# สมบัติทางเคมีกายภาพของข้าวและผลของวิธีการโม่ต่อสมบัติของฟลาวร์จากข้าว Oryza sativa L. พันธุ์ต่างๆ ในประเทศไทย

นางสาวจิตรานุช ลีวัชรารุ่งเจริญ

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต สาขาวิชาเทคโนโลยีทางอาหาร ภาควิชาเทคโนโลยีทางอาหาร

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# PHYSICOCHEMICAL PROPERTIES OF RICE AND EFFECT OF MILLING METHOD ON THE PROPERTIES OF FLOUR FROM DIFFERENT RICE *Oryza sativa* L. CULTIVARS IN THAILAND

Miss Jitranut Leewatchararongjaroen

A Thesis Submitted in Partial Fulfillment of the Requirements for the Degree of Master of Science Program in Food Technology Department of Food Technology Faculty of Science Chulalongkorn University Academic Year 2011 Copyright of Chulalongkorn University

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Ву	Miss Jitranut Leewatchararongjaroen
Field of Study	Food Technology
Thesis Advisor	Assistant Professor Jirarat Tattiyakul, Ph. D.

Accepted by the Faculty of Science, Chulalongkorn University in Partial Fulfillment of the Requirements for the Master's Degree

..... Dean of the Faculty of Science (Professor Supot Hannongbua, Dr.rer.nat.)

THESIS COMMITTEE

..... Chairman

(Assistant Professor Pasawadee Pradipasena, Sc. D.)

...... Thesis Advisor

(Assistant Professor Jirarat Tattiyakul, Ph. D.)

..... Examiner

(Kanitha Tananuwong, Ph. D.)

..... Examiner

(Daris Kuakpetoon, Ph. D.)

...... External Examiner

(Sunee Chotineeranat, Ph. D.)

จิตรานุช ลีวัชรารุ่งเจริญ : สมบัติทางเคมีกายภาพของข้าวและผลของวิธีการโม่ต่อสมบัติ ของฟลาวร์จากข้าว *Oryza sativa* L. พันธุ์ต่างๆในประเทศไทย. (Physicochemical Properties of Rice and Effect of Milling Method on the Properties of Flour from Different Rice *Oryza sativa* L. Cultivars in Thailand) อ. ที่ปรึกษาวิทยานิพนธ์หลัก: ผศ. ดร. จิรารัตน์ ทัตติยกุล, 157 หน้า.

งานวิจัยนี้ได้ศึกษาสมบัติทางเคมีกายภาพของข้าวและฟลาวร์ที่ได้จากการโม่แห้งและโม่เปียกข้าวที่ ปลูกในประเทศไทย จำนวน 9 สายพันธุ์ ได้แก่ กข41 กข45 กข47 ช่อลง97 ปราจีนบุรี1 ปราจีนบุรี2 พลายงาม ปราจีนบุรี ขาวดอกมะลิ105 และ อยุธยา1 โดยงานวิจัยแบ่งออกเป็น 2 ขั้นตอน ขั้นตอนแรก เป็นการศึกษา สมบัติทางเคมีกายภาพ และคุณภาพการหุงต้มของข้าว จากการทดลองพบว่า ข้าวทั้ง 9 สายพันธุ์มีความชื้น ในช่วงร้อยละ 11.1-12.7 โดยน้ำหนักเปียก และประกอบด้วย ไขมัน โปรตีน เถ้า เส้นใยหยาบ และคาร์โบไฮเดรต ร้อยละ 0.3-1.9, 6.3-10.7, 0.2-0.8, 0.1-0.5 และ 78.6-86.4 โดยน้ำหนักแห้ง ตามลำดับ มีช่วงเวลาในการหงต้ม ที่ 18-30 นาที และข้าวหุงสุกมีค่าความแข็งในช่วง 227.6-602.2 กรัม โดยข้าวสายพันธุ์ขาวดอกมะลิ105 ใช้เวลา ในการหงต้มน้อยที่สดและข้าวสายพันธ์ปราจีนบรี1 มีการเกาะตัวกันน้อยที่สด ส่งผลให้มีความแข็งน้อยที่สด ขั้นตอนที่ 2 เป็นการศึกษาผลของวิธีการโม่เปียกและโม่แห้งต่อสมบัติทางเคมีกายภาพของฟลาวร์ข้าว พบว่า ฟลาวร์ข้าวมีความชื้นร้อยละ 8.2-12.0 โดยน้ำหนักเปียก ฟลาวร์แห้งประกอบด้วย ไขมัน โปรตีน เถ้า และ คาร์โบไฮเดรต ร้อยละ 0.2-1.0, 5.3-9.2, 0.2-0.9 และ 87.7-92.5 ตามลำดับ สามารถแบ่งตามปริมาณแอมิโลส ได้เป็น 2 กลุ่ม คือ กข45 และ ขาวดอกมะลิ105 เป็นกลุ่มที่มีปริมาณแอมิโลสต่ำ (ร้อยละ 13.1-18.7) สายพันธุ์ ้อื่นๆที่ศึกษามีปริมาณแอมิโลสสูง (ร้อยละ 29.4-36.5) การโม่เปียกส่งผลให้ปริมาณโปรตีนในฟลาวร์มีค่าน้อย กว่าฟลาวร์โม่แห้งอย่างมีนัยสำคัญ (P ≤ 0.05) นอกจากนี้ยังพบว่าขนาดอนุภาคเฉลี่ยของฟลาวร์โม่แห้งมีขนาด ใหญ่กว่าฟลาวร์โม่เปียกอย่างมีนัยสำคัญ (P ≤ 0.05) ค่ากำลังการพองตัวที่ 60°C และค่าการละลายที่ทุก ้อุณหภูมิของฟลาวร์โม่เปียกมีค่าน้อยกว่าฟลาวร์โม่แห้งอย่างมีนัยสำคัญ (P ≤ 0.05) กระบวนการโม่แห้งส่งผลให้ โครงสร้างผลึกของแกรนลข้าวบางส่วนถกทำลาย ทำให้ค่าร้อยละความเป็นผลึกของฟลาวร์โม่แห้งมีค่าน้อย กว่าฟลาวร์โม่เปียกอย่างมีนัยสำคัญ (P ≤ 0.05) และเป็นผลให้ค่าพลังงานที่ใช้ในการเกิดเจลาติไนเซซั่น (ΔH) ของฟลาวร์โม่แห้งมีค่าน้อยกว่าฟลาวร์โม่เปียกอย่างมีนัยสำคัญ (P ≤ 0.05) จากการศึกษาสมบัติการเกิดเพสต์ พบว่า ฟลาวร์ข้าวโม่แห้งมีค่า peak viscosity น้อยกว่าฟลาวร์โม่เปียกอย่างมีนัยสำคัญ (P ≤ 0.05) เจล (ร้อยละ 12) ของฟลาวร์ข้าวทุกสายพันธุ์มีค่า G' มากกว่า G'' ที่ทุกความถี่ของการทดสอบ ซึ่งแสดงถึงลักษณะของความ เป็นของแข็งมากกว่าความเป็นของเหลว

สาขาวิชา <u>เทคโนโลยีทางอาหาร</u>	ุลายมือชื่อนิสิต
ปีการศึกษา <u>2554</u>	ลายมือชื่อ อ.ที่ปรึกษาวิทยานิพนธ์หลัก

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JITRANUT LEEWATCHARARONGJAROEN: PHYSICOCHEMICAL PROPERTIES OF RICE AND EFFECT OF MILLING METHOD ON THE PROPERTIES OF FLOUR FROM DIFFERENT RICE *Oryza sativa* L. CULTIVARS IN THAILAND. ADVISOR: ASST.PROF. JIRARAT TATTIYAKUL,Ph.D., 157 pp.

This research investigated physicochemical properties of rice and flour from Thailand grown rice cultivars, which are RD 41, RD 45, RD 47, Shaw Lung 97, Prachin Buri 1, Prachin Buri 2, Plai Ngahm Prachin Buri, Khao Dawk Mali 105 and Ayutthaya 1. The research consisted of two parts. The first part involved the investigation of physicochemical properties and cooking quality of rice. Rice grains contained 11.1-12.7% (wb) moisture, 0.3-1.9% (db) fat, 6.3-10.7% (db) protein, 0.2-0.8% (db) ash, 0.1-0.5% (db) fiber and 78.6-86.4% (db) carbohydrates. Cooking time of rice ranged from 18 to 30 min. Hardness values of cooked rice ranged from 227.6 to 602.2 gf. The second part involved the study on the effect of milling methods; wet- and dry-milling, on physicochemical properties of flour. The flours contained 8.2-12.0% (wb) moisture, 0.2-1.0% (db) fat, 5.3-9.2% (db) protein, 0.2-0.9% (db) ash and 87.7-92.5% (db) carbohydrates. The flour samples could be classified into low amylose flour that contained 13.1-18.7% amylose (RD 45 and Khao Dawk Mali 105) and high amylose flour that contained 29.4-36.5% amylose (Ayutthaya 1, Plai Ngahm Prachin Buri, Prachin Buri 1, Prachin Buri 2, RD 41, RD 47 and Shaw Lung 97 cultivars). Wet milling process resulted in flour with significantly lower (P  $\leq$  0.05) protein content. In addition, dry-milled rice flour contained granules with significantly larger average size (P  $\leq$  0.05) compared to wet-milled samples. Swelling power at 60°C and solubility of wet-milled samples were significantly lower ( $P \le 0.05$ ) than those of dry-milled flours. Dry milling process caused the destruction of the crystalline and yielded a lower crystallinity degree ( $P \leq 0.05$ ) compared to wet-milling process, which resulted in significantly lower gelatinization enthalpy ( $\Delta$ H). Dry-milled flours showed significantly (P  $\leq$  0.05) lower peak viscosity comparing to wet-milled flours. From the frequency sweep test, 12% (w/w) rice flours gel gave higher G' than G" indicating the typical characteristic of weak gel.

Field of Study : Food Technology	Student's Signature
	-
Academic Year : 2011	Advisor's Signature

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## CHAPTER 1

## INTRODUCTION

Rice *Oryza sativa* L. is a semi-aquatic, annual grass which can be grown under a broad range of climatic conditions (Marshall and Wadsworth, 1994). Rice is a staple of over approximately one-half of the world population. Nowadays, Thai rice was exported to more than 160 countries, amounting about 7 million tons per year. Rice production in Thailand has a spectacular increase in recent years and is approximately 30% of the total rice production of the world (Thai Free Trade Area, 2008). In Thailand, rice has been consumed mostly as cooked grains. It is also processed to flour and starch that can be further formulated into a variety of products. In order to successfully incorporate rice or its flour into products, the properties of the material have to be known as they will affect product qualities, consistency and also lead to good consumer satisfactory.

Studies have shown that factors influencing the physicochemical properties of rice flour include rice cultivars (Iturriaga *et al.*, 2004), amylose content (Varavinit *et al.*, 2003), protein content (Marco and Rosell, 2008) and milling methods (Suksomboon *et al.*, 2006). Studies (Suksomboon and Naivikul, 2006; Chen *et al.*, 1999; Chen *et al.*, 2003) have shown that dry milled flours retain other components such as protein, lipid and ash at a higher level than wet milled flours, but have more damaged starch and give lower peak and final viscosity. These, in turn, greatly affect the final product quality. In Thailand, numerous varieties of rice are grown nationwide. New rice varieties have been constantly bred by The Bureau of Rice Research and Development Rice Department (BRRD) and extended nationwide. This study aimed to investigate the physicochemical properties of cooked grains and flour of nine rice varieties which had been certified in 1959 (Khao Dawk Mali 105), 1994 (Plai Ngahm Prachin Buri), 1998 (Prachin Buri 1), 2002 (Prachin Buri 2), 2004 (Ayutthaya 1), 2009 (RD 41) and RD 45 RD 47 and Shaw Lung 97 that are the new varieties certified in 2010. Each developed variety has its own unique characteristics as briefly described below (BRRD, 2011).

- Khao Dawk Mali105 rice variety is photosensitive lowland rice that provides a production yield of 363 kg/rai. This rice cultivar is resistant to acidic soil, alkaline soil and drought. Milling quality is good with transparent grains. Khao Dawk Mali105 variety has a distinguished characteristic; aromatic, soft, and delicious. This rice is popular for consumption.
- Plai Ngahm Prachin Buri rice variety is native rice variety in Prachinburi province. This cultivar is photosensitive floating rice variety that provides a production yield of 380 kg/rai. This rice variety is resistant to rice blast and drought. The texture of cooked rice is hard, dry and flaky. Plai Ngahm Prachin Buri is suitable for processing into noodle and pasta products.
- Prachin Buri 1 rice variety is weakly sensitive photoperiod deepwater rice that provides a good production yield of 450 kg/rai. This rice variety is resistant to bacterial leaf streak and stem borers. The texture of cooked rice is hard, dry and flaky. Prachin Buri 1 is usually processed into noodle products, pasta and flours.
- Prachin Buri 2 rice variety is weakly sensitive photoperiod deepwater rice that provides very good production yield of 590 to 846 kg/rai. This rice variety is resistant to rice blast, bacterial leaf blight disease and acid soil. The texture of cooked rice is hard, dry and flaky. Prachin Buri 2 is suitable processed into noodle and pasta products.
- Ayutthaya 1 rice variety is weakly sensitive photoperiod deepwater rice that provides very good production yield of 546 to 842 kg/rai. This rice variety is resistant to brown plant-hopper and green rice leaf-hopper. The texture of cooked rice is hard, dry and flaky. Ayutthaya 1 is suitable for processing into noodle and pasta products.

- RD41 rice variety is non-photosensitive lowland rice that provides very good production yield of 722 kg/rai. It possesses good milling quality (100% head rice kernel) and is less chalky.
- RD45 (Hawm Prachin Buri) is the first recommended aromatic deepwater rice in Thailand. The variety yields distinctive soft and sticky texture cooked rice. The variety provides very good production yield of 520 kg/rai.
- RD47 rice variety is non-photosensitive lowland rice that provides very good production yield of 793 kg/rai. This cultivar is more resistant to brown planthopper than RD41 and also resistant to rice blast. Its milling quality is good to very good (100% head rice kernel) with less chalky rice. The texture of cooked rice is hard, dry and flaky.
- Shaw Lung 97 rice variety is photosensitive lowland rice that provides very good production yield of 564 kg/rai. This rice cultivar yields soft and sticky texture cooked rice.

The investigation in this study will provide an additional database on the properties of some existing commercial rices and some newly certified rice cultivars that will lead to appropriate and more diverse industrial applications and processes. Further, this study also focused on the effect of milling method; dry- and wet-milling, on physicochemical properties of rice flours.

## CHAPTER 2

## LITERATURE REVIEWS

## 2.1 Rice

Rice is a semiaquatic, annual grass which can be grown under a broad range of climatic conditions. Cultivated rice is designated as either *Oryza sativa* L. or *Oryza glaberrima* Steud. *O. sativa* is the predominant species; *O. glaberrima* is grown only in Africa on a limited scale (Marshall and Wadsworth, 1994). There are three groups of *O. sativa* cultivars: the short-grained "japonica" varieties, exemplified by Japanese rice; the long-grained "indica" varieties, exemplified by Jasmine and Basmati rice; and the broad-grained "javanica" varieties, which thrive under tropical conditions (Zohary and Hopf, 2000).

The principal parts of the grain consist of three major components that are hull, caryopsis, and endosperm (Figure 2.1).

#### 2.1.1 Husk

The husk is the outer covering for the caryopsis (brown rice). It consists of the lemma, which covers the dorsal part of the grain, and the palea, which covers the ventral portion. The lemma and palea are joined together longitudinally.

## 2.1.2 Caryopsis

Removal of the husk from rough rice by dehusking exposes the rice caryopsis. The outer four morphologically distinct layers of the caryopsis are the pericarp, seed coat, nucellus, and aleurone. Along with much of the embryo(germ), these layers comprise the bran portion of the rice grain.

#### 2.1.3 Endosperm

Further milling of the rice caryopsis removes the sub-aleurone layer and a small part of the starchy endosperm. This milling fraction is referred to as "polish". The end result of bran and polish removal is milled rice. The endosperm is rich in starch granules (Marshall and Wadsworth, 1994).

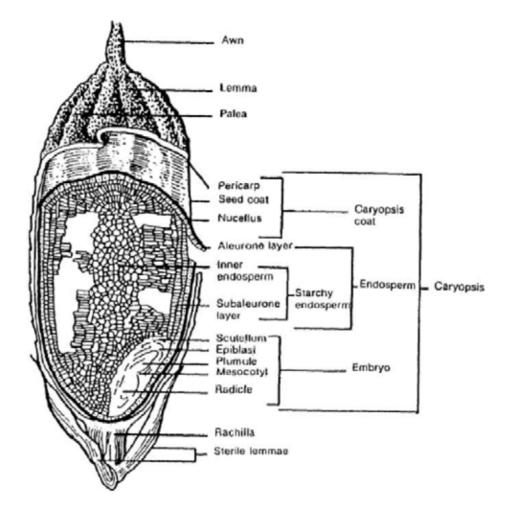


Figure 2.1 Structure of rice grain Source: Zhou *et al.* (2002)

Rice varieties are distinguished by their amylose content. There are two basic types: common and waxy. Waxy rice has an amylose content of 0 - 2%. Waxy rices are also referred to as glutinous rices, due to their sticky cooking property. Common or regular rice varieties are referred to as having amylose contents that are low (9 - 20%), medium (20 - 25%) or high (> 25%) amylose (Bemiller and Whistler, 2009).

In Thailand, numerous varieties of rice are grown nationwide. In this study nine rice varieties were investigated. Each developed variety has its own unique characteristics as described below (BRRD, 2011).

- Khao Dawk Mali105 (KDML 105) rice variety had been certified and released on May 25, 1959. This cultivar is non-glutinous and photosensitive lowland rice variety. In 1950-1951, Mr. Soontorn Sihanern, Rice Officer from Bang Khla, gathered a variety of 199 panicles of KDML from the farmers. The rice panicles were sent to Rice Breeding Division. The rows of 105<sup>th</sup> rice panicle were selected as pure line. The average yield is 363 kg/rai. Suitable cultivating area for Khao Dawk Mali105 is the areas in the Northeastern and Higher Northern of Thailand. Khao Dawk Mali105 is resistant to acidic soil, basic soil and drought but shows susceptible reaction to bacterial leaf blight disease, rice blast, rice ragged stunt disease, yellow orange leaf disease, brown plant hopper, stem borers and green rice leaf hopper. Milling quality is good with transparent grains. Khao Dawk Mali105 variety has had a distinguished characteristic; aromatic, soft, and delicious. This rice variety is popular for consumption.
- Plai Ngahm Prachin Buri rice variety had been certified on June 17, 1994. This cultivar is non-glutinous and photosensitive floating rice variety. Plai Ngahm Prachin Buri is native rice variety in Prachinburi province. The average yield is 380 kg/rai. Suitable cultivating area for Plai Ngahm Prachin Buri is the areas in the Central region and Lower Northern of Thailand where water depth is more than 100 cm. Plai Ngahm Prachin Buri is resistant to rice blast and drought but shows susceptible reaction to rice ragged stunt disease, yellow orange leaf disease, brown plant hopper and green rice leaf hopper. Rice grains have medium chalkiness. The texture of cooked rice is hard, dry and flaky. Plai Ngahm Prachin Buri is suitable for processing into noodle and pasta products.

- Prachin Buri 1 rice variety had been certified and released on September 30, 1998. This cultivar is non-glutinous and weakly sensitive photoperiod deepwater rice variety. The average yield is 450 kg/rai. Suitable cultivating area for Prachin Buri 1 is the areas in the Central region, Eastern and Lower Northern of Thailand where water depth is less than 100 cm. Prachin Buri 1 is resistant to bacterial leaf streak and stem borers but shows susceptible reaction to yellow orange leaf disease, brown plant hopper and green rice leafhopper. Milling quality is good. The texture of cooked rice is hard, dry and flaky. Due to high amylose content rice cultivar, Prachin Buri 1 is suitable for processing into noodle and pasta products.
- Prachin Buri 2 rice variety had been certified and released on January 7, 2002. This cultivar is non-glutinous and weakly sensitive photoperiod deepwater rice variety. Prachin Buri 2 had been bred between BKNFR 80086 and HTAFR 80038 at Ayutthaya Rice Research Center in 1981. The average yield is 846 kg/rai at 25 cm water level and 590 kg/rai at 100 cm water level. Suitable cultivating area for Prachin Buri 2 is the areas in the Central region and Eastern of Thailand where water depth is less than 100 cm. Prachin Buri 2 is resistant to rice blast, bacterial leaf blight disease and acidic soil but shows susceptible reaction to yellow orange leaf disease, brown plant hopper and green rice leafhopper. The texture of cooked rice is hard, dry and flaky. Prachin Buri 2 is suitable for processing into noodle and pasta products.
- Ayutthaya 1 rice variety had been certified and released on August 19, 2004. This cultivar is non-glutinous and weakly sensitive photoperiod deepwater rice variety. Ayutthaya 1 had been bred between Oo Tah Pow and Khao Dawk Mali 105 at Ayutthaya Rice Research Center in 1985. The average yield is 546 kg/rai at 100 cm water level and 842 kg/rai at 25 cm water level. Suitable cultivating area for Ayutthaya 1 is in the Central region, Eastern and Lower Northern of Thailand where water depth is less than 100 cm. Ayutthaya 1 is resistant to

brown plant hopper and green rice leaf hopper but shows susceptible reaction to rice blast, yellow orange leaf disease and bacterial leaf blight disease. The texture of cooked rice is hard, dry and flaky. Ayutthaya 1 is suitable for processing into noodle and pasta products.

- RD 41 rice variety had been certified and released on September 17, 2009. This cultivar is non-glutinous and non-photosensitive lowland rice variety. RD41 had been bred between CNT 85059-27-1-3-2 × Suphan Buri 60 and RP 217-635-8 at Chainat Rice Research Center in 1996. The average yield is 722 kg/rai. Suitable cultivating area for RD41 is the irrigated areas in the Lower Northern area of Thailand where water depth is less than 50 cm. Generally, RD 41 is resistant to rice blast and brown planthopper but shows susceptible reaction to bacterial leaf blight disease. Its brown rice is slender. Milling quality is very good (100% head rice kernel) and less chalky.
- RD 45 (Hawm Prachin Buri) is the first recommended aromatic deepwater rice in Thailand. RD45 rice variety had been certified and released on September 28, 2010. This cultivar is non-glutinous and weakly sensitive photoperiod deepwater rice variety. RD45 had been bred between PCRBR 83012-267-5 line (Hawm Nai Pon × IR 46) and Khao Dawk Mali 105 at Prachin Buri Rice Research Center in 1989. The average yield is 520 kg/rai. Suitable cultivating area for RD 45 is the rainfed areas of Central region and Eastern Thailand where water depth is less than 100 cm and water level in field should be low in November, but not in the areas of brown plant hopper. Its brown rice is slender. Milling quality is very good with transparent grains and less chalkiness. The distinct property is aromatic, soft and sticky texture cooked rice.
- RD 47 rice variety had been certified and released on September 28, 2010. This cultivar is non-glutinous and non-photosensitive lowland rice variety. RD 47 had

been bred between Suphan Buri 1 × IR 64 and CNT 86074-25-9-1 at Chainat Rice Research Center in 1996. The average yield is 793 kg/rai. Suitable cultivating area for RD 47 is the irrigated areas in the Lower Northern area of Thailand where water depth is less than 50 cm. RD 47 is more resistant to brown planthopper than RD 41 and also resistant to rice blast. However, RD 47 shows susceptible reaction to bacterial leaf blight disease and cold weather. Its brown rice is slender. Milling quality is good to very good (100% head rice kernel) and less chalky. The texture of cooked rice is hard, dry and flaky.

Shaw Lung 97 rice variety had been certified on September 28, 2010. This cultivar is non-glutinous and photosensitive lowland rice variety. Shaw Lung 97 is native rice variety in Pattani and Songkhla province. The average yield is 564 kg/rai. Suitable cultivating area for Shaw Lung 97 is the rainfed areas in the Southern area of Thailand such as Pattani, Narathiwat, Yala and Songkhla province. Shaw Lung 97 shows susceptible reaction to rice blast and very sensitive to brown plant hopper. Milling quality is good and medium chalky. The distinct property is soft and sticky texture cooked rice.

### 2.2 Cooking qualities of rice

In Thailand, rice has been consumed mostly as cooked rice. Amylose is the major factor which influences textural properties of cooked rice. During cooking, rice starch granules absorb water and swell to great extent compared to their original size. The granule expansion causes ruptures and, hence, amylose leaching (Tester and Morrison, 1990). Upon cooling, amylose undergoes a relatively re-association process and can form tightly packed structures (retrogradation) causing dry and fluffy texture in cooked rice. Therefore low amylose cooked rice has a soft and sticky texture whereas high amylose cooked rice has a dry and fluffy texture with the grains separate (Webb *et al.*, 1986).

In addition, the other components such as protein and lipid also exert an impact on textural properties of cooked rice. Ayabe *et al.* (2009) and Kongseree (1994) demonstrated that higher protein content especially around the outer layers of rice grains could provide longer cooking time because water penetration into rice grains was interrupted by protein. Rice with more protein content showed harder and less sticky cooked rice. On the other hand, lipids can form complexes with amylose and amylopectin (Biliaderis and Tonogai, 1991). These complexes are believed to restrict starch granule swelling during heating and prevent leaching of amylose during gelatinization (Saleh and Meullenet, 2005), thereby increasing cooked rice firmness and decreasing stickiness (Seneviratne and Biliaderis, 1991).

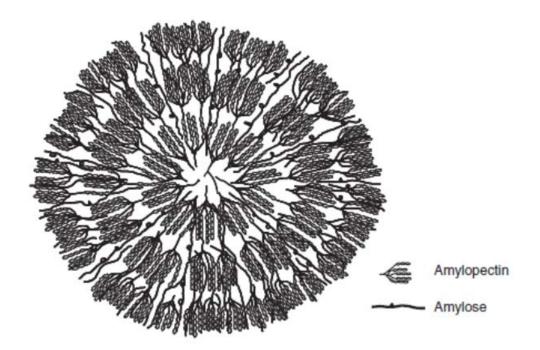
### 2.3 Flour and Starch

There are three methods used to prepare rice flour; wet milling process, semi-dry milling process, and dry milling process. For wet milling process, rice grains are soaked and ground under continuous addition of water, and dried to remove the excess water. Semi-dry milling process, rice grains are soaked overnight and dried, then ground to get rice flour without water. And for dry milling process, cleaned rice grains can be directly ground to get rice flour without generating any waste water (Ngamnikom and Songsermpong, 2011). Each milling methods have different advantages. The wet milling process yields starch containing lower amount of damaged starch and give the finest flour particle. However, the wet-milling process consumes a large amount of water, which in turn creates a lot of wastewater and also causes some components such as soluble protein, sugars, and non-starch bound lipids to leach out during soaking. In contrast, dry milling process give the flour with higher amount of damaged starch without generating any wastewater (Chiang and Yeh, 2002; Suksomboon and Naivikul, 2006).

## 2.4 Physicochemical properties of starch

### 2.4.1 Chemical composition of starch

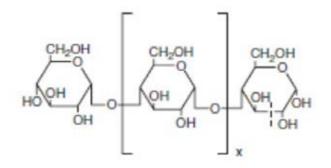
Starch is a polymer of D-glucose and usually consists of an essentially linear fraction; amylose, and a branched fraction; amylopectin. Starch molecules are aligned radially in a granule that has a semi-crystalline characteristic. Starch granules consist of amorphous and crystalline regions. The crystalline regions are formed by the short branch chain of amylopectin molecules arranged in clusters (Figure 2.2). The areas of branching points are believed to be amorphous, suggested that some amylose molecules are located in this region with some interaction with the branch chain of amylopectin (Bemiller and Whistler, 2009).



**Figure 2.2** Schematic of the organization of a starch granule Source: Bemiller and Whistler (2009)

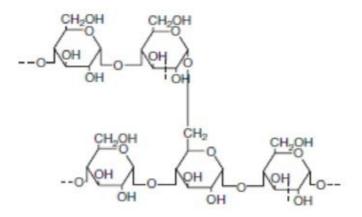
Amylose is a linear polymer made up of  $\alpha$ -1,4-linked D-glucopyranose units (Figure 2.3). The degree of polymerization (DP) is between 100 and 10,000. However, amylose from some starch sources contains about 2 to 8 branch point per molecule. The chain

length of these branch chains varies from 4 to 100 DP (Hizukuri *et al.*, 1981). Amylose chains give the molecules a right-handed spiral or helical shape. The interior of the helix is lined with hydrogen atoms and is hydrophobic, resulting in amylose complex forming with free fatty acids. There are three main forms the amylose chains can take. It can exist in a disordered amorphous conformation or two different helical forms. It can bind with itself in a double helix, or it can bind with another hydrophobic guest molecule such as iodine, a fatty acid, or an aromatic compound (Belitz *et al.*, 2009).



**Figure 2.3** Structure of amylose Source: Bemiller and Whistler (2009)

Amylopectin is a very large, highly branched molecule, and composes of  $\alpha$ -1,4linked D-glucopyranose connected by  $\alpha$ -1,6-linked branch points (Figure 2.4). The amylopectin chains can be classified into three different types of sub-chains, termed A, B and C, according to their length and branching points. The shortest A chains (DP 6-15) carry no branch points and are linked to the amylopectin molecule by a single  $\alpha$ -1,6linked. The B chains are branched by A chain or other B chains. B chains are further classified, depending on their respective length and number of cluster they span, into B1 (DP ~15-25), B2 (DP ~40-50), with B3 and B4 chains being longer. There is only one C chain per amylopectin molecule and it is identified as having the only non-reducing end (Figure 2.5). It is now widely accepted that linear branched chains with DP~15 in amylopectin are the crystalline regions present in the granules. These short chains form double helical ordered structure; part of the double helices can pack together in organized arrays in cluster form (Bemiller and Whistler, 2009; Banchathanakij, 2008).



**Figure 2.4** Structure of amylopectin Source: Bemiller and Whistler (2009)

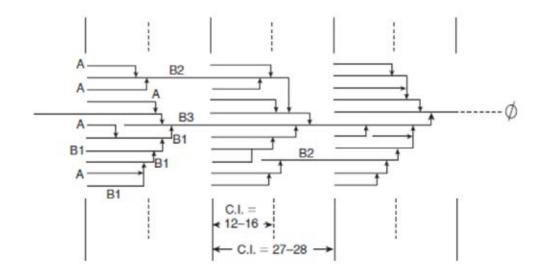


Figure 2.5 Cluster model of amylopectin Source: Hizukuri (1986)

Normal rice starch contains substantial amounts of phospholipids and some free fatty acids. Lipids and phospholipids are known to form stable complexes with long chains of starch, with both amylose and long branch chains of amylopectin (Figure 2.6, 2.7), which results in the restricted swelling of granules (Bemiller and Whistler, 2009).

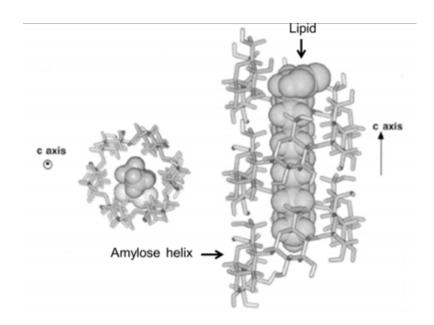


Figure 2.6 Molecular modeling of amylose-lipid complex

Source: Buléon et al. (1998)

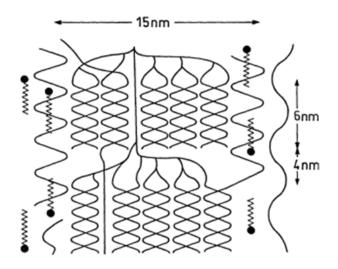


 Figure 2.7 Model of a crystalline region in a starch granule

 Amylopectin double helix

 Mixed double helix of amylose and amylopectin

 Amylose-lipid complex

Free lipid \*\*\*\*\*\*;

Free amylose

Source: Belitz et al. (2009)

2.4.2 Physical properties of starch

2.4.2.1 Morphology of starch granules

Rice consists of compound starch granules that are tightly packed together and difficult to separate. The shape of the compound starch granules is mostly polygonal but irregular, possibly as a result of space constraints during the development of starch granules. Rice starch granules have a size range from 3 to 8  $\mu$ m (Haard *et al.*, 1999; Singh *et al.*, 2003).

Most native starch granules exhibit a Maltese cross when observed under polarized light (Figure 2.8). Theoretically, the positive birefringence indicates a radial orientation of the principle axis of the crystallites. The crystallinity of starch is caused essentially by amylopectin polymer interactions. It is believed that the outer branches of amylopectin molecules interact to arrange themselves into "crystallites" forming crystalline lamellae within the granule. A small number of amylose polymers may also interact with amylopectin crystallites. This hypothetical structure has been derived based on the cluster model of amylopectin (Vandenberg, 1981).

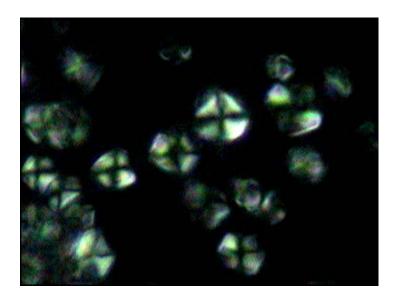


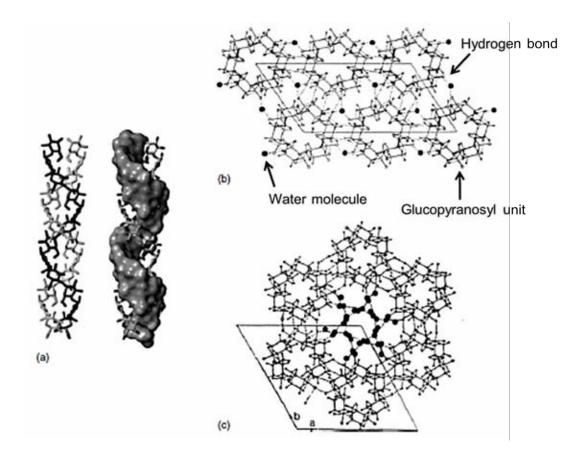
Figure 2.8 Polarized light micrographs of RD 45 wet-milled rice flours

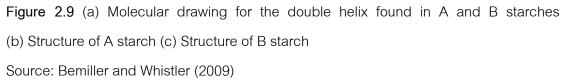
Crystalline patterns of starch are classified into three distinct groups, A, B, and C based on their polymorphism (Figure 2.9). Generally, most cereal starches give characteristic A-type pattern. Some tuber starches (e.g. potato) and cereal starches rich in amylose yield the B-type pattern, while legume starches generally give a C-type pattern (Bemiller and Whistler, 2009).

Rice starch has the A-type X-ray diffraction pattern. The structure of A-type starch indicates that there are 12 glucosyl units and 4 water molecules in each unit cell. The chain structure was left-handed, parallel-stranded, double helices. Each strand repeats in 2.138 nm, but is related to the other strand by a two-fold axis of rotation, yielding the apparent fiber repeat distance of 1.069 nm. There are no intra-chain hydrogen bonds, but there is an O-2 and O-6 hydrogen bond between the two strands. The double helix is very compact, and there is no space for water or any other molecule in the center (Bemiller and Whistler, 2009).

The structure of B-type chains are also organized in double helices, but the structure differs from A-type starch in crystal packing and water content. The crystalline unit cell is hexagonal. Double helices are connected through a network of hydrogen bonds that form a channel inside the hexagonal arrangement of six double helices. This channel is filled with water molecules, half of which are bound to amylose by hydrogen bonds and the other half to other water molecules. Thus, with a hydration of 27%, 36 water molecules are located in the unit cell between the six double helices, creating a column of water surrounded by the hexagonal network (Bemiller and Whistler, 2009).

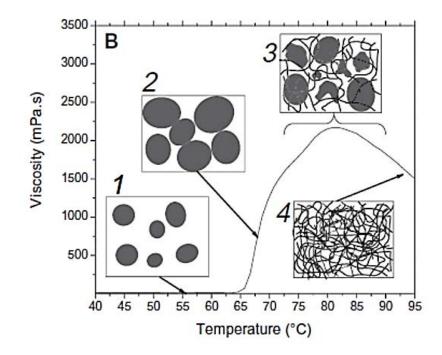
The C-type pattern is contained about 60% A-type structure and 40% B-type structure, and these two crystalline phases co-existed within the same granule. The A allomorph was essentially located in the outer part of the granules, whereas the B-type was found mostly near their center (Bemiller and Whistler, 2009).





### 2.4.2.2 Swelling and solubility

Starch is insoluble in cold water because hydrogen intermolecular bonds. But when starch is heated in excess water, the crystalline structure is disrupted due to the breakage of hydrogen bonds, and water molecules become linked by hydrogen bonding to the exposed hydroxyl group of amylose and amylopectin. This causes an increase in granule swelling and solubility (Figure 2.10). Swelling of starch granules is also accompanied by leaching of starch molecules from the granules. This material is largely amylose, although amylopectin may also solubilize depending on the nature of the starch and the severity of heat-shear conditions employed. The molecular weight of the solubilized amylose increases with increasing temperature (Eliasson, 2004). Swelling power and solubility indicate the strength of non-covalent bonding among starch molecules which depends on factors like amylose-amylopectin ratio, chain length and molecular weight distribution, degree/length of branching and conformation. Moreover, the amylose-lipid complex has been shown to restrict swelling and solubility (Hoover, 2001).



**Figure 2.10** Schematic representation explaining the swelling and solubility of starch dispersions (1) Starch granules dispersion (2) The swelling of the starch granules (3) Swollen starch granules, fragment of the starch granules and solubilized amylose and amylopectin molecules (4) Fully dispersed amylose and amylopectin molecules Source: Zuo *et al.* (2012)

### 2.4.2.3 Gelatinization

When starch granules are heated in excess water to progressively higher temperatures, a point is reached where the polarization cross starts to disappear and the granules begin to swell irreversibly. These phenomena, associated with the disruption of granular structure, are called "gelatinization". Gelatinization can be defined as the collapse (disruption) of molecular orders (breaking of H-bonds) within the granule, along with all concomitant and irreversible changes in properties such as water uptake, granular swelling, crystallite melting, birefringence loss, starch solubilization and viscosity development (Bemiller and Whistler, 2009).

Application of differential scanning calorimetry (DSC) to starch has been useful in inferring differences in starch structures, changes in physical states of starch, and interactions of starch polymers with other constituents in model systems and composite food matrices. DSC measures the direction and extent of heat energy flow while a small sample is exposed to constant rate of change in temperature. It can detect both first-order (melting, crystallization) and second-order (glass) transitions of starch. As a structural probe of starch, DSC is sensitive to the molecular order of chains (helical conformation), irrespective of their involvement in crystalline arrays. DSC measures the range in transition temperature required for gelatinization to occur. Thermal properties typically reported using DSC include onset temperature gelatinization (To), peak temperature gelatinization (Tp), conclusion temperature gelatinization (Tc) and gelatinization enthalpy ( $\Delta$ H) (Figure 2.11). The swelling usually starts at a temperature corresponding to the onset temperature (Tc) (Kim *et al.*, 1995; Ratnayake and Jackson, 2009).

Gelatinization temperature and  $\Delta H$  are indicators of the overall crystallinity of amylopectin, which is directly related to the structure of amylopectin. Yoenyongbuddhagal and Noomhorm (2002a) demonstrated that starch with a greater amount of long-chain amylopectin and smaller amount of short-chain amylopectin exhibited higher To, Tp and  $\Delta H$  than starch with fewer long branched chains. This is because the longer chains have a greater ability to form double helices, which required greater thermal energy to dissociate (Yoenyongbuddhagal and Noomhorm, 2002b).

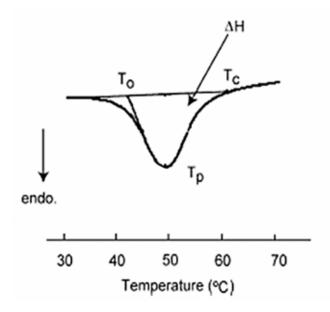


Figure 2.11 Differential scanning calorimetry thermogram Source: Kohyama *et al.* (2004)

2.4.2.4 Pasting properties

Pasting is the phenomenon following gelatinization in the dissolution of starch. It involves granular swelling, exudation of molecular components from the granule, and eventually, total disruption of the granules (Atwell *et al.*, 1998). When starch granules are heated in water, the granules can absorb a large amount of water and swell to several times of their initial size. Over a gelatinization temperature range, granules also undergo an irreversible process known as gelatinization, which is marked by crystalline melting, loss of birefringence and soluble starch. The pasting viscosity during swelling and gelatinization can be recorded using a Rapid visco-analyzer (RVA), which records the viscosity continuously as the temperature is increased, held constant for a time, and then decreased (Figure 2.12). Early in a test, the temperature is below the gelatinization temperature of the starch, and hence the viscosity is low. The increase in viscosity with temperature may be attributed to the removal of water from the exuded amylose by the granules as they swell, the shear forces caused by these swollen granules squeezing past one another results in an increase in viscosity. The temperature at the onset of this rise in viscosity is known as the pasting temperature. The pasting temperature provides

an indication of the minimum temperature required to cook a given sample, which can have implications for the stability of other components in a formula and also indicate energy costs. As the temperature increases further, the granules continue to swell and eventually rupture. The relatively soluble amylose leaches out into solution, followed at a slower rate in some cases by the amylopectin. Owing to the mechanical shear applied to the sample, these polymers tend to align themselves. These combined processes that follow gelatinization are known as pasting. The peak viscosity occurs at the equilibrium point between swelling and polymer leaching (which cause viscosity to increase) and rupture and polymer alignment (which cause viscosity to decrease). The temperature and time corresponding to the peak viscosity are referred to as the peak temperature and peak time. The peak viscosity indicates the water-binding capacity of the starch or mixture. It is often correlated with the final product. During the hold period of a test, the sample is subjected to a period of constant high temperature (typically 95°C) and mechanical shear stress. The granules undergo further disruption while the amylose molecules continue to leach out into solution and undergo alignment. This period is usually accompanied by a reduction in viscosity, which eventually reaches a minimum value. This viscosity is known either as the holding strength, hot paste viscosity or trough. The rate and extent of reduction from the peak viscosity depend on the temperature, the degree of mixing or shear stress applied to the mixture, and the nature of the material itself. The ability of a sample to withstand this heating and shear stress is an important factor for many processes. As the mixture subsequently cools, reassociation between starch molecules, especially amylose, occurs to a greater or lesser degree. Given a sufficient concentration of starch, this usually causes the formation of a gel. The viscosity will normally increase and stabilize at a final viscosity. Final viscosity increases upon cooling which might be due to the aggregation of the amylose molecules and commonly used parameter to define a particular sample quality, as it indicates the ability of the material to form a viscous paste or gel after cooking and cooling. The phase of the pasting curve between the trough and final viscosity is commonly referred to as the setback region. The difference between the two ends of this region is known as the setback viscosity. Setback value is the recovery of the viscosity during cooling of the heated starch suspension. In such cases, setback is measured as the difference between the final viscosity and peak viscosity, rather than as the difference between the final viscosity and holding strength. Setback involves retrogradation, or re-ordering, of the starch molecules. The setback of starch based samples can be quite marked. Setback has been correlated with the texture of various products. High setback is also associated with syneresis and may be due to the amount and the molecular weight of the amylose leached from the granules and the remnants of the gelatinized starch (Newport Scientific, 2001; Norbert *et al.*, 1995).

Pasting properties of starch have been reported to be affected by amylose and lipids contents and by branch chain-length distribution of amylopectin. Amylopectin contributes to swelling of starch granules and pasting, while amylose and lipids inhibit the swelling. Also, the amylopectin chain-length and amylose molecular size produce synergistic effects on the viscosity of starch pastes (Singh *et al.*, 2006).

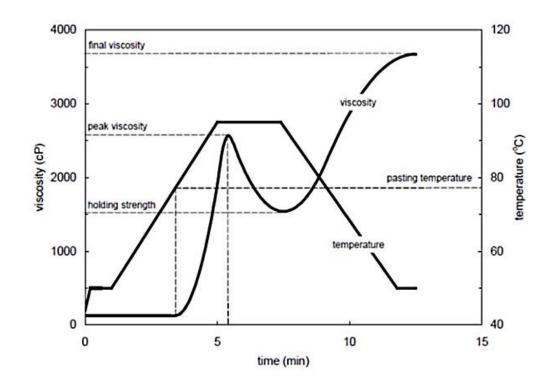


Figure 2.12 Rapid Visco Analyzer proflie Source: Newport Scientific (2001)

#### 2.4.2.5 Viscoelastic

Viscoelasticity is the property of materials that exhibit both viscous and elastic characteristics when undergoing deformation. Viscous materials resist shear flow and strain linearly with time when a stress is applied. Elastic materials strain instantaneously when stretched and just as quickly return to their original state once the stress is removed. Viscoelastic materials have elements of both of these properties and, as such, exhibit time dependent strain. Whereas elasticity is usually the result of bond stretching along crystallographic planes in an ordered solid, viscosity is the result of the diffusion of atoms or molecules inside an amorphous material. For purely viscous and elastic materials, representing the two extreme limits of viscoelastic response, the phase shift between shear strain and shear stress are 90° and 0°, respectively. Instead, a viscoelastic material responds with an intermediate phase shift between 0° and 90° (Bemiller and Whistler, 2009).

Dynamic oscillatory rheometry has been proven useful in monitoring structure development during aging of starch gels. It allows continuous assessment of dynamic moduli without breaking structural elements formed in the sample upon aging (Karim *et al.*, 2000) (Figure 2.13).

Starch pastes or gels can have both viscous (liquid-like) and elastic (solid-like) properties. Dynamic test (small deformation test or destruction free method) was used to characterize property of viscoelastic behavior. Basically, the gel specimen is subjected to a periodic, small amplitude sinusoidal torque (stress), the applied stress being altered at a given frequency (cycles s<sup>-1</sup> or  $\omega$ , radians s<sup>-1</sup>). If the behavior of a viscoelastic material is liner, the strain will be also vary sinusoidally with the stress, but will be out of phase with it. This behavior is intermediate between an ideally elastic material and a true Newtonian liquid where the stress is in phase ( $\delta = 0$ ) and 90° out of phase, respectively, with the strain. Just as modulus is defined as the stress/strain ratio in any constant deformation experiment, then, for a dynamic sinusoidal experiment it follows that two moduli can be defined: stress in-phase/strain or storage modulus (G') and stress out-of-phase/strain or loss modulus (G'') (Karim *et al.*, 2000).

Storage modulus (G') is a measure of the energy stored in the material and recovered from it per cycle. On a molecular basis, the magnitude of G' is dependent upon what rearrangements can take place within the period of oscillation, and is taken as an indication of the solid or elastic character of the material (Osada and Khokhlov, 2002).

Loss modulus (G") is an estimate of the energy dissipated as heat per cycle of deformation and is an indicator of the viscous properties of the material. Moreover, the loss modulus is defined as the stress  $90^{\circ}$  out-of-phase with the strain divided by the strain (Osada and Khokhlov, 2002).

For a gel network structure with non-permanent crosslinks, as most of the polysaccharide gels are, strong inter-chain associations (i.e. those having long relaxation times) contribute to G', whereas weak and rapidly relaxing bonds contribute only to G''. Interactions with relaxation times in the timescale of the measurements contribute to both G' and G''. The results from dynamic tests may be also presented in the form of the complex modulus (G\*) defined as  $G^* = G' + iG'' = (G'^2 + G''^2)^{1/2}$ .

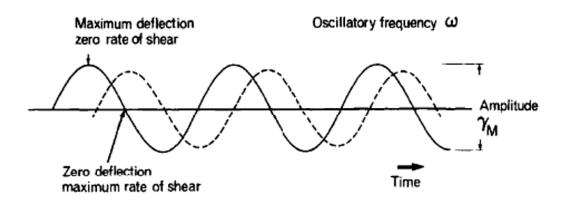
Another parameter which is often useful in indicating the physical behavior of a system is the loss tangent or tan  $\delta = G''/G'$ . This ratio is a more sensitive indicator than G' and G'' to changes in the viscoelastic character of a polymer network structure; a low loss tangent means that the material behaves more like a solid than a liquid (Gnanou and Fontanille, 2002).

Dynamic oscillatory rheometry has proved useful in monitoring structure development during aging of starch gels. It allows continuous assessment of dynamic moduli without breaking structural elements formed in the sample upon aging.

Applications for dynamic oscillatory test

- i. Deformation sweep at constant frequency (G' and G'' vs. strain) to determine the maximum deformation attainable by a sample in the linear viscoelastic region.
- ii. Frequency sweep (G' and G'' vs.  $\omega$ ) at constant deformation within the linear viscoelastic range to determine the elastic character of the gel

iii. Temperature sweep at constant frequency and deformation within the linear viscoelastic range (G' and G'' vs. Temperature) to evaluate thermal characteristics (Bemiller and Whistler, 2009; Kavanagh and Rossmurphy, 1998; Prokopowich and Biliaderis, 1995).



**Figure 2.13** The principle of applying a sinusoidal oscillation of maximum strain and oscillation frequency to a sample using parallel plate geometry Source: Kavanagh and Rossmurphy (1998)

#### 2.5 Effect of milling on physicochemical properties of starch and flour

Rice grains are usually processed into flour. Physicochemical properties need to be determined before flour use as they will affect the qualities, consistency and also lead to good consumer satisfactory. Some studies have investigated the properties of rice flour and found that milling method is an important factor which influences the physicochemical properties of rice flour.

Suksomboon and Naivikul (2006) conducted a study on starches from low amylose (Pathum Thani 1), medium amylose (RD 7) and high amylose (Leuang 11) rice varieties prepared from dry- and wet-milling processes. The results showed that dry-milled rice starches contained significantly ( $P \le 0.05$ ) higher amounts of damaged starch than wet-milled rice starches in all rice varieties. The enthalpy required to gelatinize wet-milled rice starches from all rice varieties was significantly ( $P \le 0.05$ ) higher than those of dry-

milled rice starches. The higher gelatinization enthalpy ( $\Delta$ H) suggested that a greater amount of energy was required to melt the crystallites (Patindol and Wang, 2002). The peak viscosity and final viscosity of dry-milled rice starches measured by Rapid Visco Analyser (RVA) were significantly (P ≤ 0.05) lower than those of wet-milled rice starches. The lower peak viscosity was caused by higher damage starch of dry-milled rice starches. Dry-milled rice flour contained significantly larger flour particles in the form of these aggregated starch granules, which were 50 – 150 µm in diameter compared to the separated starch granules of wet-milled samples which were 5-9 µm in diameter. Due to the leaching of protein matrix and other substances from the surface of starch granules during the soaking process of wet-milling rice flour that caused the structure of starchy endosperm to become loosen which resulted in fine particles and less damaged starch (Suksomboon and Naivikul, 2006).

Chen et al. (1999) studied on physicochemical and functional properties of waxy rice flour (TCSW1, long grain; TCW70, short grain) prepared from six milling methods. For dry-milled flour, polished rice kernels were ground with turbo, cyclone, or hammer mills. For semi-dry milled flour, rice kernels were ground with a hammer or plate mill. For wet-milled flour, the rice kernels were ground in water with a double-disk stone mill. The results showed that dry-milling maintained a higher level of the chemical components such as protein, lipid, and ash than other milling methods. When soaked rice kernels were processed by wet-milling, some soluble proteins, sugars, and nonstarch bound lipids were washed out. Hammer and semi-dry hammer milling gave higher percentages of coarse particles (100-300 µm); cyclone and turbo milling led to a more even particle-size distribution, and the wet-milling gave the finest particles (10-30 µm). Dry hammer-milled rice had higher gelatinization and pasting temperatures, and semi-dry milling resulted in the lowest pasting temperature, setback viscosity, and enthalpy value among the mills. The final quality of the two waxy rice varieties was profoundly affected by the mill type and milling method. Color measurement showed that the finer the flours, the brighter and whiter color was obtained. Due to the sample particle size affected the color, and that the smaller flour particles resulted in a smoother surface. Since the samples were different in particle size, the surface texture reflected from the sample would vary among samples. Moreover, Lamberts et al. (2007) explained the differences between the color parameters of the flour from rice that it may be from a dilution effect, grinding of rice kernels results in a mix of a small dark, coloured fraction (bran and outer endosperm) and a large light, less pigmented fraction (middle and core endosperm). Scanning electron microscopy showed that dry-milled rice flour has clump starch granules, but the starch granules from wet-milled samples were separated. The grinding-milled rice flour had higher solubility than the others. Possibly, the differences were caused by damaged starch produced during the grinding process. The waxy type starch granules unrestricted the swelling and resulted in the absence of a network structure from amylose molecules that can hold the starch molecules together. Dry hammer-milled flour showed the highest pasting temperature, and the semi-dry grinding milled flour had the lowest pasting temperature and setback viscosity. The absence of peak viscosity is due to delayed swelling of the starch granules that are embedded in the relatively large endosperm chunks in coarse flours. The finest flours had the lowest initial onset temperature, while the coarse flours had the highest. This is probably because the flour with larger particles could not hydrate or expand as rapidly and was inhibited by the starch on the particle surface. The lowest  $\Delta H$  value came from the semi-dry plate milled flour, which was in contrast with the highest  $\Delta H$  value obtained from the wet-milled flour. This reflected the considerable disruption of the native crystalline structure occurred during the grinding process. The mechanical force caused starch damage during milling, with a lower  $\Delta H$  value in rice flour.

Chen *et al.* (2003) investigated the physicochemical properties of damaged rice starch from two types of rice cultivars, indica type (Tainung Sen 19, TNuS19) and waxy (Taichung Waxy 70, TCW70) prepared from ball-milling treatment. Polarized light micrographs showed that damaged starch granules lost their birefringence, suggesting that the order structure of starch granule was disrupted. Rice cultivars and ball-milling treatment time affected all pasting parameters of the starch suspensions. The ball-

milling treatment time was negatively correlated with onset temperature, peak viscosity, hot-paste viscosity and cold-paste viscosity. Because of the ball-milling caused starch molecules (i.e. amylopectin and amylose) to breakdown into low molecular weight fragments, the paste viscosity of starches were decreased. The effects of ball milling may be attributed partly to the conversion of starch into a more amorphous form as treatment time increased; therefore  $\Delta H$  was negatively correlated with ball-milling treatment time.

# CHAPTER 3

# MATERIALS AND METHODS

# 3.1 Materials

Nine Thai rice cultivars were used in this study. They were Ayutthaya 1, Plai Ngahm Prachin Buri, Prachin Buri 1, Prachin Buri 2, RD 45 (from Prachin Buri Rice Research Center, harvested during March, 2011) Khao Dawk Mali 105 (from Pathum Thani Rice Research Center, harvested during December, 2010) Shaw Lung 97 (from Pattani Rice Research Center, harvested during March, 2011) RD 41 and RD 47 (from Phitsanulok Rice Research Center, harvested during November, 2010). The rice samples were obtained as milled rice. All of the samples were stored at 4°C until analyses.

#### 3.2 Methods

## 3.2.1 Analysis of physicochemical properties and cooking quality of rice

## 3.2.1.1 Chemical analysis

Rice samples were prepared for proximate analyses following the method in AOAC 32.2.01. Moisture (AOAC 32.1.03), fat (AOAC 32.1.13), crude fiber (AOAC 32.1.15), protein (AOAC 32.1.22), and ash (AOAC 32.1.05) content were subsequently determined and carbohydrates were calculated following the AOAC Official Methods (AOAC, 2005). The conversion factor (N×5.95) was applied to convert nitrogen content to the crude protein content (see appendix A1-7).

#### 3.2.1.2 Physical analysis

Rice grains were analyzed for their 1000-kernel weight, length/breadth ratio (L/B) and bulk density following the method of Singh *et al.* (2005) (appendix A8). Color measurement (L\* a\* b\*) was done using a chroma meter (Minolta Chroma meter, model CR 300 series, Japan) (appendix A9). The whiteness index was calculated using equation (1).

Whiteness index = 100 - 
$$\sqrt{(100 - L^*)^2 + (a^*)^2 + (b^*)^2}$$
 (1)

#### 3.2.1.3 Cooking quality analysis

Cooking time, elongation ratio, volume expansion ratio, water uptake ratio, gruel solid loss and cooked length/breadth ratio (L/B) of rice grains were analyzed following the method of Singh *et al.* (2005) as detailed in appendix A10.

### 3.2.1.4 Texture analysis

Hardness, springiness, cohesiveness, adhesiveness, gumminess and chewiness of cooked rice were measured using Texture analyzer (Stable Micro System, model TA-XT2i, UK) with the method modified from that of Park *et al.* (2001) (see appendix A11).

#### 3.2.2 Study on effect of milling on physicochemical properties of rice flour

## 3.2.2.1 Preparation of rice flour

Dry milling process

The polished rice kernels (500 g) were ground twice using a vertical disc mill. Flour samples were passed through a 100-mesh sieve (149  $\mu$ m opening), sealed in Polypropylene (PP) plastic bags and stored in a desiccator at room temperature until further analyses.

### Wet milling process

Rice (1 kg) was soaked overnight in NaHSO<sub>3</sub> solution (1.25% w/v) at rice:solution of 1:2 and ground with a stone-mill under continuous addition of water to obtain a rice slurry. The slurry was filtered through a filter bag to obtain rice cake. The cake was dried overnight in a tray dryer at 40°C. The dried rice flour was ground and sieved through a 100 mesh sifter. Flour samples were packed in PP plastic bags and stored in desiccator at room temperature for further used (Varavinit *et al.*, 2003).

### 3.2.2.2 Chemical analysis

Moisture, fat, protein, ash and carbohydrates content of rice flours were determined following the same procedure in 3.2.1.1. Amylose content was determined by using an iodine binding method (Juliano, 1971) (see appendix A12).

### 3.2.2.3 Physical analysis

## I. Particle size distribution

Particle size distribution was analyzed by using Multi-wavelength Particle Size Analyzer (Beckman Coulter, model LS 13 320, USA) with the method modified from that of Park *et al.* (2010) (see appendix A13).

## II. Color measurement

Color measurement of rice flours was determined following the same procedure in 3.2.1.2.

# III. Morphology observation

Light microscopy and scanning electron microscopy (SEM) were used to examine the morphology of starch granules. Samples were observed using a polarized light microscope (Olympus, model CH30RF200, Japan) (see appendix A14). Rice flours were also investigated using SEM (JEOL, model JSM-5800 LV, Japan) with the procedure of Scientific and Technological Research Equipment Centre (STREC), Chulalongkorn University (see appendix A15).

# IV. X-ray diffraction analysis

X-ray diffraction analysis was performed using an X-ray diffractometer (Bruker AXS, model D8 Discover, Germany) with the procedure of Scientific and Technological Research Equipment Centre (STREC), Chulalongkorn University (see appendix A16).

### V. Water binding capacity

Water binding capacity was determined following the method of Medcalf and Gilles (1965) (see appendix A17).

# VI. Swelling power and solubility

Swelling power and solubility were determined following the method of Medcalf and Gilles (1965) (see appendix A18).

VII. Pasting properties

Pasting properties were measured with a Rapid Visco Analyser (RVA) (Newport Scientific, model 4D, Australia) following the method of Norbert *et al.* (1995) (see appendix A19).

# VIII. Thermal Properties

Thermal properties of rice flours were examined by a Differential Scanning Calorimeter (Perkin-Elmer, model Diamond, USA) following the method of Kim *et al.* (1995) (see appendix A20).

# IX. Viscoelastic properties

Viscoelastic properties were determined by using a rheometer (Bohlin Instrument, model CVOR 150, UK) with the method modified from that of Lii *et al.* (1996) (see appendix A21).

# 3.2.3 Statistical analysis

Completely Randomized Design (CRD) was used as the experimental design. The experiment was performed in 3 replicates. SPSS for Windows program, version 17.0, was employed for analyzing the results. All measurements were done in triplicate, except x-ray diffraction analysis (duplicate), thermal properties (duplicate), and texture analysis (10 replicates). Analysis of variance (ANOVA) and Duncan's new multiple range test (DNMRT) were used for comparing differences in the mean values at 95% confidence level (Montgomery, 2005).

## CHAPTER 4

# **RESULTS AND DISCUSSION**

### 4.1 Physicochemical properties and cooking quality of rice

#### 4.1.1 Chemical analysis

Rice grain consists of moisture, fat, protein, ash, crude fiber and carbohydrates (Table 4.1). Moisture content of the milled rice from different cultivars was found to range between 11.1 to 12.7% (wb). The result showed that the moisture content of all nine varieties met the Thai rice standard (<14%) (Department of Agriculture, 1994) Shaw Lung 97, Prachin Buri 1 and Plai Ngahm Prachin Buri showed the highest moisture content (P  $\leq$  0.05). RD 47 had the lowest moisture content (P  $\leq$  0.05). Fat content varied from 0.31 to 1.86% (db). Khao Dawk Mali 105 showed the highest fat content (P  $\leq$ 0.05) followed by Ayutthaya 1, whereas Plai Ngahm Prachin Buri showed the lowest fat content ( $P \le 0.05$ ). Among nine cultivars, protein content of RD 47 was observed to be the highest (10.7% db) (P  $\leq$  0.05), followed by RD 45 (9.0% db). Shaw Lung 97 and Prachin Buri 1 showed the lowest protein content (6.3 and 6.4% db) ( $P \le 0.05$ ). Ash content of nine different rice cultivars ranged from 0.18 to 0.79% (db). Khao Dawk Mali 105 showed the highest ash content ( $P \le 0.05$ ) followed by Ayutthaya 1, whereas Prachin Buri 1 showed the lowest (P  $\leq$  0.05). Crude fiber of milled rice varied between 0.11 to 0.46% (db). RD 47 showed the highest (P  $\leq$  0.05), whereas Prachin Buri 2, Plai Ngahm Prachin Buri and Ayutthya 1 showed the lowest crude fiber content (P  $\leq$  0.05). Carbohydrates content was observed to be the highest for Shaw Lung 97, Prachin Buri 2 and Plai Ngahm Prachin Buri (92.2-92.5% db) (P ≤ 0.05). RD 47 cultivar showed the lowest carbohydrates content (87.7% db) (P  $\leq$  0.05). The rice cultivars could be classified into two groups; low amylose rice with an amylose content of 17.9-18.4% (RD 45 and Khao Dawk Mali 105) and high amylose rice with an amylose content of 29.5-36.5% (RD 41, RD 47, Shaw Lung 97, Prachin Buri 1, Prachin Buri 2, Plai Ngahm Prachin Buri, and Ayutthaya 1)

Ayabe *et al.* (2009) and Kongseree (1994) demonstrated that the higher protein content especially around the outer layers of rice grains could provide longer cooking time because water penetration into rice grains was interrupted by protein. In addition, the rice with more protein content showed the harder and less sticky cooked rice. Moreover, lipids can form complexes with amylose and amylopectin (Biliaderis and Tonogai, 1991). These complexes are believed to restrict starch granule swelling during heating and prevent leaching of amylose during gelatinization (Saleh and Meullenet, 2005), thereby increasing cooked rice firmness and decreasing stickiness (Seneviratne and Biliaderis, 1991).

### 4.1.2 Physical analysis

The 1000-kernel weight, bulk density and length/breadth (L/B) ratio of the milled rice obtained from various cultivars are showed in Table 4.2. Ayutthaya 1 showed the highest 1000-kernel weight (25.1 g) (P ≤ 0.05) and Shaw Lung 97 had the lowest 1000kernel weight (15.1 g) ( $P \le 0.05$ ). Among nine different cultivars, bulk density of RD 45 was observed to be the highest (0.83 g/ml) ( $P \le 0.05$ ), whereas RD 47 had the lowest bulk density of 0.77 g/ml ( $P \le 0.05$ ). L/B ratio was observed to be the highest for Prachin Buri 1 (2.9) (P  $\leq$  0.05) and RD 41 cultivar showed the lowest L/B (3.9) (P  $\leq$  0.05). The Bureau of Rice Research and Development Rice Department reported that brown rice of RD 41, RD 45, RD 47, Shaw Lung 97, Prachin Buri 2, Prachin Buri 2, Plai Ngahm Prachin Buri, Khao Dawk Mali 105 and Ayutthaya 1 are slender grain. These brown rice cultivars have L/B ratio more than 3 (3.5, 3.2, 3.8, 3.6, 3.1, 3.1, 3.1, 3.6 and 3.3 respectively) (BRRD, 2011). The results of this experiment confirmed that RD 41, RD 45, RD 47, Shaw Lung 97, Prachin Buri 2, Plai Ngahm Prachin Buri, Khao Dawk Mali 105 and Ayutthaya 1 milled rice cultivars are slender grain (L/B ratio more than 3). But Prachin Buri 1 cultivar showed the L/B ratio of 2 to 3 which indicated that the rice cultivar is of the medium grain type.

Cultivars	Moisture	Lipid	Protein	Ash	Fiber	Carbohydrates	Amylose (%
	(% wb)	(% db)	(% db)	(% db)	(% db)	(% db)	of carbohydrates)
RD 41	$12.2^{c} \pm 0.0$	$0.54^{d} \pm 0.03$	$7.9^{d} \pm 0.0$	0.23 <sup>fg</sup> ± 0.02	$0.23^{\circ} \pm 0.03$	91.1 <sup>b</sup> ± 0.1	36.5 <sup>ª</sup> ± 0.1
RD 45	$11.9^{d} \pm 0.1$	$0.60^{d} \pm 0.05$	$9.0^{b} \pm 0.2$	$0.27^{f} \pm 0.01$	$0.20^{\circ} \pm 0.01$	$89.9^{d} \pm 0.2$	$18.4^{f} \pm 0.2$
RD 47	$11.1^{f} \pm 0.0$	$0.94^{\circ} \pm 0.03$	10.7 <sup>ª</sup> ± 0.3	$0.20^{gh} \pm 0.02$	$0.46^{a} \pm 0.04$	87.7 <sup>f</sup> ± 0.3	$34.1^{\circ} \pm 0.5$
Shaw Lung 97	12.7 <sup>a</sup> ± 0.1	$0.48^{d} \pm 0.06$	$6.3^{f} \pm 0.0$	$0.62^{\circ} \pm 0.01$	$0.32^{b} \pm 0.05$	92.3 <sup>a</sup> ± 0.1	29.5 <sup>e</sup> ± 0.5
Prachin Buri 1	$12.7^{a} \pm 0.0$	$0.82^{\circ} \pm 0.03$	$8.3^{\circ} \pm 0.0$	$0.18^{h} \pm 0.03$	$0.20^{\circ} \pm 0.02$	$90.5^{\circ} \pm 0.1$	$32.7^{d} \pm 0.1$
Prachin Buri 2	$11.9^{d} \pm 0.1$	$0.58^{d} \pm 0.03$	$6.4^{f} \pm 0.2$	$0.42^{e} \pm 0.02$	$0.14^{d} \pm 0.02$	$92.5^{a} \pm 0.3$	35.1 <sup>b</sup> ± 0.1
Plai Ngahm Prachin Buri	$12.6^{a} \pm 0.1$	0.31 <sup>e</sup> ± 0.01	$6.9^{e} \pm 0.2$	$0.48^{d} \pm 0.03$	$0.11^{d} \pm 0.02$	$92.2^{a} \pm 0.3$	$33.7^{\circ} \pm 0.1$
Khao Dawk Mali 105	11.5 <sup>e</sup> ± 0.1	1.86 <sup>ª</sup> ± 0.21	$7.9^{d} \pm 0.0$	$0.79^{a} \pm 0.01$	$0.29^{b} \pm 0.04$	89.1 <sup>e</sup> ± 0.3	$17.9^{f} \pm 0.4$
Ayutthaya 1	12.5 <sup>b</sup> ± 0.1	$1.15^{b} \pm 0.09$	7.2 <sup>e</sup> ± 0.2	$0.73^{b} \pm 0.03$	$0.14^{d} \pm 0.01$	$90.8^{bc} \pm 0.3$	$32.7^{d} \pm 0.9$

 Table 4.1 Chemical composition of rice

Mean  $\pm$  standard deviation values in the same column followed by different superscripts are significantly different (P  $\leq$  0.05).

Cultivars	1000-Kernel weight	Length/breadth	bulk density
	(g)	(L/B) ratio	(g/ml)
RD 41	$20.8^{f} \pm 0.2$	$3.9^{a} \pm 0.1$	$0.78^{cd} \pm 0.01$
RD 45	$22.3^{\circ} \pm 0.0$	3.2 <sup>de</sup> ± 0.1	$0.83^{a} \pm 0.03$
RD 47	$22.9^{b} \pm 0.0$	3.7 <sup>b</sup> ± 0.1	$0.77^{d} \pm 0.01$
Shaw Lung 97	$15.1^{h} \pm 0.1$	$3.3^{d} \pm 0.1$	$0.79^{bcd} \pm 0.01$
Prachin Buri 1	$22.8^{b} \pm 0.1$	$2.9^{f} \pm 0.0$	$0.80^{bc} \pm 0.02$
Prachin Buri 2	21.1 <sup>e</sup> ± 0.0	3.1 <sup>e</sup> ± 0.0	$0.81^{ab} \pm 0.02$
Plai Ngahm Prachin Buri	$22.1^{d} \pm 0.0$	$3.3^{d} \pm 0.1$	$0.78^{cd} \pm 0.00$
Khao Dawk Mali 105	18.7 <sup>g</sup> ± 0.1	$3.4^{\circ} \pm 0.1$	$0.78^{cd} \pm 0.01$
Ayutthaya 1	25.1 <sup>ª</sup> ± 0.1	$3.3^{d} \pm 0.1$	$0.79^{bcd} \pm 0.01$

Table 4.2 Physical properties of milled rice

Mean ± standard deviation values in the same column followed by different superscripts are significantly different ( $P \le 0.05$ )

#### 4.1.3 Cooking quality analysis

Cooking quality of rice shows in Figure 4.1 and Table 4.3. Cooking time of milled rice from nine cultivars varied from 18 to 30 min. Prachin Buri 2, Plai Ngahm Prachin Buri and Ayutthaya 1 needed the longest cooking time (30 min) while Khao Dawk Mali 105 required the lowest cooking time of 18 min. The elongation ratio of cooked rice varied from 1.44 for Ayutthaya 1 to 1.60 for Shaw Lung 97. The result showed that the elongation ratio was not significantly different among these nine different rice cultivars. Volume expansion ratio was the highest in Shaw Lung 97 (4.5 g cooked rice/g milled rice) (P  $\leq$  0.05) and the lowest in Khao Dawk Mali 105 (3.0 g cooked rice/g milled rice) (P  $\leq$  0.05). The water uptake ratio ranged from 2.5 to 4.5 g water/g milled rice. Khao Dawk Mali 105 showed the lowest water uptake ratio, whereas Shaw Lung 97 showed the highest (P  $\leq$  0.05). The gruel solid loss percentage ranged from 3.0 to 6.0% of milled rice. Shaw Lung 97, Plai Ngahm Prachin Buri and Ayutthaya 1 showed the highest gruel solid loss (P  $\leq$  0.05). Prachin Buri 1 showed the lowest gruel

solid loss (P  $\leq$  0.05). Cooked length/breadth ratio was the lowest in Prachin Buri 1 (3.1) (P  $\leq$  0.05) and the highest in RD 47 (4.0).

Dipti *et al.* (2002) studied the physicochemical properties of round (L/B ratio less than 2) and slender (L/B ratio more than 3) grain rice and found that elongation ratio is an important parameter for cooked rice. If rice elongates more lengthwise it gives a finer appearance and if expands widthwise, it gives a coarse look. In addition, Elongation of cooked rice had positive correlation with L/B ratio. Basmati type cultivars (long slender grain) having higher L/B ratios than medium and slender grain, showed greater elongation ratios (Singh *et al.*, 2005). However, this study could not find differences between elongation ratio of nine different rice cultivars

During the cooking process, amylose leaches into cooking water. The amount of leached amylose depends on the amount of cell wall disruption occurring during cooking (Ogawa et al., 2003). Usually, rice with higher amylose content yields higher solid loss during cooking (Singh et al., 2005). Moreover, protein content can also affect amylose leaching. Higher protein content can inhibit water absorption and solid loss (Ayabe et al., 2009; Kongseree, 1994). In this study, the cultivars with higher amylose content showed greater gruel solids loss except Prachin Buri 1. This could be attributed to higher protein content in the Prachin Buri 1 cultivar (8.3% db). The protein content also exerted its effect on gruel solid loss of Shaw Lung 97. The Shaw Lung 97 cultivar has the lowest amylose content (29.5%) in the high amylose content rice group, yet showing the highest gruel solid loss (6.0%). This could be due to its low protein content (6.3%). It is observed that the rice cultivars with higher bulk density also required longer cooking time. This could be owing to the fact that rice with higher bulk density has compact structure and less void resulting in slower water uptake. In contrast, Singh et al. (2005) stated that the cultivars with higher amylose content required less cooking time. However, the research of Dipti et al. (2002) could not find a relationship between cooking time and amylose content, it may be because all of these varieties are non-waxy types. Similar results were found for this research.

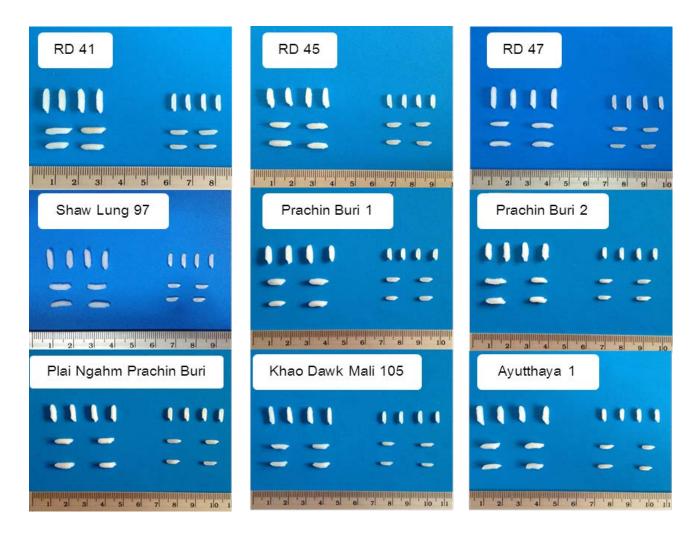


Figure 4.1 Rice Grains of nine rice cultivars (left: cooked rice grains and right: raw rice grains)

Cultivars	Cooking	Elongation	Volume expansion	Water uptake	Gruel solid loss	Cooked
	time (min)	ratio <sup>ns</sup>	ratio (g cooked rice/	Ratio (g water/	(% of milled rice)	length/breadth
			g milled rice)	g milled rice)		ratio
RD 41	23	1.50 ± 0.04	$3.6^{bc} \pm 0.2$	3.1 <sup>de</sup> ± 0.1	$4.7^{cd} \pm 0.1$	$3.8^{b} \pm 0.1$
RD 45	25	1.46 ± 0.04	$3.4^{\circ} \pm 0.1$	$3.2^{cd} \pm 0.2$	$4.6^{\circ} \pm 0.4$	$3.4^{\circ} \pm 0.1$
RD 47	24	1.50 ± 0.05	3.5 <sup>bc</sup> ± 0.1	2.7 <sup>ef</sup> ± 0.0	$4.9^{bc} \pm 0.0$	$4.0^{a} \pm 0.3$
Shaw Lung 97	26	1.60 ± 0.06	$4.5^{a} \pm 0.2$	$4.5^{a} \pm 0.3$	$6.0^{a} \pm 0.5$	3.6 <sup>bc</sup> ± 0.1
Prachin Buri 1	23	1.49 ± 0.08	3.5 <sup>bc</sup> ± 0.1	2.9 <sup>de</sup> ± 0.2	$3.0^{e} \pm 0.0$	$3.1^{d} \pm 0.2$
Prachin Buri 2	30	1.51 ± 0.05	$3.5^{bc} \pm 0.1$	$4.3^{a} \pm 0.4$	$5.3^{ab} \pm 0.5$	$3.4^{\circ} \pm 0.1$
Plai Ngahm Prachin Buri	30	1.47 ± 0.11	$3.4^{bc} \pm 0.0$	$3.5^{\circ} \pm 0.0$	$5.6^{a} \pm 0.6$	$3.4^{\circ} \pm 0.1$
Khao Dawk Mali 105	18	1.46 ± 0.01	$3.0^{d} \pm 0.1$	$2.5^{f} \pm 0.2$	$3.8^{d} \pm 0.3$	$3.8^{b} \pm 0.1$
Ayutthaya 1	30	1.44 ± 0.07	$3.7^{b} \pm 0.4$	$3.9^{b} \pm 0.2$	$5.9^{a} \pm 0.3$	$3.4^{\circ} \pm 0.1$

Table 4.3 Cooking quality of rice

Mean  $\pm$  standard deviation values in the same column followed by different superscripts are significantly different (P  $\leq$  0.05)

ns = not significantly difference

#### 4.1.4 Texture analysis

The textural characteristics of cooked rice from different rice cultivars are shown in Table 4.4. The result showed that the hardness of cooked rice ranged from 227.6 to 602.2 g, springiness varied from 0.32 to 0.58, cohesiveness ranged from 0.10 to 0.19, adhesiveness varied from 28.5 to 117.5 (g·sec), gumminess ranged between 23.4 to 105.3 g, and chewiness ranged from 7.8 to 61.6 g. The result showed that hardness, cohesiveness, adhesiveness, and chewiness of cooked RD 45, Shaw Lung 97, and Khao Dawk Mali 105 were significantly higher than other cultivars (P  $\leq$  0.05). These textural parameters had a negative correlation with amylose content. RD 45 and Khao Dawk Mali 105 were classified as low amylose rice, whereas Shaw Lung 97 was classified as high amylose rice. Nevertheless, Shaw Lung 97 has lower amylose content than other cultivars in the high amylose rice group. Usually, low amylose cooked rice has a soft and sticky texture with the grain clumping together, resulting in low hardness of a single rice grain and high values of cohesiveness, adhesiveness, and chewiness. However in this study, since the texture analysis was done on cylindrical rice bulk, the hardness of the sample reflects the force needed to separate the rice bulk rather than to crush a single grain. As a result, the hardness of rice bulk with low amylose content is high. It is the opposite for high amylose cooked rice cultivar. When cooked the rice has a dry and fluffy texture with the grains separate (Webb et al., 1986). While the texture probe contacts the sample, the rice grains sample was separated easily resulting in low value of hardness, cohesiveness, adhesiveness, and chewiness.

Cultivars	Hardness (g)	Springiness	Cohesiveness	Adhesiveness	Gumminess (g)	Chewiness (g)
				(g•sec)		
RD 41	$354.7^{b} \pm 44.2$	$0.42^{b} \pm 0.01$	$0.12^{b} \pm 0.00$	38.4 <sup>b</sup> ± 6.1	$41.4^{b} \pm 5.0$	17.6 <sup>b</sup> ± 3.0
RD 45	519.9 <sup>ª</sup> ± 55.8	$0.51^{a} \pm 0.02$	$0.17^{a} \pm 0.01$	117.5 <sup>ª</sup> ± 23.0	88.8 <sup>ª</sup> ± 15.7	46.0 <sup>°</sup> ± 10.1
RD 47	392.5 <sup>b</sup> ± 39.5	0.39 <sup>b</sup> ±0.02	$0.12^{b} \pm 0.01$	41.7 <sup>bc</sup> ± 3.8	$47.9^{b} \pm 8.0$	19.2 <sup>b</sup> ± 3.7
Shaw Lung 97	521.9 <sup>ª</sup> ± 74.7	$0.55^{a} \pm 0.03$	$0.19^{a} \pm 0.02$	100.3 <sup>ª</sup> ± 7.3	100.8 <sup>ª</sup> ± 21.7	57.1 <sup>ª</sup> ± 15.7
Prachin Buri 1	227.6 <sup>°</sup> ± 17.7	$0.32^{\circ} \pm 0.02$	$0.10^{b} \pm 0.01$	$28.5^{\circ} \pm 5.9$	$23.4^{b} \pm 4.4$	$7.8^{b} \pm 2.0$
Prachin Buri 2	$326.7^{b} \pm 53.4$	$0.43^{b} \pm 0.08$	$0.13^{b} \pm 0.02$	$42.4^{bc} \pm 7.4$	43.4 <sup>b</sup> ± 13.8	19.8 <sup>b</sup> ± 10.1
Plai Ngahm Prachin Buri	355.4 <sup>b</sup> ± 36.8	$0.44^{b} \pm 0.04$	0.11 <sup>b</sup> ± 0.01	58.9 <sup>b</sup> ± 2.5	$40.7^{b} \pm 8.5$	18.3 <sup>b</sup> ± 2.6
Khao Dawk Mali 105	$602.2^{a} \pm 39.9$	$0.58^{a} \pm 0.06$	$0.17^{a} \pm 0.01$	113.1 <sup>ª</sup> ± 18.8	105.3 <sup>ª</sup> ± 14.0	61.6 <sup>ª</sup> ± 13.5
Ayutthaya 1	$336.9^{b} \pm 61.9$	$0.38^{bc} \pm 0.03$	$0.12^{b} \pm 0.02$	54.2 <sup>b</sup> ± 22.8	42.1 <sup>b</sup> ± 13.9	$16.4^{b} \pm 6.2$

Table 4.4 Textural properties of cooked rice samples

Mean  $\pm$  standard deviation values in the same column followed by different superscripts are significantly different (P  $\leq$  0.05).

#### 4.2 Effect of milling on physicochemical properties of rice flours

#### 4.2.1 Chemical analysis

The dry milled flours contained 9.2%-12.0% (wb) moisture content, 0.38%-1.01% (db) fat, 5.8%-9.2% (db) protein, 0.45%-0.92% (db) ash, 88.9%-93.2% (db) carbohydrates (Table 4.5) and the starch consisted of 21.0%-41.1% apparent amylose (Table 4.6). The wet milled flours contained 8.2%-11.9% (wb) moisture content, 0.20%-0.83% (db) fat, 5.0%-7.8% (db) protein, 0.19%-0.27% (db) ash, 91.7%-94.6% (db) carbohydrates (Table 4.5) and the starch consisted of 21.0%-41.1% apparent amylose (Table 4.6). In the wet-milling process, rice kernels had to be soaked and ground with water. Soluble protein, which is albumin, was removed, resulting in lower protein content in wet-milled rice flours (Suksomboon and Naivikul, 2006). In combination with the removal of protein and other soluble components during wet milling process, the content of total carbohydrates of wet-milled rice flours was significantly higher than those of dry-milled rice flours (P  $\leq$  0.05).

The rice flour samples could be classified into two groups; low amylose rice with an amylose content of 13.1-18.7% (RD 45 and Khao Dawk Mali 105) and high amylose rice with an amylose content of 29.4-36.5% (RD 41, RD 47, Shaw Lung 97, Prachin Buri 1, Prachin Buri 2, Plai Ngahm Prachin Buri, and Ayutthaya 1). Dry-milled rice flours tended to possess higher amylose content than wet-milled rice flours. Dry milling process might result in the breakdown of amylopectin to low molecular weight fragmented amylopectin. The low molecular weight fragmented amylopectin has a molecular weight close to that of amylose so it can form complex with iodine. Suksomboon and Naivikul (2006) found that starches from dry-milled samples contained smaller amounts of high molecular weight fractions (amylopectin) but greater amounts of low molecular weight fractions (amylose) than those from wet-milled starch samples.

Cultivars	Moisture	Lipid	Protein	Ash	Carbohydrates
Cultivars	(% wb)	(% db)	(% db)	(% db)	(%db)
RD41(D)	10.1 <sup>fg</sup> ± 0.1	$0.53^{gh} \pm 0.05$	7.3 <sup>de</sup> ± 0.2	$0.57^{e} \pm 0.00$	91.6 <sup>g</sup> ± 0.2
RD41(W)	$8.7^{ij}$ ± 0.3	$0.83^{e} \pm 0.04$	$6.7^{fg} \pm 0.0$	$0.27^{g} \pm 0.01$	$92.2^{ef} \pm 0.0$
RD45(D)	$11.4^{bc} \pm 0.4$	$0.67^{f} \pm 0.09$	$8.6^{b} \pm 0.3$	$0.79^{b} \pm 0.02$	89.9 <sup>i</sup> ± 0.3
RD45(W)	$8.9^{hi}$ ± 0.0	$0.61^{fg} \pm 0.05$	$7.5^{cd} \pm 0.0$	$0.24^{h} \pm 0.01$	91.7 <sup>g</sup> ± 0.1
RD47(D)	$9.7^9$ ± 0.4	0.99 <sup>d</sup> ±0.03	$9.2^{a} \pm 0.4$	$0.91^{a} \pm 0.03$	$88.9^{j} \pm 0.4$
RD47(W)	$8.3^{jk} \pm 0.3$	$0.24^{j} \pm 0.02$	$7.8^{\circ} \pm 0.3$	$0.24^{h} \pm 0.01$	91.7 <sup>g</sup> ± 0.3
SL97(D)	12.0 <sup>a</sup> ± 0.1	$0.38^{i} \pm 0.03$	$5.8^{jk} \pm 0.0$	$0.60^{d} \pm 0.01$	93.2 <sup>cd</sup> ± 0.1
SL97(W)	$8.2^{k} \pm 0.2$	$0.20^{j} \pm 0.03$	$5.0^{1} \pm 0.0$	$0.23^{h} \pm 0.01$	$94.6^{a} \pm 0.0$
PB1(D)	10.8 <sup>de</sup> ± 0.3	1.00 <sup>°</sup> ± 0.11	$6.4^{ghi} \pm 0.2$	$0.75^{\circ} \pm 0.03$	$91.9^{fg} \pm 0.4$
PB1(W)	$8.2^{jk} \pm 0.1$	$0.24^{j} \pm 0.03$	$5.7^{k} \pm 0.2$	$0.23^{h} \pm 0.01$	$93.8^{b} \pm 0.2$
PB2(D)	$9.2^{h} \pm 0.2$	$0.60^{fg} \pm 0.04$	$5.9^{jk} \pm 0.4$	$0.45^{f} \pm 0.02$	$93.1^{cd} \pm 0.4$
PB2(W)	$9.0^{hi}$ ± 0.3	$0.45^{hi} \pm 0.04$	$5.3^{l} \pm 0.2$	$0.19^{i} \pm 0.01$	94.1 <sup>b</sup> ± 0.2
PNG(D)	$11.6^{ab} \pm 0.0$	$0.57^{g} \pm 0.09$	$6.4^{ghi} \pm 0.2$	$0.56^{e} \pm 0.01$	92.5 <sup>e</sup> ± 0.1
PNG(W)	$11.0^{cd} \pm 0.2$	$0.43^{hi} \pm 0.03$	$5.9^{jk} \pm 0.2$	$0.22^{h} \pm 0.00$	$93.4^{\circ} \pm 0.2$
K105(D)	11.2 <sup>bcd</sup> ± 0.1	$0.97^{d} \pm 0.10$	$7.0^{ef} \pm 0.3$	$0.92^{a} \pm 0.03$	$91.2^{h} \pm 0.4$
K105(W)	$11.9^{a} \pm 0.5$	$0.24^{j} \pm 0.02$	$6.2^{hij} \pm 0.0$	$0.24^{h} \pm 0.01$	$93.3^{cd} \pm 0.0$
AY1(D)	$11.1^{cd} \pm 0.2$	$1.01^{\circ} \pm 0.03$	$6.6^{\text{fgh}} \pm 0.1$	$0.77^{\rm bc} \pm 0.01$	91.6 <sup>g</sup> ± 0.1
AY1(W)	10.5 <sup>ef</sup> ± 0.2	$0.69^{f} \pm 0.04$	$6.1^{ijk} \pm 0.3$	$0.23^{h} \pm 0.00$	$93.0^{d} \pm 0.3$

 Table 4.5 Chemical composition of rice flours

Mean ± standard deviation values in the same column followed by different superscripts are

significantly different (P  $\leq$  0.05)

(D) = flour from dry milling method, (W) = flour from wet milling method

SL97 = Shaw Lung 97, PB1 = Prachin Buri 1, PB2 = Prachin Buri 2, PNG = Plai Ngahm Prachin Buri,

K105 = Khao Dawk Mali 105, AY1 = Ayutthaya 1

Cultivars —	Amylose content (% of carbohydrates)			
Guilivais	Dry-milled flour	Wet-milled flour		
RD 41	$36.5^{a} \pm 0.1$	$32.9^{de} \pm 0.2$		
RD 45	$18.4^{ij} \pm 0.2$	$18.7^{i} \pm 0.6$		
RD 47	$34.1^{\circ} \pm 0.5$	35.7 <sup>b</sup> ± 0.2		
Shaw Lung 97	$29.5^{fg} \pm 0.5$	$29.4^{9} \pm 0.9$		
Prachin Buri 1	32.7 <sup>de</sup> ± 0.1	$30.2^{f} \pm 0.2$		
Prachin Buri 2	$35.1^{b} \pm 0.1$	32.6 <sup>e</sup> ± 0.3		
Plai Ngahm Prachin Buri	$33.7^{\circ} \pm 0.1$	$33.4^{cd} \pm 0.4$		
Khao Dawk Mali 105	$17.9^{j} \pm 0.4$	$13.1^{k} \pm 0.4$		
Ayutthaya 1	32.7 <sup>de</sup> ± 0.9	$28.0^{h} \pm 0.2$		

 Table 4.6 Amylose content of rice flours

Mean  $\pm$  standard deviation values followed by different superscripts are significantly different (P  $\leq$  0.05)

### 4.2.2 Physical analysis

#### 4.2.2.1 Particle size distribution

The number average particle size of dry-milled and wet-milled rice flours were determined by laser particle size analyzer (Table 4.7). The data shows that the average particle size of dry-milled flours were in the range of 66.1 to 87.2  $\mu$ m and they were significantly (p ≤ 0.05) higher than those of wet-milled flours which varied in the range of 19.8 to 56.9  $\mu$ m. This was because rice starch appeared in nature as compound granules. In dry-milling process, the protein matrix and other substances that cause aggregation of starch granules were not removed. Most starch granules were, therefore, remained in their native form as large aggregates (Figure 4.2). On the contrary, starch granules in wet-milled samples were mostly separated as individual granules because, during the soaking process of wet-milling rice flour, some soluble protein, sugars, and non-starch bound lipids were washed out from the surface of starch

granules, causing the structure of starchy endosperm to become loosen which resulted in the fine particles and less damaged starch (Patindol and Wang, 2002; Chen *et al.*, 2003; Suksomboon and Naivikul, 2006).

Figure 4.4 shows that wet-milled flour expressed three major peaks when analyzed for its particle size distribution. The first, the peak at smaller size range (0.5 to 2  $\mu$ m) could possibly be the remnant of damaged starch granule or some water insoluble protein particles that remained from the wet milling process (the red circles in figure 4.19). The second peak, which covered the 3-20  $\mu$ m size range, could represent separated rice starch granules. Bemiller and Whistler (2009) demonstrated that the granule size of rice starch is around 2-10  $\mu$ m. The last peak governing the 20 to 150  $\mu$ m size range could represent starch granules aggregated. When soaking the flour samples overnight in alcohol, the average particle size were decreased (Table 4.3, 4.5). This could be because some alcohol soluble proteins (prolamin) might be dissolved resulting in the reduction of the number of starch aggregates.

Cultivars	Avera	age	Мес	lian	Mode	
Cultivars	Dry flour	Alcohol	Dry flour	Alcohol	Dry flour	Alcohol
RD41(D)	81.8 <sup>bc</sup> ± 0.7	31.5 <sup>h</sup> ± 0.1	78.8	24.2	127.6	41.7
RD41(W)	40.6 <sup>i</sup> ± 1.1	27.7 <sup>i</sup> ± 0.3	13.0	10.0	7.8	7.1
RD45(D)	$86.2^{a} \pm 0.4$	$66.7^{b} \pm 0.4$	84.9	62.4	127.6	87.9
RD45(W)	56.9 <sup>g</sup> ± 0.5	$27.2^{j} \pm 0.3$	40.0	10.9	116.3	7.8
RD47(D)	$80.2^{\circ} \pm 0.5$	57.9 <sup>°</sup> ± 0.1	77.3	50.0	127.6	87.9
RD47(W)	26.5 <sup>1</sup> ± 0.2	$24.9^{k} \pm 0.2$	9.7	8.8	7.8	7.1
SL97(D)	83.2 <sup>b</sup> ± 0.3	73.1 <sup>ª</sup> ± 0.3	79.8	68.8	127.6	87.9
SL97(W)	$38.0^{j} \pm 0.6$	18.6 <sup>°</sup> ± 0.2	10.7	7.6	8.5	7.8
PB1(D)	$76.2^{d} \pm 0.8$	$34.4^{g} \pm 0.0$	73.0	24.0	127.6	21.7
PB1(W)	$23.5^{m} \pm 0.2$	$19.3^{m} \pm 0.3$	8.5	7.5	7.8	7.1
PB2(D)	$66.1^{f} \pm 0.4$	$34.2^{g} \pm 0.2$	57.8	26.2	105.9	23.8
PB2(W)	$31.4^{k} \pm 0.6$	$22.2^{1} \pm 0.2$	11.1	9.1	9.4	8.5
PNG(D)	80.5 <sup>°</sup> ± 0.5	$53.2^{d} \pm 0.2$	77.1	46.1	116.3	66.4
PNG(W)	$22.7^{m} \pm 0.4$	19.1 <sup>mn</sup> ± 0.1	8.9	7.7	8.5	7.8
K105(D)	$87.2^{a} \pm 0.6$	$35.2^{f} \pm 0.2$	85.3	27.8	127.6	45.8
K105(W)	19.8 <sup>°</sup> ± 0.1	18.9 <sup>no</sup> ± 0.1	9.2	7.3	8.5	6.5
AY1(D)	$68.3^{e} \pm 0.4$	41.1 <sup>e</sup> ± 0.2	60.5	35.2	105.9	55.1
AY1(W)	$42.9^{h} \pm 5.6$	$24.6^{k} \pm 0.2$	12.5	8.6	9.4	7.8

Table 4.7 Particle size  $(\mu\text{m})$  of dry-milled and wet-milled flours

Mean ± standard deviation values in the same column followed by different superscripts are

significantly different ( $P \le 0.05$ )

Alcohol = flour sample soaked overnight with alcohol

(D) = flour from dry milling method, (W) = flour from wet milling method

SL97 = Shaw Lung 97, PB1 = Prachin Buri 1, PB2 = Prachin Buri 2, PNG = Plai Ngahm Prachin Buri,

K105 = Khao Dawk Mali 105, AY1 = Ayutthaya 1

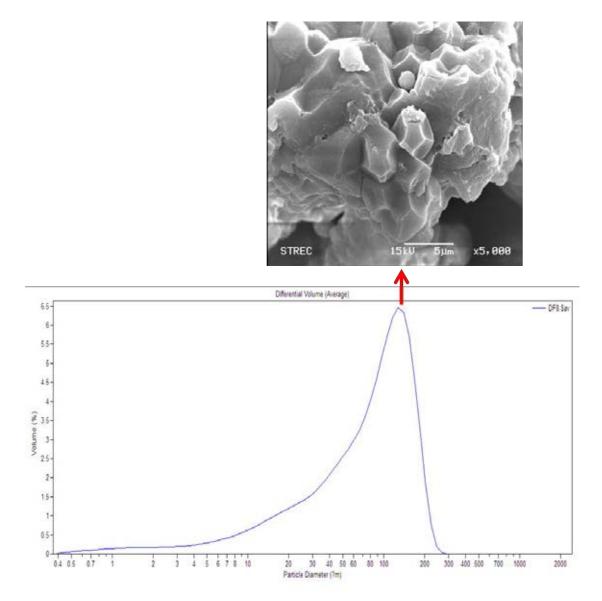


Figure 4.2 Selected particle size distribution of Khao Dawk Mali 105 cultivar in dry-milled rice flours

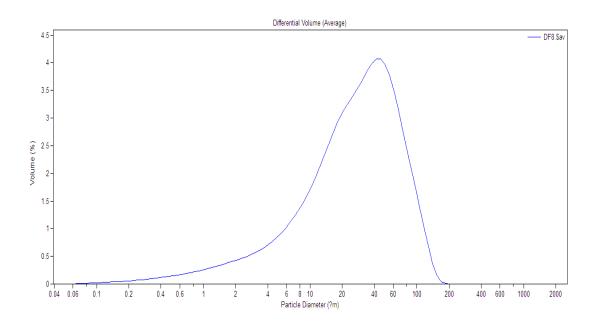


Figure 4.3 Selected particle size distribution of Khao Dawk Mali 105 cultivar in dry-milled rice flours (soaked overnight in alcohol)

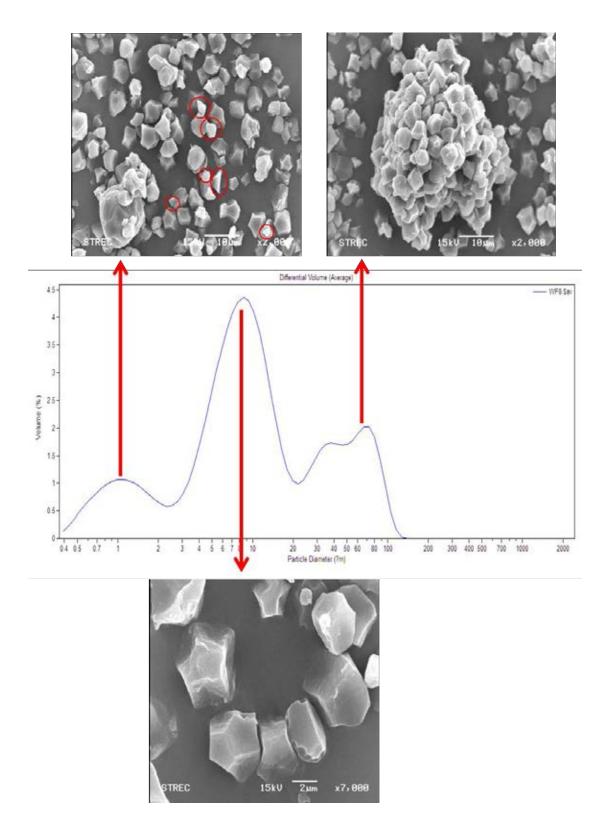


Figure 4.4 Selected particle size distribution of Khao Dawk Mali 105 cultivar in wetmilled rice flours

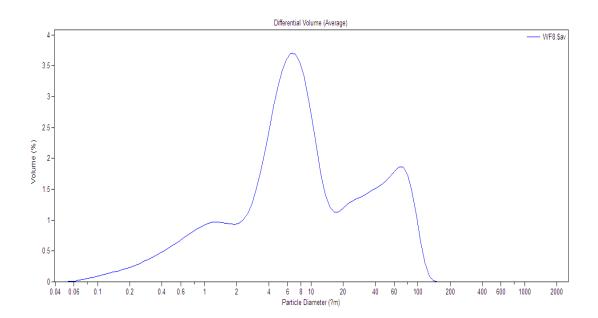


Figure 4.5 Selected particle size distribution of Khao Dawk Mali 105 cultivar in wetmilled rice flours (soaked overnight in alcohol)

## 4.2.2.2 Color measurement

Whiteness index of rice grains, dry-milled flours and wet-milled flours calculated from L\*, a\* and b\* values are shown in Table 4.8. The results showed that the whiteness index of rice grains were from 47.25 for RD 47 to 56.78 for Shaw Lung 97, 75.68 for RD 47 to 78.04 for Plai Ngahm Prachin Buri in dry-milled flours and 77.32 for RD45 to 79.99 in wet-milled flours. For all rice varieties, the whiteness index of rice grains was significantly lower than those of dry-milled and wet-milled flours ( $p \le 0.05$ ). This may be explained by a dilution effect. Grinding of rice kernels results in a mixture of a small dark, colored fraction, which could be the remnant of bran and outer endosperm, and a large light, less pigmented fraction (middle and core endosperm) (Lamberts *et al.*, 2007) (Figure 4.6). Moreover, Patindol and Wang (2002) stated that the sample particle size also affected the color, and that the finer the flours, the brighter and whiter their color. The result from laser particle size analyzer (Table 4.7) showed that dry-milled flours. This reason corresponded well with the results that the wet-milled flours tend to have higher whiteness index values than dry-milled flours.

Cultivars	Whiteness index					
Cullivars	Rice grains	Dry-milled flour	Wet-milled flour			
RD 41	50.3°± 1.1	77.1 <sup>fgh</sup> ± 0.0	$78.4^{d} \pm 0.4$			
RD 45	$48.9^{p} \pm 0.2$	77.0 <sup>gh</sup> ± 0.1	$77.3^{efg} \pm 0.6$			
RD 47	$47.3^{q} \pm 0.5$	$75.7^{i} \pm 0.5$	$79.2^{ab} \pm 0.5$			
Shaw Lung 97	$56.8^{j} \pm 0.4$	77.0 <sup>gh</sup> ± 0.1	79.1 <sup>°</sup> ± 0.2			
Prachin Buri 1	$50.6^{\circ} \pm 0.6$	77.4 <sup>efg</sup> ± 0.2	79.9 <sup>ab</sup> ± 0.1			
Prachin Buri 2	$53.2^{m} \pm 0.7$	77.8 <sup>def</sup> ± 0.1	79.3 <sup>abc</sup> ± 0.1			
Plai Ngahm Prachin Buri	$54.2^{1} \pm 0.2$	$78.0^{de} \pm 0.2$	$79.7^{abc} \pm 0.2$			
Khao Dawk Mali 105	$51.4^{n} \pm 0.5$	$76.5^{h} \pm 0.3$	$80.0^{a} \pm 0.0$			
Ayutthaya 1	$55.4^{k} \pm 0.8$	77.6 <sup>efg</sup> ± 0.1	$79.7^{abc} \pm 0.0$			

Table 4.8 Whiteness index values of rice grains and flours

Mean ± standard deviation values followed by different superscripts are significantly different

(P ≤ 0.05)

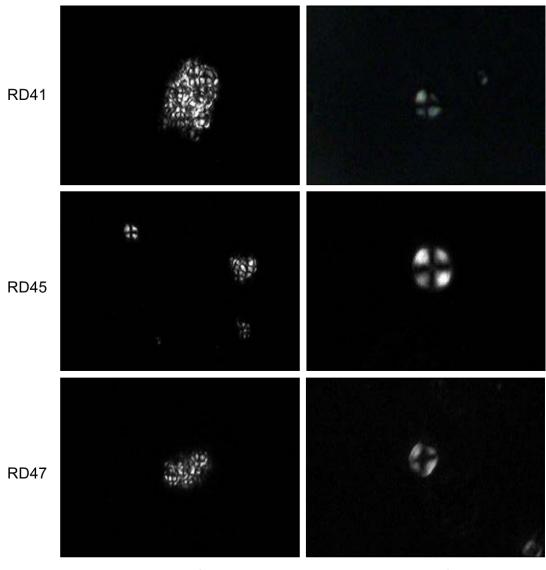


Figure 4.6 Rice grains (left), dry-milled flours (middle) and wet-milled flours (right) of different rice cultivars

#### 4.2.2.3 Morphology observation

Polarized light micrographs of flours are shown in Figures 4.7 and 4.8. Wetmilled and dry-milled rice starch granules from nine different cultivars showed birefringence. The refraction of polarized light by the intact crystalline regions in starch gives characteristic "Maltese cross" patterns on each granule. The results indicated that dry-milling disrupted the ordered structure of the starch granule causing the loss of birefringence in some part of dry-milled flours (the red circle in Figure 4.9). Similar result was found for the research of Chen *et al.* (2003), who indicated that the starch granules significantly lost their birefringence as ball-milled treatment time prolonged.

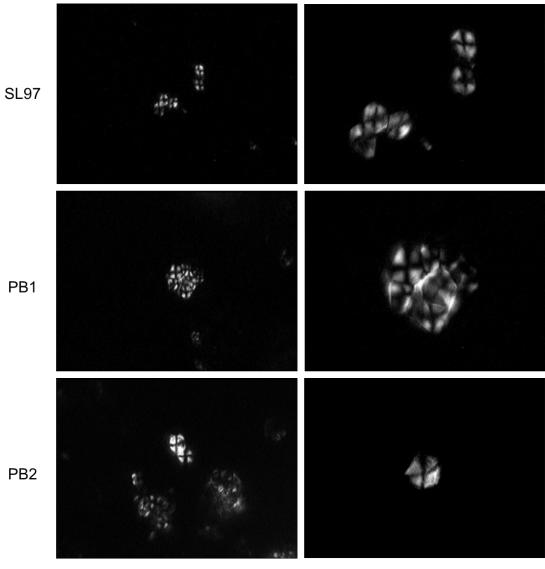
Scanning electron micrographs of dry-milled and wet-milled rice flours are shown in Figures 4.10 and 4.11. The result indicated that wet-milled and dry-milled rice starch granules are polygonal but irregular in shape. Dry milled rice flour contained significantly larger flour particles in the form of aggregated starch granules compared to the separated starch granules of wet-milled samples. Suksomboon and Naivikul (2006) stated that during the soaking process of wet-milling rice flour, protein matrix and other substances were leached out from the surface of starch granules, causing the structure of starchy endosperm to become loosen which resulted in the fine particles and less damaged starch.



40X magnification

100X magnification

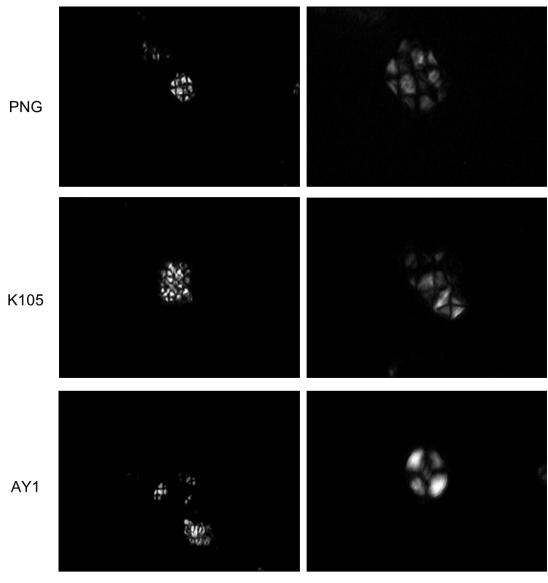
Figure 4.7 Polarized light micrographs of dry-milled flours from nine different rice cultivars at different magnifications



40X magnification

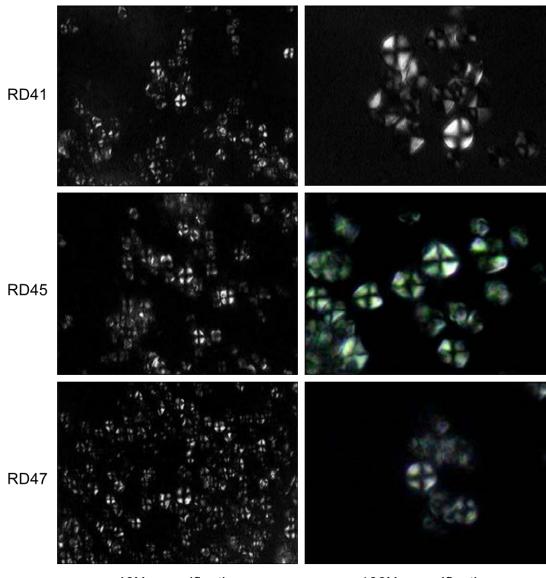
100X magnification

**Figure 4.7 (continued)** Polarized light micrographs of dry-milled flours from nine different rice cultivars at different magnifications (SL97 = Shaw Lung 97, PB1 = Prachin Buri 1, PB2 = Prachin Buri 2)



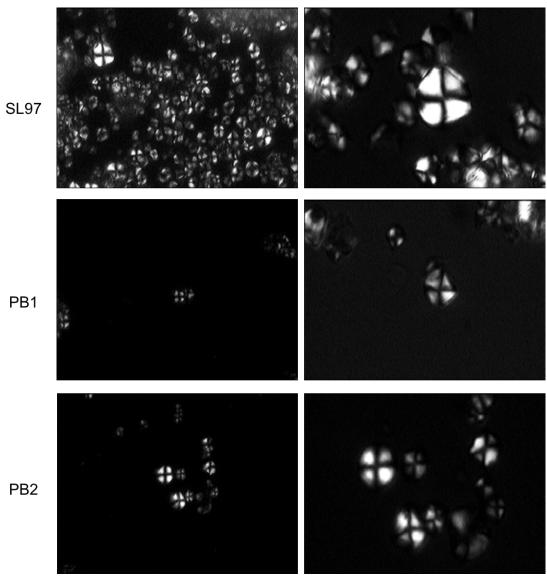
40X magnification

**Figure 4.7 (continued)** Polarized light micrographs of dry-milled flours from nine different rice cultivars at different magnifications (PNG = Plai Ngahm Prachin Buri, K105 = Khao Dawk Mali 105, AY1 = Ayutthaya 1)



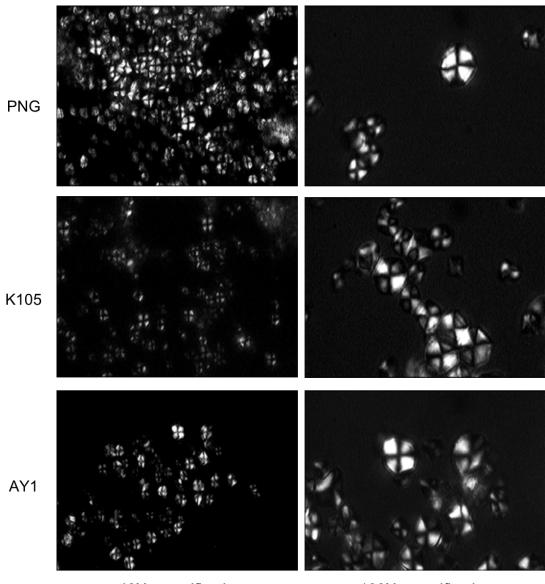
40X magnification

Figure 4.8 Polarized light micrographs of wet-milled flours from nine different rice cultivars at different magnifications



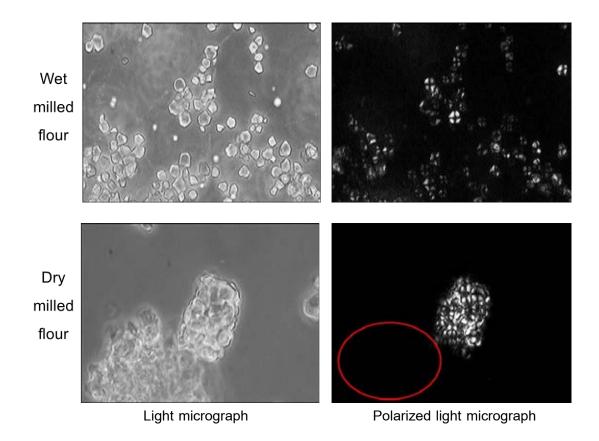
40X magnification

**Figure 4.8 (continued)** Polarized light micrographs of wet-milled flours from nine different rice cultivars at different magnifications (SL97 = Shaw Lung 97, PB1 = Prachin Buri 1, PB2 = Prachin Buri 2)



40X magnification

**Figure 4.8 (continued)** Polarized light micrographs of wet-milled flours from nine different rice cultivars at different magnifications (PNG = Plai Ngahm Prachin Buri, K105 = Khao Dawk Mali 105, AY1 = Ayutthaya 1)



**Figure 4.9** Selected normal and polarized light micrographs of RD 41 wet-milled rice flour and dry-milled rice flour (magnification 40X)

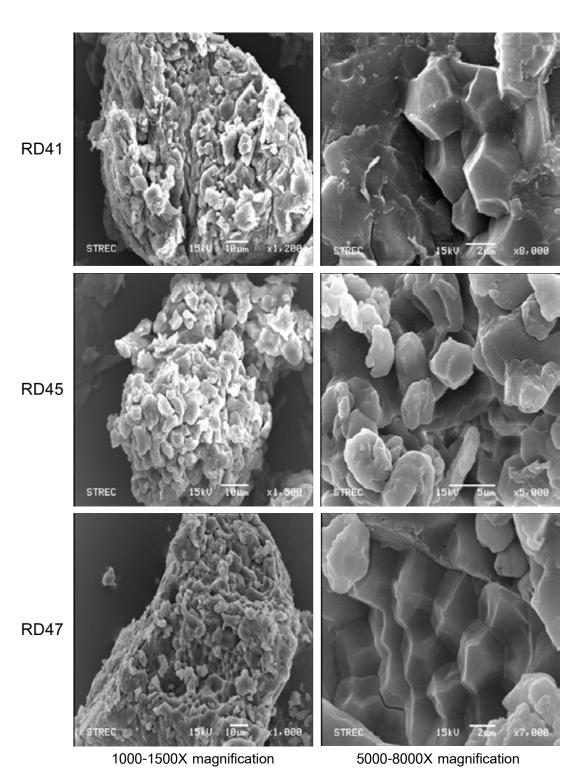
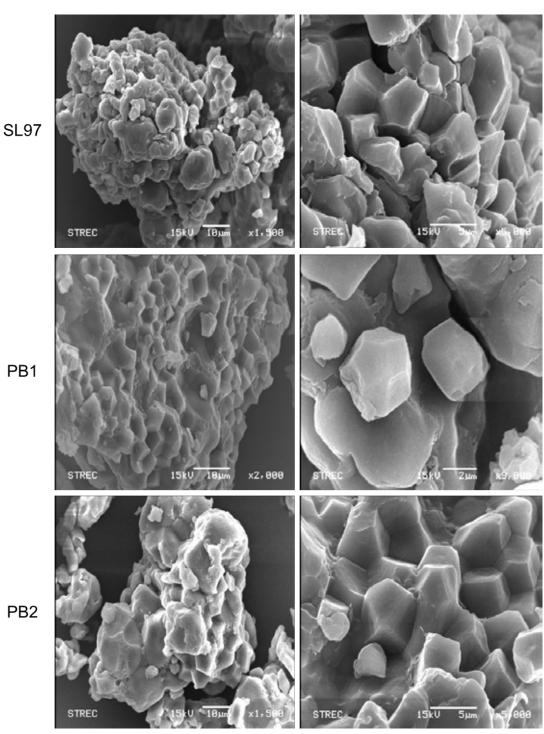


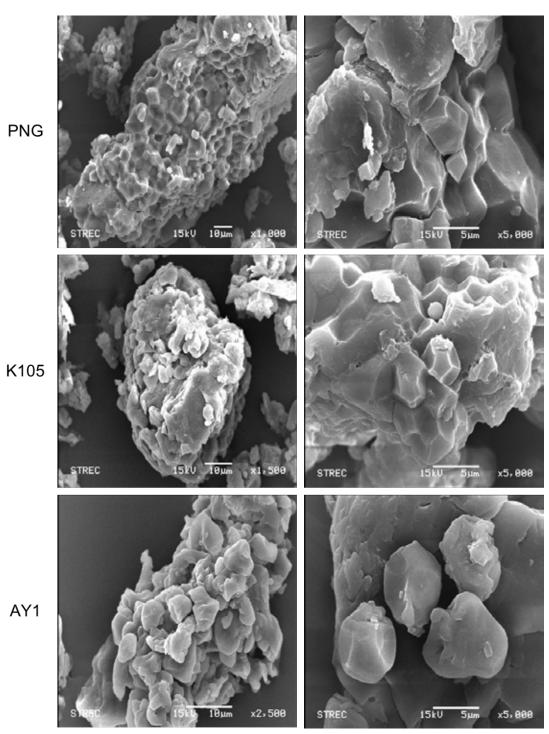
Figure 4.10 Scanning electron micrographs of dry-milled flours from nine different rice cultivars at different magnifications



1500-2000X magnification

5000-9000X magnification

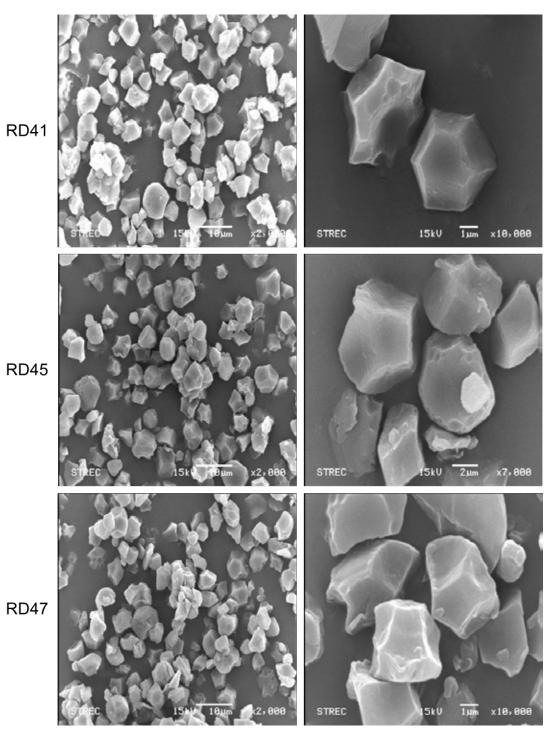
**Figure 4.10 (continued)** Scanning electron micrographs of dry-milled flours from nine different rice cultivars at different magnifications (SL97 = Shaw Lung 97, PB1 = Prachin Buri 1, PB2 = Prachin Buri 2)



1000-2500X magnification

5000X magnification

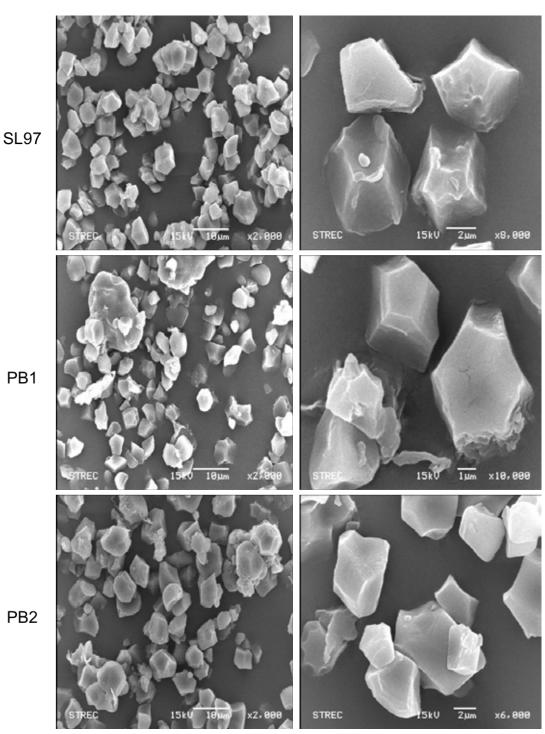
**Figure 4.10 (continued)** Scanning electron micrographs of dry-milled flours from nine different rice cultivars at different magnifications (PNG = Plai Ngahm Prachin Buri, K105 = Khao Dawk Mali 105, AY1 = Ayutthaya 1)



2000X magnification

7000-10000X magnification

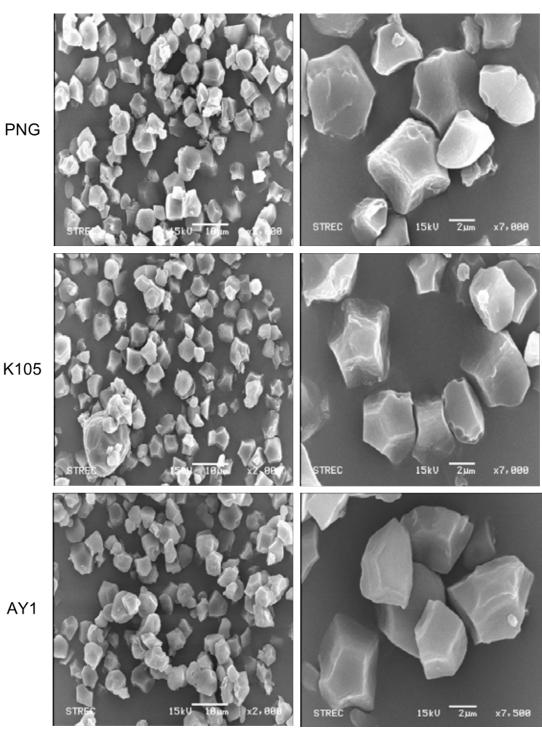
Figure 4.11 Scanning electron micrographs of wet-milled flours from nine different rice cultivars at different magnifications



2000X magnification

6000-10000X magnification

**Figure 4.11 (continued)** Scanning electron micrographs of wet-milled flours from nine different rice cultivars at different magnifications (SL97 = Shaw Lung 97, PB1 = Prachin Buri 1, PB2 = Prachin Buri 2)



2000X magnification

7000-7500X magnification

**Figure 4.11 (continued)** Scanning electron micrographs of wet-milled flours from nine different rice cultivars at different magnifications (PNG = Plai Ngahm Prachin Buri, K105 = Khao Dawk Mali 105, AY1 = Ayutthaya 1)

#### 4.2.2.4 X-ray diffraction pattern of rice flours

X-ray diffractions of rice flours showed a typical A-pattern (Figure 4.12- 4.13) and similarly strong reflections at 15°, 17°, 18°, and 23° 20 angles, which were the same as that of most ordinary rice flours (lturriaga *et al.*, 2004; Yu *et al.*, 2010). The crystallinity level calculated from the ratio of diffraction peak area and total diffraction area are given in Table 4.9. The crystallinity degree of wet-milled and dry-milled rice flours varied in the 13.0 to 24.2% range. These differences in crystallinity degree of rice flours may be attributed to rice cultivars, starch structure, the distribution of amylose and amylopectin in starch granule, and other components such as proteins and lipids. These results were similar to the earlier report of Singh et al. (2007) who reported that the difference in crystallinity in different rice starches may be attributed to difference in proportions of amylose to short and long side-chain amylopectin. Moreover, other components might influence the granule structure and crystallinity of grains (Ibáñez et al., 2007).

In addition, milling also affected crystallinity degree. The result showed that crystallinity degree of wet-milled rice flours was significantly higher than those of dry-milled rice flours ( $p \le 0.05$ ) (Table 4.9). The mechanical force from the dry milling process caused considerable disruption of the native crystalline structure (Chen *et al.*, 2003).

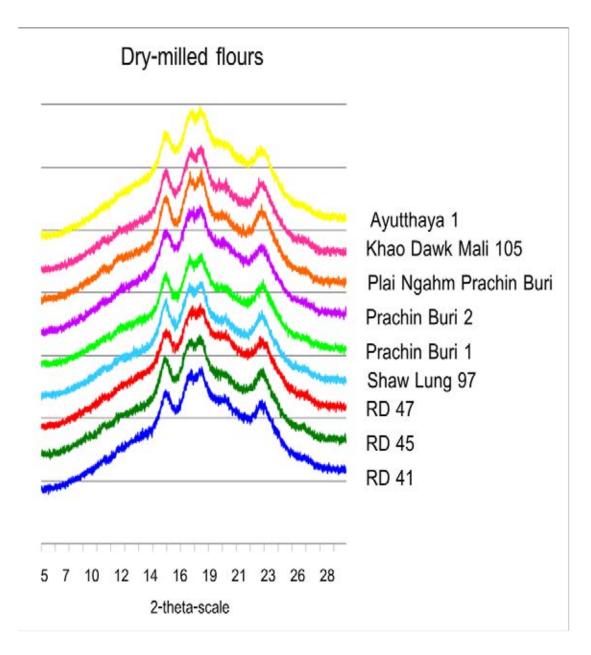


Figure 4.12 X-ray diffraction pattern of dry-milled flours in different rice cultivars

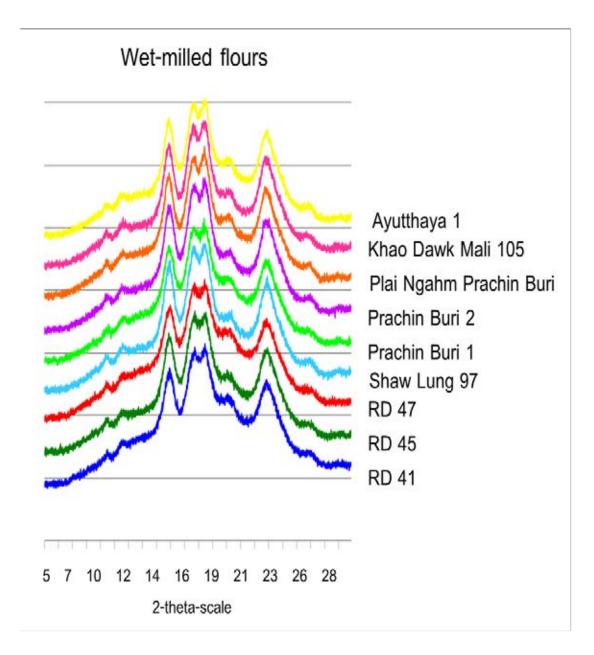


Figure 4.13 X-ray diffraction pattern of wet-milled flours in different rice cultivars

Cultivars	Crystallinity degree (%)				
Guillvars	Dry-milled flour	Wet-milled flour			
RD 41	13.0 <sup>i</sup> ± 0.1	20.4 <sup>e</sup> ± 0.4			
RD 45	$13.9^{gh} \pm 0.2$	22.5 <sup>°</sup> ± 0.1			
RD 47	13.3 <sup>hi</sup> ± 0.1	20.1 <sup>e</sup> ± 0.2			
Shaw Lung 97	$13.7^{ghi} \pm 0.0$	$23.4^{b} \pm 0.6$			
Prachin Buri 1	$13.4^{hi} \pm 0.0$	20.6 <sup>e</sup> ± 0.2			
Prachin Buri 2	$13.3^{hi} \pm 0.3$	23.3 <sup>b</sup> ± 0.1			
Plai Ngahm Prachin Buri	$14.7^{f} \pm 0.6$	$22.5^{\circ} \pm 0.5$			
Khao Dawk Mali 105	$14.2^{fg} \pm 0.4$	$24.2^{a} \pm 0.1$			
Ayutthaya 1	$13.4^{hi} \pm 0.1$	$21.7^{d} \pm 0.0$			

Table 4.9 Crystallinity characteristics of flours

Mean  $\pm$  standard deviation values followed by different superscripts are significantly different (P  $\leq 0.05$ )

### 4.2.2.5 Water binding capacity

Starch is less soluble in water at below gelatinization temperature because hydrogen intermolecular bonds. The results of water binding capacity are shown in Table 4.10. The water binding capacity of wet-milled flours was in the range of 1.29 to 1.47 g water/g dry sample and they were significantly ( $P \le 0.05$ ) lower than those of dry-milled flours that had the water binding capacity varying in the range of 2.69 to 3.87 g water/g dry sample. The mechanical damage of starch granules from dry milling process caused the absence of a network structure and increases in free hydroxyl groups.

Cultivars	Dry-milled flour	Wet-milled flour
RD 41	$3.14^{\circ} \pm 0.08$	$1.30^{f} \pm 0.05$
RD 45	$3.41^{b} \pm 0.11$	$1.44^{f} \pm 0.01$
RD 47	$2.69^{e} \pm 0.08$	$1.47^{f} \pm 0.03$
Shaw Lung 97	$3.37^{b} \pm 0.17$	$1.29^{f} \pm 0.05$
Prachin Buri 1	$3.05^{cd} \pm 0.14$	$1.37^{f} \pm 0.02$
Prachin Buri 2	$3.87^{a} \pm 0.26$	$1.31^{f} \pm 0.00$
Plai Ngahm Prachin Buri	$2.89^{d} \pm 0.11$	$1.37^{f} \pm 0.02$
Khao Dawk Mali 105	$3.07^{\circ} \pm 0.06$	$1.45^{f} \pm 0.03$
Ayutthaya 1	$2.98^{cd} \pm 0.08$	$1.41^{f} \pm 0.03$

Table 4.10 Water binding capacity of flours (g of water/g of dry sample)

Mean  $\pm$  standard deviation values followed by different superscripts are significantly different (P  $\leq 0.05$ )

## 4.2.2.6 Swelling power and solubility

The swelling power of the rice flour samples at different temperatures are presented in Figure 4.14 and Table B.22. The swelling power of flours increased with increasing temperature. The increase of swelling power of RD 41, RD 45, RD 47, Shaw Lung 97, Prachin Buri 1, Prachin Buri 2, Plai Ngahm Prachin Buri, Khao Dawk Mali 105 and Ayutthaya 1 dry-milled rice flours was 6.02 to 8.35, 6.88 to 10.75, 5.90 to 8.52, 5.86 to 11.38, 6.75 to 9.35, 5.60 to 12.43, 5.47 to 10.70, 6.26 to 11.10 and 5.46 to 10.81 g/g dry flour at 60°C to 90°C, respectively. For wet-milled flour, the swelling power of RD 41, RD 45, RD 47, Shaw Lung 97, Prachin Buri 1, Prachin Buri 2, Plai Ngahm Prachin Buri 2, Plai Ngahm Prachin Buri, Khao Dawk Mali 105 and Ayutthaya 1 were 4.14 to 9.25, 4.28 to 12.39, 4.70 to 9.03, 2.89 to 11.25, 4.76 to 9.56, 2.75 to 11.62, 3.05 to 10.85, 3.84 to 13.55 and 3.21 to 10.39 g/g dry flour at 60°C to 90°C, respectively.

These differences in swelling powers of rice flour might be attributed to the molecular characteristics of amylopectin content. Yoenyongbuddhagal and Noomhorm (2002a) reported that rice starch containing high proportions of long chain amylopectin

showed high swelling power. The swelling power of starch depends on the capacity of starch molecules to hold water via hydrogen bonding, when the hydrogen bonds between starch molecules were broken after complete gelatinization they were replaced by hydrogen bonds with water. The amylose content and the proportion of outside-chains of amylopectin were thought to be the major factors stabilizing the gel structure to retain water. The distribution of amylose in starch granule was not uniform, which also affects the swelling power of starch. The starch granules mainly contained amylose and amylopectin, during gelatinization, some amylose molecule leaked out; however, the quantity of amylose which leaked out was related to the molecular structure or hydrogen bonding. So the swelling power is not only strictly correlated with amylose content, but also related with amylopectin content the structure of granules and the distribution of starch molecule in rice starch granules (Lee and Osman, 1991; Singh *et al.*, 2006; Yu *et al.*, 2010).

The results indicated that dry milling resulted in the conversion of large ordered (crystalline) regions into essentially disordered amorphous which was confirmed by the crystallinity degree (Table 4.9). Dry milling process causes the absence of a network structure from amylose molecules that can hold the starch molecules together and increases in free hydroxyl groups (Chen et al., 2003; Patindol and Wang, 2002). Therefore the starch granules of dry-milled flour unrestricted the swelling resulting in a higher swelling power of dry-milled flours at below gelatinization temperature than wet milled flours (Figure 4.14). However, when temperature increased to 90°C, the swelling power of wet-milled and dry-milled flours became equivalent, except in wet-milled flour from RD 41, RD 45, RD 47 and Khao Dawk Mali 105 that possessed higher swelling power than the dry-milled flour at 90°C. Wet milled flour of RD 41 and RD 47 possessed slightly higher swelling power than the dry milled flour (0.9 and 0.5 g/g of dry sample differences), while that of RD 45 and Khao Dawk Mali 105 had considerably higher swelling power than the dry milled flour (1.6 and 2.5 g/g of dry sample differences) at 90°C. This could be resulted from the lower amylose content, hence higher

amylopectin, that could promote swelling at the later stage of heating in RD 45 and Khao Dawk Mali 105.

These results indicated that solubility was positively correlated with temperature (Figure 4.15 and Table B.23). The increase of solubility of RD 41, RD 45, RD 47, Shaw Lung 97, Prachin Buri 1, Prachin Buri 2, Plai Ngahm Prachin Buri, Khao Dawk Mali 105 and Ayutthaya 1 dry-milled rice flours was 5.63% to 15.21%, 7.74% to 14.35%, 5.86% to 16.69%, 7.62% to 19.26%, 4.81% to 12.64%, 6.47% to 20.22%, 5.03% to 14.78%, 8.42% to 15.62% and 5.31% to 14.55%, at 60°C to 90°C respectively. For wet-milled flour, the solubility of RD 41, RD 45, RD 47, Shaw Lung 97, Prachin Buri 1, Prachin Buri 2, Plai Ngahm Prachin Buri, Khao Dawk Mali 105 and Ayutthaya 1 was 1.31% to 11.01%, 1.10% to 6.97%, 1.67% to 11.13%, 0.40% to 9.47%, 1.09% to 8.32%, 0.52% to 11.27%, 0.73% to 9.58%, 1.87% to 6.37% and 0.66% to 7.69%, at 60°C to 90°C respectively.

As stated earlier, dry milling process caused considerable disruption of the native crystalline structure of the rice flours. The higher amount of free hydroxyl groups was, thus, inevitably increased following the increase in damaged starch. In previous research studies, higher water retention capacity was observed for the dry-milled flours (Chen *et al.*, 2003; Suksomboon and Naivikul, 2006). However, in this study, as a result of damaged granules, higher solubility were observed for dry-milled flours (Figure 4.15).

Furthermore, other compositions especially proteins and lipids also influence the swelling power and solubility of flours. Chanapamokkhot and Thongngam (2007) demonstrated that the protein and lipid could inhibit the swelling of starch granules. Protein with disulfide bonds in the rice flour restricts starch granule swelling during gelatinization and make the swollen granules less susceptible to disruption by shear (Hamaker and Griffin, 1993). Moreover, lipids can form the complexes with amylose and amylopectin (Biliaderis and Tonogai, 1991). These complexes are believed to restrict starch granule swelling during heating and prevent leaching of amylose during gelatinization (Saleh and Meullenet, 2005),

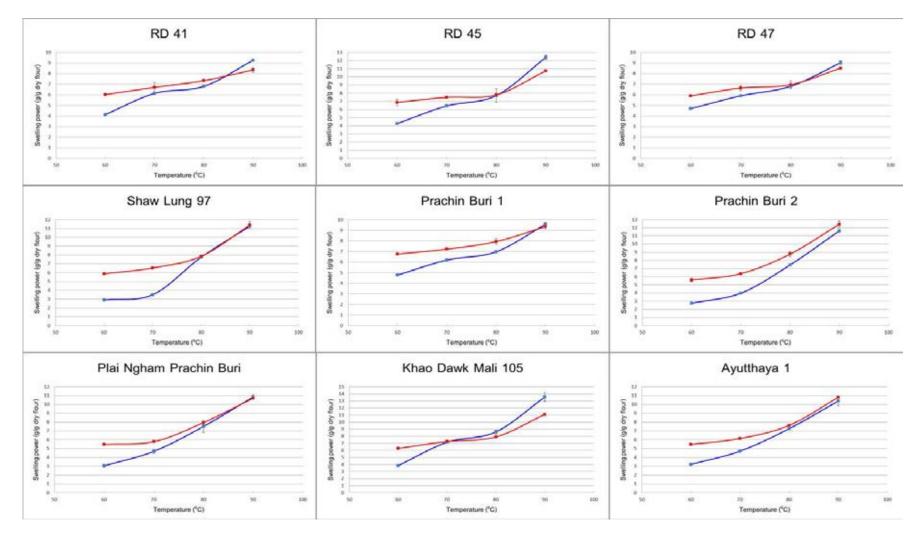


Figure 4.14 Swelling power of rice flours at different temperatures ( ----Wet milling ----Dry milling )

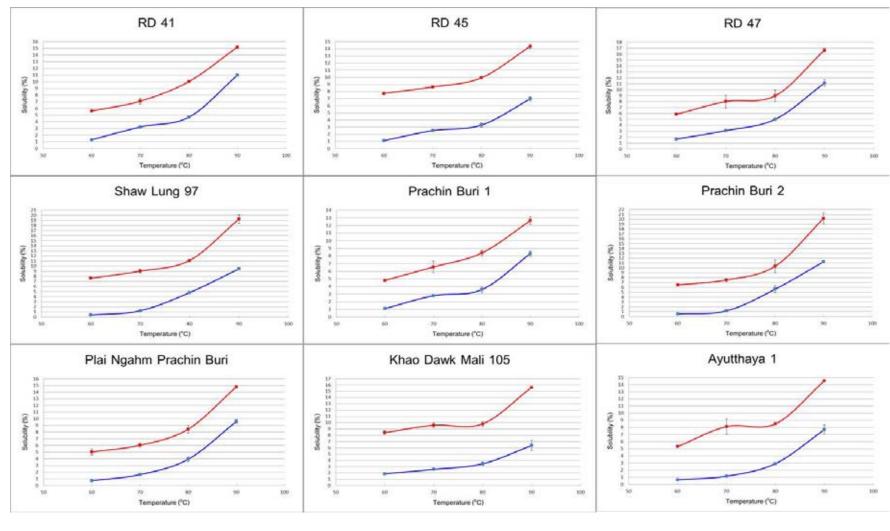


Figure 4.15 Solubility properties of rice flours at different temperatures ( ----Wet milling ----Dry milling )

75

## 4.2.2.7 Pasting properties

Pasting properties differed significantly among different rice flours. Pasting temperature of different rice flours ranged from 76.2 to 91.9°C (Table 4.11 and 4.12). Trough viscosity of different rice flours ranged from 684 to 2892 cP. Final viscosity increased upon cooling which might be due to the aggregation of the amylose molecules. Final viscosity and setback varied from 1650-4960 to 836-2687 cP, respectively. Pasting properties of starch have been reported to be affected by amylose and lipids contents and by branch chain-length distribution of amylopectin. Amylopectin contributes to swelling of starch granules and pasting, while amylose and lipids inhibit the swelling. Also, the amylopectin chain-length and amylose molecular size produce synergistic effects on the viscosity of starch pastes (Singh *et al.*, 2006).

Moreover, the milling process influences pasting viscosity properties by the mechanical damage of starch granules. The result showed that dry-milled rice flours provided significantly lower peak viscosity (1010 to 2624 cP) than those of wet-milled rice flours (2062 to 3818 cP) ( $p \le 0.05$ ). The lower peak viscosity was caused by a higher starch damage of dry-milled rice flours, similar to the result reported by Yoenyongbuddhagal and Noomhorm (2002a).

From Table 4.11, dry milled flour of Prachin Buri 1 had the highest peak viscosity while that of RD 47 had the lowest peak viscosity. It was observed that the two dry milled flours, which were RD 41 and RD 47, that gave the lowest peak viscosities (1235 and 1010 cP) also had the lowest swelling powers at 90°C (8.4 and 8.5 g/g of dry sample). Among dry milled flours, the flour of RD 45 and Khao Dawk Mali 105 gave relatively low setback-to-trough viscosity ratio of 1.02 and 1.01 respectively, while other dry milled flour has the setback-to-trough viscosity ratio from 1.17 to 1.61. This could be because of the low amylose content in these flours (18.4 and 17.9%).

In wet milled flours, RD 41 and RD 47, which possessed lower swelling power at 90°C also had the lowest peak viscosity (2298 and 2062 cP). RD 45 and Khao Dawk Mali 105 gave relatively higher breakdown-to-peak viscosity ratio of 0.44 and 0.48, respectively, which could be related to their low amylose content (18.7 and 13.1%).

Cultivars	Peak time (min)	Pasting	Pasting viscosity (cP)				
		temperature (°C)	Peak viscosity	Trough viscosity	Final viscosity	Breakdown	Setback
RD 41	$5.6^{gh} \pm 0.0$	$87.2^{\circ} \pm 0.8$	1235 <sup>k</sup> ± 52	875 <sup>j</sup> ± 27	1901 <sup>j</sup> ± 105	$360^{9} \pm 25$	1026 <sup>i</sup> ± 81
RD 45	$5.5^{hi} \pm 0.0$	83.7 <sup>e</sup> ± 0.5	2095 <sup>i</sup> ± 105	$1248^{h} \pm 68$	2518 <sup>hi</sup> ± 82	$847^{d} \pm 40$	1270 <sup>gh</sup> ± 18
RD 47	$5.6^{gh} \pm 0.1$	$88.4^{b} \pm 0.4$	$1010^{1} \pm 64$	$684^{k} \pm 62$	$1650^{k} \pm 65$	325 <sup>g</sup> ± 5	966 <sup>i</sup> ± 19
Shaw Lung 97	$5.5^{i} \pm 0.1$	$81.1^{f} \pm 0.4$	1711 <sup>j</sup> ± 123	$1024^{i} \pm 70$	2394 <sup>i</sup> ± 139	687 <sup>e</sup> ± 53	1370 <sup>g</sup> ± 72
Prachin Buri 1	$5.9^{f} \pm 0.0$	84.3 <sup>e</sup> ± 0.5	2624 <sup>ef</sup> ± 139	1993 <sup>de</sup> ± 100	4680 <sup>b</sup> ± 222	$631^{ef} \pm 40$	2687 <sup>ª</sup> ± 122
Prachin Buri 2	$5.2^{j} \pm 0.1$	78.3 <sup>g</sup> ± 0.1	2003 <sup>i</sup> ± 89	1005 <sup>ij</sup> ± 72	2399 <sup>i</sup> ± 87	997 <sup>°</sup> ± 32	1394 <sup>fg</sup> ± 25
Plai Ngahm Prachin Buri	$5.6^{ghi} \pm 0.0$	$78.9^{g} \pm 0.4$	$2569^{fg} \pm 25$	1430 <sup>g</sup> ± 33	3528 <sup>f</sup> ± 21	1139 <sup>b</sup> ± 13	2098 <sup>°</sup> ± 21
Khao Dawk Mali 105	$5.6^{gh} \pm 0.1$	$86.2^{cd} \pm 0.4$	$1720^{j} \pm 78$	1164 <sup>h</sup> ± 82	2338 <sup>i</sup> ± 99	$556^{f} \pm 50$	1174 <sup>h</sup> ± 19
Ayutthaya 1	5.7 <sup>g</sup> ± 0.1	$78.9^{g} \pm 0.6$	2444 <sup>gh</sup> ± 144	1482 <sup>9</sup> ± 120	3871 <sup>e</sup> ± 201	962 <sup>°</sup> ± 37	2389 <sup>b</sup> ± 112

 Table 4.11 Pasting properties of dry-milled rice flours

Mean  $\pm$  standard deviation values in the same column in table 4.11 and 4.12 followed by different superscripts are significantly different (P  $\leq$  0.05)

Cultivars	Peak time (min)	Pasting	Pasting viscosity (cP)				
		temperature (°C)	Peak viscosity	Trough viscosity	Final viscosity	Breakdown	Setback
RD 41	$6.3^{b} \pm 0.1$	91.1 <sup>ª</sup> ± 0.5	2298 <sup>h</sup> ± 137	1680 <sup>f</sup> ± 122	3287 <sup>g</sup> ± 165	621 <sup>ef</sup> ± 16	1611 <sup>de</sup> ± 68
RD 45	$6.6^{de} \pm 0.1$	$86.2^{cd} \pm 0.6$	$3467^{\circ} \pm 63$	1932 <sup>e</sup> ± 119	3198 <sup>g</sup> ± 118	1536 <sup>ª</sup> ± 56	1266 <sup>gh</sup> ± 1
RD 47	$6.5^{a} \pm 0.0$	$91.9^{a} \pm 0.4$	$2062^{i} \pm 61$	1406 <sup>g</sup> ± 53	$2632^{h} \pm 56$	657 <sup>e</sup> ± 15	1227 <sup>h</sup> ± 27
Shaw Lung 97	$6.1^{e} \pm 0.0$	$81.6^{f} \pm 0.1$	2742 <sup>e</sup> ± 42	$1648^{f} \pm 36$	3149 <sup>g</sup> ± 45	1094 <sup>b</sup> ± 39	1501 <sup>ef</sup> ± 40
Prachin Buri 1	$6.6^{a} \pm 0.0$	91.3 <sup>ª</sup> ± 1.4	3818 <sup>ª</sup> ± 123	2892 <sup>a</sup> ± 100	4615 <sup>b</sup> ± 162	926 <sup>°</sup> ± 37	1723 <sup>d</sup> ± 72
Prachin Buri 2	$5.9^{f} \pm 0.1$	$80.8^{f} \pm 0.8$	$3183^{d} \pm 67$	$2083^{cd} \pm 36$	$4100^{d} \pm 61$	1100 <sup>b</sup> ± 82	2017 <sup>°</sup> ± 64
Plai Ngahm Prachin Buri	$6.2^{cd} \pm 0.1$	83.8 <sup>e</sup> ± 1.0	$3740^{ab} \pm 67$	2215 <sup>°</sup> ± 116	4338 <sup>°</sup> ± 82	1525 <sup>ª</sup> ± 105	2123 <sup>°</sup> ± 82
Khao Dawk Mali 105	$6.2^{bcd} \pm 0.1$	$76.2^{h} \pm 0.5$	$3235^{d} \pm 78$	$1696^{f} \pm 77$	2532 <sup>hi</sup> ± 92	1539 <sup>ª</sup> ± 33	836 <sup>j</sup> ± 53
Ayutthaya 1	$6.3^{bc} \pm 0.0$	$85.7^{d} \pm 0.8$	3626 <sup>b</sup> ± 37	2677 <sup>b</sup> ± 52	4960 <sup>°</sup> ± 138	949 <sup>°</sup> ± 32	2283 <sup>b</sup> ± 170

Table 4.12 Pasting properties of wet-milled rice flours

Mean ± standard deviation values in the same column in table 4.11 and 4.12 followed by different superscripts are significantly different (P ≤ 0.05)

#### 4.2.2.8 Thermal Properties

The thermal parameters from endotherms are shown in Table 4.13. The onset temperature (To) of the rice flour samples varied from 61.7°C for dry-milled rice flour RD 47 to 75.4°C for dry-milled rice flour Shaw Lung 97, whereas the peak temperature (Tp) range was 68.3°C for wet-milled rice flour RD 47 to 80.5°C for wet-milled rice flour Shaw Lung 97 and the conclusion temperature (Tc) range was 74.0°C for dry-milled rice flour RD 47 to 85.6°C for dry-milled rice flour Shaw Lung 97. The results showed that the Tc-To of dry-milled flours were significantly higher than those of wet-milled flour ( $P \le 0.05$ ). This is probably because dry milled flours had larger particle distribution. The smaller particle started to hydrate and gelatinize rapidly when gelatinization temperature was reached. On the other hand, the larger particles or aggregates could not hydrate or expand as rapidly resulting in delayed gelatinization, hence a larger Tc-To. The gelatinization enthalpy of dry-milled flours was in the range of 2.5 to 5.9 J/g and they were significantly lower than those of wet-milled flours which varied in the range of 7.6 to 12.1 J/g (P  $\leq$  0.05). The crystalline structure of dry-milled rice flours were disrupted due to the grinding process. The mechanical force caused starch damage during milling, with a lower gelatinization enthalpy in dry-milled rice flour (Chen et al., 1999).

Gelatinization temperature and  $\Delta H$  are indicators of the overall crystallinity of amylopectin, which is directly related to the structure of amylopectin. Yoenyongbuddhagal and Noomhorm (2002a) demonstrated that starch with a greater amount of long-chain amylopectin and smaller amount of short-chain amylopectin exhibited higher To, Tp and  $\Delta H$  than starch with fewer long branched chains. This is because the longer chains have a greater ability to form double helices, which required greater thermal energy to dissociate (Yoenyongbuddhagal and Noomhorm, 2002b).

Cultivars	To (°C)	Tp (°C)	Tc (°C)	<b>Δ</b> Η (J/g)	Tc - To (°C)
RD41(D)	$63.2^{k} \pm 0.1$	$71.3^{i} \pm 0.1$	77.1 <sup>f</sup> ± 0.1	$5.2^{h} \pm 0.2$	$13.8^{ab} \pm 0.2$
RD41(W)	$64.6^{i} \pm 0.2$	$70.5^{j} \pm 0.2$	$75.8^{h} \pm 0.3$	$9.6^{d} \pm 0.1$	11.2 <sup>e</sup> ± 0.1
RD45(D)	$64.4^{i} \pm 0.4$	$71.7^{h} \pm 0.3$	77.9 <sup>e</sup> ± 0.1	$5.3^{d} \pm 0.2$	13.5 <sup>b</sup> ± 0.5
RD45(W)	$65.1^{h} \pm 0.1$	$70.5^{j} \pm 0.0$	$75.5^{h} \pm 0.2$	$9.9^{cd} \pm 0.2$	$10.4^{f} \pm 0.2$
RD47(D)	61.7 <sup>n</sup> ± 0.1	$69.9^{k} \pm 0.1$	$75.8^{h} \pm 0.3$	$5.3^{h} \pm 0.3$	14.1 <sup>ª</sup> ± 0.2
RD47(W)	62.1 <sup>m</sup> ± 0.2	$68.3^{m} \pm 0.2$	$74.0^{j} \pm 0.1$	$8.6^{e} \pm 0.3$	$11.9^{d} \pm 0.1$
SL97 (D)	$75.4^{a} \pm 0.2$	$80.5^{a} \pm 0.0$	$85.6^{a} \pm 0.1$	$4.2^{i} \pm 0.2$	$10.2^{f} \pm 0.3$
SL97 (W)	75.4 <sup>ª</sup> ± 0.1	$79.5^{b} \pm 0.2$	$83.7^{b} \pm 0.1$	$11.4^{b} \pm 0.4$	$8.3^{h} \pm 0.0$
PB1 (D)	$62.7^{1} \pm 0.0$	$70.1^{k} \pm 0.1$	$76.7^{g} \pm 0.0$	$5.9^{9} \pm 0.0$	$14.0^{a} \pm 0.0$
PB1 (W)	$63.8^{j} \pm 0.0$	$69.2^{1} \pm 0.0$	$74.8^{i} \pm 0.1$	$10.3^{\circ} \pm 0.3$	11.0 <sup>e</sup> ± 0.1
PB2 (D)	73.0 <sup>e</sup> ± 0.2	78.9 <sup>°</sup> ± 0.1	83.6 <sup>b</sup> ± 0.1	$3.3^{j} \pm 0.1$	$10.5^{f} \pm 0.2$
PB2 (W)	$74.0^{cd} \pm 0.0$	78.1 <sup>d</sup> ± 0.0	$82.4^{\circ} \pm 0.0$	12.1 <sup>ª</sup> ± 0.2	$8.4^{h} \pm 0.0$
PNG (D)	$74.3^{bc} \pm 0.0$	78.9 <sup>°</sup> ± 0.1	$83.5^{b} \pm 0.0$	$5.1^{h} \pm 0.2$	$9.3^{g} \pm 0.0$
PNG (W)	$73.2^{e} \pm 0.2$	77.5 <sup>e</sup> ± 0.2	82.1 <sup>°</sup> ± 0.2	$11.2^{b} \pm 0.1$	$9.0^{g} \pm 0.0$
K105 (D)	$67.2^{f} \pm 0.1$	$74.1^{f} \pm 0.0$	$79.9^{d} \pm 0.1$	$3.9^{i} \pm 0.1$	$12.7^{\circ} \pm 0.2$
K105 (W)	$66.3^{g} \pm 0.2$	$72.5^{g} \pm 0.3$	77.8 <sup>e</sup> ± 0.2	11.5 <sup>b</sup> ± 0.4	$11.4^{e} \pm 0.0$
AY1 (D)	74.5 <sup>b</sup> ± 0.1	79.3 <sup>b</sup> ± 0.1	$83.5^{b} \pm 0.3$	$2.5^{k} \pm 0.0$	$9.0^{9} \pm 0.2$
AY1 (W)	$73.7^{d} \pm 0.0$	$78.0^{d} \pm 0.0$	82.0 <sup>°</sup> ± 0.1	$7.6^{f} \pm 0.3$	$8.3^{h} \pm 0.1$

Table 4.13 Thermal properties of rice flours

Mean ± standard deviation values in the same column followed by different superscripts are

significantly different ( $P \le 0.05$ )

(D) = flour from dry milling method, (W) = flour from wet milling method

SL97 = Shaw Lung 97, PB1 = Prachin Buri 1, PB2 = Prachin Buri 2, PNG = Plai Ngahm Prachin Buri, K105 = Khao Dawk Mali 105, AY1 = Ayutthaya 1

To = onset temperature, Tp = peak temperature, Tc = conclusion temperature,  $\Delta$ H = gelatinization enthalpy

### 4.2.2.9 Viscoelastic properties

The mechanical spectra of the formed gel have been determined by measuring different oscillation indices such as storage modulus (G') and loss modulus (G''). The storage dynamic modulus (G') is a measure of the energy stored in the material and recovered from it per cycle while the loss modulus (G'') is a measure of the energy dissipated or lost per cycle of sinusoidal deformation (Ferry, 1980).

All of nine rice varieties showed similar flow behavior and the storage modulus (G') was much larger than the loss modulus (G'') about one log cycle (Figure 4.16 and 4.17). Low amylose content rice flours such as RD 45 and Khao Dawk Mali 105 have higher slope of both modulus (G' and G'') more than the high amylose flours (RD 41, RD 47, Shaw Lung 97, Prachin Buri 1, Prachin Buri 2, Plai Ngahm Prachin Buri, Ayutthaya 1). Besides, the low amylose flours also showed the less difference between storage and loss modulus (< 1 log cycle). These behaviors indicated that low amylose flours gave weaker gel structure characteristic than high amylose flours.

Figure 4.18 showed complex modulus (G\*) of dry-milled and wet-milled flours from different rice cultivars. The result showed that the G\* of wet-milled flours were higher than dry-milled flours in all nine rice cultivars. Dry-milled flours contained larger amount of damaged starch than wet-milled flours (Suksomboon and Naivikul, 2006). Higher levels of damaged starch led to higher water absorption and enzymatic hydrolysis. Damaged starch increased dispersion of amylopectin resulting in reduced gel forming capacity of amylose (Arora, 2003). In addition, the low amylose of dry-milled and wet-milled rice flours provided higher slope of G\* than those of high amylose rice flours. This behavior indicated the weaker gel structure and more fluid behavior.

Rheological behavior of gelatinized starch dispersions is primarily because of inter-granular interactions, and to the viscoelasticity and compressibility of the granules themselves. It is generally accepted that both the continuous phase (amylose) and the swollen granules contribute to the mechanical properties of the gel composite. Thus, granular size, amylose-amylopectin ratio as well as minor constituents (lipid and protein) are all important determinants of the viscoelastic behavior of starch dispersions

(Biliaderis, 1993). Differences in the amounts of starch lipids among the rice samples could also affect their gel properties. Biliaderis and Tonogai (1991) have recently shown that removal of internal granular lipids from wheat and rice starches results in increased gel firmness (G') and viscosity. A likely explanation is that granular lipids inhibit leaching of amylose and associate with amylopectin during gelatinization because of their ability to complex with both polymers, thereby lowering the tendency of this polymer to retrograde. Protein contributes to the rigidity of swollen starch granules (Han et al., 2002). Eliasson and Tjerneld (1990) studied on wheat flour and found that the proteins on the surface of starch granules influence the elastic properties of gelatinized starch causing an increase in the storage modulus (G').

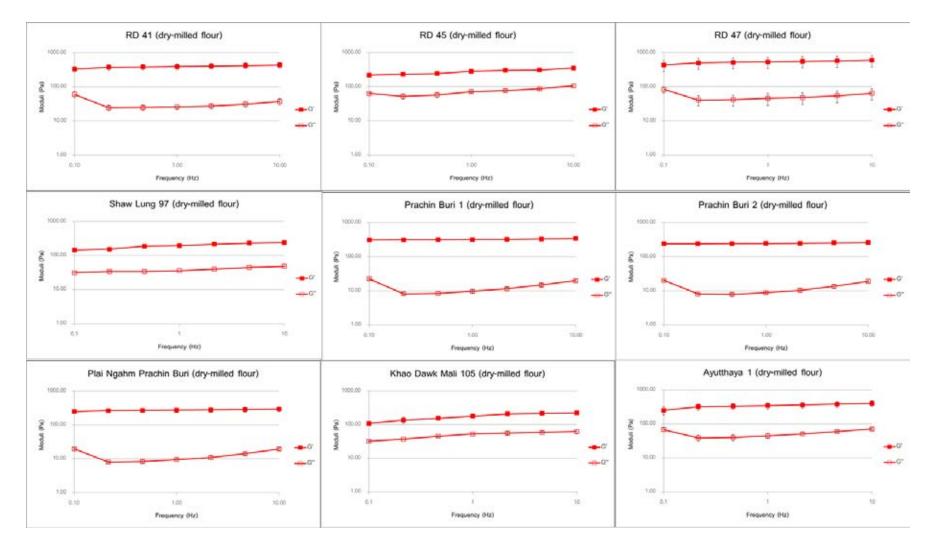


Figure 4.16 Modulus of dry-milled flours in different rice cultivars (+G', +G')

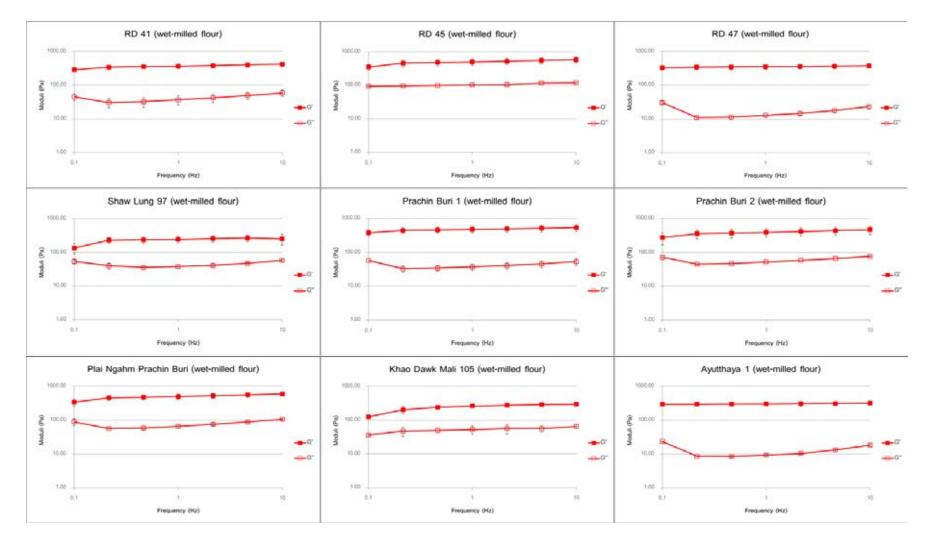
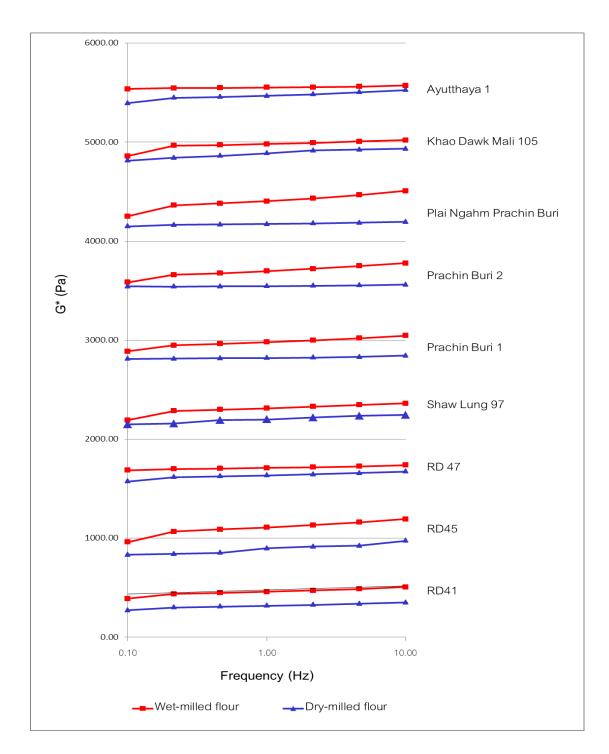


Figure 4.17 Modulus of wet-milled flours in different rice cultivars (  $\bullet$  G ,  $\Rightarrow$  G )



**Figure 4.18** Complex modulus of dry and wet milled flours in different rice cultivars (The data of RD 41 is in real scale. That of RD 45, RD 47, Shaw Lung 97, Prachin Buri 1, Prachin Buri 2, Plai Ngahm Prachin Buri, Khao Dawk Mali 105 and Ayutthaya 1 was added with constant values of 600, 1300, 2000, 2500, 3300, 3900, 4700 and 5200 Pa, respectively.)

## CHAPTER 5

# CONCLUSIONS

Among the nine rice varieties studied in this work, Khao Dawk Mali 105 and RD 45 had lower amylose content and gave higher cohesiveness, adhesiveness, chewiness and hardness while Prachin Buri 1 gave the lowest values of the cooked rice in bulk. When processed into flours, wet milled and dry milled flour of RD 41 and RD 47 had the lowest swelling at 90°C resulting in the lowest peak viscosity while dry milled flours of RD 45 and Khao Dawk Mali 105 had the lowest setback.

Wet milling process removed soluble proteins and the other components. Therefore wet milled rice flours contained lower protein content than dry milled rice flours. The wet milled flours also possessed smaller particle size compared to dry milled flours which retained higher amount of starch granule aggregates. In addition, wet milling caused the flour to retain more crystallinity and, hence, higher gelatinization enthalpy than dry milling process. The flours from wet milling process were able to give higher peak viscosity and form better gel with higher complex modulus at 12% concentration.

However, dry milled flours had greater solubility and water binding capacity. At below gelatinization temperature ( $60^{\circ}$ C) the damaged starch in dry milled flour gave rise to higher swelling power.

# References

- AOAC. 2005. <u>Official Methods of Analysis</u>. 17<sup>th</sup>ed. Association of Official Analysis Chemists. Washington, D.C.
- Arora, S. 2003. <u>The effect of enzymes and starch damage on wheat flour tortilla quality</u>.
   Master's Thesis, Department of Food Science and Technology, Graduate
   School, Texas A&M University.
- Atwell, W.A., Hood, L.F., Lineback, D.R., Marston, V.E., and Zobel, H.F. 1988. The terminology and methodology associated with basis starch phenomena. <u>Cereal Foods World</u> 33: 306-311.
- Ayabe, S., Kasai, M., Ohishi, K., and Hatae, K. 2009. Textural properties and structures of starches from indica and japonica rice with similar amylose content.
   <u>Food Science and Technology Research</u> 15(3): 299-306.
- Banchathanakij, R. 2008. Effect of different *β*-glucans on the gelatinization and retrogradation of rice starch. Master's Thesis, Department of Biotechnology, Graduate School, Mahidol University.
- Belitz, H.D., Grosch, W., and Schieberle, P. 2009. <u>Food Chemistry</u>. 4<sup>th</sup> ed. German: Springer Verlay Berlin Heidelberg.
- Bemiller, J., and Whisler, R. 2009. <u>Starch: Chemistry and Technology</u>. 3<sup>rd</sup> ed. USA: Macmillan Publishing Solutions.
- Biliaderis, C.G., and Tonogai, J.R. 1991. Influence of lipids on the thermal and mechanical properties of concentrated starch gels. <u>Journal of Agriculture and</u> <u>Food Chemistry</u> 39: 833-840.
- Biliaderis, C.G., and Juliano, B.O. 1993. Thermal and mechanical properties of concentrated rice starch gels of varying composition. <u>Food Chemistry</u> 48: 243-250.
- Buléon, A., Colonna, P., Planchot, V., and Ball, S. 1998. Starch granules: structure and biosynthesis. <u>International Journal of Biological Macromolecules</u> 23: 85-112.

Bureau of Rice Research and Development Rice Department (BRRD).

- Rice Knowledge Bank. [Online]. Available from: http://www.brrd.in.th/ rkb/data\_002/rice\_xx2-02\_New\_index.html [14 August 2011].
- Chanapamokkhot, H., and Thongngam, M. 2007. The chemical and physico-chemical properties of sorghum starch and flour. <u>Kasetsart Journal</u> 41: 343-349.
- Chen, J.J., Lu, S., and Lii, C.Y. 1999. Effects of milling on the physicochemical characteristics of waxy rice in Taiwan. <u>Cereal Chemistry</u> 76(5): 796-799.
- Chen, J.J., Lii, C.Y., and Lu, S. 2003. Physicochemical and morphological analyses on damaged rice starches. <u>Journal of Food and Drug Analysis</u> 11(4): 283-289.
- Chiang, P.Y., and Yeh. A.I. 2002. Effect of soaking on wet-milling of rice. <u>Journal of</u> <u>Cereal Science</u> 35: 85-94.
- Dipti, S.S., Hossain, S.T., Bari, M.N., and Kabir, K.A. 2002. Physicochemical and cooking properties of some fine rice varieties. <u>Pakistan Journal of Nutrition</u> 1(4): 188-190.
- Eliasson, A.C., and Tjerneld E. 1990. Adsorption of wheat proteins on wheat starch granules. <u>Cereal Chemistry</u> 67: 366-372.
- Eliasson, A.C. 2004. Starch in food. New York: Woodhead Publishing Limited.
- Ferry, J.D. 1980. <u>Viscoelastic properties of polymers</u>. 3<sup>rd</sup> ed. New York: John Wiley and Sons, Inc.
- Gnanou, Y., and Fontanille, M. 2002. <u>Organic and physical chemistry of polymers</u>. Canada: John Wiley and Sons, Inc.
- Haard, N.F., Odunfa, S.A., and Lee, C.H. 1999. <u>Fermented cereals: global perspective</u>. Rome: Food and Agriculture Organization of the United Nations.
- Hamaker B.R., and Griffin V.K. 1993. Effect of disulfide bond containing protein on rice starch gelatinization and pasting. <u>Cereal chemistry</u> 70(4): 377-380.
- Han, X.Z., Campanella, O.H., Guan, H., Keeling, P.L., and Hamaker, B.R. 2002.
   Influence of maize starch granule-associated protein on the rheological properties of starch pastes. Part II. Dynamic measurements of viscoelastic properties of starch pastes. <u>Carbohydrate Polymers</u> 49(3): 323-330.

- Hizukuri, S., Takeda, Y., and Yasuda, M. 1981. Multi-branched nature of amylose and the action of debranching enzymes. <u>Carbohydrate Research</u> 94: 205-213.
- Hizukuri, S. 1986. Polymodal distribution of the chain lengths of amylopectin, and its significance. <u>Carbohydrate Research</u> 147: 342-347.
- Hoover, R. 2001. Composition, molecular structure, and physicochemical properties of tuber and root starches. <u>Carbohydrate Polymer</u> 45: 253-267.
- Juliano, B.O. 1971. A simplified assay for milled rice amylose. <u>Cereal Science Today</u> 16(10): 334-360.
- Ibáñez, A. A., Wood, D. F., Yokoyama, W. H., Park, I. M., Tinoco, M. A., and
   Hudson, C. A. 2007. Viscoelastic properties of waxy and nonwaxy rice flours,
   their fat and protein-free starch, and the microstructure of their cooked kernels.
   Journal of Agriculture and Food Chemistry 55: 6761-6771.
- Iturriaga, L., Lopez, B., and Añon, M. 2004. Thermal and physicochemical characterization of seven argentine rice flours and starches. <u>Food Research</u> <u>International</u> 37: 439-447.
- Karim, A.A., Norziah, M.H., and Seow, C.C. 2000. Methods for the study of starch retrogradation. <u>Food Chemistry</u> 71: 9-36.
- Kavanagh, G.M., and Rossmurphy, S. 1998. Rheological characterisation of polymer gels. <u>Progress in Polymer Science</u> 23: 533-562.
- Kim, Y.S., Wiesenborn, D.P., Orr, P.H., and Grant, L.A. 1995. Screening potato starch for novel properties using differential scanning calorimetry. <u>Journal of Food Science</u> 60(5): 1060-1065.
- Kohyama, K., Matsuki, J., Yasui, T., and Sasaki, T. 2004. A differential thermal analysis of the gelatinization and retrogradation of wheat starches with different amylopectin chain lengths. <u>Carbohydrate Polymers</u> 58: 71-77.
- Kongseree N. 1994. Cooked rice quality. <u>Quality and investigation of Thai jasmine rice</u>. Department of Agriculture, Ministry of Agriculture and Cooperatives and The Office of Industrial economics, Ministry of Industrial (in Thai).

- Lamberts, L., Bie, E.D., Vandeputte, G.E., Veraverbeke, W.S., Derycke, V., Man, W.D., and Delcour, J. A. 2007. Effect of milling on colour and nutritional properties of rice. <u>Food Chemistry</u> 100: 1496–1503.
- Lee, Y. E., and Osman, E.M. 1991. Correlation of morphological changes of rice starch granules with rheological properties during heating in excess water. <u>Journal of Korean Agricultural Chemical Society</u> 34: 379-385.
- Lii, C.Y., Tsai, M.L., and Tseng K.H. 1996. Effect of amylose content on the rheological property of rice starch. <u>Cereal Chemistry</u> 73(4): 415-420.
- Marco, C., and Rosell, C.M. 2008. Effect of different protein isolates and transglutaminase on rice flour properties. <u>Journal of Food Engineering</u> 84:132-139.
- Marshall, W.E., and Wadsworth, J.I. 1994. <u>Rice Science and Technology</u>. New York: Marcel Dekker, Inc.
- Medcalf, D.G. and Gilles, K.A. 1965. Wheat starches I. Comparison of physicochemicals properties. <u>Cereal Chemistry</u> 42: 558-568.
- Montgomery, C.D. 2005. <u>Design and Analysis of Experiments</u>, 6<sup>th</sup> ed. John Wiley and Sons, Inc.
- Newport Scientific. 2001. <u>Rapid Visco Analyser Series 4 (RVA-4) Installation and</u> <u>operation manual</u>. Australia: Newport Scientific Pty Limited.
- Ngamnikom, P., and Songsermpong, S. 2011. The effects of freeze, dry, and wet grinding processes on rice flour properties and their energy consumption. Journal of Food Engineering 104: 632-638.
- Norbert, U.H., Mintus, T., and Detmold, D.W. 1995. Viscosity measurement of potato starch paste with Rapid Visco Analyzer. <u>Starch/Stärke</u> 47: 123-126.
- Ogawa, Y., Glenn, G. M., Orts, W. J., and Wood, D. F. 2003. Histological structures of cooked rice grain. <u>Journal of Agriculture and Food Chemistry</u> 51: 7019-7023.
- Osada, Y., and Khokhlov, A.R. 2002. <u>Polymer gels and networks</u>. New York: Marcel Dekker, Inc.

- Park J.K., Kim, S.S., and Kim K.O. 2001. Effect of milling ratio on sensory properties of cooked rice and on physicochemical properties of milled and cooked rice. <u>Cereal Chemistry</u> 78(2): 151-156.
- Park, C.S., Kang C.S., Cheong, Y.K. Jung, W., and Woo, S.H. 2010. Influence of puroindoline genotypes on grain characteristics, physico-chemical properties of flour and end-use quality of Korean wheats. <u>Breeding Science</u> 60: 233-242.
- Patindol, J., and Wang, Y.J. 2002. Fine structures of starches from long-grain rice cultivars with different functionality. <u>Cereal Chemistry</u> 79(3): 465-469.
- Prokopowich, D.J., and Biliaderis, C.G. 1995. A comparative study of the effect of sugars on the thermal and mechanical properties of concentrated waxy maize, wheat, potato and pea starch gels. <u>Food Chemistry</u> 52: 255-262.
- Ratnayake, W.S., and Jackson, D.S. 2009. Starch Gelatinization. <u>Advances in Food and</u> <u>Nutrition Research</u> 55: 221-268.
- Schoch, T.J. 1964. Swelling power and solubility of granular starches. <u>Method in</u> <u>Carbohydrates Chemistry</u> 6: 106-108.
- Saleh, M.I., and Meullenet J.F. 2005. Effect of surface lipids on cooked long-grain rice textural properties. <u>Arkansas Agricultural Experiment Station (AAES) Research</u> <u>Series</u> 540: 387-395.
- Seneviratne, H.D., and Biliaderis, C.G. 1991. Action of **α**-amylases on amylose-lipid complex superstructures. Journal of Cereal Science 13: 129-143.
- Singh, N., Singh, J., Kaur, L., Sodhi, N.S., and Gill, B.S. 2003. Morphological, thermal and rheological properties of starches from different botanical sources. <u>Food Chemistry</u> 81: 219-231.
- Singh, N., Kaur, L., Sodhi, N.S., and Sekhon, K.S. 2005. Physicochemical, cooking and textural properties of milled rice from different Indian rice cultivars. <u>Food Chemistry</u> 89: 253-259.
- Singh, N., Kaur. L., Sandhu, K.S., Kaur, J., and Nishinari, K. 2006. Relationships between physicochemical, morphological, thermal, rheological properties of rice starches. <u>Food Hydrocolloids</u> 20: 532–542.

- Singh, N., Nakaura, Y., Inouchi, N., and Nishinari, K. 2007. Fine structure, thermal and viscoelastic properties of starches separated from indica rice cultivars. <u>Starch/Stärke</u> 59: 10-20.
- Suksomboon, A., and Naivikul, O. 2006. Effect of dry- and wet-milling processes on chemical, physicochemical properties and starch molecular structures of rice starches. <u>Kasetsart Journal</u> 40: 125-134.
- Tester, R.F., and Morrison, W.R. 1990. Swelling and gelatinization of cereal starches. <u>Cereal Chemistry</u> 67:558-563.
- Thai Free Trade Area (FTA). <u>Thai rice export</u> [Online]. Available from: http://www.thaifta.com/thaifta/Portals/0/File/ascn\_rice1.doc [2 November 2008].
- Vandenberg, C. 1981. <u>Vapour sorption equilibria and other water-starch interaction</u>; <u>a physico-chemical approach</u>. Doctoral's thesis, Department of Food Science, Agricultural University, Wageningen, the Netherlands.
- Varavinit, S., Shobsngob, S., Varanyanond, W., Chinachoti, P., and Naivikul, O. 2003.
  Effect of amylose content on gelatinization, retrogradation and pasting properties of flours from different cultivars of Thai rice. <u>Starch/Stärke</u> 55: 410-415.
- Webb, B.D., Pomeranz, Y., Afework, S., Lai, F.S., and Bollich, C.N. 1986. Rice grain hardness and its relationship to some milling, cooking, and processing characteristics. <u>Cereal Chemistry</u> 63(1): 27-30.
- Yoenyongbuddhagal, S., and Noomhorm, A. 2002a. Effect of physicochemical properties of high-amylose Thai rice flours on vermicelli quality. <u>Cereal Chemistry</u> 79(4): 481-485.
- Yoenyongbuddhagal, S., and Noomhorm, A. 2002b. Effect of raw material preparation on rice vermicelli quality. <u>Starch/Stärke</u> 54(11): 534-539.
- Yu, S., Ma, Y., Menager, L., and Sun, D.W. 2010. Physicochemical properties of starch and flour from different rice cultivars. <u>Food Bioprocess Technology</u> 10: 1-12.

- Zhou, Z., Robards, K., Helliwell, S., and Blanchard, C. 2002. Composition and functional properties of rice. <u>International Journal of Food Science and Technology</u> 37: 849-868.
- Zohary, D., and Hopf, M. 2000. <u>Domestication of Plants in the Old World</u>. 3<sup>rd</sup> ed. New York: Oxford University Press, Inc.
- Zuo, Y.Y.J., Hébraud, P., Hemar, Y., and Ashokkumar M. 2012. Quantification of high-power ultrasound induced damage on potato starch granules using light microscopy. <u>Ultrasonics Sonochemistry</u> 19: 421-426.

APPENDICES

# APPENDIX A

# Analytical methods

A.1 Preparation of proximate analysis test sample for rice grain (AOAC section 32.2.01,

2005)

## <u>Apparatus</u>

- 1. No. 35 sieve
- 2. Blender

## **Procedure**

Grind test sample to pass No. 35 sieve and mix thoroughly.

A.2 Moisture content (AOAC section 32.1.03, 2005)

## <u>Apparatus</u>

- 1. Hot-air oven (Memmert, model W350, Germany)
- 2. Aluminum dish
- 3. Weighing machine (Scaltec, model SBC series, Germany)
- 4. Desiccator

# <u>Procedure</u>

- 2 g of the sample is transferred to an aluminum dish of pre-established constant weight (W<sub>1</sub>g)
- 2. The sample is set in a hot-air circulating oven regulated at 130°C for 1 hour
- 3. Dish is weighed after cooling  $(W_3g)$ .
- 4. Drying is repeated until constant weight is achieved

# Calculation

Moisture (%) = 
$$\frac{W_2 - W_3}{W_2 - W_1} \times 100$$
 (2)

W1: Weight (g) of aluminum dish

W<sub>2</sub>: Weight (g) of sample with aluminum dish before drying

W<sub>3</sub>: Weight (g) of sample with aluminum dish after drying

A.3 Protein content (AOAC section 32.1.22, 2005)

## <u>Reagent</u>

- 1. Concentrated sulfuric acid (A.R. grade)
- 2. 4 % (w/v) Boric acid solution (A.R. grade)
- 3. 45% (w/v) Sodium hydroxide solution
- 4. Selenium reagent mixture (A.R. grade)
- 5. 0.5 M Hydrochloric acid standard solution (A.R. grade)
- Mixed indicator solution: 1:1 mixed of bromocresol green solution (dissolve 0.1 g of bromocresol green in 100 ml of ethanol (95% v/v)) and methyl red solution (dissolve 0.1 g of methyl red in 100 ml of ethanol (95% v/v))

#### <u>Apparatus</u>

- 1. Buchi digestion unit (Buchi, model K-424, Switzerland)
- 2. Buchi scrubber (Buchi, model B-414, Switzerland)
- 3. Distillation apparatus (Buchi, model B-324, Switzerland)
- 4. Weighing machine (Scaltec, model SBC series, Germany)

- i. Digestion
  - 1. Weigh 1 g of the sample on a piece of filter paper (Whatman No.41)
  - 2. Transfer the paper and the test portion to the Kjeldahl tube.
  - 3. Add 5 g of selenium reagent mixture and 20 ml of sulfuric acid
  - 4. Conduct a blank test following the procedure except for addition of the sample.
  - Place the tube on the Buchi digestion unit with scrubber and heat at heating level 8
  - 6. Heat the tube until the contents have become completely liquefied.
- ii. Distillation
  - Add 50 ml of the boric acid solution to 250 ml flask, add 2 drops of the indicator solution, mix and place the flask under the condenser of the distillation apparatus so that the outlet of the adapter dips into the liquid.

- 2. Digest mode
  - Distilled water: 50 ml
  - 45% (w/v) NaOH solution: 60 ml
  - Distillation time: 5 min
  - Steam: 100%
  - Aspiration: SAM
- iii. Titration
  - 1. Titrate the contents of the flask with 0.5 M hydrochloric acid standard solution.
  - 2. Record the volume of the hydrochloric acid standard solution required.

## Calculation

Total nitrogen (%) = 
$$\frac{(V-B) \times N \times 1.4}{S}$$
 (3)

Protein (%) = total nitrogen (%) × 5.95

V = Volume (ml) of hydrochloric acid standard solution required for the test portion

B = Volume (ml) of hydrochloric acid standard solution required for the blank test

N = Normality factor of hydrochloric acid standard solution

A.4 Ash content (AOAC section 32.1.05, 2005)

#### <u>Apparatus</u>

- 1. Muffle furnace (Fisher Scientific, model Isotemp, USA)
- 2. Crucible
- 3. Hot plate
- 4. Weighing machine (Scaltec, model SBC series, Germany)
- 5. Desiccator

- 1. Place the crucibles into a muffle furnace at 550°C for 30 minutes or more
- Remove the crucibles, cool to room temperature in a desiccator for at least 1 hour and weigh each crucible accurately. (W1)

- 3. Accurately weigh 3 g of sample into each crucible.  $(W_2)$
- 4. Place the crucibles on a hot plate under a fume-hood and slowly increase the temperature until the samples become thoroughly charred.
- 5. Place the crucibles inside the muffle furnace and ash at 550°C.
- 6. Remove the crucibles from the muffle and place in a desiccator for at least 1 hour to allow to cool. (The ash should be clean and white in appearance.)
- 7. By difference, calculate the weight of ash.  $(W_3)$

## Calculation

Ash (%) = 
$$\frac{W_3 - W_1}{W_2 - W_1} \times 100$$
 (4)

W1: Weight (g) of crucible

 $W_2$ : Weight (g) of sample with crucible before ashing

W<sub>3</sub>: Weight (g) of sample with crucible after ashing

### A.5 Lipid content (AOAC section 32.1.13, 2005)

#### Reagent

Petroleum ether (A.R. grade)

#### <u>Apparatus</u>

- 1. Soxhlet (Gerhardt, model HC61, Germany)
- 2. Extraction thimbles
- 3. Evaporator (Eyela, model SB-651, Japan)
- 4. Weighing machine (Scaltec, model SBC series, Germany)

- Weigh 4 g of the dried sample, i.e. the dried residue obtained in the determination of moisture content, on a piece of filter paper (Whatman No.1) and transfer to an extraction thimble.
- 2. Place the thimble in the extractor and connect a weighed flask containing 250 ml petroleum ether. Connect the extractor to a reflux condenser.
- 3. Extract fat, under reflux, for 4 hours.
- 4. Remove the solvents by evaporation.

5. Dry the flask in an air oven at  $100^{\circ}$ C for 1 hour, cool in a desiccator and weigh. *Calculation* 

Fat (%) = 
$$\frac{W - W_0}{S} \times 100$$
 (5)

S: Weight (g) of sample before drying

W<sub>0</sub>: Weight (g) of flask without fat

W: Weight (g) of flask with fat

A.6 Crude fiber (AOAC section 32.1.15, 2005)

### Reagent

- 1. 1.25% (v/v) Sulfuric acid solution (A.R. grade)
- 2. 1.25 % (w/v) Sodium hydroxide solution (A.R. grade)
- 3. 95% Ethanol

#### <u>Apparatus</u>

- 1. Büchner funnel
- 2. Crucible
- 3. Hot-air oven (Memmert, model W350, Germany)
- 4. Muffle furnace (Fisher Scientific, model Isotemp, USA)
- 5. Weighing machine (Scaltec, model SBC series, Germany)
- 6. Desiccator

- 1. Transfer the defat residue obtained from the determination of lipid content ( $W_1$ ) into 600 ml beaker.
- 2. Add 200 ml of 1.25% sulfuric acid and heat to boiling within 30 minutes.
- 3. Filter the contents of the beaker through a Büchner funnel prepared with a filter paper (Whatman No.1).
- 4. Wash with boiling water until acid-free.
- 5. Wash the sample back into the original beaker with 200 ml of 1.25% sodium hydroxide and heat to boiling within 30 minutes.

- 6. Filter the contents of the beaker through a Büchner funnel prepared with a filter paper (Whatman No.42).
- 7. Wash with boiling water until base-free.
- 8. Wash twice with 25 ml of alcohol
- Transfer all unsoluble matter to the aluminum dish and dry at 100°C until constant weight (W<sub>2</sub>).
- Transfer the dried sample to a crucible and ash in muffle furnace at 550°C until the ash should be clean and white in appearance.
- 9. Cool the crucible in a desiccator and weigh  $(W_3)$ .

# Calculation

Crude fiber (%) = 
$$\frac{W_2 - W_3}{W_1} \times 100$$
 (6)

W<sub>1</sub>: Weight (g) of sampleW<sub>2</sub>: Weight (g) of unsoluble matter

W<sub>3</sub>: Weight (g) of ash

#### A.7 Carbohydrates

Calculation

Carbohydrates (%) = 
$$100 - \%$$
 (Protein + Ash + Crude fiber + Fat) (7)

A.8 Physical properties of rice (Singh et al., 2005)

### <u>Apparatus</u>

- 1. Weighing machine (Scaltec, model SBC series, Germany)
- 2. Vernier Caliper (Mitutoyo, Japan)

### A.8.1 1000-Kernel weight

### Procedure

One thousand head rice kernels of milled rice were counted randomly in triplicate and weighed separately. Mean of three replications was reported.

#### A.8.2 Length-breadth ratio (L/B)

#### **Procedure**

Length- and breadth-wise arrangement of milled rice was done and their cumulative measurements (in mm) were taken. The value of L/B was determined by dividing length by breadth. A mean of 10 replications was reported.

#### A.8.3 Bulk density

#### <u>Procedure</u>

Milled rice kernels from different cultivars were poured into a certain known volume from a fixed height and mass of samples occupying the volume was determined. Ratio was calculated as g/ml.

#### A.9 Color measurement (L\* a\* b\*)

#### <u>Apparatus</u>

1. Chroma meter (Minolta Chroma meter, model CR 300 series, Japan)

#### <u>Procedure</u>

- 1. A sample is placed on the granular materials attachment and compacted.
- 2. The Minolta Chroma Meter is inserted into the granular materials attachment.
- 3. Measurements are taken and recorded.

L\* value = whiteness (100 white, 0 black)

a\* value = red or green color (positive values are red color,

negative values are green color)

b\* value = yellow or blue color (positive values are yellow color, negative values are blue color)

### A.10 Cooking properties of rice (Singh et al., 2005)

#### <u>Apparatus</u>

- 1. Weighing machine (Scaltec, model SBC series, Germany)
- 2. Vernier Caliper (Mitutoyo, Japan)
- 3. Hot-air oven (Memmert, model W350, Germany)

- 4. Aluminum dish
- 5. Desiccator
- 6. Water bath (Memmert, model WNB 22, Germany)

#### A.10.1 Minimum cooking time

#### **Procedure**

Head rice (1 g) samples were taken in a test tube from each variety and cooked in 10 ml distilled water in a boiling water bath. The cooking time was determined by removing a few kernels at different time intervals during cooking and pressing them between two glass plates until no white core was left.

#### A.10.2 Water uptake ratio

#### <u>Procedure</u>

Head rice samples (1 g) for each cultivar were cooked in 10 ml distilled water for a minimum cooking time in a boiling water bath. The contents were drained and the superficial water on the cooked rice was sucked by pressing the cooked samples in filter paper sheets. The cooked samples were then weighed accurately and the water uptake ratio was calculated.

#### A.10.3 Elongation ratio

#### Procedure

Cumulative length of 10 cooked rice kernels was divided by length of 10 uncooked raw kernels and the result was reported as elongation ratio.

### A.10.4 Gruel solid loss

#### Procedure

Head rice samples (1 g) in 10 ml distilled water, for each cultivar, were cooked for minimum cooking time in a boiling water bath. The gruel were transferred to aluminum dish and evaporated at 100°C in an oven until completely dry. The solids were weighed and percent gruel solids were reported.

### A.10.5 Cooked length-breadth ratio

### **Procedure**

This was determined by dividing the cumulative length of 10 cooked kernels by the breadth of 10 cooked kernels. A mean of 10 replications was reported.

### A.10.6 Volume expansion ratio

#### **Procedure**

Head rice samples (1 g) in 10 ml distilled water, for each cultivar, were cooked for minimum cooking time in a boiling water bath. Volume expansion ratio as calculated as the ratio of the volume of the cooked rice to the initial volume of the raw rice.

A.11 Textural properties (the method modified from that of Park et al., 2001)

#### <u>Apparatus</u>

1. Texture analyzer (Stable Micro System, model TA-XT2i, UK)

- Cooked rice (5 g) was molded into a block using cylindrical container (2 cm diameter × 1 cm depth) for testing.
- 2. Calibrate force with 1 kg load cell.
- 3. Use 36 mm cylindrical probe (P36/R).
- 4. Calibrate height
  - Return distance 20 mm
  - Return speed 10 mm
  - Contact force 10
- 5. Use mode two-cycle compression
  - Mode TPA
  - Test speed 1 mm/sec
  - Target mode 50% strain
  - Time 3 sec
  - Post-test speed 10 mm/sec
  - Pre-test speed 1 mm/sec

- Trigger force 2 g

- 6. Parameters which recorded from the test curve were hardness adhesiveness springiness cohesiveness chewiness and gumminess by Exponent Lite Express software, version 3.11.
- 7. Use 10 sample test cell for 1 replicate.

A.12 Amylose content (lodine binding method; Juliano, 1971)

# Reagent

- 1. Amylose type III: from potato (Sigma- ALDRICH Company, Germany)
- 2. 1N Sodium hydroxide solution (A.R. grade)
- 3. 95% Ethanol
- 4. 1N Acetic acid solution
- 5. Iodine solution (0.2%  $I_2$  in 2% KI)

## <u>Apparatus</u>

- 1. Spectrophotometer (Thermo Spectronic, model Genesys 10 UV, USA)
- 2. Weighing machine (Scaltec, model SBC series, Germany)

### **Procedure**

# For standard curve

- 1. Weigh 0.0400 g of amylose and transfer to 50 ml flask.
- Add 1 ml of 95% ethanol and 9 ml of 1N sodium hydroxide solution, mix thoroughly.
- Heat in boiling water bath within 10 minutes, and cool down to room temperature.
- Transfer to 100 ml volumetric flask, dilute to volume with distilled water and mix well.
- 5. Pipette 1 2 3 4 and 5 ml aliquot into 100 ml volumetric flask.
- 6. Add 0.2 0.4 0.6 0.8 and 1.0 ml of acetic acid solution.
- 7. Add 2 ml of iodine solution, dilute to volume with distilled water.
- 8. Mix well and stand for 20 min.

9. Read absorbance at 620 nm against the blank.

#### For samples

- 1. Weigh 100 mg of sample and transfer to a 50 ml flask.
- Add 1 ml of 95% ethanol and 9 ml of 1N sodium hydroxide solution, mix thoroughly.
- 3. Heat in boiling water bath within 10 minutes to gelatinize the starch, and cool down to room temperature.
- 4. Transfer entire content into 100 ml volumetric flask, dilute to volume with distilled water and mix well.
- 5. Pipette 5 ml aliquot into 100 ml volumetric flask.
- Add 1N acetic acid solution and 2 ml iodine solution the volume was adjusted to 100 ml with distilled water
- 7. Mix well and incubate at room temperature for 20 min.
- 8. Read absorbance at 620 nm.
- The amylose content was determined from a previous standard curve of potato amylose.

Calculation

Amylose content (%) = 
$$\frac{A}{W} \times 100 \times 20$$
 (8)

A: Amylose content (g) from standard curve (X-axis)

W: Weight (g) of sample

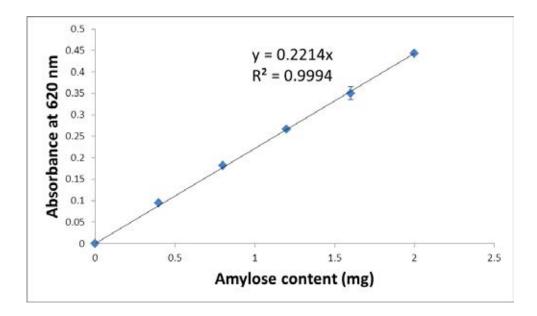


Figure A.1 Standard curve for determining amylose content

A.13 Particle size analysis (the method modified from that of Park et al., 2010)

# <u>Reagent</u>

1. 95% Ethanol

### <u>Apparatus</u>

- Multi-wavelength Particle Size Analyzer (Beckman Coulter, model LS 13 320, USA)
- 2. Universal Liquid Module
- 3. Sonication control Unit
- 4. Sonicator

### Procedure

### For sample powder

- 1. Connect the particle size analyzer with the universal liquid module.
- 2. Universal liquid module run cycle
  - Include PIDS data (Polarization Intensity Differential Scattering)
  - Auto rinse
  - Measure offsets
  - Align

- Measure background
- Measure loading
- Enter sample info
- Enter run setting
- Start run(s)
- 3. Sample is sonicated for 90 seconds on the sonication control unit.
- 4. Pump speed: 60%

Fluid: Ethyl alcohol

R.I.(Refractive index): 1.3611

- 5. The samples are loaded into the universal liquid module until PIDS settings are attained.
- 6. The sample is re-circulated in a closed-loop system.
- Measurements are taken and recorded with Beckman Coulter LS software, version 5.01.

#### For sample solution

- 1. Samples are pretreated with 95% ethanol overnight.
- 2. Sonicate each sample with the independent sonicator for 20 minute to disperse the sample before analyst.
- 3. Run method same the sample powder.

### A.14 Microscopy

#### Reagent

50% glycerol solution

### <u>Apparatus</u>

1. Microscope (Olympus, model CH30RF200, Japan)

- 1. Drop 1-2 drops of 50% glycerol solution on glass slide.
- 2. Suspend the flour sample in the solution.
- 3. View under a polarized light microscope equipped with a camera set.

A.15 Examination of starch granules (Scientific and Technological Research Equipment Centre (STREC), Chulalongkorn University)

## <u>Apparatus</u>

- 1. Scanning electron microscope (SEM) (JEOL, model JSM-5800 LV, Japan)
- 2. Ion sputter (Balzers Union, model SCD 040, Liechtenstein)

### <u>Procedure</u>

- 1. Samples were mounted on aluminum stubs using double-sided tape.
- 2. Sputter-coated with gold.
- 3. Investigated using SEM at an accelerated voltage of 20 kV.

A.16 X-ray Diffraction pattern (Scientific and Technological Research Equipment Centre (STREC), Chulalongkorn University)

### <u>Apparatus</u>

1. X-ray diffractometer (Bruker AXS, model D8 Discover, Germany)

### Procedure

- 1. Rice flours were packed tightly into the aluminum sample holder.
- 2. operated at condition

Target: Cu

Voltage: 40 kV

Current: 40 mA

Angle: 5 – 30 degree

Increment: 0.02 degree/ step

Scan speed: 0.5 sec/ step

Detector: VÅNTEC-1 Detector (Super Speed Detector)

 Relative crystallinity degree (%) was estimated from the ratio of the peak area to the total area of a diffractogram by using Topas software, version 3. Calculation

Degree of crystallinity (%) = 
$$\frac{A_C}{A_L} \times 100$$
 (9)

 $A_c$  = Area under curve of crystalline region

 $A_t$  = Area under curve of total from baseline

A.17 Water binding capacity (Medcalf and Gilles, 1965)

#### <u>Apparatus</u>

- 1. Centrifuge (Centrifuge Thermo IEC, model IEC Multi-RF, USA)
- 2. Centrifuge tube
- 3. Shaker water bath (Thermo Scientific, model NESLAB EX 10, USA)
- 4. Weighing machine (Scaltec, model SBC series, Germany)

### **Procedure**

- 1. Weigh 1 g of sample  $(W_1)$  and transfer to centrifuge tube.
- 2. Add 15 ml distilled water and vortex.
- 3. Incubate in a shaker water bath at 30°C, 174 rpm for 30 min.
- 4. Centrifuge at 5000 × g for 20 min.
- 5. Supernatant was decanted carefully and the residue was weighed  $(W_2)$ .

#### Calculation

Water binding capacity = 
$$\frac{W_2 - W_1}{W_1}$$
 (10)

W<sub>1</sub>: Weight (g) of sample before analyst

W<sub>2</sub>: Weight (g) of sample after analyst

A.18 Swelling power and solubility (Schoch, 1964)

### <u>Apparatus</u>

- 1. Centrifuge (Centrifuge Thermo IEC, model IEC Multi-RF, USA)
- 2. Centrifuge tube
- 3. Shaker water bath (Thermo Scientific, model NESLAB EX 10, USA)
- 4. Weighing machine (Scaltec, model SBC series, Germany)

- 5. Hot-air oven (Memmert, model W350, Germany)
- 6. Aluminum dish

#### **Procedure**

- 1. Weigh 0.5 g of sample  $(W_1)$  and transfer to centrifuge tube.
- 2. Add 15 ml distilled water and vortex.
- Incubate in a shaker water bath at 60°C 70°C 80°C and 90°C, 174 rpm for 30 min.
- 4. Cooled to room temperature and centrifuge at  $6000 \times g$ ,  $4^{\circ}C$  for 20 min.
- Supernatant was decanted carefully and kept, and the residue was weighed (W<sub>2</sub>).
- 6. The supernatant was poured out from the tube to an aluminum dish.
- 7. The dish was dried at  $105^{\circ}$ C in hot-air oven to constant and weighed (W<sub>3</sub>). *Calculation*

Swelling power = 
$$\frac{W_2 \times 100}{W_1 \times (100 - \% \ solubility)}$$
(11)

% Solubility = 
$$\frac{W_3}{W_1} \times 100$$

W<sub>1</sub>: Weight (g) of sample before analyst

W<sub>2</sub>: Weight (g) of the wet sediment

 $W_3$ : Weight (g) of the dried supernatant

A.19 Pasting properties (Norbert et al., 1995)

### <u>Apparatus</u>

- 1. RVA (Rapid Visco Analyzer) (Newport Scientific, model 4D, Australia)
- 2. Metal RVA canister with paddle
- 3. Weighing machine (Mettler Toledo, model AB204, Switzerland)

## Procedure

1. 25 ml of distilled water was added directly to a metal RVA canister.

- 3.00 ± 0.01 g of rice flour was weighed, added to the water, and immediately measured by RVA.
- 3. Temperature profile

Time (min.sec)	Temperature (°C)	Speed (rpm)
0.00	50	960
0.10	50	160
1.00	50	160
4.42	95	160
7.12	95	160
11.00	50	160
13.00	50	160

4. Viscosity parameters (peak, trough, final, breakdown, and setback viscosity) were expressed in centipoise (cP) by Thermocline software, version 3.11.

## A.20 Thermal properties (Kim et al., 1995)

### <u>Apparatus</u>

- 1. Differential Scanning Calorimeter (Perkin-Elmer, model Diamond, USA)
- 2. Intracooler unit (Perkin-Elmer, model 2P, USA)
- 3. Nitrogen gas purge
- 4. Aluminum volatile sample pan (Perkin-Elmer, USA)
- 5. Micropipette
- 6. Weighing machine (Ohaus, model Explorer, Switzerland)

- 1. Weigh 3 mg of sample and transfer to aluminum volatile sample pan.
- 2. Add distilled water to give a flour : water ratio of 3 : 7.
- 3. The pans were sealed hermetically to prevent moisture loss and kept overnight.
- 4. A sealed empty aluminum pan was used as reference.

- After equilibration at room temperature overnight, the sample was heated from 30 to 95°C at a rate of 10 °C/min.
- 6. The onset (To), peak (Tp) and conclusion (Tc) temperatures of gelatinization, and the gelatinization enthalpy ( $\Delta$ H in J/g) were recorded by Pyris software, version 8.0.0.0172.

A.21 Viscoelastic properties (the method modified from that of Lii *et al.*, 1996) <u>Apparatus</u>

1. Rheometer with geometry (Bohlin Instrument, model CVOR 150, UK)

### Procedure

- Flour suspensions (12% w/w) was prepared and kept for 1 hour at room temperature.
- 2. Equip geometry (parallel plate 40 mm diameter) with rheometer.
- 3. Set zero and set gap at 500  $\mu m.$
- 4. Use mode oscillation

Temperature sweep test

- Stress 40 Pa
- Frequency 1 Hz
- Temperature

 $30^{\circ}C - 85^{\circ}C$  for 4 min

 $85^{\circ}C - 30^{\circ}C$  for 3 min

Frequency sweep test

- Stress 10 Pa
- Minimum frequency 0.1 Hz
- Maximum frequency 100 Hz
- Isothermal 25°C
- 5. Load the sample suspension into the rheometer plate and adjust the parallel plate into the gap size.

- 6. Drop 3-4 drops of immersion oil to prevent the evaporation and cover with closure.
- 7. Start to run the temperature sweep test to gelatinized starch and continue to the frequency sweep test.
- 8. Storage and loss modulus were recorded with Bohlin software, version 6.32.2.0.

# APPENDIX B

# Additional data and figure

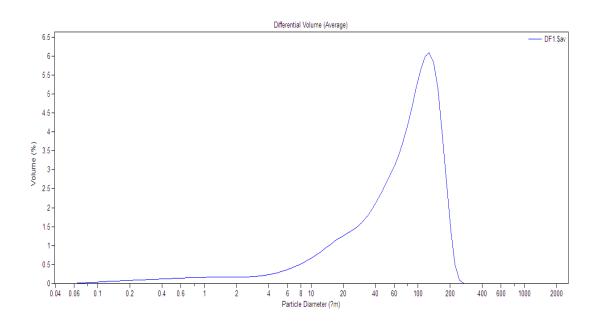


Figure B.1 Particle size distribution (µm) of RD 41 cultivar in dry-milled rice flour

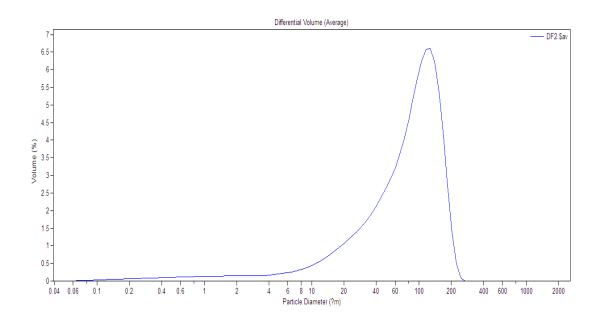


Figure B.2 Particle size distribution ( $\mu$ m) of RD 45 cultivar in dry-milled rice flour

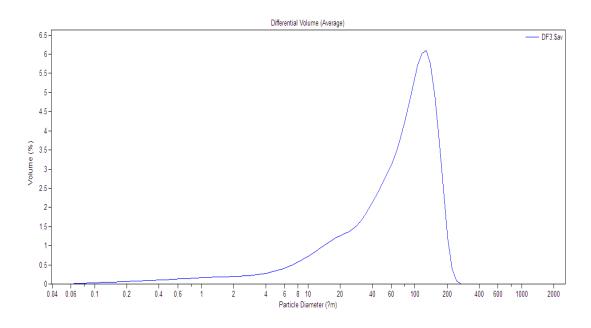


Figure B.3 Particle size distribution  $(\mu\text{m})$  of RD 47 cultivar in dry-milled rice flour

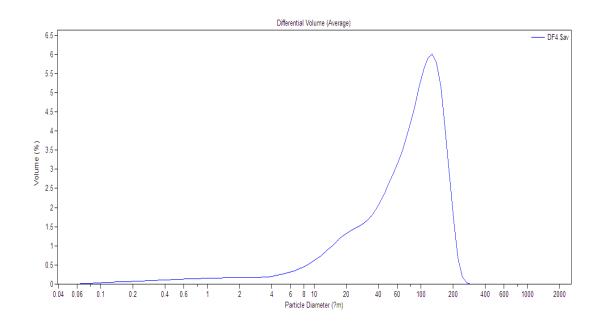


Figure B.4 Particle size distribution ( $\mu$ m) of Shaw Lung 97 cultivar in dry-milled rice flour

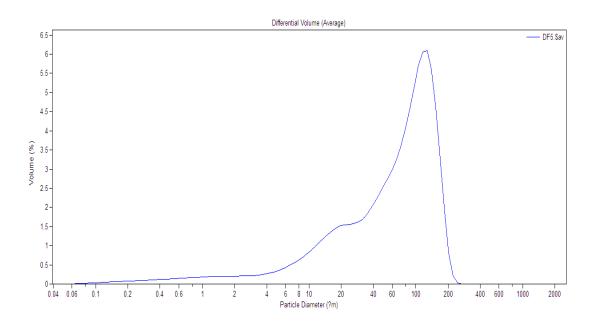


Figure B.5 Particle size distribution ( $\mu$ m) of Prachin Buri 1 cultivar in dry-milled rice flour

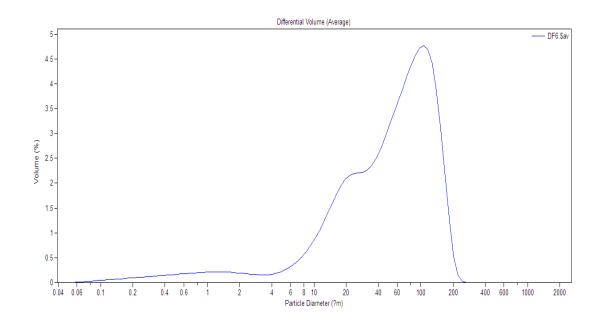


Figure B.6 Particle size distribution ( $\mu$ m) of Prachin Buri 2 cultivar in dry-milled rice flour

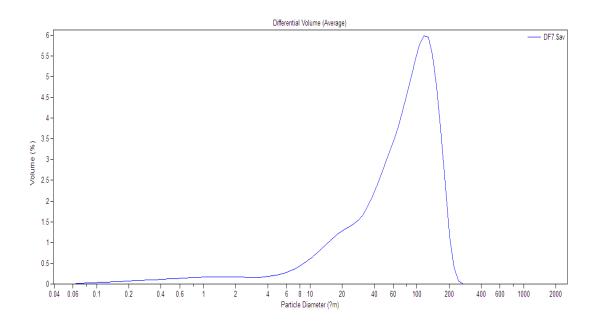


Figure B.7 Particle size distribution ( $\mu$ m) of Plai Ngahm Prachin Buri cultivar in dry-milled rice flour

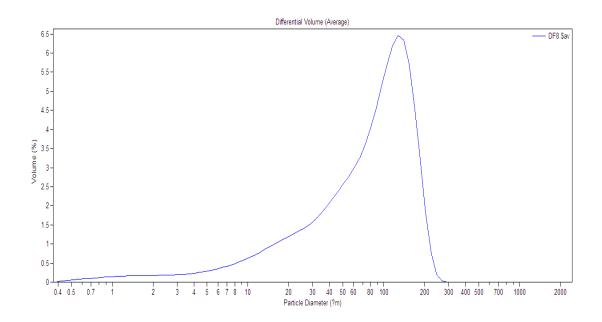


Figure B.8 Particle size distribution ( $\mu$ m) of Khao Dawk Mali 105 cultivar in dry-milled rice flour

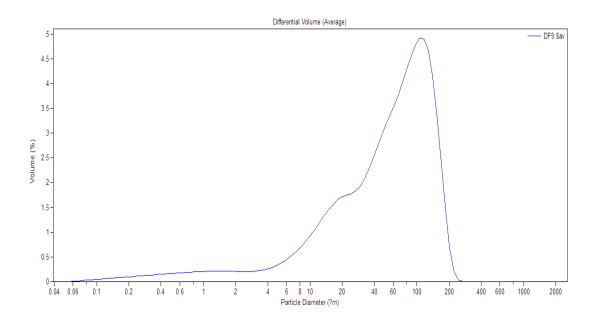


Figure B.9 Particle size distribution (µm) of Ayutthaya 1 cultivar in dry-milled rice flour

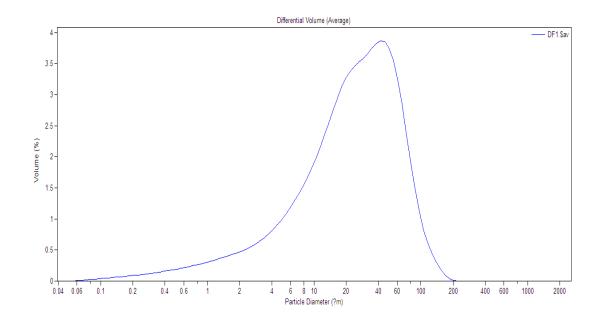


Figure B.10 Particle size distribution ( $\mu$ m) of RD 41 cultivar in dry-milled rice flours (soaked overnight in alcohol)

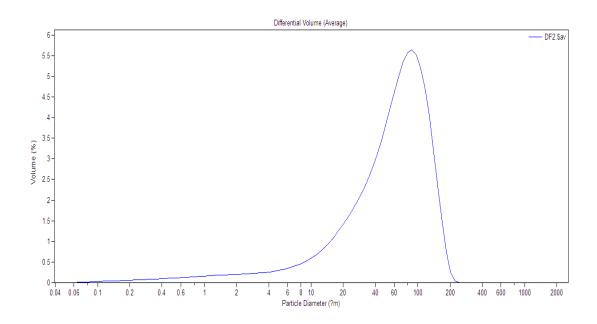


Figure B.11 Particle size distribution ( $\mu$ m) of RD 45 cultivar in dry-milled rice flours (soaked overnight in alcohol)

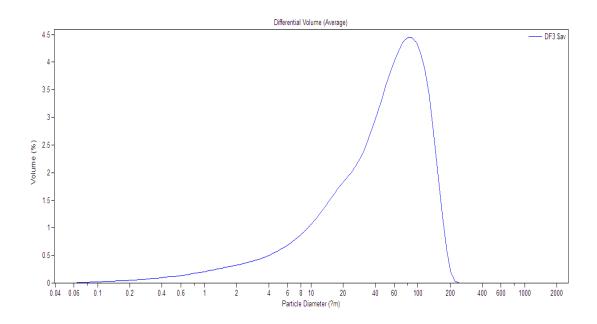


Figure B.12 Particle size distribution ( $\mu$ m) of RD 47 cultivar in dry-milled rice flours (soaked overnight in alcohol)

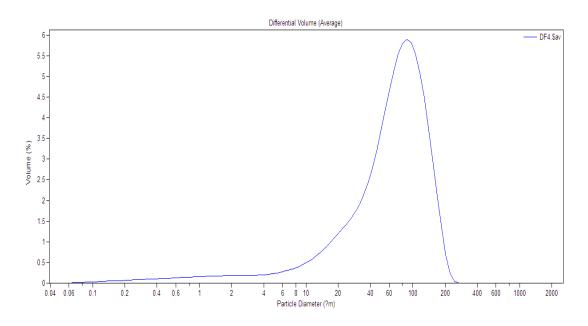


Figure B.13 Particle size distribution ( $\mu$ m) of Shaw Lung 97 cultivar in dry-milled rice flour (soaked overnight in alcohol)

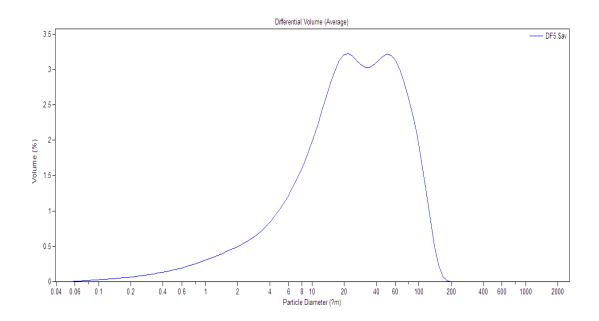


Figure B.14 Particle size distribution ( $\mu$ m) of Prachin Buri 1 cultivar in dry-milled rice flour (soaked overnight in alcohol)

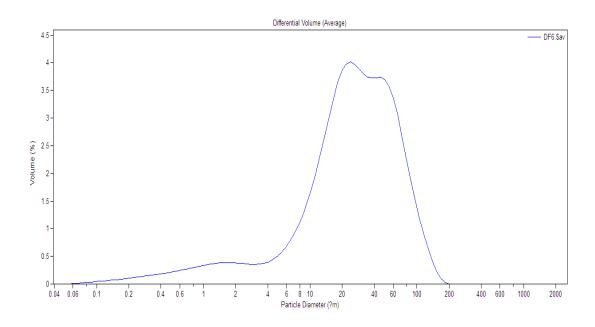


Figure B.15 Particle size distribution ( $\mu$ m) of Prachin Buri 2 cultivar in dry-milled rice flour (soaked overnight in alcohol)

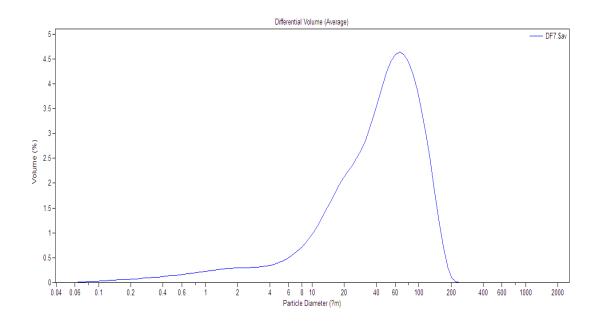


Figure B.16 Particle size distribution ( $\mu$ m) of Plai Ngahm Prachin Buri cultivar in drymilled rice flour (soaked overnight in alcohol)

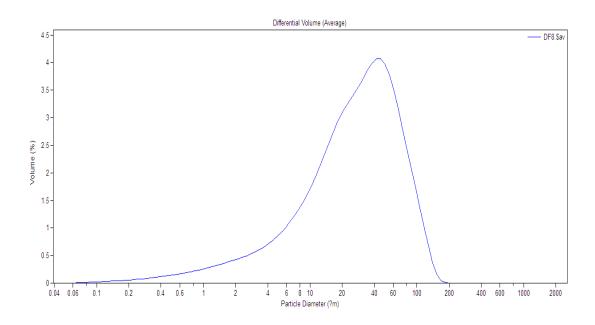


Figure B.17 Particle size distribution ( $\mu$ m) of Khao Dawk Mali 105 cultivar in dry-milled rice flour (soaked overnight in alcohol)

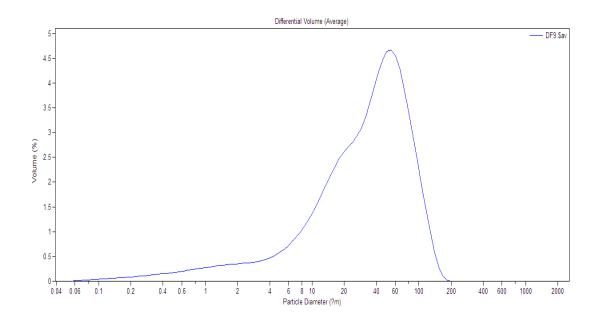


Figure B.18 Particle size distribution ( $\mu$ m) of Ayutthaya 1 cultivar in dry-milled rice flour (soaked overnight in alcohol)

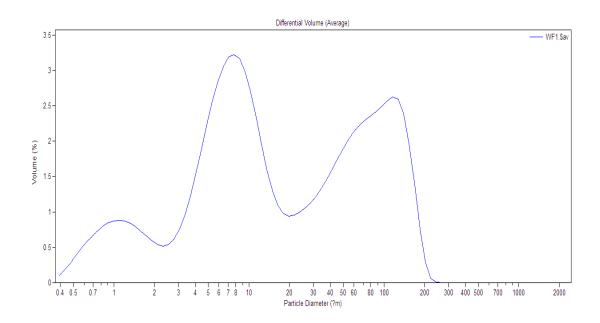


Figure B.19 Particle size distribution (µm) of RD 41 cultivar in wet-milled rice flours

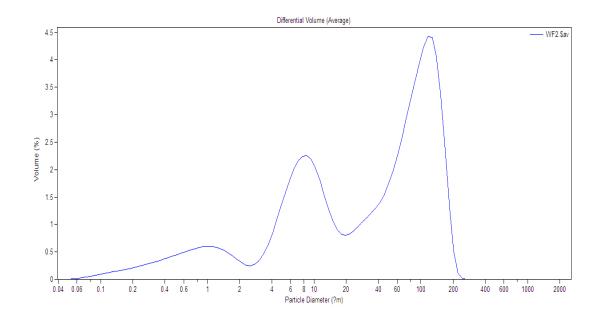


Figure B.20 Particle size distribution ( $\mu$ m) of RD 45 cultivar in wet-milled rice flours

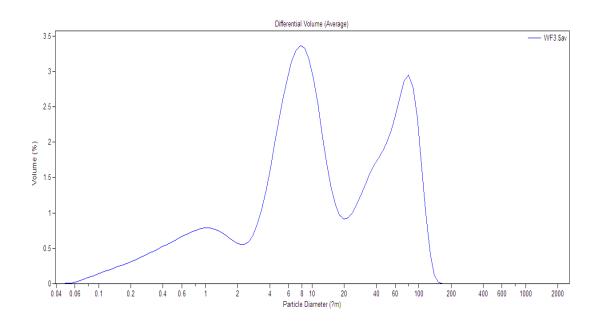


Figure B.21 Particle size distribution ( $\mu$ m) of RD 47 cultivar in wet-milled rice flours

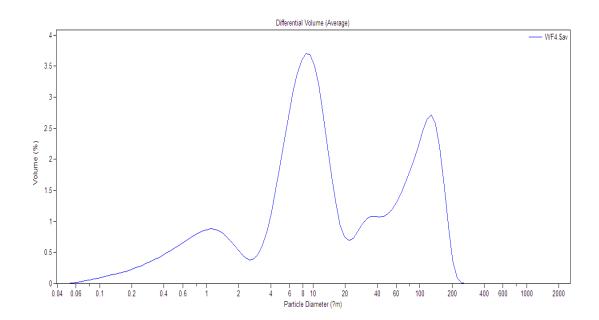


Figure B.22 Particle size distribution ( $\mu$ m) of Shaw Lung 97 cultivar in wet-milled rice flour

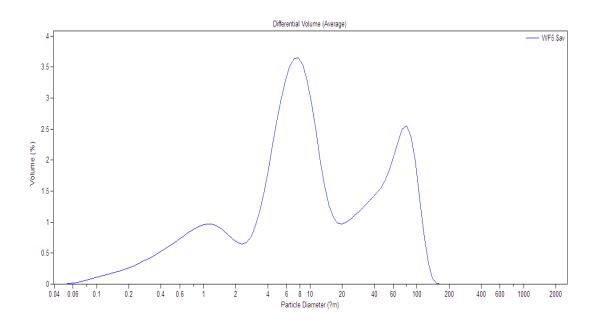


Figure B.23 Particle size distribution ( $\mu$ m) of Prachin Buri 1 cultivar in wet-milled rice

flour

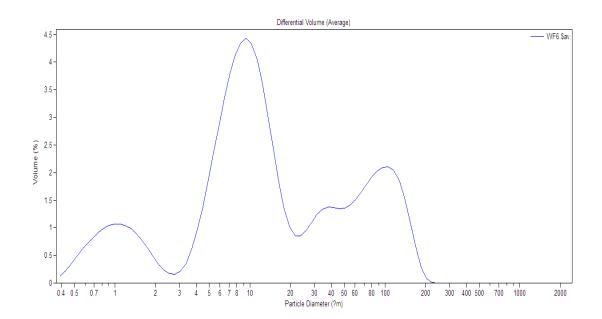


Figure B.24 Particle size distribution ( $\mu$ m) of Prachin Buri 2 cultivar in wet-milled rice flour

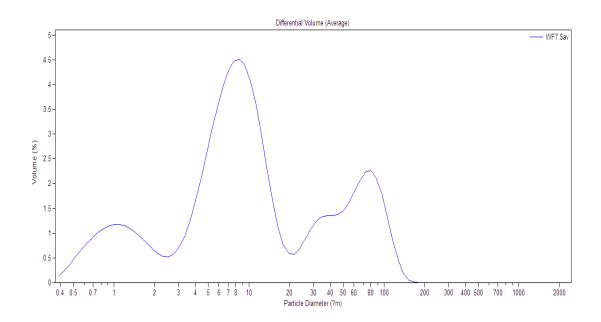


Figure B.25 Particle size distribution ( $\mu$ m) of Plai Ngahm Prachin Buri cultivar in wet-milled rice flour

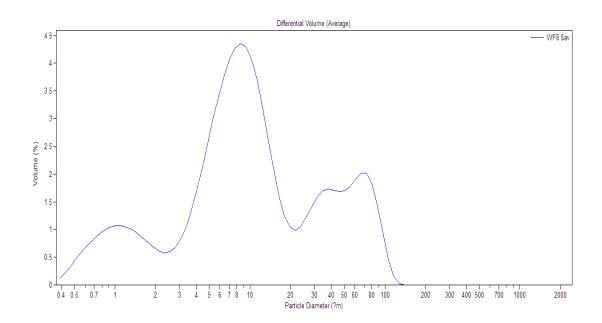


Figure B.26 Particle size distribution ( $\mu$ m) of Khao Dawk Mali 105 cultivar in wet-milled rice flour

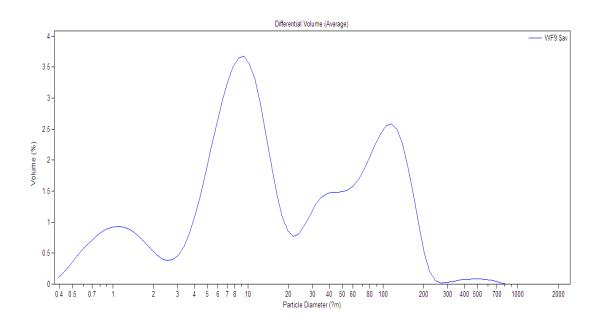


Figure B.27 Particle size distribution  $(\mu m)$  of Ayutthaya 1 cultivar in wet-milled rice flour

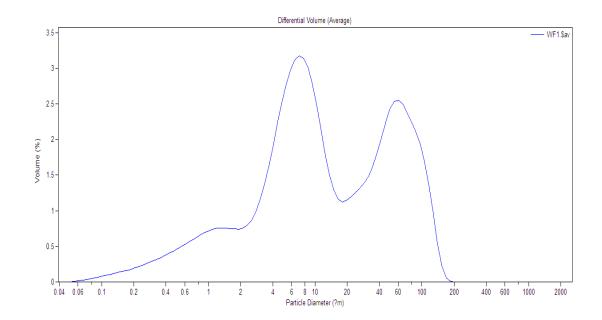


Figure B.28 Particle size distribution ( $\mu$ m) of RD 41 cultivar in wet-milled rice flours (soaked overnight in alcohol)

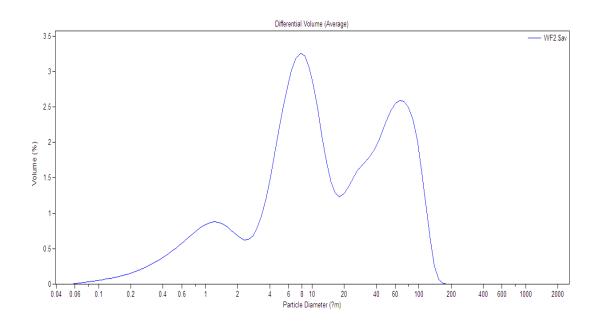


Figure B.29 Particle size distribution ( $\mu$ m) of RD 45 cultivar in wet-milled rice flours (soaked overnight in alcohol)

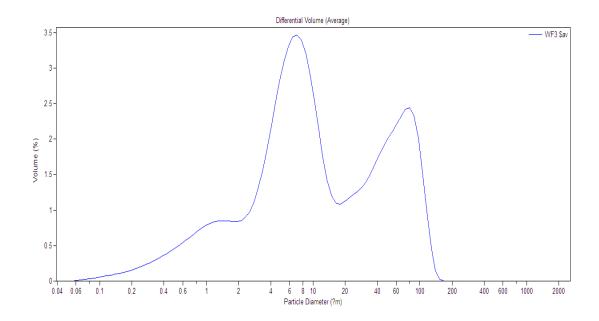


Figure B.30 Particle size distribution ( $\mu$ m) of RD 47 cultivar in wet-milled rice flours (soaked overnight in alcohol)

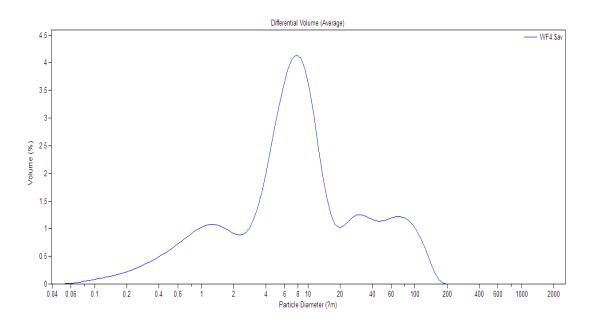


Figure B.31 Particle size distribution ( $\mu$ m) of Shaw Lung 97 cultivar in wet-milled rice flour (soaked overnight in alcohol)

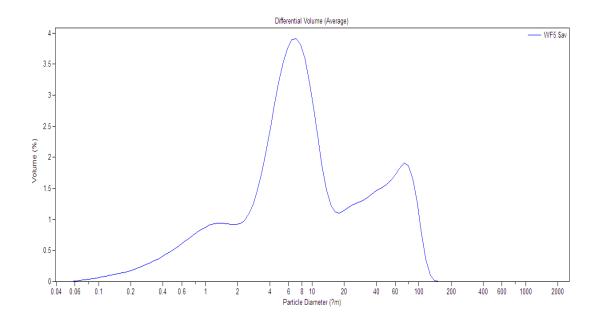


Figure B.32 Particle size distribution ( $\mu$ m) of Prachin Buri 1 cultivar in wet-milled rice flour (soaked overnight in alcohol)

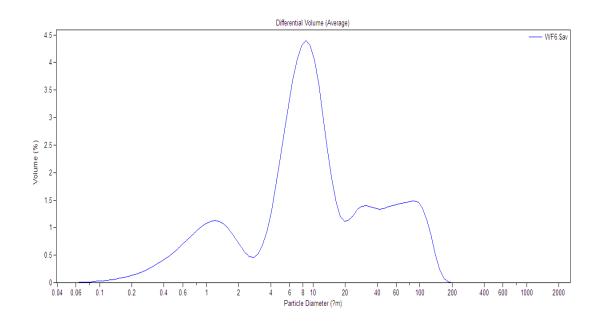


Figure B.33 Particle size distribution ( $\mu$ m) of Prachin Buri 2 cultivar in wet-milled rice flour (soaked overnight in alcohol)

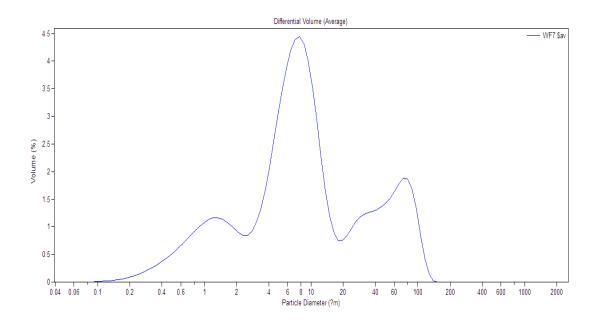


Figure B.34 Particle size distribution ( $\mu$ m) of Plai Ngahm Prachin Buri cultivar in wetmilled rice flour (soaked overnight in alcohol)

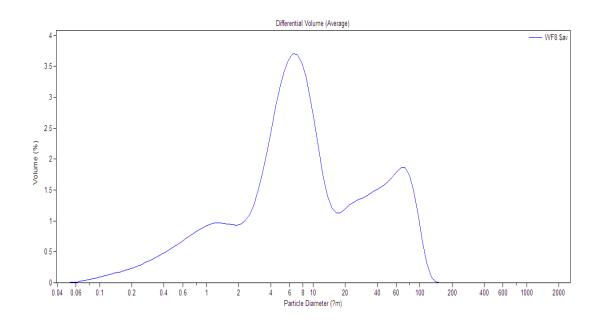


Figure B.35 Particle size distribution ( $\mu$ m) of Khao Dawk Mali 105 cultivar in wet-milled rice flour (soaked overnight in alcohol)

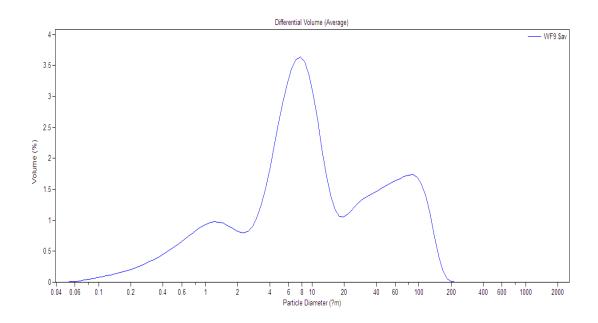


Figure B.36 Particle size distribution ( $\mu$ m) of Ayutthaya 1 cultivar in wet-milled rice flour (soaked overnight in alcohol)

Cultivars	L*	a*	b*
RD 41	50.7 <sup>e</sup> ± 1.1	$0.13^{\circ} \pm 0.02$	6.46 <sup>°</sup> ± 0.31
RD 45	$49.2^{f} \pm 0.3$	$0.21^{b} \pm 0.03$	$5.96^{abc} \pm 0.48$
RD 47	$47.6^{g} \pm 0.5$	$0.35^{a} \pm 0.04$	$5.67^{bcd} \pm 0.16$
Shaw Lung 97	$57.2^{a} \pm 0.4$	$-0.47^{f} \pm 0.01$	$6.32^{ab} \pm 0.31$
Prachin Buri 1	$50.9^{de} \pm 0.6$	$0.15^{\circ} \pm 0.02$	$5.69^{bcd} \pm 0.08$
Prachin Buri 2	$53.5^{\circ} \pm 0.7$	$0.11^{\circ} \pm 0.02$	$5.48^{cd} \pm 0.14$
Plai Ngahm Prachin Buri	$54.4^{\circ} \pm 0.2$	$-0.46^{f} \pm 0.05$	$5.05^{d} \pm 0.72$
Khao Dawk Mali 105	$51.8^{d} \pm 0.5$	$-0.26^{d} \pm 0.03$	$6.27^{ab} \pm 0.21$
Ayutthaya 1	$55.8^{b} \pm 0.8$	-0.35 <sup>e</sup> ± 0.02	$6.50^{a} \pm 0.28$

Table B.1 Color parameter of rice grains

Mean ± standard deviation values in the same column followed by different superscripts are

significantly different (P  $\leq$  0.05)

Cultivars	L*	a*	b*
RD 41	$77.3^{\circ} \pm 0.0$	$-0.83^{a} \pm 0.03$	$2.86^{d} \pm 0.06$
RD 45	$77.2^{\circ} \pm 0.1$	-0.95 <sup>°</sup> ± 0.03	$3.11^{bc} \pm 0.09$
RD 47	75.9 <sup>e</sup> ± 0. 5	-0.84 <sup>a</sup> ±0.04	$3.21^{b} \pm 0.13$
Shaw Lung 97	$77.3^{\circ} \pm 0.1$	$-0.83^{a} \pm 0.03$	$3.48^{a} \pm 0.04$
Prachin Buri 1	$77.6^{bc} \pm 0.2$	$-0.90^{b} \pm 0.02$	$2.99^{cd} \pm 0.06$
Prachin Buri 2	77.9 <sup>ab</sup> ± 0.1	-0.96 <sup>°</sup> ± 0.01	$2.40^{e} \pm 0.02$
Plai Ngahm Prachin Buri	$78.2^{a} \pm 0.3$	$-1.07^{d} \pm 0.02$	$2.16^{f} \pm 0.04$
Khao Dawk Mali 105	$76.8^{d} \pm 0.3$	$-0.83^{a} \pm 0.03$	3.53 <sup>ª</sup> ± 0.17
Ayutthaya 1	77.8 <sup>ab</sup> ± 0.1	$-1.06^{d} \pm 0.01$	$2.85^{d} \pm 0.06$

### Table B.2 Color parameter of dry-milled flours

Mean  $\pm$  standard deviation values in the same column followed by different superscripts are

Cultivars	L*	a*	b*
RD 41	$78.5^{\circ} \pm 0.4$	-0.97 <sup>a</sup> ± 0.01	$1.34^{b} \pm 0.05$
RD 45	$77.4^{d} \pm 0.6$	$-0.99^{a} \pm 0.04$	$1.26^{\circ} \pm 0.07$
RD 47	$79.3^{b} \pm 0.5$	-1.10 <sup>b</sup> ±0.03	$1.11^{d} \pm 0.01$
Shaw Lung 97	$79.2^{b} \pm 0.2$	$-1.00^{a} \pm 0.03$	$1.46^{a} \pm 0.03$
Prachin Buri 1	$80.0^{a} \pm 0.1$	-1.16 <sup>°</sup> ± 0.01	$1.07^{de} \pm 0.01$
Prachin Buri 2	$79.3^{b} \pm 0.1$	-1.14 <sup>°</sup> ± 0.01	$1.09^{de} \pm 0.06$
Plai Ngahm Prachin Buri	$79.7^{ab} \pm 0.2$	$-1.20^{d} \pm 0.01$	$0.84^{g} \pm 0.04$
Khao Dawk Mali 105	$80.1^{a} \pm 0.0$	$-1.09^{b} \pm 0.03$	1.03 <sup>ef</sup> ± 0.01
Ayutthaya 1	$79.8^{ab} \pm 0.0$	-1.23 <sup>d</sup> ± 0.01	$0.98^{f} \pm 0.02$

Table B.3 Color parameter of wet-milled flours

Mean ± standard deviation values in the same column followed by different superscripts are

Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
15.3218	5.7783	2437	86.4
17.3804	5.0982	2779	98.5
17.7452	4.9942	2711	96.1
18.1967	4.8713	2821	100.0
20.1110	4.4118	2346	83.2
20.3566	4.3591	2342	83.0
23.0075	3.8625	2236	79.3
23.1616	3.8371	2267	80.4
23.3410	3.8080	2206	78.2
26.6351	3.3441	1412	50.1

Table B.4 X-ray diffractogram data of RD 41 dry-milled rice flour

Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
10.1368	8.7193	1416	46.1
11.3353	7.7999	1586	51.6
15.0675	5.8752	2678	87.1
15.2050	5.8224	2741	89.2
17.0569	5.1942	3000	97.6
17.3054	5.1201	3069	99.8
18.0913	4.8995	3074	100.0
19.9148	4.4548	2322	75.5
23.1208	3.8438	2548	82.9
26.6794	3.3386	1396	45.4

Table B.5 X-ray diffractogram data of RD 41 wet-milled rice flour

Table B.6 X-ray diffractogram data of RD 45 dry-milled rice flour

Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
15.0875	5.8675	2391	86.8
17.3089	5.1191	2677	97.2
18.0406	4.9131	2753	100.0
18.3078	4.8420	2650	96.3
20.1684	4.3993	2188	79.5
22.2004	4.0010	2009	73.0
22.8419	3.8942	2243	81.5
23.2411	3.8242	2228	80.9
26.4650	3.3652	1393	50.6

Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
10.2053	8.6608	1396	45.6
11.2536	7.8563	1510	49.3
15.0103	5.8975	2570	83.9
15.1971	5.8254	2659	86.8
17.2542	5.1352	3048	99.5
17.5634	5.0455	2908	94.9
17.7871	4.9826	2923	95.4
18.0888	4.9001	3064	100.0
18.2104	4.8677	3041	99.3
19.8384	4.4717	2141	69.9
23.2541	3.8221	2502	81.7
26.5653	3.3527	1380	45.0

Table B.7 X-ray diffractogram data of RD 45 wet-milled rice flour

Table B.8 X-ray diffractogram	data of RD 47	dry-milled rice flour
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Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
15.0286	5.8903	2408	86.9
15.2027	5.8233	2433	87.8
16.9240	5.2347	2602	94.0
17.2654	5.1319	2770	100.0
18.1075	4.8951	2770	100.0
18.7876	4.7194	2531	91.4
20.1908	4.3945	2370	85.6
23.0726	3.8517	2280	82.3
26.4087	3.3722	1427	51.5

Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
10.0788	8.7693	1506	48.1
11.4172	7.7441	1677	53.5
15.2867	5.7914	2765	88.3
17.1457	5.1675	3114	99.4
17.3510	5.1068	3072	98.1
18.0580	4.9084	3132	100.0
19.7138	4.4997	2435	77.7
20.0308	4.4292	2377	75.9
22.9552	3.8711	2526	80.6
23.1156	3.8446	2592	82.7
26.7928	3.3247	1443	46.1

Table B.9 X-ray diffractogram data of RD 47 wet-milled rice flour

Table B.10 X-ray diffractogram data of Shaw Lung 97 dry-milled rice flour

Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
15.2378	5.8099	2294	90.6
17.1446	5.1678	2492	98.4
18.0055	4.9226	2532	100.0
18.2286	4.8629	2520	99.5
20.1613	4.4009	2031	80.2
22.8927	3.8816	2105	83.1
23.0995	3.8473	2086	82.4
26.6230	3.3456	1294	51.1

Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
9.9854	8.8511	1393	45.2
11.4233	7.7400	1543	50.1
15.2627	5.8005	2792	90.6
17.1924	5.1536	3046	98.8
17.3955	5.0938	3007	97.6
18.1021	4.8966	3081	100.0
19.4777	4.5537	2069	67.2
20.2896	4.3733	2087	67.7
23.1694	3.8358	2549	82.7
26.7487	3.3301	1374	44.6

Table B.11 X-ray diffractogram data of Shaw Lung 97 wet-milled rice flour

Table B.12 X-ray diffractogram data of Prachin Buri 1 dry-milled rice flour

Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
15.1142	5.8572	2184	86.1
15.3416	5.7709	2208	87.1
17.2194	5.1455	2433	96.0
17.9935	4.9259	2485	98.0
18.1975	4.8711	2536	100.0
20.0931	4.4156	2073	81.7
23.1778	3.8345	2094	82.6
26.4951	3.3614	1308	51.6
26.7523	3.3297	1286	50.7

Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
10.2327	8.6378	1419	45.1
11.4935	7.6928	1610	51.1
15.1065	5.8601	2692	85.5
15.2929	5.7891	2683	85.2
17.1820	5.1566	3063	97.3
17.9249	4.9446	3104	98.6
18.0616	4.9075	3148	100.0
20.0763	4.4193	2246	71.4
23.0927	3.8484	2535	80.5
23.3294	3.8099	2506	79.6
23.6048	3.7661	2329	74.0
26.5048	3.3602	1435	45.6

Table B.13 X-ray diffractogram data of Prachin Buri 1 wet-milled rice flour

Table B.14 X-ray diffractogram data of Prachin Buri 2 dry-milled rice flour
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Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
15.3631	5.7628	2550	87.9
16.9892	5.2147	2801	96.6
17.3892	5.0957	2839	97.9
17.8456	4.9664	2876	99.2
18.1957	4.8716	2900	100.0
19.8546	4.4681	2385	82.2
23.2398	3.8244	2317	79.9
26.6149	3.3466	1438	49.6

Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
10.2517	8.6218	1426	44.6
11.3165	7.8128	1543	48.2
15.2477	5.8062	2846	89.0
17.1697	5.1603	3156	98.7
17.3765	5.0994	3141	98.2
17.9720	4.9317	3198	100.0
18.1325	4.8884	3198	100.0
20.2315	4.3857	2115	66.1
23.1161	3.8446	2616	81.8
26.7102	3.3348	1396	43.7

Table B.15 X-ray diffractogram data of Prachin Buri 2 wet-milled rice flour

Table B.16 X-ray diffractogram data of Plai Ngahm Prachin Buri dry-milled rice flour

Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
10.0299	8.8119	1462	49.3
11.3401	7.7966	1625	54.8
15.2083	5.8211	2588	87.2
15.3852	5.7546	2588	87.2
17.2576	5.1342	2967	100.0
17.8236	4.9724	2887	97.3
18.0376	4.9139	2957	99.7
18.3118	4.8410	2917	98.3
20.0753	4.4195	2343	79.0
23.0949	3.8480	2396	80.8
26.7690	3.3277	1409	47.5

Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
10.0516	8.7930	1489	45.4
11.4336	7.7331	1644	50.1
15.1534	5.8421	2878	87.7
17.0565	5.1943	3158	96.3
17.3499	5.1071	3137	95.6
18.0633	4.9070	3280	100.0
18.2297	4.8626	3133	95.5
20.1444	4.4045	2243	68.4
23.0009	3.8636	2694	82.1
23.1434	3.8401	2690	82.0
26.5976	3.3487	1464	44.6

Table B.17 X-ray diffractogram data of Plai Ngahm Prachin Buri wet-milled rice flour

Table B.18 X-ray diffractogram data of Khao Dawk Mali 105 dry-milled rice flour

Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
15.2012	5.8238	2461	85.8
17.2774	5.1284	2804	97.8
18.1081	4.8949	2868	100.0
18.5375	4.7825	2685	93.6
20.4121	4.3473	2287	79.7
23.1187	3.8441	2310	80.6
26.0988	3.4116	1450	50.6
26.9352	3.3075	1386	48.3

Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
10.1204	8.7333	1469	45.6
11.4266	7.7378	1619	50.3
15.1194	5.8552	2806	87.1
17.0841	5.1860	3148	97.7
17.9893	4.9270	3200	99.3
18.1334	4.8882	3221	100.0
20.0541	4.4241	2125	66.0
23.1480	3.8393	2604	80.8
26.3849	3.3752	1431	44.4
26.6492	3.3423	1385	43.0

Table B.19 X-ray diffractogram data of Khao Dawk Mali 105 wet-milled rice flour

Table B.20 X-ray diffractogram data of Ayutthaya 1 dry-milled rice flour

Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
15.1180	5.8557	2573	86.7
15.3676	5.7612	2574	86.7
17.2587	5.1339	2945	99.2
18.0445	4.9121	2969	100.0
19.9363	4.4500	2457	82.8
22.2044	4.0003	2165	72.9
22.7114	3.9122	2301	77.5
23.0338	3.8581	2329	78.4
23.2957	3.8153	2305	77.6
23.6408	3.7604	2196	74.0
26.5816	3.3507	1436	48.3

Angle 2-Theta $^{\circ}$	d value Angstrom	Intensity Count	Intensity (%)
10.0714	8.7757	1369	45.1
11.3068	7.8195	1533	50.5
15.2034	5.8230	2679	88.3
17.2229	5.1445	2967	97.8
17.9073	4.9494	2976	98.1
18.0784	4.9029	3035	100.0
19.8911	4.4600	2095	69.0
23.1671	3.8362	2576	84.9
23.3956	3.7993	2431	80.1
26.5703	3.3521	1380	45.5

Table B.21 X-ray diffractogram data of Ayutthaya 1 wet-milled rice flour

Quiltinger	Sw	elling power (	g / g of dry sar	mple)
Cultivars	60°C	70°C	80°C	90°C
RD41(D)	$6.0^{bc} \pm 0.1$	$6.7^{\circ} \pm 0.1$	7.3 <sup>cdef</sup> ± 0.2	$8.4^{j} \pm 0.0$
RD41(W)	$4.1^{f} \pm 0.0$	$6.1^{g} \pm 0.0$	$6.8^{f} \pm 0.1$	$9.3^{hi} \pm 0.0$
RD45(D)	$6.9^{a} \pm 0.4$	$7.5^{a} \pm 0.1$	$7.8^{bcd} \pm 0.2$	10.8 <sup>efg</sup> ± 0.1
RD45(W)	$4.3^{f} \pm 0.1$	$6.4^{de} \pm 0.1$	$7.7^{\text{bcd}} \pm 0.8$	$12.4^{b} \pm 0.3$
RD47(D)	5.9 <sup>°</sup> ± 0.1	$6.6^{cd} \pm 0.2$	$7.0^{ef} \pm 0.4$	8.5 <sup>j</sup> ± 0.1
RD47(W)	$4.7^{e} \pm 0.0$	$5.9^{h} \pm 0.1$	$6.8^{f} \pm 0.0$	$9.0^{i}$ ± 0.2
Shaw Lung 97 (D)	$5.9^{\circ} \pm 0.1$	$6.5^{cde} \pm 0.1$	$7.9^{bcd} \pm 0.1$	$11.4^{cd} \pm 0.4$
Shaw Lung 97 (W)	2.9 <sup>ij</sup> ± 0.1	$3.5^{k} \pm 0.0$	$7.8^{\text{bcd}} \pm 0.0$	11.3 <sup>cde</sup> ± 0.3
Prachin Buri 1 (D)	$6.8^{a} \pm 0.1$	$7.2^{b} \pm 0.1$	$7.9^{bc} \pm 0.3$	$9.4^{hi} \pm 0.2$
Prachin Buri 1 (W)	4.8 <sup>e</sup> ± 0.1	$6.2^{fg} \pm 0.1$	7.0 <sup>ef</sup> ± 0.1	$9.6^{h} \pm 0.2$
Prachin Buri 2 (D)	$5.6^{d} \pm 0.2$	$6.4^{ef} \pm 0.1$	$8.8^{a} \pm 0.3$	$12.4^{b} \pm 0.4$
Prachin Buri 2 (W)	$2.8^{j} \pm 0.0$	$4.0^{j} \pm 0.1$	$7.4^{\text{bcde}} \pm 0.2$	$11.6^{\circ} \pm 0.2$
Plai Ngahm Prachin Buri (D)	$5.5^{d} \pm 0.1$	$5.8^{h} \pm 0.1$	$7.9^{b} \pm 0.3$	10.7 <sup>fg</sup> ± 0.1
Plai Ngahm Prachin Buri (W)	$3.1^{hi} \pm 0.2$	$4.7^{i} \pm 0.2$	$7.5^{\text{bcde}} \pm 0.7$	10.9 <sup>efg</sup> ± 0.3
Khao Dawk Mali 105 (D)	$6.3^{b} \pm 0.2$	$7.3^{b} \pm 0.2$	$7.9^{bc} \pm 0.0$	11.1 <sup>def</sup> ± 0.2
Khao Dawk Mali 105 (W)	3.8 <sup>g</sup> ± 0.1	7.1 <sup>b</sup> ± 0.0	$8.6^{a} \pm 0.3$	$13.6^{a} \pm 0.6$
Ayutthaya 1 (D)	$5.5^{d} \pm 0.1$	$6.1^{g} \pm 0.2$	$7.6^{bcd} \pm 0.1$	10.8 <sup>efg</sup> ± 0.1
Ayutthaya 1 (W)	$3.2^{h} \pm 0.2$	$4.7^{i} \pm 0.1$	7.3 <sup>def</sup> ± 0.1	10.4 <sup>g</sup> ± 0.5

Table B.22 Swelling power properties of rice flours at different temperatures

Mean  $\pm$  standard deviation values in the same column followed by different superscripts are

significantly different (P  $\leq$  0.05)

(D) = flour from dry milling method, (W) = flour from wet milling method

	Solubility (%) at different temperature			ature
Cultivars	60°C	70°C	80°C	90°C
RD41(D)	$5.6^{d} \pm 0.1$	$7.1^{ef} \pm 0.5$	$10.0^{b} \pm 0.1$	15.2 <sup>de</sup> ± 0.2
RD41(W)	1.3 <sup>h</sup> ± 0.1	$3.2^{h} \pm 0.1$	4.7 <sup>ef</sup> ± 0.1	11.0 <sup>g</sup> ± 0.2
RD45(D)	$7.7^{b} \pm 0.2$	8.7 <sup>bc</sup> ± 0.2	$9.9^{b} \pm 0.1$	$14.4^{e} \pm 0.3$
RD45(W)	$1.1^{h} \pm 0.0$	$2.5^{h} \pm 0.1$	$3.3^{gh} \pm 0.3$	$7.0^{jk} \pm 0.3$
RD47(D)	5.7 <sup>d</sup> ± 0.1	8.0 <sup>cd</sup> ± 1.1	9.0 <sup>°</sup> ± 1.0	$16.7^{\circ} \pm 0.3$
RD47(W)	1.7 <sup>g</sup> ± 0.1	$3.1^{h} \pm 0.2$	5.0 <sup>de</sup> ± 0.1	11.1 <sup>g</sup> ±0.6
Shaw Lung 97 (D)	$7.6^{b} \pm 0.2$	$9.0^{ab} \pm 0.4$	$11.1^{a} \pm 0.2$	19.3 <sup>b</sup> ± 0.9
Shaw Lung 97 (W)	$0.4^{j} \pm 0.0$	$1.2^{i} \pm 0.1$	$4.8^{de} \pm 0.1$	$9.5^{h} \pm 0.2$
Prachin Buri 1 (D)	$4.8^{f} \pm 0.1$	$6.6^{fg} \pm 0.7$	$8.4^{\circ} \pm 0.4$	$12.6^{f} \pm 0.5$
Prachin Buri 1 (W)	$1.1^{h} \pm 0.1$	$2.8^{h} \pm 0.1$	$3.6^{gh} \pm 0.4$	$8.3^{i} \pm 0.4$
Prachin Buri 2 (D)	$6.5^{\circ} \pm 0.2$	7.5 <sup>de</sup> ± 0.4	10.3 <sup>ab</sup> ± 1.3	20.2 <sup>a</sup> ± 1.1
Prachin Buri 2 (W)	$0.5^{ij} \pm 0.0$	1.1 <sup>i</sup> ± 0.1	$5.6^{d} \pm 0.7$	11.3 <sup>g</sup> ± 0.1
Plai Ngahm Prachin Buri (D)	5.0 <sup>ef</sup> ± 0.5	$6.0^{g} \pm 0.3$	$8.4^{\circ} \pm 0.6$	14.8 <sup>de</sup> ± 0.2
Plai Ngahm Prachin Buri (W)	$0.7^{i} \pm 0.1$	1.6 <sup>i</sup> ± 0.1	$3.9^{fg} \pm 0.3$	$9.6^{h} \pm 0.3$
Khao Dawk Mali 105 (D)	$8.4^{a} \pm 0.3$	$9.6^{a} \pm 0.4$	$9.8^{b} \pm 0.4$	$15.6^{d} \pm 0.2$
Khao Dawk Mali 105 (W)	$1.9^{g} \pm 0.0$	$2.6^{h} \pm 0.2$	$3.5^{gh} \pm 0.3$	$6.4^{k} \pm 0.8$
Ayutthaya 1 (D)	5.3 <sup>e</sup> ± 0.2	8.1 <sup>cd</sup> ± 1.1	8.5 <sup>°</sup> ± 0.1	$14.6^{e} \pm 0.1$
Ayutthaya 1 (W)	$0.7^{ij} \pm 0.0$	$1.2^{i} \pm 0.1$	$2.9^{h} \pm 0.1$	$7.7^{ij} \pm 0.7$

Table B.23 Solubility properties of rice flours at different temperatures

Mean ± standard deviation values in the same column followed by different superscripts are

significantly different (P  $\leq$  0.05)

(D) = flour from dry milling method, (W) = flour from wet milling method

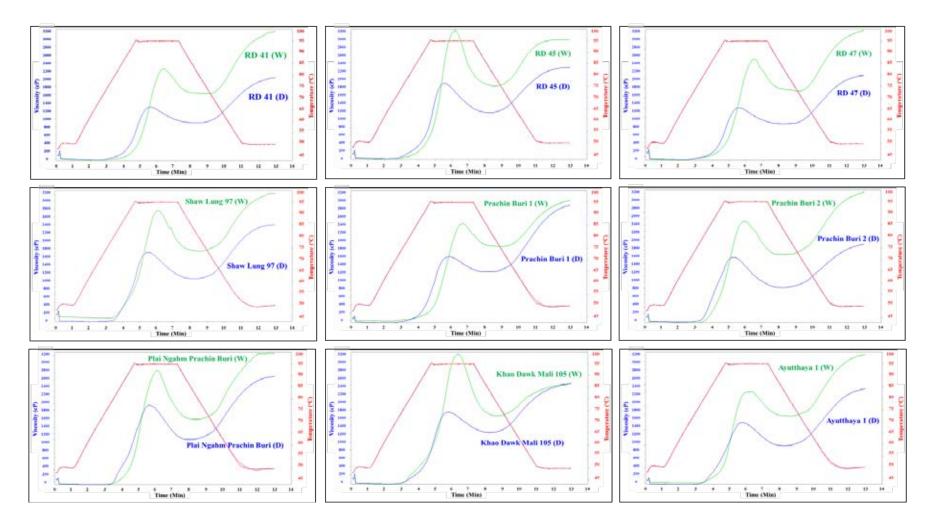


Figure B.37 Pasting properties of different rice flours cultivars (D = dry-milled flour and W = wet-milled flour)

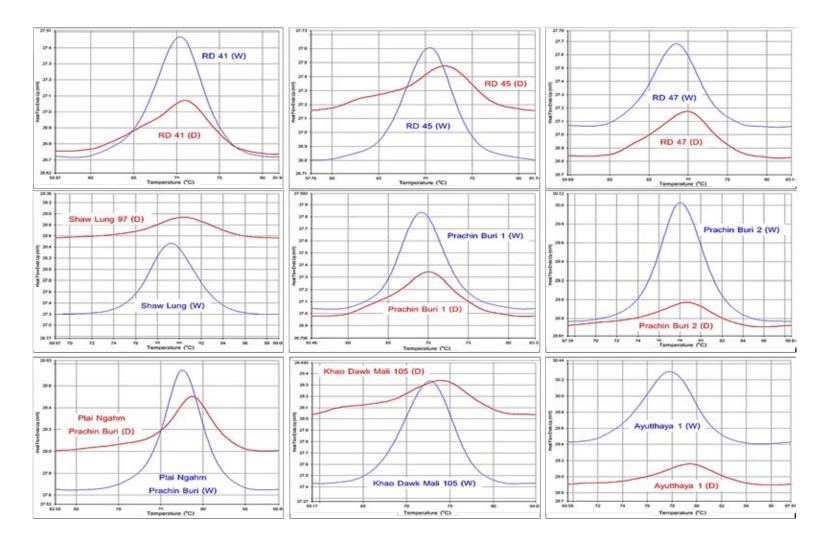


Figure B.38 DSC thermogram of flours from different rice cultivars (D = dry-milled flour and W = wet-milled flour)

## APPENDIX C

# Analysis of variance

Table C.1 Analysis of variance for proximate composition of rice (for Table 4.1)

F-test	SOV	df	MS
Proximate composition	Moisture	8	0.98*
	Error	18	0.00
	Lipid	8	0.66*
	Error	18	0.01
	Protein	8	5.74*
	Error	18	0.03
	Ash	8	0.16*
	Error	18	0.00
	Crude fiber	8	0.04*
	Error	18	0.00
	Carbohydrate	8	7.42*
	Error	18	0.05
	Amylose	8	148.83*
	Error	18	0.17

F-test	SOV	df	MS
Physical properties	1000-Kernel weight	8	25.05*
	Error	18	0.00
	Length-breadth ratio	8	0.27*
	Error	18	0.00
	Bulk density	8	0.001*
	Error	18	0.000

Table C.2 Analysis of variance for physical properties of rice (for Table 4.2)

\* Significantly different (P  $\leq$  0.05)

Table C.3 Analysis	of variance for	cooking quality	of rice (for Table 4.3)

F-test	SOV	df	MS
Cooking quality	Elongation ratio	8	0.01 <sup>ns</sup>
	Error	18	0.00
	Volume expansion ratio	8	0.48*
	Error	18	0.03
	Water uptake ratio	8	1.46*
	Error	18	0.04
	Gruel solid loss	8	2.91*
	Error	18	0.13
	Cook length-breadth ratio	8	0.22*
	Error	18	0.02

\* Significantly different (P  $\leq$  0.05), ns = not significant difference

F-test	SOV	df	MS
Color parameter	L*	8	29.91*
	Error	18	0.37
	a*	8	0.30*
	Error	18	0.00
	b*	8	0.001*
	Error	18	0.000

Table C.4 Analysis of variance for color parameter of rice (for Table B.1)

\* Significantly different ( $P \le 0.05$ )

 Table C.5 Analysis of variance for textural properties of cooked rice samples (for Table

4.4)

F-test	SOV	df	MS
Textural properties	Hardness	8	42430.23*
	Error	18	2462.09
	Adhesiveness	8	3581.81*
	Error	18	177.59
	Springiness	8	0.02*
	Error	18	0.00
	Cohesiveness	8	0.003*
	Error	18	0.000
	Gumminess	8	2749.04*
	Error	18	163.86
	Chewiness	8	1189.97*
	Error	18	78.03

F-test         SOV         df         MS           Proximate composition         Moisture         17         5.35*           Error         36         0.07           Lipid         17         0.24*           Error         36         0.00           Protein         17         3.60*           Error         36         0.05           Ash         17         0.21*           Error         36         0.00
Error360.07Lipid170.24*Error360.00Protein173.60*Error360.05Ash170.21*
Lipid       17       0.24*         Error       36       0.00         Protein       17       3.60*         Error       36       0.05         Ash       17       0.21*
Error       36       0.00         Protein       17       3.60*         Error       36       0.05         Ash       17       0.21*
Protein         17         3.60*           Error         36         0.05           Ash         17         0.21*
Error         36         0.05           Ash         17         0.21*
Ash 17 0.21*
Error 36 0.00
Carbohydrate 17 6.14*
Error 36 0.06

**Table C.6** Analysis of variance for proximate composition of dry-milled and wet-milledrice flour (for Table 4.5)

\* Significantly different (P  $\leq$  0.05)

**Table C.7** Analysis of variance for amylose content of dry-milled and wet-milled rice flour(for Table 4.6)

SOV	df	MS
Amylose content	17	152.07*
Error	36	0.17

F-test	SOV	df	MS
Particle size	Without Soaking	17	1892.87*
	Error	36	1.54
	Soaked in alcohol	17	860.73*
	Error	36	0.05

**Table C.8** Analysis of variance for average particle size of dry-milled and wet-milled riceflour (for Table 4.7)

\* Significantly different (P  $\leq$  0.05)

F-test	SOV	df	MS
Color parameter	L*	8	1.39*
	Error	18	0.06
	a*	8	0.03*
	Error	18	0.00
	b*	8	0.62*
	Error	18	0.01

Table C.10 Analysis of variance for color parameter of wet-milled rice flour (for TableB.3)

F-test	SOV	df	MS
Color parameter	L*	8	2.16*
	Error	18	0.09
	a*	8	0.03*
	Error	18	0.00
	b*	8	0.11*
	Error	18	0.00

\* Significantly different (P  $\leq$  0.05)

Table C.11 Analysis of variance for white index value of rice, dry-milled flour and wet-milled flour (for Table 4.8)

SOV	df	MS
White index value	26	486.39*
Error	54	0.17

\* Significantly different (P  $\leq$  0.05)

 Table C.12 Analysis of variance for crystallinity degree of dry-milled and wet-milled rice

 flour (for Table 4.9)

SOV	df	MS
Crystallinity degree	17	40.05*
Error	18	0.09

**Table C.13** Analysis of variance for water binding capacity of dry-milled and wet-milledrice flour (for Table 4.10)

SOV	df	MS
Water binding capacity	17	2.71*
Error	36	0.01

\* Significantly different ( $P \le 0.05$ )

**Table C.14** Analysis of variance for swelling power of dry-milled and wet-milled rice flour(for Table B.22)

F-test	SOV	df	MS
Swelling power	60°C	17	5.40*
	Error	36	0.02
	70°C	17	3.87*
	Error	36	0.01
	80°C	17	0.91*
	Error	36	0.10
	90°C	17	6.02*
	Error	36	0.08

F-test	SOV	df	MS
Solubility	60°C	17	24.96*
	Error	36	0.03
	70°C	17	28.71*
	Error	36	0.21
	80°C	17	25.29*
	Error	36	0.25
	90°C	17	50.25*
	Error	36	0.24

**Table C.15** Analysis of variance for solubility of dry-milled and wet-milled rice flour(for Table B.23)

F-test	SOV	df	MS
Pasting properties	Peak time	17	0.49*
	Error	36	0.00
	Pasting temperature	17	66.47*
	Error	36	0.41
	Peak viscosity	17	2160655.23*
	Error	36	8169.82
	Trough viscosity	17	1087149.49*
	Error	36	6543.44
	Final viscosity	17	3054232.45*
	Error	36	14416.02
	Breakdown	17	414457.39*
	Error	36	2117.98
	Setback	17	872022.82*
	Error	36	5338.87

**Table C.16** Analysis of variance for pasting properties of dry-milled and wet-milled riceflour (for Table 4.11 and 4.12)

F-test	SOV	df	MS
Thermal properties	Onset temperature	17	57.17*
	Error	18	0.03
	Peak temperature	17	37.30*
	Error	18	0.02
	Conclusion temperature	17	28.35*
	Error	18	0.03
	Gelatinization enthalpy	17	20.64*
	Error	18	0.06
	Conclusion – Onset	17	8.31*
	temperature	17	0.01
	Error	18	0.04

**Table C.17** Analysis of variance for thermal properties of dry-milled and wet-milled riceflour (for Table 4.13)

### BIOGRAPHY

Name	Miss Jitranut Leewatchararongjaroen
Date of birth	1 <sup>st</sup> January 1988
Institutions attended	Mahidol University, 2006-2009: Bachelor Degree of Science (Food Technology) with the Second Class Honors
	Chulalongkorn University, 2010-2011: Master Degree of Science (Food Technology)
Scholarship	Sri-Trung-Tong Scholarship, Faculty of Science, Mahidol University
	H.M. King Bhumibol Adulyadej's 72 <sup>nd</sup> Birthday Anniversary Scholarship, Graduate School, Chulalongkorn University
Home address	34 Soi Naknivas 21 Naknivas Road Ladprao 71 Bangkok 10230
	Tel. 086-829-3353
	Email: jitranut.lee@gmail.com

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