sup>2</sup> <sub>cal</sub>	R <sup>2</sup> <sub>val</sub>	RMSEC	RMSEP	BIAS	RPD
Adhesiveness	1 <sup>st</sup> derivative	6102-4597	9	0.897	0.920	0.379	0.322	0.005	3.53
Hardness	MSC	9403-7498, 6102-5446	7	0.879	0.883	0.364	0.340	0.021	2.93
Dryness	Straight line subtraction	7506-5446, 4605-4242	7	0.910	0.876	0.319	0.359	0.009	2.83
stickiness	Vector normalization	9403-4597	10	0.924	0.900	0.324	0.353	0.084	3.26
Aroma	Raw spectrum	9403-5446	9	0.910	0.898	0.327	0.318	0.079	3.23
Whiteness	Straight line subtraction	9403-7498, 6102-4242	9	0.940	0.880	0.272	0.360	-0.038	2.90

PC - Optimal number of principal components

R<sup>2</sup><sub>cal</sub> - The coefficient of determination of calibration set

R<sup>2</sup><sub>val</sub> - The coefficient of determination of validation set

RMSEE – Root mean square error of calibration

RMSEP - Root mean square error of prediction

RPD - Ratio of standard deviation of validation data to Root mean square error of prediction

**Table 4.5** Means and standard deviations (SD) of the differences between the duplicates and ratios of standard error of prediction (SEP) to standard deviation (i.e., SEP/SD) of repeatability and reproducibility tests of sensory quality of rice.

Quality	Repeatability test		Reproducibility test		
	Mean	SD	Mean	SD	SEP/SD
Adhesiveness	-0.1	0.2	0.0	0.2	2
Hardness	0.0	0.3	-0.1	0.2	2
Dryness	0.1	0.2	-0.1	0.2	1
stickiness	0.0	0.2	0.1	0.3	1
Aroma	-0.2	0.2	0.2	0.2	1
Whiteness	-0.1	0.2	0.0	0.5	1

#### 4.5.2 Prediction results of sensory quality of cooked rice using cooked rice spectra obtained from FT-NIR spectrometer

Mean, SD, Max, and Min values of the sensory qualities of cooked rice for calibration and validation groups are shown in Table 4.6. The range of stickiness values was widest, i.e., 1.0 to 5.0., whereas the hardness range was narrowest, 1.3 to 4.7. The range of adhesiveness was equal to those of aroma, and whiteness (1.0 to 4.8), while the dryness range was between 1.2 and 4.7. The sensory quality has an SD of validation between 1.09 and 1.23. The SD of adhesiveness and stickiness was highest at 1.23.

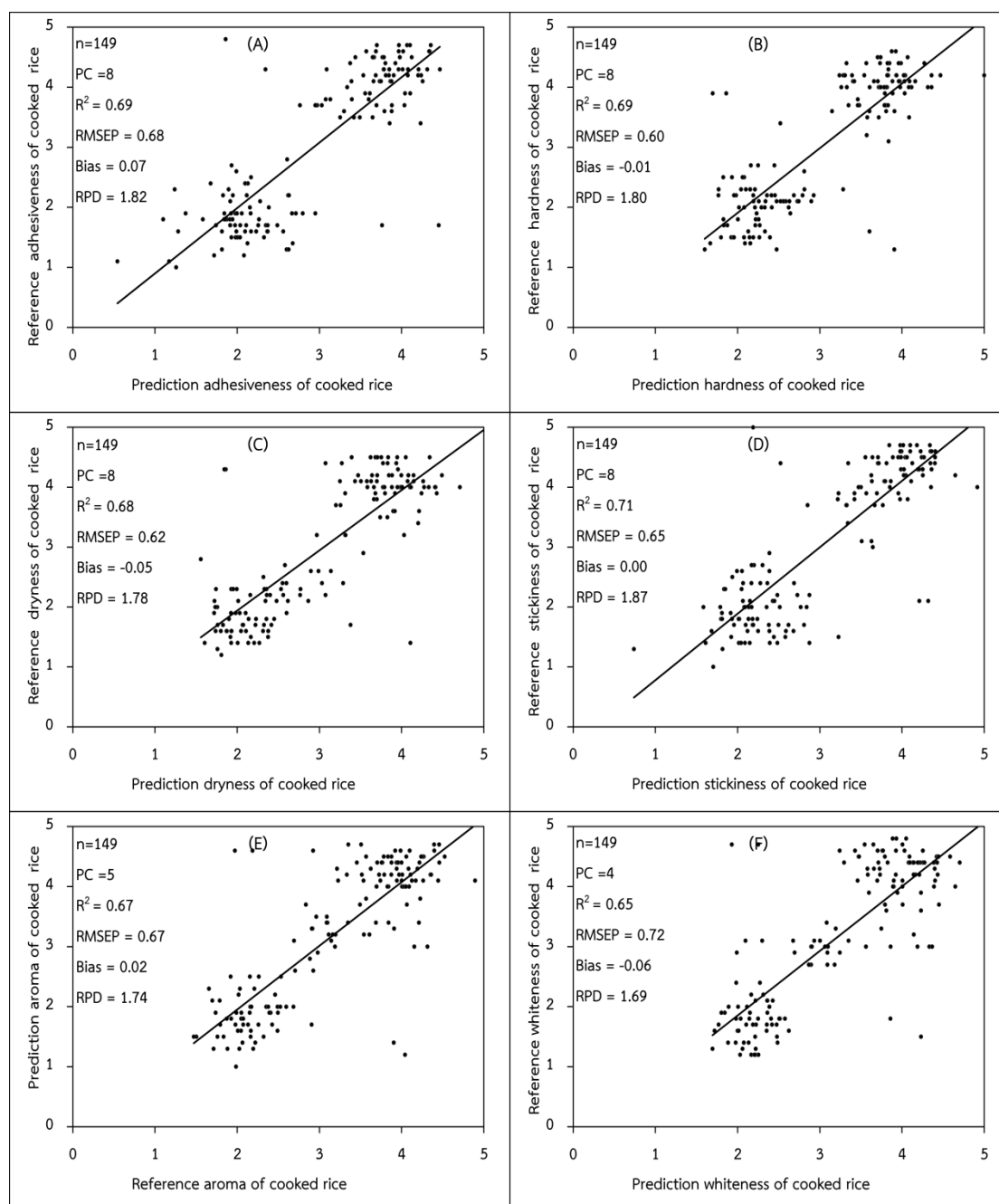
The prediction statistics for the PLS models to predict the sensory quality of cooked rice are shown in Table 4.7. The optimum model for the prediction of adhesiveness was established from the raw spectra. The max–min normalization method led to the most predictive models for hardness and stickiness. The models for dryness were derived from the spectra preprocessed using the first derivative and straight line subtraction method. The models for aroma and whiteness were created using spectra preprocessed using the multiplicative scattering correction (MSC) and vector normalization methods, respectively. The prediction results of all sensory quality indices showed  $R^2_{\text{val}}$  values between 0.65 and 0.71. The results for stickiness

model displayed the highest predictive capability of all the sensory quality indices (i.e.  $R^2_{\text{val}} = 0.71$ ; RMSEP = 0.65; Bias = 0.00; RPD = 1.87). The  $R^2_{\text{val}}$  values for adhesiveness and hardness, indicative of texture properties of cooked rice, were both 0.69. The dryness and whiteness parameters, both of which are evaluated visually, showed  $R^2_{\text{val}}$  values of 0.68 and 0.65, respectively. The aroma based models exhibited an  $R^2_{\text{val}}$  of 0.67. The scatter plots (reference data (X) with prediction data (Y)) of each of the sensory quality indices are shown in Figure 4.6. The lower ability of prediction of the models developed from cooked rice spectra than those developed from milled rice spectra might be due to the influence of high water content where the water band was large.

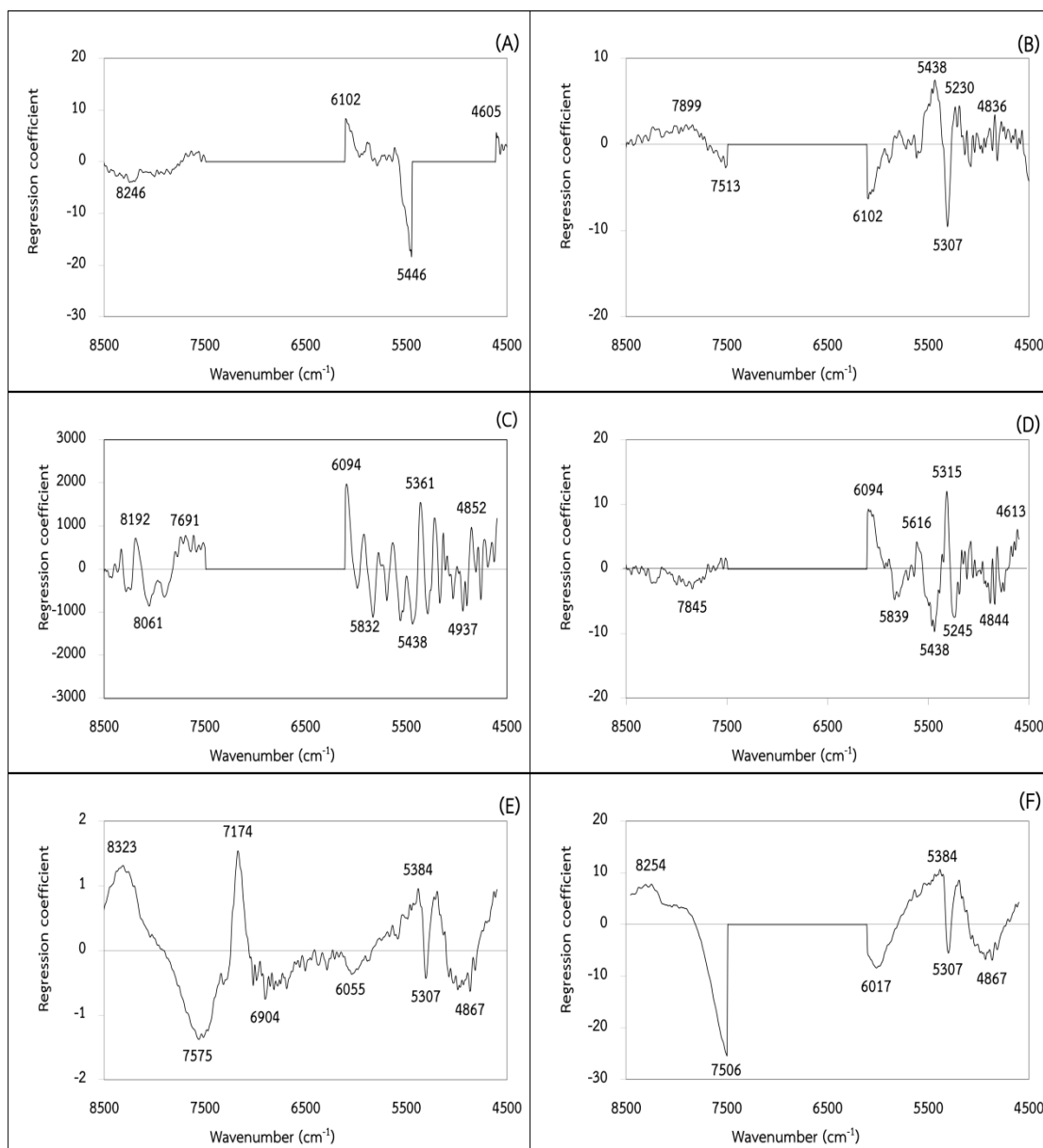
The regression coefficient plots of the optimum models for evaluating the sensory quality of cooked rice are shown in Figure 4.7. The regression coefficient plots for the sensory quality models do not show absorption bands associated with water (13158, 10309, 6897 and 5155  $\text{cm}^{-1}$ ) (Osborne & Fearn, 1986). It was clear that the water content was not closely related to the sensory quality of cooked rice. The regression coefficient plots of all sensory quality indices show peaks around 8840–8655  $\text{cm}^{-1}$  (1131–1155 nm). The prominent features around 8840–8655  $\text{cm}^{-1}$  are the absorption peaks associated with the first overtone of C–H stretching of the starch (i.e., 8841–8658  $\text{cm}^{-1}$  or 1131–1155 nm) (Shao et al., 2011). It is perhaps obvious that carbohydrates are the main chemical component of rice that affects the sensory quality.

Table 4.8 shows mean and standard deviation (SD) of the differences between the duplicates and SEP/SD of the overall precision tests. The SD values of the tests are the indication of precision of sensory panelist on evaluation of sensory quality of cooked rice. The repeatability of all sensory quality indices was less than 0.3 and the reproducibility was between 0.2–0.3 except for whiteness (0.5) which was 4–6% of maximum value (5). This indicated that the evaluation by panelist was fairly precise. The ratios of SEP/SD of reproducibility tests imply the performance of NIR spectroscopy model for practical application. The SEP/SD values of stickiness and whiteness model was 2 indicated that the model was good. The values for models of adhesiveness, hardness and dryness were 3 indicated the poor performance of the

models. The model for aroma could not be recommended because the SEP/SD was 4.



**Figure 4.6** The scatter plots of reference data with prediction data of sensory quality of cooked rice of prediction sensory quality of rice using NIR spectral of cooked rice scanning by FT-NIR spectrometer. (A) Adhesiveness; (B) Hardness; (C) Dryness; (D) Stickiness; (E) Aroma; (F) Whiteness.



**Figure 4.7** Regression coefficient plots of optimum models for evaluation of sensory quality of cooked rice developed by using NIR spectra of cooked rice scanned by FT-NIR spectrometer. (A) Adhesiveness; (B) Hardness; (C) Dryness; (D) Stickiness; (E) Aroma; (F) Whiteness.

**Table 4.6** Minimum (Min), maximum (Max), mean, and standard deviation (SD) of sensory quality indices of rice of calibration and validation groups used for model development where the NIR spectra of cooked rice scanned by FT-NIR spectrometer.

Parameter	Calibration					Prediction				
	Number of Samples	Min	Max	Average	SD	Number of Samples	Min	Max	Average	SD
Adhesiveness	601	1.0	4.8	2.4	1.10	149	1.0	4.8	3.0	1.23
Hardness	601	1.3	4.7	3.5	0.98	149	1.3	4.6	3.0	1.09
Dryness	601	1.2	4.7	3.5	0.98	149	1.2	4.5	3.0	1.11
Stickiness	601	1.0	5.0	2.5	1.08	149	1.0	5.0	3.1	1.23
Aroma	601	1.0	4.8	3.7	0.98	149	1.0	4.7	3.2	1.16
Whiteness	601	1.2	5.0	3.7	1.01	149	1.2	4.8	3.1	1.21

**Table 4.7** Statistics of prediction of sensory quality of cooked rice by PLS models using NIR spectra of cooked rice scanned by FT-NIR spectrometer.

Parameter	Pre-treatment	Wavenumber range (cm <sup>-1</sup> )	PC	R <sup>2</sup> <sub>cal</sub>	R <sup>2</sup> <sub>val</sub>	RMSEE	RMSEP	BIAS	RPD
Adhesiveness	Raw spectra	9403.8-7498.3, 6102-5446.3, 4605.4-4242.9	8	0.71	0.69	0.60	0.68	0.07	1.82
Hardness	Min-max normalization	9403.8-7498.3, 6102-4242.9	8	0.69	0.69	0.55	0.60	-0.01	1.80
Dryness	First derivative + Straight line subtraction	9403.8-7498.3, 6102-4597.7	8	0.69	0.68	0.55	0.62	-0.05	1.78
Stickiness	Min-max normalization	9403.8-7498.3, 6102-4597.7	8	0.71	0.71	0.59	0.65	0.00	1.87
Aroma	Multiplicative scattering correction	9403.8-4597.7	5	0.65	0.67	0.59	0.67	0.02	1.74
Whiteness	Vector normalization	8454.9-7498.3, 6012-4597.7	4	0.63	0.65	0.62	0.72	-0.06	1.69

PC - Optimal number of principal components

R<sup>2</sup><sub>cal</sub> - The coefficient of determination of calibration set

R<sup>2</sup><sub>val</sub> - The coefficient of determination of validation set

RMSEE – Root mean square error of calibration

RMSEP - Root mean square error of prediction

RPD - Ratio of standard deviation of validation data to Root mean square error of prediction

**Table 4.8** Means and standard deviations (SD) of the differences between the duplicates and ratios of standard error of prediction (SEP) to standard deviation (i.e., SEP/SD) of repeatability and reproducibility tests of sensory quality of rice.

Quality	Repeatability test		Reproducibility test		
	Mean	SD	Mean	SD	SEP/SD
Adhesiveness	-0.1	0.2	0	0.2	3
Hardness	0	0.3	-0.1	0.2	3
Dryness	0.1	0.2	-0.1	0.2	3
stickiness	0	0.2	0.1	0.3	2
Aroma	-0.2	0.2	0.2	0.2	4
Whiteness	-0.1	0.2	0	0.5	2

#### 4.5.3 Prediction results of sensory quality of cooked rice using milled rice spectra obtained from short wavelength diode array

Mean, SD, Max, and Min values of the sensory qualities of cooked rice for calibration and validation groups are shown in Table 4.9. The range of stickiness was widest (1.0–5.0), that of hardness was narrowest (1.3–4.7), that of adhesiveness, aroma, and whiteness was 1.0–4.8, and that of dryness was 1.2–4.7.

The statistics regarding prediction of the sensory qualities of cooked rice by the PLS models are shown in Table 4.10. The general optimum models (i.e., adhesiveness, stickiness, aroma, and whiteness) were derived from spectra preprocessed by the range normalization method. The de-trending method was found to be the spectral preprocessed method that gave the optimum model for prediction of hardness. The models for prediction of dryness were created by the spectra that were preprocessed by the baseline offset method. The prediction results of all sensory quality indices showed a range of  $R^2_{\text{val}}$  between 0.842 and 0.918. The results for aroma indicated the highest prediction ability ( $R^2_{\text{val}} = 0.918$ ; SEP = 0.284; bias = -0.043; RPD = 3.535), while  $R^2_{\text{val}}$  for adhesiveness, hardness, and

stickiness parameters indicating the textural properties of cooked rice ranged from 0.842 to 0.904. Dryness and whiteness, which were evaluated visually, yielded  $R^2_{\text{val}}$  of 0.895 and 0.866, respectively. The scatter plots (reference data with prediction data) of sensory qualities are shown in Figure 4.8. This is the first time that VIS and SW-NIR in the range of 600–1100 nm have been applied to evaluate the sensory qualities of rice where a wide variation matrix was applied to different varieties and processed rice. Our overall sensory quality prediction ( $R^2_{\text{val}} = 0.842\text{--}0.918$ ) was better than that reported by Qingyun et al. (2007) who recorded 0.31–0.77, likely because their prediction model was developed by multiple linear regression using wavelengths of 540, 640, and 970 nm only. In addition, our prediction of certain texture parameters was better than that obtained by Meullenet et al. (2002) where wavelengths of 400–2500 nm were used ( $R^2_{\text{val}}$  for hardness was 0.67); and that reported by Champagne et al. (2001) ( $R^2_{\text{val}}$  for hardness and stickiness was 0.67 and 0.58, respectively). The better prediction found in our work might be due to the wider range of VIS and SWNIR (600–1100 nm), the higher number of samples and enhanced VIS and SW-NIR spectral correlation of our sensory method. Compared with the research findings of Srisawas et al. (2007) where longwave NIR (1100–2500 nm) was used and those of Meullenet et al. (2002), our RPDs were higher (>2.5) for all prediction models. The SD in this study ranges from 1.00 to 1.15, which is nearly twice that reported in the literature cited above, thus yielding a higher RPD. Our higher SD value was obtained because of the wider variation matrix of different varieties and processed rice, whereas other studies included no processed products, such as parboiled rice, in their modeling.

**Table 4.9** Minimum (Min), maximum (Max), mean, and standard deviation (SD) of sensory quality indices of rice of calibration and validation groups used for model development where the NIR spectra of milled rice scanned by short wavelength diode array.

	Calibration					Prediction				
	Number of Sample	Min	Max	Average	SD	Number of Sample	Min	Max	Average	SD
Adhesiveness	201	1.0	4.8	2.5	1.16	49	1.1	4.8	2.5	1.15
Hardness	201	1.3	4.7	3.4	1.03	49	1.5	4.7	3.4	1.01
Dryness	201	1.2	4.7	3.4	1.04	49	1.4	4.7	3.4	1.03
stickiness	201	1.0	5.0	2.6	1.14	49	1.2	4.7	2.6	1.13
Aroma	201	1.0	4.8	3.6	1.05	49	1.4	4.7	3.6	1.00
Whiteness	201	1.2	5.0	3.6	1.09	49	1.4	4.9	3.7	1.05

**Table 4.10** Statistics of prediction of sensory quality of cooked rice by PLS models using NIR spectra of milled rice scanned by short wavelength diode array.

Parameter	Pre-treatment	PC	$R^2_{cal}$	$R^2_{val}$	SEC	SEP	BIAS	RPD
Adhesiveness	Range Normalization	4	0.923	0.904	0.320	0.352	-0.051	3.259
Hardness	Detrending	3	0.875	0.842	0.364	0.399	0.023	2.524
Dryness	Baseline offset	3	0.892	0.895	0.343	0.327	0.062	3.144
stickiness	Range Normalization	4	0.913	0.891	0.337	0.368	0.053	3.068
Aroma	Range Normalization	4	0.911	0.918	0.314	0.284	-0.043	3.535
Whiteness	Range Normalization	4	0.928	0.866	0.292	0.370	0.102	2.838

PC - Optimal number of principal components

$R^2_{cal}$  - The coefficient of determination of calibration set (full cross-validation)

$R^2_{val}$  - The coefficient of determination of validation set

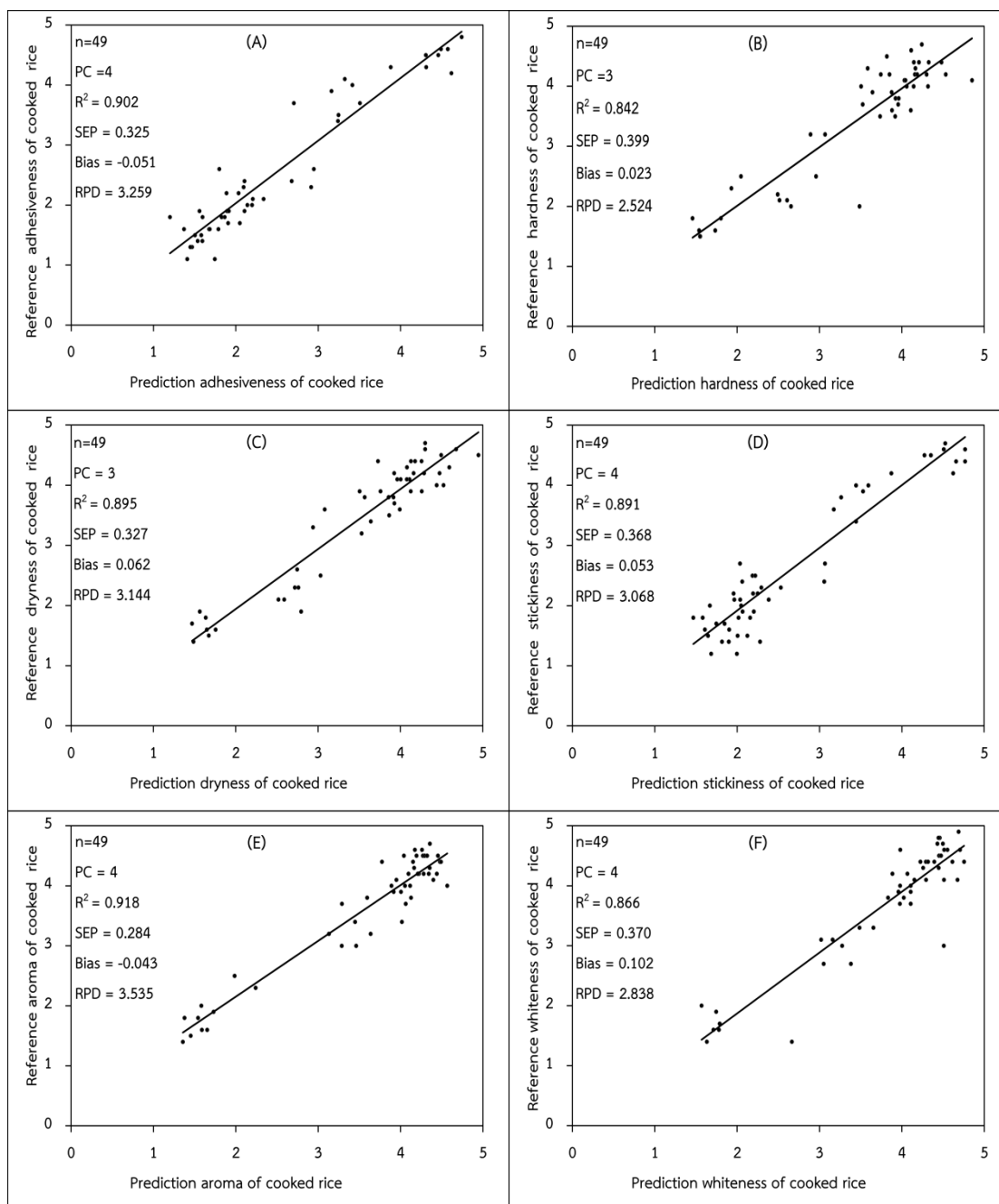
SEC - Standard error of calibration set (full cross-validation)

SEP - Standard error of prediction

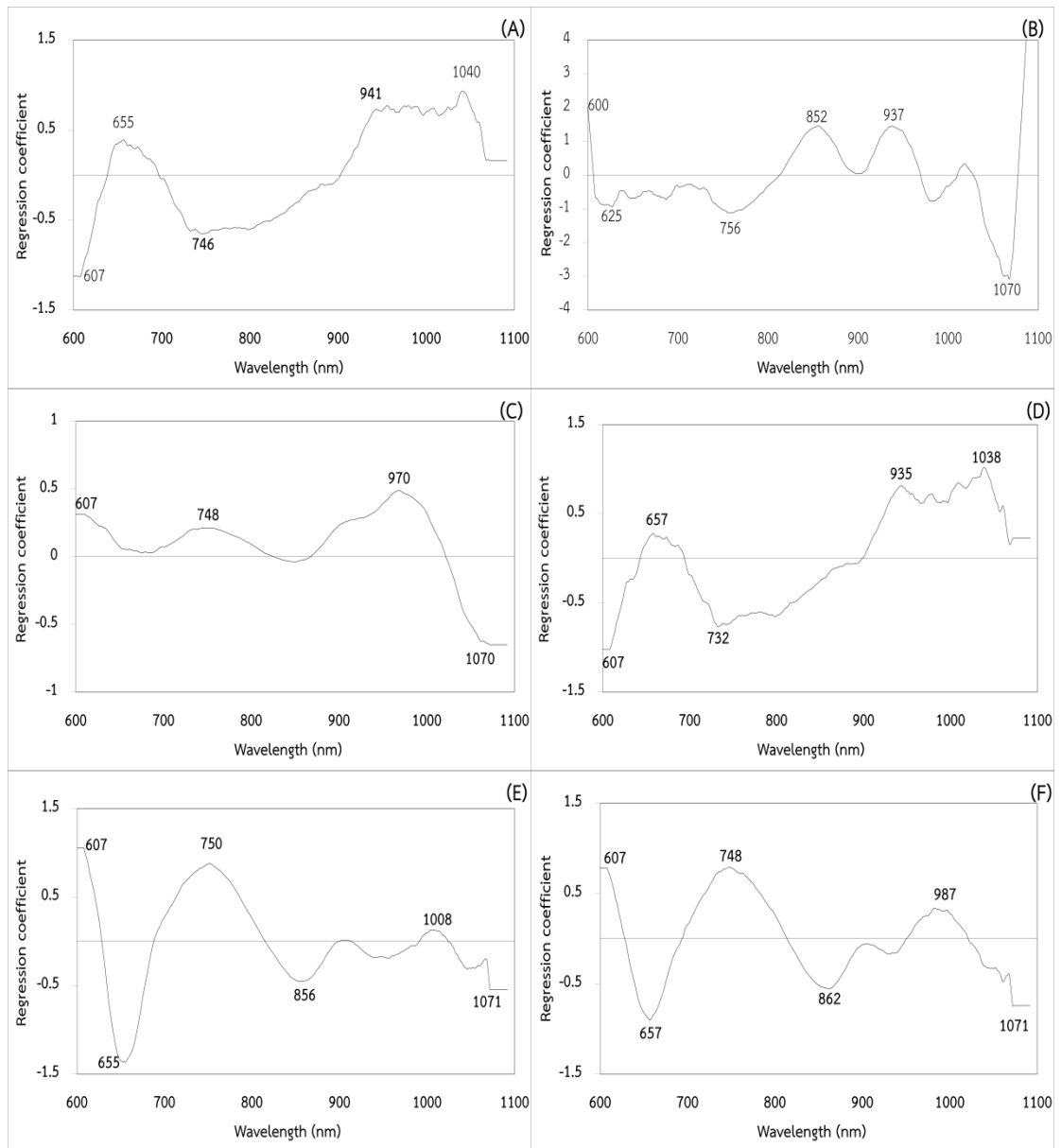
RPD - Ratio of standard deviation of validation data to standard error of prediction

The regression coefficient plots of optimum models for evaluation of the sensory qualities of cooked rice are shown in Figure 4.9. The peak regression coefficient appeared at around 746–750 nm for all sensory quality indices. The peaks at 746 and 747 nm show the vibration frequency of the C–H stretch fourth overtone of CH<sub>2</sub> and O–H stretch third overtone of ArOH (Osborne and Fearn, 1986). ArOH is the chemical structure of phenolic compounds in rice (Huang and Ng, 2012). Adhesiveness, hardness, and stickiness, which was the textural qualities of cooked rice, showed the common peak of regression coefficient (935–990 nm). The prominent features at 938 nm are the absorption peaks associated with the C–H stretch third overtone of CH<sub>2</sub> (Osborne and Fearn, 1986). In addition, the peak at 990 nm is the vibration band of the O–H stretch second overtone of starch (Osborne and Fearn, 1986). The absorption peak of water (970 nm) is shown on a regression coefficient plot of texture attributes and dryness, indicating that the attributes were affected by starch and water. This is consistent with the work of Srisawas et al. (2007) who studied the application of long-wave NIRS in the evaluation of sensory textural attributes where the identical strong absorption peak of NIR spectra was at 1894 nm (O–H stretch + 2 × C–O stretch [1900 nm] [starch]) for both milled and cooked rice.

The mean and SD of the differences between the duplicates and SEP/SD of repeatability and reproducibility tests are shown in Table 4.11. The SEP/SD values for general sensory quality (adhesiveness, hardness, dryness, stickiness, and aroma) of cooked rice were found to be more than 1. The results indicated the robustness of the models and indicating that SW-NIR spectroscopy of milled rice can satisfactorily be applied to evaluate the sensory qualities of cooked rice.



**Figure 4.8** The scatter plots of reference data with prediction data of sensory quality of cooked rice of prediction sensory quality of rice using NIR spectral of milled rice scanning by short wavelength diode array. (A) Adhesiveness; (B) Hardness; (C) Dryness; (D) Stickiness; (E) Aroma; (F) Whiteness.



**Figure 4.9** Regression coefficient plots of optimum models for evaluation of sensory quality of cooked rice developed by using NIR spectra of milled rice scanned by short wavelength diode array. (A) Adhesiveness; (B) Hardness; (C) Dryness; (D) Stickiness; (E) Aroma; (F) Whiteness.

**Table 4.11** Means and standard deviations (SD) of the differences between the duplicates and ratios of standard error of prediction (SEP) to standard deviation (i.e., SEP/SD) of repeatability and reproducibility tests of sensory quality of rice.

Sensory quality	Repeatability		Reproducibility		
	Mean	SD	Mean	SD	SEP/SD
Adhesiveness	-0.1	0.2	0.0	0.2	2
Hardness	0.0	0.3	-0.1	0.2	2
Dryness	0.1	0.2	-0.1	0.2	2
stickiness	0.0	0.2	0.1	0.3	1
Aroma	-0.2	0.2	0.2	0.2	2
Whiteness	-0.1	0.2	0.0	0.5	1

#### 4.5.4 Prediction results of sensory quality of cooked rice using cooked rice spectra obtained from short wavelength diode array

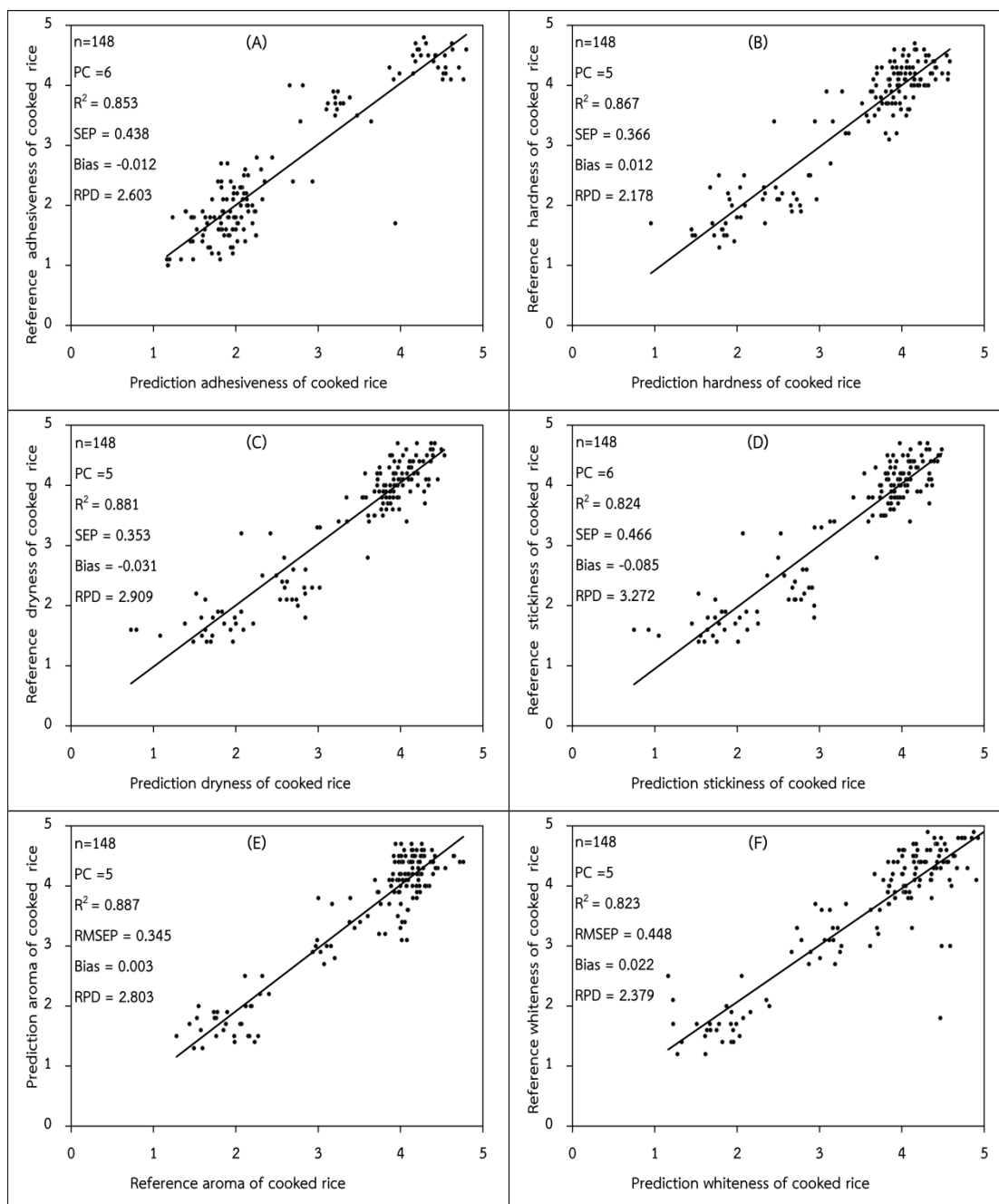
Mean, SD, Max, and Min values of the sensory qualities of cooked rice for calibration and validation groups are shown in Table 4.12. The range of stickiness was widest, i.e. 1.0 to 5.0., whereas the dryness range was narrowest, 1.2 to 4.7. The range of adhesiveness was equal to those of aroma and whiteness (1.0 to 4.8), likewise range of hardness and whiteness was 1.2 to 5.0.

The statistics of prediction of the sensory quality of cooked rice by PLS models are shown in Table 4.13. The optimum models for stickiness evaluation were derived from the spectra that were preprocessed by the baseline offset method. The 1st derivative (with 2 smoothing points on both left and right sides) method was the spectra preprocessed method that gave the optimum model for prediction of hardness and dryness. The optimum model for prediction of adhesiveness was established from spectra that pre-processed by range normalization method. The models for prediction of aroma and whiteness were created by the spectra that were preprocessed respectively with MSC and SNV+detrending. The prediction results of all sensory quality indices showed  $R^2_{\text{val}}$  between 0.823 and 0.887. The results of

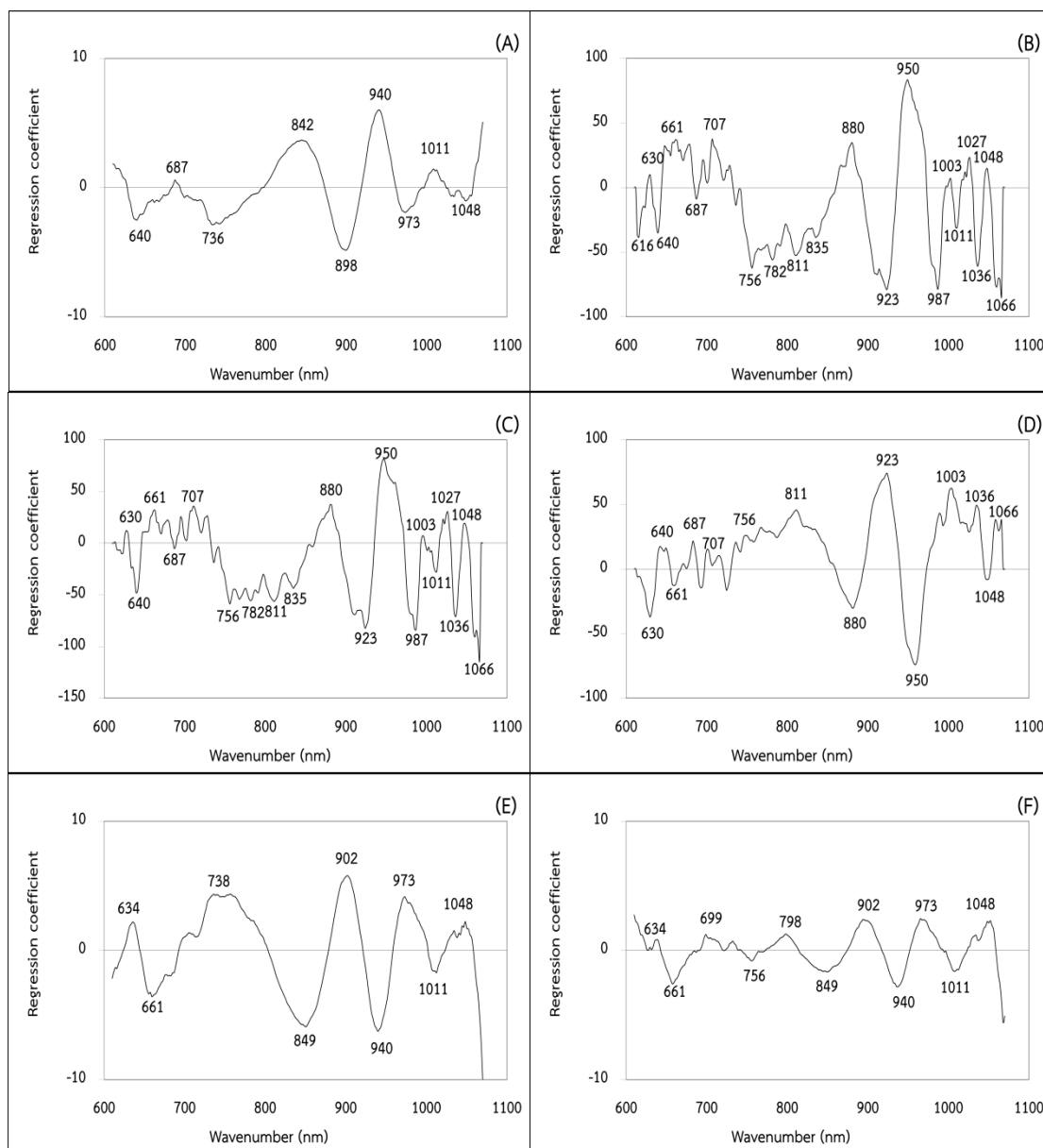
aroma indicated its highest prediction ability of all the sensory quality indices (i.e.  $R^2_{\text{val}} = 0.887$ ; SEP = 0.345; Bias = 0.003; RPD = 2.803).  $R^2_{\text{val}}$  for adhesiveness, hardness and stickiness, all indicating the texture properties of cooked rice ranged from 0.824 to 0.867. The dryness and whiteness, which were evaluated visually, showed  $R^2_{\text{val}}$  of 0.881 and 0.823 respectively. The aroma of cooked rice exhibited  $R^2_{\text{val}}$  of 0.887. The scatter plots (reference data with prediction data) of the sensory quality are shown in Figure 4.10.

The regression coefficient plots of optimum models for evaluation of the sensory qualities of cooked rice are shown in Figure 4.11. The peak regression coefficient appeared at 1048 nm for all sensory quality indices. Adhesiveness, hardness, and stickiness, the textural qualities of cooked rice, showed the same peak of regression coefficient (640, 687 and 1048 nm). The peak at 1047 is related to the alkyl alcohols (O-H with hydrogen bonding (R-C-OH)) (Workman and Weyer, 2008). Adhesiveness, aroma and whiteness showed the same peak of regression coefficient about 940 nm. The prominent features at 938 nm are the absorption peaks associated with the C-H stretch third overtone of  $\text{CH}_2$  (Osborne & Fearn, 1986). The regression coefficient plots for all sensory quality models do not show absorption bands associated with water (760 and 970 nm) (Osborne & Fearn, 1986). It was clear that the water content of the rice samples was not related to the sensory quality of the cooked rice.

The mean and standard deviation (SD) of the differences between the duplicates and SEP/SD of repeatability and reproducibility tests are shown in Table 4.14. The SEP/SD values of whiteness were of 1. This indicated the robustness of whiteness model. Adhesiveness, hardness, dryness, stickiness and aroma showed SEP/SD values of 2, thereby indicating that the NIR spectroscopy of milled rice could satisfactorily be applied to evaluate adhesiveness, hardness, dryness, stickiness and aroma of cooked rice.



**Figure 4.10** The scatter plots of reference data with prediction data of sensory quality of cooked rice of prediction sensory quality of rice using NIR spectral of cooked rice scanning by FT-NIR spectrometer. (A) Adhesiveness; (B) Hardness; (C) Dryness; (D) Stickiness; (E) Aroma; (F) Whiteness.



**Figure 4.11** Regression coefficient plots of optimum models for evaluation of sensory quality of cooked rice developed by using NIR spectra of cooked rice scanned by short wavelength diode array. (A) Adhesiveness; (B) Hardness; (C) Dryness; (D) Stickiness; (E) Aroma; (F) Whiteness.

**Table 4.12** Minimum (Min), maximum (Max), mean, and standard deviation (SD) of sensory quality indices of rice of calibration and validation groups used for model development where the NIR spectra of cooked rice scanned by short wavelength diode array.

	Calibration					Validation				
	Number of Samples	Min	Max	Mean	SD	Number of Samples	Min	Max	Mean	SD
Adhesiveness	600	1.0	4.8	2.5	1.15	148	1.0	4.8	2.5	1.14
Hardness	600	1.2	5.0	3.6	1.08	148	1.2	4.9	3.6	1.07
Dryness	600	1.2	4.7	3.4	1.04	148	1.4	4.7	3.4	1.03
Stickiness	600	1.0	5.0	2.6	1.14	148	1.0	4.7	2.6	1.13
Aroma	600	1.0	4.8	3.6	1.05	148	1.3	4.7	3.6	1.03
Whiteness	600	1.2	5.0	3.6	1.08	148	1.2	4.9	3.6	1.07

**Table 4.13** Statistics of prediction of sensory quality of cooked rice by PLS models using NIR spectra of cooked rice scanned by short wavelength diode array.

Parameters	Preprocessing	PC	$R^2_{cal}$	$R^2_{val}$	SEC	SEP	Bias	RPD
Adhesiveness	Range Normalization	6	0.860	0.853	0.431	0.438	-0.012	2.603
Hardness	1 <sup>st</sup> Derivative <sup>a</sup>	5	0.823	0.867	0.431	0.366	0.012	2.178
Dryness	1 <sup>st</sup> Derivative <sup>a</sup>	5	0.845	0.881	0.410	0.353	-0.031	2.909
Stickiness	Baseline offset	6	0.832	0.824	0.427	0.466	-0.085	3.272
Aroma	MSC	5	0.817	0.887	0.447	0.345	0.003	2.803
Whiteness	SNV+detrending	5	0.863	0.823	0.401	0.448	0.022	2.379

<sup>a</sup>Number of smoothing points: 2 points on the left and the right sides.

PC - Optimal number of principal components

$R^2_{cal}$  - The coefficient of determination of calibration set

$R^2_{val}$  - The coefficient of determination of validation set

SEC – Standard error of calibration

SEP - Standard error of prediction

RPD - Ratio of standard deviation of validation data to Root mean square error of prediction

**Table 4.14** Means and standard deviations (SD) of the differences between the duplicates and ratios of standard error of prediction (SEP) to standard deviation (i.e., SEP/SD) of repeatability and reproducibility tests of sensory quality of rice.

Sensory quality	Repeatability test		Reproducibility test		
	Mean	SD	Mean	SD	SEP/SD
Adhesiveness	-0.1	0.2	0	0.2	2
Hardness	0	0.3	-0.1	0.2	2
Dryness	0.1	0.2	-0.1	0.2	2
stickiness	0	0.2	0.1	0.3	2
Aroma	-0.2	0.2	0.2	0.2	2
Whiteness	-0.1	0.2	0	0.5	1

#### 4.5.5 Prediction results of sensory quality of cooked rice using milled rice spectra obtained from long wavelength diode array

Mean, SD, Max, and Min values of the sensory qualities of cooked rice for calibration and validation groups are shown in Table 4.15. The range of stickiness was widest (1.0–5.0), that of hardness was narrowest (1.3–4.7), that of adhesiveness and aroma, was 1.0–4.8, and that of dryness was 1.2–4.7. The whiteness was 1.2–5.0.

The statistics regarding prediction of the sensory qualities of cooked rice by the PLS model are shown in Table 4.16. Every model was derived from spectra preprocessed by the Savitzky Golay smoothing average (polynomial order 2 with number of smoothing point of 5). The prediction results of all sensory quality indices showed a range of  $R^2_{\text{val}}$  between 0.194 and 0.389. The results for aroma indicated the highest prediction ability ( $R^2_{\text{val}} = 0.389$ ; SEP = 0.782; bias = -0.098; RPD = 1.292), while  $R^2_{\text{val}}$  for adhesiveness, hardness, and stickiness parameters indicating the textural properties of cooked rice ranged from 0.297 to 0.386. Dryness and whiteness, which were evaluated visually, yielded  $R^2_{\text{val}}$  of 0.360 and 0.400, respectively. The scatter plots (reference data with prediction data) of sensory qualities are shown in Figure 4.12.

The regression coefficient plots of optimum models for evaluation of the sensory quality of cooked rice are shown in Figure 4.13. The regression coefficient plots of all sensory qualities present many sharp peaks but they were not the absorption band of water. It indicated that the moisture content of milled rice had no relation to the sensory qualities of rice. This fact is consistent with the work by Srisawas et al. (2007), who studied the application of NIRS in evaluation of sensory textural attributes. Consistent with our study, the work by Srisawas et al. (2007) reported the identical strong absorption peaks of NIR spectra at 1404 and 1894 nm for both milled rice and cooked rice. All sensory qualities showed the regression coefficient peak at 1528 which is vibration bond of starch (Osborne & Fearn, 1986). It was obvious that starch as the main chemical component of rice, was the dominant factor.

The mean and SD of the differences between the duplicates and SEP/SD of repeatability and reproducibility tests are shown in Table 4.17. The SEP/SD values of all sensory quality of cooked rice were more than 2. The results indicated the weakness of both models.

**Table 4.15** Minimum (Min), maximum (Max), mean, and standard deviation (SD) of sensory quality indices of rice of calibration and validation groups used for model development where the NIR spectra of milled rice scanned by long wavelength diode array.

	Calibration					Prediction				
	Number of Sample	Min	Max	Average	SD	Number of Sample	Min	Max	Average	SD
Adhesiveness	197	1.0	4.8	2.5	1.15	48	1.1	4.7	2.5	1.14
Hardness	197	1.3	4.7	3.4	1.03	48	1.5	4.6	3.4	1.00
Dryness	197	1.2	4.7	3.4	1.04	48	1.4	4.7	3.5	1.02
Stickiness	197	1.0	5.0	2.6	1.14	48	1.2	4.6	2.6	1.12
Aroma	197	1.0	4.8	3.6	1.05	48	1.4	4.7	3.6	1.01
Whiteness	197	1.2	5.0	3.6	1.09	48	1.4	4.9	3.7	1.05

**Table 4.16** Statistics of prediction of sensory quality of cooked rice by PLS models using NIR spectra of milled rice scanned by long wavelength diode array.

Parameter	Pre-treatment	PC	$R^2_{cal}$	$R^2_{val}$	SEC	SEP	BIAS	RPD
Adhesiveness	Savitzky golay smoothing <sup>a</sup>	4	0.323	0.297	0.946	0.957	-0.040	1.191
Hardness	Savitzky golay smoothing <sup>a</sup>	9	0.484	0.349	0.740	0.811	0.015	1.233
Dryness	Savitzky golay smoothing <sup>a</sup>	5	0.360	0.368	0.834	0.812	0.046	1.256
Stickiness	Savitzky golay smoothing <sup>a</sup>	5	0.345	0.386	0.924	0.875	-0.039	1.280
Aroma	Savitzky golay smoothing <sup>a</sup>	8	0.532	0.389	0.721	0.782	-0.098	1.292
Whiteness	Savitzky golay smoothing <sup>a</sup>	4	0.400	0.194	0.844	0.944	0.037	1.112

<sup>a</sup>Number of smoothing points: 2 points on the left and the right sides.

PC - Optimal number of principal components

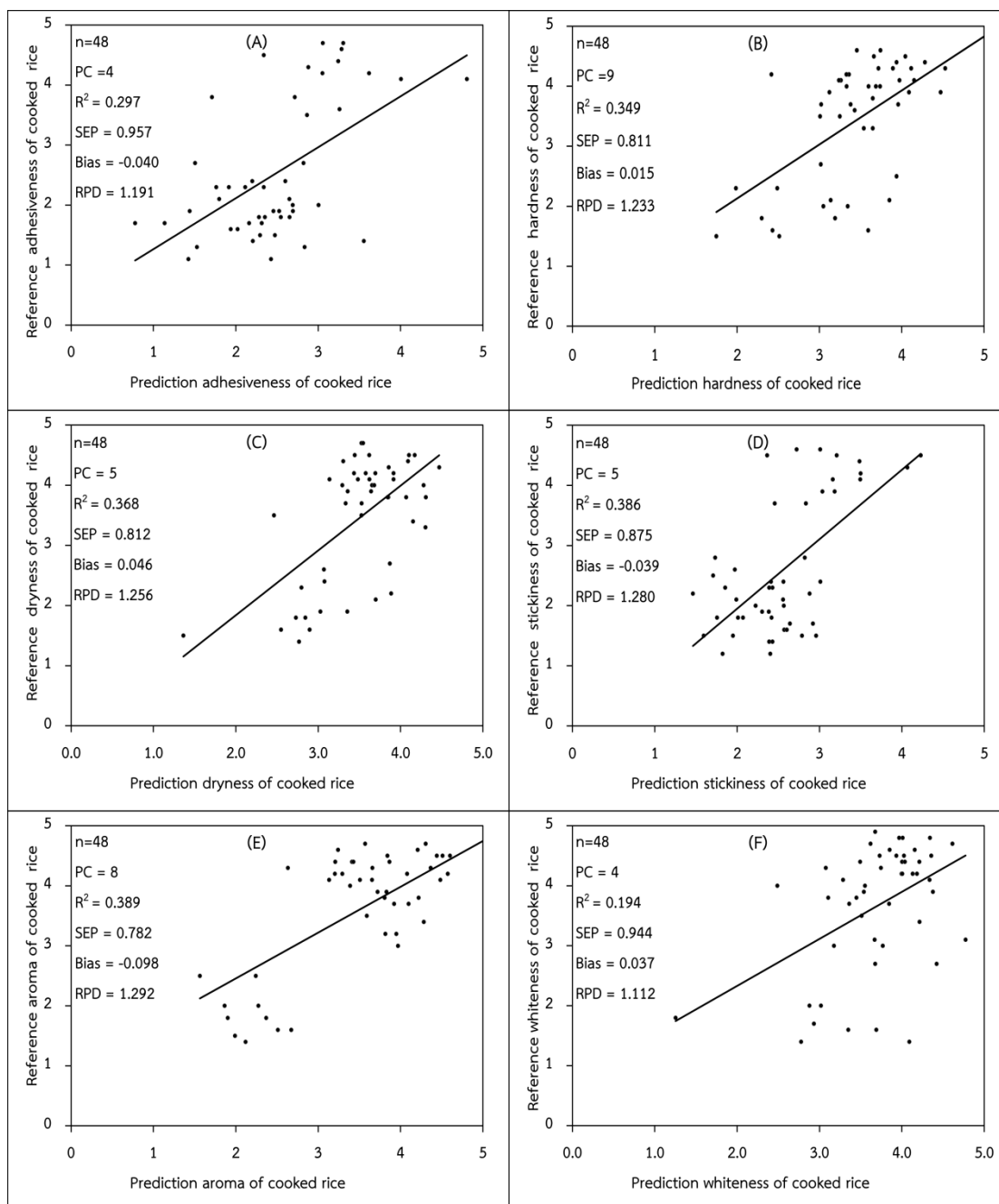
$R^2_{cal}$  - The coefficient of determination of calibration set

$R^2_{val}$  - The coefficient of determination of validation set

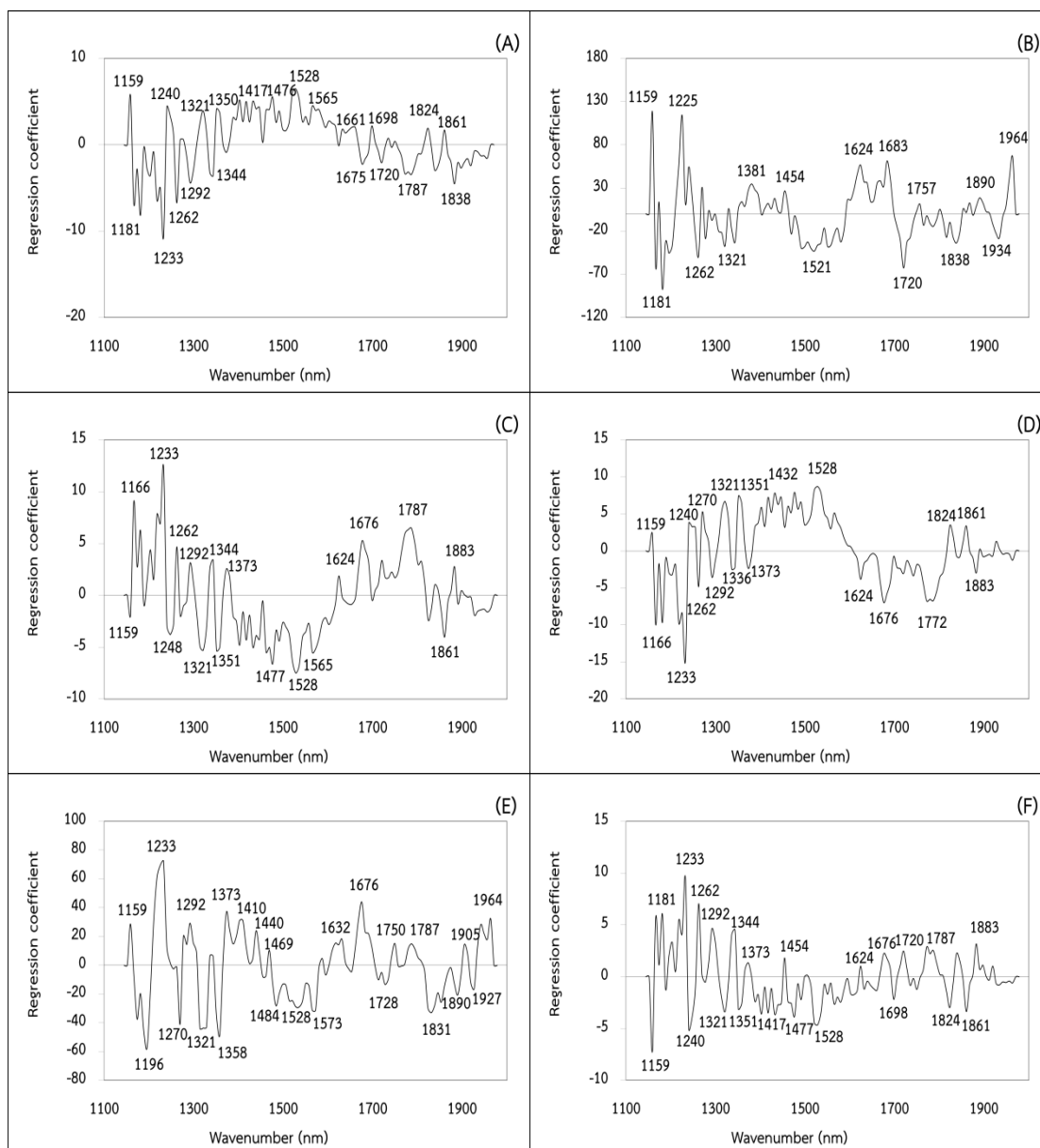
SEC – Standard error of calibration

SEP - Standard error of prediction

RPD - Ratio of standard deviation of validation data to Root mean square error of prediction



**Figure 4.12** The scatter plots of reference data with prediction data of sensory quality of cooked rice of prediction sensory quality of rice using NIR spectral of milled rice scanning by long wavelength diode array. (A) Adhesiveness; (B) Hardness; (C) Dryness; (D) Stickiness; (E) Aroma; (F) Whiteness.



**Figure 4.13** Regression coefficient plots of optimum models for evaluation of sensory quality of cooked rice developed by using NIR spectra of milled rice scanned by long wavelength diode array. (A) Adhesiveness; (B) Hardness; (C) Dryness; (D) Stickiness; (E) Aroma; (F) Whiteness.

**Table 4.17** Means and standard deviations (SD) of the differences between the duplicates and ratios of standard error of prediction (SEP) to standard deviation (i.e., SEP/SD) of repeatability and reproducibility tests of sensory quality of rice.

Sensory quality	Repeatability test		Reproducibility test		
	Mean	SD	Mean	SD	SEP/SD
Adhesiveness	-0.1	0.2	0	0.2	5
Hardness	0	0.3	-0.1	0.2	4
Dryness	0.1	0.2	-0.1	0.2	4
stickiness	0	0.2	0.1	0.3	3
Aroma	-0.2	0.2	0.2	0.2	4
Whiteness	-0.1	0.2	0	0.5	2

#### 4.5.6 Prediction results of sensory quality of cooked rice using cooked rice spectra obtained from long wavelength diode array

Mean, SD, Max, and Min values of the sensory qualities of cooked rice for calibration and validation groups are shown in Table 4.18. The range of stickiness values was widest, i.e., 1.0 to 5.0., whereas the hardness range was narrowest, 1.3 to 4.7. The range of adhesiveness was equal to those of aroma, and whiteness (1.0 to 4.8), while the dryness range was between 1.2 and 4.7. The sensory quality has an SD of validation between 1.09 and 1.23. The SD of adhesiveness and stickiness was highest at 1.23.

The prediction statistics for the PLS models to predict the sensory quality of cooked rice are shown in Table 4.19. All the models for the prediction of sensory quality was established from spectra preprocessed by the Savitzky Golay smoothing average (polynomial order 2 with number of smoothing point of 5). The prediction results of all sensory quality indices showed  $R^2_{\text{val}}$  values between 0.473 and 0.544. The results for stickiness displayed the highest predictive capability of all the sensory quality indices (i.e.  $R^2_{\text{val}} = 0.544$ ; RMSEP = 0:758; Bias = -0:074; RPD = 1:491). The  $R^2_{\text{val}}$  values for adhesiveness and hardness, indicative of texture properties of cooked rice, were 0.537 and 0.504 respectively. The dryness and whiteness parameters, both of

which are evaluated visually, showed  $R_{\text{val}}^2$  values of 0.523 and 0.508, respectively. The aroma based models exhibited an  $R_{\text{val}}^2$  of 0.473. The scatter plots (reference data (X) with prediction data (Y)) of each of the sensory quality indices are shown in Figure 4.14.

The regression coefficient plots of optimum models for evaluation of the sensory qualities of cooked rice are shown in Figure 4.15. All sensory qualities of rice presented the peak of regression coefficient at 1440 nm. This sharp peak is vibration bond of starch (O-H str. first overtone) (Osborne & Fearn, 1986). There was no absorption band of water. It indicated that the moisture content of milled rice had no relation to the sensory qualities of rice.

The mean and SD of the differences between the duplicates and SEP/SD of repeatability and reproducibility tests are shown in Table 4.17. The SEP/SD values of all sensory quality of cooked rice were more than 2. The results indicated the weakness of both models.

**Table 4.18** Minimum (Min), maximum (Max), mean, and standard deviation (SD) of sensory quality indices of rice of calibration and validation groups used for model development where the NIR spectra of milled rice scanned by long wavelength diode array.

	Calibration					Prediction				
	Number of Sample	Min	Max	Average	SD	Number of Sample	Min	Max	Average	SD
Adhesiveness	600	1.0	4.8	2.5	1.15	148	1.0	4.8	2.5	1.16
Hardness	600	1.3	4.7	3.4	1.03	148	1.3	4.7	3.4	1.01
Dryness	600	1.2	4.7	3.4	1.04	148	1.4	4.7	3.4	1.03
Stickiness	600	1.0	5.0	2.6	1.14	148	1.0	4.7	2.6	1.13
Aroma	600	1.0	4.8	3.6	1.05	148	1.3	4.7	3.6	1.03
Whiteness	600	1.2	5.0	3.6	1.08	148	1.2	4.9	3.6	1.07

**Table 4.19** Statistics of prediction of sensory quality of cooked rice by PLS models using NIR spectra of cooked rice scanned by long wavelength diode array.

Parameter	Pre-treatment	PC	$R^2_{cal}$	$R^2_{val}$	SEC	SEP	BIAS	RPD
Adhesiveness	Savitzky golay smoothing <sup>a</sup>	7	0.550	0.537	0.773	0.777	-0.031	1.493
Hardness	Savitzky golay smoothing <sup>a</sup>	7	0.530	0.504	0.702	0.714	0.014	1.415
Dryness	Savitzky golay smoothing <sup>a</sup>	5	0.500	0.523	0.735	0.709	0.006	1.453
stickiness	Savitzky golay smoothing <sup>a</sup>	6	0.519	0.544	0.791	0.758	-0.074	1.491
Aroma	Savitzky golay smoothing <sup>a</sup>	5	0.505	0.473	0.736	0.743	0.054	1.386
Whiteness	Savitzky golay smoothing <sup>a</sup>	5	0.511	0.508	0.756	0.744	0.080	1.438

<sup>a</sup>Number of smoothing points: 2 points on the left and the right sides.

PC - Optimal number of principal components

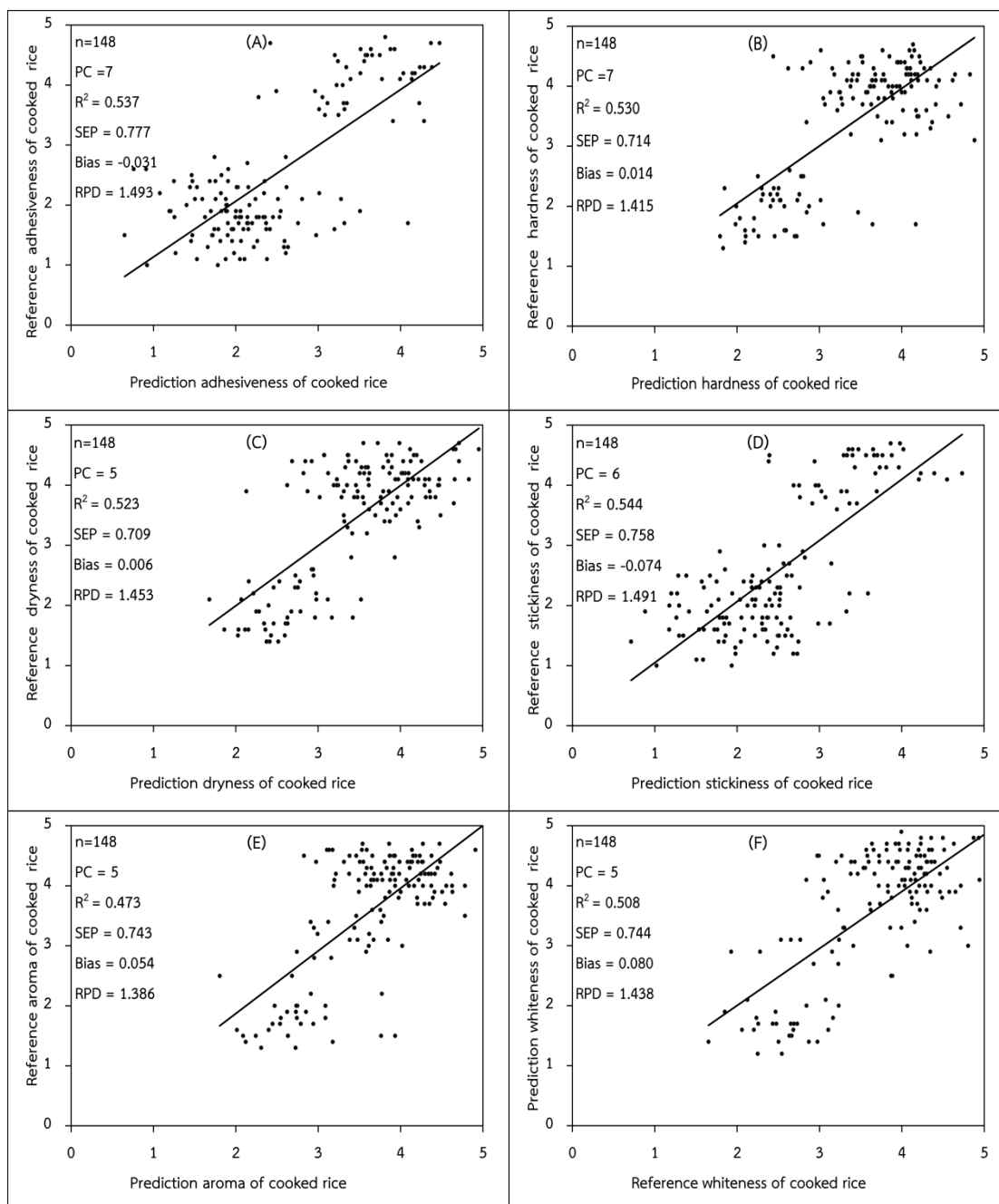
$R^2_{cal}$  - The coefficient of determination of calibration set

$R^2_{val}$  - The coefficient of determination of validation set

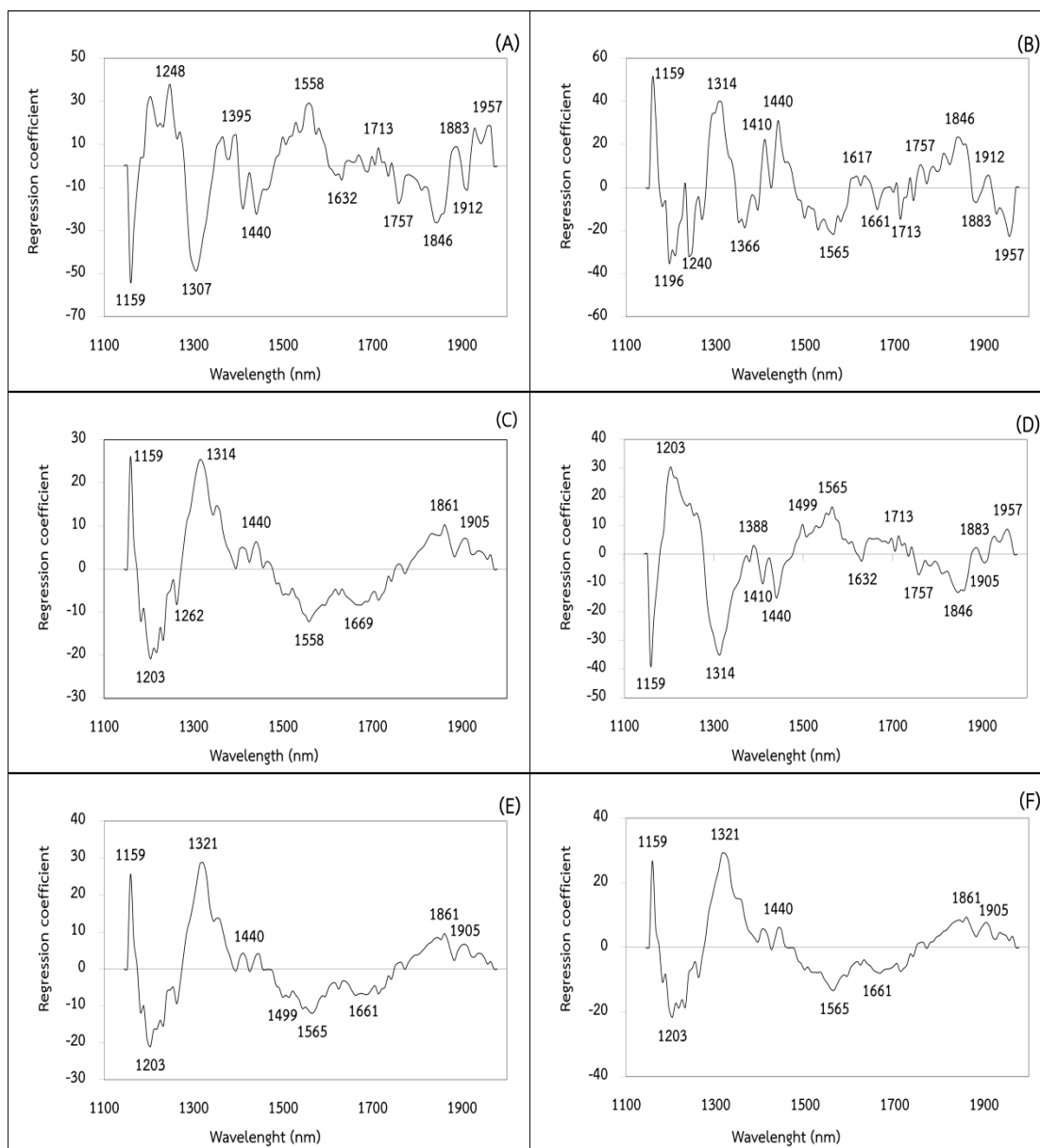
SEC – Standard error of calibration

SEP - Standard error of prediction

RPD - Ratio of standard deviation of validation data to Root mean square error of prediction



**Figure 4.14** The scatter plots of reference data with prediction data of sensory quality of cooked rice of prediction sensory quality of rice using NIR spectral of cooked rice scanning by long wavelength diode array. (A) Adhesiveness; (B) Hardness; (C) Dryness; (D) Stickiness; (E) Aroma; (F) Whiteness.



**Figure 4.15** Regression coefficient plots of optimum models for evaluation of sensory quality of cooked rice developed by using NIR spectra of cooked rice scanned by long wavelength diode array. (A) Adhesiveness; (B) Hardness; (C) Dryness; (D) Stickiness; (E) Aroma; (F) Whiteness.

**Table 4.20** Means and standard deviations (SD) of the differences between the duplicates and ratios of standard error of prediction (SEP) to standard deviation (i.e., SEP/SD) of repeatability and reproducibility tests of sensory quality of rice

Sensory quality	Repeatability test		Reproducibility test		
	Mean	SD	Mean	SD	SEP/SD
Adhesiveness	-0.1	0.2	0	0.2	4
Hardness	0	0.3	-0.1	0.2	4
Dryness	0.1	0.2	-0.1	0.2	4
stickiness	0	0.2	0.1	0.3	3
Aroma	-0.2	0.2	0.2	0.2	4
Whiteness	-0.1	0.2	0	0.5	2

## 4.6 RESULTS OF NIR SPECTROSCOPY FOR QUALITATIVE EVALUATION

### 4.6.1 Classification results of sensory quality of cooked rice by SIMCA method using milled rice and cooked spectra from FT-NIR spectrometer

Classification results of sensory qualities of cooked rice by SIMCA method using milled rice spectra scanned by FT-NIR spectrometer are shown in table 4.21. Classification models of all sensory quality showed that the overall error of false positive (1.1 to 11.6%) was lower than the overall error of false negative (40.0 to 96.5%). This indicated that these classification models could be better for self-classification of sensory quality but not for other group classification. The total error of all classification models were more than 10% (26.5 – 79.0%). Therefore, the models were not applicable.

Classification results of sensory qualities of cooked rice by SIMCA method using cooked rice spectra scanned by FT-NIR spectrometer are shown in table 4.22. Classification models of sensory quality showed the overall error of false positive

between 2.6 and 6.2% but the percentage of false negative results were more than 86.8%. However, the total error of all classification models was too high more than 10% (62.5 – 86.7%). Therefore, classification models of sensory quality were not applicable.

#### **4.6.2 Classification results of sensory quality of cooked rice by PLS-DA method using milled rice and cooked rice spectra from FT-NIR spectrometer**

Classification results of sensory qualities of cooked rice by PLS-DA method using milled rice spectra scanned by FT-NIR spectrometer are shown in table 4.23. Classification models of all sensory quality showed the overall error of false positive from 15.6. to 42.6%. The results of overall error of false negative were between 6.6 and 21.5%. The overall error of false positive of dryness was more than their overall error of false negative. On the other hand, other sensory qualities showed the overall error of false negative more than the overall error of false positive. This finding point indicated that dryness classification models could be better for self-classification but other sensory qualities classification models could be better for other group classification. The total error of all classification models were more than 10% (10.2 – 18.4%) therefore, the models were not applicable.

Classification results of sensory qualities of cooked rice by PLS-DA method using milled rice spectra scanned by FT-NIR spectrometer are shown in table 4.24. Classification models of all sensory quality showed the overall error of false positive from 37.3 to 83.8. The results of overall error of false negative were between 5.8 and 10.4. This indicated that these classification models could be better for other group classification than self-classification. The total error of all classification models were more than 10% (12.3 – 19.7%); therefore, the models were not applicable.

**Table 4.21** Classification results of sensory quality of cooked rice by SIMCA method using milled rice spectra from FT-NIR spectrometer.

Sensory Quality	Model	Error rate (%)		
		FP	FN	Total
Adhesiveness	very low	4.5	37.0	22.4
	low	0.0	56.8	22.4
	High	0.0	68.2	61.2
	Very high	0.0	44.9	44.9
	<b>Overall error</b>	<b>1.1</b>	<b>51.7</b>	<b>37.8</b>
Hardness	very low	0.0	69.8	61.2
	low	0.0	214.1	179.1
	High	8.3	67.6	53.1
	Very high	8.7	34.6	22.4
	<b>Overall error</b>	<b>4.3</b>	<b>96.5</b>	<b>79.0</b>
Dryness	very low	12.5	12.2	12.2
	low	0.0	46.5	40.8
	High	0.0	51.4	36.7
	Very high	4.8	50.0	30.6
	<b>Overall error</b>	<b>4.3</b>	<b>40.0</b>	<b>30.1</b>
Stickiness	very low	5.6	41.9	28.6
	low	6.3	26.2	36.7
	High	0.0	60.0	55.1
	Very high	0.0	60.5	46.9
	<b>Overall error</b>	<b>3.0</b>	<b>47.2</b>	<b>41.8</b>
Aroma	very low	0.0	61.9	53.1
	low	0.0	60.9	57.1
	High	0.0	62.2	46.9
	Very high	7.4	31.8	18.4
	<b>Overall error</b>	<b>1.9</b>	<b>54.2</b>	<b>43.9</b>
Whiteness	very low	42.9	2.4	8.2
	low	0.0	23.9	22.4
	High	0.0	78.4	59.2
	Very high	3.7	31.8	16.3
	<b>Overall error</b>	<b>11.6</b>	<b>34.1</b>	<b>26.5</b>

FP – False positive

FN - False negative

**Table 4.22** Classification results of sensory quality of cooked rice by SIMCA method using cooked rice spectra from FT-NIR spectrometer.

Sensory Quality	Model	Error rate (%)		
		FP	FN	Total
Adhesiveness	very low	4.4	92.5	52.0
	low	2.8	91.1	69.6
	High	7.1	96.3	87.8
	Very high	3.3	94.1	75.7
	<b>Overall error</b>	<b>4.4</b>	<b>93.5</b>	<b>71.3</b>
Hardness	very low	4.8	89.8	78.4
	low	4.3	98.4	84.5
	High	5.4	87.5	67.6
	Very high	4.4	93.8	53.4
	<b>Overall error</b>	<b>4.7</b>	<b>92.4</b>	<b>70.9</b>
Dryness	very low	0.0	90.3	75.7
	low	0.0	100.0	86.5
	High	10.5	83.6	87.8
	Very high	6.1	93.9	96.6
	<b>Overall error</b>	<b>4.1</b>	<b>92.0</b>	<b>86.7</b>
Stickiness	very low	3.6	89.3	52.0
	low	6.7	88.3	58.1
	High	0.0	96.2	85.1
	Very high	0.0	94.4	68.9
	<b>Overall error</b>	<b>2.6</b>	<b>92.1</b>	<b>66.0</b>
Aroma	very low	4.5	93.3	76.4
	low	0.0	96.9	85.8
	High	6.9	93.8	73.0
	Very high	1.3	91.9	39.2
	<b>Overall error</b>	<b>3.2</b>	<b>94.0</b>	<b>68.6</b>
Whiteness	very low	9.5	78.0	63.5
	low	0.0	91.5	79.7
	High	6.5	90.7	66.9
	Very high	9.0	86.9	39.9
	<b>Overall error</b>	<b>6.2</b>	<b>86.8</b>	<b>62.5</b>

FP – False positive

FN - False negative

**Table 4.23** Classification results of sensory quality of cooked rice by PLS-DA method using milled rice spectra from FT-NIR spectrometer.

Sensory Quality	Model	Error rate (%)		
		FP	FN	Total
Adhesiveness	very low	13.6	22.2	18.4
	low	50.0	13.5	22.4
	High	0.0	4.5	4.1
	Very high	20.0	0.0	4.1
	<b>Overall error</b>	<b>20.9</b>	<b>10.1</b>	<b>12.2</b>
Hardness	very low	16.7	4.7	6.1
	low	37.5	2.4	8.2
	High	75.0	18.9	32.7
	Very high	13.0	26.9	20.4
	<b>Overall error</b>	<b>35.6</b>	<b>13.2</b>	<b>16.8</b>
Dryness	very low	12.5	0.0	2.0
	low	16.7	2.3	4.1
	High	14.3	51.4	40.8
	Very high	19.0	32.1	26.5
	<b>Overall error</b>	<b>15.6</b>	<b>21.5</b>	<b>18.4</b>
Stickiness	very low	22.2	9.7	14.3
	low	37.5	12.1	20.4
	High	0.0	4.4	4.1
	Very high	18.2	0.0	4.1
	<b>Overall error</b>	<b>19.5</b>	<b>6.6</b>	<b>10.7</b>
Aroma	very low	0.0	7.1	6.1
	low	100.0	0.0	6.1
	High	66.7	0.0	16.3
	Very high	3.7	22.7	12.2
	<b>Overall error</b>	<b>42.6</b>	<b>7.5</b>	<b>10.2</b>
Whiteness	very low	0.0	2.4	2.0
	low	100.0	0.0	6.1
	High	58.3	5.4	18.4
	Very high	0.0	31.8	14.3
	<b>Overall error</b>	<b>39.6</b>	<b>9.9</b>	<b>10.2</b>

FP – False positive

FN - False negative

**Table 4.24** Classification results of sensory quality of cooked rice by PLS-DA method using cooked rice spectra from FT-NIR spectrometer.

Sensory Quality	Model	Error rate (%)		
		FP	FN	Total
Adhesiveness	very low	20.6	33.8	27.7
	low	97.2	0.0	23.6
	High	21.4	0.0	2.0
	Very high	10.0	0.0	2.0
	<b>Overall error</b>	<b>37.3</b>	<b>8.4</b>	<b>13.9</b>
Hardness	very low	33.3	3.9	8.1
	low	78.3	0.8	12.8
	High	100.0	0.0	25.0
	Very high	29.4	34.6	32.4
	<b>Overall error</b>	<b>60.3</b>	<b>9.8</b>	<b>19.6</b>
Dryness	very low	4.2	5.6	5.4
	low	95.0	0.0	12.8
	High	94.7	0.0	24.3
	Very high	36.4	32.9	34.5
	<b>Overall error</b>	<b>57.6</b>	<b>9.6</b>	<b>19.3</b>
Stickiness	very low	100.0	0.0	14.9
	low	100.0	0.0	8.1
	High	100.0	0.0	20.9
	Very high	35.1	23.1	27.7
	<b>Overall error</b>	<b>83.8</b>	<b>5.8</b>	<b>17.9</b>
Aroma	very low	18.2	5.8	7.4
	low	100.0	0.0	7.4
	High	82.8	3.5	18.9
	Very high	10.0	32.3	18.9
	<b>Overall error</b>	<b>52.7</b>	<b>10.4</b>	<b>13.2</b>
Whiteness	very low	9.5	3.4	4.1
	low	100.0	0.0	6.1
	High	100.0	0.9	21.6
	Very high	6.4	34.4	17.6
	<b>Overall error</b>	<b>54.0</b>	<b>9.7</b>	<b>12.3</b>

FP – False positive

FN - False negative

#### **4.6.3 Classification results of sensory quality of cooked rice by SIMCA method using milled rice and cooked rice spectra from short wavelength diode array**

Classification results of sensory qualities of cooked rice by SIMCA method using milled rice spectra scanned by short wavelength diode array are shown in table 4.25. Classification models of all sensory quality showed the overall error of false positive from 2.8 to 12.6%. The results of overall error of false negative were between 25.7 and 48.4%. This finding point indicated that these classification models could be better for self-classification of sensory quality but not for other group classification. The total error of all classification models were more than 10% (21.4 – 38.3%) therefore, the models were not applicable.

Classification results of sensory qualities of cooked rice by SIMCA method using cooked rice spectra scanned by FOA-NIR spectrometer are shown in table 4.26. Classification models of sensory quality showed the overall error of false positive between 2.7 and 26.7% but those of false negative were more than 51.3%. However, the total error of all classification models was too high more than 10% (39.7 – 52.0%). Therefore, the models were not applicable.

#### **4.6.3 Classification results of sensory quality of cooked rice by PLS-DA method using milled rice and cooked rice spectra from short wavelength diode array**

Classification results of sensory qualities of cooked rice by PLS-DA method using milled rice spectra scanned by short wavelength diode array are shown in table 4.27. Classification models of all sensory quality showed the overall error of false positive from 27.7 to 52.8%. The results of overall error of false negative were between 4.0 and 13.7%. Most classification models except aroma showed the overall

error of false positive more than the overall error of false negative. This finding point indicated that these classification models could be better for other group classification than self-classification. The total error of adhesiveness, dryness and stickiness was less than 10% (8.2 – 9.7%) and those of other classification models were more than 10% (11.2 to 16.8). Therefore, the model of adhesiveness, dryness and stickiness was fairly applicable and the other models were not applicable.

Classification results of sensory qualities of cooked rice by PLS-DA method using milled rice spectra scanned by short wavelength diode array are shown in table 4.28. Classification models of all sensory quality showed the overall error of false positive from 30.7 to 50.1%. The results of overall error of false negative were between 4.7 and 10.8%. This finding point indicated that these classification models could be better for other group classification than self-classification. The total error of adhesiveness and aroma was less than 10% (9.1 and 8.1%, respectively) and other classification models, were more than 10% (10.8 – 13.5%). Therefore, the model of adhesiveness and aroma was fairly applicable while the other models were not applicable.

**Table 4.25** Classification results of sensory quality of cooked rice by SIMCA method using milled rice spectra from short wavelength diode array.

Sensory Quality	Model	Error rate (%)		
		FP	FN	Total
Adhesiveness	very low	9.1	29.6	20.4
	low	16.7	48.6	40.8
	High	0.0	62.2	57.1
	Very high	0.0	10.5	8.2
	<b>Overall error</b>	<b>6.4</b>	<b>37.8</b>	<b>31.6</b>
Hardness	very low	0.0	14.0	12.2
	low	12.5	22.0	20.4
	High	8.3	51.4	40.8
	Very high	4.3	38.5	22.4
	<b>Overall error</b>	<b>6.3</b>	<b>31.4</b>	<b>24.0</b>
Dryness	very low	12.5	2.4	4.1
	low	0.0	30.2	26.5
	High	25.0	56.8	49.0
	Very high	13.0	46.2	30.6
	<b>Overall error</b>	<b>12.6</b>	<b>33.9</b>	<b>27.6</b>
Stickiness	very low	5.6	35.5	24.5
	low	5.6	58.1	38.8
	High	0.0	80.0	73.5
	Very high	0.0	20.0	16.3
	<b>Overall error</b>	<b>2.8</b>	<b>48.4</b>	<b>38.3</b>
Aroma	very low	0.0	9.3	8.2
	low	0.0	24.4	22.4
	High	8.3	51.4	40.8
	Very high	3.7	27.3	14.3
	<b>Overall error</b>	<b>3.0</b>	<b>28.1</b>	<b>21.4</b>
Whiteness	very low	16.7	4.7	6.1
	low	25.0	24.4	24.5
	High	0.0	67.4	59.2
	Very high	3.0	6.3	4.1
	<b>Overall error</b>	<b>11.2</b>	<b>25.7</b>	<b>23.5</b>

FP – False positive

FN - False negative

**Table 4.26** Classification results of sensory quality of cooked rice by SIMCA method using cooked rice spectra from short wavelength diode array.

Sensory Quality	Model	Error rate (%)		
		FP	FN	Total
Adhesiveness	very low	7.4	50.0	30.4
	low	2.8	78.6	60.1
	High	7.1	50.7	46.6
	Very high	3.3	32.2	26.4
	<b>Overall error</b>	<b>5.2</b>	<b>52.9</b>	<b>40.9</b>
Hardness	very low	0.0	52.3	45.3
	low	4.5	57.9	50.0
	High	2.6	73.6	55.4
	Very high	5.9	41.3	25.0
	<b>Overall error</b>	<b>3.3</b>	<b>56.3</b>	<b>43.9</b>
Dryness	very low	4.2	41.1	35.1
	low	70.0	72.7	72.3
	High	20.5	66.1	54.1
	Very high	12.3	73.5	46.6
	<b>Overall error</b>	<b>26.7</b>	<b>63.3</b>	<b>52.0</b>
Stickiness	very low	0.0	53.3	33.1
	low	4.2	69.0	48.0
	High	8.3	55.9	52.0
	Very high	15.6	28.4	25.7
	<b>Overall error</b>	<b>7.0</b>	<b>51.6</b>	<b>39.7</b>
Aroma	very low	4.5	40.5	35.1
	low	0.0	69.1	63.5
	High	0.0	79.3	62.2
	Very high	6.1	39.4	20.9
	<b>Overall error</b>	<b>2.7</b>	<b>57.1</b>	<b>45.4</b>
Whiteness	very low	13.6	27.0	25.0
	low	16.7	66.2	62.2
	High	5.9	75.4	59.5
	Very high	13.8	36.8	24.3
	<b>Overall error</b>	<b>12.5</b>	<b>51.3</b>	<b>42.7</b>

FP – False positive

FN - False negative

**Table 4.27** Classification results of sensory quality of cooked rice by PLS-DA method using milled rice spectra from short wavelength diode array.

Sensory Quality	Model	Error rate (%)		
		FP	FN	Total
Adhesiveness	very low	9.1	14.8	12.2
	low	41.7	2.7	12.2
	High	100.0	0.0	8.2
	Very high	27.3	0.0	6.1
	<b>Overall error</b>	<b>44.5</b>	<b>4.4</b>	<b>9.7</b>
Hardness	very low	0.0	4.7	4.1
	low	100.0	0.0	16.3
	High	25.0	8.1	12.2
	Very high	17.4	11.5	14.3
	<b>Overall error</b>	<b>35.6</b>	<b>6.1</b>	<b>11.7</b>
Dryness	very low	14.3	0.0	2.0
	low	50.0	0.0	6.1
	High	33.3	8.1	14.3
	Very high	13.0	7.7	10.2
	<b>Overall error</b>	<b>27.7</b>	<b>4.0</b>	<b>8.2</b>
Stickiness	very low	33.3	3.2	14.3
	low	6.3	15.2	12.2
	High	100.0	0.0	8.2
	Very high	18.2	0.0	4.1
	<b>Overall error</b>	<b>39.4</b>	<b>4.6</b>	<b>9.7</b>
Aroma	very low	0.0	9.3	8.2
	low	100.0	0.0	8.2
	High	100.0	0.0	24.5
	Very high	11.1	45.5	26.5
	<b>Overall error</b>	<b>52.8</b>	<b>13.7</b>	<b>16.8</b>
Whiteness	very low	16.7	4.7	6.1
	low	100.0	0.0	8.2
	High	58.3	2.7	16.3
	Very high	0.0	31.8	14.3
	<b>Overall error</b>	<b>43.8</b>	<b>9.8</b>	<b>11.2</b>

FP – False positive

FN - False negative

**Table 4.28** Classification results of sensory quality of cooked rice by PLS-DA method using cooked rice spectra from short wavelength diode array.

Sensory Quality	Model	Error rate (%)		
		FP	FN	Total
Adhesiveness	very low	4.4	13.8	9.5
	low	44.4	6.3	15.5
	High	100.0	0.0	9.5
	Very high	6.7	0.8	2.0
	<b>Overall error</b>	<b>38.9</b>	<b>5.2</b>	<b>9.1</b>
Hardness	very low	40.0	0.0	5.4
	low	68.2	2.4	12.2
	High	42.1	6.4	15.5
	Very high	13.2	7.5	10.1
	<b>Overall error</b>	<b>40.9</b>	<b>4.1</b>	<b>10.8</b>
Dryness	very low	4.2	2.4	2.7
	low	100.0	5.5	18.2
	High	38.5	7.3	15.5
	Very high	15.4	12.0	13.5
	<b>Overall error</b>	<b>39.5</b>	<b>6.8</b>	<b>12.5</b>
Stickiness	very low	17.9	23.9	21.6
	low	43.8	9.0	20.3
	High	100.0	0.0	8.1
	Very high	12.5	1.7	4.1
	<b>Overall error</b>	<b>43.5</b>	<b>8.7</b>	<b>13.5</b>
Aroma	very low	4.5	3.2	3.4
	low	100.0	0.0	8.1
	High	15.6	4.3	6.8
	Very high	2.4	28.8	14.2
	<b>Overall error</b>	<b>30.7</b>	<b>9.1</b>	<b>8.1</b>
Whiteness	very low	9.1	6.3	6.8
	low	100.0	0.0	8.1
	High	91.2	0.0	20.9
	Very high	0.0	36.8	16.9
	<b>Overall error</b>	<b>50.1</b>	<b>10.8</b>	<b>13.2</b>

FP – False positive

FN - False negative

#### **4.6.4 Classification results of sensory quality of cooked rice by SIMCA method using milled rice and cooked rice spectra from long wavelength diode array**

Classification results of sensory qualities of cooked rice by SIMCA method using milled rice spectra scanned by long wavelength diode array are shown in table 4.29. The classification models of hardness showed the overall error of false positive of 0 while the overall error of false positive of other sensory quality (adhesiveness, dryness, stickiness, aroma and whiteness) ranged 1.1 and 5.6%. On the other hand, the results of overall error of false negative were between 83.0 and 89.6%. This finding point indicated that these classification models just could be better used for self- classification of sensory quality but not for other group classification. However, the total error was more than 10% too high (61.5 – 68.8). Therefore, the models were not applicable.

Classification results of sensory qualities of cooked rice by SIMCA method using cooked rice spectra scanned by long wavelength diode array are shown in table 4.30. The similar result was obtained as the classification models using milled rice spectra. Classification models of sensory quality showed the overall error of false positive between 0.9 and 32.2% but the percentage of false negative results were more than 79.3%. However, the total error was more than 10% too high (59.8 – 66.6). Therefore, the models were not applicable.

#### **4.6.5 Classification results of sensory quality of cooked rice by PLS-DA method using milled rice and cooked rice spectra from long wavelength diode array**

Classification results of sensory qualities of milled rice by PLS-DA method using cooked rice spectra scanned by long wavelength diode array are shown in table 4.31. The overall error of false positive and false negative results were between

44.5 – 93.6% and 2.9 – 22.7%, respectively. The overall error rate of all sensory quality was more than 10% too high (18.9 – 31.1%). Therefore, the PLS-DA models were not applicable.

Classification results of sensory qualities of cooked rice by PLS-DA method using cooked rice spectra scanned by long wavelength diode array are shown in table 4.32. The overall error rate of false positive for every attribute was more than 66.0% and that of false negative was between 7.7 and 11.7%. The classification models showed the total overall error rate of 17.6 – 21.6%. These results indicated the models were not applicable.

**Table 4.29** Classification results of sensory quality of cooked rice by SIMCA method using milled rice spectra from long wavelength diode array.

Sensory Quality	Model	Error rate (%)		
		FP	FN	Total
Adhesiveness	very low	4.5	84.6	47.9
	low	0.0	86.1	64.6
	High	0.0	61.4	56.3
	Very high	0.0	100.0	79.2
	<b>Overall error</b>	<b>1.1</b>	<b>83.0</b>	<b>62.0</b>
Hardness	very low	0.0	97.6	85.4
	low	0.0	90.0	75.0
	High	0.0	88.9	66.7
	Very high	0.0	80.8	43.8
	<b>Overall error</b>	<b>0.0</b>	<b>89.3</b>	<b>67.7</b>
Dryness	very low	0.0	97.5	81.3
	low	0.0	92.9	81.3
	High	0.0	83.3	62.5
	Very high	4.5	84.6	47.9
	<b>Overall error</b>	<b>1.1</b>	<b>89.6</b>	<b>68.2</b>
Stickiness	very low	11.1	83.3	56.3
	low	11.1	76.7	52.1
	High	0.0	68.2	62.5
	Very high	0.0	92.5	77.1
	<b>Overall error</b>	<b>5.6</b>	<b>80.2</b>	<b>62.0</b>
Aroma	very low	16.7	76.2	68.8
	low	0.0	81.8	75.0
	High	0.0	88.9	66.7
	Very high	0.0	77.3	35.4
	<b>Overall error</b>	<b>4.2</b>	<b>81.0</b>	<b>61.5</b>
Whiteness	very low	0.0	83.3	72.9
	low	0.0	95.5	87.5
	High	16.7	97.6	87.5
	Very high	3.1	75.0	27.1
	<b>Overall error</b>	<b>4.9</b>	<b>87.9</b>	<b>68.8</b>

FP – False positive

FN - False negative

**Table 4.30** Classification results of sensory quality of cooked rice by SIMCA method using cooked rice spectra from long wavelength diode array.

Sensory Quality	Model	Error rate (%)		
		FP	FN	Total
Adhesiveness	very low	0.0	91.3	49.3
	low	5.6	81.3	62.8
	High	14.3	64.2	59.5
	Very high	3.3	83.9	67.6
	<b>Overall error</b>	<b>5.8</b>	<b>80.1</b>	<b>59.8</b>
Hardness	very low	0.0	88.3	76.4
	low	9.1	65.1	56.8
	High	2.6	94.5	70.9
	Very high	1.5	95.0	52.0
	<b>Overall error</b>	<b>3.3</b>	<b>85.7</b>	<b>64.0</b>
Dryness	very low	4.2	95.2	80.4
	low	90.0	65.6	68.9
	High	0.0	89.9	66.2
	Very high	0.0	90.4	50.7
	<b>Overall error</b>	<b>23.5</b>	<b>85.3</b>	<b>66.6</b>
Stickiness	very low	3.6	96.7	61.5
	low	0.0	92.0	62.2
	High	0.0	60.3	55.4
	Very high	0.0	86.2	67.6
	<b>Overall error</b>	<b>0.9</b>	<b>83.8</b>	<b>61.7</b>
Aroma	very low	4.5	81.0	69.6
	low	0.0	63.2	58.1
	High	3.1	90.5	71.6
	Very high	0.0	86.4	38.5
	<b>Overall error</b>	<b>1.9</b>	<b>80.3</b>	<b>59.5</b>
Whiteness	very low	0.0	67.5	57.4
	low	25.0	62.5	59.5
	High	100.0	93.0	94.6
	Very high	3.8	94.1	45.3
	<b>Overall error</b>	<b>32.2</b>	<b>79.3</b>	<b>64.2</b>

FP – False positive

FN - False negative

**Table 4.31** Classification results of sensory quality of cooked rice by PLS-DA method using milled rice spectra from long wavelength diode array.

Sensory Quality	Model	Error rate (%)		
		FP	FN	Total
Adhesiveness	very low	68.2	34.6	49.0
	low	0.0	0.0	0.0
	High	100.0	0.0	8.2
	Very high	10.0	23.7	20.4
	<b>Overall error</b>	<b>44.5</b>	<b>14.6</b>	<b>19.4</b>
Hardness	very low	83.3	0.0	10.2
	low	100.0	0.0	16.3
	High	100.0	0.0	24.5
	Very high	40.9	11.5	24.5
	<b>Overall error</b>	<b>81.1</b>	<b>2.9</b>	<b>18.9</b>
Dryness	very low	87.5	0.0	14.3
	low	100.0	0.0	12.2
	High	100.0	0.0	24.5
	Very high	54.5	26.9	38.8
	<b>Overall error</b>	<b>85.5</b>	<b>6.7</b>	<b>22.4</b>
Stickiness	very low	94.4	23.3	49.0
	low	100.0	6.7	40.8
	High	100.0	4.8	12.2
	Very high	80.0	7.9	22.4
	<b>Overall error</b>	<b>93.6</b>	<b>10.7</b>	<b>31.1</b>
Aroma	very low	83.3	2.4	12.2
	low	100.0	0.0	8.2
	High	91.7	0.0	22.4
	Very high	0.0	72.7	32.7
	<b>Overall error</b>	<b>68.8</b>	<b>18.8</b>	<b>18.9</b>
Whiteness	very low	83.3	0.0	10.2
	low	100.0	0.0	8.2
	High	91.7	0.0	22.4
	Very high	38.5	90.9	61.2
	<b>Overall error</b>	<b>78.4</b>	<b>22.7</b>	<b>25.5</b>

FP – False positive

FN - False negative

**Table 4.32** Classification results of sensory quality of cooked rice by PLS-DA method using cooked rice spectra from long wavelength diode array.

Sensory Quality	Model	Error rate (%)		
		FP	FN	Total
Adhesiveness	very low	31.9	39.2	35.8
	low	100.0	0.0	25.7
	High	100.0	0.0	7.4
	Very high	30.0	2.5	8.1
	<b>Overall error</b>	<b>65.5</b>	<b>10.4</b>	<b>19.3</b>
Hardness	very low	95.0	0.0	12.8
	low	95.5	0.0	14.2
	High	100.0	0.0	25.7
	Very high	29.4	35.0	32.4
	<b>Overall error</b>	<b>80.0</b>	<b>8.8</b>	<b>21.3</b>
Dryness	very low	50.0	4.0	11.5
	low	100.0	0.0	13.5
	High	100.0	1.8	27.7
	Very high	35.4	32.5	33.8
	<b>Overall error</b>	<b>71.3</b>	<b>9.6</b>	<b>21.6</b>
Stickiness	very low	58.9	18.5	33.8
	low	77.1	9.0	31.1
	High	100.0	0.0	8.1
	Very high	28.1	3.4	8.8
	<b>Overall error</b>	<b>66.0</b>	<b>7.7</b>	<b>20.4</b>
Aroma	very low	77.3	1.6	12.8
	low	100.0	0.0	8.1
	High	90.6	1.7	20.9
	Very high	24.4	39.4	31.1
	<b>Overall error</b>	<b>73.1</b>	<b>10.7</b>	<b>18.2</b>
Whiteness	very low	50.0	4.8	11.5
	low	100.0	0.0	8.1
	High	100.0	0.9	23.6
	Very high	15.0	41.2	27.0
	<b>Overall error</b>	<b>66.3</b>	<b>11.7</b>	<b>17.6</b>

FP – False positive

FN - False negative

## Chapter 5

### CONCLUSION

#### 5.1 CONCLUSION

The correlation coefficient ( $r$ ) between sensory texture of cooked rice and instrumental texture of cooked rice were higher than 0.54. The high  $r$  values point to the fact that the texture properties measured by the texture instrument followed the method used in this study could imply the texture perception of human. The linear equations related the texture properties by instrument and by sensory test were calculated where the  $r$  were between 0.54 – 0.94.

Predictive models for rice quality have been generated using input from FT-NIR spectroscopy of milled rice and cooked rice. Models of all sensory qualities showed the high predictive performance (for milled rice, showed  $R^2_{\text{val}}$  of adhesiveness = 0.920,  $R^2_{\text{val}}$  of hardness = 0.883,  $R^2_{\text{val}}$  of dryness = 0.876,  $R^2_{\text{val}}$  of stickiness = 0.900,  $R^2_{\text{val}}$  of aroma = 0.898, and  $R^2_{\text{val}}$  of whiteness = 0.880; for cooked rice showed  $R^2_{\text{val}}$  of adhesiveness = 0.690,  $R^2_{\text{val}}$  of hardness = 0.690,  $R^2_{\text{val}}$  of dryness = 0.680,  $R^2_{\text{val}}$  of stickiness = 0.710,  $R^2_{\text{val}}$  of aroma = 0.670, and  $R^2_{\text{val}}$  of whiteness = 0.650). The  $R^2_{\text{val}}$  was 0.66-0.81 indicating that the model is OK for screening and some other “approximate” calibrations; 0.83-0.90 indicated that the model can be usable with caution for most applications, including research and 0.92-0.96 indicated that the model can be usable in most applications, including quality assurance. The FT-NIR spectroscopic methods could be used to evaluate some sensory quality indices of milled rice and cooked rice. The SEP/SD values of adhesiveness, hardness, dryness, stickiness, aroma and whiteness were 2, 2, 1, 1, 1, and 1 respectively. Cooked rice model showed the SEP/SD values more than 2. (i.e., adhesiveness = 3, hardness = 3, dryness = 3, stickiness = 2, aroma = 4 and whiteness = 2). The some(The model? Some model? The same model?) model of milled rice and cooked rice were a good model (for milled rice was dryness, stickiness, aroma and whiteness and cooked rice was stickiness and whiteness) because the SEP/SD < 1.0, 1.0–2.0 a good model, 2.1–2.4 a fair model, 2.5–3.0 a poor model. In addition, by regression

coefficient plot, it was clear that the water content of the rice samples was not related to the sensory quality of the cooked rice. However, it was clearly apparent that starch as the main chemical component of rice was the dominant factor.

SW-NIR spectra of milled rice were recorded using short wavelength diode array, represent a potential alternative technique for the industrial evaluation of the sensory qualities of cooked rice. The sensory quality indices of cooked rice models yielded  $R^2_{\text{val}}$  between 0.823 and 0.918 and SEP/SD values lower than 2, which supported the hypothesis that these indices correlated well with the spectral properties of milled rice. The regression coefficient plot of texture properties was affected by starch and water. These findings indicated the potential application of NIR spectroscopy, a non-destructive method, for evaluating the sensory qualities of cooked rice at different random sampling stations for quality assurance and control at the rice quality improvement plant. In practice, this method based on VIS and SW-NIR spectroscopy could reduce the time, number of procedures, and costs previously inherent in the rice industry.

Prediction results for sensory quality of rice using spectra from long wavelength diode array of milled rice and cooked rice showed  $R^2_{\text{val}}$  between 0.194 and 0.544. These results indicated the rough screening application of long wavelength diode array for evaluating the sensory qualities of cooked rice. The inferior prediction results might be due to temperature of instrument which increased during samples scanning.

Classification results of sensory qualities of cooked rice by SIMCA method using milled rice and cooked rice spectra scanned by FT-NIR spectrometer showed the total error of all classification models were more than 10% (26.5 – 86.7%). Therefore the classification models were not applicable.

Classification results of sensory qualities of milled rice by PLS-DA method using milled rice and cooked rice spectra scanned by FT-NIR spectrometer showed the total error of all classification models were more than 10% (10.2 – 19.6%); therefore, the models were not applicable.

Classification results of the models for sensory qualities of cooked rice by SIMCA method using milled rice and cooked rice spectra scanned by short wavelength diode array showed the total error of all classification models were more

than 10% (21.4 – 52.0%). Therefore the classification models from spectra of cooked and milled rice were not applicable.

Classification results of the models for sensory qualities of cooked rice by PLS-DA method using milled and cooked rice spectra scanned by short wavelength diode array showed the total error of most classification models were more than 10%. For milled rice, the total error of adhesiveness, dryness and stickiness were less than 10% (8.2 – 9.7%) and those of other classification models were more than 10% (11.2 to 16.8). Therefore, the model of adhesiveness, dryness and stickiness was fairly applicable and the other models were not applicable. For cooked rice, the total error of adhesiveness and aroma were less than 10% (9.1 and 8.1%, respectively) and other classification model, were more than 10% (10.8 – 13.5%). Therefore, the model of adhesiveness and aroma was fairly applicable while the other models were not applicable.

Classification results of the models for sensory qualities of cooked rice by SIMCA method using milled rice and cooked rice spectra scanned by long wavelength diode array showed the total error between 59.8 and 68.8%. This indicated that these models could not be used for classification sensory quality of rice.

Classification results of the models for sensory qualities of cooked rice by PLS-DA method using milled rice and cooked rice spectra scanned by long wavelength diode array are shown showed the total error of 17.6 to 37.1%. This indicated that the PLS-DA classification models using both cooked rice spectra scanned by MICRONIR spectrometer were not applicable.

## 5.2 RECOMMENDATIONS

To further explore the possibility of near infrared spectroscopy in evaluation of sensory quality of rice in the rice quality improvement plant, future research should focus on:

1. Though the FT-NIR spectrometer and short wavelength diode array could be used to evaluate sensory quality of rice alternatively to trained panelist sensory method, the long wavelength diode array instrument could not which might be due

to its increased temperature during long scanning period. Therefore the temperature should be controlled.

2. More types of milled rice should be included in NIR spectroscopy models.
3. The relationship between sensory properties and instrumental properties of cooked rice should be studied.

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APPENDIX A  
Published paper



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## EATING QUALITY OF COOKED RICE DETERMINATION USING FOURIER TRANSFORM NEAR INFRARED SPECTROSCOPY

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The goal of this research was to study the relationship between the eating quality of cooked rice and near infrared spectra measured by a Fourier Transform near infrared (FT-NIR) Spectrometer. Samples of milled: parboiled rice, white rice, new Jasmine rice (harvested in 2012) and aged Jasmine rice (harvested in 2006 or during the period 2007–2011) were used in this study. The eating quality of the cooked rice, i.e., adhesiveness, hardness, dryness, whiteness and aroma, were evaluated by trained sensory panelists. FT-NIR spectroscopy models for predicting the eating quality of cooked rice were established using the partial least squares regression. Among the eating quality, the stickiness model indicated its highest prediction ability (i.e.,  $R_{\text{val}}^2 = 0.71$ ; RMSEP = 0.65; Bias = 0.00; RPD = 1.87) and SEP/SD of 2. In addition, it was clear that the water content did not affect the eating quality of cooked rice, rather the main chemical component implicated was starch.

*Keywords:* Rice; FT-NIR spectroscopy; eating quality.

### 1. Introduction

Rice (*Oryza sativa* L) is one of the principle foods consumed by human around the world especially in Asia. Important rice products include milled rice and parboiled rice. Parboiled rice is unprocessed rice that has undergone partial steaming. In contrast,

milled rice is produced by removing the husk, bran layer and the germ.

Consumer appreciation of rice depends upon the quality of the cooked rice product. The determination of the eating quality of cooked rice is closely monitored by the rice processing and milling

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industry. During the rice production process sampling is conducted to determine the six key eating quality attributes: adhesiveness, hardness, dryness, stickiness, aroma and whiteness. At present the eating quality of samples are determined using a descriptive analytical sensory analysis by trained human panelists. Unfortunately, sensory based analysis methods are slow, complex and lead to the destruction of the test objects. Therefore, a novel analytical method that is fast, easy and non-destructive for evaluating eating quality of rice is highly sought after within the rice processing industry.

Near infrared (NIR) spectroscopy is a non-destructive method that has been applied to evaluate a variety of properties of agricultural products and foods. NIR spectroscopy is efficient because it dramatically reduces the time needed for experimental analysis, thereby leading to lower overall costs. Researchers have studied the application of NIR spectroscopy to analyze the amylose content, protein content, and lipid content of rice, with satisfactory outcomes.<sup>1-4</sup> Researchers also reported the use of NIR spectroscopy to predict the sensory characteristics of foods such as virgin olive oil,<sup>5</sup> apple,<sup>6</sup> table grape,<sup>7</sup> lamb meat<sup>8</sup> and beef steaks.<sup>9</sup>

In recent years, methods relying on Fourier transform near-infrared (FT-NIR) spectroscopy have been developed to overcome limitations associated with the NIR spectroscopic instrument. FT-NIR records the intensity of absorbance across the entire spectrum as a function of the optical path differences (OPD) between two NIR beams in an interferometer.<sup>10</sup> The two beams are created by splitting the measurement beam, i.e., the beam that is transmitted through or reflected from the specimen.<sup>10</sup> One split beam travels over a different optical path length, via a moving mirror, and is recombined with the second beam to create an interference signal.<sup>10</sup> The total interference signal results from the mirror traveling through a range of wavelengths and is transformed to spectral components via a fast Fourier transform.<sup>10</sup> The FT-NIR spectrometer has a number of advantages over the conventional grating NIR spectrometer including; (1) higher signal to-noise ratios, (2) extremely high resolutions and (3) fast and accurate frequency determinations.<sup>11</sup> FT-NIR spectroscopy was successfully applied to analyze properties of rice such as the lipid content of milled rice (long, medium and

short grains)<sup>12</sup> and the optimal cooking time of rice.<sup>13</sup> However, to date FT-NIR spectroscopy has not been used to evaluate the eating quality of rice where the reference method was the sensory approach.

The goal of this research was to study the relationship between the eating quality of cooked rice and its near infrared spectral data measured using an FT-NIR spectrometer. The results from this FT-NIR spectroscopy study could prove useful in real world applications associated with the rice processing and marketing.

## 2. Materials and Methods

### 2.1. Determination of criteria of eating quality indices

Samples of five different types of milled rice, which cover the full of eating quality range, were prepared by the quality control section of a rice processing plant (C.P. Rice Co., Ltd., Thailand) and delivered to the Near Infrared Spectroscopy Research Center for Agricultural Product and Food at King Mongkut's Institute of Technology Ladkrabang, Bangkok, Thailand. In this research, we assess the principal eating quality indices of rice which encompass adhesiveness, hardness, stickiness, dryness, whiteness and aroma, all of which have been used by the rice quality processing plant.

The five types of the rice samples were cooked according to the water to rice ratios suggested by the processing plant. A team of panelists consisting of five males and five females were each served with the five types of cooked rice. The panelists were allowed to confer among one another before collectively deciding on the score to give to each of the samples. The scoring system for adhesiveness, hardness, dryness and stickiness are on a scale from 1 to 5, where 1 denotes the maximum level and 5 the lowest level. For aroma and whiteness, the scoring is reversed, where 1 represents the lowest level while 5 represents the maximum level. All scores provided by panelists were integers only (i.e., 1, 2, 3, 4 or 5).

### 2.2. The training of sensory panelists

The 10-member panelists were firstly trained using multiple training sessions to acquaint themselves with the scoring criteria needed to perform the rice

sensory evaluation in a consistent fashion. Each panelist was served with a scoop of cooked rice of five different types. Upon being served with the cooked rice, the panelists first smelled the samples so as to perform the aromatic assessment. The panelists were trained to assess the cooked rice adhesiveness by either shaking a portion in a small closed-lid plastic container or by gently scraping the top surface of the rice samples with a small plastic spoon, or both. For hardness and stickiness, the panelists were asked to chew all the five types of cooked rice and summarize their conclusions using concise words and scores related to their hardness (e.g., extremely hard (1), hard (2), etc.) and stickiness (e.g., extremely sticky (1), sticky (2), etc.). The panelists were also trained to determine sample dryness by visually observing the moistness of the cooked rice. Whiteness was visually assessed. These eating quality criteria were then used in the subsequent scoring of the samples in the experimental stage.

The sensory evaluation of each of the five rice types were performed in duplicate. The training session continued until the 10 participating panelists were skilled at the quality assessment process. It should be noted that before each assessment, the panelists would be served with drinking water to wash out the aftertaste and rice residues from the previous assessment. Note the cooked rice samples in the training sessions were of different lots from those used in the main experimental stage.

### 2.3. Rice samples

The 250 samples of milled rice were received from the same rice quality improvement plant. The rice samples were randomly garnered from three locations inside the plant premises, i.e., from the raw material receiving station, behind the color sorter machine, and under the storage bin. These three locations were selected for sample collection since they are the sites at which rice samples are typically collected for the internal quality inspection. The samples of milled rice were of parboiled rice, white rice, new Jasmine rice (harvested in 2012) and aged Jasmine rice (harvested in 2006 or during 2007–2011). A total of 51, 23, 12 and 164 samples of each variety were selected, respectively. The weight of each milled rice sample was approximately 200 g, which was stored in a plastic zipper bag until the experimental analyses were performed.

### 2.4. Cooked rice preparation

Home electronic rice cookers (RC-10 MM, Toshiba, Thailand) were used to cook 250 samples of five types of milled rice, each weighing 200 g. The five rice types were cooked according to the different water to rice ratios recommended by the plant, i.e., 2.5:1 for parboiled rice; 1.6:1 for white rice; 1:1 for new Jasmine rice harvested in 2012 and 1.2:1 and 1.4:1 respectively for aged Jasmine rice harvested in 2007–2011 and in 2006. The rice was considered fully cooked once the rice cooker automatically switched to the warm mode. The cooked rice was gently but thoroughly mixed using a plastic ladle before transferring to the small closed-lid plastic containers for sensory evaluation. The cooked rice samples were then presented to the sensory panelists for evaluation.

### 2.5. Sample scanning

Each cooked rice sample was scooped into a Petri dish with the following dimensions (53.5 mm diameter and 15 mm height). Three replicates were assessed per sample. The NIR spectrum of the cooked rice samples were measured using an FT-NIR spectrometer (MPA, Bruker Ltd., Germany) in reflection mode between 12,500–4000  $\text{cm}^{-1}$  (800–2500 nm) at a resolution of 8  $\text{cm}^{-1}$ . An average spectrum for each sample was obtained from 64 separate scans recorded in absorption mode ( $\log 1/R$ ).

### 2.6. Sensory evaluation

The trained sensory panelists of five males and five females were asked to separately score each sample on a scale of 1–5 for all six eating quality indices. As discussed previously, 5 denotes the minimum level and 1 the maximum level, for adhesiveness, hardness, dryness and stickiness. This scale is reversed for the whiteness and aroma.

The scores of the eating quality indices given by the panelists were allowed to contain only one decimal point. The evaluation of the 250 cooked rice samples followed the same procedures established during the training stage. Each sample was assigned a three-digit random number. About 8–10 samples were served to the panelist at a time. Moreover, after each sample evaluation, the panelists were served with drinking water to wash out any aftertaste or rice residue remaining from the previous assessment. Prior to each sensory evaluation day,

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the panelists were re-trained on the sensory evaluation criteria to ensure evaluation consistency.

### 2.7. Spectrum pre-treatment and NIR spectroscopy model establishment

The NIR spectroscopy models for predicting the eating quality of cooked rice were established using partial least squares regression (PLS). The multivariate analysis software (OPUS, v. 7.0, Bruker, Germany) was used in both spectrum preprocessing and model creation. About 80% of the experimental samples (i.e., 601 samples of 750 analyzed) were used in the calibration group while the remainder (149 samples) was placed in the validation group. The calibration group was used for the model creation. The model error was then calculated using the full cross-validation method. The same model was then used to predict the samples in the validation group. The optimum wavenumber range and preprocessing method were selected using the default optimization procedures of the software. The predictive capability of the models were assessed using the coefficient of determination of the calibration and validation groups ( $R^2_{\text{cal}}$  and  $R^2_{\text{val}}$ , respectively), root mean square error of estimation (RMSEE), root mean square error of prediction (RMSEP), bias, and ratio of standard deviation of validation data to RMSEP (RPD).

### 2.8. Overall precision test

The overall precision or reproducibility was assessed by conducting tests on 27 pairs of blind duplicates from the experiments on the 750 samples. The reproducibility is defined as the standard deviation (SD) of the differences between the values of the blind duplicates. In addition, another 18 rice

samples were selected as a new set of duplicates (27 pairs) to determine the repeatability of the reference tests. This is defined as the SD of the differences between the values of these duplicates. The ratio of standard error of prediction (SEP) to the SD from the reproducibility test, SEP/SD, was then computed to evaluate the predictive capability of all the calibration models, where SEP/SD < 1.0 represents an excellent NIR calibration model; SEP/SD = 1.0 to 2.1 a good NIR calibration model; SEP/SD = 2.1 to 2.4 a fair NIR calibration model; SEP/SD = 2.5 to 3.0 a poor NIR calibration model and SEP/SD > 3.0 an unreliable NIR calibration model (Tony, Pietronittonio, Caltest, USA, personal communication).

## 3. Result and Discussion

### 3.1. NIR spectroscopy model

The mean, SD, maximum (Max), and minimum (Min) values of the eating quality of cooked rice for calibration and validation groups are shown in Table 1. The range of stickiness values was widest, i.e., 1.0 to 5.0., whereas the hardness range was narrowest, 1.3 to 4.7. The range of adhesiveness was equal to those of aroma, and whiteness (1.0 to 4.8), while the dryness range was between 1.2 and 4.7. The eating quality has an SD of validation between 1.09 and 1.23. The SD of adhesiveness and stickiness was highest at 1.23.

The prediction statistics for the PLS models to predict the eating quality of cooked rice are shown in Table 2. The optimum model for the prediction of adhesiveness was established from the raw spectra. The max-min normalization method led to the most predictive models for the prediction of hardness and stickiness. The models for dryness were derived from the spectra preprocessed using the first

Table 1. Minimum (Min), maximum (Max), mean, and SD of the six eating quality indices associated with cooked rice for the calibration and validation groups.

Parameter	Calibration					Prediction				
	Number of samples	Min	Max	Average	SD	Number of samples	Min	Max	Average	SD
Adhesiveness	601	1.0	4.8	2.4	1.10	149	1.0	4.8	3.0	1.23
Hardness	601	1.3	4.7	3.5	0.98	149	1.3	4.6	3.0	1.09
Dryness	601	1.2	4.7	3.5	0.98	149	1.2	4.5	3.0	1.11
Stickiness	601	1.0	5.0	2.5	1.08	149	1.0	5.0	3.1	1.23
Aroma	601	1.0	4.8	3.7	0.98	149	1.0	4.7	3.2	1.16
Whiteness	601	1.2	5.0	3.7	1.01	149	1.2	4.8	3.1	1.21

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Table 2. Statistics of prediction for the eating quality indices of cooked rice by PLS models.

Parameter	Pre-treatment	Wavenumber range (cm <sup>-1</sup> )	PC	$R_{cal}^2$	$R_{val}^2$	RMSEE	RMSEP	BIAS	RPD
Adhesiveness	Raw spectra	9403.8–7498.3, 6102–5446.3, 4605.4–4242.9	8	0.71	0.69	0.60	0.68	0.07	1.82
Hardness	Min–max normalization	9403.8–7498.3, 6102–4242.9	8	0.69	0.69	0.55	0.60	–0.01	1.80
Dryness	First derivative + Straight line subtraction	9403.8–7498.3, 6102–4597.7	8	0.69	0.68	0.55	0.62	–0.05	1.78
Stickiness	Min-max normalization	9403.8–7498.3, 6102–4597.7	8	0.71	0.71	0.59	0.65	0.00	1.87
Aroma	MSC	9403.8–4597.7	5	0.65	0.67	0.59	0.67	0.02	1.74
Whiteness	Vector normalization	8454.9–7498.3, 6012–4597.7	4	0.63	0.65	0.62	0.72	–0.06	1.69

PC — Optimal number of principal components,  
 $R_{cal}^2$  — The coefficient of determination of calibration set,  
 $R_{val}^2$  — The coefficient of determination of validation set,  
 RMSEE — Root mean square error of calibration,  
 RMSEP — Root mean square error of prediction, and  
 RPD — Ratio of SD of validation data to RMSEP.

derivative and straight line subtraction method. The models for the prediction of aroma and whiteness were created using spectra preprocessed using the multiplicative scattering correction (MSC) and vector normalization methods, respectively.

The prediction results of all eating quality indices showed  $R_{val}^2$  values between 0.65 and 0.71. The results for stickiness displayed the highest predictive capability of all the eating quality indices (i.e.,  $R_{val}^2 = 0.71$ ; RMSEP = 0.65; Bias = 0.00; RPD = 1.87). The  $R_{val}^2$  values for adhesiveness and hardness, indicative of texture properties of cooked rice, were both 0.69. The dryness and whiteness parameters, both of which are evaluated visually,

showed  $R_{val}^2$  values of 0.68 and 0.65, respectively. The aroma based models exhibited an  $R_{val}^2$  of 0.67.

The scatter plots (reference data ( $X$ ) with prediction data ( $Y$ )) of each of the eating quality indices are shown in Fig. 1. The regression coefficient plots of the optimum models for evaluating the eating quality of cooked rice are shown in Fig. 2.

The regression coefficient plots for the eating quality models do not show absorption bands associated with water (13,158, 10,309, 6897 and 5155 cm<sup>-1</sup>).<sup>14</sup> It was clear that the water content was not closely related to the eating quality of cooked rice. The regression coefficient plots of all eating quality indices show peaks around 8840–8655 cm<sup>-1</sup>

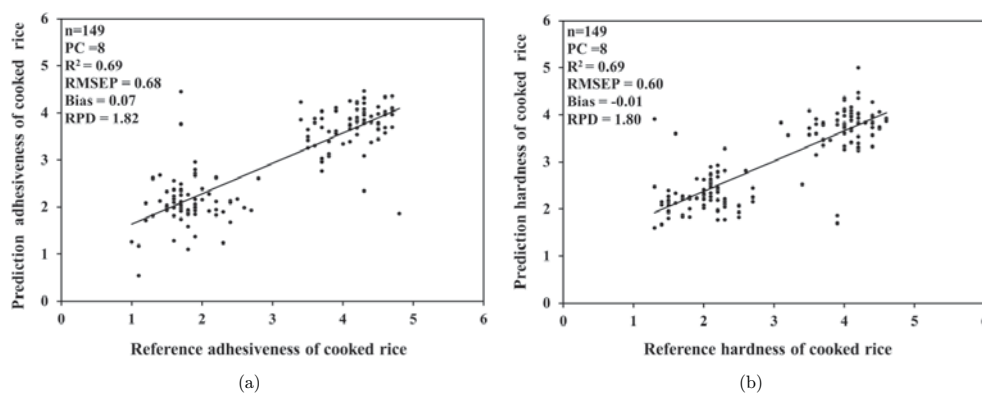


Fig. 1. Scatter plots of the reference data ( $X$ ) against the prediction data ( $Y$ ) for the six eating quality indices associated with cooked rice. These are: (a) Adhesiveness; (b) Hardness; (c) Dryness; (d) Stickiness; (e) Aroma and (f) Whiteness.

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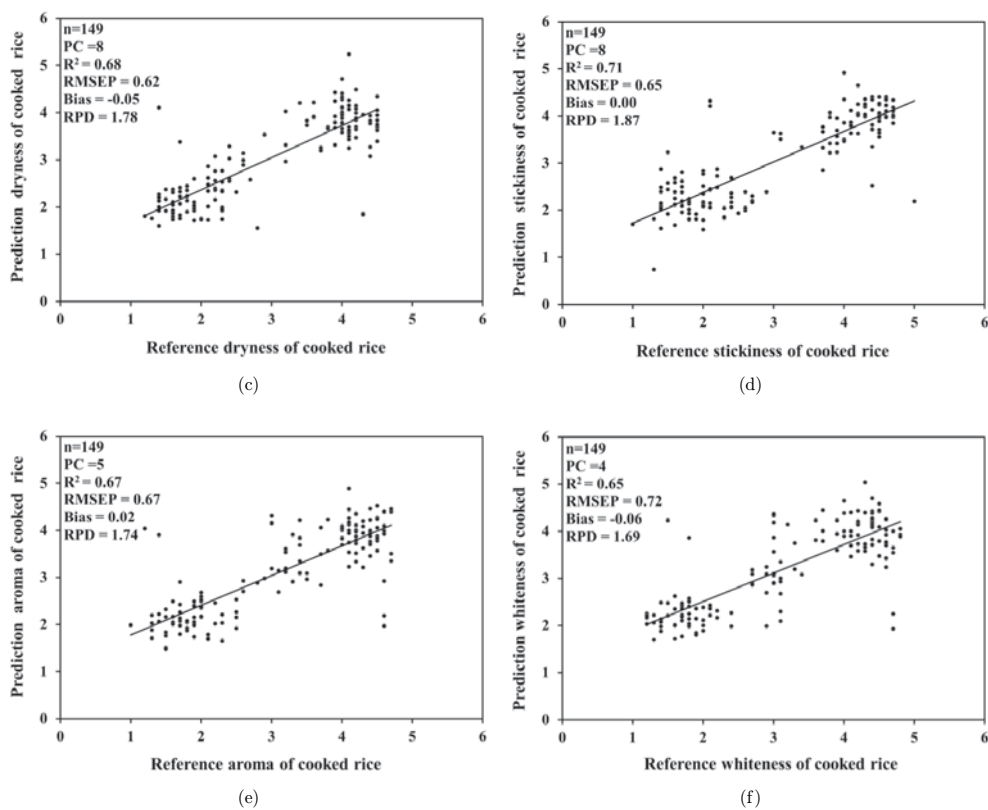


Fig. 1. (Continued)

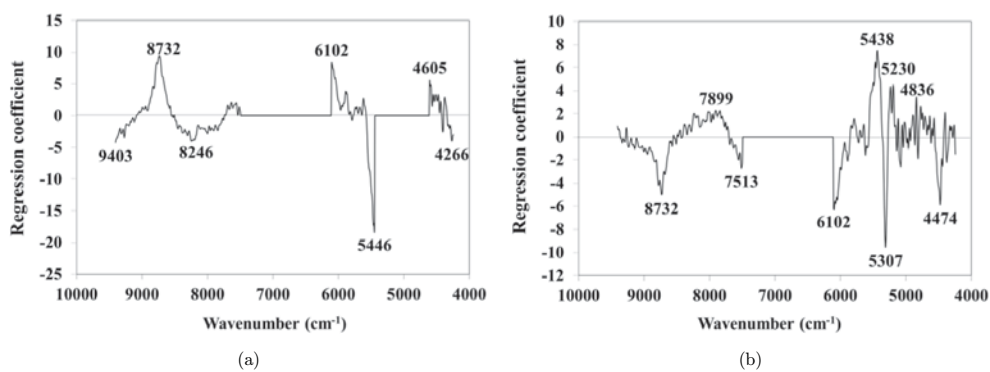


Fig. 2. Regression coefficient plots for the optimal PLS models associated with the six eating quality indices of cooked rice. (a) Adhesiveness; (b) Hardness; (c) Dryness; (d) Stickiness; (e) Aroma and (f) Whiteness.

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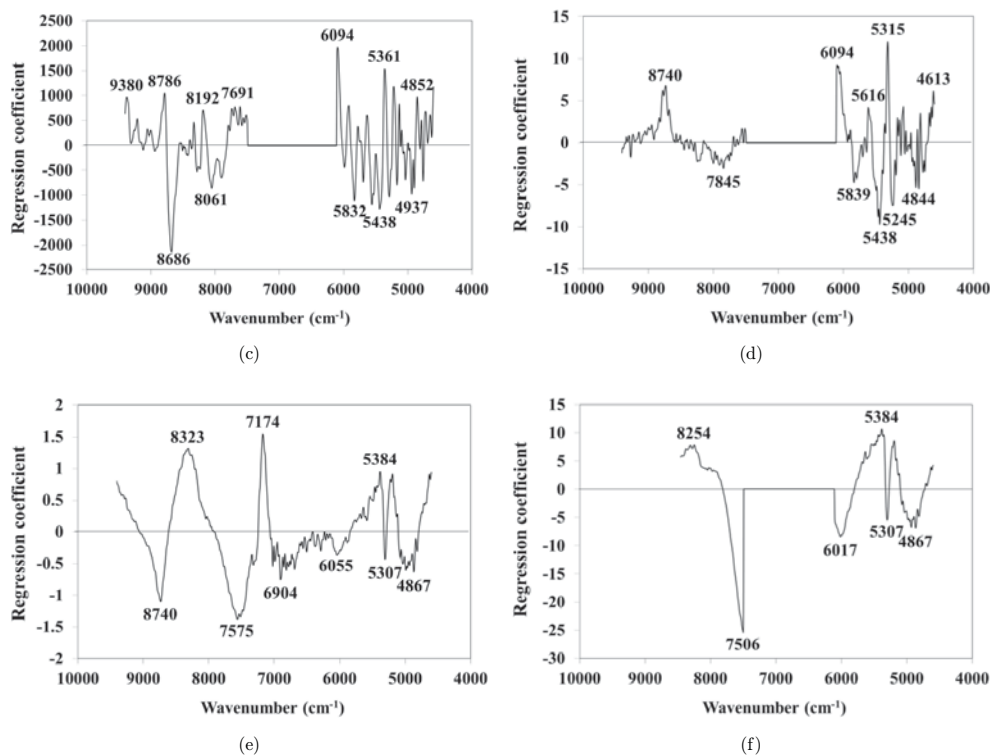


Fig. 2. (Continued)

(1131–1155 nm). The prominent features around 8840–8655  $\text{cm}^{-1}$  are the absorption peaks associated with the first overtone of C–H stretching of the starch (i.e., 8841–8658  $\text{cm}^{-1}$  or 1131–1155 nm).<sup>15</sup> It is perhaps obvious that carbohydrates are the main

Table 3. Means, SDs and ratios of SEP to SD (i.e., SEP/SD) for duplicate measurements (i.e., repeatability and reproducibility tests).

Quality	SEP	Repeatability test		Reproducibility test		
		Mean	SD	Mean	SD	SEP/SD
Adhesiveness	0.68	-0.1	0.2	0	0.2	3
Hardness	0.65	0	0.3	-0.1	0.2	3
Dryness	0.67	0.1	0.2	-0.1	0.2	3
Stickiness	0.70	0	0.2	0.1	0.3	2
Aroma	0.71	-0.2	0.2	0.2	0.2	4
Whiteness	0.75	-0.1	0.2	0	0.5	2

SEP — Standard error of prediction.

chemical component of rice that affects the eating quality. Table 3 shows means and standard deviations (SD) of the differences between the duplicates and ratios of standard error of prediction (SEP) to standard deviation (i.e., SEP/SD) of the overall precision tests. The SD values of the tests are the indication of precision of sensory panelist on evaluation of eating quality of cooked rice. The repeatability of all eating quality indices was less than 0.3 and the reproducibility was between 0.2–0.3 except for whiteness (0.5) which was 4–6% of maximum value (5). This indicated that the evaluation by panelist was fairly precise. The ratios of SEP/SD of reproducibility tests imply the performance of NIR spectroscopy model for practical application. The SEP/SD values of stickiness and whiteness model was 2 indicated that the model was good. The values for models of adhesiveness, hardness and dryness were 3 indicated the poor

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performance of the models. The model for aroma could not be recommended because the SEP/SD was 4.

#### 4. Conclusion

Predictive models for rice quality have been generated using input from FT-NIR spectroscopy. Models for stickiness and whiteness showed the highest predictive performance ( $R^2_{\text{val}} = 0.71$  and  $0.65$ ; RMSEP =  $0.65$  and  $0.72$  and; Bias =  $0.00$  and  $-0.06$ ; RPD =  $1.87$  and  $1.69$ , respectively) and SEP/SD of 2 and 2. This indicated that FT-NIR spectroscopic methods applied to cooked rice could be used to evaluate certain eating quality indices of cooked rice (i.e., stickiness and whiteness). In addition, it was clear that the water content of the rice samples was not related to the eating quality of the cooked rice. However, it was clearly apparent that starch as the main chemical component of rice, was the dominant factor.

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### Sensory Quality Evaluation of Rice Using Visible and Shortwave Near-Infrared Spectroscopy

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## Sensory Quality Evaluation of Rice Using Visible and Shortwave Near-Infrared Spectroscopy

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This research aimed to develop both visible and shortwave near-infrared spectroscopy to evaluate the sensory qualities of cooked rice. In this study, four different types of milled rice were used: parboiled, white, new Jasmine, and aged Jasmine. The sensory qualities of cooked rice (adhesiveness, hardness, stickiness, dryness, whiteness, and aroma) were evaluated by a trained sensory panel. The results demonstrated that these sensory attributes correlated with visible and shortwave near-infrared spectral data. Both visible and shortwave near-infrared spectroscopy models used for predicting the sensory qualities of cooked rice were established using partial least squares regression. All prediction results for sensory qualities showed a range of  $R^2_{val}$  between 0.837 and 0.918, with the highest found for aroma (0.918). The proposed models can be utilized in quality control by the rice industry.

*Keywords:* Rice, Sensory qualities, Visible, Shortwave, Near-infrared spectroscopy.

### INTRODUCTION

The improvement process for milled rice raw materials processed in quality improvement plants includes cleaning, destoning and polishing, grading by size, sorting by color, winnowing, silo storage, and packaging. Quality-improved rice products are developed according to formulas that are a combination of different categories of raw materials designed by the plant's research and development section. The various quality attributes of raw materials, such as moisture, instrument texture parameters, color, and tested sensory quality, were previously measured by traditional methods before the rice formulas were developed.

This article focuses on those sensory quality attributes required for quality assurance and control in the plant that affect consumer acceptance. During the improvement processes, rice is sampled for sensory quality attributes that are evaluated by trained panelists using sensory methods. Sensory qualities evaluated include adhesiveness, hardness, dryness, stickiness, aroma, and whiteness. In addition, sensory testing is carried out by panelists for sensory qualities on behalf of other

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product industries, such as those producing rice-shaped extruded kernels,<sup>[11]</sup> Japonica rice,<sup>[12]</sup> par-boiled rice,<sup>[13]</sup> whole meal bread,<sup>[14]</sup> mashed potato,<sup>[15]</sup> and cheese.<sup>[16]</sup> Because this system using trained specialists is complicated and requires destruction of the test objects, an alternative method of efficiently and conveniently evaluating the sensory qualities of rice is needed.

The use of near-infrared spectroscopy (NIRS) has now become widespread in industry because of its non-destructive nature, rapidity, low cost, and environmental friendliness. The NIRS method has been applied in the assessment of the sensory attributes of various agricultural products and foods. Several researchers have evaluated its use in regard to foodstuff parameters, such as the acidity, bitterness, flavor, cleanliness, body, and overall quality of coffee;<sup>[17]</sup> the taste, texture, and visual and olfactory attributes of and consumer preferences in regard to grapes;<sup>[18,9]</sup> the presence of holes, saltiness, buttery flavor, hardness, chewiness, creaminess, rancid flavor, pungency, and retronasal sensation in cheese;<sup>[10]</sup> the classification of extra virgin olive oil;<sup>[11]</sup> and the tenderness, juiciness, flavor intensity, abnormal flavor intensity, and overall acceptance of beef.<sup>[12]</sup> In 1998, shortwave near-infrared spectroscopy (SW-NIRS) was applied to evaluate the physical and chemical properties (i.e., head rice ratio, apparent density, whiteness, moisture, protein, and amylose contents) of Japonica rice.<sup>[13]</sup> A whole range of visible (VIS) and NIR (400–2500 nm) and long-wave (LW)-NIRS (1100–2500 nm) was applied to predict the texture of cooked rice varieties: 14 long-grain Thai;<sup>[14]</sup> 76 short-grain, Bengal, Cypress, Keybonnet, and Drew;<sup>[15]</sup> medium- and long-grain US;<sup>[16]</sup> and M401, Koshihikari, and Calrose<sup>[17]</sup>. The sensory qualities of Chinese indica rice in different areas (Hubei Province [mid-China], Zhejiang Province [East China], Sichuan Province [Southwest China], and Guangdong Province [South China]) were predicted using VIS and SW-NIRS,<sup>[18]</sup> but the wavelengths used were limited to 540, 640, and 970 nm. However, to date there has been no report published on the use of VIS and SW-NIRS in evaluating the sensory quality of cooked rice for quality assurance and control in a rice quality improvement plant where a wide range of varieties, and processed rice, are used. The raw material in such plants is milled rice, with various varieties including Jasmine, other white rice, and parboiled rice, which is derived from white rice.

The purpose of this research was to develop VIS and SW-NIRS models to determine the sensory qualities of cooked rice in regard to quality assurance. In addition, it was aimed at exploring the possibility of NIRS application to a wide range of rice varieties and processed milled rice. The hypothesis of the research was that the sensory qualities of cooked rice would correlate with the near-infrared spectral properties of milled rice.

## MATERIALS AND METHODS

### Determination of Criteria of Sensory Quality Indices

Methodology for determination of criteria of sensory quality indices followed Lapcharoensuk and Sirisomboon.<sup>[19]</sup> Samples of five types of milled rice were delivered to the Near Infrared Spectroscopy Research Center for Agricultural Product and Food at King Mongkut's Institute of Technology Ladkrabang, Bangkok, Thailand. These five varieties are the most representative of the range of sensory qualities as recommended by the quality control section of the plant, because they cover the range routinely processed by the plant. In this research, the sensory quality indices of rice encompassed adhesiveness, hardness, stickiness, dryness, whiteness, and aroma, all of which are used by the rice quality improvement plant for quality evaluation.

The rice samples were cooked according to the water:rice ratios suggested by the plant. A team of 10 panelists (5 males and 5 females) were each served with the five types of cooked rice. The panelists conferred among each other before collectively deciding on the score allocated to each of the sensory quality indices. Scoring was on a scale of 1–5 (whole integers only), where 1 denoted maximum level and 5 minimum, except in the case of whiteness and aroma where 1 denoted minimum and 5 maximum.

### Training of Panelists

All panelists were given multiple training sessions to acquaint them with the scoring criteria. Each panelist was served with one scoop each of all five cooked rice types, with aromatic assessment performed first. They were trained to assess adhesiveness by shaking small, covered plastic containers containing cooked rice and/or gently scraping the surface of the cooked samples with a small plastic spoon. In regard to the assessment of hardness and stickiness, the panelists were asked to chew the samples and then to explain their perceptions in simple words (e.g., for hardness, extremely hard, hard; for stickiness, extremely sticky, sticky). The panelists were trained to determine dryness and whiteness by visual observation. The overall sensory quality criteria were then used in the subsequent scoring at the experimental stage.

Two replicates of each type of cooked rice were used in each sensory evaluation training session, and these sessions continued until all participating panelists had become skilled at quality assessment. It should be noted that before each subsequent assessment, the panelists were served with drinking water to eliminate the aftertaste and rice residues from the previous assessment; moreover, the 5 cooked rice samples in the training sessions were of different types to those (i.e., 250 samples) used in the experimental stage. A flow diagram illustrating the sensory method used for evaluation of cooked rice is shown in Fig. 1.

### NIR Spectroscopy Experiment

#### *Rice samples*

All samples ( $n = 250$ ) of milled rice were received from the rice quality improvement plant (C.P. Rice Co., Ltd., Thailand). The rice samples were randomly garnered from three locations within the plant premises (the raw material receiving station, behind the color sorter machine, and under the storage bin). These three locations were selected for sample collection because they are the sites at which rice samples are typically collected for internal quality inspection. The samples of milled rice tested were parboiled (processed from white rice;  $n = 51$ ), white rice (from varieties other than Jasmine;  $n = 23$ ), new Jasmine (Khao Dawk Mali 105 variety;  $n = 12$ ; harvested in 2012 and sold to the plant where it was evaluated that year), and aged Jasmine ( $n = 164$ ; harvested either in 2006 or 2007–2011; "aged" means the rice was kept for at least 1 year postharvest before processing and evaluation). The weight of each milled rice sample was approximately 200 g, and the samples were retained in a plastic zipper bag until experimentation.

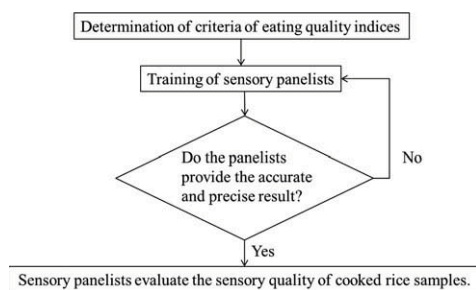


FIGURE 1 Flow diagram of sensory method for cooked rice.

### Sample scanning

Milled rice samples was transferred from zipper bags to glass beakers (250 ml). The NIR spectrum was measured with a portable FQA-NIR GUN unit (FANTEC, Japan; reflection mode, 600–1100 nm; Fig. 2). Scan results were recorded in absorption mode ( $\log 1/R$ ) for all samples. For every scan, the NIR GUN and sample were covered with black cloth to minimize interference by light.

### Cooked Rice Preparation

Home electronic rice cookers (RC-10 MM; Toshiba, Thailand) were used to cook the 250 rice samples, and cooking was performed according to the water:rice ratios recommended by the plant (i.e., 2.5:1 for parboiled rice, 1.6:1 for white rice, 1:1 for new Jasmine rice harvested in 2012, and 1.2:1 and 1.4:1, respectively, for aged Jasmine rice harvested in 2007–2011 and in 2006). After the rice was fully cooked and the rice cooker automatically switched to warm mode, the cooked rice was gently and thoroughly mixed using a plastic ladle before transfer to small, covered plastic containers for sensory evaluation. The cooked rice samples were then presented to the sensory panelists for evaluation.

### Sensory Evaluation

Sensory evaluation followed Lapcharoensuk and Sirisomboon.<sup>[19]</sup> Scoring by panelists was as described above. The scores of the sensory quality indices allocated by the panelists contained only one decimal point. Evaluation of the 250 cooked rice samples followed the same procedures as those at the training stage, with 8–10 samples being served to each panelist simultaneously. Prior to tasting



FIGURE 2 VIS-NIR spectral measurement by portable FQA-NIR GUN (FANTEC, Japan).

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by the panelists, each sample was assigned a random 3-digit number. Prior to the commencement of sensory evaluation each day, the panelists received retraining on the sensory evaluation criteria.

### Spectrum Pre-Treatment and NIR Spectroscopy Model Establishment

The NIR spectroscopy models for prediction of the sensory qualities of cooked rice were established using partial least squares regression (PLS). The software for multivariate analysis (Unscrambler, v. 9.8; CAMO, Norway) was used in both spectrum preprocessing and model creation. The NIR spectra were preprocessed by mean normalization, maximum normalization, range normalization, multiplicative scatter correction (MSC), first and second derivatives each with two smoothing points on both left and right sides and five smoothing points on both sides, baseline offset, de-trending (D), and standard normal variate (SNV) and de-trending (SNV+D). Further, 80% of the experimental samples (201 samples) were used in the calibration group and the remainder (49) in the validation group. The calibration group was used for model creation and the model error was calculated by the full cross-validation method. The same model was then used to predict the sensory qualities of rice in the validation group. The optimum model was selected by coefficients of determination of the calibration and validation groups ( $R^2_{\text{cal}}$  and  $R^2_{\text{val}}$ ), standard error of calibration (SEC), standard error of prediction (SEP), bias, and ratio of standard deviation of validation data to SEP (RPD). The flow diagram of NIR spectroscopy methodology is shown in Fig. 3.

### Overall Precision Test

The overall precision test is important for the precision of reference test—in this case, sensory evaluation by the panelists. The NIR model cannot evaluate properties more accurate than the reference test. The overall accuracy or reproducibility of reference tests was determined by conducting tests on nine pairs of blind duplicates alongside the regular experiment. The duplicates were selected from 18 out of the 250 rice samples, and reproducibility is the standard deviation (SD) of the differences between the values of the blind duplicates. In addition, another 18 rice samples were selected as a new set of duplicates (9 pairs) to determine the repeatability of the reference tests, which is the SD of the differences between the values of these duplicates. The ratio of SEP to the SD of the reproducibility test (SEP/SD) was computed to determine the evaluation capability of all the calibration models, where SEP/SD < 1.0 represents an excellent NIR model, 1.0–2.0 a good model, 2.1–2.4 a fair model, 2.5–3.0 a poor model, and >3.0 an unreliable model (Tony Pietroutonio, Caltest, USA, personal communication).

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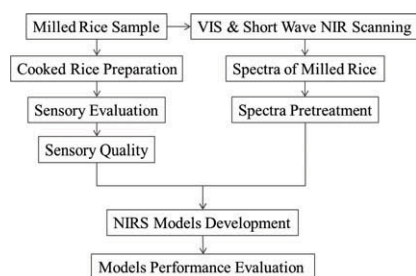


FIGURE 3 Flow diagram of NIR spectroscopy methodology.

## RESULTS AND DISCUSSION

## NIR Spectra of Milled Rice

The spectra of milled rice determined are shown in Fig. 4. In Fig. 4a, all five types of milled rice showed a high absorbance peak ( $\log 1/R$ ) of 950–1000 nm. However, spectral data may have baseline shift and/or baseline offset that led to dislocation of absorbances. These problems can be resolved by use of the second derivative method (Fig. 4b). The spectral plot shows obvious peaks at 744, 900, and 970 nm. Different types of milled rice affected the spectral level of the absorbance peak, especially at 970 nm, which is the absorbance band of water. The intensity of the peak was lowest for aged Jasmine rice (harvested in 2006) while those of other types tested were similar to each other, indicating the dryness of milled rice after long-term storage. The raw and second derivative spectra of milled rice at the three different stations are shown in Figs. 4c and 4d for aged Jasmine rice (harvested 2006–2011) and Figs. 4e and 4f for parboiled rice. By the second derivation, there was no difference in intensity level of the peaks illustrated, indicating that rice spectra were not affected by the different processing stations in the plant.

## NIR Spectroscopy Model

Mean, SD, maximum (Max), and minimum (Min) values of the sensory qualities of cooked rice for calibration and validation groups are shown in Table 1. The range of stickiness was widest (1.0–5.0), that of hardness was narrowest (1.3–4.7), that of adhesiveness, aroma, and whiteness was 1.0–4.8, and that of dryness was 1.2–4.7. The statistics regarding prediction of the sensory qualities of cooked rice by the PLS model are shown in Table 2. The general optimum models (i.e., adhesiveness, stickiness, aroma, and whiteness) were derived from spectra preprocessed by the range normalization method. The de-trending method was found to be the spectral preprocessed method that gave the optimum model for prediction of hardness. The models for prediction of dryness were created by the spectra that were preprocessed by the baseline offset method.

The prediction results of all sensory quality indices showed a range of  $R^2_{\text{val}}$  between 0.842 and 0.918. The results for aroma indicated the highest prediction ability ( $R^2_{\text{val}} = 0.918$ ; SEP = 0.284; bias = -0.043; RPD = 3.535), while  $R^2_{\text{val}}$  for adhesiveness, hardness, and stickiness—parameters indicating the textural properties of cooked rice—ranged from 0.842 to 0.904. Dryness and whiteness, which were evaluated visually, yielded  $R^2_{\text{val}}$  of 0.895 and 0.866, respectively. The scatter plots (reference data (X) with prediction data (Y)) of sensory qualities are shown in Fig. 5.

This is the first time that VIS and SW-NIR in the range of 600–1100 nm have been applied to evaluate the sensory qualities of rice where a wide variation matrix was applied to different varieties and processed rice. Our overall sensory quality prediction ( $R^2_{\text{val}} = 0.842$ – $0.918$ ) was better than that reported by Qingyun et al.<sup>[18]</sup> who recorded 0.31–0.77, likely because their prediction model was developed by multiple linear regression using wavelengths of 540, 640, and 970 nm only. In addition, our prediction of certain texture parameters was better than that obtained by Meullenet et al., where wavelengths of 400–2500 nm were used ( $R^2_{\text{val}}$  for hardness was 0.67);<sup>[15]</sup> and that reported by Champagne et al. ( $R^2_{\text{val}}$  for hardness and stickiness was 0.67 and 0.58, respectively).<sup>[16]</sup> The better prediction found in our work may have been due to the wider range of VIS and SW-NIR (600–1100 nm), the higher number of samples and the effectiveness, reduced complexity, and enhanced VIS and SW-NIR spectral correlation of our sensory method. Compared with the research findings of Srisawas et al. where longwave NIR (1100–2500 nm) was used<sup>[14]</sup> and those of Meullenet et al.,<sup>[15]</sup> we found a higher RPD (>2.5) for all prediction models. The SD in this study ranges from 1.00 to 1.15, which is nearly twice that reported in the literature cited above, thus yielding a higher RPD. Our higher SD value was obtained because of the wider variation matrix of different varieties and processed rice, whereas other studies included no processed products, such as parboiled rice, in their modeling.

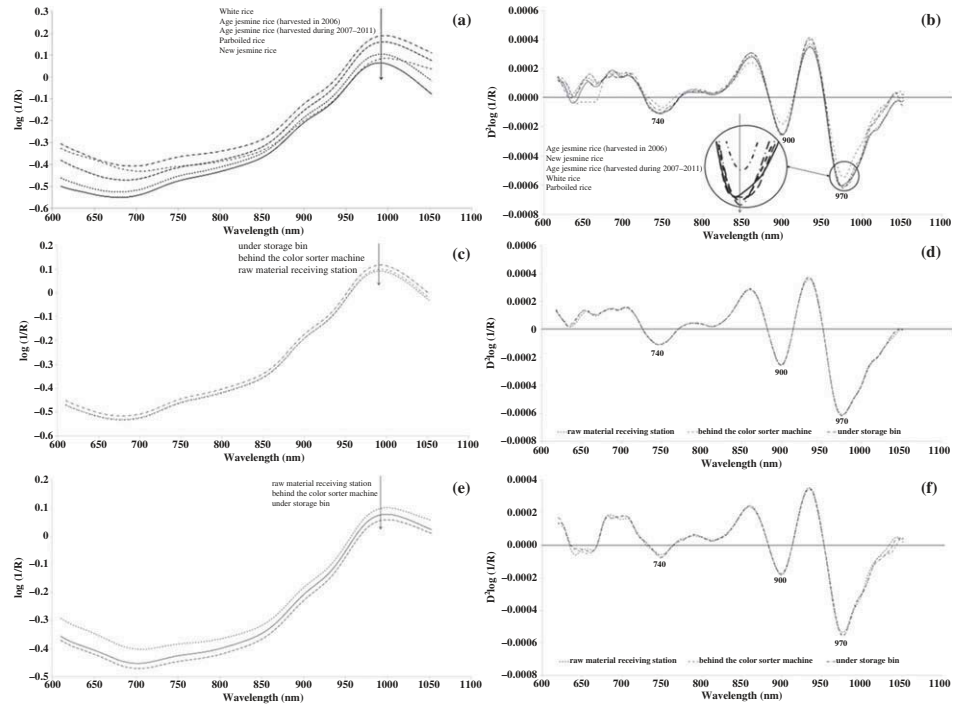


FIGURE 4 VIS-NIR spectra of milled rice. (a) Absorbance spectra ( $\log 1/R$ ) of different types of milled rice; (b) second derivative spectra ( $D^2\log 1/R$ ) of different types of milled rice; (c) absorbance spectra ( $\log 1/R$ ) of aged Jasmine rice (harvested 2007–2010) from different stations; (d) second derivative spectra ( $D^2\log 1/R$ ) of aged Jasmine rice (harvested 2007–2010) from different stations; (e) absorbance spectra ( $\log 1/R$ ) of parboiled rice from different stations; and (f) second derivative spectra ( $D^2\log 1/R$ ) of parboiled rice from different stations.

TABLE 1  
Minimum (Min), maximum (Max), mean, and SD of sensory quality indices of cooked rice in calibration and validation groups

	Calibration					Prediction				
	Number of sample	Min	Max	Average	SD	Number of sample	Min	Max	Average	SD
Adhesiveness	201	1.0	4.8	2.5	1.16	49	1.1	4.8	2.5	1.15
Hardness	201	1.3	4.7	3.4	1.03	49	1.5	4.7	3.4	1.01
Dryness	201	1.2	4.7	3.4	1.04	49	1.4	4.7	3.4	1.03
Stickiness	201	1.0	5.0	2.6	1.14	49	1.2	4.7	2.6	1.13
Aroma	201	1.0	4.8	3.6	1.05	49	1.4	4.7	3.6	1.00
Whiteness	201	1.2	5.0	3.6	1.09	49	1.4	4.9	3.7	1.05

TABLE 2  
Statistics of prediction of eating quality of cooked rice by PLS models

Parameter	Pre-treatment	PC	$R^2_{cal}$	$R^2_{val}$	SEC	SEP	BIAS	RPD
Adhesiveness	Range normalization	4	0.923	0.904	0.320	0.352	-0.051	3.259
Hardness	Detrending	3	0.875	0.842	0.364	0.399	0.023	2.524
Dryness	Baseline offset	3	0.892	0.895	0.343	0.327	0.062	3.144
Stickiness	Range normalization	4	0.913	0.891	0.337	0.368	0.053	3.068
Aroma	Range normalization	4	0.911	0.918	0.314	0.284	-0.043	3.535
Whiteness	Range normalization	4	0.928	0.866	0.292	0.370	0.102	2.838

PC: Optimal number of principal components;  $R^2_{cal}$ : the coefficient of determination of calibration set (full cross-validation);  $R^2_{val}$ : the coefficient of determination of validation set; SEC: standard error of calibration set (full cross-validation); SEP: standard error of prediction; RPD: ratio of standard deviation of validation data to standard error of prediction.

The regression coefficient plots of optimum models for evaluation of the sensory qualities of cooked rice are shown in Fig. 6. The peak regression coefficient appeared at around 746–750 nm for all sensory quality indices. The peaks at 746 and 747 nm show the frequency characteristic of the C–H stretch fourth overtone of CH<sub>2</sub> and O–H stretch third overtone of ArOH.<sup>[20]</sup> ArOH is the chemical structure of phenolic compounds in rice.<sup>[21]</sup> Adhesiveness, hardness, and stickiness, which mirror the textural qualities of cooked rice, showed the same peak of regression coefficient (935–990 nm). The prominent features at 938 nm are the absorption peaks associated with the C–H stretch third overtone of CH<sub>2</sub>.<sup>[20]</sup> In addition, the peak at 990 nm is the vibration band of the O–H stretch second overtone of starch.<sup>[20]</sup> The absorption peak of water (970 nm) is represented on a regression coefficient plot of texture attributes and dryness, indicating that the textural attributes were affected by starch and water. This is consistent with the work of Srisawas et al.,<sup>[14]</sup> who studied the application of long-wave NIRS in the evaluation of sensory textural attributes where the identical strong absorption peak of NIR spectra was at 1894 nm (O–H stretch + 2 × C–O stretch [1900 nm] [starch]) for both milled and cooked rice. The dryness characteristic of cooked rice was evaluated by visual observation.

#### Repeatability and Reproducibility

The means and SD of the differences between the duplicates and ratios of SEP to standard deviation (i.e., SEP/SD) of repeatability and reproducibility tests are shown in Table 3. The SEP/SD values for general sensory quality (adhesiveness, hardness, dryness, stickiness, and aroma) of cooked rice

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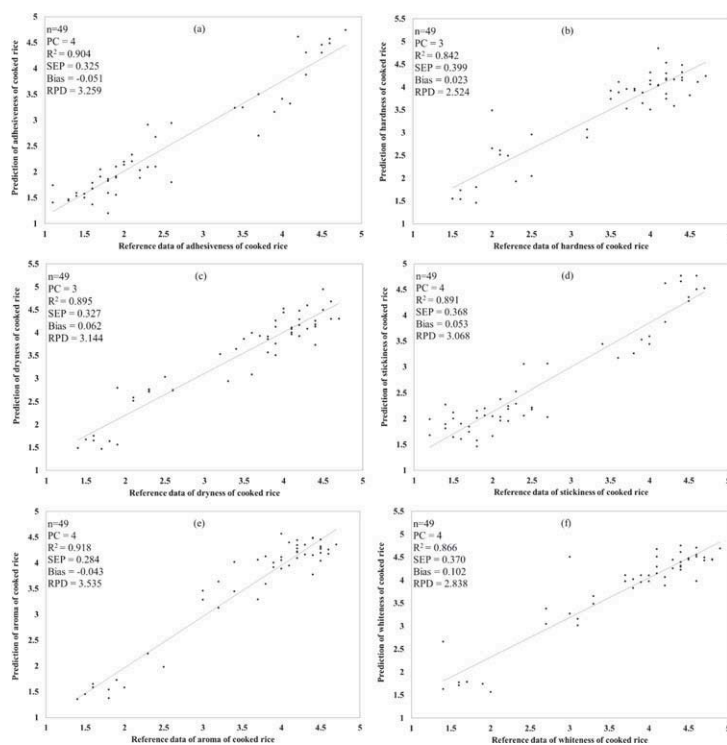


FIGURE 5 Scatter plots of reference data (X) with prediction data (Y) of sensory quality of cooked rice. (a) Adhesiveness; (b) hardness; (c) dryness; (d) stickiness; (e) aroma; and (f) whiteness.

were found to be less than 1. In addition, the prediction model of whiteness showed SEP/SD values of 1.3, indicating the robustness of the models and indicating that SW-NIR spectroscopy of milled rice can satisfactorily be applied to evaluate the sensory qualities of cooked rice.

## CONCLUSION

VIS and SW-NIR spectroscopy of milled rice represent a potential alternative technique for the industrial evaluation of the sensory qualities of cooked rice. The sensory quality indices of cooked rice models yielded  $R^2_{\text{val}}$  between 0.842 and 0.918, which supports the hypothesis that these indices correlate well with the spectral properties of milled rice. The findings in regard to aroma indicated its optimum prediction ability among the sensory quality indices, because the NIR spectroscopy model for aroma showed the highest  $R^2_{\text{val}}$  and lowest SEP values. The sensory quality indices of cooked

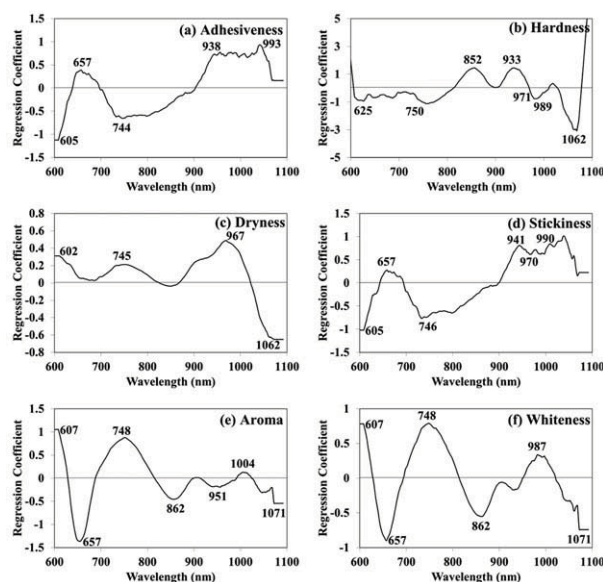


FIGURE 6 Regression coefficient plots of optimum models for evaluation of sensory quality of cooked rice. (a) Adhesiveness; (b) hardness; (c) dryness; (d) stickiness; (e) aroma; and (f) whiteness.

TABLE 3  
Means and SD of differences between duplicates and ratios of standard error of prediction (SEP) to standard deviation (SEP/SD) of repeatability and reproducibility tests

Eating quality	Repeatability		Reproducibility		SEP/SD
	Mean	SD	Mean	SD	
Adhesiveness	-0.1	0.2	0.0	0.2	0.4
Hardness	0.0	0.3	-0.1	0.2	0.4
Dryness	0.1	0.2	-0.1	0.2	0.7
Stickiness	0.0	0.2	0.1	0.3	0.8
Aroma	-0.2	0.2	0.2	0.2	0.9
Whiteness	-0.1	0.2	0.0	0.5	1.3

rice with SEP/SD values less than 1 could also be evaluated by VIS and SW-NIR spectroscopy. These findings indicate the potential application of NIRS, a non-destructive method, for evaluating the sensory qualities of cooked rice at different random sampling stations for quality assurance and better control at the rice quality improvement plant. In practice, this method based on VIS and SW-NIRS could reduce the time, number of procedures, and costs previously inherent in the rice industry.

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