EFFECTS OF BROKEN RICE NOODLE FLOUR AND HEAT-MOISTURE TREATED TAPIOCA STARCH ON QUALITY OF DRIED NOODLE

Mr. Yafei Liu

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ผลของฟลาวร์จากเส้นก๋วยเตี๋ยวหักและสตาร์ชมันสำปะหลังดัดแปรด้วยวิธีความร้อนชื้นต่อคุณภาพของก๋วยเตี๋ยวอบแห้ง

Mr. Yafei Liu

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต

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By Mr. Yafei Liu

Field of Study Food Science and Technology

Thesis Advisor Associate Professor Saiwarun Chaiwanichsiri, Ph.D.

Thesis Co-advisor Associate Professor Kalaya Laohasongkram, 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
(Daris Kuakpetoon, Ph.D.)

……………………………………… Thesis Advisor
(Associate Professor Saiwarun Chaiwanichsiri, Ph.D.)

……………………………………… Thesis Co- advisor
(Associate Professor Kalaya Laohasongkram, Ph.D.)

……………………………………… Examiner
(Kanitha Tananuwong, Ph.D.)

……………………………………… External Examiner
(Professor Vanna Tulyathan, Ph.D.)

YAFEI LIU:

ผลของฟลาวร์จากเส้นก๋วยเตี๋ยวหักและสตาร์ชมันสำปะหลังดัดแปรด้วยวิธีความร้อนชื้นต่อคุณภาพของก๋วยเตี๋ยวอบแห้ง (EFFECTS OF BROKEN RICE NOODLE FLOUR AND HEAT-MOISTURE TREATED TAPIOCA STARCH ON QUALITY OF DRIED NOODLE) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: รศ.ดร.สายวรุฬ ชัยวานิชศิริ, อ.ที่ปรึกษาวิทยานิพนธ์ร่วม: รศ.ดร.กัลยา เลาหสงคราม, 114 หน้า.

วัตถุประสงค์ของงานวิจัยนี้คือการศึกษาสมบัติทางเคมีกายภาพของแป้งฟลาวร์จากเส้นก๋วยเตี๋ยวหักและสตาร์ชมันสำปะหลังดัดแปรด้วยความร้อนชื้น และศึกษาการใช้แป้งและสตาร์ชทั้งสองชนิดในการผลิตก๋วยเตี๋ยวเส้นเล็กอบแห้ง โดยแป้งฟลาวร์ของเส้นก๋วยเตี๋ยวหัก (BRNF) เตรียมจากการบดเส้นหักของก๋วยเตี๋ยวอบแห้ง สตาร์ชมันสำปะหลังดัดแปร(HMT-TS) เตรียมจากการดัดแปรสตาร์ชแป้งมันสำปะหลัง (TS) ที่ภาวะความชื้น (17, 20, และ 23%) และอุณหภูมิต่างๆ (95, 110 และ 125 °C) เป็นเวลานาน 16 ชั่วโมง ผลการทดลองพบว่า BRNF มีระดับแป้งทนย่อยสูงกว่า แต่สมบัติการพองตัว การเกิดเพส และลักษณะเนื้อสัมผัสของเจลต่ำกว่าแป้งฟลาวร์ข้าว (Rice flour, RF) นอกจากนี้ยังมีลักษณะผลึกและสมบัติการเกิดรีโทรเกรเดชันแตกต่างจาก RF การดัดแปรสตาร์ชมันสำปะหลังด้วยความร้อนชื้นมีผลต่อปริมาณอมิโลสปรากฏ ระดับของแป้งทนย่อย ลักษณะผลึก สมบัติทางความร้อน สมบัติการพองตัว สมบัติทางการเกิดเพสและเนื้อสัมผัสของเจล จากการผสมแป้งฟลาวร์และสตาร์ชดัดแปรกับ RF และสตาร์ช มันสำปะหลังที่ดัดแปรด้วยวิธีเคมีแบบเชื่อมข้าม (Cross-linked tapioca starch, CTS) ในสัดส่วนต่างๆ กันพบว่าแป้งผสม (RF/TS, RF/HMT-TS, RF/CTS, RF/CF-CTS) มีสมบัติการพองตัวที่สูงขึ้น แต่ไม่มีผลต่อสมบัติการเกิดเพสและลักษณะเจล การเพิ่มปริมาณ BRNF ลงในแป้งผสมให้ผลทางเชิงลบ โดยแป้งผสม RF/HMT-TS, RF/BRNF/HMT-TSไม่สามารถขึ้นรูปเป็นเส้นก๋วยเตี๋ยวได้ เนื่องจาก HMT-TS มีสมบัติการเกิดเพส และการเกิดเจลที่ต่ำ จากการเปรียบเทียบกับตัวอย่างทางการค้าพบว่าเส้นก๋วยเตี๋ยวที่เตรียมจากแป้งผสม RF/BRNF/TS มีคุณภาพการหุงสุก (cooking quality) และลักษณะเนื้อสัมผัสที่ใกล้เคียงกัน

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Broken rice noodles from noodle factory were milled into flour (BRNF) and combined with rice flour (RF), tapioca starch (TS) and its derivatives (heat-moisture treated tapioca starch, HMT-TS, and cross-linked tapioca starch, CTS) to make composite rice noodles. Heat-moisture treatment (HMT) was applied to TS having various moisture contents (17, 20 and 23%, wb) at different temperature levels (95, 110 and 125 °C) for 16 h. HMT displayed influence on the apparent amylose contents, resistant starch levels, X-ray diffraction characteristics, thermal behaviors, swelling power and solubility, pasting characteristics and gel textures of TS. BRNF displayed some distinct physicochemical properties from RF such as higher resistant starch level, unique X-ray diffraction characteristics, retrogradation behavior, suppressed swelling and pasting, and weaker gel texture. Although the partial substitution of BRNF for RF did not change the composition-property relationships of RF-starch blends, decrease in swelling, pasting and gelling of RF combinations were observed. Among RF combinations, those that contained HMT-TS (20-125) failed to be used for noodle making, which was due to the inferior pasting properties of HMT-TS (20-125) to those of TS and CTS. Composite rice noodles prepared from flour combinations containing BRNF showed cooking and textual qualities close to those of commercial one. And flour blend of RF/BRNF/TS was considered preferable to reusing broken rice noodles.

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INTRODUCTION

Rice noodles, preceded only by rice grains, are widely consumed in Asian countries. Produced mainly from rice flour, rice noodles could be divided into two types according to the processing methods: extruded rice vermicelli and slurry-sheeted flat rice noodles. Flat rice noodles can be prepared in either fresh or dried products. In contrast to fresh types, dried rice noodles with less amount of moisture (10-12%) could contribute to a longer shelf life. Occurrence of broken rice noodle in dried rice noodle making process is, however, inevitable due to improper handling and can be as high as 20% (data from local factories). Instead of being disposed as waste, broken rice noodles were milled into flour in this study and further used as raw material to study its effect on rice noodle making.

Due to the limitation of gel quality, fresh rice flour failed to be used for rice noodle making. Although ageing of rice grain could be applied to rice noodle making, its time-consuming disadvantage forced researchers to seek for more economic solutions for manufacturers. Rice flour and starch have been combined with native and modified starches to make noodles with comparable qualities. Furthermore, native and chemically modified tapioca starches have been used in noodle making including rice noodles. Hydrothermally modified tapioca starches, however, have rarely been applied to noodle making, especially rice noodle making. Heat-moisture treatment of starches has also been shown to increase resistant starch levels, which would provide physiological benefits to foods containing heat-moisture treated starches. In this study, native TS was heat-moisture treated under various conditions, and the impacts of HMT on some physicochemical properties of TS, as well as the suitability of HMT-TS for noodle making, were studied.

The objective of this research was, therefore, to study the reusability of broken rice noodles in the form of BRNF by combining with RF, TS and its derivatives (HMT-TS, CTS) to make composite rice noodles.

CHAPTER II

LITERATURE REVIEWS

2.1 Rice

Rice (*Oryza sativa*) production in the world is only second to wheat for food use (Bean, Elliston-Hoops, and Nishita, 1983), and about 90% of the world's rice is produced and consumed in Asia (Marshall and Wadsworth, 1993). As a staple food for most Asian countries, nearly 80-90% of the daily caloric intake for people in these areas has come from the consumption of rice (Luh, 1999). Globally, among thousands of rice varieties, those consumed as diet account for 21, 14 and 2% of energy, protein and fat supply, respectively (Kennedy and Burlingame, 2003). The diet preference for rice types differs among countries such as the preferred Indica rice for Indians, and Japonica and waxy rices for Japanese (Singh et al., 2000). Preceded only to milled rice, rice products in the forms of rice noodles, puffed rice, breakfast cereals, rice cakes, fermented sweet rice, and snack foods are largely consumed in Asian countries (Luh, 1999).

Study on quality profiles of 177 rice samples has classified rices into eight varieties based on amylose contents and viscogram patterns (Bhattacharya, Sowbhagya, and Indudhara Swamy, 1982). Rices distinguished by amylose contents are generally divided into two basic types: common/non-waxy and waxy rices; common rice could be further divided into low (9 - 20%), medium (20 - 25%) or high (> 25%) amylose rices, and waxy rice is almost amylose free (0 - 2%) (Mitchell, 2009).

2.1.1 Rice milling

The principal parts of the rice grain are the hull, pericarp, seed coat, nucellus, embryo, aleurone layer, and endosperm (Marshall and Wadsworth, 1993). Rice grain with the intact hull is called rough rice, and brown rice is produced by removing the hull; while, partial or entire removal of the bran layer gives partially polished rice or white rice (Mitchell, 2009). Chemical compositions of protein, fat, fiber, ash and starch in each part of the rice grain structure are listed in Table 2.1.

Table 2.1 Range of proximate composition of rough rice, brown rice, milled rice, rice hulls, rice bran, rice embryo, and rice polish (% dry basis).

Constituent	Rough	Brown	Milled	Hulls	Bran	Embryo	Polish
Protein (N × 5.95)	6.7–8.3	8.3–9.6	7.3–8.3	2.3–3.2	13.2–17.3	17.7–23.9	13.0–14.4
Crude fat	2.1–2.7	2.1–3.3	0.4–0.6	0.4–0.7	17.0–22.9	19.3–23.8	11.7–14.4
Crude fiber	8.4–12.1	0.7–1.2	0.3–0.6	40.1–53.4	9.5–13.2	2.8–4.1	2.7–3.7
Crude ash	3.4–6.0	1.2–1.8	0.4–0.9	15.3–24.4	9.2–11.5	6.8–10.1	6.1–8.5
Starch	62.1	77.2	90.2	1.8	16.1	2.4	48.3–55.4
Dietary fiber	19.1	4.5	2.7	77.3	27.6–33.3		

Source: Marshall and Wadsworth (1993)

The outer layer of brown rice consists of bran and polish, known as rice bran, and the two parts are not separated under commercial milling to produce white rice (Marshall and Wadsworth, 1993). The residual milled white rice is further divided into several fractions based on the size: head rice and broken rice. Typical mill yields and their compositions are listed in Table 2.2 (Mitchell, 2009).

Rice flour could be prepared from polished or broken rices by dry-, wet- and semi-dry-milling processes which mainly differ in the use of water to soak and grind rice kernels (Chiang and Yeh, 2002). Dry-milling of rice kernels was usually conducted by commercial mills or grinders such as hammer, turbo and roller mills to get ground residuals which were screened through specific mesh sieves to get rice flour (Nishita and Bean, 1982). Different from dry-milling process, wet-milled rice flour required water-soaking of rice kernels and grinding with excess water to obtain slurry; the slurry was then centrifuged to get rice cake which was dried and milled prior to sieving.

Table 2.2 Typical mill yields and composition of products and by-products from rough rice.

Fraction	Yield from rough rice (%)	Protein, N × 5.95 (%)	Crude fat (%)	Crude fiber (% d.b.)	Crude ash (%)	Nitrogen-free extract (%)	Free sugars (%)
Hull	18–28	2–4	0.4–0.8	48–53	15–20	26–34	
Brown rice	72–82	7–15	2–4	1 1–12	79–90	1.3–1.5	
Bran	4–5	12–17	15–22	9–16	9–16	40–49	6.4–6.5
Polish	3	13–16	9–15	2–5	5–9	54–71	
Milled rice	64–74	6–13	0.3–0.6	0.1–0.6	0.3–0.7	84–93	0.2–0.5
Head	56						
Seconds	9						
Brewers	3						

Source: Mitchell (2009)

Effects of rice milling on food applications of corresponding flours have been studied (Nishita and Bean, 1982; Jomduang and Mohamed, 1994; Hemavathy and Bhat, 1994; Suksomboon and Naivikul, 2006a). In contrast to dry-milled flour, wet-milled rice flour was believed to be superior for making baked products and Thai rice-based snack food; while semi-dry-milled rice flour showed inferior pasting properties to those of wet-and dry-milled ones and had highest degree of gelatinization (Chen, Lu, and Lii, 1999). Hemavathy and Bhat (1994) studied the textural qualities of rice vermicelli prepared from rice flours with various average particle sizes and found that rice vermicelli produced from middle particle size fraction (138 and 165 μm) of rice flour had better textural profile. Rice flour with smallest particle size distribution (100-150 μm) demonstrated resistance to the heat-induced disintegration rendering it suitable for rice noodle making (Chen et al., 1999). Effects of starch granule size fractions on the processibility and qualities of potato and sweet potato noodles have also been reported by Chen, Schols, and Voragen (2003), who found that both dried and cooked starch noodles made from small-size granule fractions were significantly better than those made from their initial starch preparations and much better than those made from the large-size granule fractions.

2.1.2 Rice flour/starch

Rice flour/starch is preferably prepared from broken rices for economic reasons (Mitchell, 2009). And as a specialty by-product, rice flour has been used as a dusting flour or anticaking agent for refrigerated biscuit doughs, or as an ingredient in starchy foods such as in baby foods (Nishita and Bean, 1982; Bean et al., 1983).

Proximate composition differed in the milling fractions of brown rice, which was attributed to the loss of nutrients, especially the substantial removal of minerals and vitamins during the preparation of well-milled white rice (Marshall and Wadsworth, 1993). As seen from Table 2.1, composition of starch in milled rice reached to a content of 90% followed by protein (8.3-9.6%) as the second main constituent; moreover, fat, fiber, ash and minerals have also been reported as the ingredients found in some rice varieties (Kennedy and Burlingame, 2003). As the most common nutrient analyzed in rice, protein content in rice varied significantly by varieties, ranging from 4.5-15.9% as the greatest overall variation for Asian rice varieties (Kennedy and Burlingame, 2003). Rices for daily consumption or common eating generally contained 7% protein, and did not fluctuate widely from this level (Hamaker and Griffin, 1990). Nevertheless, rice starch was still counted up to 90% in rice flour, which made rice starch the decisive factor in the functionality of rice flour (Suksomboon and Naivikul, 2006). Detailed study on the amylose content, chain length distribution of amylopectin debranching, and gelatinization behavior of normal and waxy rice starch has been reported (Vandeputte et al., 2003) and these characteristics are listed in Table 2.3 and Table 2.4.

Some superior properties of rice starch to those of other starches, including hypoallergenicity, digestibility, consumer acceptance, bland flavor, small granules (2-10 μm), white color, greater freeze-thaw stability of pastes, greater acid resistance and a wide range of amylaoe:amylopectin ratios, has rendered rice starch preferably used in balanced foods, infant and geriatric foods, etc.(Mitchell, 2009). Food uses of rice starch, in terms of rice starch based food production or property altering ingredient in non-starch foods, have been shown great dependence on rice starch composition, gelatinization and pasting properties, enzyme susceptibility, crystallinity, swelling and solubility, which in turn could be all affected by its granule size (Lindeboom, Chang, and Tyler, 2004).

Method widely used to extract rice starch was to solubilize rice protein by alkali, in which glutelin (approximately 80% of the protein in rice) was extracted out to obtain a starch containing < 1% protein (Mitchell, 2009). Protein content in rice starch played a significant role in determining the pasting properties, evidenced by the negative correlation between peak viscosity (PV) and protein content (Hamaker and Griffin, 1990; Lim et al., 1999).

Table 2.3 Amylose contents and relative chain length distribution of amylopectin of waxy and normal rice starches

Sample	Amylose			Amylopectin chain					
	AAM (%)	FAM (%)	LAM (%)	F1	F2	F3	F4	F5	F6
Waxy rice starches of low Tp									
Thai glutinous I	2.8	2.4	0.4	13.0	13.5	47.5	3.5	6.0	16.5
Thai glutinous II	2.9	2.6	0.3	14.0	13.0	45.0	3.5	6.5	18.0
Sanpathong	2.9	2.8	0.1	15.0	13.5	47.0	3.5	6.0	15.0
Black rice	3.4	2.9	0.5	13.0	12.0	44.5	3.5	7.0	20.0
IR65	1.9	1.9	0.0	14.5	13.0	45.5	3.5	6.0	17.5
Normal rice starches of low Tp									
IR42	28.2	27.5	0.6	12.0	11.5	44.5	3.5	7.5	21.0
IR48	23.0	22.4	0.5	12.0	11.5	44.0	3.5	7.5	21.5
IR24	17.0	15.5	1.4	11.0	11.0	45.5	3.5	7.5	21.5
Normal rice starches of intermediate Tp									
Thaibonnet	27.8	27.1	0.6	8.0	12.0	52.0	3.5	6.3	18.0
Puntal	26.3	23.3	3.0	9.0	12.0	51.5	3.5	6.0	18.0
IR5	29.3	28.1	1.2	9.0	12.0	50.5	3.5	6.5	18.5
PSBRc18	22.2	20.6	1.5	9.5	12.0	49.0	3.5	7.0	19.0
Normal rice starches of high Tp									
Pelde	18.6	15.9	2.7	8.0	12.0	52.5	3.5	6.0	17.5
Century Patna 231	15.6	13.0	2.6	8.0	11.5	51.5	3.5	6.5	19.0
IR2071	13.6	10.6	3.0	8.0	12.5	54.0	3.5	5.5	16.5

AAM, absolute amylose; FAM, free amylose; LAM, lipid complexed amylose; Tp, gelatinization peak temperature; F, amylopectin chain fraction; F1 with degree of polymerization 6-9 (DP, 6-9), F2 (DP 10-11), F3 (DP 12-22), F4 (DP 23-24), F5 (DP 25-32) and F6 (DP > 32).

Source: Vandeputte et al. (2003)

Table 2.4 Gelatinization behaviors of waxy and normal rice starches

Sample	Gelatinization behavior						
	To (°C)	Tp (°C)	Tc (°C)	Tc – To (°C)	ΔH (J/g)	ΔH_{AP} (J/g)	ΔH_{LAM} (J/g)
Waxy rice starches of low Tp							
Thai glutinous I	57.9	65.2	75.5	17.7	18.6	19.1	–
Thai glutinous II	58.6	65.3	75.8	17.2	19.6	20.2	–
Sanpathong	58.7	65.5	75.5	16.8	18.4	18.9	–
Black rice	58.9	65.8	75.7	16.8	19.1	19.8	–
IR65	59.6	65.5	75.8	16.3	18.8	19.2	–
Normal rice starches of low Tp							
IR42	58.3	62.8	71.0	12.8	17.3	24.1	2.0
IR48	56.6	63.2	75.7	19.2	19.8	25.7	2.3
IR24	62.1	67.0	74.9	12.8	17.8	21.4	2.1
Normal rice starches of intermediate Tp							
Thaibonnet	66.4	71.7	79.0	12.6	16.8	23.3	1.7
Puntal	67.6	72.1	79.2	11.6	17.6	23.9	2.0
IR5	69.5	73.0	78.7	9.2	16.9	23.9	1.0
PSBRc18	69.0	73.5	80.6	11.6	17.9	23	2.1
Normal rice starches of high Tp							
Pelde	73.0	76.7	82.3	9.3	18.9	23.2	1.7
Century Patna 231	74.8	77.8	83.3	8.5	18.8	22.3	1.7
IR2071	75.6	78.5	83.3	7.7	18.7	21.7	1.4

To, Tp, Tc, Tc – To, ΔH, ΔH_{AP}, ΔH_{LAM} represents gelatinization onset, peak, conclusion temperatures, gelatinization ranges, gelatinization enthalpies, gelatinization enthalpies expressed on amylopectin content, and enthalpies of the melting of amylose–lipid complexes respectively.

Source: Vandeputte et al. (2003)

2.2 Tapioca starch

Obtained from the roots of cassava plants, tapioca starch is differentiated from other commercial starches by its low levels of residual materials (fat, protein, ash), high molecular weights of amylose and amylopectin, and lower amylose content than other amylose-containing starches (Breuninger, Piyachomkwan, and Sriroth, 2009). Amylose content of tapioca starch, as compared with some cereal starches, has been shown less variation ranging from 17-24%, and other significant features found for tapioca starch was its low contents of lipid (0.1%), protein/nitrogen (around 0.01%), and phosphorous (0.001-0.008%) (Swinkels, 1985; Defloor, Dehing, and Delcour, 1998; Lim, Kasemsuwan, and Jane, 1994). Approximate composition, characteristics of amylose, and amylopectin chain length distribution of tapioca starch as compared with other starches are presented in Table 2.5, 2.6, and 2.7.

Table 2.5 Tapioca starch in comparison to some other commercial starches

Source	Granule diameter (μm)	Average diameter (μm)	Amylose content (%)	Amylose average DP	Phosphorus (%)	Ash (%)	Protein (%)	Lipid (%)
Tapioca	4–35	15	17	3000	0.01	0.2	0.1	0.1
Potato	5–100	27	21	3000	0.08	0.4	0.06	0.05
Normal corn	2–30	10	28	800	0.02	0.1	0.35	0.7
Waxy corn	2–30	10	0	not available	0.01	0.1	0.25	0.15
Wheat	1–45	bimodal	28	800	0.06	0.2	0.4	0.8

DP, degree of polymerization

Source: Breuninger et al. (2009)

Table 2.6 Properties of amylose from different botanical sources

Property	Corn	Potato	Rice	Tapioca	Wheat
Iodine binding capacity (g/100 g)	20	20.5	20.0–21.1	20	19.9
Degree of polymerization by weight, DP w (range)	390–13100	840–21800	210–12900	580–22400	not available
Degree of polymerization by weight, DP w (mean)	2550	6360	2750–3320	6680	not available
Degree of polymerization by number, DP n (mean)	960	4920	920–1110	2660	1290
DP w /DP n	2.66	1.29	2.64–3.39	2.51	not available
Chain length, CL	305	670	250–370	340	270
Chain number	3.1	7.3	2.5–4.3	7.8	4.8
Unbranched amylose (mol%)	52		69	58	73
Phosphorus (μg/g)	not available	3	None	7	not available

Source: Breuninger et al. (2009)

Table 2.7 Chain length distribution of components from amylopectin debranching

Starch types		Chain					
		A1	A2	B1	B2	B3	B4
Wheat	Number-average DP	10	15	24	49	84	110
	Weight-average DP	11	15	25	51	84	111
	Weight%	18.5	21.4	35.6	20.9	2.3	0.3
	Molar%	35.4	27.4	28.3	8.2	0.6	0.1
Waxy rice	Number-average DP	10	15	24	44	69	86
	Weight-average DP	11	16	24	46	69	87
	Weight%	20.2	26.2	27.2	21.9	3.9	0.5
	Molar%	36.9	32	20.7	9.1	1.1	0.1
Potato	Number-average DP	10	14	24	44	69	86
	Weight-average DP	10	14	24	46	69	87
	Weight%	6.2	18.5	33.7	29.5	11.6	0.5
	Molar%	15	31.8	35.2	14.5	3.4	0.1
Cassava	Number-average DP	10	13	21	48	74	90
	Weight-average DP	10	13	22	49	74	91
	Weight%	9.4	17	45.6	24.7	2.7	0.6
	Molar%	18.9	26.3	43.6	10.4	0.7	0.1

DP, degree of polymerization

Source: Breuninger et al. (2009)

Long-term and wide use of tapioca starch in food manufacturing, especially baby food, has been attributed to the physical properties of its paste (clarity and stability) and bland flavor contribution. Another food application of tapioca starch reliant upon its flavor absence and fast meltaway (due to the low retrogradation tendency) is in pudding products (Breuninger et al., 2009). Furthermore, tapioca starch in either native or modified (cross-linked) forms has been used in Asian-style noodle making such as extruded vermicelli (Kasemsuwan, Bailey, and Jane, 1998; Charutigon et al., 2008), and Chinese noodles (Charles et al., 2007).

2.3 Physicochemical properties of starch

2.3.1 Starch swelling and pasting

Food applications of starch usually involve the heating of starch granules in the presence of water. Several phenomena accompanied the heating of starch aqueous suspension were observed from the loss of birefringence, swelling of starch granules and partial solubilization of starch (Subramanian, Hoseney, and Bramel-Cox, 1994). When starch granules were heated in excess water, water molecules would penetrate into the amorphous regions to initiate starch swelling, which was, however, restricted as the failure of water penetration into the crystalline regions (Donovan, Lorenz, and Kulp, 1983). Starch swelling has been proven to be mainly a property of amylopectin, while amylose and lipids played a role in suppressing starch swelling (Tester and Morrison, 1990a). Negative correlation was found between the amylose content and wheat starch swelling; whereas, the lipid content did not negatively correlate with starch swelling (Sasaki and Matsuki, 1998). Measurements of the swelling power, also called as water-absorption index (Anderson, 1982; Ruales, Valencia, and Nair, 1993) or water-binding capacity (Medcalf and Gillies, 1965), of starches have been designated as percentage of volume increase (swollen volume/initial volume of dry sample) (Tester and Morrison, 1990a; Hoover and Manuel, 1996b), percentage of weight increase (weight of the wet sediment/initial weight of dry sample) (Takashi and Seib, 1988; Subramanian et al., 1994; Eerlingen et al., 1997), and others (Collado and Corke, 1999; Holm, Lundquist, and Björck, 1988). Correlation between swelling power and heating temperature has been studied by incubating starches-water suspensions at different temperatures for various periods of time. By conducting this Tester and Morrison (1990a) found that swelling factor increased with the water/starch ratio ranging from 10:1 to 20:1, but leveled off with the water/starch ratio higher than 25:1. As the progressive increase in the incubation temperature such as 30-85 °C (Tester and Morrison, 1990a) and 60-95 °C (Hoover and Manuel, 1996b), increase in swelling power was noticed, and the different magnitudes of increase among different starches have been attributed to the amylose/amylopectin ratio and amount of lipid-complexed amylose chains. Moreover, at specific heating temperature, when various incubation times were employed to starch, swelling exhibited several phases: an initial slight swelling followed by a rapid swelling and a final maximum swelling (Tester and Morrison, 1990a).

As starch swelling proceeded the hydrogen bonds that held the integrity of starch granules were disrupted by water molecules (Hoover, 2001), which caused leaching of polysaccharide (amylose and/or amylopectin, depending on the starch) (Tester and Morrison, 1990a), and further increase in granule swelling. Starch solubilization by leaching during starch swelling, or gelatinization, has largely been expressed as the measurement of solubilized starch content and amylose leaching (Anderson, 1982; Takashi and Seib, 1988;

Holm et al., 1988; Eerlingen et al., 1997). On the water-insoluble nature of amylose-lipid complex (Morrison, 1988), the complexing of amylose and starch lipids during granule swelling could prevent partial amylose leaching leading to decrease in the solubilization of water soluble carbohydrates (Swinkels, 1985; Tester and Morrison, 1990a; Bhatnagar and Hanna, 1994). As an indication of the degree of starch chain interactions, starch swelling power and solubility would be affected by factors that influenced these chain interactions such as amylose/amylopectin ratio, characteristics of amylose and amylopectin (Hoover, 2001), as well as the aforementioned amylose-lipid complexing.

When the temperature applied to heating of starch dispersions increased, swelling of starch granule caused the thickening of the suspension. And further increase in heating temperature would disintegrate starch granules, especially in the presence of shear force, resulting in a continuous phase of solubilized polysaccharides (amylose/amylopectin) filling an interpenetrating discontinuous phase of swollen starch granules/granule remnants, granule ghosts and fragments. And starch was pasted as a highly viscous paste was achieved due to the disruption of most starch granules under high temperature shearing (Eliasson and Gudmundsson, 2006). Accompanied starch pasting was the changes in viscosity of starch suspensions, which could be measured by two apparatuses: Brabender Viscoamylograph (BVA), and Rapid Visco-Analyzer (RVA). Parameters used to characterize starch pasting behavior were usually the pasting temperature (P_T), peak viscosity (PV), hot paste viscosity (HPV), cold paste viscosity (CPV), breakdown (BD) and setback (SB). By comparison between these two instruments for viscosity detection, RVA has been proven to be an alternative for measuring starch pasting properties (Deffenbaugh and Walker, 1989). Apart from the factors influencing starch swelling, pasting of starch-water suspension under constant shear force was also affected by the initial temperature, the heating rate, and the final holding time (Batey and Curtin, 2000).

2.3.2 Starch gelatinization

When the dispersion of starch granules and water was heated, water absorption capability of starch increased as granule swelling occurred, which, however, was hindered at low heating temperature by the ending of amorphous region in more ordered crystalline domains (Donovan et al., 1983). Increase in heating temperature until a level (onset of gelatinization), the water uptake of granules was reversible, but further increase in heating temperature approaching the onset of gelatinization would cause the disruption or melting of crystallites due to thermal motion and swelling force, and the water uptake became irreversible (Eliasson and Gudmundsson, 2006). Changes in starch granule throughout gelatinization involves loss of birefringence, loss of crystallinity, morphological changes, granule swelling, solutes leaching (Eliasson and Gudmundsson, 2006), and alterations in rheology (Rao et al., 1997), and susceptibility toward enzymatic hydrolysis/digestibility (Holm et al., 1988; Escarpa et al., 1996) etc. Differential scanning calorimetry (DSC) has become the most widely used tool to study the gelatinization phenomenon of starches on account of the intended melting transition parameters provided by it: onset (To), peak (Tp), conclusion (Tc) temperatures of gelatinization, gelatinization temperature range (ΔT_G), and gelatinization enthalpy (ΔH_G).

Factors affecting starch gelatinization by DSC included starch granule structure organization, heating rate, moisture content, mechanical damage, thermal history of the sample, extraction conditions of starch, etc. (Biliaderis, 2009). Starches from various botanical sources would exhibit different gelatinization characteristics even under same testing conditions (Singh et al., 2003). Starch crystallinity has displayed close correlation with gelatinization parameters (gelatinization temperatures, Tm, ΔT_G and ΔH_G) (Krueger et al., 1987; Barichello et al., 1990). Since starch crystallinity is associated with amylopectin, a higher degree of crystallinity was expected from maize starches with higher amylopectin content and this was evidenced by a higher ΔH_G and narrower ΔT_G (Krueger et al., 1987). Vandeputte et al. (2003) found that gelatinization temperatures positively correlated with relatively crystallinity of starches from normal rices. Apart from starch crystallinity, molecular structure of amylopectin, starch composition, and granule architecture have also shown effects on starch gelatinization. Furthermore, comparison between A- and B-type starches demonstrated that the former had a higher ΔH_G; while the latter displayed a lower To but higher Tp and Tc (Singh et al., 2003).

Biphasic endothermic transitions during starch gelatinization have been observed from both single starch melting in the presence of limited water content (Biliaderis, Maurice, and Vose, 1980) and mixed starches from same [rice (Hagenimana, Pu, and Ding, 2005)] and different [rice and wheat (Liu and Lelièvre, 1992), potato, waxy maize and barley (Ortega-Ojeda and Eliasson, 2001)] botanical sources melting under excess water content. The overlapped two melting endotherms occurred at low moisture content was due to the limited extent of granule swelling and melting of crystallites as water became a limiting factor (Donovan, 1979); whereas, in the case of mixed starches, granule melting position in the DSC trace was governed by the distinct granular stabilities of mixing counterparts resulting in independent gelatinization (Liu and Lelièvre, 1992). The extent of overlapping or superimposing of these two melting endotherms was, however, dependent on the similarities of the gelatinization temperatures of each starch (Ortega-Ojeda and Eliasson, 2001).

In contrast to only traces of lipids present in legume and tuber starch, lipids contained in non-waxy cereal starches have been shown roughly proportional to their amylose contents, and the existence of lipids (both surface and internal lipids) might be in free state or in the form of inclusion complex with amylose (Morrison, 1988), or linked to hydroxyl groups of starch components by ionic or hydrogen bonding (Vasanthan and Hoover, 1992). Naturally existing amylose-lipid complexes (commonly found in starch genotypes with amylose content at least 30%) could diffract X-rays to display a V pattern; however, such V structure could also be formed in granular, amylose-bearing starches between their amyloses and naturally occurring lipids or added complexing ligands under selected heat/moisture treatments (Zobel, 1988). Formation of amylose-lipid complexes in non-waxy starches by heat/moisture treatment has been confirmed through starch gelatinization by DSC manifested by the melting transitions at temperatures above 95 °C (Biliaderis et al., 1986). In the presence of excess lipids, disorganized or melted amylose-lipid complexes could re-complex during cooling observed from the exothermic transitions suggesting a thermally reversible behavior (Kugimiya, Donovan, and Wong, 1980; Eliasson, 1985). Reoccurrence of the melting transition (> 95 °C) of amylose-lipid complexes in cooled gelatinized starches by DSC was indicative of the thermal reversibility of these inclusion complexes (Biliaderis et al., 1986).

X-ray crystallography has been employed to study the crystallinity change of granular starches during gelatinization; original X-ray pattern disappeared due to the melting of crystallites under heating (Zobel, Young, and Rocca, 1988), and a V pattern was observed for fresh bread or fresh gelatinized starches, which was due to the diffraction of X-ray by amylose-lipid complex (Dragsdorf and Varriano-Marston, 1980; Miyoshi, 2002; Zobel, 1988).

2.3.3 Starch retrogradation

On account of the non-thermodynamic equilibrium state of starch paste obtained from starch gelatinization, structural transition of gelatinized starch from an amorphous state to a more ordered or crystalline state would occur upon cooling and storage of gelatinized starch, and this phenomenon was collectively termed retrogradation (Gudmundsson, 1994; Eerlingen and Delcour, 1995; Fredriksson et al., 1998).

Amylose and amylopectin differed in their retrogradation behaviors; retrogradation of amylose proceeded rapidly with the melting enthalpy of recrystallized amylose approaching toward maximum value within short time; the extent of the retrogradation of amylopectin, however, increased with storage along with an increment in the melting enthalpy of recrystallized amylopectin (Miles et al., 1985; Gudmundsson, 1994). Furthermore, when melting transition and thermal reversibility were taken into consideration, recrystallized amylose was disordered by heating at temperature above 100 °C and shown to be thermally reversible, but melting of recrystallized amylopectin occurred at lower temperature (< 100 °C) and thermal reversibility was noticed (Miles et al., 1985; Orford et al., 1987). Transitional temperatures and melting enthalpies of retrograded starches (< 100 °C, disintegration of recrystallized amylopecins) showed to be lower than those of native starch gelatinization (White, Abbas, and Johnson, 1989; Shi and Seib, 1992; Jane et al., 1999), which has been ascribed to the weaker crystallinity of retrograded starches (Sasaki, Yasui, and Matsuki, 2000) resulting from the less ordered manner in amylopectin chain associations (Singh et al., 2003). Cui and Oates (1997) observed that prolonged storage (under the same low temperature) only increased the melting enthalpies of recrystallized amylpectins, but preferable retrogradation conditions (low temperature for nucleation followed by high temperature for propagation) would facilitate the recrystallization of amylopectin resulting in perfect crystallites and increase in melting transition temperatures.

Chain structure of amylopectin played a significant role in amylopectin recrystallization (Ring et al., 1987; Kalichevsky, Orford, and Ring, 1990; Orford et al., 1987); reassociation of amylopectin chains during retrogradation depended on the crystallization of the outermost short branch chains with a degree of polymerization (DP) of 15. Study on the retrogradation behavior of waxy cereal starches showed that amylopectin retrogradation was directly proportional with mole fraction of unit chains with DP 14-24; chains of DP 6-9, however, would hinder starch retrogradation (Shi and Seib, 1992). Study on the retrogradation of amylopectins isolated from corn and wheat starches demonstrated that higher proportion of chains of DP 15-20 of corn amylopectin facilitated its recrystallization process resulting in higher retrograded crystallinity, while wheat amylopectin of greater proportions of short chains (DP 9-13) retarded its retrogradation (Ward, Hoseney, and Seib, 1994).

When starch retrogradation enthalpy was compared among A-, B- and C-type starches, cereal starches exhibited the lowest value, which was attributed to the weight-average DP of amylopectins (Fredriksson et al., 1998). Jane et al. (1999) determined the retrogradation rate as percentage of retrogradation ($\Delta H_{retrogradation}/\Delta H_{gelatinization}$) of A-, B- and C-type starches and found that cereal starches generally retrograded more rapidly (e.g.

40.5% of normal rice) than tuber and root starches (e.g. 25.3% of tapioca). These authors attributed this to the promotion effect of lipids present in cereal starches and inhibition impact due to phosphate monoesters in tuber and root starches.

A small fraction in retrograded starches that has demonstrated resistance to the hydrolysis by amylolytic enzymes, along with the melting transition similar to that of recrystallized amylose, was corporately recognized as resistant starch type III (RS 3) that mainly comprised retrograded amylose (Siljeström, Eliasson, and Björck, 1989; Sievert and Pomeranz 1989, 1990; Czuchajowska, Sievert, and Pomeranz, 1991; Eerlingen, Crombez, and Delcour, 1993). Czuchajowska et al. (1991) also reported that during starch retrogradation, the presence of lipids would reduce the yield of RS (recrystallized amylose) by complexation with amylose. And this was based on a competitive mechanism between amylose recrystallization and amylose-lipid complexing that crystallization of amylose and lipids preceded amylose reassociation (Czuchajowska et al., 1991; Biliaderis, 2009). Although reduction in enzymatic susceptibility of amylose by complexing with lipids was observed, amylose-lipid complex was till enzymatically degradable (Holm et al., 1983).

The aforementioned recrystallization process of starch retrogradation also involved changes in X-ray crystallography. Dragsdorf and Varriano-Marston (1980) observed a V-hydrate (V_h) pattern for the starch from fresh bread which, after one-day aging, exhibited a mixed V_h and B structure, however a seven-day staling of bread did not alter the mixed X-ray pattern but increased the degree of crystallinity. Starch gels from maize and potato both gave a V pattern immediately after gelatinization (0 h); while the developing recrystallization rendered a gradual recovery of B pattern (Miyoshi, 2002). Thus, starch retrogradation process regarding to changes in X-ray pattern was expressed as a gradual decrease in V structure and an increase in B structure (Zobel et al., 1988).

2.4 Heat-moisture treatment

Heat-moisture treatment has been defined as a physical treatment that involves incubation of starch granules at low moisture levels (<35% moisture, w/w), during a certain period of time (15min-16h), at a temperature (84-120 °C) above the glass transition temperature but below the gelatinization temperature (Jacobs and Delcour, 1998; Gunaratne and Hoover, 2002).

Researchers found that starches from different botanical origin such as cereal starches (e.g. wheat, maize and oat), tuber starches (e.g. potato and yam), root starches (e.g. cassava, arrowroot) and legume starches (e.g. lentil, pea) reacted differently to moist heat treatment (Hoover and Vasanthan, 1994; Hoover and Manuel, 1996ab; Jacobs and Delcour, 1998; Gunaratne and Hoover, 2002; Mason, 2009). Hoover (2010) summarized the impacts of HMT on physicochemical properties of different starches under various HMT conditions (Table 2.8) and showed that HMT applied to starches varied less in heating temperature and time than in the moisture content. Temperatures of HMT used in most studies ranged from 90-135 °C with few ones lower or higher than this range; the duration of heating time was largely fixed for 16 h, which was especially common for tuber, root, and legume starches, and even for some cereal starches. Different types of moisture were employed in HMT including the most commonly used vaporized steam (adjusted moisture content before HMT) as well as live injection of steam, and the range of moisture content varied from 10-40%. Reasons for the larger variations in heating temperatures and moisture levels than heating times could be ascribed to that increased heating temperatures and moisture levels more significantly changed the physicochemical properties than heating time (Cham and Suwannaporn, 2010). Furthermore, HMT condition in higher heating temperature and longer holding time has shown to affect flour properties more effectively than that in low heating temperature and short holding time (Lorlowhakarn and Naivikul, 2006).

Table 2.8 Heat-moisture treatment parameters for tuber, root, legume, and cereal starches

Starch source	Temp. (EC)	Time (h)	Moisture (%)	Reference
Tuber and root				
Potato	100	0.5	100% RH	Miyoshi, 2000
	100	16	20–30	Shin et al., 2005
	100	16	18–27	Donovan et al., 1983
	100	16	30	Hoover et al., 1994
	100	16	10–30	Hoover and Vasanthan, 1994
Sweet potato	120	1	20, 25	Lim et al., 2001
	40–100	12	20–90	Shin et al., 2005
	110	3	27–30	Collado et al., 2001
Cassava	110	3–16	18–24	Abraham, 1993
	100	16	18–27	Lorenz and Kulp, 1982
	100	10	30	Gunaratne and Hoover, 2002
Arrowroot	100	16	18–27	Lorenz and Kulp, 1982
Taro	100	10	30	Gunaratne and Hoover, 2002
New cocoyam	100	10	30	Gunaratne and Hoover, 2002
Yam	100	16	10–30	Hoover and Vasanthan, 1994
	100	10	30	Gunaratne and Hoover, 2002
Cereal				
Maize (normal, waxy, high amylose)	100	20 min	100% RH	Miyoshi, 2000
	100–140	80 min	30	Brumovsky and Thompson, 2001
	100, 120	2	30	Chung et al., 2009b
	120	24	30	Chung et al., 2009a

Table 2.8 Heat-moisture treatment parameters for tuber, root, legume, and cereal starches (*Continued*)

Starch source	Temp. (EC)	Time (h)	Moisture (%)	Reference
Maize (normal, waxy, high amylose)	110	4	45	Sang and Seib, 2006
	100	16	30	Hoover and Manuel, 1996a
	120	1	25, 30	Lim et al., 2001
Barley	100	1	18–27	Lorenz and Kulp, 1982
Oat	100	16	10–30	Hoover and Vasanthan, 1994
Triticale	100	16	18–27	Lorenz and Kulp, 1982
Red millet	100	16	20–30	Lorenz and Kulp, 1982
Rice (waxy, normal)	100	16	18–27	Khunae et al., 2007
	110	2	20–40	Shih et al., 2007
	110	1	15–25	Zavareze et al., 2010
Wheat	100	16	18–27	Donovan et al., 1983
	100	16	30	Hoover et al., 1994
	100	16	10–30	Hoover and Vasanthan, 1994
Legume				
Pea	100, 120	2	30	Chung et al., 2009b
Lentil	100, 120	2	30	Chung et al., 2009b
	100	16	30	Hoover et al., 1994
	100	16	10–30	Hoover and Vasanthan, 1994
Pigeon pea	100	16	30	Hoover and Manuel, 1996b
Pinto bean	100	16	30	Hoover and Manuel, 1996b
Wrinkle pea	100	16	30	Hoover and Manuel, 1996b
Smooth pea	100	16	30	Hoover and Manuel, 1996b
Black bean	100	16	30	Hoover and Manuel, 1996b
Jack bean	100	16	18–27	Lawal and Adebowale, 2005

Source: Hoover (2010)

Increase in amylose content on HMT has been reported by Miyoshi (2002) for maize and potato starches under the HMT conditions shown in Table 2.8, which has been explained by the theory proposed by Lu, Chen, and Lii (1996) that HMT could cause degradation of exterior linear chains of amylopectin to form newly created amylose.

HMT has also displayed influence on starch susceptibility toward enzyme hydrolysis; both increase and decrease in enzyme susceptibility by porcine pancreatic α-amylase (PPA) and/or amyloglucosidase were observed on heat-moisture treated cereals, tuber, root and legume starches (Hoover, 2010). When the physiological benefits of resistant starch were concerned, resistance of starch to enzymatic hydrolysis has been modified by HMT. On a large scale, method used to determine the RS levels present in starches was the one proposed by Englyst, Kingman, and Cummings (1992) (Figure 2.1) or modified ones from it. Brumovsky and Thompson (2001) found that HMT would increase the boiling-stable RS level but decrease the total RS content of a high-amylose maize starch (ae-VII). Boiling-stable RS was indicative of a portion of resistant starch surviving starch gelatinization. Similarly, increase in the amount of thermostable RS on HMT, RS determined in gelatinized HMT samples, has been reported by Chung, Liu, and Hoover (2009b) for corn, pea and lentil starches. Along with the increment in the RS content of HMT granular starches (ungelatinized samples), starch chain interactions (AM-AM, AM-AMP and AMP-AMP), especially those formed between amylose chains (disruption under high temperature), were postulated to be responsible for the decreased susceptibility toward enzyme hydrolysis (Chung et al., 2009b). Chung, Hoover, and Liu (2009a) have also investigated the impacts of single (HMT or annealing, ANN) or dual (HMT-ANN, ANN-HMT) hydrothermal modifications on amount of RS in corn starch, and found that either HMT or ANN, or dual treatment of ANN followed by HMT (ANN-HMT) caused decrement in RS content, whereas ANN of HMT corn starch showed RS level same as that of native corn starch. Increase in enzymatic hydrolysis on both HMT and ANN has been ascribed to crystallite disruption and enlarged granular porosity respectively, while crystalline perfection and chain interactions of ANN and HMT could also influence starch susceptibility toward enzyme hydrolysis, which accounted for the increase in RS content of HMT-ANN corn starch compared with ANN-HMT(Chung et al., 2009a). Sang and Seib (2006) found that HMT would decrease the RS content of raw Hylon VII (~ 70% amylose) maize starch from 72% to 37%, whereas increase in the RS level of cooked Hylon VII maize starch (25% to 32%) was observed on HMT. Among all the HMT conditions (40, 55 and 100 °C at 20, 50, and 90% moisture for 12 h) employed to sweet potato starch, modification of 40 °C and 90% moisture level resulted in higher RS content (68.9%) than native starch (67.3%) (Shin et al., 2005). However, no reason was given for the increase in RS content under this condition by the above authors.

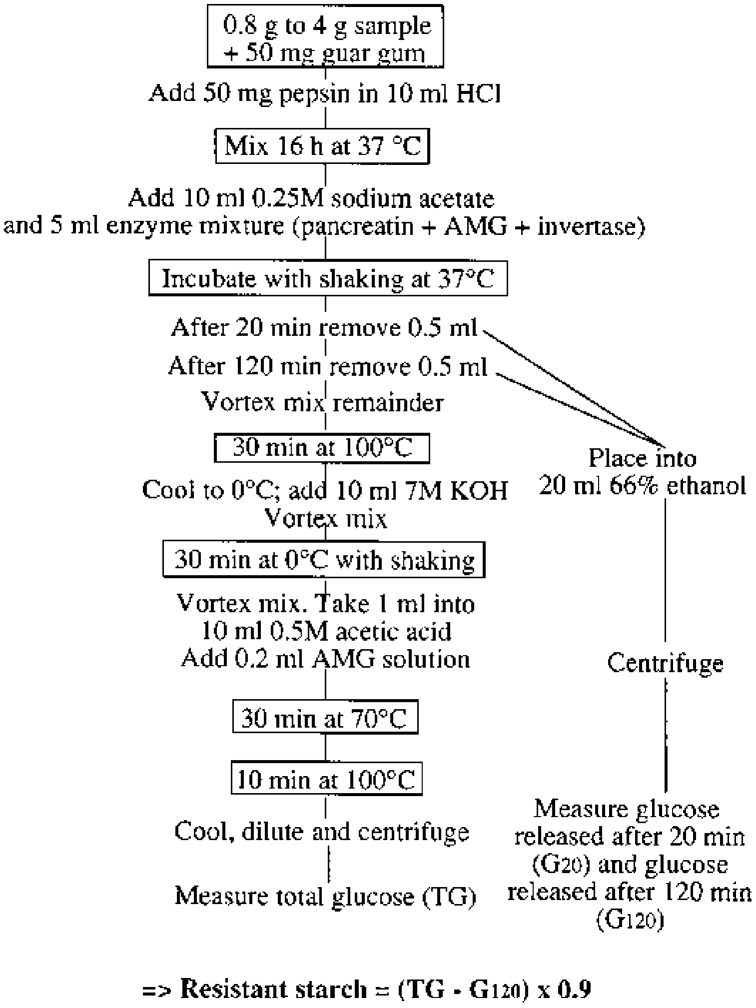

Figure 2.1 Resistant starch determination procedure according to Englyst et al. (1992).

Source: Champ et al. (1999)

When a retrograded RS 3 (40.4% of RS 3, generated from the hydrolyzed products of corn starch) was subject to HMT (autoclaving at 105-121 °C with 10-40% moisture for 1h, followed by cooling at room temperature for 2 h and freezing at -20 °C for 1 h), a final RS 3 content up to 75% was obtained on account of the modification (Jacobasch et al., 2006).

Alternation in X-ray diffraction patterns of starches subjected to HMT has been observed for B type starches (B⟶A or A + B), which, however, has rarely been reported for A- (typical for cereal) and C- types (typical for legume) starches (Hoover, 2010). On account of the presence of lipids, Shih et al. (2007) found that HMT of rice starches at higher moisture levels (30% and 40%) induced the formation of amylose-lipid complex resulting in an A pattern superimposed by a V type peak. Such mixed X-ray crystallography was, however, not observed for rice starches on HMT reported by Khunae, Tranb, and Sirivongpaisala (2007), which was probably due to the less amount of moisture (18-27%) present during HMT retarding chain association. Tapioca starch has been reported to possess a C (mixed A+B) or A pattern with the crystallinity of around 38% (Hoover, 2001). Tapioca starch subjected to HMT (100 °C at 30% moisture for 10 h) has not shown change in its A X-ray diffraction pattern (Gunaratne and Hoover, 2002).

In contrast to the decrease in starch crystallinity on HMT (90–120 °C at 20–30% moisture for 16 h) for B-type tuber (true yam, potato) and A-type cereal (rice) starches (Hoover, 2001), starch crystallinity of A-type tuber (new coco yam, cassava, taro) starches remained unchanged on HMT (100 °C at 30% moisture for 10 h) (Table 2.8; Gunaratne and Hoover, 2002). However, Abraham (1993) observed decrease in starch crystallinity of cassava after HMT. Furthermore, decrease in starch crystallinity with increase in moisture levels during HMT has been observed for rice starches (Shih et al., 2007; Khunae et al., 2007; Zavareze et al., 2010).

Under specific HMT conditions, the X-ray diffraction intensities of major peaks have been increased in maize (Hoover and Manuel, 1996a), wheat and oat (Hoover and Vasanthan, 1994), lentil (Hoover and Vasanthan, 1994), jack bean (Lawal and Adebowale, 2005) and wrinkle pea (Hoover and Manuel, 1996b) but decreased in potato (Hoover and Vasanthan, 1994; Lim, Chang, and Chung, 2001), yam (Hoover and Vasanthan, 1994), cassava (Abraham, 1993), pea and bean (Hoover and Manuel, 1996b) starches. Mainly noticed was the enhanced major peak intensities in A type starches, and this was attributed to the perfection of double helices (more closely packed and ordered) on HMT, however movement/detachment of adjacent double helices of B type starches on HMT accounted for the weaker X-ray diffraction (Hoover, 2010).

HMT has been shown to increase the gelatinization temperatures (To, Tp, Tc) and broaden the gelatinization temperature range (ΔT_G) of starches; the gelatinization enthalpy (ΔH_G) of starch on HMT displayed lower or unchanged values compared to that of native starch (Hoover, 2001). Narrowed ΔT_G of starches on HMT (100 °C at 24 and 27% moisture for 16 h), however, has also been reported for waxy rice starches (Khunae et al., 2007). One of the many intrinsic factors that influencing gelatinization of granular starches was the destabilization effect of surrounding amorphous regions on the melting of crystallites (Gunaratne and Hoover, 2002). Such destabilization effect was attributed to the swelling of amorphous areas in starch granule exerting a stress on crystalline regions, which striped polymer chains from the surface of crystallites (Adebowale et al., 2009). Starch chain interactions of amylose-amylose (AM-AM) and amylose-amylopectin (AM-AMP), and amylose-lipid complexing on HMT reduced the mobility of starch chains in the amorphous regions, which consequently would cause a higher temperature for amorphous regions to swelling and further disruption of crystalline regions (Hoover, 2001; Zavareze, and Dias, 2011). Decreased amylose leaching and appearance of V pattern suggested the occurrence of chain interactions and AM-L complexing, whereas, on account of failing to observe these, Chung et al. (2009a) ascribed the increase in gelatinization temperature on HMT of corn starch to aggregation of disrupted crystallites forming larger ones.

Gelatinization enthalpy (ΔH_G) measured the overall crystallinity of amylopectins, representing the number of double helices unraveling and melting during gelatinization (Tester and Morrison, 1990b). ΔH_G of starches decreased on HMT was ascribed to the disruption of double helices present in both crystalline and amorphous regions (Gunaratne and Hoover, 2002). Reduction in ΔH_G has also been attributed to the partial gelatinization of less thermostable crystallites on HMT (Hormdok and Noomhorm, 2007), resulting in less energy required during gelatinization.

Since gelatinization endotherm was indicative of the homogeneity of ordered structures inside the starch granules (Yuan, Thompson, and Boyer, 1993), broadened ΔT_G of HMT starches suggested melting of crystallites of different stability (Chung et al., 2009a), which was due to the AM-AM interactions and AM-L complexing on HMT leading to newly formed crystallites with varied stabilities (Hoover and Manuel, 1996a). Whereas HMT under some conditions has also been reported to favor the development of more homogeneous crystallites (Hoover et al., 1994), which explained the decreased ΔT_G of rice starch on HMT reported by Khunae et al. (2007).

Influence of HMT on starch retrogradation behavior has largely been studied by DSC to compare the appearance and development of retrogradation endotherms with native counterparts (Lu et al., 1996; Miyoshi, 2000; Gunaratne and Hoover, 2002). HMT has increased the retrogradation enthalpy (ΔH_R) of cereal (wheat and oat) and legume (lentil) starches, which was ascribed to the interplay of enhanced starch crystallinity and AM-AMP interactions on HMT (Hoover and Vasanthan, 1994). Furthermore, Hoover and Vasanthan (1994) also observed that HMT under the same condition has caused decrease in ΔH_R of potato starch. Similarly, Gunaratne and Hoover (2002) found that HMT exerted decrease in ΔH_R of potato and true yam (B-type) but no significant change in those of taro, new cocoyam and cassava (A-type) starches. Upon HMT, the influence of AM-AMP could be nullified by starch crystallite disruption (Hoover and Vasanthan, 1994), and the latter, more prominent for B-type starches, would cause separation between the outer branches of adjacent amylopectin chains hindering the recrystallization/reassociation of amylopectins and affect the stabilities of newly formed crystallites (Gunaratne and Hoover, 2002).

From the reviewed literatures on the impact of HMT on swelling and pasting properties of starches from different botanical sources (Hoover, 2010; Jacobs and Delcour, 1998), crystallite disruption on HMT accounted for the decrease in starch swelling of cereal, legume and tuber starches, and as an indicative of starch solubilization, starch solubility and amylose leaching has been found increased on HMT of some cereal starches. Rheological properties during starch pasting determined by either BVA or RVA of tuber and root, cereal, and legume starches have been influenced by HMT, on which increase was found in PT and thermal stability i.e. HPV, and decrease was noticed in PV but SB was increased or decreased. These changes in pasting properties on HMT has been attributed to decreased starch swelling and amylose leaching, and increased starch chain interactions.

Utilization of heat-moisture treated starches in starchy or starch-contained foods processing has been studied. Abraham (1993) employed HMT to cassava starch to stabilize the paste and used modified starches to produce a sweet meat product with good organoleptic properties. Moreover, heat-moisture treated tapioca starch has also been used to make tapioca pearls (Breuninger et al., 2009). Applications of HMT of starches to starch noodle making have been conducted on sweet potato noodle (Collado et al., 2001), sago starch noodle (Purwani et al., 2006), and rice noodles (Hormdok and Noomhorm, 2007; Cham and Suwannaporn, 2010). Hoover (2010) has also reviewed the utilization of HMT of starches in baking and processed foods.

2.5 Flour/starch blends and noodle making

In order to avoid the excessive use of chemically modified starches in starch-based food manufacturing, combining different starches to get at least some properties close to those derived from chemical modification has been achieved (Obanni and BeMiller, 1997). Starch blends from same cultivar (with different amylose content) or various botanical sources (unmodified and/or modified) have been shown thermal, pasting, rheological and swelling properties that were either additive or nonadditive types depending on the blending components and mixing ratios (Liu and Lelièvre, 1992; Sasaki et al., 2000; Ortega-Ojeda and Eliasson, 2001; Gunaratne and Corke, 2007; Puncha-arnon et al., 2008; Park et al., 2009; Zhang et al., 2011). And the approach to imparting desired properties to starch blends has been evidenced by the increased total dietary fiber content (Novelo-Cen and Betancur-Ancona, 2005), improved pasting characteristics (Obanni and BeMiller, 1997; Chen, Lai, and Lii, 2003), retarded retrogradation tendency (Obanni and BeMiller, 1997), improved gel structure (Karam et al., 2005), increased paste elasticity (Oh, Kim, and Yoo, 2010; Sun and Yoo, 2011), and elevated freeze-thaw stability/lowered syneresis (Karam et al., 2005). Based on these findings, researchers concluded that such distinct physicochemical properties of starch blends could be due to the interactions among starch molecules and granules, which cannot be simply interpreted by the final amylose content, sum of outputs of each individual component, or mixing ratio.

Due to the limitation of gel quality, flour of fresh rice grain failed to be used for rice noodle making (Hormdok and Noomhorm, 2007). Although ageing of rice grain could be applied to rice noodle making, its time-consuming disadvantage forced researchers to seek for more economic solutions for manufacturers. Rice flour and starch have been combined with corn (Wang et al., 2000), potato (Sandhu, Kaur, and Mukesh, 2010), pigeon pea (Yadav, Yadav, and Kumar, 2011), and hydrothermally modified rice (Hormdok and Noomhorm, 2007; Cham and Suwannaporn, 2010) starches/flours to make noodles, and composite noodles displayed acceptable qualities. Apart from hydrothermal modifications, cross-linking as the widely used chemical modification has been shown impacts on starch properties (Singh, Kaur, and McCarthy, 2007). Cross-linking has increased the granular rigidity under high shear force, which accounted for the use of cross-linked tapioca starch in clear noodle making (Kasemsuwan et al., 1998) as well as rice noodle making (Charutigon et al., 2008).

Produced mainly from rice flour, rice noodles could be divided into two types according to the processing methods: extruded rice vermicelli and slurry-sheeted flat rice noodles (Fu, 2008). Classification of rice noodles according to the moisture content of the final products could divide into three types: fresh rice noodles with the highest moisture content of 62-64% but shortest shelf-life of only 1-2 days, semi-dried type having around 37% moisture content and 2-3 days shelf-life, and dried rice noodles with the lowest moisture content (< 13%) and longest shelf-life (approx. 1 year) (Lu and Collado, 2010). The procedure of making rice vermicelli noodles usually includes partial gelatinization of rice dough, and the kneaded dough is then extruded into noodle strands which are fully gelatinized and cooled prior to drying. While the sheeted or flat rice noodles are prepared from slurry sheeting by gelatinizing rice slurry to form noodle sheets (about 1mm thickness) which can be cut into strips and sold as fresh or dried noodles (Fu, 2008). Dried rice noodles should show acceptable characteristics such as uniform and straight strand, white and translucent color, and absence of broken strands, while desired cooking qualities of rice noodle should manifest a short cooking time hydration with minimum turbidity and surface stickiness, and little solid loss in the cooking water (Fu, 2008; Hormdok and Noomhorm, 2007).

Juliano and Sakurai (1985) stated that rice varieties with high amylose content (> 25 g/100 g) were traditionally used to prepare rice noodles, which was due to the retrogradation of amylose after starch gelatinization contributing to a gel network and setting the noodle structure (Mestres, Colonna, and Buleon, 1988). It was widely accepted that amylose content of starch was positively correlated with starch gel hardness, which accounted for the findings that rice starches with high gel firmness would produce rice noodles with superior textual qualities (Yoenyongbuddhagal and Noomhorm, 2002). In addition to starch gel strength, swelling power and pasting parameters have also been shown correlation with rice noodle textures (Bhattacharya, Zee, and Corke, 1999; Hormdok and Noomhorm, 2007; Han, Cho, and Koh, 2011).

On account of the full utilization of polished rice in rice noodle making, the final product contained certain nutritional benefits and generally showed higher protein content than other starch noodles (Lu and Collado, 2010). Compared with cooked waxy rice, cooked nonwaxy rice has been shown lower glycemic index response (Juliano et al., 1989), and accordingly rice noodles prepared from nonwaxy rice would show physiological benefits, since starch noodles through retrogradation can be a source of resistant starch (Collado et al., 2001).

CHAPTER III

MATERIALS AND METHODS

3.1 Materials

Raw materials were kindly provided by one local noodle company (NIT noodle factory, Sukhothai province, Thailand), including broken rice, broken rice noodles, tapioca starch, corn flour and cross-linked tapioca starch. Commercial rice noodles from the same noodle factory were used for quality comparison. Broken rice was first cleaned by running water, and then steeped in tap water (10 times the weight of rice) for 2 h at room temperature (~25 °C). Soaked broken rice was drained and milled with tap water (10 times the weight of rice) by a local double stone mill. Rice slurry was then centrifuged to get rice cake which was dried at 45 °C in a tray drier until the moisture content reached 10–12 g/100 g. Dried rice cakes were ground by a commercial blender and sieved through a 120-mesh screen to get rice flour. Broken rice noodles were first chopped by a commercial blender to get small fragments and then ground by a commercial grain grinder (Lita® brand, Thailand) before sieving through a 120-mesh screen to get broken rice flour. Flour and starch samples were airtightly sealed in aluminum laminated bags (purchased from local market) and stored at 4 °C prior to further use.

3.2 Heat-moisture treatment

Heat-moisture treatment used in this study was modified from Abraham (1993). The moisture content of tapioca starch was adjusted to 17, 20 and 23 g/100 g by spraying the calculated amount of distilled water. Tapioca starch was weighed and immediately transferred to a polypropylene bag, and distilled water was sprayed by a foggy while shaking the starch sample quickly. After mixing thoroughly, samples were transferred to aluminum laminated bags and airtightly sealed. Samples were equilibrated at room temperature for 24 h and the accurate moisture content was determined before being transferred to metal cans (half size filling) and sealed. Sealed samples were then incubated in hot-air ovens with the set temperatures of 95, 110 and 125 °C for 16 h to complete the modification. After removal from the oven, samples were unsealed and spread on stainless steel trays, and then dried at 45 °C in a hot-air oven till the moisture content reached around 10 g/100 g. Dried samples were ground by a commercial blender and sieved through a 120-mesh screen to obtain heat-moisture treated tapioca starch. Modified tapioca starch samples were airtightly sealed in aluminum laminated bags and stored at 4 °C prior to further use. In this study tapioca starch under specific heat-moisture treatment condition was signed as HMT-TS (moisture content-heating temperature), such as HMT-TS (17-95). HMT conditions of temperature and moisture employed to native tapioca starch was a factorial design. The modification of heat-moisture treatment was replicated twice. The modified samples were analyzed for the following properties.

3.2.1 Proximate analysis

Moisture content was determined by the method of AOAC (2005). Apparent amylose content was measured by colorimetric method (Juliano, 1971; Appendix A.1). Chemical analyses were replicated three times.

3.2.2 *In vitro* starch digestibility

In vitro starch digestibility was expressed as the level of resistant starch present in samples. Both ungelatinized and gelatinized samples were determined for the resistant starch content by Megazyme® resistant starch assay kit (Megazyme International Ireland Ltd., Wicklow, Ireland). Sample was incubated by pancreatic α-amylase and amyloglucosidase (AMG) to hydrolyze non-resistant starch; the resultant resistant starch was further hydrolyzed to glucose by AMG. D-Glucose was measured by glucose oxidase/peroxidase reagent (GOPOD), and resistant starch content was calculated by the Megazyme *Mega-Calc*™ provided with the assay procedure (Appendix A.2).

Resistant starch content in gelatinized samples was also analyzed and the gelatinization procedure followed that of Chung et al. (2009b). The ungelatinized sample (100 mg) and distilled water (2 ml) were added to test tubes. The tubes were capped, and the contents were mixed on a vertex mixer for 1 min. The tubes were then heated in boiling water for 20 min while gently stirring on a magnetic stirrer with hot plate. After heating, the tubes were placed in a water bath at 37 °C to equilibrate for 10 min. Determination of resistant starch content was immediately conducted following the above resistant starch assay procedure. Results of resistant starch content in ungelatinized and gelatinized samples were averaged from three replicates.

3.2.3 X-ray diffraction

X-ray diffractograms of samples were obtained with an X-ray diffractometer (Bruker AXS Model D8 Discover, Bruker AXS, USA). The samples were analyzed under the operating conditions as: target voltage 40 kV, current 40 mA, increment 0.02 degree/step, scan speed 0.5 sec/step and a Super Speed Detector (VÅNTEC-1 Detector). Sample X-ray patterns were determined by comparing with the proposed features of each starch X-ray pattern (Zobel, 1988). Crystallinity of sample was determined by the ratio of crystalline area to the total area of crystalline and amorphous regions.

$$\text{Degree of crystallinity (\%)} = \frac{\text{Crystalline area} \times 100}{\text{Total area}_{\text{(Crystalline area + Amorphous area)}}}$$

3.2.4 Thermal properties

Both gelatinization and retrogradation behaviors were determined by Differential Scanning Calorimetry (Diamond DSC, Perkin Elmer, Norwalk, Connecticut, USA) equipped with an intercooler unit (Perkin Elmer, Model 2P, USA.) and using nitrogen as purge gas. Parameters of thermal properties were recorded and/or calculated by a Pyris® manager software. Sample (9 mg, db) was weighed directly in stainless steel pan with rubber O-ring (P/N 0319-0218), and deionized water was added to obtain a sample-water ratio of 1:3 (w/w). The pan was hermetically sealed by a Quick Press (NO. 0990-8467) and equilibrated at room temperature for 2 h prior to analysis. Sample pan was then heated from 30–150 °C at the rate of 10 °C/min. An empty pan was used as a reference.

After gelatinization sample pan was stored at 4 °C for seven days, which was subsequently equilibrated at room temperature for 4 h, and then rescanned by the same profile as gelatinization. Thermal transition onset (T_o), peak (T_p) and conclusion (T_c) temperatures were recorded. The energy (enthalpy, ΔH) required to melt starch crystallites was measured from the areas of endothermic peaks in the thermograms on a dry weight basis (J/g), and the temperature range (ΔT) between onset and conclusion temperatures was calculated as $T_c - T_o$. For DSC analysis, each sample was replicated at least twice.

3.2.5 Swelling power and solubility

Swelling power and solubility was determined following the method of Subramanian et al. (1994) with minor modifications. Mixture of sample (1 g) and distilled water (30 ml) was heated in plastic centrifuge tubes (NALGENE®) at 95 °C for 1 h in a water bath. Intermittent stirring by a vortex mixer was applied to prevent clumping during incubation. Removed from the water bath, sample tubes were centrifuged at 3,500 rpm for 15 min. Immediately after centrifuging, the supernatant was carefully decanted to a pre-weighed glass beaker with subsequent drying at 110 °C to get constant weight of dry matter, and the weight of wet sediment was also recorded. Swelling power (SP, g/g) was calculated as the ratio in weight of the wet sediment to initial weight of dry sample. Solubility (%) was the percentage of sample dissolved in the supernatant. The determination of swelling power and solubility was conducted at least three times for each sample.

$$\text{Swelling power (g/g)} = \frac{\text{weight of wet sediment (g)}}{\text{weight of dry sample (g)}}$$

$$\text{Solubility (\%)} = \frac{\text{weight of dry matter in supernatant (g)}}{\text{weight of dry sample (g)}} \times 100\%$$

3.2.6 Pasting characteristics

Pasting characteristics were determined by Rapid Visco-Analyzer (RVA; Series S4A, Newport Scientific, Australia). Sample (3.0 g, 12 g/100 g moisture basis) was weighed directly in the RVA canister and distilled water was added to obtain a suspension with total weight of 28.0 g. The suspension was held at 50 °C for 1 min, heated to 95 °C within 3 min 45 s, then held at 95 °C for 2 min 30 s, cooled to 50 °C within 3 min 51 s and held at 50 °C for 1 min 24 s (AACC, 2000). A high rotation speed (960 rpm) was used to sufficiently mix the suspension for 10 s before lowering to a constant stirring speed (160 rpm) during the measurement. Parameters of pasting characteristics included peak viscosity (PV), the highest viscosity during heating; hot paste viscosity (HPV) at the end of holding at 95 °C; cold paste viscosity (CPV) at the end of holding at 50 °C; breakdown (BD = PV – HPV); setback (SB = CPV – HPV) and pasting temperature (P_T) (Hormdok and Noomhorm, 2007). All measurements were processed in triplicate.

3.2.7 Gel texture

TA-XT*i*2 texture analyzer (Stable Micro Systems, Godalming, UK) provided with an Exponent Lite Express software was used to determine the texture of gels formed in the RVA canisters after pasting test. The profile or condition used to determine the texture of gels was adapted from Collado and Corke (1999) with minor modifications. After removal of RVA paddle, the canister was covered with Parafilm®, and stored at 4 °C overnight. Sample was then equilibrated at room temperature for 4 h before the texture analysis was carried out. Puncture test of the gel was conducted by using a 6.0 mm diameter cylinder stainless steel probe to puncture 10.0 mm depth of the gel at 1.0 mm/s. Only one cycle was applied to the puncture test. Gel hardness was the maximum force to break the gel, and the value was automatically recorded by the Exponent software. Each gel was tested at five different positions on the surface, and the result was obtained from all the data for each sample.

3.3 Preparation of rice noodles

Rice noodles were prepared from various flour combinations as below. Apart from RF and BRNF, certain amount of TS, HMT-TS or CTS was incorporated into the flour combinations. All flour combinations were determined for swelling power and solubility, pasting characteristics and TPA of gels according to 3.2.5, 3.2.6 and 3.2.7. Flour combinations were mixed with water to obtain a solid concentration of 40 g/100 g; the slurries consisted of 30 g/100 g of RF/BRNF (30%:0%, 25%:5%) and 10 g/100 g of TS, or HMT-TS or CTS. After 1 h equilibration at room temperature, appropriate amount (65 g) of slurry was spread evenly on a stainless steel tray (12 x 21 cm) and steamed for 5 min to form a noodle sheet with approximate thickness of 1 mm. The noodle sheet was scraped out and dried at 70 °C for 10 min before storing at 4 °C for 24 h. The noodle sheet was then cut into strips of 3-mm width and dried at 45 °C till the final moisture content was around 10–12 g/100 g. Preparation of composite dried rice noodles followed a complete randomized design, and was replicated twice. Dried noodle samples were sealed in polypropylene bags and kept under room temperature. The qualities of dried noodle samples were analyzed for the following properties.

3.3.1 Cooking quality

Cooking qualities including cooking time, cooking loss and rehydration ratio were determined by the method of Collado et al. (2001). Dried noodles (5.0 g) were cut into 5-cm length and boiled in 200 ml water. Optimum cooking time was determined by removing a strand of noodle every 30 s and pressing between 2 pieces of watch glass until the center of noodles became transparent. Cooking was stopped by rinsing briefly in water.

Cooking loss (%) was determined by evaporating to dryness of cooking water and rinse water at 110 °C. The dried residue was weighed and reported as a percentage of the weight of dried noodles before cooking. As for rehydration ratio (%), cooked noodles were drained in a strainer for 2 min and excess moisture on the surface was wiped with paper towel; the ratio in weight of wet mass to weight of dried noodles before cooking was expressed as rehydration ratio. The determination of cooking qualities was replicated three times for each noodle sample.

$$\text{Cooking loss (\%)} = \frac{\text{weight of dry matter in cooking water (g)}}{\text{weight of dry noodle (g)}} \times 100\%$$

$$\text{Rehydration ratio (\%)} = \frac{\text{weight of cooked noodle (g)}}{\text{weight of dry noodle (g)}} \times 100\%$$

3.3.2 Texture quality

Texture profile analysis (TPA) of cooked noodles was carried out following the method of Kim and Seib (1993) with some modifications. Upon optimum cooking time, cooked noodles were drained in a strainer for 2 min, and several strands were laid individually in a covered petri dish to equilibrate for 10 min and minimize dehydration. TPA of cooked noodles was conducted within 15 min by the TA-XT*i*2 texture analyzer. One strand of noodle was placed on the filter paper which was affixed to the platform by double-sided adhesive tape. TPA was operated in the compression mode by a cylindrical probe (P/100, 100.0 mm diameter) with test speed of 1.0 mm/s and 75% compression of the noodle thickness. The pause between first and second cycles was 1 s. TPA parameters of hardness (g force), adhesiveness (g·s) and chewiness (g·mm) were calculated by the Exponent Lite Express software from the force-time curve (Bhattacharya et al., 1999). As a variable, adhesiveness was recorded automatically by the software, while chewiness was calculated from gumminess and springiness as below. A typical TPA force-time curve was shown (Figure 3.1) to define texture parameters used in calculation. Ten strands of each noodle sample were tested.

Chewiness = gumminess × springiness (Figure 3.1)

where, gumminess = hardness × cohesiveness (Figure 3.1)

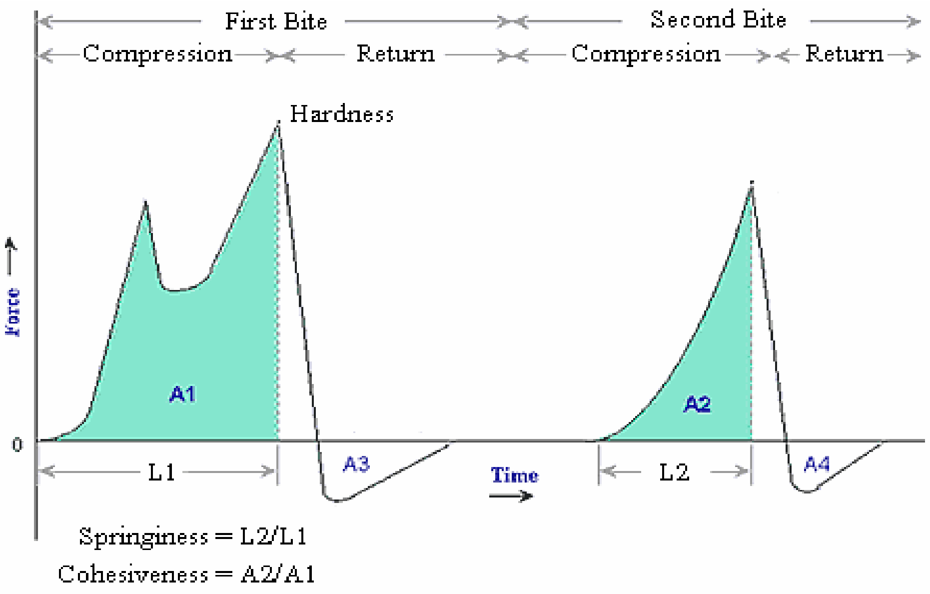

Figure 3.1 Texture profile curve and definitions of standard TPA terms.

Source: Bourne (1978).

As for the tensile strength of cooked noodles, the TA-XTi2 texture analyzer was equipped with a spaghetti-noodle tensile grip analyzer (Stable Micro Systems, Godalming, England). Profile of determining noodle tensile strength was followed that of Bhattacharya et al. (1999). A single strand of noodle was fixed on the analyzer by positioning through slots and winding two or three times around the parallel friction rollers to anchor the sample ends and reduce any slippage. The upper roller was set to move up at a speed of 3.0 mm/s from the lower one. Tensile strength (g force) was the maximum force required to break the noodle. Ten strands of each noodle sample were tested within 15 min.

3.3.3 Sensory evaluation

Sensory evaluation was carried out to compare the qualities of commercial noodle and composite noodles prepared from flour combination containing BRNF. The noodles were presented to the panelists (30) as plainly boiled noodles, which were cooked based on the optimum cooking time earlier determined and served within 20. A 5-point quality scoring, the sensory attributes color, firmness, clarity, flavor, and general acceptability were evaluated (Appendix B) (Collado et al., 2001). The statistical analysis was performed according to randomized complete block design (RCBD).

3.4 Statistical analysis

Analysis of variance (ANOVA) was performed to compare treatment means, and differences were reported at the confidence level of 95% ($P < 0.05$) by Duncan's multiple range test (DMRT) using SPSS v. 16 software.

CHAPTER IV

RESULTS AND DISCUSSION

4.1 Flour and starch properties

Apparent amylose contents of RF, BRNF, TS and HMT-TS were listed in Table 4.1. HMT induced increase in the amylose contents from 17.42 g/100 g (TS) to 17.87–19.92 g/100 g (HMT-TS). It was postulated that newly created amylose could arise from exterior linear chains of amylopectin degraded by HMT (Miyoshi, 2002). And HMT at 110 °C led to the highest amylose content at each moisture level, followed by those at 95 °C and 125 °C. Furthermore, at each modification temperature, HMT having 20 g/100 g moisture level showed the highest amylose content. These trends in amylose content change could be due to varied extents of amylopectin degradation under different modification conditions.

Lowest RS content of RF (0.49%) among all the ungelatinized samples could be caused by its highest susceptibility toward enzyme hydrolysis. Weak structure due to the branch linkages in crystalline region of A-type (cereal) starches rendered starch granule highly susceptible to enzyme hydrolysis (Jane et al., 1997). Differentiating from RF, BRNF went through rice noodle making process, which would induce starch retrogradation. Retrograded starch has been regarded as the third type RS (RS 3) (Englyst et al., 1992), which mainly consisted of retrograded amylose (Biliaderis et al., 1985; Sievert and Pomeranz, 1989, 1990; Szczodrak and Pomeranz, 1991); while retrograded amylopectin isolated or in the form of waxy starch has also showed resistant to enzyme hydrolysis (Berry, 1986; Sievert and Pomeranz, 1989; Escarpa et al., 1996). And this would explain the higher RS content present in BRNF (1.14%) compared to that in RF. Heating of

BRNF suspension, however, caused less reduction of RS content in BRNF (1.14% to 1.03%) compared with those in RF (0.49% to 0.31%). Decrease of RS content in BRNF after heating was probably due to the enzymatic hydrolysis of melted recrystallized amylopectin, which displayed lower melting point (<100 °C) contrasting with thermally stable fraction of recrystallized amylose (>100 °C) (Miles et al., 1985; Gudmundsson, 1994).

Table 4.1 Apparent amylose and resistant starch contents of flours and starches

Sample	AC (%)	RS^{UN} (%)	RS^{G} (%)
RF	15.98 ± 0.74	0.49 ± 0.09	0.31 ± 0.15
BRNF	17.97 ± 0.65	1.14 ± 0.19	1.03 ± 0.20
TS	17.42 ± 1.39d	17.88 ± 3.95a	0.19 ± 0.11e
HMT-TS (17-95)	19.08 ± 0.71ab	4.66 ± 0.26c	0.29 ± 0.02de
HMT-TS (17-110)	19.66 ± 0.49a	15.67 ± 1.02ab	0.44 ± 0.11cde
HMT-TS (17-125)	17.87 ± 0.83cd	5.29 ± 0.05c	0.64 ± 0.05bc
HMT-TS (20-95)	19.47 ± 1.00a	5.39 ± 0.23c	0.35 ± 0.01cde
HMT-TS (20-110)	19.92 ± 1.88a	4.49 ± 0.54c	0.60 ± 0.27bcd
HMT-TS (20-125)	18.54 ± 0.52bc	3.78 ± 0.73c	0.82 ± 0.18b
HMT-TS (23-95)	19.37 ± 0.58a	12.50 ± 1.20b	0.33 ± 0.01cde
HMT-TS (23-110)	19.72 ± 0.73a	3.71 ± 0.23cd	0.56 ± 0.11bcd
HMT-TS (23-125)	18.41 ± 0.48bc	5.15 ± 1.60c	1.13 ± 0.19a

RF, rice flour; BRNF, broken rice noodle flour; TS, tapioca starch; HMT, heat-moisture treatment; AC, apparent amylose content; RS^{UN}, resistant starch in ungelatinized samples; RS^{G}, resistant starch in gelatinized samples.

Means followed by different letters in a column are significantly different at $P<0.05$.

For TS, its granule possesses multiple layers and a tightly organized structure resulting in less access for the penetration of chemical reagents and enzymes to the starch granule (Breuninger et al., 2009), which accounts for the high RS content present in it (Table 4.1). HMT-TS, however, showed increased susceptibility toward enzyme hydrolysis, manifesting in the significant decrease in RS contents from 3.71% of HMT-TS (23-110) to 15.67% of HMT-TS (17-110). Increase in enzyme susceptibility on HMT has also been reported for some tuber and root starches, which was attributed to the disruption of double helices in amorphous regions during HMT (Gunaratne and Hoover, 2002). Reported studies also attributed increased enzyme hydrolysis of starch on HMT to the damage of starch granule (Miyoshi, 2002) or crystallite disruption near granule surface (Hoover and Vasanthan, 1994), which caused more access to enzyme hydrolysis.

Starch gelatinization would cause the disintegration of starch granules, which facilitated the further enzyme hydrolysis resulting in decrease in RS content for TS and HMT-TS (Table 4.1). By comparing, gelatinized HMT-TS had higher RS content (0.29%-1.13%) than gelatinized TS (0.19%); meaning HMT would induce increase in the RS content of gelatinized HMT-TS. And this was attributed to more of the thermostable RS formed by starch chain interactions during HMT, which showed resistant to gelatinization (Chung et al., 2009b).

4.2 X-ray diffraction

Rice flour exhibited a characteristic A-type X-ray diffraction pattern (Figure 4.1 and Table 4.2) with strong diffraction intensities at 5.8, 5.2 and 3.8 Angstroms (Å) (Table 4.3) (Zobel, 1988). However, by comparing, the broken rice noodle flour showed distinct pattern from that of rice flour expressed as a diffuse pattern typical of amorphous materials (Ottenhof, Hill and Farhat, 2005).

X-ray diffraction has been used to study bread staling (Dragsdorf and Varriano-Marston, 1980) and starch gel retrogradation (Miyoshi, 2002) by observing changes in the X-ray diffraction patterns, peak intensities and starch crystallinity following storage time. The retrogradation kinetics of starch in both gel and powder forms studied by X-ray diffraction showed that the latter prepared by dehydration of starch gels terminating starch retrogradation at any time during the process provided a rapid and convenient means of measuring specific ‘real time’ recrystallization (Roulet et al., 1988). BRNF was dehydrated and powdered from a highly concentrated flour gel (i.e. noodle sheet) after pre-drying and subsequent cooling. Availability of water to starch chains in gelatinized noodle sheet for aging was influenced by the pre-drying causing moisture content in noodle sheet during cooling much lower than that during gelatinization (60% water). Moreover, the cooling process was shorter than most experimental conditions which would further affect the recrystallization of starch chains. Accordingly, due to the low water content, high glass transition temperature and a highly viscous glassy state of the amorphous gel would effectively hinder molecular mobility (Gudmundsson, 1994), which caused insufficient recrystallization of starch chains. The impact of use of mixed starches and possible addition of additives (e.g. monoglyceride) in noodle making, and insufficient recrystallization of noodle sheet on the X-ray diffraction of BRNF exhibited unique characteristics differing from those of single starch gels or powdered gels from pure starches (Figure 4.1). By comparison, the X-ray diffraction pattern of BRNF displayed more similarity to that of fresh bread (Dragsdorf and Varriano-Marston, 1980) than those of fresh and one-day stored starch gels (Miyoshi, 2002).

Starch crystallinity in this study was determined by the percentage of crystalline area in the total area of both crystalline and amorphous regions. Crystallinity of RF was 21.75%, while the much lesser crystalline area of BRNF caused the corresponding crystallinity far lower (8.01%) than that of RF (Table 4.2). In addition to the pre-drying of fresh noodle sheets and ephemeral duration of cooling for starch chain recrystallization, drying of fresh noodle strands ceased the retrogradation process as the moisture content of end product reached around 10% (Longton and Legrys, 1981; Zeleznak and Hoseney, 1986; Marsh and Blanshard, 1988). Additionally, for a fully retrograded starch sample, only about 15% of the available amylopectin could be crystallized (Roulet et al., 1988). Since the X-ray diffraction measured the crystalline fraction of the amylopectin in starch, the limited retrogradation extent and availability of amylopectin would account for the less crystalline area and lower degree of crystallinity of BRNF.

A-type diffraction pattern has been shown for both native TS and HMT-TS (Figure 4.1), indicating that HMT demonstrated no change in the X-ray pattern of TS, which was in conformity with the result observed by Gunaratne and Hoover (2002). However, the degree of crystallinity of HMT-TS was higher than that of TS (Table 4.2), and, in comparison, both of the crystalline and amorphous areas of HMT-TS were larger than those of TS. Starch crystallinity has been reported (Gunaratne and Hoover, 2002) to be influenced by crystallite size, double helices orientation within the crystallite, average chain length of amylopectin, and amount of short chain fraction of amylopectin.

Based on the decrease in gelatinization enthalpy and reduction in starch crystallinity, Chung et al. (2009a) reported that HMT may increase the mobility of double helices in the crystalline regions causing disruption of hydrogen bonds among double helices and between adjacent double helices. Increased crystalline and amorphous areas suggested that perfection of the remaining crystallites due to HMT negated the disruption of less thermostable double helices in both crystalline and amorphous regions, which might account for the increment in intensities of major peaks and starch crystallinity.

Increase in the crystalline area of TS on HMT was probably due to the rearrangement of double helices within the crystalline regions forming more ordered crystalline array to diffract X-rays (Hoover and Vasanthan, 1994; Hoover and Manuel, 1996a, b). The exterior linear chains (mainly A chains) of amylopectin degraded during HMT caused increase in amylose content (Lu, Chen, and Lii., 1996; Miyoshi, 2002), which would contribute to the enlarged amorphous area in starch granules. Furthermore, diffraction intensities of major peaks of HMT-TS were higher than those (at same 2-theta angles) of TS (Table 4.3).

Table 4.2 X-Ray diffraction patterns, crystalline and amorphous areas and crystallinities of flours and starches

Sample	X-ray pattern	Crystalline area	Amorphous area	Crystallinity (%)
RF	A	3679	13235	21.75
BRNF	-	1300	14921	8.01
TS	A	3769	12777	22.78
HMT-TS (17-95)	A	4882	14954	24.61
HMT-TS (17-110)	A	4639	14654	24.05
HMT-TS (17-125)	A	8634	23227	27.10
HMT-TS (20-95)	A	8756	23737	26.95
HMT-TS (20-110)	A	8136	23038	26.10
HMT-TS (20-125)	A	4033	13442	23.08
HMT-TS (23-95)	A	8274	23318	26.19
HMT-TS (23-110)	A	7258	21555	25.19
HMT-TS (23-125)	A	7743	22612	25.51

RF, rice flour; BRNF, broken rice noodle flour; TS, tapioca starch; HMT, heat-moisture treatment.

Table 4.3 X-Ray diffraction intensities of the major peaks of flours and starches

Sample	Interplanar spacings (*d*) in Å with intensities (CPS)^{a} of 2-theta at				
	15.1	16.9	17.2	20.0	23.0
RF	5.85(1136)	5.25(1153)	5.14(1289)	4.44 (887)	3.86(1051)
BRNF				4.40(996)	3.85 (807)
TS	5.86(1142)	5.21(1257)	5.15(1281)		3.84(1002)
HMT-TS (17-95)	5.84(1418)	5.23(1486)	5.19(1553)		3.87(1274)
HMT-TS (17-110)	5.86(1406)	5.26(1528)	5.20(1531)		3.88(1217)
HMT-TS (17-125)	5.86(2461)	5.22(2623)	5.18(2708)		3.86(2125)
HMT-TS (20-95)	5.87(2484)	5.23(2701)	5.19(2719)		3.86(2128)
HMT-TS (20-110)	5.88(2400)	5.22(2614)	5.18(2607)		3.87(1989)
HMT-TS (20-125)	5.83(1253)		5.15(1377)		3.86(1065)
HMT-TS (23-95)	5.87(2437)		5.19(2663)		3.86(2112)
HMT-TS (23-110)	5.88(2189)	5.23(2343)	5.19(2389)		3.88(1898)
HMT-TS (23-125)	5.87(2403)	5.21(2523)	5.17(2538)		3.86(1994)

[a] Counts per second.

RF, rice flour; BRNF, broken rice noodle flour; TS, tapioca starch; HMT, heat-moisture treatment.

Both RF and TS showed intensities of major diffraction peaks similar to each other; while HMT has increased the intensities of corresponding major diffraction peaks, and specific trends in the change in peak intensities were also noticed among HMT-TS. For TS modified at both 95 and 110 °C, highest peak intensity was found on HMT at moisture level of 20% followed by those at 23% and 17%. However, HMT at 125 °C altered the highest to lowest peak intensities into the order of modification at moisture levels of 17%, 23% and 20% (Table 4.3). Influence of HMT moisture levels and temperatures on changes in peak intensities has been ascribed to the thermal energy and moisture rearranging double helices or rupture of linking between adjacent double helices on different HMT conditions (Hoover and Vasanthan, 1994). In addition, similar effects of HMT on slight shift in major existing *d*-spacings and lack of major new *d*-spacings of starches (Hoover and Vasanthan, 1994) has also been shown for TS in our study, which further verified the suggestion that increase in peak intensities were largely due to structural changes within the crystalline domains of the granule.

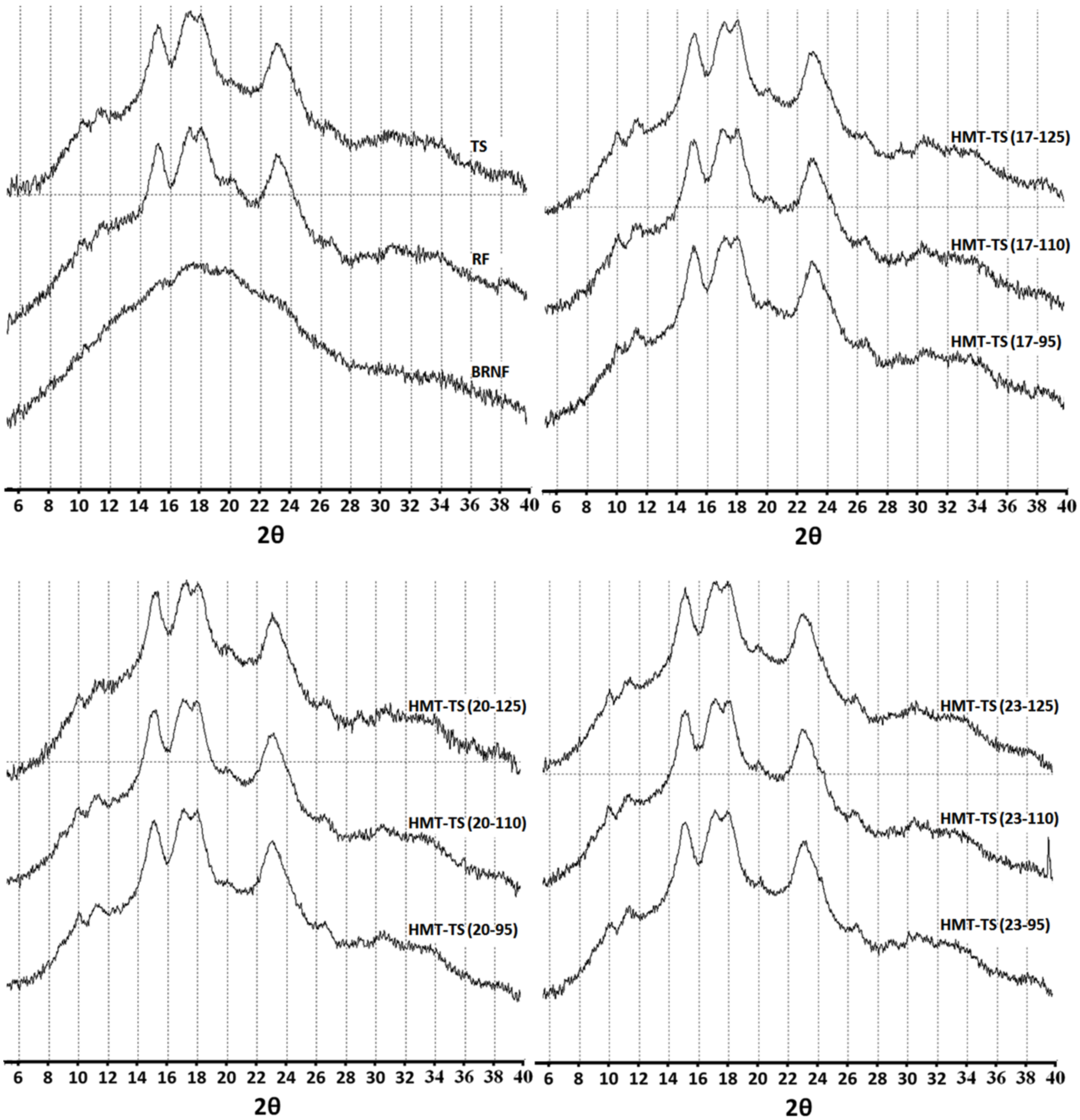

Figure 4.1 X-ray diffraction patterns of flours and starches. RF, rice flour; BRNF, broken rice noodle flour; TS, tapioca starch; HMT, heat-moisture treatment.

4.3 Thermal properties

Gelatinization of RF showed two overlapped peaks (Figure 4.2 and Table 4.4) at the temperature range of 60–90 °C suggesting that the rice flour sample contained more than one rice varieties with distinct granular thermal stabilities (Hagenimana et al., 2005). In addition, multi-modal melting profile of minor endothermic peaks was detected on the DSC trace as well (Figure 4.2). These endothermic transitions within high temperature region (100–150 °C) were representative of the melting of amylose-lipid (AM-L) complexes which displayed different degrees of helical chain organization in their solid state (Biliaderis and Seneviratne, 1990a, b; Czuchajowska et al., 1991; Biliaderis et al., 1993; Karkalas et al., 1995).

Table 4.4 Thermal properties of rice and broken rice noodle flours

Sample	Thermal behaviors		Endothermic transition parameters				
			T_o (°C)	T_p (°C)	T_c (°C)	ΔH (J/g)	ΔT (°C)
RF	Heating	Gelatinization	65.20	73.00 80.86	86.60	13.19	21.40
		AM-L complex (1st peak)	93.63	100.20	106.44	0.81	12.81
		AM-L complex (2nd peak)		117.16			
		AM-L complex (3rd peak)		131.61			
	Reheating	Retrogradation	50.80	61.42	70.56	4.24	19.76
		AM-L complex (1st peak)	94.10	101.31	108.06	1.43	13.97
BRNF	Heating	Retrograded amylopectin	51.42	59.34	69.37	1.80	17.96
		AM-L complex (1st peak)	93.36	101.16	108.57	0.93	15.21
		AM-L complex (2nd peak)		124.53			
		AM-L complex (3rd peak)		131.07			
	Reheating	Retrograded amylopectin	50.71	60.57	69.70	3.13	19.00
		AM-L complex (1st peak)	95.79	101.47	109.55	1.04	13.76

RF, rice flour; BRNF, broken rice noodle flour; AM-L, amylose-lipid; T_o, onset temperature; T_p, peak temperature; T_c, conclusion temperature; ΔH, endothermic transition enthalpy; ΔT, endothermic transition temperature range

After storage of 7 days at 4 °C, reheating of RF and water mixture exhibited typical thermal transition related to starch retrogradation (Figure 4.2). A single endothermic peak with lesser melting enthalpy appeared in low temperature region (50–70 °C), which represented the thermal transition of recrystallized amylopectin. Minor endothermic peaks present at the same high temperature area (100–150 °C, Figure 4.2) as in gelatinization denoted the melting of AM-L complexes; differences in the thermal transitional characteristics of AM-L complexes from those of RF gelatinization could be attributed to the recrystallization process.

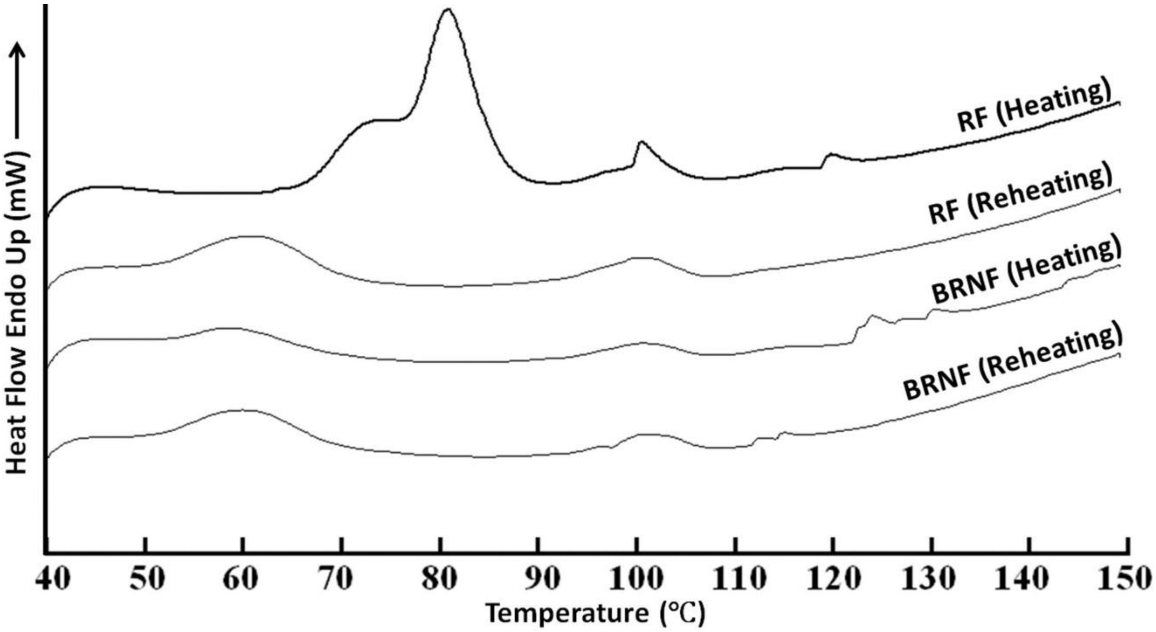

Figure 4.2 DSC thermograms of RF (rice flour) and BRNF (broken rice noodle flour).

Upon heating, thermoanalysis of BRNF exhibited thermal characteristics different from those of RF, expressed as one single endothermic peak occurring at low temperature area (50–70 °C) (Figure 4.2 and Table 4.4). Deduced from the comparable thermograms of RF retrogradation (reheating) and BRNF gelatinization (heating) that rice noodle making facilitated complete gelatinization and imparted retrogradation characteristics to BRNF.

Accordingly, heating of BRNF would show thermal behaviors related to recrystallized starch. Different from the thermally irreversible fraction of recrystallized amylose, recrystallization of amylopectin has been shown to be thermally reversible (<100 °C) (Gudmundsson, 1994). The first peak on the DSC trace of BRNF (heating) was, therefore, the melting of recrystallized amylopectin (Sievert and Pomeranz, 1990; Cui and Oates, 1997). Minor melting peaks on the DSC thermal curve of BRNF (heating) appeared in high temperature range (100–150 °C) almost same as those of RF (Figure 4.2). Similarly, these endothermic transitions would correspond to the melting of inclusion complexes of AM-L formed during noodle making.

BRNF was obtained by dehydrating and powdering the freshly cut noodles which essentially were highly concentrated gels. Noodle sheets were pre-dried before they were cooled to strengthen the firmness and cut into strips. Dehydration of freshly cut noodles would effectively hinder or even cease the retrogradation process as the moisture content was lowered due to drying (Gudmundsson, 1994), which caused insufficient recrystallization of amylopectin in BRNF. However, under the presence of thermal energy and moisture in DSC test, water would penetrate into the structure of BRNF resulting in the melting of retrograded crystallites, and water within the BRNF mixture after DSC test would perform as plasticizer again to initiate the recrystallization of amylopectin in BRNF during refrigeration. Due to its thermal reversibility, melting transition of recrystallized amylopectin in BRNF after storage (7 days at 4 °C) was detected on reheating (Figure 4.2). In addition, as the mixture was refrigerated, resultant increase in the melting enthalpy of recrystallized amylopectin was also observed on reheating (Figure 4.2) indicating that further recrystallization or crystal growth of recrystallized amylopectin occurred during refrigeration, which was consistent with reported findings (Cui and Oates, 1997). Dissociation of AM-L complexes was noticed on reheating as well (Figure 4.2); differences with regard to their melting transitions might be ascribed to the changes in the complexing process during recrystallization.

Upon gelatinization, endothermic transitions of HMT-TS with a shift toward higher temperatures than that of TS were observed (Figure 4.3 and Table 4.5), and this showed consistency with the results of HMT reported by other researchers (Donovan et al., 1983; Hoover and Vasanthan, 1994; Hoover and Manuel, 1996a, b). Amorphous regions within starch granule have been shown influence on starch gelatinization, attributable to the destabilization effect on crystallite melting (Donovan, 1979; Gunaratne and Hoover, 2002). Crystallite reorientation and starch chain interactions induced by HMT would restrict the swelling and mobility of starch chains in amorphous regions causing higher temperature for endothermic transitions (Donovan et al., 1983; Hoover and Vasanthan, 1994; Hoover and Manuel, 1996a, b). Degradation effect of HMT on amylopectin molecules has also been reported (Lu, Chen, and Lii., 1996) that degradation of weak structures in amylopectin crystallites during HMT resulted in more thermally stable amylopectin crystallites which required higher temperature to gelatinize (Miyoshi, 2002). Furthermore, gelatinization temperature of HMT-TS increased as the modification temperature of HMT increased at each moisture level, and vice versa (Table 4.5). This indicated that both high temperature and high moisture content during HMT promoted the recrystallization of starch granules.

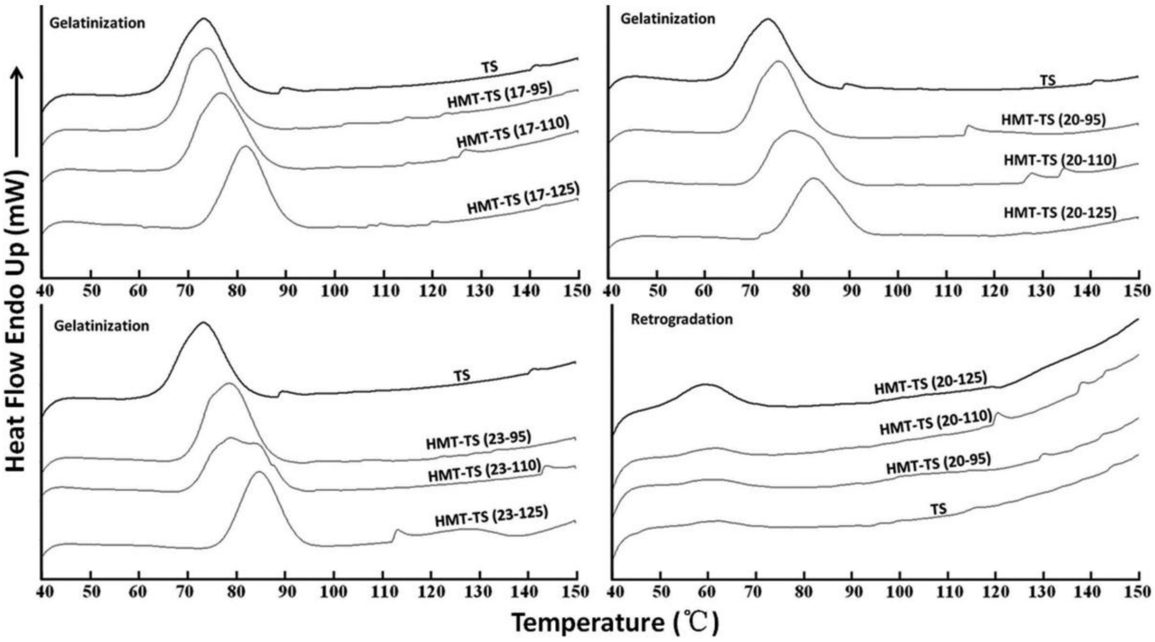

Figure 4.3 DSC thermograms of TS (tapioca starch) and HMT-TS (heat-moisture treated tapioca starch).

Table 4.5 Thermal properties of native and heat-moisture treated tapioca starches

Sample	DSC gelatinization properties					ΔH_R (J/g)
	T_o (°C)	T_p (°C)	T_c (°C)	ΔH_G (J/g)	ΔT_G (°C)	
TS	64.26 ± 0.05f	73.14 ± 0.11e	81.78 ± 0.18f	14.66 ± 0.86a	17.53 ± 0.23bc	0.40 ± 0.04g
HMT-TS (17-95)	66.42 ± 0.35e	74.05 ± 0.57e	82.42 ± 0.64f	14.47 ± 0.33a	16.00 ± 0.52ef	0.59 ± 0.05fg
HMT-TS (17-110)	68.04 ± 0.45d	76.19 ± 0.56d	85.31 ± 1.39e	14.12 ± 0.41a	17.27 ± 0.96bcd	0.66 ± 0.21ef
HMT-TS (17-125)	74.10 ± 0.58b	82.09 ± 0.35b	90.24 ± 0.32b	13.65 ± 0.24a	16.14 ± 0.34e	2.49 ± 0.05b
HMT-TS (20-95)	68.29 ± 0.84d	76.14 ± 0.92d	84.66 ± 1.13e	14.44 ± 0.23a	16.38 ± 0.38de	0.85 ± 0.05de
HMT-TS (20-110)	71.01 ± 1.13c	79.31 ± 0.90c	89.14 ± 0.33c	13.93 ± 0.58a	18.13 ± 1.43ab	0.94 ± 0.01d
HMT-TS (20-125)	74.24 ± 0.62b	82.22 ± 0.60b	92.04 ± 0.13a	11.31 ± 1.19b	17.80 ± 0.51ab	2.79 ± 0.13a
HMT-TS (23-95)	70.58 ± 0.10c	78.94 ± 0.18c	87.22 ± 0.26d	13.95 ± 0.41a	16.64 ± 0.23cde	1.20 ± 0.13c
HMT-TS (23-110)	71.36 ± 0.11c	80.28 ± 2.12c	90.07 ± 0.19bc	11.32 ± 0.39b	18.71 ± 0.28a	1.26 ± 0.28c
HMT-TS (23-125)	77.27 ± 0.27a	84.71 ± 0.15a	92.27 ± 0.39a	10.76 ± 0.80b	15.00 ± 0.61f	2.86 ± 0.02a

TS, tapioca starch; HMT, heat-moisture treatment; T_o, onset temperature; T_p, peak temperature; T_c, conclusion temperature; ΔH_G, gelatinization enthalpy; ΔT_G, gelatinization temperature range; ΔH_R, retrogradation enthalpy.

Means followed by different letters in a column are significantly different at $P<0.05$.

Decrease in gelatinization enthalpy (ΔH_G) of TS on HMT could be explained by the disruption of double helices present in crystalline and amorphous regions, resulting in less number double helices unraveling and melting during gelatinization (Gunaratne and Hoover, 2002). Cooke and Gidley (1992) reported, further verified by Hoover and Vasanthan (1994), that gelatinization enthalpy primarily reflected the loss of double helical order rather than the loss of crystalline register. And this accounted for the decreased melting endothermic enthalpy in the presence of increased crystalline order of HMT-TS (Table 4.2 and Table 4.5). Effect of increase in the moisture level prevailing during HMT on facilitating the movement of double-helical chains influenced the disruption of double helices (Hoover and Vasanthan, 1994). Consequently, progressive decrease in ΔH_G with increase in moisture content as well as in heating temperature was also observed in our study (Table 4.5).

Similar to those of RF and BRNF, multiphasic endothermic transitions of HMT-TS were also observed on the DSC profiles (Figure 4.3). As a result of the low lipid content (0.1%) present in tapioca starch (Hoover, 2001); these minor but noticeable melting peaks might be the disordering of newly formed structures instead of AM-L complexes on HMT. Donovan et al. (1983) reported that co-crystallization between amylose and amylopectin could possibly occur on HMT, which would further manifest itself as decreased amylose leaching and suppressed granular swelling. As for the thermal stability of these chain interactions formed during hydrothermal treatments, amylose-amylose (AM-AM) was reported to be much stronger than amylose-amylopectin (AM-AMP) and amylopectin-amylopectin (AMP-AMP) (Chung et al., 2009b). Furthermore, phase transition of recrystallized amylose has been shown in the temperature range of 110–150 °C (Eberstein et al., 1980 cited in Donovan et al., 1983). Therefore, these minor endothermic transitions in high temperature region (100–150 °C) might be the dissociation of recrystallized starch chains, or cocrystallites, with different thermal stabilities due to varied degrees of chain association on HMT.

Gelatinization temperature range (ΔT_G) has been interpreted as the indication of homogeneity of ordered structures inside starch granules (Yuan et al., 1993). Differences in the ΔT_G among HMT-TS suggested varied homogeneity of crystallites within the starch granules (Gunaratne and Hoover, 2002). Both broadened and narrowed ΔT_G caused by HMT were observed in our study (Table 4.5). Increase in ΔT_G of starches on HMT has been reported by other researchers (Donovan et al., 1983; Hoover and Vasanthan, 1994; Hoover and Manuel, 1996a, b; Gunaratne and Hoover, 2002). Narrowed ΔT_G of starches on HMT, however, could be due to modification in these conditions benefiting the development of more homogeneous crystallites (Hoover et al., 1994). Similar results of decrease in ΔT_G on HMT were also observed for rice starch (Khunae et al., 2007).

The total enthalpy change for gelatinization (ΔH_G) was found decreased for all HMT-TS (Table 4.5), and the same effect of HMT on reducing gelatinization enthalpy has been reported for other starches (Hoover and Vasanthan, 1994; Gunaratne and Hoover, 2002; Adebowale, Afolabi, and Olu-Owolabi, 2005). The magnitude of decrease in ΔH_G was greater as TS was modified under high temperatures and/or high moisture levels (Table 4.5), implying that more penetration of water molecules into starch granules during HMT could cause more disruption of starch molecules resulting in less energy required to melt the remaining crystallites.

Retrogradation of TS, after storage for 7 days at 4 °C, displayed minute endothermic transition in the DSC thermogram (Figure 4.3). This could be attributed to the long unit chain length of TS amylopectin demanding extended period of time to recrystallize (Hoover, 2001). In contrast, HMT-TS displayed distinguishable phase transition peaks (Figure 4.3) indicating that HMT promoted the recrystallization process of starch chains facilitating the retrogradation of starch gels. Same effect of HMT on increasing starch retrogradation has also been reported (Lu et al., 1996; Miyoshi, 2002). Furthermore, at each temperature level of HMT, higher moisture contents during the modification resulted in larger melting enthalpy for retrogradation (ΔH_R), and vice versa (Table 4.5). The foregoing degradation effect of HMT on amylopectin structure, as well as the enhanced starch crystallinity and amylose interaction due to HMT (Hoover and Vasanthan, 1994), have been postulated to be responsible for the increase in melting enthalpy of retrograded starches on HMT.

4.4 Swelling power and solubility

It has been reported that factors influencing starch swelling properties included amylopectin structure, AM-L complexing (Tester and Morrison, 1990a, b; Morrison et al., 1993), starch chain interaction and phosphate content (Gunaratne and Hoover, 2002). Starch swelling has been proven to be mainly a property of amylopectin (Tester and Morrison, 1990a), and widely known is that starch crystallinity is associated with amylopectin. RF showed higher SP but lower solubility than those of TS (Table 4.6 and 4.7), which could be ascribed to the interplay of different degrees of starch crystallinity, AM-L complexing and phosphorous content (Gunaratne and Hoover, 2002).

Impact of HMT on the swelling properties of TS exhibited decrement in SP but increment in solubility (Table 4.7). Jacobs and Delcour (1998) reviewed the influence of hydrothermal treatments on physicochemical properties of starches that decreased SP and increased solubility of starches as a result of HMT have been reported. However, in our study trend in the change in SP and solubility of HMT-TS was not found between modification temperatures and moisture levels employed in HMT, as was in conformity with the findings by Eerlingen et al. (1996). Reduction in SP of HMT-TS was probably due to starch chain interactions induced by HMT, which would suppress the swelling of starch granules (Hoover and Vasanthan, 1994; Hoover and Manuel, 1996a, b); while crystallite disruption of starches on HMT was also reported (Lu et al., 1996; Miyoshi, 2002) and accordingly this would be responsible for the increase in solubility of HMT-TS.

Table 4.6 Swelling power, solubility and gel hardness of flours and starches

Sample	SP (g/g)	Solubility (%)	Gel hardness (g force)
RF	16.22 ± 0.57c	12.87 ± 0.56c	18.39 ± 0.81c
BRNF	8.59 ± 0.11d	25.62 ± 0.55a	13.67 ± 0.67d
CF	17.07 ± 0.90c	14.57 ± 0.71b	50.78 ± 3.48a
CTS	23.50 ± 1.33a	9.74 ± 0.40e	19.03 ± 0.78c
CF-CTS	20.46 ± 0.71b	11.74 ± 0.31d	32.53 ± 1.17b

RF, rice flour; BRNF, broken rice noodle flour; CF, corn flour; CTS, cross-linked tapioca starch; CF-CTS, mixture (1:1) of corn flour and cross-linked tapioca starch; SP, swelling power.

Means followed by different letters in a column are significantly different at $P<0.05$.

Table 4.7 Swelling power, solubility and gel hardness of native and heat-moisture treated tapioca starches

Sample	SP (g/g)	Solubility (%)	Gel hardness (g force)
TS	5.00 ± 0.24a	49.94 ± 0.67h	24.15 ± 1.24a
HMT-TS (17-95)	4.24 ± 0.23cd	70.46 ± 4.73g	16.28 ± 1.02cd
HMT-TS (17-110)	3.20 ± 0.18e	86.88 ± 2.28b	10.65 ± 0.50h
HMT-TS (17-125)	2.77 ± 0.14f	89.86 ± 1.42a	16.99 ± 1.05c
HMT-TS (20-95)	4.58 ± 0.35fb	74.43 ± 2.61f	14.83 ± 0.46e
HMT-TS (20-110)	4.11 ± 0.26d	82.94 ± 0.70c	11.60 ± 0.71g
HMT-TS (20-125)	4.33 ± 0.39bcd	79.39 ± 2.48de	19.31 ± 1.18b
HMT-TS (23-95)	4.26 ± 0.15cd	83.85 ± 1.02c	13.40 ± 0.33f
HMT-TS (23-110)	4.55 ± 0.24bc	80.38 ± 1.49d	11.96 ± 0.69g
HMT-TS (23-125)	4.46 ± 0.29bc	77.71 ± 1.57e	15.69 ± 0.83d

TS, tapioca starch; HMT, heat-moisture treatment; SP, swelling power.

Means followed by different letters in a column are significantly different at $P<0.05$.

4.5 Pasting characteristics

Pasting characteristics of samples were presented in Table 4.8 and Table 4.9. Compared with RF, the prominent feature of the pasting profile of BRNF was the absence of P_T with the pasting rising slowly to low PV, HPV and CPV (Figure 4.4). This may be because gelatinization during steaming in rice noodle making caused the disintegration of starch granules, which accounted for the missing of P_T and failure of BRNF to sufficiently absorb water and swell to develop high viscosities.

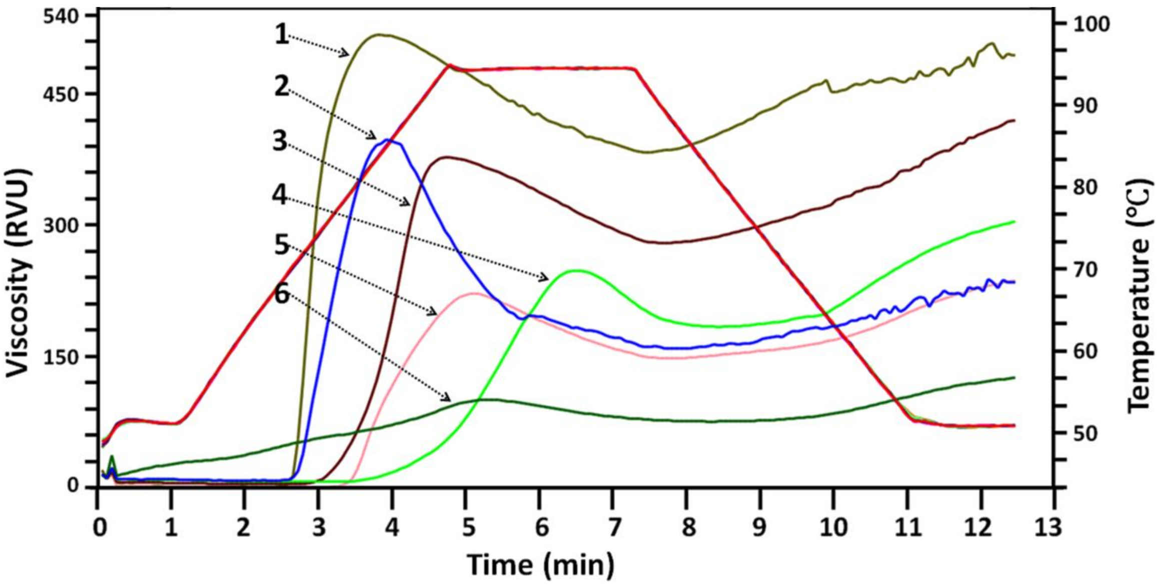

Figure 4.4 RVA viscograms of flours and starches. 1, CTS (cross-linked tapioca starch); 2, TS (tapioca starch); 3, CF-CTS [mixture (1:1) of corn flour and cross-linked tapioca starch]; 4, RF (rice flour); 5, CF (corn flour); 6, BRNF (broken rice noodle flour).

Table 4.8 RVA pasting characteristics of flours and starches

Sample	RVA pasting properties					
	PV (RVU)	HPV (RVU)	BD (RVU)	CPV (RVU)	SB (RVU)	P_T (°C)
RF	252.50 ± 0.93c	190.65 ± 1.95c	61.85 ± 1.79d	320.69 ± 0.97c	130.04 ± 1.95a	85.98 ± 0.03a
BRNF	100.50 ± 2.88d	75.60 ± 3.22e	24.90 ± 0.39e	122.06 ± 0.75e	46.46 ± 3.49d	–
CF	223.29 ± 2.77c	146.58 ± 0.83d	76.71 ± 1.94c	237.88 ± 2.06d	91.29 ± 1.23c	77.98 ± 0.04b
CTS	524.08 ± 2.24a	385.54 ± 0.18a	138.54 ± 2.42a	498.46 ± 0.88a	112.91 ± 0.71b	68.85 ± 0.64d
CF-CTS	380.12 ± 0.18b	280.09 ± 0.59b	100.04 ± 0.76b	422.59 ± 0.47b	42.46 ± 0.65d	73.98 ± 0.04c

RF, rice flour; BRNF, broken rice noodle flour; CF, corn flour; CTS, cross-linked tapioca starch; CF-CTS, mixture (1:1) of corn flour and cross-linked tapioca starch; PV, peak viscosity; HPV, hot paste viscosity; BD, breakdown; CPV, cold paste viscosity; SB, setback; P_T, pasting temperature.

Means followed by different letters in a column are significantly different at $P<0.05$

Table 4.9 RVA pasting characteristics of native and heat-moisture treated tapioca starches

Sample	RVA pasting properties					
	PV (RVU)	HPV (RVU)	BD (RVU)	CPV (RVU)	SB (RVU)	P_T (°C)
TS	399.42 ± 1.13a	156.73 ± 0.79b	242.69 ± 0.76a	234.73 ± 4.33b	78.00 ± 5.03a	69.44 ± 0.35h
HMT-TS (17-95)	355.21 ± 28.64b	169.21 ± 3.85a	186.00 ± 24.83b	227.44 ± 3.59b	58.23 ± 0.86b	70.95 ± 0.73g
HMT-TS (17-110)	191.48 ± 19.56d	100.64 ± 5.42c	90.83 ± 14.15e	141.42 ± 10.20c	40.77 ± 4.79c	72.00 ± 0.52f
HMT-TS (17-125)	20.29 ± 3.21g	9.94 ± 1.63e	10.35 ± 1.62g	14.06 ± 1.91e	4.13 ± 0.40e	81.64 ± 0.48c
HMT-TS (20-95)	318.60 ± 28.31c	168.23 ± 7.93a	150.38 ± 20.40c	233.04 ± 1.98b	64.82 ± 9.39b	71.76 ± 0.69f
HMT-TS (20-110)	134.44 ± 3.06f	81.54 ± 2.64d	52.90 ± 0.44f	114.27 ± 2.28d	32.73 ± 0.41d	74.64 ± 0.39e
HMT-TS (20-125)	22.02 ± 3.09g	12.07 ± 1.22e	9.95 ± 1.92g	17.70 ± 1.44e	5.63 ± 0.30e	82.58 ± 0.22b
HMT-TS (23-95)	304.41 ± 22.60c	172.64 ± 7.67a	131.77 ± 14.98d	246.88 ± 1.97a	74.23 ± 9.52a	74.24 ± 0.41e
HMT-TS (23-110)	166.23 ± 19.80e	104.34 ± 11.56c	61.90 ± 8.27f	150.27 ± 16.53c	45.93 ± 4.99c	75.55 ± 0.61d
HMT-TS (23-125)	12.09 ± 1.20g	6.96 ± 1.05e	5.13 ± 0.20g	11.08 ± 1.24e	4.12 ± 0.38e	83.52 ± 0.34a

TS, tapioca starch; HMT, heat-moisture treatment; PV, peak viscosity; HPV, hot paste viscosity; BD, breakdown; CPV, cold paste viscosity; SB, setback; P_T, pasting temperature. Means followed by different letters in a column are significantly different at $P<0.05$.

In contrast, TS showed higher PV, BD and SB but lower P_T than HMT-TS; however, HMT under some conditions increased the thermal stability of starch granules, expressed as the higher HPV and lower BD (Figure 4.5 and Table 4.9). HMT has also caused increase or decrease in CPV for TS. Results from our study suggested that the general effects of HMT on the pasting properties of TS were an increase in P_T, suppression in PV and increase or decrease in CPV, which were in correspondence with those of HMT reported on cassava starch (Lorenz and Kulp, 1982, Abraham, 1993). In addition, at each moisture level, decrement in viscosities but increment in P_T was observed as the temperature applied in HMT increased (Table 4.9). Restricted starch swelling and starch chain interactions occurred during HMT would cause higher P_T and resultant suppression in the development of peak viscosity. Furthermore, the magnitude of increase in P_T was consistent with that in DSC gelatinization temperatures indicating that severer HMT conditions would have greater effect on the interplay of starch chain interactions and starch crystallinity. HMT at 95 °C increased the HPV of TS exhibiting stronger thermal stability of swollen granules under high temperature shearing. This suggested that HMT at low modification temperature (i.e. 95 °C) contributed less to crystallite disruption, and starch chain interactions would then increase the granule rigidity resulting in higher thermal stability than those modified at 110 °C and 125 °C. Higher BD of TS than those of HMT-TS indicated that cooked tapioca starch granules had greater susceptibility to disintegration, which further verified the effect of HMT on increasing the rigidity of starch granules. CPV has been shown to largely depend on the retrogradation tendency of solubilized amylose upon cooling (Bhattacharya et al., 1999). Low CPV of HMT-TS (Table 4.9) could be, therefore, attributed to the decrease in amylose leaching on HMT (Hoover and Vasanthan, 1994; Hoover and Manuel, 1996a, b; Gunaratne and Hoover, 2002), rendering less solubilized amylose during pasting. SB measured the degree of retrogradation of cooked starches under cooling, and from our results the reduction in SB of HMT-TS varied significantly from modification conditions (Table 4.9). As seen from the measure condition of SB (cooling from high temperature to 50 °C and holding for short time), the value of SB was , therefore, only reflective of the formation of amylose network due to the recrystallization of amylose. In contrast, ΔH_R in DSC test measured the melting enthalpy of recrystallized amylopectin reassociated under low temperature (4 °C) for a long time (7 days), which was also responsible for the long-term changes in starch retrogradation (Gudmundsson, 1994).

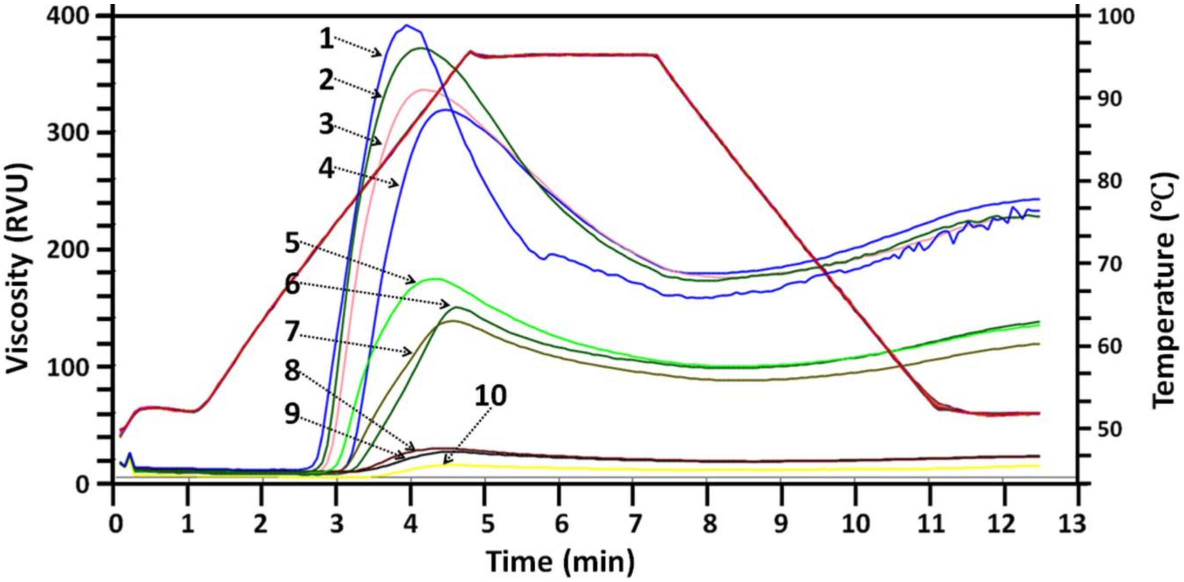

Figure 4.5 RVA viscograms of native and heat-moisture treated tapioca starches. 1, TS (tapioca starch); 2, HMT-TS (17-95); 3, HMT-TS (20-95); 4, HMT-TS (23-95); 5, HMT-TS (17-110); 6, HMT-TS (20-110); 7, HMT-TS (23-110); 8, HMT-TS (17-125); 9, HMT-TS (20-125); 10, HMT-TS (23-125).

4.6 Gel texture

TS gel in our study showed higher gel hardness than those of RF and BRNF (Table 4.6 and 4.7). Gel hardness of HMT-TS determined by the same test profile gave smaller values than that of TS (Table 4.7 and Figure 4.6) implying the impact of HMT on starch gel texture. Besides, gel hardness of TS modified at 125 °C had the highest value, followed by modification at 95 °C and 110 °C, which could be due to the different extents of starch chain interactions induced by HMT causing varied junction zones in the continuous phase of the gels (Hormdok and Noomhorm, 2007). Influence of increase in modification severity was also observed from the more solid-like gel textures of TS modified at higher temperatures, especially at 125 °C, manifested as the appearance of fracture prior to the first peak (Figure 4.6). Since gel hardness of starch has been shown closely correlated with the textural qualities of starch noodles (Bhattacharya et al., 1999; Yoenyongbuddhagal and Noohorm, 2002; Chen et al., 2002; Hormdok and Noomhorm, 2007), TS modified at 125 °C with 20 g/100 g moisture level having the highest gel hardness was applied to rice noodle making.

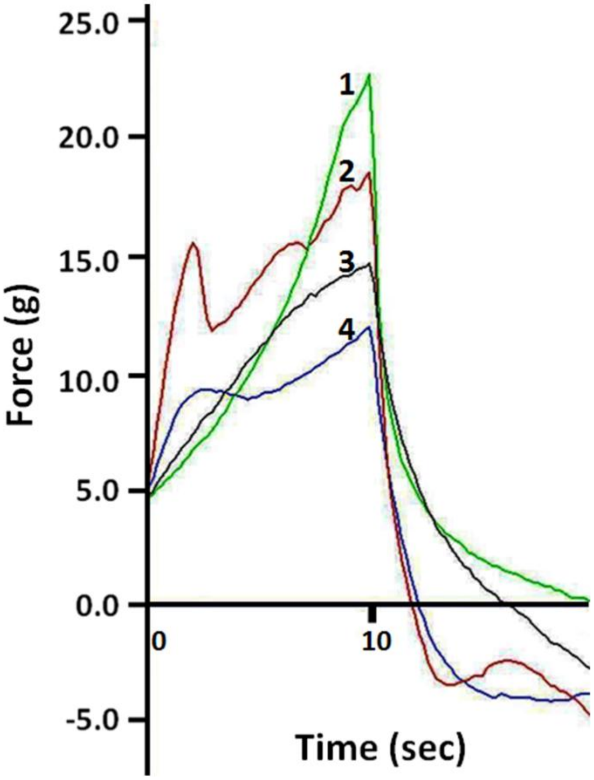

Figure 4.6 Gel harness of TS (tapioca starch) and HMT-TS (20-95, 110, 125). 1, 2, 3 and 4 represent TS, HMT-TS (20-125), HMT-TS (20-95) and HMT-TS (20-110) respectively.

4.7 Properties of flour combinations

Commercially, rice flours from fresh rice or low-amylose rice varieties are not practically suitable for rice noodle making. In addition to aging of rice (Hormdok and Noomhorm, 2007), RF blending with high-amylose starches (Wang et al., 2000; Sandhu et al., 2010; Yadav et al., 2011) and chemically modified starches (Charutigon et al., 2008) has been used in rice noodle making. Simulation of mung bean starch by mixing high-amylose maize starch and cross-linked tapioca starch to make clear noodles has been conducted (Kasemsuwan et al., 1998). In our study, corn flour (CF) (20.2 g amylose/100 g) was also provided by the noodle company, and the mixture (1:1) of CF and CTS was used in rice noodle making.

4.7.1 Swelling power and solubility

SP and solubility of RF-starch blends in the presence of TS, HMT-TS (20-125) and CTS lay between those of the single RF and blending counterparts, and the swelling power and solubility had values approaching toward those of RF, which was due to the higher proportion of RF incorporated in the blends. Nevertheless, blend of RF and CF-CTS had SP and solubility lower and higher than those of the each component, respectively (Table 4.10). In general, practical SP and solubility of RF-starch blends corresponded to the theoretical values calculated according to the weight fraction of each individual component in the blends, implying that blended counterparts swelled independently in the mixtures. Although, on account of its low SP and high solubility, partial substitution of BRNF for RF in RF-starch blend systems caused decrease in SP and increase in solubility, the composition-swelling relationship of RF/BRNF-starch blends still showed an additive type.

Table 4.10 Swelling power, solubility and gel hardness of flour combinations

Flour combinations	SP (g/g)	Solubility (%)	Gel hardness (g force)
RF/TS (30%:10%)	16.05 ± 1.80abc	24.59 ± 1.18c	17.14 ± 0.48d
RF/BRNF/TS (25%:5%:10%)	14.47 ± 1.78cd	29.09 ± 0.84b	15.59 ± 0.54f
RF/HMT-TS (30%:10%)	13.23 ± 0.79de	29.06 ± 0.04b	16.30 ± 0.77e
RF/BRNF/HMT-TS (25%:5%:10%)	12.43 ± 0.31e	31.34 ± 1.46a	15.78 ± 0.47ef
RF/CTS (30%:10%)	17.24 ± 0.31a	12.33 ± 0.98e	20.16 ± 0.22a
RF/BRNF/CTS (25%:5%:10%)	16.52 ± 0.47ab	14.78 ± 0.48d	18.23 ± 0.64c
RF/CF-CTS (30%:10%)	15.58 ± 1.24bc	14.52 ± 0.68d	19.08 ± 0.14b
RF/BRNF/CF-CTS (25%:5%:10%)	15.03 ± 0.71bc	15.42 ± 1.47d	18.99 ± 0.49b

RF, rice flour; BRNF, broken rice noodle flour; TS, tapioca starch; HMT, heat-moisture treatment; CTS, cross-linked tapioca starch; CF, corn flour; CF-CTS, mixture (1:1) of corn flour and cross-linked tapioca starch; SP, swelling power; PV, peak viscosity; HPV, hot paste viscosity; BD, breakdown; CPV, cold paste viscosity; SB, setback; P_T, pasting temperature.

Means followed by different letters in a column are significantly different at *P*<0.05.

4.7.2 Pasting characteristics

Pasting characteristics of flour combinations were presented in Table 4.11 and Figure 4.7. In general, pasting viscosities and P_T of RF-starch blends fell into the ranges of those of each individual component in their mixtures. Although RF-starch blend system showed RVA pasting viscosities more or less than those of single RF, replacement of TS, HMT-TS (20-125), CTS and CF-CTS for RF resulted in lower P_T than that of RF. An inexact additive composition-pasting relationship was noticed for blends of RF/TS, RF/CTS and RF/CF-CTS, evidenced by the slightly higher or lower practical pasting parameters than the sum of outputs of each component. Whereas, blended RF and HMT-TS (20-125) had practical pasting viscosities and P_T much lower than those of the theoretical values. Such inconsistency in the pasting characteristics of RF-starch blends could be attributed to the greater interactions between different molecules, granules, swollen granules, and granule fragments than those between the same from a single starch (Obanni and BeMiller, 1997). Due to the absence of P_T and low pasting viscosities, the addition of BRNF to RF-starch blends caused decrement in RVA pasting parameters. Moreover, the above composition-pasting relationships were not impacted by the incorporation of BRNF into RF-starch blends, implying that no further significant mutual interference between BRNF and mixing components occurred during pasting.

Table 4.11 RVA pasting characteristics of flour combinations

Flour combinations	RVA pasting properties					
	PV (RVU)	HPV (RVU)	BD (RVU)	CPV (RVU)	SB (RVU)	P_T (°C)
RF/TS (30%:10%)	262.75 ± 1.41c	169.21 ± 0.06d	93.54 ± 1.47a	278.30 ± 0.18e	109.09 ± 0.23c	78.03 ± 0.04e
RF/BRNF/TS (25%:5%:10%)	253.88 ± 2.41d	160.50 ± 2.47e	93.38 ± 0.06a	262.46 ± 2.06f	101.96 ± 0.41d	74.80 ± 0.00f
RF/HMT-TS (30%:10%)	113.04 ± 0.65e	86.63 ± 1.00f	26.42 ± 0.35c	150.80 ± 0.18g	64.17 ± 0.82e	81.18 ± 0.04a
RF/BRNF/HMT-TS (25%:5%:10%)	96.38 ± 0.29f	72.46 ± 0.41g	23.92 ± 0.71c	131.46 ± 0.18h	59.00 ± 0.23f	80.70 ± 0.57ab
RF/CTS (30%:10%)	303.25 ± 0.35a	226.54 ± 0.53a	76.71 ± 0.88b	377.84 ± 0.47a	151.29 ± 1.00b	81.10 ± 0.07a
RF/BRNF/CTS (25%:5%:10%)	291.25 ± 3.30b	216.00 ± 4.24b	75.25 ± 0.95b	368.25 ± 3.07b	152.25 ± 1.17ab	80.33 ± 0.04bc
RF/CF-CTS (30%:10%)	262.75 ± 0.95c	186.12 ± 2.76c	76.63 ± 3.71b	340.91 ± 4.12c	154.79 ± 1.36a	79.98 ± 0.60cd
RF/BRNF/CF-CTS (25%:5%:10%)	253.30 ± 5.48d	181.83 ± 0.47c	71.46 ± 5.01b	331.21 ± 3.59d	149.38 ± 3.12b	79.58 ± 0.04d

RF, rice flour; BRNF, broken rice noodle flour; TS, tapioca starch; HMT, heat-moisture treatment; CTS, cross-linked tapioca starch; CF, corn flour; CF-CTS, mixture (1:1) of corn flour and cross-linked tapioca starch; SP, swelling power; PV, peak viscosity; HPV, hot paste viscosity; BD, breakdown; CPV, cold paste viscosity; SB, setback; P_T, pasting temperature.

Means followed by different letters in a column are significantly different at $P<0.05$.

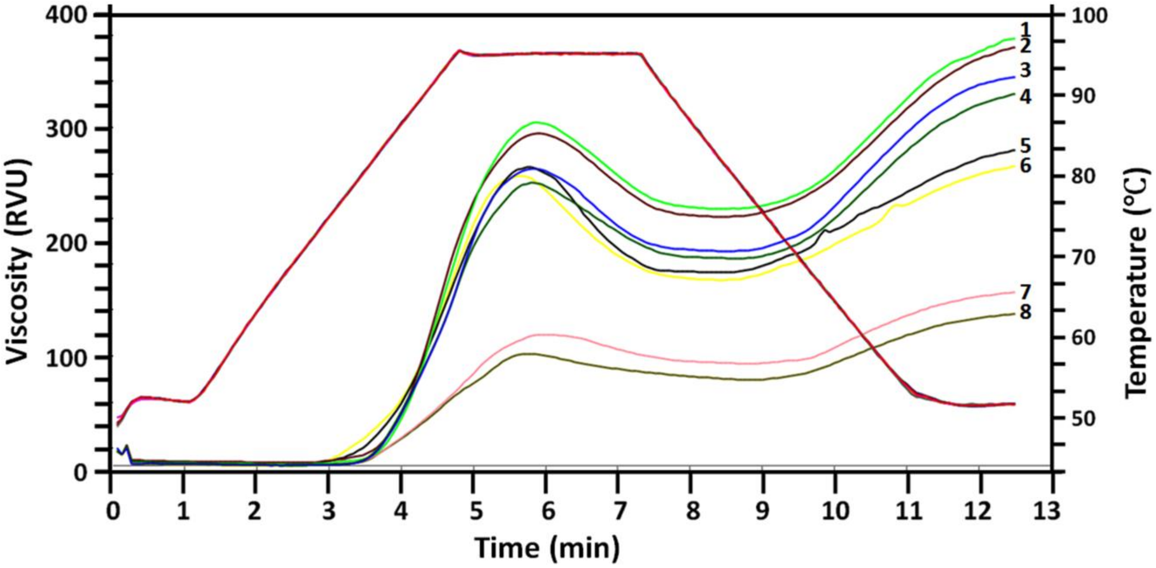

Figure 4.7 RVA viscograms of flours combinations. 1, RF/CTS (30%:10%); 2, RF/BRNF/CTS (25%:5%:10%); 3, RF/CF-CTS (30%:10%); 4, RF/BRNF/CF-CTS (25%:5%:10%); 5, RF/TS (30%:10%); 6, RF/BRNF/TS (25%:5%:10%); 7, RF/HMT-TS (30%:10%); 8, RF/BRNF/HMT-TS (25%:5%:10%).

4.7.3 Gel texture

Similar to the pasting characteristics, gel strength of flour combinations showed great dependence upon the blending components. Prior to mixing with RF, CF-CTS exhibited the highest gel hardness followed by TS, HMT-TS (20-125) and CTS (Table 4.6 and 4.7). Replacing of these counterparts for RF influenced the gel strength of RF-starch mixtures; blend of RF/CTS displayed the highest gel hardness followed by those of RF/CF-CTS, RF/TS and RF/HMT-TS (20-125) (Table 4.10). Among RF-starch blends, only RF/CF-CTS mixture had gel hardness falling between those of RF and CF-CTS, and the value of gel strength approached toward that of RF. Both blends of RF/TS and RF/HMT-TS (20-125) had gel hardness lower than those of either single component; whereas, RF blending with CTS gave gel hardness higher than those of the single RF and CTS. Discrepancy between the practical gel hardness of RF-starch blends and the sum of outputs of each component denoted an inexact additive retrogradation behavior. Blends of various starches with resulting decrease in gel hardness or starch retrogradation have been reported, which was attributed to the greater differences in granular morphology and associations of leached materials from blending components influencing starch recrystallization and resultant gel structure (Obanni and BeMiller, 1997; Puncha-arnon et al., 2008). Results from our study also suggested that the occurrence of molecular interactions within starch blends could exert influence on strengthening or weakening the gel texture of corresponding starch mixtures. Although the nonadditive composition-gelling nature of RF-starch blends was not affected by the further incorporation of BRNF, decrease in gel hardness was observed upon the partial substitution of BRNF due to its poor gelling property (Table 4.10).

4.8 Effect of various flour combinations on noodle qualities

Composite rice noodles were prepared from flour combinations and their cooking and textural qualities were compared with commercial one (Table 4.12). Among all the mixtures, flour combinations containing HMT-TS (20-125) were unable to form noodle sheets that were strong enough to be scraped out of the tray. After steaming, noodle sheets of HMT-TS (20-125) combinations adhered to the tray bottom causing broken pieces when attempted to pull the intact noodle sheets out. Noodle sheet formed by steaming flour slurry (40 g/100 g) was considered to be a highly concentrated starch paste. And from the results of pasting test, HMT-TS (20-125) combinations showed poor pasting capacity and short paste texture, which led to the weak structure of noodle sheets from the combination and failed to make noodles.

4.8.1 Cooking quality of dried noodles

Desired cooking qualities of rice noodle were manifested in short cooking time, little solid loss, and sufficient rehydration with minimum surface stickiness (Hormdok and Noomhorm, 2007; Fu, 2008). Optimum cooking time of composite rice noodles appeared almost the same as that of commercial one (approx. 4 min). Cooking qualities of cooking loss and rehydration ratio of all noodle samples were compared (Table 4.12). Commercial rice noodle exhibited the best cooking qualities expressed as low cooking loss and high cooking weight. Composite rice noodles, however, displayed cooking qualities inferior to those of commercial one evidenced by the higher cooking loss and lower rehydration ratio. Generally, for each RF-starch blend system the incorporation of BRNF caused decrease in both cooking loss and rehydration ratio; whereas the difference was not significant ($P > 0.05$). Among composite rice noodles, those prepared from TS combinations (i.e. RF/TS and RF/BRNF/TS) had the highest cooking loss, which might be due to the high solubility of TS combinations.

4.8.2 Texture quality of cooked noodles

Textural qualities of composite and commercial rice noodles were presented in Table 4.12. Rice noodles prepared from CTS combinations (i.e. RF/CTS and RF/BRNF/CTS) showed hardness lower than that of commercial one; while the rest composite rice noodles were determined to demonstrate higher firmness. Differences in noodle hardness between commercial rice noodle and those prepared from RF/BRNF/TS and RF/BRNF/CF-CTS were found not significant ($P > 0.05$). Effect of further addition of BRNF to RF-starch blends has been shown to be decrease in noodle firmness.

Composite rice noodles showed to be more adhesive than the commercial one ($P < 0.05$). Similarly, further incorporation of BRNF into RF-starch blends caused decrease in adhesiveness for noodles, among which the one prepared from RF/BRNF/TS showed the lowest adhesiveness closest to that of commercial one.

In addition to hardness and adhesiveness, chewiness is another important factor affecting the mouthfeel of noodles. Chewiness of composite rice noodles was lower than that of commercial one ($P < 0.05$). Chewiness in TPA test could be influenced by hardness, cohesiveness and springiness (i.e. chewiness = hardness × cohesiveness × springiness) of samples. Composite rice noodles with higher or lower hardness than that of commercial one showed less chewy ($P < 0.05$), which could be ascribed to the differences in cohesiveness and springiness among composite and commercial noodle samples. Moreover, composite rice noodles prepared from CTS combinations (with lower hardness) showed higher chewiness than those prepared from CF-CTS combinations, which was due to the substitution of CF for CTS causing increase in cohesiveness and springiness. With the addition of BRNF to RF-starch blends, decrease in chewiness was observed for noodles prepared from all flour combinations.

The highest tensile strength of commercial rice noodle ($P < 0.05$) indicated its greater resistance to tearing force than composite ones. Among composite rice noodles, those prepared from TS combinations exhibited the highest tensile strength followed by those prepared from CF-CTS and CTS combinations. Substitution of BRNF for RF in RF-starch blends decreased the tensile strength of corresponding noodles.

Table 4.12 Cooking and texture qualities of rice noodles

Noodles	Cooking quality		Textural quality			
	Cooking loss (%)	Rehydration ratio (%)	Hardness (g force)	Adhesiveness (g s)	Chewiness (g mm)	Tensile strength (g force)
RF/TS (30%:10%)	9.43 ± 0.42a	213.07 ± 5.14bc	3801.70 ± 100.63a	76.23 ± 5.75bc	2069.26 ± 115.57b	20.40 ± 1.81b
RF/BRNF/TS (25%:5%:10%)	8.55 ± 0.39ab	211.14 ± 2.16c	3104.20 ± 122.39c	69.67 ± 10.91c	1963.54 ± 47.65c	20.16 ± 2.92b
RF/CTS (30%:10%)	6.65 ± 0.81cd	219.63 ± 2.94b	2869.00 ± 93.48d	79.30 ± 14.44abc	1934.66 ± 73.84c	14.50 ± 1.01d
RF/BRNF/CTS (25%:5%:10%)	7.67 ± 0.25bc	209.99 ± 0.71c	2369.50 ± 144.21e	72.10 ± 10.24c	1897.81 ± 78.99cd	14.03 ± 1.20d
RF/CF-CTS (30%:10%)	6.62 ± 1.06cd	219.09 ± 5.02b	3636.90 ± 100.40b	89.23 ± 11.40a	1838.63 ± 76.86d	17.24 ± 1.41c
RF/BRNF/CF-CTS (25%:5%:10%)	6.38 ± 0.36d	219.09 ± 3.41b	3073.60 ± 144.06c	86.22 ± 8.35ab	1592.85 ± 66.48e	14.95 ± 0.94d
Commercial Noodle	5.02 ± 0.36e	231.24 ± 5.37a	3044.40 ± 82.92c	56.17 ± 7.45d	2206.53 ± 53.15a	23.47 ± 2.89a

RF, rice flour; BRNF, broken rice noodle flour; TS, tapioca starch; CTS, cross-linked tapioca starch; CF, corn flour; CF-CTS, mixture (1:1) of corn flour and cross-linked tapioca starch. Means followed by different letters in a column are significantly different at $P<0.05$.

Table 4.13 Sensory attributes scores of plainly boiled noodles

Sample	Color	Clarity	Firmness	Flavor	Acceptability
Commercial Noodle	2.4 ± 0.9a	2.5 ± 0.9b	2.3 ± 0.8b	2.7 ± 1.0ab	3.1 ± 1.0a
RF/BRNF/TS (25%:5%:10%)	2.3 ± 1.1a	3.2 ± 1.0a	2.9 ± 1.2ab	2.5 ± 0.9b	2.2 ± 0.9b
RF/BRNF/CTS (25%:5%:10%)	2.3 ± 0.9a	3.2 ± 1.2a	3.1 ± 1.2a	2.6 ± 1.0b	2.3 ± 1.2b
RF/BRNF/CF-CTS (25%:5%:10%)	2.4 ± 1.0a	3.0 ± 1.4ab	2.5 ± 1.2b	3.2 ± 1.1a	2.9 ± 0.9a

RF, rice flour; BRNF, broken rice noodle flour; TS, tapioca starch; CTS, cross-linked tapioca starch; CF, corn flour; CF-CTS, mixture (1:1) of corn flour and cross-linked tapioca starch. Means followed by different letters in a column are significantly different at $P<0.05$.

Range of Scores: Color: 1-white to 5-yellow; Clarity: 1-opaque to 5-clear; Firmness: 1-firm to 5-soft; Flavor: 1- characteristic to 5-not characteristic; Acceptability: 1- very acceptable to 5-not acceptable

4.8.3 Sensory evaluation of cooked noodles

Sensory evaluation of composite rice noodles was conducted to noodles prepared from flour combinations containing BRNF i.e. RF/BRNF/TS, RF/BRNF/CTS and RF/BRNF/CF-CTS, which corresponded to the aim of this research utilizing BRNF to produce rice noodles with comparable qualities to commercial one. As listed in Table 4.13, color of composite noodles was not significantly different from that of commercial one, and flour combinations of TS or CTS produced noodles with whiter color than noodle prepared from RF/BRNF/CF-CTS and commercial one, which was due to the use of CF in both noodle makings. When clarity was compared between commercial noodle and composite ones, noodles prepared from RF/BRNF/TS and RF/BRNF/CTS displayed clearer than noodle prepared from RF/BRNF/CF-CTS and commercial one, and this could be also explained by the mixing of CF during noodle making.

Firmness of noodle samples followed the sequence of commercial noodle, noodles prepared from RF/BRNF/CF-CTS, RF/BRNF/TS and RF/BRNF/CTS, which was basically in agreement with the order of noodle hardness determined by the texture analyzer (Table 4.12). The above discrepancy between noodle TPA and sensory evaluation could be attributed to personal mouthfeel of panelists during sensory evaluation. Lower score of noodle flavor indicated a blander flavor. Seen from the results noodle prepared from RF/BRNF/TS showed the least characteristic flavor, while RF/BRNF/CF-CTS produced noodle having the most characteristics flavor. Use of CF and/or CTS in noodle making might be responsible for the flavor contribution.

Overall acceptability of all noodle samples exhibited significant difference ($P < 0.05$) between noodles prepared from RF/BRNF/TS, RF/BRNF/CTS and noodle produced from RF/BRNF/CF-CTS, and commercial one. All composite noodles surprisingly demonstrated higher overall acceptability than commercial one. The highest acceptability (lowest score) of RF/BRNF/TS noodle was accordingly ascribed to its whitest color, higher clarity, moderate hardness, and blandest flavor. General acceptability has furthermore shown that composite noodles prepared from flour combinations of RF/BRNF/TS, RF/BRNF/CTS and RF/BRNF/CF-CTS were comparable to commercial noodle.

CHAPTER V

CONCLUSION

5.1 CONCLUSIONS

The intention of reusing broken rice noodles in the form of broken rice noodle flour to make composite rice noodles was proposed in this study. Comparison of some physicochemical properties between native rice flour and broken rice noodle flour showed some significant differences. BRNF displayed both of higher total RS and thermostable RS contents than those of RF, while the X-ray diffraction crystallography of BRNF showed to be a pattern of amorphous materials expressed as much lower crystalline area and crystallinity. BRNF upon heating exhibited endothermic melting characteristics of retrograded starches instead of the native starch as demonstrated by RF, and both RF and BRNF gave melting endotherms of amylose-lipid complexes. Swelling power and solubility of BRNF were lower and higher than those of RF, respectively. Distinct pasting behaviors of BRNF were the absence of pasting temperature and low pasting viscosities in comparison with those of RF. Various conditions of HMT were applied to TS which largely altered some physicochemical properties and resultant noodle making applicability. HMT caused decrease in total RS content, but increase in thermostable RS and apparent amylose contents. No change in X-ray diffraction pattern of TS was observed on HMT, but increase in crystallinity due to HMT occurred. Increase in gelatinization temperatures and decrease in gelatinization enthalpy of TS on HMT were noticed, and the retrogradation enthalpy of TS was also increased by HMT. HMT-TS showed lower SP but higher solubility. Some pasting characteristics of TS related to noodle making applicability such as CPV and SB were reduced by HMT under certain conditions especially severe treatment i.e. high heating temperatures and/or high moisture levels. And the resultant gel hardness values of HMT-TS were smaller than that of TS.

Swelling, pasting and gelling properties of flour combinations were also compared to evaluate their noodle making applicability. Among all the flour blends, HMT-TS (20-125) combinations failed to be used for noodle making, which was attributed to the declined pasting properties (i.e. CPV and SB). Composite rice noodles prepared from different flour combinations displayed varied cooking and textural qualities. In terms of utilizing BRNF, composite rice noodles prepared from flour combinations of RF/BRNF/TS, RF/BRNF/CTS and RF/BRNF/CF-CTS had cooking and textural qualities close to those of commercial rice noodle. Amid all these three composite rice noodles, the one prepared from RF/BRNF/CF-CTS exhibited superior cooking qualities, but also highest adhesiveness, lowest chewiness and lesser tensile strength. Noodle from RF/BRNF/CTS showed moderate cooking qualities; however, its hardness and tensile strength were the lowest. Although it showed high cooking loss, noodle prepared from RF/BRNF/TS had textural qualities closest to those of commercial one, evidenced by its lowest adhesiveness, highest chewiness and tensile strength. Results of noodle cooking, texture, and sensory qualities showed that in terms of using BRNF to make noodles with comparable qualities, flour combination of RF/BRNF/TS would be the preferable one than the others. Furthermore, composite rice noodle prepared from RF/BRNF/TS was considered safer since the CTS blended in other two flour mixtures still required chemical modification.

Suggestion could be given to the future work for considering other pasting parameters such as CPV and SB of starches as prerequisites along with gel hardness for noodle making.

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APPENDICES

APPENDIX A

ANALYTICAL METHODS

A.1. Apparent amylose determination

100 mg of rice powder is put into a 100 ml volumetric flask and 1 ml of 95% ethanol and 9 ml of 1N sodium hydroxide are added. The contents are heated on a boiling water bath to gelatinize the starch. After cooling for one hour, distilled water is added and contents are mixed well.

Five ml of the starch solution is put in a 100-ml volumetric flask with a pipette. One ml of 1N acetic acid, 2 ml of iodine solution (0.2 g iodine and 2.0 g potassium iodine in 100 ml of aqueous solution) are added and volume is made up with distilled water. Contents are shaken well and let stand for 20 minutes. Absorbance of the solution is measured at 620 nm with a spectrophotometer. Amylose content is determined by using a conversion factor and the results are expressed on a dry weight basis.

For the standard curve 40 mg of potato amylose (Sigma Chemical Co. Inc.) are wet with 1 ml ethanol and 9 ml 1N sodium hydroxide. Heated for 5-10 minutes in a boiling water bath, cooled and made up to volume. 1, 2, 3, 4, 5 ml of solution are placed with a pipette in 100 ml volumetric flasks. The solution is acidified with 1N acetic acid (0.2, 0.4, 0.6, 0.8 and 1.0 ml, respectively) and treated as above. The absorbance values are plotted at 620 nm against the concentration of anhydrous amylose (mg) and the conversion factor is determined. The dilution factor of 20 for the samples is included in the conversion factor.

A.2. Megazyme® Resistant Starch Assay Procedure

(a) Hydrolysis and solubilization of non-resistant starch.

Accurately weigh a 100 ± 5 mg sample directly into each screw cap tube (Corning culture tube; 16 x 125 mm) and gently tap the tube to ensure that the sample falls to the bottom.

1. Add 4.0 mL of pancreatic α-amylase (10 mg/ml) containing AMG (3 U/mL) to each tube.
2. Tightly cap the tubes, mix them on a vortex mixer and attach them horizontally in a shaking water bath, aligned in the direction of motion.
3. Incubate tubes at 37 °C with continuous shaking (200 strokes/min) for exactly 16 hr.
4. Remove the tubes from the water bath and remove excess surface water with paper towel. Remove the tube caps and treat the contents with 4.0 mL of ethanol (99 % v/v) or IMS (99 % v/v) with vigorous stirring on a vortex mixer.
5. Centrifuge the tubes at 1,500 *g* (approx. 3,000 rpm) for 10 min (non-capped).
6. Carefully decant the supernatants and re-suspend the pellets in 2 mL of 50 % ethanol or 50 % IMS with vigorous stirring on a vortex mixer. Add a further 6 mL of 50 % IMS, mix the tubes and centrifuge again at 1,500 *g* for 10 min.
7. Decant the supernatants and repeat this suspension and centrifugation step once more.
8. Carefully decant the supernatants and invert the tubes on absorbent paper to drain excess liquid.

(b) Measurement of Resistant Starch.

i. Add a magnetic stirrer bar (5 x 15 mm) and 2 mL of 2 M KOH to each tube and re-suspended the pellets (and dissolve the RS) by stirring for approx. 20 min in an ice/water bath over a magnetic stirrer.
ii. Add 8 mL of 1.2 M sodium acetate buffer (pH 3.8) to each tube with stirring on the magnetic stirrer. Immediately add 0.1 mL of AMG (solution 1; 3300 U/ml), mix well and place the tubes in a water bath at 50 °C.

iii. Incubate the tubes for 30 min with intermittent mixing on a vortex mixer.

iv. ***For samples containing > 10 % RS content***; quantitatively transfer the contents of the tube to a 100 mL volumetric flask (using a water wash bottle). Use an external magnet to retain the stirrer bar in the tube while washing the solution from the tube with the water wash bottle. Adjust to 100 mL with distilled water and mix well. Centrifuge an aliquot of the solution at 1,500 *g* for 10 min.

v. ***For samples containing < 10 % RS content***; directly centrifuge the tubes at 1,500 *g* for 10 min (no dilution). For such samples, the final volume in the tube is approximately 10.3 mL (however, this volume will vary particularly if wet samples are analyzed, and appropriate allowance for volume should be made in the calculations).

vi. Transfer 0.1ml aliquots (in duplicate) of either the diluted (step iv) or the undiluted (step v) supernatants into glass test tubes (16 x 100 mm), add 3.0 mL of GOPOD reagent and incubate at 50 °C for 20 min.

vii. Measure the absorbance of each solution at 510 nm against the reagent blank.

CALCULATIONS

Calculate resistant starch (%, on a dry weight basis) in test samples as follows:

Resistant Starch (g/100 g sample) (samples containing > 10 % RS):

$= \Delta E \times F \times 100/0.1 \times 1/1000 \times 100/W \times 162/180$

$= \Delta E \times F/W \times 90.$

Resistant Starch (g/100 g sample) (samples containing < 10 % RS):

$= \Delta E \times F \times 10.3/0.1 \times 1/1000 \times 100/W \times 162/180$

$= \Delta E \times F/W \times 9.27.$

where:

ΔE = absorbance (reaction) read against the reagent blank.

F = conversion from absorbance to micrograms

W = dry weight of sample analyzed

APPENDIX B

Quantitative Descriptive Analysis

Assessor……………… Date………………

Product: Plain boiled rice noodles

Instruction:

1) Please rinse your mouth thoroughly before and after assessing each boiled sample
2) Please mark the intensity for each attribute of the sample by the category scale

Sample code	Color (1-5)	Clarity (1-5)	Firmness (1-5)	Flavor (1-5)	Acceptability (1-5)

Range of Scores:

Color: 1-white to 5-yellow;

Clarity: 1-opaque to 5-clear;

Firmness: 1-firm to 5-soft;

Flavor: 1- characteristic to 5-not characteristic;

Acceptability: 1- very acceptable to 5-not acceptable

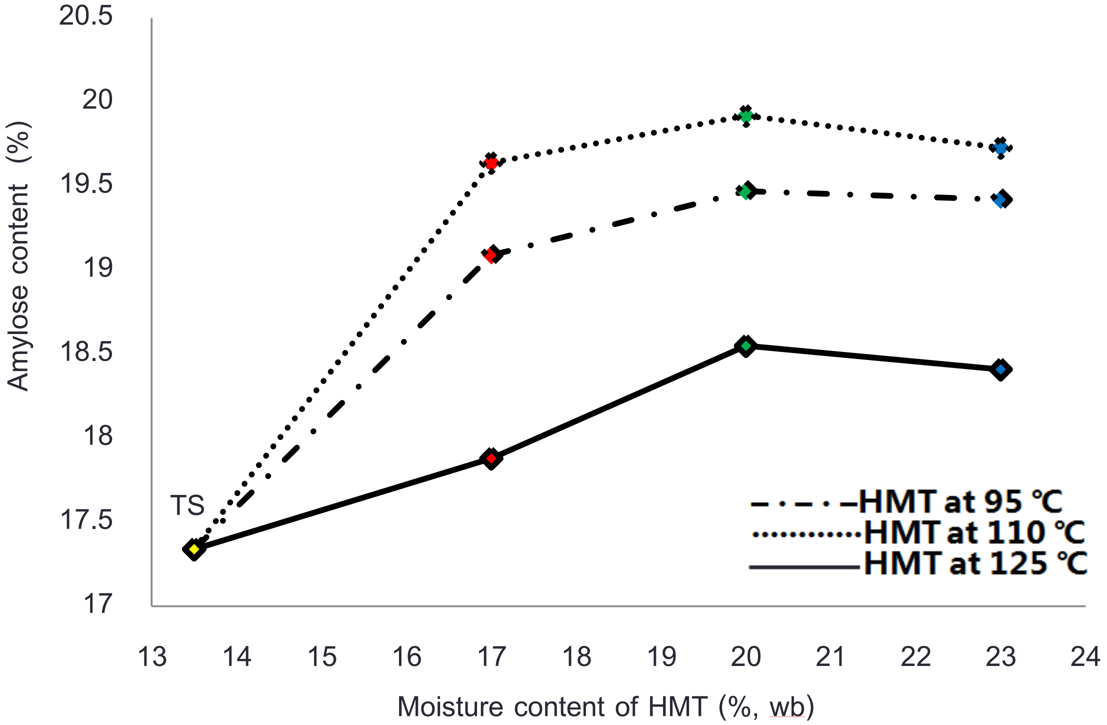
APPENDIX C

ADDITIONAL DATA

C.1 ANOVA for the different HMT temperatures and times on apparent amylose of tapioca starch

Source	df	Mean Square	F
Moisture content	2	1.801	5.112*
Temperature	2	11.353	32.213*
Moisture content * Temperature	4	0.647	1.835

*Sig<0.05

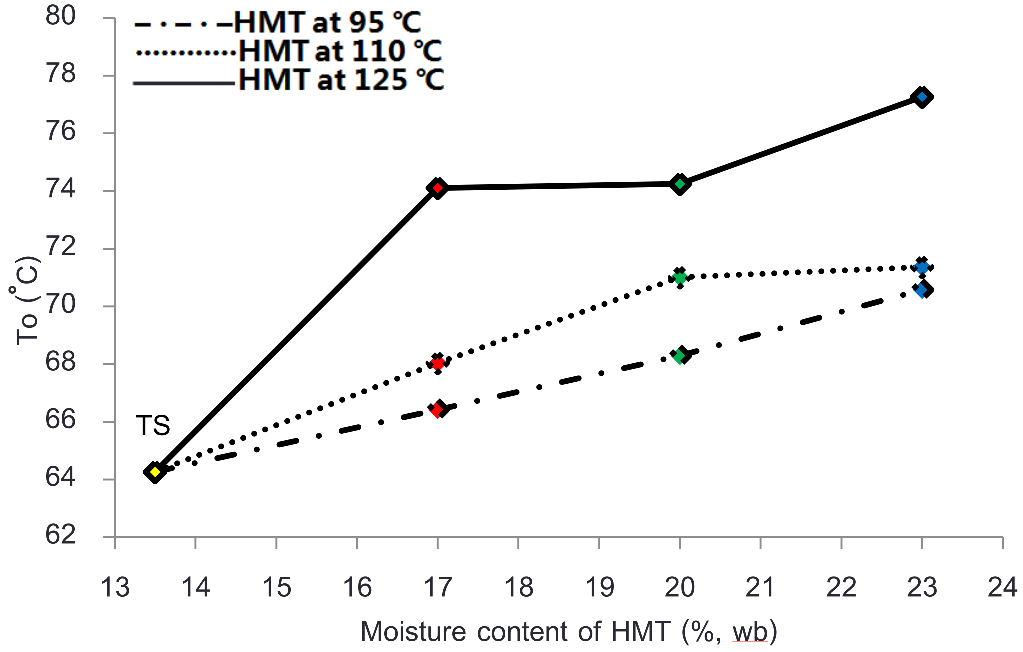


C.2 Effect of HMT moisture contents and temperatures on apparent amylose content of TS.

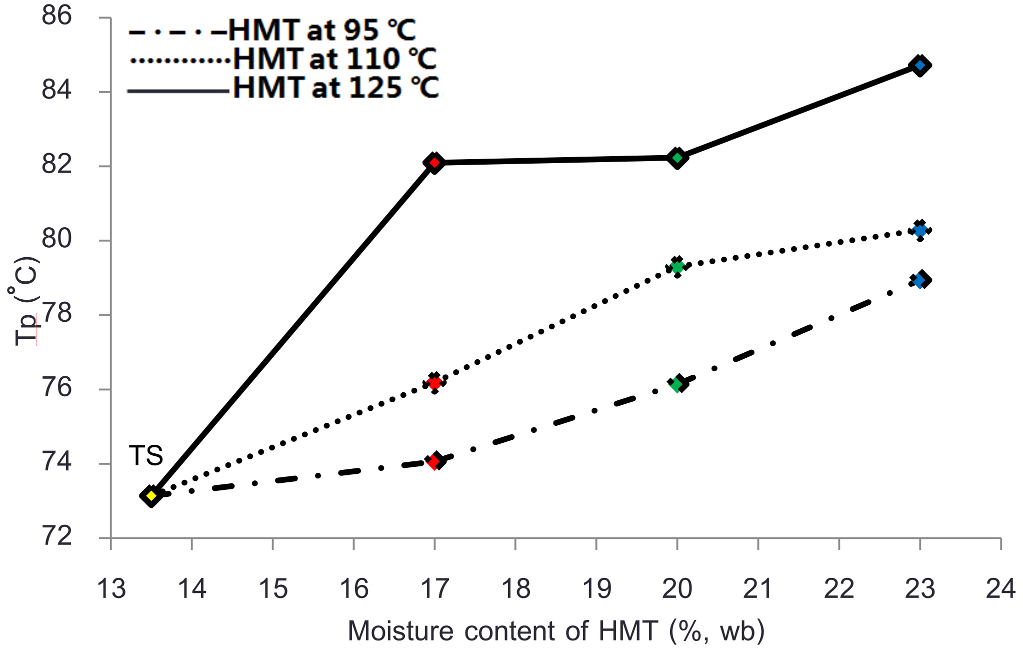
C.3 ANOVA for the different HMT temperatures and times on thermal properties of tapioca starch

Dependent Variable	Source	df	Mean Square	F
To	Moisture content	2	37.895	109.502*
	Temperature	2	149.099	430.837*
	Moisture content * Temperature	4	2.832	8.183*
Tp	Moisture content	2	44.968	55.047*
	Temperature	2	136.873	167.551*
	Moisture content * Temperature	4	3.056	3.741*
Tc	Moisture content	2	46.725	102.235*
	Temperature	2	136.725	299.157*
	Moisture content * Temperature	4	3.383	7.403*
ΔH_G	Moisture content	2	12.998	37.805*
	Temperature	2	17.008	49.470*
	Moisture content * Temperature	4	3.272	9.516*
ΔT	Moisture content	2	2.909	6.128*
	Temperature	2	11.733	24.713*
	Moisture content * Temperature	4	3.753	7.905*
ΔH_R	Moisture content	2	0.828	45.219*
	Temperature	2	12.928	705.744*
	Moisture content * Temperature	4	0.028	1.547

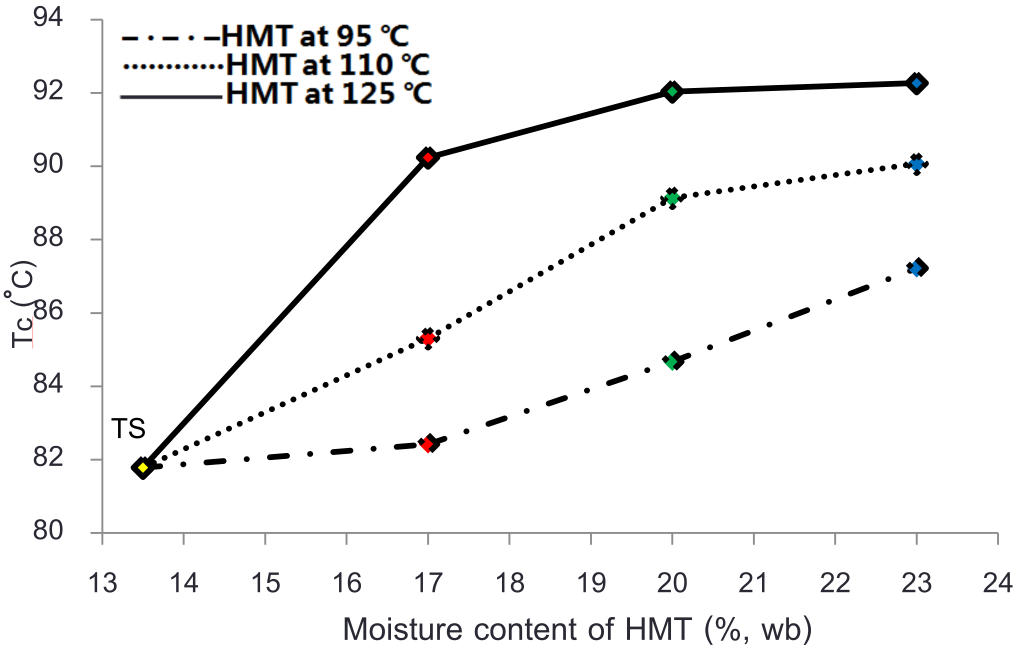
*Sig<0.05



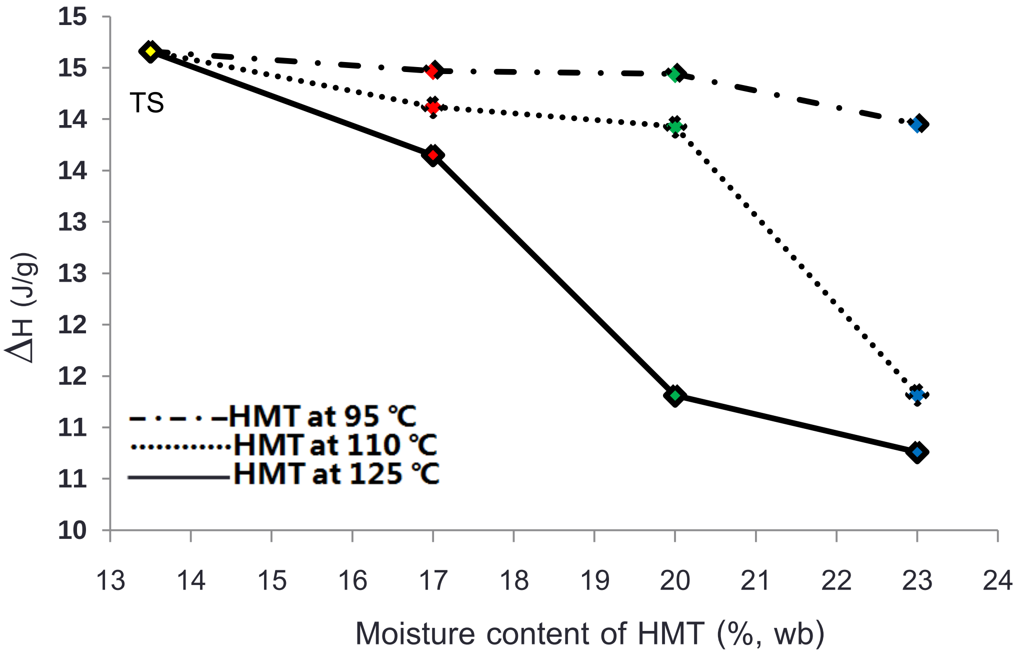
C.4 Effect of HMT moisture contents and temperatures on gelatinization onset temperature (To) of TS.



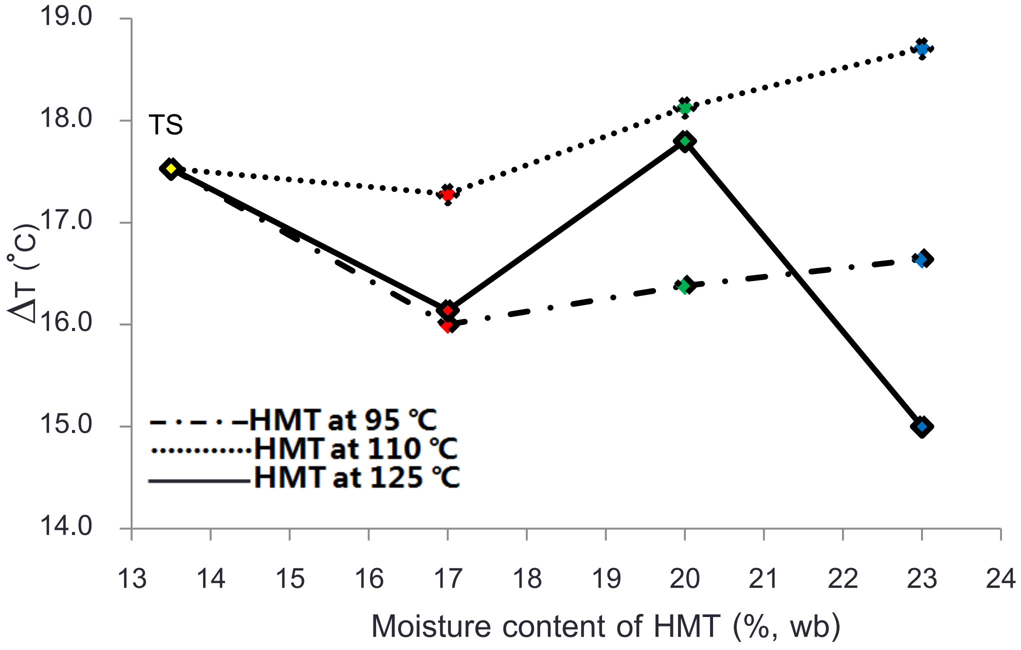
C.5 Effect of HMT moisture contents and temperatures on gelatinization peak temperature (Tp) of TS.



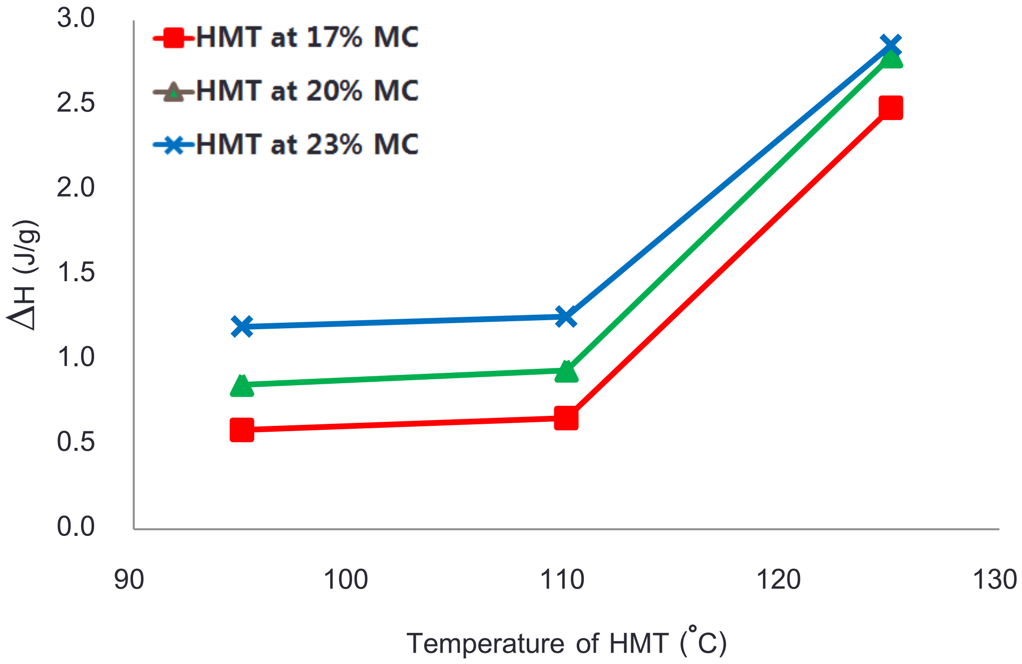
C.6 Effect of HMT moisture contents and temperatures on gelatinization conclusion temperature (Tc) of TS.



C.7 Effect of HMT moisture contents and temperatures on gelatinization enthalpy (ΔH) of TS.



C.8 Effect of HMT moisture contents and temperatures on gelatinization temperature range (ΔT) of TS.

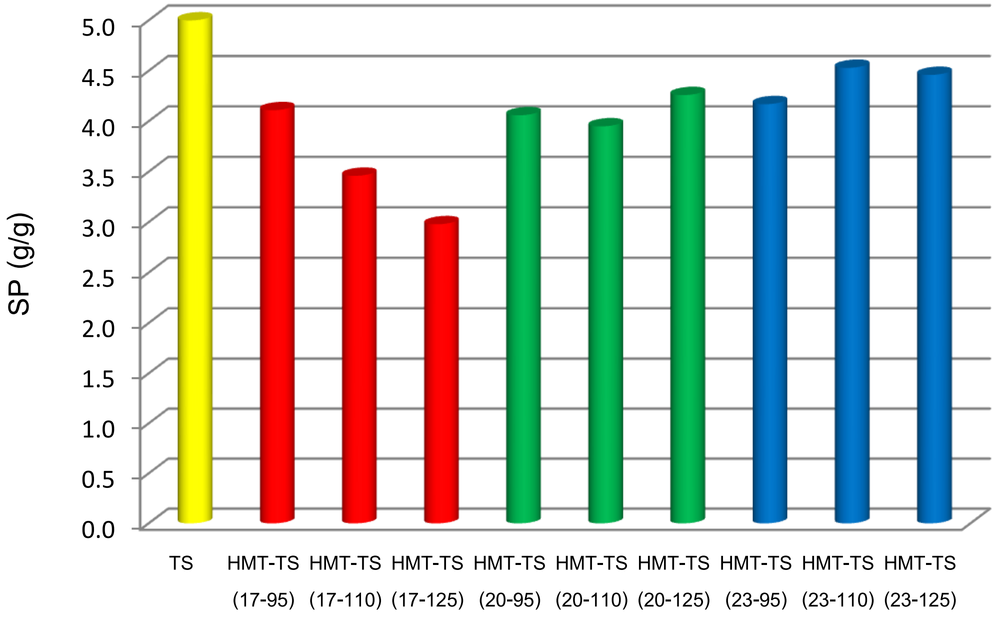


C.9 Effect of HMT moisture contents (MC) and temperatures on retrogradation enthalpy (ΔH) of TS.

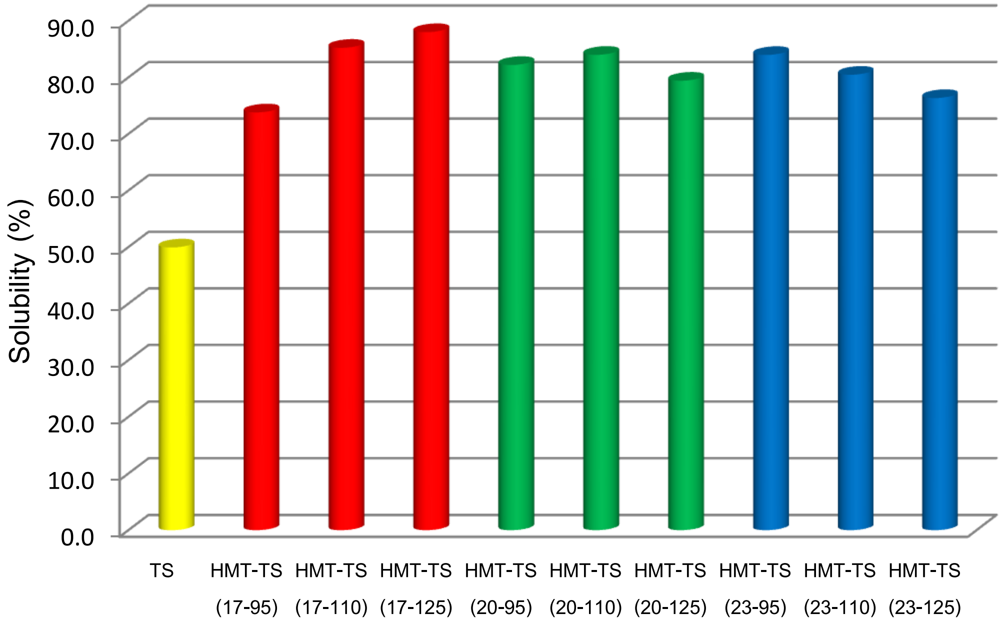
C.10 ANOVA for the different HMT temperatures and times on swelling power, solubility and gel hardness of tapioca starch

Dependent Variable	Source	df	Mean Square	F
SP	Moisture content	2	8.408	118.348*
	Temperature	2	1.925	27.099*
	Moisture content * Temperature	4	1.987	27.961*
Solubility	Moisture content	2	81.929	16.650*
	Temperature	2	408.266	82.972*
	Moisture content * Temperature	4	423.645	86.097*
Gel hardness	Moisture content	2	20.321	30.217*
	Temperature	2	289.974	431.186*
	Moisture content * Temperature	4	21.749	32.341*

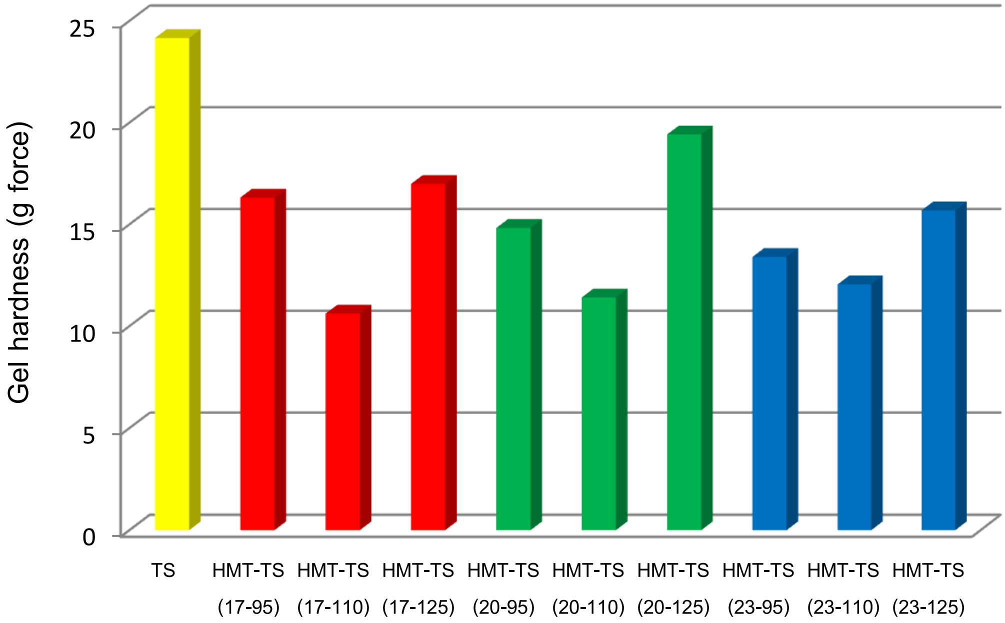
*Sig<0.05



C.11 Effect of HMT moisture contents and temperatures on swelling power (SP) of TS.



C.12 Effect of HMT moisture contents and temperatures on solubility of TS.

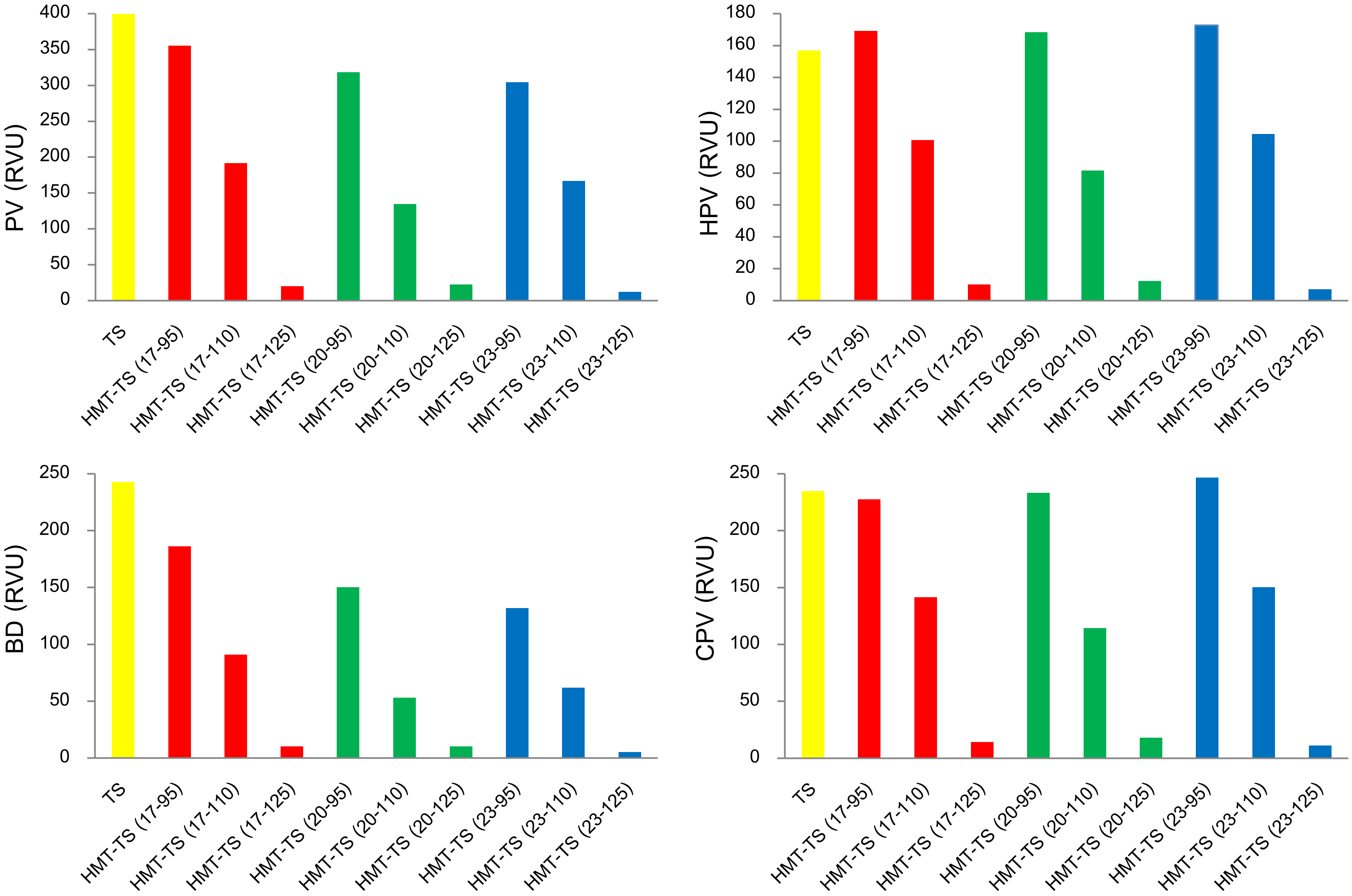


C.13 Effect of HMT moisture contents and temperatures on gel hardness of TS.

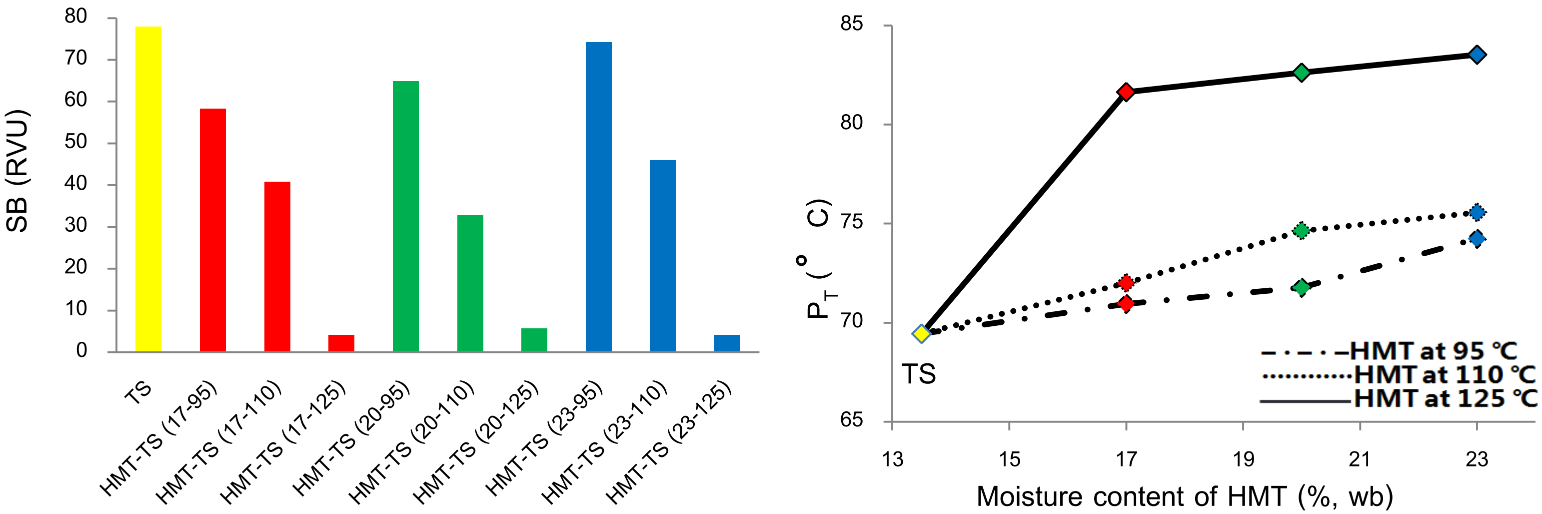
C.14 ANOVA for the different HMT temperatures and times on RVA pasting characteristics of tapioca starch

Dependent Variable	Source	df	Mean Square	F
PV	Moisture content	2	3519.075	11.167*
	Temperature	2	294328.986	933.996*
	Moisture content * Temperature	4	1379.074	4.376*
HPV	Moisture content	2	192.337	5.749*
	Temperature	2	80106.921	2.394E3*
	Moisture content * Temperature	4	241.656	7.223*
BD	Moisture content	2	3016.518	18.358*
	Temperature	2	68091.140	414.381*
	Moisture content * Temperature	4	836.677	5.092*
CPV	Moisture content	2	647.833	14.701*
	Temperature	2	153071.684	3.474E3*
	Moisture content * Temperature	4	634.482	14.398*
SB	Moisture content	2	200.778	8.221*
	Temperature	2	11727.506	480.210*
	Moisture content * Temperature	4	122.636	5.022*
PT	Moisture content	2	25.347	98.367*
	Temperature	2	378.099	1.467E3*
	Moisture content * Temperature	4	1.760	6.832*

*Sig<0.05



C.15 Effect of HMT moisture contents and temperatures on PV, HPV, BD, and CPV of TS.



C.16 Effect of HMT moisture contents and temperatures on SB and P_T of TS.

C.17 ANOVA for the different blending counterparts and BRNF on swelling power, solubility and gel hardness of flour combinations

Dependent Variable	Source	df	Mean Square	F
SP	Blending counterparts	3	26.388	21.632*
	BRNF	1	7.739	6.344*
	Blending counterparts * BRNF	3	0.499	0.409
Solubility	Blending counterparts	3	601.741	537.620*
	BRNF	1	55.575	49.653*
	Blending counterparts * BRNF	3	4.780	4.271*
Gel hardness	Blending counterparts	3	31.112	119.119*
	BRNF	1	11.249	43.069*
	Blending counterparts * BRNF	3	2.074	7.941*

*****Sig<0.05

C.18 ANOVA for the different blending counterparts and BRNF on RVA pasting characteristics of flour combinations

Dependent Variable	Source	df	Mean Square	F
PV	Blending counterparts	3	29075.870	4.631E3
	BRNF	1	552.133	87.931
	Blending counterparts * BRNF	3	12.584	2.004*
HPV	Blending counterparts	3	14404.079	3.446E3
	BRNF	1	355.511	85.050
	Blending counterparts * BRNF	3	16.867	4.035*
BD	Blending counterparts	3	3439.781	635.294
	BRNF	1	21.553	3.981
	Blending counterparts * BRNF	3	4.506	0.832*
CPV	Blending counterparts	3	41571.569	7.576E3
	BRNF	1	741.609	135.155
	Blending counterparts * BRNF	3	23.078	4.206
SB	Blending counterparts	3	7520.507	4.037E3
	BRNF	1	70.183	37.674
	Blending counterparts * BRNF	3	12.536	6.729
PT	Blending counterparts	3	17.518	202.736
	BRNF	1	5.941	68.761
	Blending counterparts * BRNF	3	1.815	21.007

*Sig<0.05

C.19 ANOVA for the different blending counterparts and BRNF on cooking and texture qualities of composite rice noodles

Dependent Variable	Source	df	Mean Square	F
Hardness	Blending counterparts	2	3313195.129	224.739*
	BRNF	1	3945657.774	267.640*
	Blending counterparts * BRNF	2	37909.770	2.571
Adhesiveness	Blending counterparts	2	1271.618	10.474*
	BRNF	1	460.430	3.793
	Blending counterparts * BRNF	2	27.235	0.224
Chewiness	Blending counterparts	2	472088.738	83.125*
	BRNF	1	212569.758	37.429*
	Blending counterparts * BRNF	2	52886.972	9.312*
Tensile strength	Blending counterparts	2	225.694	65.346*
	BRNF	1	15.777	4.568*
	Blending counterparts * BRNF	2	6.008	1.740

*Sig<0.05

VITAE

Mr. Yafei Liu was born on November 11th, 1986 in Anyang, China. He studied Food Quality and Security in China Agricultural University from 2006 to 2010, and he obtained the bachelor degree of science in July, 2010. From 2010 to 2012 he studied master degree in Department of Food Technology, Faculty of Science, Chulalongkorn University.

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Liu, Y.F., Laohasongkram, K., and Chaiwanichsiri, S. 2012. **Effects of broken rice noodle flour, tapioca starch and cross-linked tapioca starch on quality of composite rice noodle.** Presented at the *38th Congress on Science and Technology of Thailand (oral presentation)*, 17-19 October, 2012, Chiang Mai, Thailand.