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Effects of Crystallinity on Puffing Qualities of Rice Starch-Based Product

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งานวิจัยนี้มีสมมติฐานว่าปริมาณผลึกสัมพัทธ์ (RC) ของเจลสตาร์ชที่รีโทรเกรด (RG) และแผ่นสตาร์ชแห้ง (P) ส่งผลต่อสมบัติทางกายภาพและลักษณะทางประสาทสัมผัสของผลิตภัณฑ์พองตัวจากสตาร์ช (PD) ซึ่งผลการทดลองได้พิสูจน์สมมติฐานนี้และได้สร้างโมเดลทางคณิตศาสตร์เพื่อใช้ในการทำนายลักษณะทางประสาทสัมผัสและสมบัติทางกายภาพของ PD จาก RC ของ P เพื่อให้บรรลุวัตถุประสงค์ดังกล่าวงานวิจัยนี้ได้ศึกษาผลของปริมาณแอมิโลส (AC)ในช่วงร้อยละ 0.12-19.00 (น้ำหนักแห้ง) อัตราการทำให้เย็น (1, 3, 5 และ 9 องศาเซลเซียสต่อนาที) และระยะเวลาการบ่มที่ 4 ºC (24, 48 และ 72 ชั่วโมง) ต่อปริมาณผลึกสัมพัทธ์ของ RG และ P สมบัติทางกายภาพของ RG, P และ PD รวมทั้งลักษณะทางประสาทสัมผัสของ PD สตาร์ชข้าวที่ใช้สกัดจากข้าวเหนียวพันธุ์กข 6 และข้าวเจ้าพันธุ์เสาไห้เจ็กเชย โดยนำมาผสมที่สัดส่วนโดยน้ำหนักต่างกัน ทำการวิเคราะห์ชนิดของผลึกและ RC ของ RG และ P ด้วย X-ray diffraction pattern และพลังงานเอนทาลปีที่ใช้ในการหลอมผลึกด้วยเครื่อง Differential Scanning Calorimeter จากผลการทดลองไม่พบผลึกและเอนโดเทิร์มใน RG และ P ที่ได้จากสตาร์ชที่มี AC เท่ากับร้อยละ 0.12 และผลการทดลองพบว่าผลึกของ RG และ P เป็นแบบบีและวีและพบเอนโดเทิร์มในตัวอย่างที่ได้จากสตาร์ชที่มีแอมิโลส ≥ ร้อยละ 4.00 โดยพบว่า RC และพลังงานที่ใช้ในการหลอมผลึกของแอมิโลเพคติน (ΔH_{ru})ของ RG และ P เพิ่มขึ้นเป็นเส้นตรงกับ AC ด้วยความชันเท่ากับ 0.57 และ 0.27 ร้อยละ RC ต่อร้อยละ AC และ 0.34 และ 0.15 จูลต่อกรัมต่อร้อยละ AC ตามลำดับ นอกจากนี้ยังพบว่า RC ของ RG และ P เพิ่มขึ้นตามระยะเวลาการบ่มเย็นจาก 24 ถึง 72 ชั่วโมงอย่างน้อยร้อยละ 7 และ 15 และ ΔH_rของ RG และ P เพิ่มขึ้นอย่างน้อยร้อยละ 4 และ 7 ตามลำดับ แต่อัตราการทำให้เย็นไม่มีผลต่อ RC และΔH_r สำหรับ P พบว่ามีความแข็งและความเปราะเพิ่มขึ้น 0.53 และ 0.22 นิวตันต่อร้อยละ RC ตามลำดับ สำหรับผลิตภัณฑ์พองตัวพบว่า ค่าความแข็งและความเปราะเพิ่มขึ้นเท่ากับ 1.20 และ 0.89 นิวตันต่อร้อยละ RC และค่าความหนาแน่นจำเพาะเพิ่มขึ้นเท่ากับ 0.03 กรัมต่อมิลลิลิตรต่อร้อยละ RC ในขณะที่อัตราการขยายตัวลดลงเท่ากับ 0.134 ต่อร้อยละ RC RC ของ P ที่เพิ่มขึ้นส่งผลทำให้ขนาดโพรงอากาศเล็กลง และการขยายตัวของผลิตภัณฑ์ลดลงแต่ทำให้ความกรอบ ความแข็งและความเปราะของ PD เพิ่มขึ้น โมเดลที่พัฒนาขึ้นมาแสดงว่าค่าปริมาณผลึกสัมพัทธ์ในแผ่นแป้งแห้งที่มีค่าระหว่าง 0.95 ถึง 6.96 สามารถใช้เป็นตัวทำนายสมบัติทางกายภาพ ได้แก่ ค่าความแข็ง ความเปราะ ความหนาแน่นจำเพาะ และอัตราการขยายตัว รวมทั้งลักษณะทางประสาทสัมผัส ได้แก่ ความหนาแน่นและขนาดของโพรงอากาศ การขยายตัว ความพอง ความกรอบ ความแข็งและความเปราะของผลิตภัณฑ์ได้ดี โดยมีค่าสัมประสิทธ์ของการตัดสินใจมากกว่า 0.85 และยังพบว่าสมบัติทางกายภาพ ได้แก่ ความแข็ง ความเปราะ และความพองมีความสัมพันธ์กับค่าลักษณะทางประสาทสัมผัส ได้แก่ ค่าความแข็ง ความเปราะ และความกรอบ โดยมีค่าสัมประสิทธ์สหสัมพันธ์ ≥ 0.85 ค่าความพองและความกรอบสัมพันธ์กับค่าการขยายตัวและความเปราะซึ่งมีค่าสัมประสิทธิ์สหสัมพันธ์ ≥ 0.88 แผ่นสตาร์ชแห้งที่มีค่าปริมาณผลึกสัมพัทธ์ในช่วงร้อยละ 2.8 ถึง 3.7 ซึ่งผลิตจากสตาร์ชที่มีปริมาณแอมิโลสเท่ากับร้อยละ 9 มีคะแนนการยอมรับทางด้านลักษณะปรากฏและเนื้อสัมผัสสูงที่สุดโดยมีค่าความเข้มที่พอดีทางด้านความกรอบและความแข็งจากผู้บริโภคร้อยละ 78.3 และ 76.3

ภาควิชา	เทคโนโลยีทางอาหาร	ลายมือชื่อนิสิต .....
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ROSSAPORN JIAMJARIYATAM: Effects of Crystallinity on Puffing Qualities of Rice Starch-Based Product. ADVISOR: ASST. PROF. PASAWADEE PRADIPASENA, Sc.D., CO-ADVISOR: ASST. PROF. VARAPHA KONGPENSOOK, Ph.D., 150 pp.

The influence of relative crystallinity (RC) of retrograded starch gel (RG) and pellet (P) on physical properties as well as sensory attributes of rice starch-based puffed product (PD) was determined. Mathematical models for predicting of the sensory attributes of PD from either RC of the P or physical properties of the PD were developed. In order to achieve these aims, the effects of amylose content (AC in the range of 0.12-19.00% w/w on dry basis); cooling rate (1, 3, 5, and 9 °C/min) and aging time (24, 48, and 72 h) on RC, physical properties of RG; P and PD, and sensory attributes of PD were evaluated. Starches, isolated from waxy rice (RD 6) and non-waxy rice (Jeckchey-Saohai), were mixed to obtain an AC of 0.12, 4.00, 9.00, 14.00, and 19.00%. The crystalline type and RC of RG and P were determined by X-ray diffraction pattern (XRD) while retrogradation enthalpy (ΔH_{rt}) was determined using Differential Scanning Calorimetry (DSC) thermogram. For all cooling rates and aging times, the polymorph (B and V) and endotherms were found for AC ≥4.00%. For gels and P, RC and ΔH_{rt} increased linearly with AC with slopes of 0.57 and 0.27 %RC/%AC, and 0.34 and 0.15 J/g%AC, respectively. They also increased with aging time from 24 to 72 h at least 7 and 15% for RC gel and P while ΔH_{rt} increased at least 4% and 7%. The cooling rate did not affect RC and ΔH_{rt}. An increase in hardness was found to be 0.53 and 1.20 N/%RC for P and PD, respectively. The fracturability of P increased 0.22 N/%RC. For the PD, the hardness, fracturability, and bulk density increased 1.20 N/%RC, 0.89 N/%RC and 0.03g/ml/%RC; while expansion ratio decreased 0.134/%RC. A higher RC of P provided smaller and denser air cell structures and lower expansion, but higher crispness, hardness and fracturability of the PD. The developed models showed that the RC of P in a range of 0.95-6.96% can be an excellent predictor of physical properties (hardness, fracturability, bulk density, and expansion ratio) and sensory attributes (air cell density, air cell size, expansion, puffiness, crispness, hardness, and brittleness) of PD ($R^2 \geq 0.85$). It also found that physical properties (hardness, fracturability and puffiness) and their match sensory attributes (hardness, brittleness, and crispness) did correlate ($r \geq 0.85$). Puffiness and crispness correlated to expansion and brittleness ($r \geq 0.88$). P having RC in the range of 2.8-3.7%, made from a starch of 9% AC, got the highest scores for appearance and texture while also having just the right intensity of crispness and hardness by 76.3-78.3% of consumers.

Department:	Food Technology	Student's Signature	.....
Field of Study:	Food Technology	Advisor's Signature	.....
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I dedicate this Thesis to my beloved father. Finally, I would like to extend my special thanks to my beloved mother, brother (Mr. Pichit Sangthaun) and sisters for their love, encouragement, and emotional support. Without them, I would not have been able to achieve this degree.

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CHAPTER I
INTRODUCTION

Starch-based snacks have gained great popularity worldwide. In 2013, in response to consumer demand, thousands of new puffed snack products were debuted. Upon the processes of cooling and aging, the gelatinized starch was retrograded (Goodfellow and Wilson, 1990) which would consecutively affect the properties of gel, dried pellet and final product. The amylose content (AC), cooling rate and aging time are the main factors affecting the degree of retrogradation. Most research has reported that the higher AC and aging time led to the higher degree of retrogradation, and hence the higher gel hardness (Iturriaga et al., 2010; Vandeputte et al., 2003a; Vandeputte et al., 2003b, c; Varavinit et al., 2003; Yu et al., 2009). Therefore, the extent of this new crystallite formation determined in terms of relative crystallinity (RC) could be considered as a critical parameter determining the quality of starch-based products. Prior to puffing, the drying used to dense starch networks might affect the crystallites and RC. However, how the RC affects puffed product properties and characteristics has not yet been reported. Moreover, the effects of AC, cooling rate and aging time on crystalline structure, physical properties of pellet and puffed product and sensory attributes of puffed product have not been found.

The hypothesis of this research is that "the degree of retrogradation is responsible for the properties and sensory characteristics of puffed products". Therefore, this research

aimed to:

1. identify effects of AC, aging time and cooling rate on

   a. the crystalline type, RC, ΔH_r and the hardness of starch gel and pellet,
   b. the physical properties of puffed products, and
   c. the sensory attributes of puffed products,
2. compare the instrumental and sensory values of puffed products,
3. correlate the effects of pellet RC on sensory attributes of puffed products, and devise prediction models, and
4. determine the acceptability of puffed products.

CHAPTER II
LITERATURE REVIEW

2.1 Puffed starch-based product

Nowadays puffed starch-based products are popular for breakfast and as a snack. They are made from cereal, flour or starch, using either a conventional process or extrusion. This research is focused on the conventional process, which consists of cooking, cooling/aging, drying and explosive puffing. Puffing can be done by deep-frying, baking or gun-puffing. These procedures create an aerated and porous structure, resulting in a light (low density) and crispy product (Mariotti et al., 2006; Nath and Chattopadhyay, 2008). The lightness and crispness are the most appreciated sensory attributes for this type of products. Table 2.1 shows puffed starch-based snacks made from various starches. In South East Asia, cassava starch is widely used for making traditional puffed snacks called Keropok in Indonesia, Krupuk in Malaysia, Bahn Nam Tom in Vietnam, and Khao-Kriab in Thailand. Japanese and Korean traditional puffed starch-based snacks are usually made from rice. Japanese baked rice crackers are generally classified by texture. The softer texture and ready soluble in the mouth, Arare and Okaki, is made from waxy rice flour while the harder and more porous texture, Senbei, is made from non-waxy rice flour (or wheat flour in western Japan) (Nagao, 2001). The different types of Arare and Okaki classified by texture, which relates to specific volume, are soft (3.5-4.5 ml/g), medium (2.5-3.5 ml/g) and hard (2.0-2.5 ml/g). Commonly, Arare has soft texure and Okaki has medium texture. For Arare and Okaki, prior to drying, steamed dough is aged at 2-5 ˚C for 2-3 days and cut. For Senbei, steamed dough is cooled down to 60-65 ˚C

without aging, cut and then dried to 20 % moisture content. Korean traditional puffed product, Yukwa, is made from waxy rice and puffed in hot oil. Yukwa is softer than Arare and Okaki (Cho et al., 2003). The long steeping time (2-14 days) is considered to be the critical factor for volume expansion affecting crispiness and puffiness of Yukwa (Cho et al., 2003). For Yukwa, steamed dough is cut and dried to 17% moisture content without aging (Cho et al., 2003; Park et al., 2002). Thai rice crackers, Khao-Kriab-Waw, are made from waxy rice. While Japanese and Korean rice were wet ground first, and are then steamed and kneaded. The Thai rice cracker is made by steaming rice, kneading, spreading, drying without aging and baking. Khao-Kriab-Waw has a very light and brittle texture and is easily dissolved in the mouth (Sottirattanapan, 2007). In USA, corn puffed snacks have been popular since they were created in 1930's. Nowadays, both American and Mexican/Latin American-type puffed snacks made from corn are consumed and produced worldwide. American-type corn puffs are made from extruded corn starch, while Latin American type are made from alkaline cooked corn flour which use extrusion for the formation of dough and shape, and are then puffed by frying or baking (Matz, 1976). In Europe, commercial wafers are baked from a batter from soft (white) wheat flour. Batter is deposited onto the wafer-baking plates. After baking, the product is cut into "books" and the filling placed between the various layers (Wade, 1988).

Rice is one of the world's most important cereals for human consumption. It is the most widely produced and consumed cereal crop staple food in Asia. Compared to wheat worldwide, food use for rice is lower, however, even though it is considered as a low allergenicity food, or a hypoallergenic food. Hence, rice starch is typically recommended to be used as a main ingredient for babies' first foods and as

elimination diets for patients who have food allergies, intolerances or sensitivities. More than 75% of globally traded rice is Indica rice. Indica rice starch, therefore, is used in this research. In international trade, normal indica rices (non-waxy and non-aromatic) are the most commercialized (at 80%), while waxy rices are commercialized at 1%. Traditional Asian puffed rice-based products are mainly produced from waxy rice/rice starch. However, the price for non-waxy rice is only half of that of waxy rice. According to the Thai Rice Exporter Association, the trade price of rice is about 1050, 880 and 430 US dollars per metric ton for aromatic, waxy and non-waxy rices, respectively. For this reason, mixture of non-waxy rice starch and waxy rice starch should be used to produce affordable products for consumers as well as to make a profitable business. Amylose-amylopectin ratio is one of the most important factors for texture and consumer's acceptability in puffed starch-based products. On this account, the proper understanding of structural and physical properties of rice starch is crucial. Thus, their inherent properties were reviewed in the following section.

Table 2.1 Appearance and characteristic of puffed snack product from various starch type

Botanical Source	Cereal/flour/ starch	Amylose content (%)	Product name	Country	Method	Appearance	Characteristic	Source
Cassava	starch	18-24	Keropok	Southeast Asia	Forming a dough, shaping, boiling, cutting, drying, and frying		Crunchy, hard	Tongdang et al. (2008) The Vietnamese cusine (2014)
Waxy rice	cereal	0-2	Khao-Kriab-Waw	Thailand	Water steeping, grinding, shape, drying, and grill		Brittle, more expand, light and low density	Sottirattanapan (2007) Rice cracker (2014)
Waxy rice	cereal	0-2	Khoa Tung/ Khoa Tan/ Nang Led	Thailand	Water steeping, steaming, molding, drying, and frying		Hard and crispy texture	Intharapongnuwat et al. (2008) Asian supper (2014)

Table 2.1 (continue) Appearance and characteristic of puffed snack product from various starch type

Botanical Source	Cereal/flour/ starch	Amylose content (%)	Product name	Country	Method	Appearance	Characteristic	Source
Waxy rice	cereal	0-2	Yukwa	Korea	Water steeping, pulverizing, steaming, kneading, drying, and frying		Porous texture, brittle, light	Cho et al. (2004) Wikipedia the free encyclopedia Yumilgawa: Yugwa (2014)
Waxy rice	cereal	0-2	Arare/Okaki	Japan	Steeping, grounding, kneading, steaming, aging, rolling, cutting, drying, and baking.		Easily dissolved in the mouth	Keeratipibul et al. (2008) Martínez-Navarrete et al. (2004) Nagao (2001) Okaki and Senbei (2014)
Non-waxy rice	cereal	15-35	Senbei	Japan	Steeping, grounding, kneading, steaming, rolling, cutting, drying, and baking.		Hard and rough texture	

Table 2.1 (continue) Appearance and characteristic of puffed snack product from various starch type

Botanical Source	Cereal/flour/ starch	Amylose content (%)	Product name	Country	Method	Appearance	Characteristic	Source
Corn	starch	0-35%	Corn puffs	USA	Extrusion		Light	Matz (1976) Chinnaswamy and Hanna (1988) Sell corn puffs machine (2014)
Wheat	flour	24-27%	Wafer	UK	Batter mixing, baking of wafer sheets, cooling		Spongy texture, crunchiness and crispness	Wade (1988) Wafer boxes packing in shrink pack machine (2014)

2.2 Rice starch

Starch is the major component, comprising about 90% of rice solid and is present in the starchy endosperm as compound granules (Pomeranz, 1987). Compound granules, cluster of rice starch granules containing between 20 and 60 individual granules and having diameters up to 150 μm, fill most of the central space within the endosperm cell. Individual rice starch granules are the smallest known to exist in cereal grains, with the size reported ranging from 3 to 10 μm. Rice starch granules have polygonal or round shapes and smooth surfaces (Liu, 2005). Amylose and amylopectin are the main components of starch. In general, starch granules are rich in amylopectin, the layers being interlaced with strands of open amylose chains. Rice is classified from its AC as waxy (0-2% amylose), very low (5-12%), low (12-20%), intermediate (20-25%) or high (25-33%). Normal rice starch has AC of 12-35%. Pomeranz (1987) reported that indica rices (long-grain type) generally contain more amylose than japonica rices (short-grain type). Various rices with specific amylose-amylopectin ratios are grown and used for different rice products around the world.

Amylose is a mostly linear molecule of α(1-4)-linked D-glucopyronosyl units, containing approximately 1.4%wt of α(1-6) branching points (Takeda et al., 1987). Amylose greatly influences gelatinization, retrogradation, gelation and others physico-chemical properties of starch (Sasaki et al., 2002). Thus, the fine structure and content of amylose affects starch properties and functionality. Compared to amylopectin, the fine structure of amylose is much more simple, however, it has not been fully elucidated and has been studied to a lesser extent for different varieties of rice. The reported fine structure of amylose are number average degree of

polymerization (DP_n), chain length (the total amount of carbohydrate divided by the number of non-reducing end groups, CL), and percentage β-amylolysis limit (percentage of conversion to maltose).

It was reported that DP_n, CL, β-amylolysis limit of amylose from Indica rice and Japonica rice were not significantly different and were in the range 980 -1110, 230-370 glucose unit (GU) and 73-84%, respectively (Takeda et al., 1986; Vandeputte and Delcour, 2004). For Japonica rice starch, the weight ratio of branched amylose to linear amylose was 0.32:0.68, and DP_n was 740 for linear amylose and 1180 for branched amylose (Takeda et al., 1993). For rice starch sold in USA, amylose had DP_n of 2911 and branch linkages of 1.4%, and branch chain of amylose had DP_n of 72 (Mukerjea and Robyt, 2010).

Amylose content (AC) was reported to be one of the main factors affecting properties of starch gel and puffed products. Therefore, the determination of AC in starch is crucial. In this research amylose content was determined using amperometric titration. Amperometric titration is used to determine the equivalence point by measuring the electric current between the electrodes. During amperometric titration, the reaction occurs as shown in Equation [1]. The galvanometer was set to zero with water, reagent blank, and amylopectin blank. The changes in current depend on the quantity of KIO_3 used to tritrate. The equivalence point in the amperogram is shown in Figure 2.1. The details of procedure and calculation are shown in Appendix A1.

$$KIO_3 + 5KI + 6H^+ ==> 3I_2 + 6K^+ + 3H_2O \qquad [1]$$

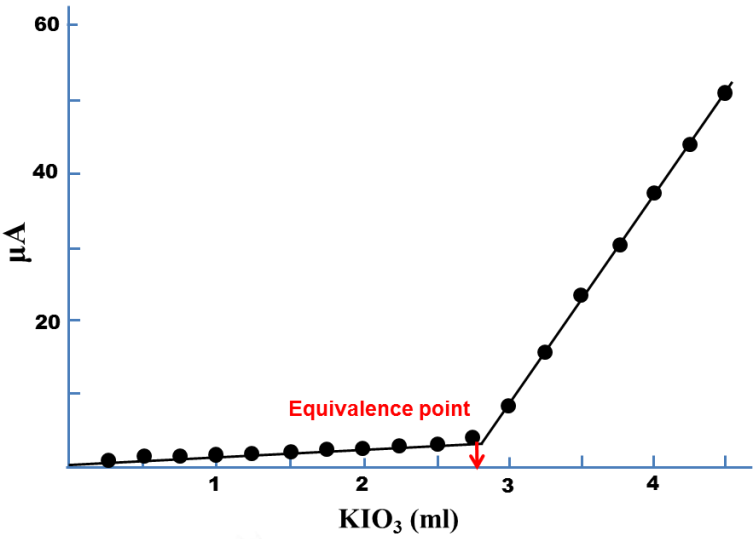


Figure 2.1 Amperogram

Amylopectin is a huge extensively branched macromolecule. Lu et al. (1997) reported that rice amylopectin had an average DP_n ranging from 2,700 to 12,000 (2,700 to 2,900 for high amylose indica rice and 6,500 to 12,000 low amylose indica rice and japonica rice). The current accepted model for the fine structure of amylopectin is polymodal distributions proposed by Hizukuri (1986) as shown in Figure 2.2. From this model, amylopectin consists of a lot of clusters jointed together by α(1-6)-linkage at reducing end side, and each cluster is composed of many short α(1-4)-D-glucopyronosyl chains that are bound together by α(1-6)-linkage at reducing end side. Average CL determined by debranching of rice starch with isoamylase was found to be 17.1-19.4 (Hizukuri et al., 1983). Among several rice cultivars, waxy rice had the average CL of amylopectin of 18 to 22, 15 to 18 and 17 to 20 for indica, japonica and waxy rice, respectively (Lu et al., 1997). Similarly, research of Takeda et al. (1987) reported the average CL of amylopectin was 19 to 20 and 21 to 22 for

japonica and indica rice, respectively. The japonica and indica rice has β-amylolysis of 58-59% and 56-59%, respectively (Tester et al., 2004).

In Figure 2.2, chains in amylopectin molecule were classified as follows (Hizukuri, 1986; Vandeputte and Delcour, 2004):

- C-chain (C) is the chain that contains the single reducing group of the amylopectin molecule and which carries other chains,
- B-chain (B) is the chain that connects to the C-chain and/or other B-chains, which is further classified as
  - B1: B-chains within a single cluster,
  - B2: B-chains extend in to 2 clusters,
  - B3: B-chains extend in to 3 clusters, and
  - B4: B-chains extend in to 4 clusters.
- A-chain (A) is the chain that connects to the B-chain and carries no other chains.

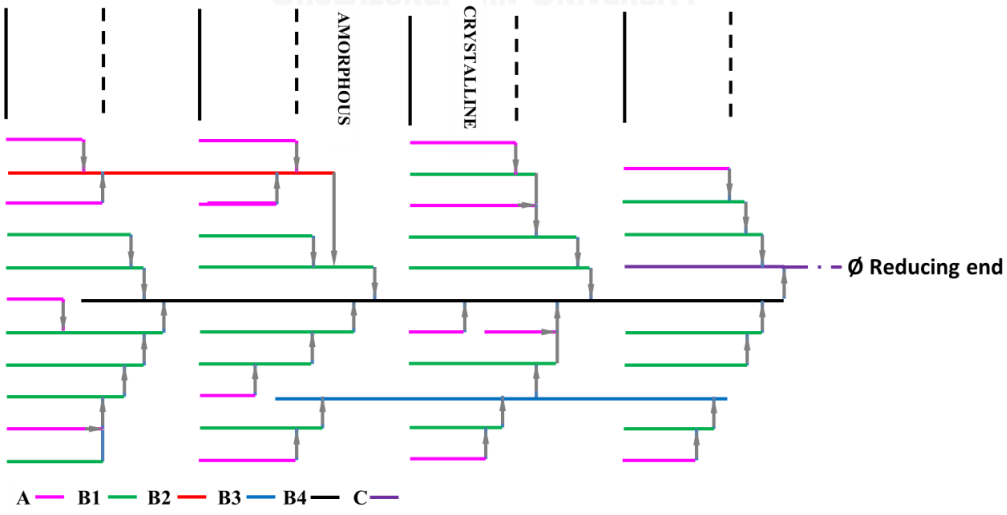


Figure 2.2 Amylopectin cluster model.
Modified from: Hizukuri (1986)

The range of CL for A, B1, B2, B3 and B4 was found to be 12-16, 20-24, 42-48, 69-75 and 104-140, respectively. Amylopectin of waxy and non-wax rice starches was reported to have similar molar ratios of A to B of 1.1:1-1.5:1 (Vandeputte and Delcour, 2004). Amylopectin having high proportion of A chain was reported to give a low tendency to retrogradation (Hizukuri, 1986). Wansuksri et al. (2004) determined the distribution of CL for amylopectin of rice starches used in this research, Saohai (Jechchey) and RD6. They reported that the percentage distribution for A (DP 6-12), B1 (DP 13-24), B2 (DP 25-36) and B>3 (B3 and B4, DP>37) were 27.74%, 59.39%, 9.07%, and 3.81% for Saohai-Jeckchey, and 31.6%, 25.97%, 9.24%, and 4.03% for RD6.

Like all cereal native starch, native rice starch is partially crystalline of A-type (Vandeputte and Delcour, 2004). Crystallites in A-type form by double helices of amylopectin short chains (DP 11-13) which pack tightly in the orthorhombic unit cell (dimensions of a = 1.17 nm, b = 1.77 nm, and c = 1.05 nm) with 8 molecules of water per cell as shown in Figure 2.3 (Tester et al., 2004; Wang et al., 1998). The four peaks at the diffraction angles (2θ) of 14.2º, 17º, 18º and 23.17º the crystalline portion of "A"-pattern starch (Arambula-Villa et al., 2001; Zobel, 1988). The degree of crystallinity of rice starch was reported to range from 29.2% to 39.3% (Ong and Blanshard, 1995a; Vandeputte et al., 2003b). Crystallinity was likely influenced by amylose-amylopectin ratios; however, a clear association between these two properties has never been reported (Ong and Blanshard, 1995b). Vandeputte et al. (2003b) reported that waxy rice starch had higher crystallinity than non-waxy varieties.

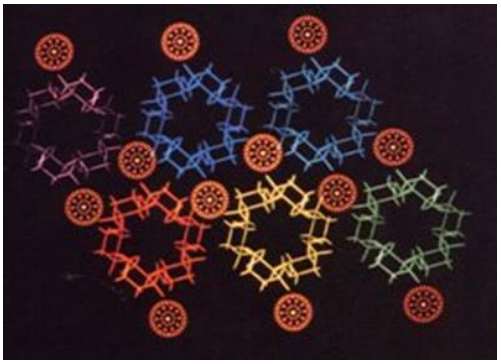


Figure 2.3 The A polymorph contains double helices that are densely packed in a monoclinic lattice.
Source: Starch structure and morphology (2014)

2.3 Effect of amylose content on starch, gel and puffed product properties

AC is widely recognized as the most important determinant of properties of starches (Lu et al., 2009) and their gels and puffed products. Processes for making starch-based products include gelatinization, cooling, aging, drying and puffing/explosive expansion. For each aforementioned process, an appropriate influence of AC on the properties of starches, and their gels and puffed products are reviewed in the following sections.

2.3.1 Gelatinization

Gelatinization is an order-disorder phase transition of starch during heating in the presence of excess water. Figure 2.4 shows the phenomena occurring throughout cooking starch (Jansson, 2008). The water penetrates the starch from the outside to the inside of granule. The swelling of granules increases as water uptake and temperature increase. Starch granules are heated up to the gelatinization temperature. The gelatinization temperature depends on the botanical source of starch

(Stevens and Elton, 1971; Varavinit et al., 2003). The hydrogen bonds of the starch chain are weak, and the crystallites/double helices are broken and melted. Amylose leaches out of the swollen granules while amylopectin is inside and holds the integrity of the swollen granules (Keetels et al., 1996b; Lu et al., 2009).

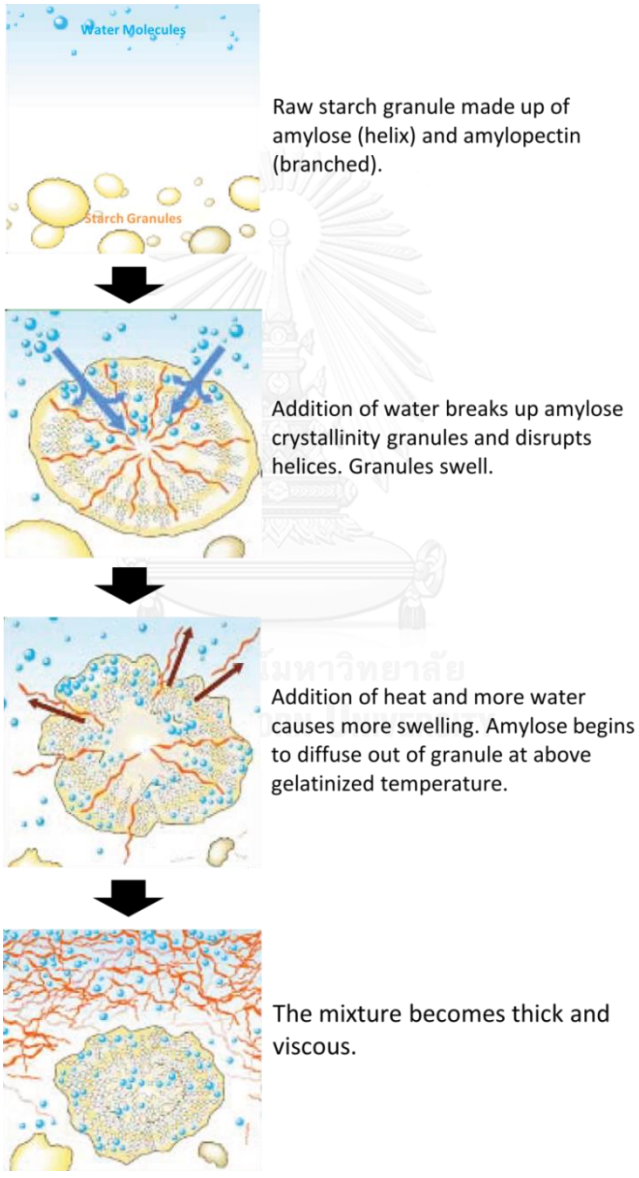


Figure 2.4 Schematic representation of starch gelatinization.
Source: Jansson (2008)

During gelatinization, thermal properties of starch are determined by DSC as changing in endothermic heat flow with increasing temperature. For rice starch, the onset (T_0), peak (T_p), and conclusion (T_c) gelatinization temperature and enthalpy of gelatinization was in a range of 52-75 ºC, 63-79 ºC, 71-89 ºC and 13-19 J/g, respectively (Lu et al., 2009; Vandeputte et al., 2003b; Varavinit et al., 2003). From the endothermic heat flow-temperature profile (DSC thermogram), the gelatinization phenomena is a multiphase transition of starch (Liu et al., 2007; Liu et al., 2006; Russell, 1987; Shogren, 1992; Yu and Christie, 2001). Figure 2.5 shows the DSC thermogram for rice starch of 22% AC at 50% w/w moisture content (Biliaderis, 2009). It shows four distinct endotherms; M1, M2, M3 and M4. The endotherms for the melting of amylopectin, M1 and M2, were found below 100 ºC, and those for an order and disorder processes of amylose-lipid complexes, M3 and M4, were found above 100 ºC (Biliaderis, 2009; Biliaderis et al., 1985; Jovanovich and Añón, 1999; Liu et al., 2007; Liu et al., 2006; Vandeputte et al., 2003b). The melting of short amylopectin with DP_n of 6-9 (M1) occurs at a lower temperature than that of the disruption of longer double helices of amylopectin with a DP_n of 12-22 (M2) (Vandeputte et al., 2003a). Sievert and Holm (1993) and Sievert and Würsch (1993) reported that a peak temperature of 108.4 and 154.2 ºC for the M4 endotherm was melting non-lipid complex amylose and dissociation (melting) of amylose instead of melting amylose-lipid complexes. Therefore, it should be verified by cooling and reheating the sample. The re-appearance of the endotherm at this temperature range suggested that endotherm M4 was melting amylose-lipid complexes (Chung and Liu, 2009; Russell, 1987).

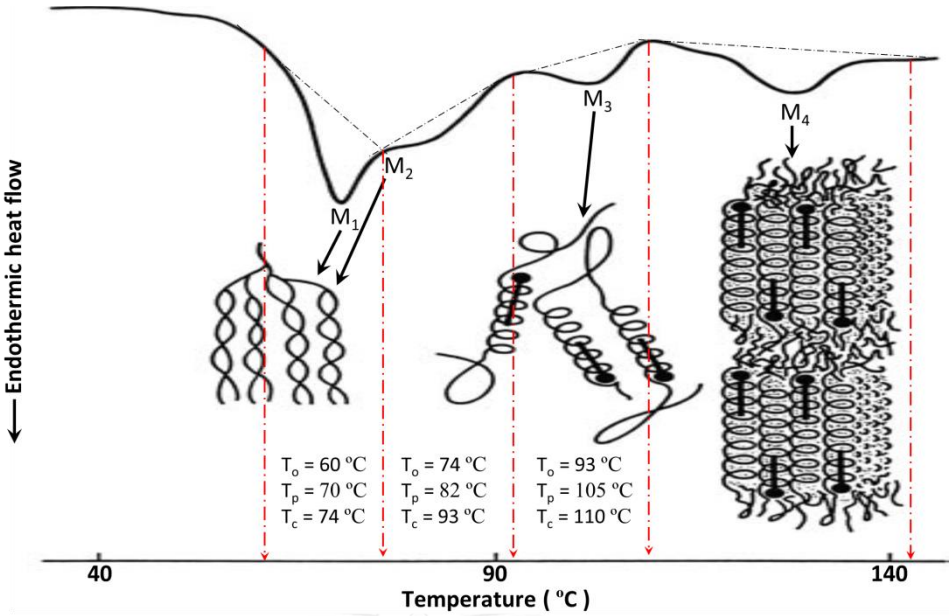


Figure 2.5 DSC thermogram with models described amylopectin structure and amylose-lipid complex of native rice starch.
Source: Biliaderis (2009)

The effects of AC and heating temperature on swelling power were determined using starches from 15 rice varieties (Vandeputte et al., 2003a). At temperature 65-75 ºC, the swelling power of waxy rice starch (AC of 0-5%) was found to be higher than that of non-waxy rice starch (AC of 15-30%). However, within the waxy rice starch group or non-waxy rice starch group, AC did not significantly affect swelling power. Heating at 95-125 ºC, swelling power decreased linearly as AC increased. This indicated that amylose acted as a restraint against swelling.

From the Rapid Visco Analyser (RVA) profile, the higher amylose rice starch gave a lower breakdown and peak viscosity (Vandeputte et al., 2003a; Varavinit et al., 2003). From DSC thermogram, the rice starch with a lower AC led to a lower

gelatinization enthalpy and gelatinization temperature (Vandeputte et al., 2003b; Varavinit et al., 2003).

2.3.2 Cooling and aging

During cooling and aging, the molecules composing gelatinized starch (non-crystalline gels) rearranged into a more ordered structure of amylose molecules (Hug-Iten et al., 2003; Keetels et al., 1996c; Putaux et al., 2000). Rapid gelation (or association) of amylose occurs upon cooling of starch paste, and slow recrystallization (or re-associate) of the amylopectin chains occurs during aging. These phenomena are called retrogradation (Aguirre et al., 2011; Atwell et al., 1988; Keetels et al., 1996a; Keetels et al., 1996c; Orford et al., 1987; Yuan et al., 1993).

Starch retrogradation is recrystallization of starch polymers upon cooling and aging after starch gelatinization. It is a time and temperature dependent process (Kibar et al., 2011). During gelatinization, starch granules swell and amylose leaches out of starch granule. By cooling, the leached-out amylose recrystallizes, and finally forms a network when the leached-out amylose concentration is sufficiently (Miles et al., 1985; Tester and Morrison, 1990). Starch paste/gel composes of porous, gelatinized and swollen granules with an amylopectin skeleton which suspend in an amylose solution/network (Richardson et al., 1981; Ring, 1985). While recrystallization of amylose is dominant at the initial stages of retrogradation, long term changes in starch gels are generally due to amylopectin.

Recrystallization process includes the nucleus formation and crystal growth. The nucleation step is the first step of recrystallization. The existent of nucleus is required for crystal growth. While crystal growth of existing nucleus occurs, new nucleuses also form. The crystal growth step consists of the propagation

and maturation. Propagation is growth of crystals from the nuclei, while maturation is crystal perfection or continuing slow growth (Park et al., 2009; Silverio et al., 2000). Nucleation, which is enhanced at lower temperatures, is the rate limit of recrystallization rather than propagation, which is enhanced at higher temperatures (Kibar et al., 2011). Recrystallization occurred above glass transition temperature (T_g) and below melting temperature (T_m). For starch gel of 50% moisture content, T_g was -5 ºC and Tm was 60 ºC (Biliaderis, 2009).

Tamaki et al. (2011) postulated the mechanism of retrogradation of amylose recrystallization, amylose-amylopectin co-recrystallization and intermolecular interaction of amylopectin as shown in Figure 2.6. This figure describes the following:

A. Amylose recrystallization

   A1. Both intramolecular and intermolecular hydrogen bonding of amylose chains occurred. Intermolecular hydrogen bonding of amylose chains resulted in amylose-amylose aggregates.

   A2. Retrogradation of amylose in continuous phase of starch solution/gel occurred from intermolecular hydrogen bonding of amyloses between amylose-amylose aggregates formed in A1 step.

B. Amylose-amylopectin co-recrystallization

   B1. Intermolecular hydrogen bonding between amylose and short amylopectin side chains, A and B1, took place

resulting in amylose-amylopectin aggregates. While intramolecular hydrogen bonding of amylopectin molecule occurred within long side chains, B2-B4 (not shown in this figure).

B2. Amylose chain of amylose-amylopectin aggregates (from B1) formed hydrogen bonding with another amylopectin molecule through A and B1 side-chains, which formed amylopectin-amylose-amylopectin aggregates.

C. Intermolecular interaction of amylopectin occurred between amylopectin of amylopectin-amylose-amylopectin aggregates (from B2) by forming intermolecular hydrogen bonding through the outer most side chains.

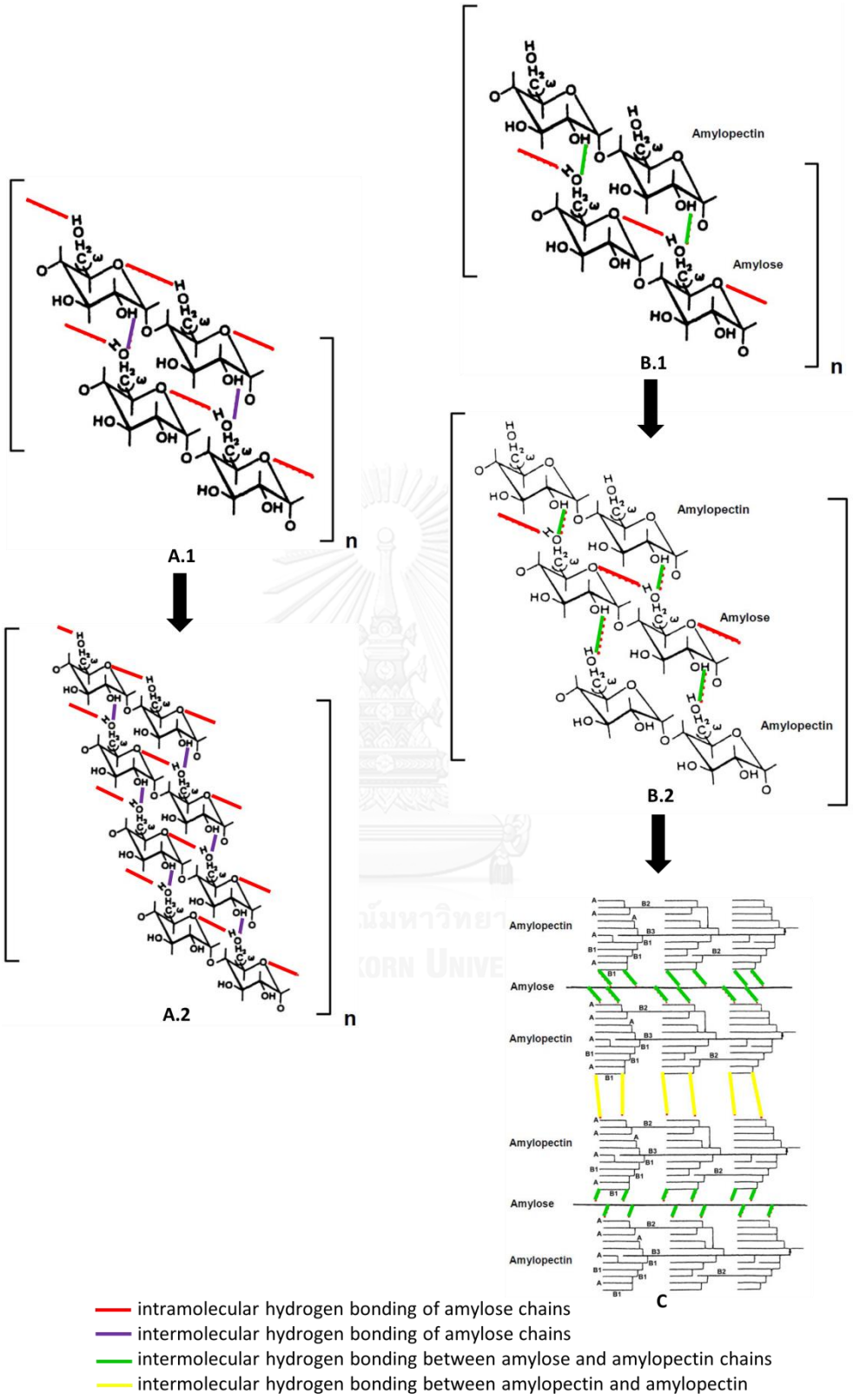


Figure 2.6 Possible model of retrogradation mechanism of rice starch. Dotted lines represent hydrogen bonding. The chains of amylopectin are short side-chains (A or B1).

The X-ray diffraction pattern obtained for amylose gels is known as a B-type pattern (Miles et al., 1985). It was reported that amylose chains of gelatinized starch having DP_n of 40-110 had intermolecular interaction and formed double helical associations. While amylose recrystallization has been reported to be very rapid with in a day, amylopectin recrystallization takes longer time (Zhang et al., 2008a; Zhang et al., 2008b). The amylopectin recrystallization was reported to form double helical associations mainly with the outer short chains of the molecules having DP_n of 15-20 (Gidley, 1989; Jane and Robyt, 1984; Ring et al., 1987; Sandhu and Singh, 2007; Vandeputte et al., 2003c). Lu et al. (2009) described melting and retrogradation of amylopectin as shown in Figure 2.7. Native crystalline structure of amylopectin (Figure 2.7a) is destroyed during heating in water and its short-branched chains form gel-balls (Figure 2.7b), V-type single helix crystals of amylose form upon cooling (Figure 2.7c), and new crystallines structures form during aging (Figure 2.7d). Moreover, Primo-Martin et al. (2007) reported that V-type was attributed to amylose-lipid complex.

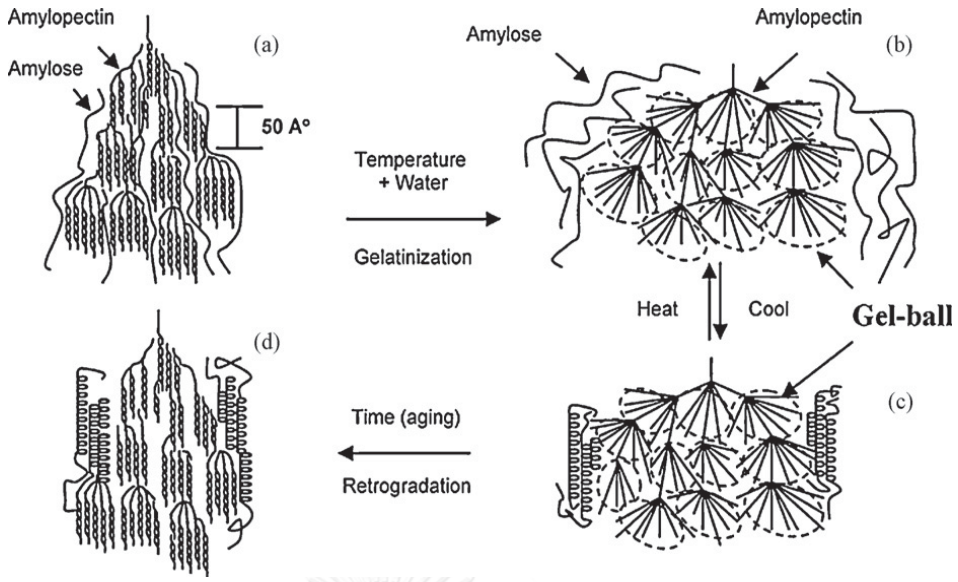


Figure 2.7 Schematic representation for phase transitions of starch during heating and cooling and aging.
Source: Yu and Christie (2005)

It was reported that an X-ray diffraction pattern of rice starch changed from A-type crystalline found in native to B-type crystalline after retrogradation as shown in Figure 2.8 (Biliaderis, 2009; Lopez-Rubio et al., 2008). For B-type crystalline (Figure 2.9), double helices of amylopectin short chains (DP 16-18) pack loosely in the hexagonal unit cell (dimensions of a =b = 1.85 nm, c = 1.04 nm) and each cell contains 36 molecules of water (Imberty and Pérez, 1989; Liu, 2005; Singh et al., 2006). The peak at 17 º2θ, for B-type crystalline, is due to the crystallization of the amorphous starch melt, mainly of the amylopectin (Keetels et al., 1996a; Zobel Henry F. and Kulp, 1996). The peak at 20 º2θ, for V-type crystalline of helical amylose complexes, was also found after retrogradation (Putseys et al., 2010; Zobel et al., 1988). The amylose-lipid complex is thermo reversible thus reheating did not change this V-type crystalline (Jovanovich and Añón, 1999; Russell, 1987). An increase in

the degree of crystallinity as a function of aging time was followed by an increase in intensity of the B-type and V-type XRD peaks (Aguirre et al., 2011; Primo-Martin et al., 2007). However, the effect of AC on degree of crystallinity of retrograded starch has not been found.

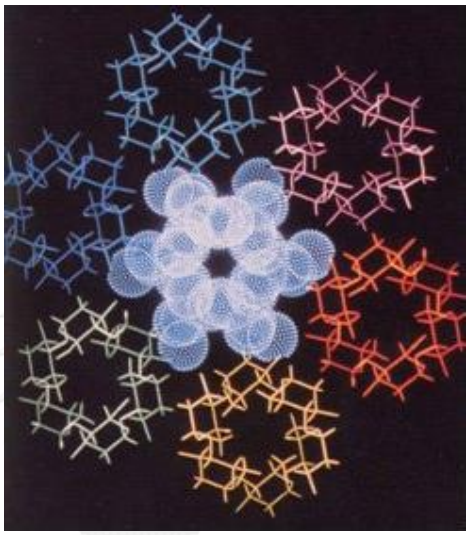


Figure 2.8 The B polymorph has double helices packed in a hexagonal lattice.
Source: Starch structure and morphology (2014)

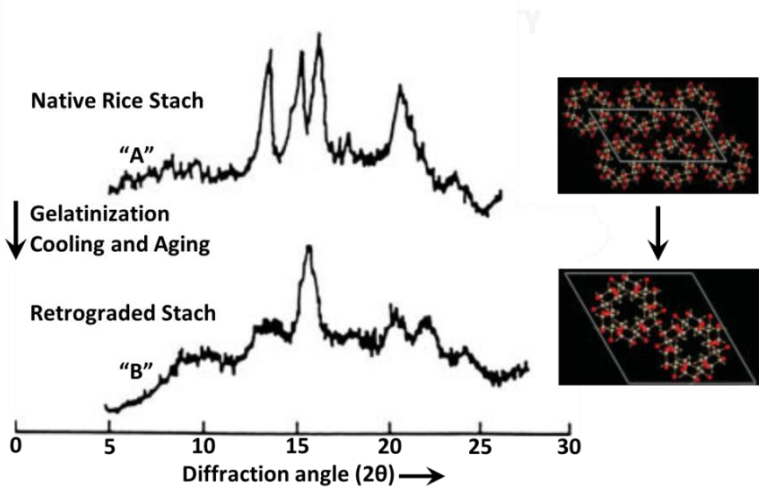


Figure 2.9 Polymorphic transition from the A to B starch.
Source: Starch structure and morphology (2014)

Thermal properties of retrograded starch were also determined using DSC. Compared to native starch, retrograded starch has wider endotherms, indicating that crystallites formed during cooling and aging were large, non-uniform and imperfect. For rice starch, T_o, T_p,T_c and enthalpy of retrograded starch were lower than those of native starch, suggesting that the crystallites in retrograded starch were less stable and easier to break down (Biliaderis, 1992; Chung et al., 2006; Paredes-López et al., 1994; Sandhu and Singh, 2007; Vandeputte et al., 2003b, c; White et al., 1989; Zhou et al., 2010). For aging at 5 ºC for 3 days, percentage retrogradation was found to be 51-60% for rice starches of 21-26% AC, and 18-30% for rice starches of 5% AC, indicating higher AC starch formed more or stronger crystallites (Sasaki et al., 2000; Varavinit et al., 2003). For aged cooked rice at 4 ºC for 1 day, T_o, T_p and T_c and enthalpy were 46, 55, 65 ºC and 9.71 J/g for rice of 35.7% AC, and 44, 52, 60 ºC and 5.72 J/g for rice of 1.2% AC (Yu et al., 2009). This indicated that retrograded starch from high AC starch had more stable, stronger and harder to destroy crystallites than that from low AC starch. The melting temperature of retrograded starch was found to be in a range of 55-85 ºC which was amylopectin retrogradation (Liu et al., 2007). There was a different effect of AC on retrogradation enthalpy. For aging at 6 ºC for 4 weeks, waxy rice starch gave lower enthalpy (1-4% AC) than normal rice starch (17-28% AC), which had enthalpy of 5-7J/g (Vandeputte et al., 2003b, c). The enthalpy is for amylopectin retrogradation. However, this result indicated that the higher AC resulted in the higher retrogradation. In low amylopectin contents, amylose is a nucleus for co-crystallization with amylopectin (Vandeputte et al., 2003a). However, earlier studies reported that the amylopectin retrogradation enthalpies decreased (10 J/g) with higher AC (Sasaki et al., 2000). Vandeputte et al. (2003c) explained that at

higher amylose content 1) amylose could penetrate in between amylopectin chains resulting in restriction of partially re-crystallization of amylopectin or 2) amylose co-crystallized with amylopectin resulting in a less perfect of crystalline. This result of DSC suited with B-type structure found in XRD pattern. Conformed to V-type structure presented in XRD pattern, DSC thermogram also showed an amylose lipid complex endotherm in a range of 90-120 ºC and 120-150 ºC (Biliaderis et al., 1985). Biliaderis et al. (1985) also reported that amylose-lipid was affected by moisture content. A single endothermic (96-104 ºC) was shown at excess water while low water contents (≤60%) showed two melting endotherms (90-130 ºC and 120-150 ºC). Thermograms for waxy starch did not show amylose-lipid complex endotherms (Biliaderis, 2009; Sasaki et al., 2000). By heating, cooling and reheating rice starch of 22% AC in the range of 20-160 ºC in DSC, the amylose–lipid complex endotherm was found at 100-110 ºC for all three thermograms (Figure 2.10). A higher enthalpy of the melting of amylose-lipid complex resulted from a higher AC (Sasaki et al., 2000).

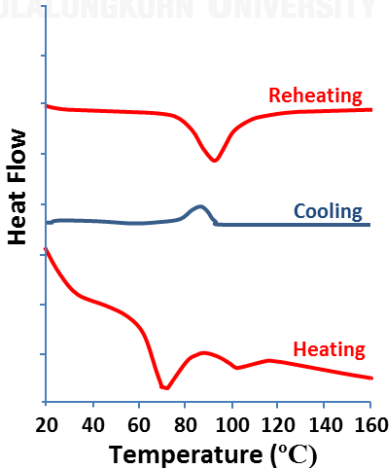


Figure 2.10 DSC heating, cooling and reheating rice starch
Source: modified from Chung and Liu (2009)

AC has been reported to have a strong impact on paste and gel properties. From pasting properties determined by RVA, the higher amylose (21-26%) rice starch gave higher setback (29-45 RVU) than lower amylose (4-5%) rice starch which gave lower setback (7-11 RVU) (Varavinit et al., 2003). This suggested that the higher setbacks is due to higher amylose forming a network in starch gel (Vandeputte et al., 2003a). The gel hardness was consistent with setback that increased with AC (Vandeputte et al., 2003a). The cooling rate and aging time also affected the crystallite mobility in starch gel and resulted in gel properties which will be reviewed in Sections 2.4 and 2.5.

2.3.3 Drying

The objective of drying is to reduced moisture content and dense starch networks. The mechanism for puffing will be reviewed in Section 2.3.4. The moisture content for dried starch pellets suitable for puffing was found to be in the range of 10-17% (Intharapongnuwat et al., 2008; Xie et al., 2008). Moisture content plays a critical role in the puffing of pellets as vaporization of the water causes an expansion of starch networks. Starch networks serve as a wall matrix of air cells; therefore, the strength of the starch network of pellets is also important. However, the hardness of pellets, which reflect the strength of the starch network of pellets, could not be found in the literature review as a function of AC. Instead of hardness, density of pellet were determined and found to be increase linearly with AC (Chen and Yeh, 2001). It is expected that the higher AC will result in the denser and harder pellet.

2.3.4 Puffing

Puffing is done by vaporization of water in dried pellets. Deep-frying or baking is commonly used for puffing. During heating, water in dried pellets rapidly

vaporizes and are trapped in starch networks, resulting in expansion and air cell formation as shown in Figure 2.11. The expansion is needed for the suitable balance between the driving forces from water evaporation inside the pellet and resistant force from starch networks to obtain the cellular porous structure (Bhat and Bhattacharya, 2001; Krokida et al., 2000; Saeleaw and Schleining, 2011). The properties and characteristics of puffed products are affected by expansion and air cell formation (Tongdang et al., 2008). The expansion is determined in terms of expansion ratio or linear expansion. The expansion ratio is the volume ratio of puffed product to pellet, while linear expansion is the percentage difference in dimensions or volume between puffed product and pellet. The impact of AC on expansion ratio has been studied by using starches from different botanical sources and by mixing low and high amylose starches.

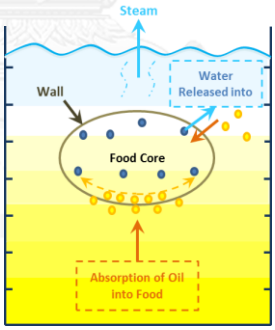


Figure 2.11 Puffing by frying

Puffed products made from waxy corn (<2% AC), cassava (19-27% AC), wheat (27-28% AC), and sago (22-29% AC) were found to have linear expansion of 300-400%, 120-190%, 100-115%, and 50-70%, respectively (Boischot et al., 2003; Cheow et al., 2004; Saeleaw and Schleining, 2010; Tongdang et al., 2008). Rice crackers made from waxy rice flour (<3% AC) normally have linear expansion of 250-300 % (Cho et al., 2003; Lu and Lin, 2001). For puffed rice grain,

the expansion ratio was reported to be about 2-3 for waxy rice and 1.5-2.5 for normal rice (Villareal and Juliano, 1987). A mixture of cassava-sago starches and mixtures of waxy-non-waxy rice flour resulted in expansion ratio decreasing as AC increased (Chen and Yeh, 2001; Tongdang et al., 2008). This result suggests that amylose or amylopectin content are responsible for expansion. This might be due to the structures of amylose and amylopectin, because the linear chain structure of amylose forms strong networks that resist expansion (Chen and Yeh, 2001). The highly branched chain structure of amylopectin led to the formation of weaker networks, resulting in large expansion and low density (Chen and Yeh, 2001). Furthermore, as was previously mentioned, the higher AC promoted retrogradation, resulting in a stronger gel, thus resisting expansion. Large expansion and low density led to light product texture, which is more desirable for consumers (Lu and Lin, 2001). Tongdang et al. (2008) reported that the amount of air cells increased with AC. Oil absorption decreased with increasing AC (Mohamed and Abd Hamid, 1994). The sensory characteristics have been widely studied in puffed products, such as extruded puffs and oil-puffed snack. Surprisingly, the effects of AC on sensory characteristics has not been found. The characteristics and texture of puffed snacks will be reviewed in Section 2.6.

2.4 Effect of cooling rate on starch gel and product properties

Upon cooling, a formation of new crystallites occurs. The cooling rate affected the mobility of crystalline and re-aggregation of amylose to form junctions (Creek, 2007). For gelatinized maize starch, a cooling rate at 10 ºC/min provides a broader endotherm and enthalpy of 8 J/g, while a slower cooling rate at 1 ºC/min gave a narrow endotherm and enthalpy of 25-30 J/g (Nordmark and Ziegler, 2002). This

indicated that the slower cooling rate provided higher retrogradation with a smaller and more stable crystallite than a faster cooling rate. At slow cooling rates, an increasing number of crystal junction form, resulting in an interconnection of the polymer chains (Creek, 2007). For cooked rice, rapid cooling rates (3.36 ºC/min) retarded starch retrogradation, resulting in low retrogradation enthalpy (0.85 J/g) and gel hardness (29.35 N) (Yu et al., 2010). For rapid cooling rates, there was no time for the amylose and amylopectin to form a gel network. However, the effect of cooling rates on puffed product properties and characteristics has not been found.

2.5 Effect of aging time on starch gel and product properties

Gelation of amylose was rapid less than 1 day, while amylopectin recrystallization continued for weeks (Aguirre et al., 2011; Ji et al., 2007; Orford et al., 1987). The prolonged time of aging affected the higher amylopectin retrogradation (Yu et al., 2009). The effect of aging time at different AC provided different quantities of retrogradation. At constant AC, a longer aging time provided higher retrogradation. At high AC rice (27%), a longer aging time (0 to 63 days) provided higher enthalpy (1 to 10 J/g) (Iturriaga et al., 2010) while waxy rice (1-3% AC) gave lower enthalpy (0 to 4 J/g). This indicated that longer aging time needed higher energy to melt the new crystallite, which had a lot more stable and stronger crystallites than those produced with a shorter aging time (Sasaki et al., 2000). Moreover, the retrogradation also depended on aging temperature. For rice starch aged at ambient temperature, the high AC (13-18%) gave a higher percentage of retrogradation (35% to 50%) of retrograded starch increased with increasing aging time (2 to 4 weeks) while waxy rice with 1-3% AC found no amylopectin retrogradation during 4 weeks, but was found at aging at 6 ºC for 4 weeks

(Vandeputte et al., 2003c). The hardness of gel was affected by aging time. A longer aging time resulted in a harder gel. Vandeputte et al. (2003c) reported that rice starch gel with high AC (29%) increased in firmness (425 to 719 g) and decreased in elasticity (76-42) when aged for 2 days to 2 weeks. This could suggest that during the aging, the higher the degree of retrogradation, the higher the hardness of starch gel could be obtained (Park et al., 2012; Perdon et al., 1999; Zhou et al., 2007). However, the effect of aging time on puffed product characteristics has not been found.

2.6 Texture of snack products

Textural properties are the group of physical characteristics, as the feeling of touch and the feeling related to deformation, and are measured objectively by functions of mass, time and distance. Texture is comprised of a number of different parameters (or attributes). The mechanical textural properties can be analyzed by observing the deformation (strain) of samples under applied stress (force per unit area) (Matz, 1962; Sherman et al., 1979). Texture is a key critical quality of expanded cereals and snack foods for consumer acceptance (Bourne, 2002). The "puffed" characteristic has multidimensional meaning. These have been subjectively described as crisp and homogeneous expanded structure, appropriate hardness, and attractive appearance. The profile composes of terms and definitions, intensity, and order of attributes. The various texture attributes evaluated in the texture profile method are grouped in mechanical, geometrical attributes and attributes related to moisture and fat content (Muñoz et al., 1992).

Texture is one of the most important qualities attribute and it is particularly true for snack foods. The importance of texture on the overall acceptability of the foods is well known and it directly affects the liking of the consumer. Consumers

expect tasty and crispy snacks. The main role of snack foods which attract the consumer are light food eaten between principal meals and providing wide varieties to the consumer. Besides the purposes, snack also provided sensory satisfaction to the consumers. Crispness, the most important quality attribute of a puffed product, relates directly to percentage (or ratio) of expansion (Kyaw et al., 2001; Siaw et al., 1985). It is perceived through a combination of touch, visual and auditory sensations and represents the key texture characteristics of snack products.

2.6.1 Descriptive sensory analysis of snack product

Descriptive sensory analysis involves in the detection of sensory components and quantifying their intensities by trained panels of judges in order to achieve greater consistency and accuracy (Meilgaard, 1991; Murray et al., 2001). The texture profile method is one of the descriptive sensory method specificity on classification of textural characteristics. With the texture profile method, it was found that the standard texture reference materials have been used for the development of definitions and evaluation procedures of mechanical parameters during training (Muñoz et al., 1992; Muñoz, 1986). The texture profile of snack products has been reported as crispy, crunchy, hardness, brittleness, expansion, cohesiveness, tooth packing, and oiliness by many researchers (Hong et al., 2014; Lazou and Krokida, 2010; Lazou et al., 2010; Murray et al., 2001) as shown in Table 2.2. Occasionally, there have been misunderstandings in the basic concept of the developed standard texture scales, due to the assumption that the originally published examples must be adhered to very strictly. Many standard references, published for the texture profile analysis, may not be available in every country. Furthermore, differences in language

and culture could pose limitations on the description of certain attributes (Cherdchu et al., 2013). Such deviation occurs from difficulties in understanding and interpreting previously established terms (Murray et al., 2001). In addition, sensory expressions unique in a particular language might not be accurately reflected in English because the attributes were developed before effective translation into English was available (Hong et al., 2014). Therefore, new references with modified definitions and evaluation procedures for selected textural attributes are needed (Hong et al., 2014; Muñoz, 1986). Jenkins (2008) used commercial snacks sold in Thailand and Korea such as Hanami™, Pringle™, Pocky™, rice cracker, sticky rice, and crispy green pea as new references for rating products. As a result, new texture references and terms (such as adhesiveness to lips, roughness, hardness, crispness, fracturability, cohesiveness of mass, denseness, moisture absorption and adhesiveness to teeth) were established for Thai and South Korean. Hong et al. (2014) also used local commercial products as a modified reference to create definitions for texture profile attributes of Korean cookies as shown in Table 2.3.

Table 2.2 Textural attributes of snack products

Product	Attributes	Source
Extruded snack	Crispy, Crunchy, Hardness, Brittle, Melt in the Mouth, Sticky Mouth Coating, Tooth packing	Murray et al. (2001)
Extruded potato puffs	Expansion, Fracturability, Hardness, Crispness, Oiliness	Faller and Heymann (1996)
Corn extruded puffs	Crunchiness, Crispness, Hardness, Cohesiveness	Lazou and Krokida (2010)
Korean oil puffed snack	Hardness, Crispness, Cohesiveness, Tooth packing Oiliness	Hong et al. (2014)
Potato Chips	Crispness	Zampini and Spence (2004)
Waxy rice extruded snacks	Hardness, Crispness, Brittleness, Noise	Chaiyakul et al. (2008)

Table 2.3 Definitions of the textural attributes and modified reference used in oil puffed snack product

Textural attributes	Definitions	Reference
Hardness	The force to attain a given deformation, such as force to compress between molars	Brownie Biscuit
Cripness	The noise with which a product breaks or fractures (rather than defoms) when chewed with the molar teeth	Brownie Energy bar
Cohesiveness	The degree to which sample defoms rather than crumbles, cracks or breaks	Agar gel Brownie White bread
Denseness	The compactness of the cross section	Pie Biscuit/Agar gel

Source: Hong et al. (2014)

2.6.2 Relationship between instrumental and sensory attributes

Because texture is one of the major acceptability factors of snack food, instrumental measurement of texture has progressed to investigate the relation

(Bourne, 2002). Early investigation have been examined the relationship between instrumental and sensory attributes. Lazou et al. (2010) reported that crunchiness can be related to appearance attributes of the extrudates and overall acceptability. Furthermore, the hardness of extrudates showed a correlation with crispness and melting (Lazou et al., 2010). In addition, the texture of cereal puffed products has been related to their moisture content (Roopa et al., 2009). Chaiyakul et al. (2008) also correlated the feed moisture content and barrel temperature with hardness, crispness, brittleness, and noise of extruded snacks from waxy rice flour. However, the perceived sensory properties and their relationships with instrumental measurements, which provide the practical knowledge for determining product properties, have only been studied to a limited extent. To save time and costs required for descriptive sensory analysis there has been a concern effort to determine relationships between sensory qualities and instrumental data.

CHAPTER III
MATERIALS AND METHODS

3.1 Starch isolation

Waxy rice (RD6, Royal Umbrella Brand, Ayutthaya, Thailand) and non-waxy rice (Soa-hi, Pin-Ngern Brand, Nonthaburi, Thailand) were soaked in water (weight ratio of hulled grain to water = 1:5) at 6±1°C for 16 h. After draining, they were washed twice. Then, they were wet-milled with a stone mill using a weight ratio of hulled grain to water of 1:5. The slurry was filtered through 50, and then 100 mesh sieves (Lavallab Inc., Canada). The cake was washed twice and filtered through 50 and 100 mesh sieves again. After sedimentation of starch at 6±1°C for 3 h, the supernatant was drained and the yellow upper layer was removed. The cake was suspended in water (weight ratio of wet cake to water = 1:2). For protein removal, the pH of the suspension was adjusted to 8.5 with 0.1 M NaOH (analytical grade, Ajax Finechem, NZ), stirred for 30 min, and then allowed to sediment at 6±1 °C for 5 h. After draining, the precipitate (starch granule) was washed twice (weight ratio of precipitate to water = 1:1). The pH of starch suspension was adjusted to pH 7 with 1.0 M HCl (analytical grade, Ajax Finechem, NZ), and then allowed to sediment at 6±1°C for 5 h. The cake was suspended in distilled water (weight ratio of precipitate to water = 1:2) and filtered through a 100-mesh sieve. The filtrate containing starch was collected and precipitated at 6±1°C for 3 h. The starch precipitate was dried in hot air oven (HA – 100S, Yeo Heng Co., Ltd., Bangkok, Thailand) at 65±1°C for 14 h. The dried starch was milled with a blender (HR2001, Philips, Belgium) and sieved through a 200-mesh wire sieve, packed in aluminum laminated bags and stored at

5±1°C. The moisture content of dried rice starch was determined using the hot air oven method at 105 ºC (AOAC, 1995). Moisture content of all starch mixtures was 11.89±0.20% w/w.

3.2 Starch mixture preparation

The rice starch mixtures were prepared by mixing waxy starch and non-waxy starch at weight ratio of 100:0, 75:25, 50:50, 25:75, and 0:100, respectively. The starch mixtures were sieved through 100 mesh wire sieve three times for homogeneous mixture. These starch mixtures were separately packed in aluminum laminated bags and stored at 4±1 °C.

3.3 Starch pellet preparation

Gelatinization: Starch (30 g dry weight) was dispersed in water (28 g). This dispersion was poured in hot water at 95±2 °C to obtain a total suspension weight of 100 g. Starch paste was prepared by heating starch suspension at 110±1 °C for 20 min in the closed system (1L capacity) equipped with hand mixer (BUO-153263, Buono, Germany) for continuous stirring at speed level 5. The moisture content of the starch paste was determined.

Spreading: Starch paste was spread on wax paper (Pankraft paper, Daiso, Japan) to obtain a thin slab (17.8x28.0x0.2 cm). The temperature of the starch paste was determined and found to be 40±3 °C after spreading.

Cooling: The spread pastes were cooled from 40±3°C to 4 °C in the polystyrene foam box (35.5x30.5x29.5 cm with 2.3 cm foam thickness) containing 6 blocks of dry ice (block size and weight were 20.0x15.0x2.5 cm and 1.17±0.02 kg). The electric operated fan (9 V direct current power supply, Suksapanpanit Scientific Apparatus and Instruments, Bangkok, Thailand) was used for

circulation inside the box. The cooling box was shown in Figure 3.1. Cooling rates varied by fan speed (high, medium, and low) were used to vary the time of cooling. The fan speed in Hz was measured by using a frequency counter (Leybold Didactic GMBH, Germany), and found to be 49±0.33, 40±0.33, 30±0.33 Hz, respectively. After storing the starch paste in a box, the fan was controlled at high, medium, and low the speeds for 3, 6, and 9 min, respectively. The control sample was kept in an incubator (MIR-153, Sanyo, Japan) at 4 °C for 42 min. The temperature and time was recorded by a data logger (Wisco AI2100, Thailand). The 4 points of sample were measured by sensor until the temperature dropped down to 4 °C. The cooling rate found varying 1, 3, 5, and 9 °C/min, respectively.

Aging: After cooling to 4 °C, it was wrapped with Saran® wrap and kept in plastic box enclosing silica gel (1/2 cm high of box) and aged at 4±1°C for 24, 48, and 72 h. The gel was kept in an incubator (MIR-153, Sanyo, Japan) at 4 °C. The moisture content was determined using the hot air oven method at 105 ºC (AOAC, 1995) and found to be63±2% w/w.

Drying: The starch gel was cut to obtain a square shape of 2 x 2 cm. The square paste (2x2 cm) was dried using a hot air oven at 40°C for 10 h. The moisture content of pellets was 15% w/w.

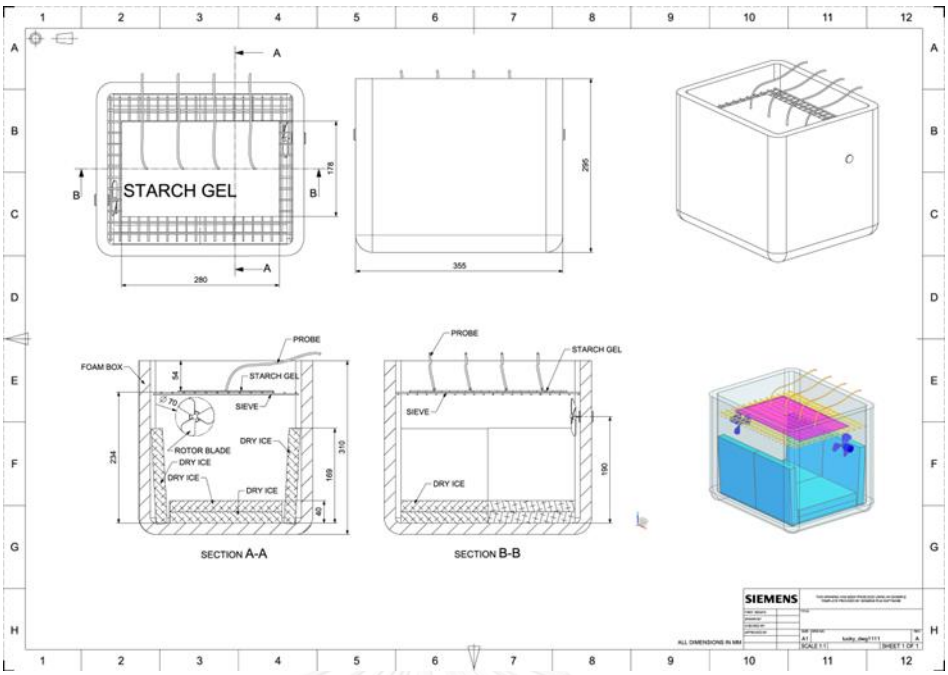


Figure 3.1 Cooling box

3.4 Puffing

For each batch, 6 g dried starch pellets were puffed by deep frying in 500 g palm oil (Morrakot, Morrakot Industries Co., Ltd., Thailand). The oil was heated on a hot plate (IH-EOV-40B-4, Alfa Kitch, Alfa One Trading Co., Ltd., Korea) that turned to the highest setting. The oil temperature was monitored and recorded using a calibrated thermocouple (Type K, SK PCR-1, Sang Chai Meter Co., Ltd., Thailand) and data logger (AI2100, Wisco, Thailand). The pellets were dropped in oil when it reached 210 °C, and they were fried for 50 s. The recorded temperature during frying was found to be 210±1 °C.

3.5 Amylose content determination

AC of all starch samples was determined by the amperometric method (Larson et al., 1953; Takeda et al., 1987; Williams et al., 1970). The mercury-mercuric oxide-

barium hydroxide half-cell was used, together with a hook-type rotating platinum electrode. The rotating platinum electrode was fitted with a suitable geared motor and the circuit was laid out as described the detail in Appendix A.

3.6 Crystallinity pattern and relative crystallinity determination

To preserve crystalline structure of gel, gels were freeze-dried at -50 ºC and 50x10-3 mbar for 12 h (Labconco, USA.). The freeze-dried gels and pellets were kept over a saturated NaCl solution (relative humidity about 75%) in a desiccator under vacuum for 2 weeks to obtain sample with moisture content about 15% w/w (wet basis, wb), and then were ground by blender prior to determination the X-ray diffraction pattern. Crystallinity of the starch granule was analyzed using a wide angle X-ray diffractometer (D5005, Siemens AXS, Germany) equipped with a copper source operating at 40 kV and 50 mA, producing X-rays as monochromatic copper Kα radiation with a wavelength of 0.154 nm. The diffraction data were collected over an angular range from 5 to 30° (2θ) at 0.1° intervals with a scanning rate of 60 s/°. The X-ray pattern was compared with the peak characteristic of theoretical diffractogram given by Zobel (1964) cited in Zobel et al. (1988). The relative crystallinity was determined quantitatively from the ratio of the sharp peak area to the total peak area (Nara and Komiya, 1983), using a peak-fitting software (Origin-version 8.0, Microcal Inc., Northampton, MA, USA.).

3.7 Thermal property determination

For starch gels, which had moisture content of 63±2% w/w, approximately 20 mg of gel was transferred to the pre-weighed large volume DSC stainless steel pans with O-ring and weighed to determine the actual sample mass with 1 mg accuracy. The sample pan was hermetically sealed and held isothermally at 20ºC for 5 min prior

to heating in the Differential Scanning Calorimeter, DSC (DSC 8000, Perkin- Elmer, Norwalk, VA, USA) from 20-180 ºC at 10 ºC/min. Verification of the peak, which occurred in the temperature range of 90-120 ºC, was carried out with starch gels obtained at cooling rate of 5 °C/min by immediately cooling down to 20 °C at 10 °C/min, and then reheating up to 180 °C at 10 °C/min.

For starch pellets and puffed product, the powdered samples (≈10 mg) were placed and weighed for determination of the actual sample mass with 1 mg accuracy in pre-weighed stainless steel pan. Deionized water was added at a weight ratio of starch to water of 1:1. The sample pan was hermetically sealed and equilibrated for 1 day prior to heating in DSC from 20-180 ºC at 10 ºC/min. For pellets, the moisture content was determined and found to be 15±3% w/w, therefore, the moisture content of the sample in DSC pan was 65±3% w/w. Thermal properties of puffed products were determined only for those obtained from starch gels which were cooled at the rate of 5 °C/min and aged for 48 h. Puffed products were stored at 25 °C for 1 week before thermal property determination.

Empty pan was used as reference. The DSC was calibrated using indium as standard reference material. After heating, all sample pans were re-weighed.

3.8 Mechanical property, bulk density and expansion ratio determination

3.8.1 Gel

The gel obtained from 3.4 was determined the hardness. Hardness of gel was determined using Texture Analyzer with cylinder probe (P/25) with test speed of 1 mm/s up to a distance of 2 mm, using a trigger force of 5 g, and a post-test speed of 1 mm/s at compression rate of 1.0 mm/s. Twenty replications were determined for each treatment.

3.8.2 Pellet and puffed product

The pellet and puffed product hardness and fracturability were determined using Texture Analyzer with spherical probe (P/0.25s) with test speed of 1 mm/s, trigger force of 5 g, and post-test speed of 10 mm/s. The samples were punched through completely. The highest peak resistance force was reported as the hardness and first peak breaking force was reported as the fracturability (Noomhorm et al., 1997). Twenty replications were made for each treatment.

The bulk density of puffed product was determined using rapeseed displacement method (Park, 1976). Millet seed (Lemonfarm, Thailand) was used instead of rapeseed. A 250-ml beaker was filled with millet seed and the volume of millet seed was recorded by cylinder (V1). The puffed products were put in and then millet seed was full to the beaker while tapping gently, and the volume of replaced millet seed (V2) and weight of the puffed product (W) was recorded. Ten replications were performed for each sample. Bulk density (BD) was calculated as follows:

$$BD\ (g/ml) = W/(V1-V2)$$

Where W is the weight of sample (g), V1 is the initial volume of beaker (ml), and V2 is the volume of beaker with sample (ml).

The puffing characteristic in term expansion ratio (E) were calculated using Equation [1] (Saeleaw and Schleining, 2011). The width, length and thickness of both pellet and puffed product were measured using a digital vernier caliper (Series 500, Mitutoyo, Japan). The volume of puffed product and pellet was calculated from these dimensions.

$$E = \frac{\text{Volume of puffed product}}{\text{Volume of pellet}} \qquad [1]$$

3.9 Image of gel and puffed product

The gel was freeze dried (at -50°C and $50x10^{-3}$ mbar for 12 h) and prepared as described in Utrilla-Coello et al. (2013) prior to obtaining an image using SEM (JEOL: JSM-5800LV, Jeol Ltd., Tokyo, Japan). The image of the puffed product was taken by an image analyzer (Nikon SMZ 1000, Japan).

3.10 Descriptive sensory analysis

3.10.1 Panelists

A descriptive panel was composed of 10 Thai panelists (24-35 years old, 3 males and 7 females) who were graduate students in the Department of Food Technology at Chulalongkorn University, and who had taken at least one class in sensory evaluation and were willing to be panelists in this research.

3.10.2 Training and lexicon development

An intensive 60 h of descriptive sensory panel training, using a barrage of samples and sensory techniques through scaling (with a 15-point intensity scale) was conducted. The panelists discussed the sensory attributes of the puffed products. Eighteen attributes (roughness, porosity, puffiness, denseness, crispness, noise, hardness, crystallinity, brittleness, light, flakiness, tendency to crumble, cohesiveness, moisture absorption, residue, oiliness in the mouth, ease of swallowing, tooth stickiness) were generated in the vocabulary development session. The panelists discussed definitions and evaluation methods, to reduce redundancy through consensus. Finally, 11 sensory attributes and their respective reference intensities were established based on panel consensus. The panel was also discussed on the anchoring references for each attribute. Many puffed product samples from markets and lab-made samples (from the previous session) were reviewed.

Each selected reference product was well represented at least one of the 11 attributes from the vocabulary development session. For the 4 appearance attributes, five puffed lab-made samples were shown in Figure. 3.2. These 5 samples were resin coated for visual evaluation throughout the test sessions. For the 6 textural attributes and the after-taste, 9 commercial snack products (Table 3.1.) available in Thai markets were selected. Moreover, all references were quantified the intensity scores for the specific attributes by consensus. The lexicon will be shown in Section 4. The training focus on these 11 sensory attributes continued until all panelists were consistent in their evaluation.

Table 3.1 Reference products for sensory attributes

Brand	Snack Product Type	Manufacturer
Corn Puff	Corn starch	Friendship Co., Ltd., Bangkok, Thailand
Roller Coaster	Ring-shaped potato	URC Co., Ltd, Bangkok, Thailand
Poppo	Corn chips	Aiwthaisubrungreung Co., Ltd., Samutsakorn, Thailand
Tawan	Rice and corn starch	Pepsi-Cola (Thai) Trading, Lumpoon, Thailand
Pringle	Potato chips	The Procter and Gamble Company, Ohio, USA
Nalie	Potato cracker	Siangchun Foods Co., Ltd., Jongsan, China
Mashita	Fried-Seaweed	KJC Interfood, Co. Ltd., Pathumtani, Thailand
Bourbon	Rice cracker	Bourbon Co. Ltd., Nikata, Japan
Entrée	Crispy pork	S.Khonkaen Foods Public Co., Ltd., Bangkok, Thailand

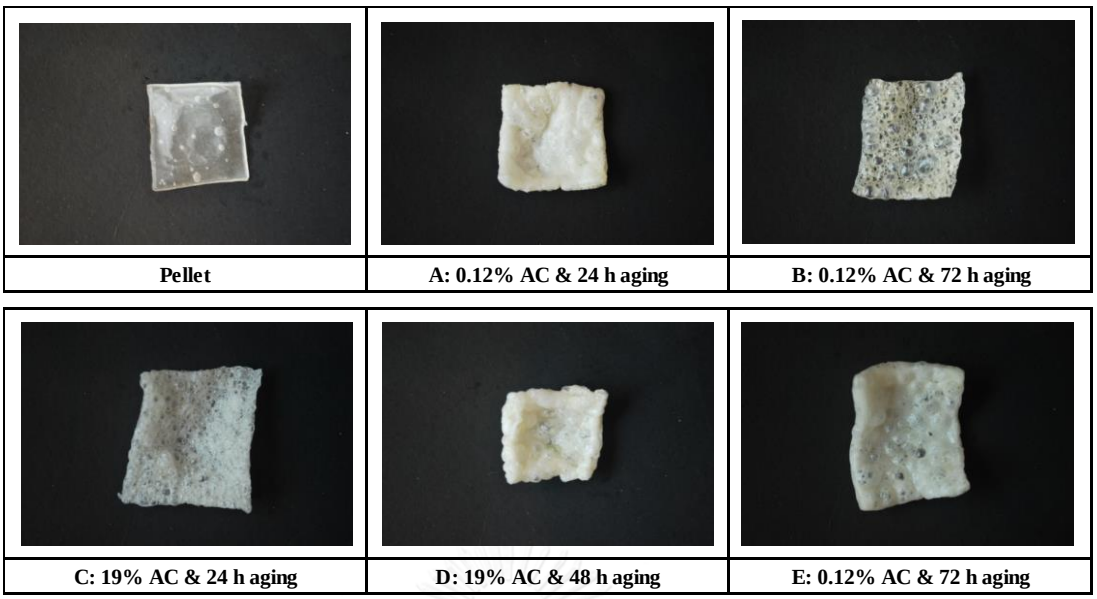


Figure 3.2 Images of puffed product references for appearance attributes

3.10.3 Product evaluation

Twenty seven puffed product samples were selected from the previous process for the descriptive evaluation. As a 3x3x3 factorial treatment, they were prepared by varying 3 levels of AC (4, 9, and 14%), 3 levels of cooling rate (3, 5, and 9 ºC/min), and 3 levels of aging time (24, 48, and 72 h). The evaluation of all 27 puffed product samples was conducted in 9 sessions taking approximately 36 h. Each sample was individually evaluated in duplicate over the evaluation session held on different days. The evaluation was conducted separately in a climate-controlled laboratory. Panelists evaluated the warm up samples and came to consensus through discussion before individual evaluation. For sample evaluation, 5 pieces of each test sample were presented in a white dish coded with three-digit numbers. All test samples were monadically served in random order to the panel. However, each panelist received the same samples in every serving. Each sample was evaluated for

11 attributes in 3 procedural stages for appearance, texture and after taste using 15-point numerical scales with anchored references. The references for each panelist were available throughout the sensory analysis. The samples were served at room temperature (25±1 ºC) and the evaluation was conducted under normal lighting conditions. The panelists cleansed their palates between samples with drinking water.

3.11 Validation of models

After the descriptive analysis, the 9 samples were selected for model validation and an acceptance test. As a 3x3 factorial treatment, they were prepared by varying 3 levels of AC (4, 9, and 14%), and 3 levels of aging time (24, 48, and 72 h). The RC of pellets and physical properties (hardness, fracturability, bulk density, and expansion ratio) of puffed product were also determined. These experimental values were compared to the predicted value which was calculated from RC using the developed models

3.12 Acceptance test

Sixty staff and students of the Faculty of Science in Chulalongkorn University who are puffed-snack eaters were recruited. The 9 test samples were randomly presented to each assessor in 2 sessions (5 samples and 4 samples). Three pieces of each sample in a white dish coded with 3-digit random number were presented in monadic servings to each assessor under normal lighting conditions at room temperature. Puffed products were evaluated for liking of appearance and texture using seven-point hedonic scales (1= like not at all to 7= like very much). Two attributes (crispness and hardness) were evaluated using 5-point just about right scales (1 = much too weak, 2 = little too weak, 3 = just right, 4 = little too strong, 5 = much

too strong). Drinking water was provided to clean and rinse the mouth between samples.

3.13 Statistical analysis

3.13.1 Crystallinity and thermal properties of gel and pellet and mechanical properties of pellets and puffed product

All experiments were done in duplicate. SPSS software version 17 (SPSS Inc., USA.) was used for statistical analysis. The analysis of variance (ANOVA) was used to determine the effects of factors and interactions. Duncan's new multiple range test was used to compare means (α=0.05).

3.13.1.1The crystallinity and thermal properties of gel and pellets and the mechanical properties of pellets and puffed product determination were conducted with 5x4x3 factorial in Completely Randomized Design (CRD). Regression models and correlations between RC of pellets and physical properties of pellet and the puffed product were performed.

3.13.1.2 Descriptive sensory analysis

The descriptive sensory evaluation was conducted with 3x3x3 factorial in Randomized Complete Block Designs (RCBD). The panelists were treated as blocks. Regression models for predicting puffed product properties from pellets' RC were established.

3.13.1.3 Acceptance test

The acceptance test was conducted with 3x3 factorial in Randomized Complete Block Designs (RCBD). Assessors were treated as blocks. Analysis of variance (ANOVA) was used to determine effects.

3.13.2 Prediction model and data validation

The pearson's correlations between instrumental and sensory properties were determined. The difference of mean values between the experimental data and the values from the prediction models were compared by t-test (α=0.05).

CHAPTER IV

RESULTS AND DISCUSSION

4.1 Chemical properties of rice starch

The AC was found to be 0.12% w/w dry starch for waxy rice starch and 19.00% w/w dry starch for non-waxy rice starch. The AC for their mixtures was found to be 4.00, 9.00, and 14.00% w/w dry starch for a weight ratio of waxy starch to non-waxy starch of 75:25, 50:50, and 25:75, respectively.

4.2. Starch gel and pellet

4.2.1 Crystallinity structure and relative crystallinity

The XRD patterns for all retrograded starch gels (Figures 4.1-4.4) and pellets (Figures 4.5-4.8) of 0.12% AC were typical for amorphous systems. This indicated that the crystallite was disrupted during gelatinization and starch molecules did not recrystallize during cooling and aging. For gels and pellets of AC $\geq$ 4.00%, there were peaks at 17º 2θ for the B-structure was attribute to amylose and the amylopectin recrystallization and 20º 2θ for the V-structure indicating existence of amylose-lipid complex and amylose single helix crystal (Koksel et al., 1993; Yu and Christie, 2005).

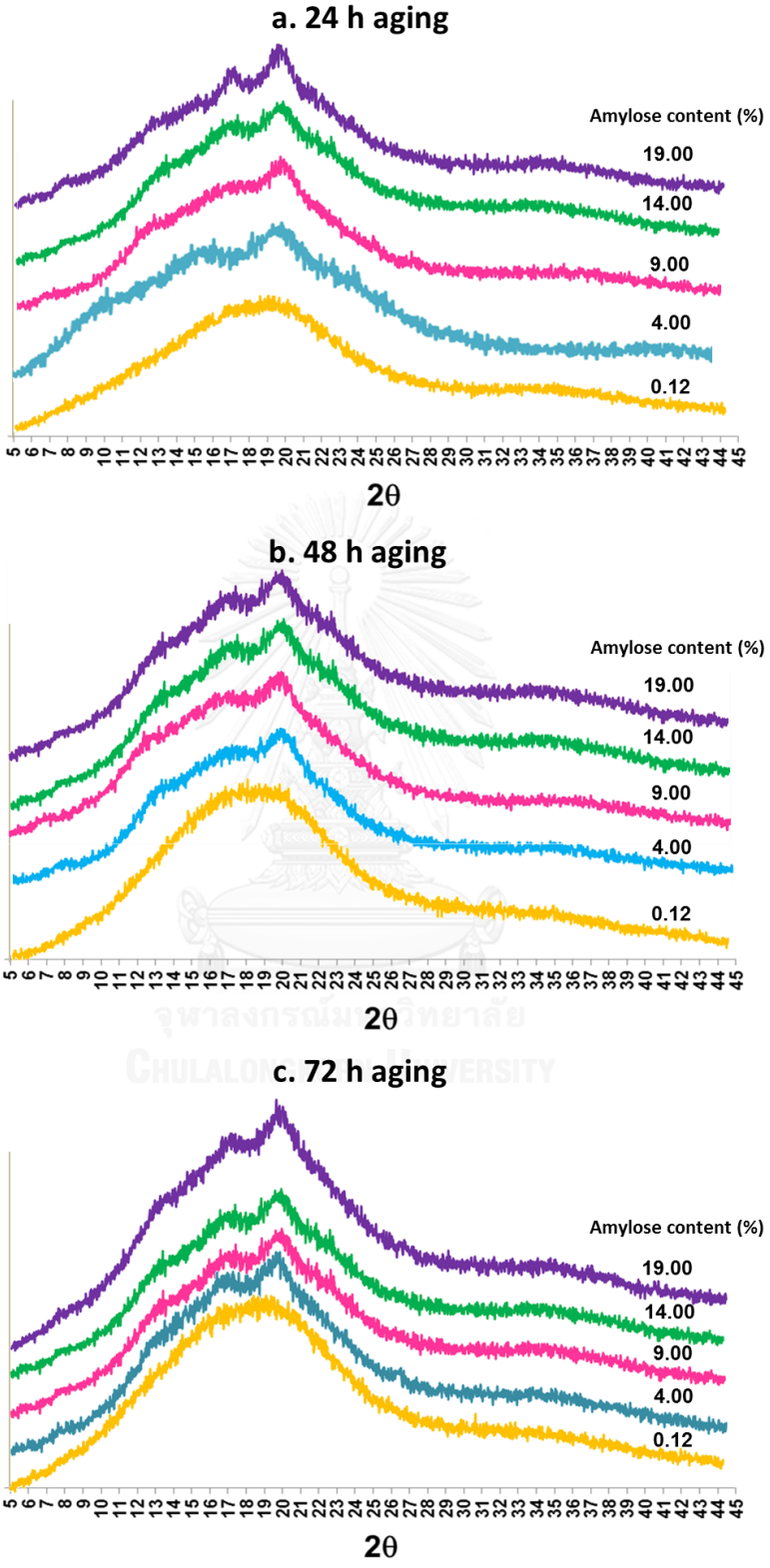


Figure 4.1 XRD pattern of rice starch gels cooled at 1 ºC/min.

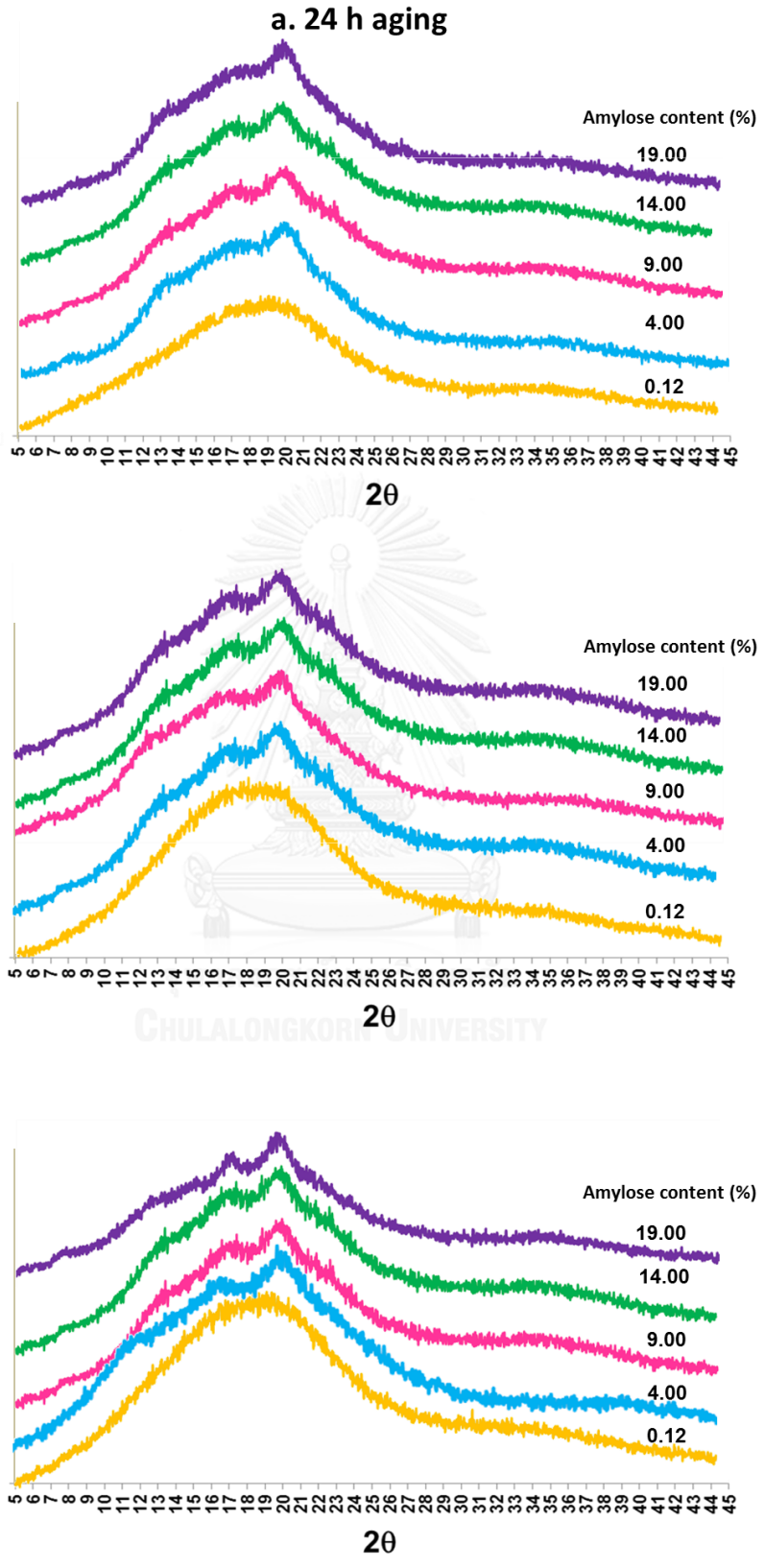


Figure 4.2 XRD pattern of rice starch gels cooled at 3 ºC/min.

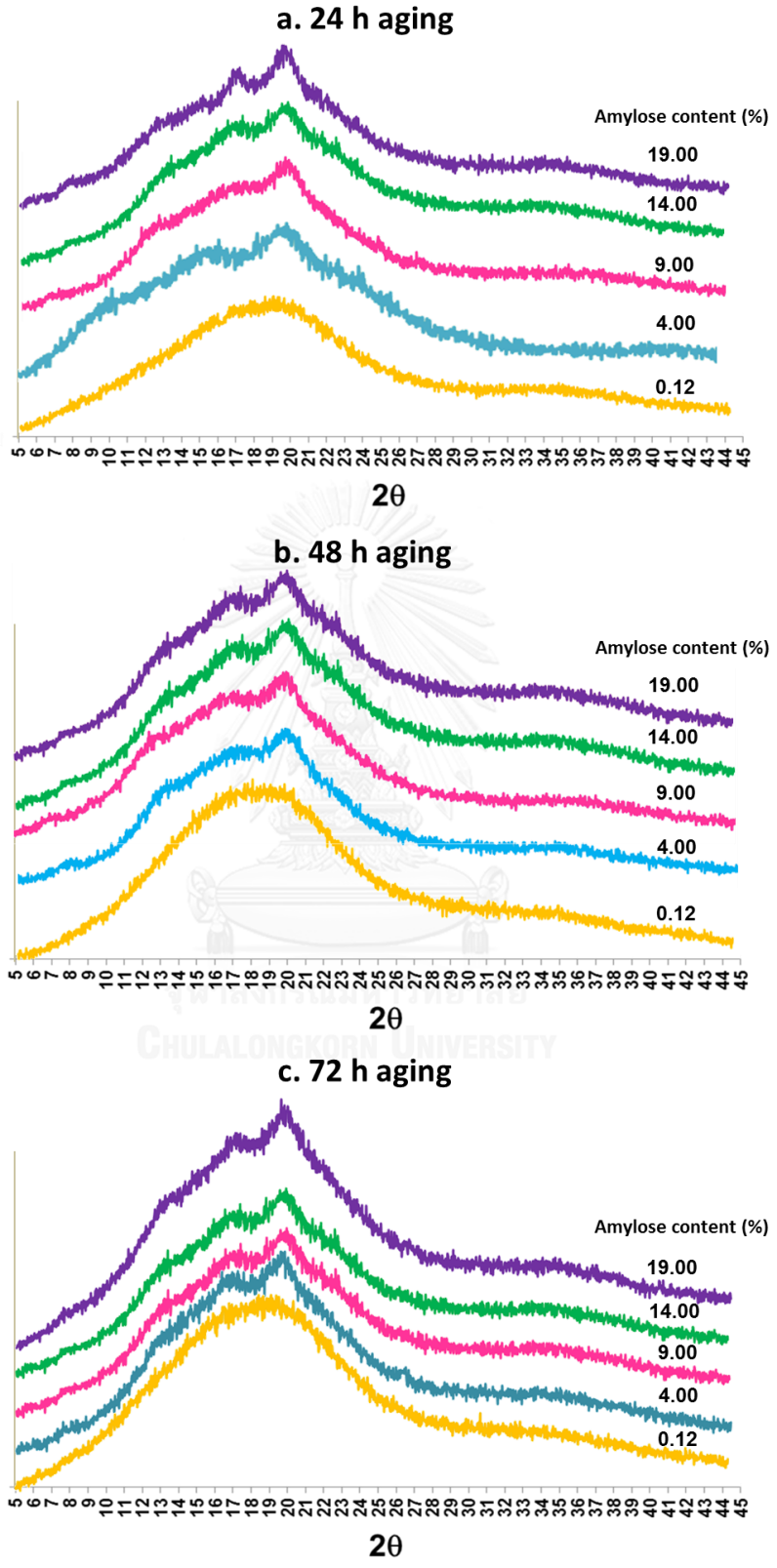


Figure 4.3 XRD pattern of rice starch gels cooled at 5 ºC/min.

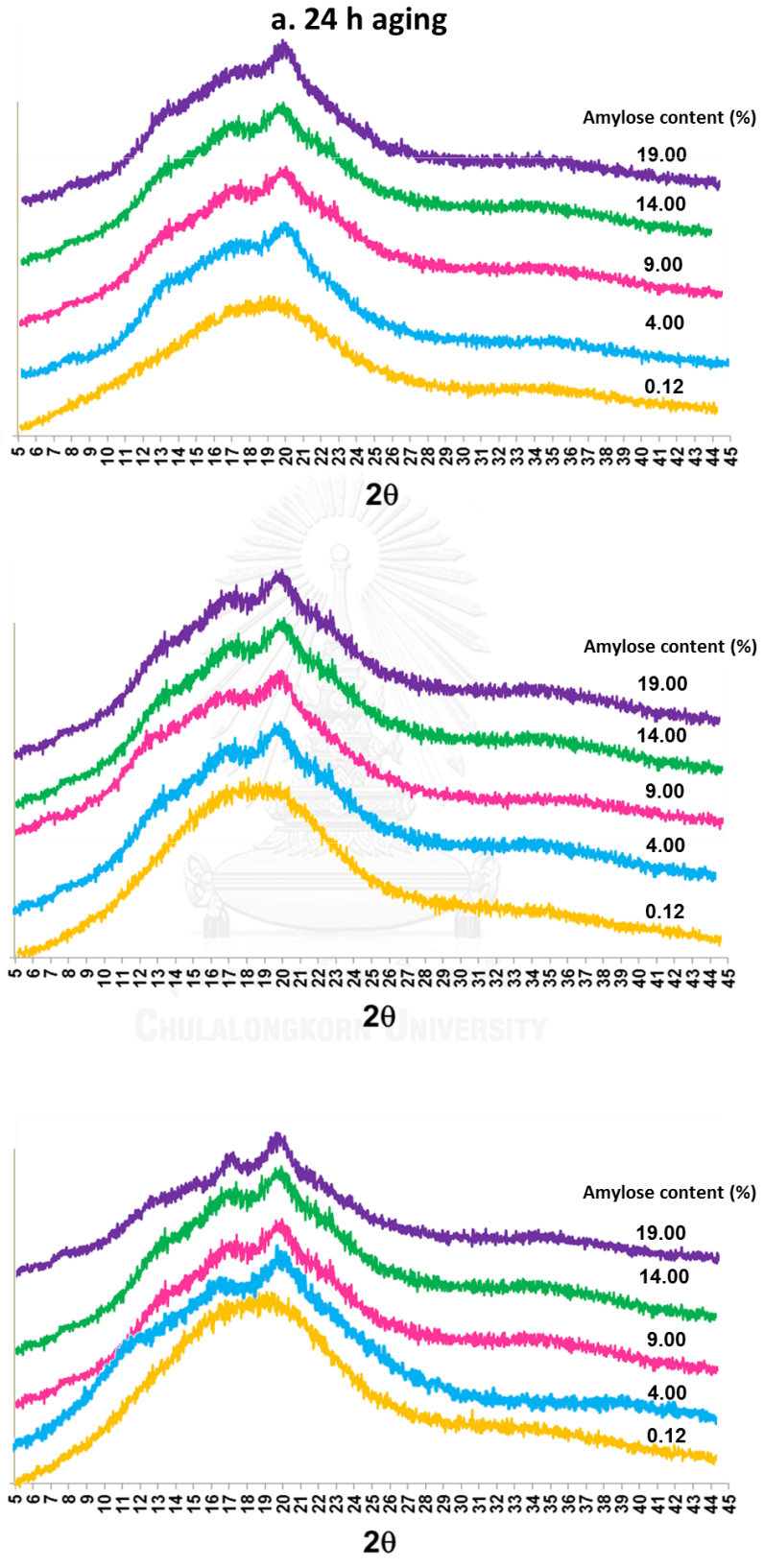


Figure 4.4 XRD pattern of rice starch gels cooled at 9 ºC/min.

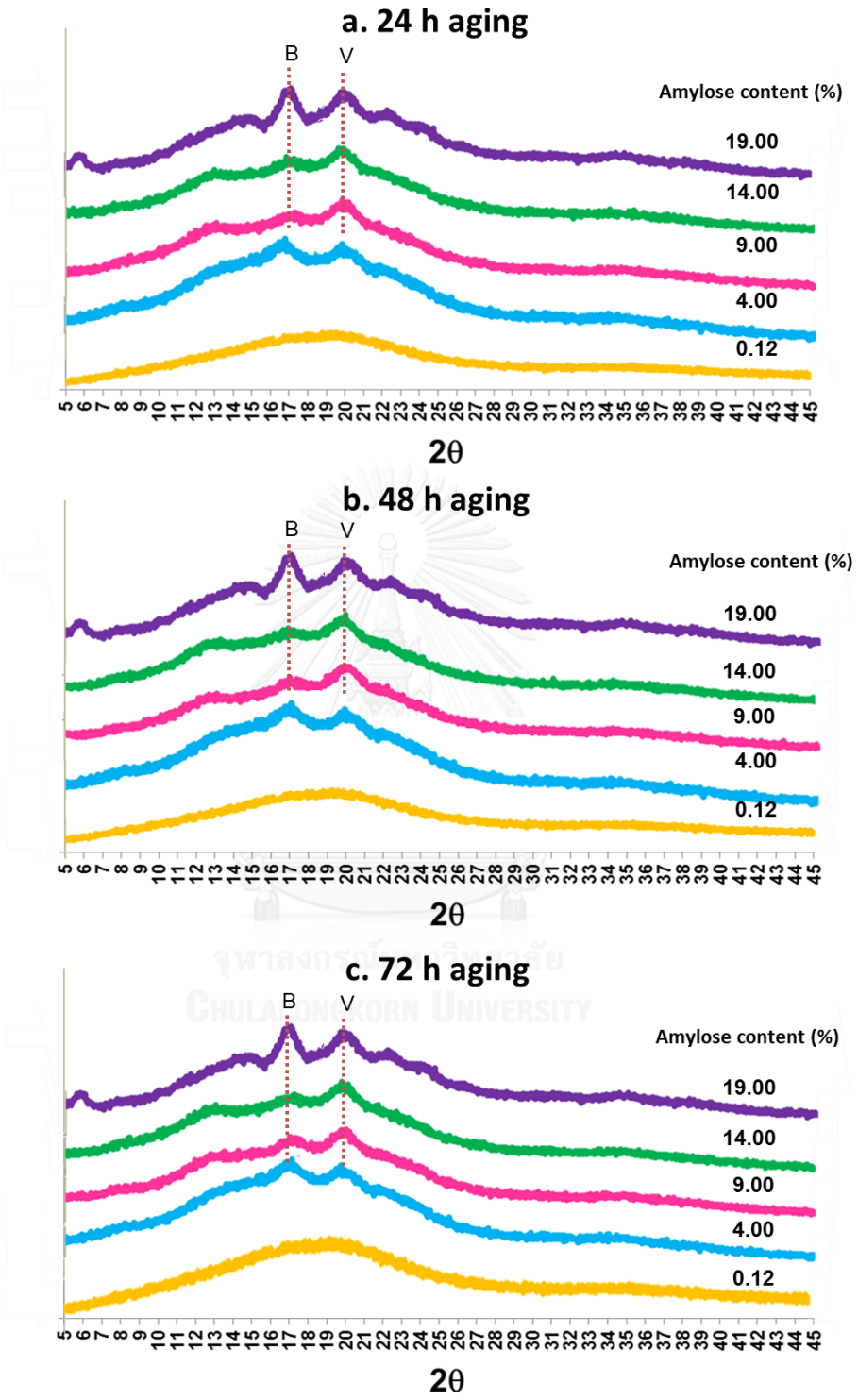


Figure 4.5 XRD pattern of pellets from starch gel cooled at 1 ºC/min.

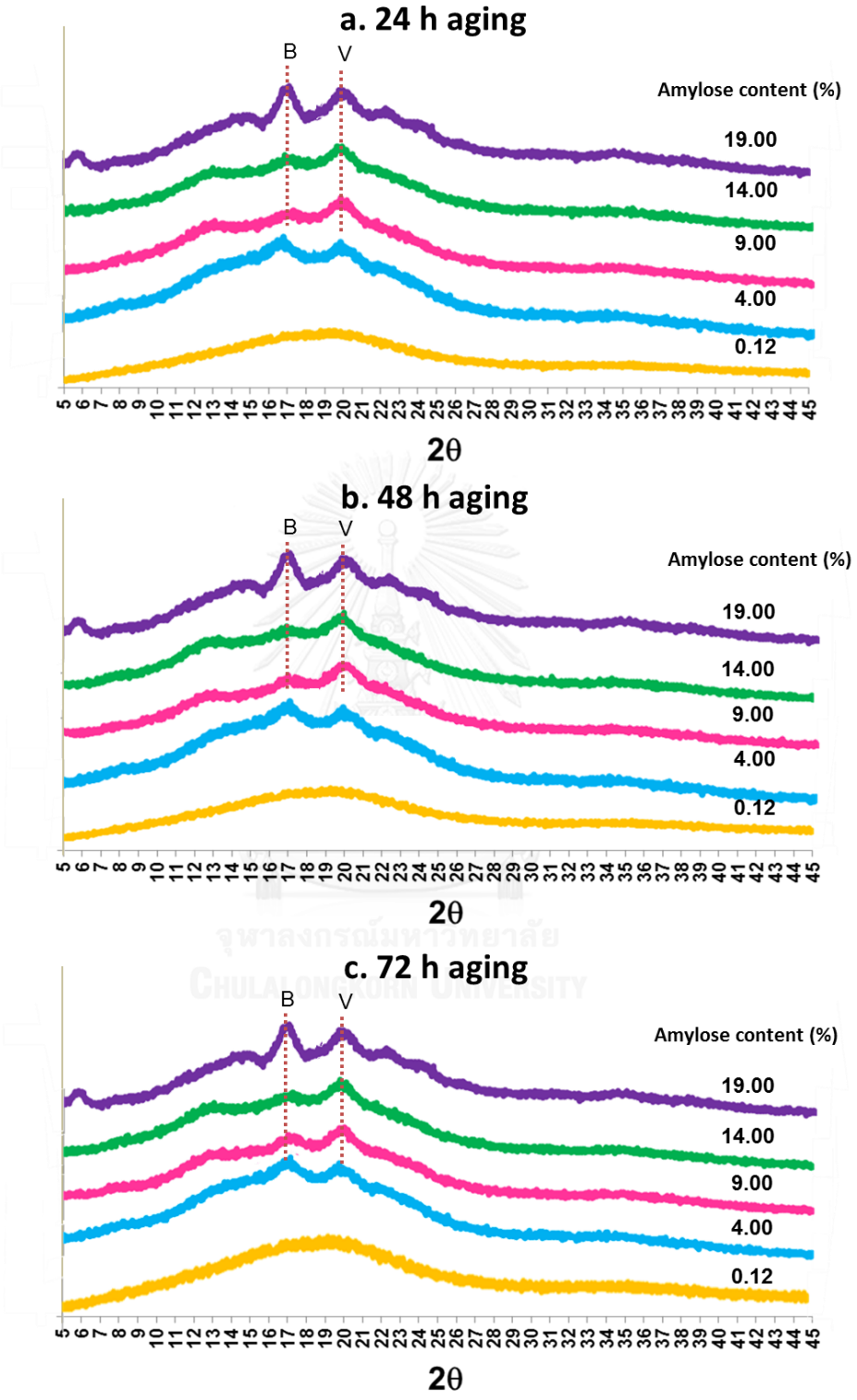


Figure 4.6 XRD pattern of pellets from starch gel cooled at 3 ºC/min.

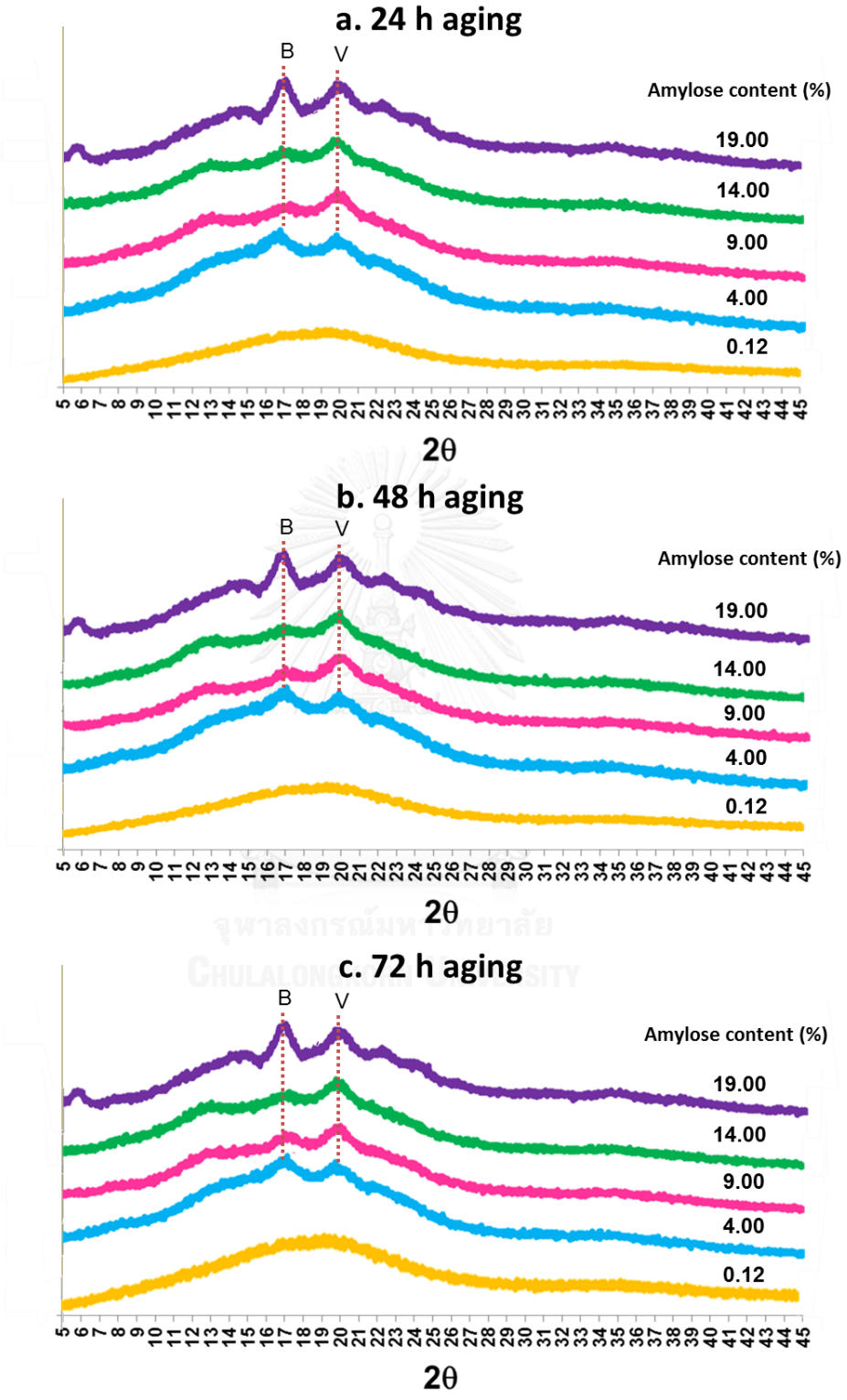


Figure 4.7 XRD pattern of pellets from starch gel cooled at 5 ºC/min.

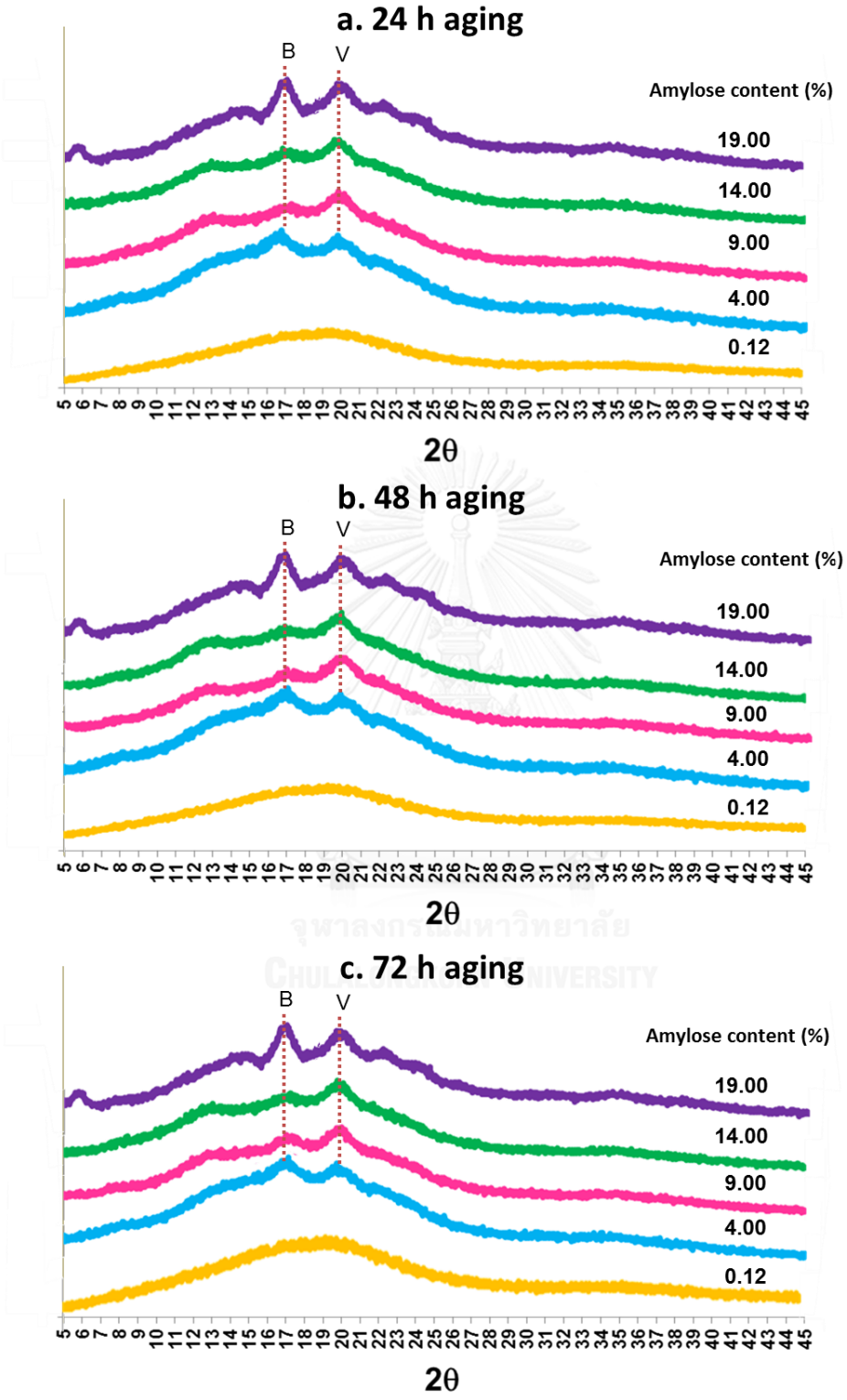
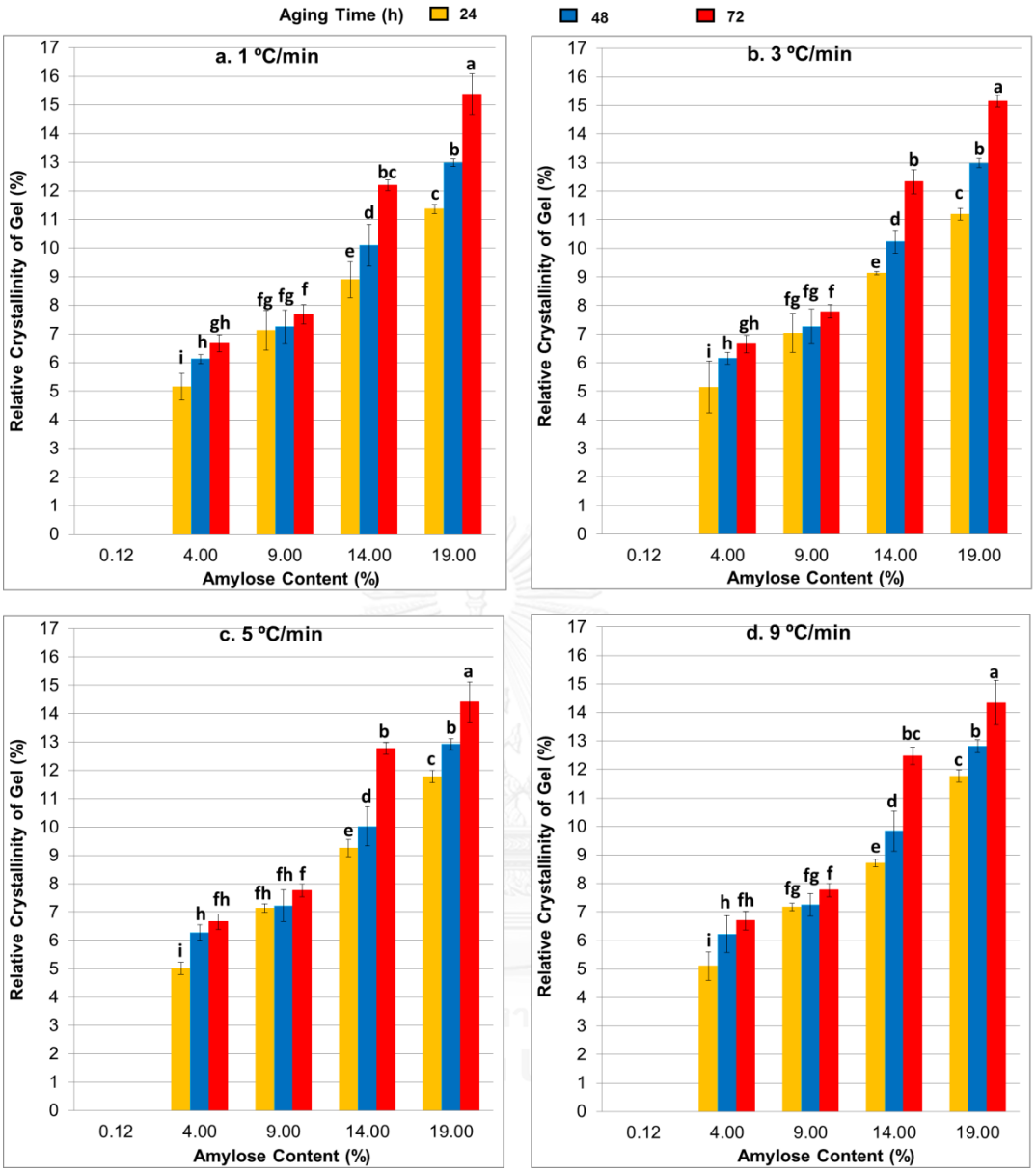


Figure 4.8 XRD pattern of pellets from starch gel cooled at 9 ºC/min.

Overall, the results showed that AC was the most significant factor affecting RC, while cooling rate had the least effect (as shown in Appendix C). Therefore the data were presented as the effect of AC, aging time and their interaction in each cooling rate.

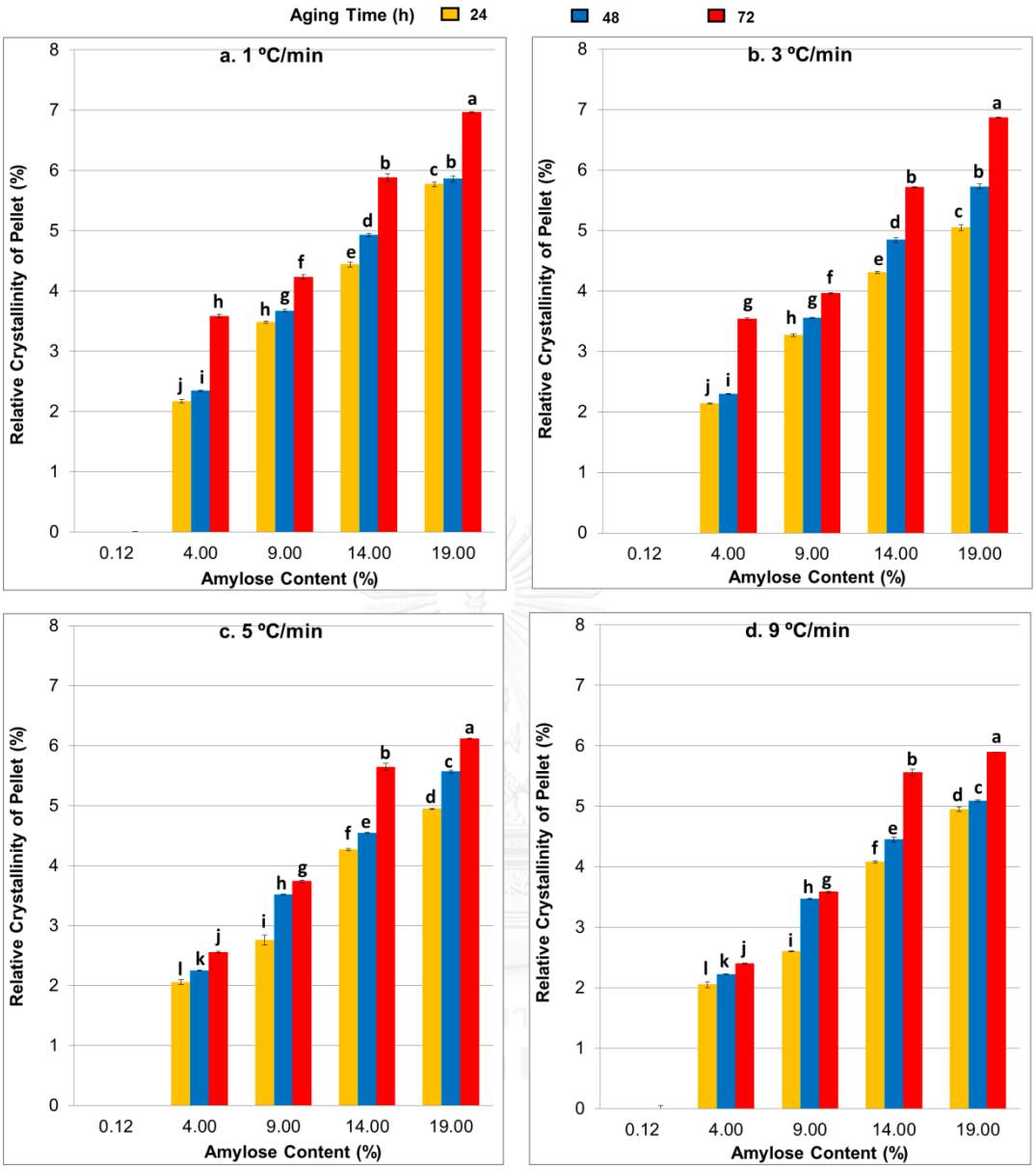
RC of both gel and pellet significantly increased with AC and aging time ($p \leq 0.05$) and its interaction as shown in Figure 4.9 and 4.10. For 0.12% AC, the RC was 0 and was not showed in these figures. All cooling rates, the samples with 14% and 19% AC showed the effect of aging time more than 4% and 9% AC samples by increasing RC. However, the RC of pellet was affected by cooling rate. The slower cooling rate resulted in higher RC of pellet as shown in Figure 4.10.

The well-ordered structure (crystalline) is detected by XRD. Compared to gel, pellet had lower RC suggesting that the order-disorder transition occurred during drying (Figure 4.11a). Moreover, the difference in RC between gel and pellet increased with AC. The result also showed that the RC of pellets strongly correlated to that of gels (Figure 4.11b).



The same letters are not significantly different ($p>0.05$) in each cooling rate.

Figure 4.9 Effects of amylose content and aging time at each cooling rate on relative crystallinity of gel.



The same letters are not significantly different ($p>0.05$) in each cooling rate.

Figure 4.10 Effects of amylose content and aging time at each cooling rate on relative crystallinity of pellet.

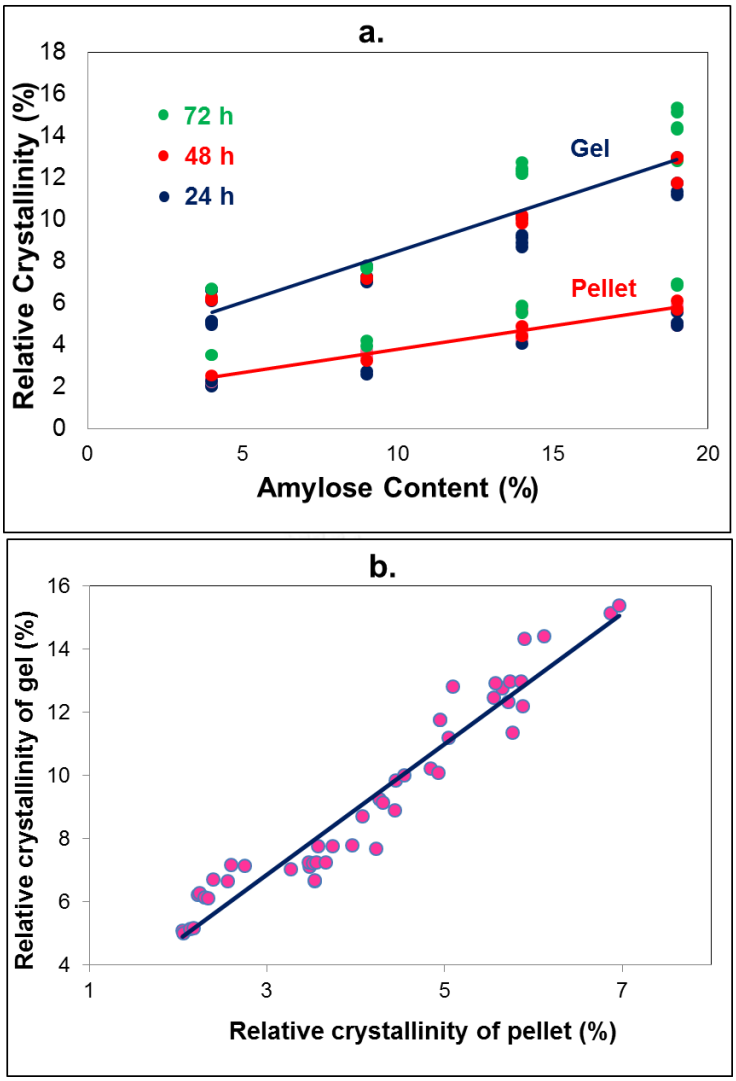


Figure 4.11 Effect of amylose content on relative crystallinity of gel and pellet (a) and relationship between relative crystallinity of gels and pellets (b).

After gelatinization, starch molecules were re-associated to generate new crystallites upon cooling and aging (Aguirre et al., 2011; Biliaderis, 2009; Lopez-Rubio et al., 2008; Putaux et al., 2000). The B-type and V-type structures were reported to be found in the retrograded rice starch gel (Shih et al., 2007). The B-type is the crystallization of the amylopectin and amylose (Miles et al., 1985) and the V-type is an amylose-lipid complex (Aguirre et al., 2011; Becker et al., 2001) and

amylose single helix crystal (Yu and Christie 2005). For waxy rice starch, the amylose-lipid complex was not found (Shih et al., 2007). Native rice starch was found to have A-type crystalline, which double helices of amylopectin pack tightly, while double helices of amylopectin in the retrograded rice starch (B-type) looser pack. Therefore, crystallite in retrograded starch was weaker than those of native starch. The RC increased with AC because amylose served as a nucleus for crystallization of amylopectin and amylose-amylopectin co-crystallization and amylose-amylose crystallization might occur (Smits et al., 1999; Vandeputte et al., 2003b). It was reported that amylose retrograded within an hour while amylopectin retrogradation took several days to retrograde (Aguirre et al., 2011; Ji et al., 2007; Orford et al., 1987). Therefore, RC increased with aging time because amylopectin retrogradation increased with aging time (Aguirre et al., 2011; Ji et al., 2007). The effect of cooling rate on RC was also expected because the cooling rate affected the mobility and rearrangement of starch molecules to form crystallites (Creek, 2007; Nordmark and Ziegler, 2002). However, the cooling rate used in this research did not affect RC ($p>0.05$).

4.2.2 Thermal properties

Examples of thermograms were shown for retrograded starch gels (Figure 4.12) and pellets (Figure 4.13) which were obtained at cooling rate of 5 ^{o}C/min and aging time of 24 h. From all obtained thermograms in Appendix D, thermal properties, T_o, T_p, T_c, ΔH_r and ΔH_{A-L}, for each endotherm were presented in Tables 4.1-4.8. No endotherm appeared for gels and pellets of 0.12% AC, except for those obtained at aging time of 72 h for every cooling rate which Endotherm I was found.

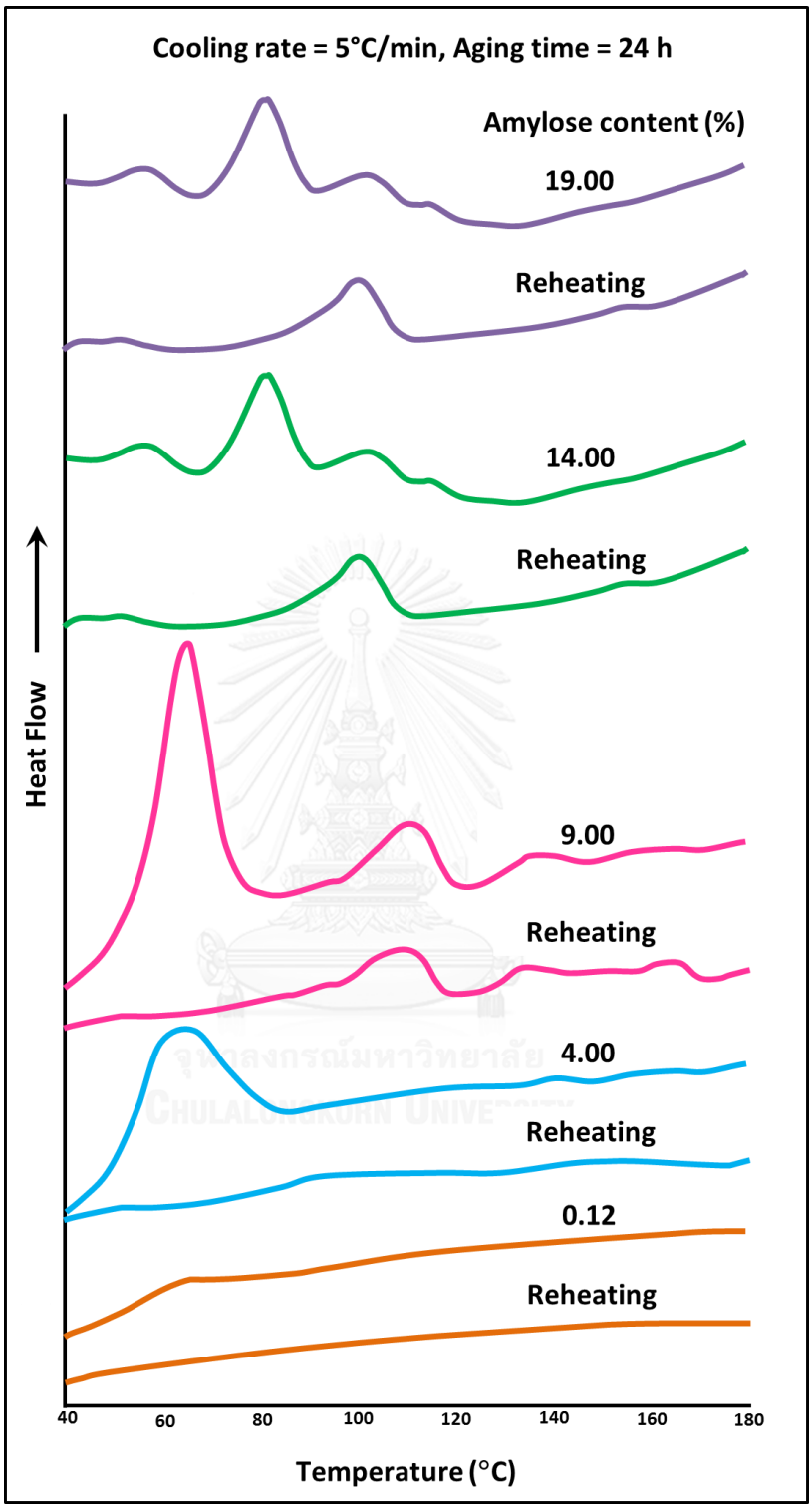


Figure 4.12 Thermograms of starch gel and reheating.

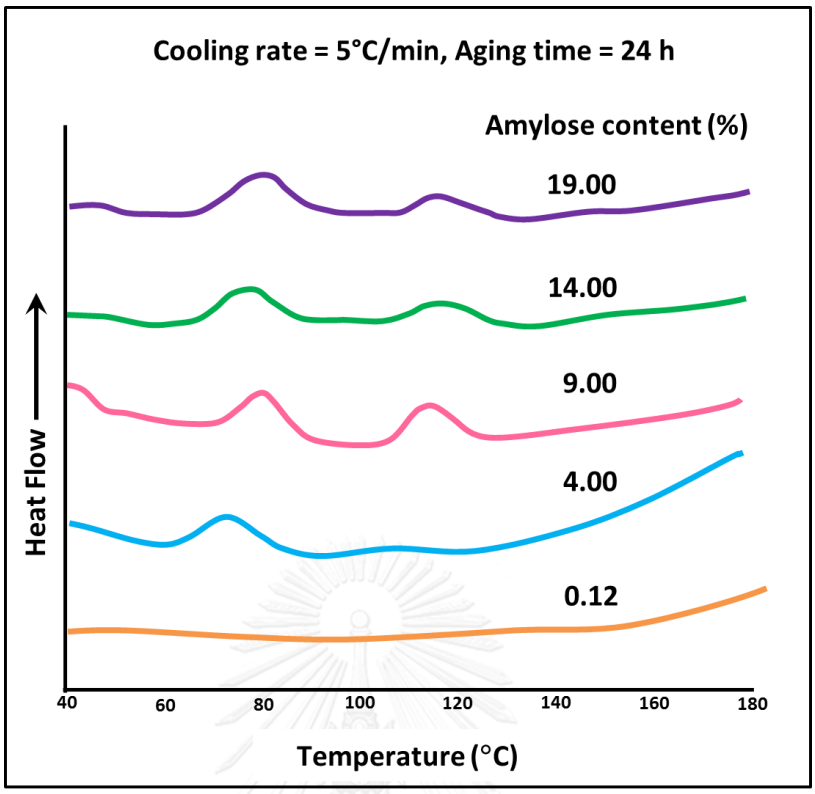


Figure 4.13 Thermograms of dried starch pellets.

For gel, Endotherm I (42-75 ºC) was found in all gel having AC≥4.00%. Endotherm II (71-88 C) was found for AC of ≥14.00% except for AC of 19.00% which cooled at 1 and 3 ºC/min and aged for 72 h. Endotherm III (86-121 ºC) was found in all gel having AC≥9.00%. Endotherm IV (110-120 ºC) was found in gel having AC≥14.00% except for 19.00% AC which cooled at 1 and 3 ºC/min and aged for 72 h. After reheating, only Endotherm III (85-121 ºC) appeared in every gels having AC of ≥9.00%. Endotherms I and II were melting of amylopectin retrogradation while Endotherms III and IV were melting of amylose-lipid complex. For all endotherms, there was no specific trend for change in T_o, T_p, T_c, T_c-T_o (ΔT), and ΔH_r with change in AC. However, gel having 9.00% AC had the highest T_p, T_c,

ΔT, and ΔH_r for endotherm I. At constant cooling rate, ΔHr of Endotherm II increased with AC and aging time. Because of the least significant effect of the cooling rate as shown in Appendix C., the results were shown as interactions between AC and aging time at each cooling rate on the thermal properties (Figures 4.14 and 4.15). Effects of all factors and their interactions were significant on the total ΔH_r (summation of ΔHr from Endotherms I and II, ΔH_{rt}). The ΔH_{rt} also increased as AC and aging time increased and vice versa for cooling rate. The interaction between AC and aging time ($P \leq 0.05$) was shown in Figure 4.11. For 19.00% AC, the ΔH_{rt} did not change as aging time increased from 24 to 48 h. Endotherm III and Endotherm IV resulted from the amylose-lipid complex which occurred at low and high temperatures, respectively (Le Bail et al., 1999; Liu et al., 2007; Primo-Martin et al., 2007). ΔH_{A-L} of Endotherm III increased with AC and aging time, while that of Endotherm IV was not affected by AC and aging time. Therefore, total ΔH_{A-L} (summation of ΔH_{A-L} from Endotherms III and IV) increased with AC and aging time.

For pellets, AC≥4.00%, Endotherms I and III appeared in the range of 51-77 ºC and 85-123 ºC, respectively. For Endotherm I, there was no specific trend for change in T_o, T_p, and T_c with change in AC. There were the effects of AC, aging time and cooling rate and interaction between AC and aging time on ΔH_{rt}. The effect of AC was the highest follow by aging time and cooling rate as shown in Appendix C. At constant cooling rate, ΔH_r significantly increased with AC and aging time ($p \leq 0.05$). The interaction between AC and aging time ($p \leq 0.05$) was showed in Figure 4.13, for pellet with 19.00% AC, there was no effect from aging time for 24 and 48 h on ΔH_r. T_o, T_p, T_c and ΔT significantly increased with AC and aging time. ΔH_{A-L} was

not affected by AC and aging time at cooling rate of ≤ 3 ºC/min, however, it increased with AC and aging time at faster cooling.

While XRD peaks indicate only crystalline structures, the endothermic peaks from DSC indicate the melting of both crystallines and aggregates in the samples (Biliaderis, 2009). The results from the thermograms conformed with those from the XRD patterns for both gels and pellets. The effects of AC and aging time on ΔH_{rt} can use the same explanations as the effects of AC aging time on RC. Endotherms I and II were reported to be attributed to a short-chain double helix amylopectin retrogradation and a long-chain double helix amylopectin retrogradation, respectively (Liu et al., 2007). It was reported that even under the gentlest condition, drying caused the starch granules to shrink and crack, and upon rehydration, dried starch did not resume its fully original structure (Grant, 1998). This might also happen when starch gel was dried and pellet was rehydrated. The higher or rapid cooling rate, the lower starch retrogradation. This was agreement with the RC and researches of Yu et al. (2010); and Nordmark and Ziegler (2002) who found faster cooling rates gave lower ΔH_r when compare to the slower cooling rates. At slow cooling rates, an increasing number of nuclei form as the system slowly cools, resulting in an interconnection of the polymer chains throughout the system (Creek, 2007). The XRD detects only well-order crystalline while the DSC thermogram obtains from both order crystalline and aggregation of crystal in samples. The slower cooling rate used in this study might provide the sufficient time only for formation of aggregates, which could be detected by DSC. Therefore, effect of cooling rate on ΔH_{rt} was detected.

Table 4.1 Thermal properties of rice starch gels cooled at 1 °C/min

Amylose Content (%)	Aging Time (h)	Endotherm I				Endotherm II				Endotherm III				Endotherm IV			
		T_o (°C)	T_p (°C)	T_c (°C)	ΔH_r (J/g)	T_o (°C)	T_p (°C)	T_c (°C)	ΔH_r (J/g)	T_o (°C)	T_p (°C)	T_c (°C)	ΔH_{A-L} (J/g)	T_o (°C)	T_p (°C)	T_c (°C)	ΔH_{A-L} (J/g)
0.12	24	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
4.00	24	53.52^{ab} (0.32)	61.27^{c} (0.33)	68.47^{c} (0.33)	2.61^{e} (0.15)	-	-	-	-	-	-	-	-	-	-	-	-
9.00	24	50.36^{de} (0.49)	62.69^{b} (0.28)	74.57^{a} (0.21)	6.45^{c} (0.73)	-	-	-	-	85.54^{g} (0.50)	103.93^{b} (0.06)	120.67^{a} (0.25)	1.00^{fg} (0.01)	-	-	-	-
14.00	24	54.21^{a} (0.27)	59.25^{de} (0.29)	63.73^{d} (0.19)	1.13^{c} (0.03)	76.25^{a} (0.25)	81.25^{ab} (0.28)	87.34^{a} (0.26)	4.79^{e} (0.06)	93.63^{d} (0.18)	102.47^{d} (0.45)	107.84^{e} (0.16)	1.25^{de} (0.01)	112.26^{a} (0.37)	115.03^{b} (0.01)	119.31^{b} (0.20)	0.31^{a} (0.07)
19.00	24	53.67^{ab} (0.13)	58.67^{de} (0.21)	63.77^{d} (0.19)	1.29^{f} (0.36)	71.05^{c} (0.59)	81.39^{a} (0.14)	87.88^{a} (0.47)	5.76^{b} (0.09)	94.87^{c} (0.43)	102.53^{cd} (0.74)	107.91^{e} (0.71)	1.52^{bc} (0.02)	112.21^{a} (0.50)	115.59^{a} (0.07)	120.57^{c} (0.06)	0.32^{a} (0.03)
0.12	48	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
4.00	48	49.47^{e} (0.11)	59.50^{d} (0.52)	68.55^{c} (0.33)	5.02^{d} (0.33)	-	-	-	-	-	-	-	-	-	-	-	-
9.00	48	50.45^{d} (0.62)	62.54^{b} (0.59)	74.29^{a} (0.19)	6.42^{c} (0.13)	-	-	-	-	89.43^{f} (0.30)	103.62^{b} (0.08)	117.82^{b} (0.24)	1.08^{fg} (0.14)	-	-	-	-
14.00	48	51.56^{c} (0.56)	57.20^{f} (0.11)	63.23^{d} (0.31)	1.30^{f} (0.04)	75.15^{b} (0.04)	81.15^{ab} (0.16)	87.67^{a} (0.17)	5.32^{d} (0.06)	96.73^{b} (0.18)	103.29^{bc} (0.23)	108.76^{d} (0.29)	1.33^{cd} (0.05)	112.44^{a} (0.33)	115.61^{a} (0.08)	118.33^{c} (0.43)	0.29^{a} (0.05)
19.00	48	51.73^{c} (0.27)	57.22^{f} (0.14)	63.21^{d} (0.28)	1.53^{f} (0.23)	71.25^{c} (0.24)	80.67^{b} (0.14)	87.35^{a} (0.28)	6.02^{a} (0.09)	96.34^{b} (0.44)	102.37^{d} (0.14)	108.92^{d} (0.04)	1.56^{b} (0.07)	112.35^{a} (0.62)	115.40^{a} (0.28)	119.21^{b} (0.01)	0.30^{a} (0.01)
0.12	72	50.37^{de} (0.26)	58.26^{e} (1.15)	63.47^{d} (1.32)	1.31^{f} (0.02)	-	-	-	-	-	-	-	-	-	-	-	-
4.00	72	54.39^{a} (0.64)	63.09^{b} (0.11)	71.30^{b} (0.86)	5.23^{d} (0.31)	-	-	-	-	-	-	-	-	-	-	-	-
9.00	72	50.54^{d} (0.13)	64.61^{a} (0.35)	75.29^{a} (0.24)	7.13^{b} (0.17)	-	-	-	-	98.65^{a} (0.00)	111.31^{a} (0.03)	116.73^{c} (0.35)	0.81^{ef} (0.27)	-	-	-	-
14.00	72	51.51^{c} (0.65)	57.17^{f} (0.16)	63.30^{d} (0.22)	1.36^{f} (0.07)	75.21^{b} (0.08)	81.30^{ab} (0.37)	87.32^{a} (0.37)	5.53^{c} (0.27)	96.39^{b} (0.29)	103.30^{bc} (0.25)	109.34^{d} (0.45)	1.38^{bcd} (0.00)	112.41^{a} (0.25)	115.52^{a} (0.04)	118.88^{b} (0.04)	0.27^{a} (0.05)
19.00	72	53.14^{b} (0.17)	63.26^{b} (0.26)	72.27^{b} (0.17)	8.78^{a} (0.28)	-	-	-	-	90.77^{e} (0.69)	103.32^{bc} (0.28)	116.60^{c} (0.22)	1.76^{a} (0.17)	-	-	-	-

Means followed by the same superscript in each column are not significantly different ($p>0.05$).
Values shown in parentheses are standard deviation.

Table 4.2 Thermal properties of rice starch gels cooled at 3 °C/min

Amylose Content (%)	Aging Time (h)	Endotherm I				Endotherm II				Endotherm III				Endotherm IV			
		T_o (°C)	T_p (°C)	T_c (°C)	ΔH_r (J/g)	T_o (°C)	T_p (°C)	T_c (°C)	ΔH_r (J/g)	T_o (°C)	T_p (°C)	T_c (°C)	ΔH_{A-L} (J/g)	T_o (°C)	T_p (°C)	T_c (°C)	ΔH_{A-L} (J/g)
0.12	24	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
4.00	24	54.25$_b$ (0.38)	61.22$_c$ (0.23)	68.00$_f$ (0.11)	2.36$_e$ (0.01)	-	-	-	-	-	-	-	-	-	-	-	-
9.00	24	51.69$_d$ (0.25)	63.22$_b$ (0.28)	74.24$_a$ (0.31)	6.30$_b$ (0.52)	-	-	-	-	88.59$_e$ (0.46)	103.43$_c$ (0.04)	116.58$_c$ (0.30)	1.00$_a$ (0.01)	-	-	-	-
14.00	24	54.27$_b$ (0.21)	59.23$_d$ (0.27)	63.47$_i$ (0.49)	1.10$_f$ (0.02)	76.04$_a$ (0.04)	81.25$_a$ (0.28)	87.31$_b$ (0.21)	4.79$_e$ (0.03)	93.65$_c$ (0.19)	102.30$_e$ (0.21)	107.77$_g$ (0.11)	1.25$_a$ (0.02)	111.53$_b$ (0.39)	115.14$_c$ (0.17)	119.34$_c$ (0.45)	0.35$_a$ (0.02)
19.00	24	50.97$_d$ (0.47)	58.31$_e$ (0.21)	64.75$_g$ (0.25)	1.28$_f$ (0.35)	70.92$_c$ (0.23)	81.12$_a$ (0.16)	87.95$_a$ (0.18)	5.72$_b$ (0.05)	91.84$_d$ (0.35)	102.63$_{cd}$ (0.14)	109.12$_{ef}$ (0.16)	1.47$_a$ (0.02)	111.30$_b$ (0.50)	115.57$_b$ $_c$(0.01)	120.58$_b$ (0.05)	0.31$_{ab}$ (0.01)
0.12	48	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
4.00	48	45.63$_h$ (0.37)	59.04$_{de}$ (0.77)	69.33e (0.23)	3.63$_d$ (0.23)	-	-	-	-	-	-	-	-	-	-	-	-
9.00	48	51.35$_d$ (0.38)	63.37$_b$ (0.50)	73.64$_b$ (0.26)	6.45$_b$ (0.18)	-	-	-	-	89.31$_e$ (0.16)	104.20$_b$ (0.09)	120.49$_a$ (0.65)	1.02$_a$ (0.10)	-	-	-	-
14.00	48	49.54$_e$ (0.39)	57.28$_f$ (0.35)	64.39$_{gh}$ (0.23)	1.23$_f$ (0.11)	73.47$_b$ (0.03)	81.41$_a$ (0.48)	88.30$_a$ (0.25)	5.09$_d$ (0.06)	93.66$_c$ (0.11)	103.43$_c$ (0.59)	109.61$_e$ (0.04)	1.22$_a$ (0.10)	111.89$_{ab}$ (0.08)	115.64$_b$ (0.23)	120.18$_b$ (0.25)	0.24$_b$ (0.11)
19.00	48	47.57$_g$ (0.30)	57.20$_f$ (0.21)	64.18$_h$ (0.12)	1.35$_f$ (0.37)	72.74$_b$ (0.68)	81.23$_a$ (0.32)	88.27$_a$ (0.21)	5.97$_a$ (0.14)	93.73$_c$ (0.34)	103.18$_{cd}$ (0.06)	109.42$_e$ (0.07)	1.44$_a$ (0.25)	110.43$_c$	116.22$_a$ (0.07)	121.86$_a$ (0.02)	0.22$_b$ (0.13)
0.12	72	55.59$_a$ (0.20)	59.14$_d$ (0.18)	62.47$_j$ (0.04)	1.18$_f$ (0.11)	-	-	-	-	-	-	-	-	-	-	-	-
4.00	72	48.43$_f$ (0.40)	59.50$_d$ (0.47)	69.87$_d$ (0.22)	5.56$_c$ (0.07)	-	-	-	-	-	-	-	-	-	-	-	-
9.00	72	53.39$_c$ (0.45)	64.62$_a$ (0.19)	74.43$_a$ (0.28)	6.54$_b$ (0.64)	-	-	-	-	95.48$_b$ (0.14)	107.43$_a$ (0.04)	117.47$_b$ (0.31)	1.09$_a$ (0.58)	-	-	-	-
14.00	72	51.45d (0.60)	57.36f (0.11)	63.09i (0.11)	1.35f (0.00)	75.29a (0.19)	81.24a (0.30)	87.74ab (0.22)	5.40c (0.09)	96.47$_a$ (0.45)	103.36$_c$ (0.33)	108.72$_f$ (0.18)	1.25$_a$ (0.10)	112.31$_a$ (0.11)	115.75$_b$ (0.28)	118.32$_d$ (0.45)	0.27$_{ab}$ (0.04)
19.00	72	54.19$_{bc}$ (0.25)	61.12$_c$ (0.13)	71.55$_c$ (0.13)	8.52$_a$ (0.59)	-	-	-	-	87.49$_f$ (0.41)	103.52$_c$ (0.09)	115.42$_d$ (0.04)	1.43$_a$ (0.41)	-	-	-	-

Means followed by the same superscript in each column are not significantly different ($p>0.05$).
Values shown in parentheses are standard deviation.

Table 4.3 Thermal properties of rice starch gels cooled at 5 °C/min

Amylose Content (%)	Aging Time (h)	Endotherm I				Endotherm II				Endotherm III				Endotherm IV			
		T_o (°C)	T_p (°C)	T_c (°C)	ΔH_r (J/g)	T_o (°C)	T_p (°C)	T_c (°C)	ΔH_r (J/g)	T_o (°C)	T_p (°C)	T_c (°C)	ΔH_{A-L} (J/g)	T_o (°C)	T_p (°C)	T_c (°C)	ΔH_{A-L} (J/g)
0.12	24	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
4.00	24	48.48f (0.41)	58.36d (0.17)	71.06b (0.59)	2.43d (0.14)	-	-	-	-	-	-	-	-	-	-	-	-
9.00	24	53.66bc (0.28)	59.55c (0.14)	66.26d (0.34)	5.63bc (0.37)	-	-	-	-	97.44bc (0.54)	104.28b (0.24)	110.67b (0.28)	0.88cd (0.13)	-	-	-	-
14.00	24	42.46g (0.28)	58.70d (0.35)	65.12e (0.14)	0.99g (0.04)	74.95a (0.01)	81.34a (0.12)	88.13ab (0.16)	4.71f (0.01)	93.25f (0.29)	101.72d (0.33)	108.59de (0.12)	1.19b (0.07)	111.18b (0.12)	115.51a (0.91)	120.33 a (0.18)	0.30a (0.11)
19.00	24	53.27c (0.25)	58.34d (0.21)	63.67f (0.23)	1.37ef (0.10)	71.09b (0.36)	81.31a (0.23)	88.19a (0.60)	5.58c (0.07)	94.91e (0.12)	102.21d (0.04)	108.16e (0.05)	1.42a (0.06)	111.7ab (0.40)	115.56a (0.09)	120.46 a (0.73)	0.33a (0.05)
0.12	48	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
4.00	48	54.2b (0.12)	61.16b (0.16)	67.39c (0.67)	2.78d (0.10)	-	-	-	-	-	-	-	-	-	-	-	-
9.00	48	53.40c (0.22)	59.54c (0.16)	67.13c (0.13)	6.00c (0.35)	-	-	-	-	97.73b (0.13)	104.45b (0.47)	107.25f (0.31)	0.70d (0.12)	-	-	-	-
14.00	48	51.47e (0.58)	57.16e (0.18)	63.27f (0.25)	1.25ef (0.14)	70.44b (0.30)	81.28a (0.23)	88.41a (0.21)	5.09e (0.01)	96.73cd (0.21)	103.44c (0.45)	108.51e (0.21)	1.18b (0.12)	112.36a (0.18)	115.62a (0.09)	118.71 c (0.10)	0.29a (0.05)
19.00	48	51.69e (0.38)	57.20e (0.18)	63.22f (0.29)	1.54e (0.14)	70.64b (0.84)	80.61bc (0.23)	87.31b (0.30)	5.58b (0.07)	96.29d (0.37)	102.22d (0.07)	108.85d (0.35)	1.54a (0.09)	112.48a (0.57)	115.19b (0.01)	119.8b (0.80)	0.24a (0.10)
0.12	72	48.23f (0.26)	57.25e (0.28)	65.66e (0.01)	1.03g (0.00)	-	-	-	-	-	-	-	-	-	-	-	-
4.00	72	53.37c (0.32)	59.35c (0.42)	67.05c (0.12)	5.29c (0.35)	-	-	-	-	-	-	-	-	-	-	-	-
9.00	72	55.22a (0.28)	64.60a (0.49)	72.46a (0.13)	6.89a (0.08)	-	-	-	-	98.56a (0.42)	108.74a (0.40)	112.63a (0.30)	0.92c (0.25)	-	-	-	-
14.00	72	51.55e (0.58)	57.15e (0.18)	63.30f (0.21)	1.34ef (0.04)	75.30a (0.17)	81.15ab (0.16)	87.74ab (0.25)	5.32d (0.05)	96.42d (0.38)	103.29c (0.23)	109.21c (0.11)	1.34b (0.04)	112.49a (0.37)	115.56a (0.01)	118.64 bc (0.30)	0.27ab (0.04)
19.00	72	52.47d (0.13)	57.25e (0.28)	61.58g (0.48)	1.45ef (0.28)	74.54a (0.77)	80.37c (0.42)	87.31b (0.16)	6.35a (0.02)	96.13d (0.16)	103.39c (0.35)	108.48e (0.16)	1.51a (0.00)	112.52a (0.18)	115.20b (0.00)	118.37 c (0.01)	0.16b (0.08)

Means followed by the same superscript in each column are not significantly different ($p>0.05$).
Values shown in parentheses are standard deviation.

Table 4.4 Thermal properties of rice starch gels cooled at 9 °C/min

Amylose Content (%)	Aging Time (h)	Endotherm I				Endotherm II				Endotherm III				Endotherm IV			
		T_o (ºC)	T_p (ºC)	T_c (ºC)	ΔH_r (J/g)	T_o (ºC)	T_p (ºC)	T_c (ºC)	ΔH_r (J/g)	T_o (ºC)	T_p (ºC)	T_c (ºC)	ΔH_{A-L} (J/g)	T_o (ºC)	T_p (ºC)	T $(ºC)_c$	ΔH_{A-L} (J/g)
0.12	24	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
4.00	24	54.78a (0.48)	61.18b (0.13)	66.26d (0.14)	1.99d (0.06)	-	-	-	-	-	-	-	-	-	-	-	-
9.00	24	49.63c (0.49)	59.56c (0.42)	68.47c (0.53)	5.26b (0.62)	-	-	-	-	93.28d (0.24)	103.39b (0.01)	111.61b (0.06)	0.60d (0.05)	-	-	-	-
14.00	24	49.23c (0.30)	58.15ef (0.11)	65.36e (0.21)	0.90e (0.12)	72.52c (0.10)	81.47a (0.23)	89.24ab (0.13)	4.17e (0.02)	91.31e (0.20)	101.42e (0.04)	108.47de (0.02)	1.25abc (0.01)	111.61b (0.18)	116.51a (0.02)	121.49a (0.14)	0.26ab (0.18)
19.00	24	53.16b (0.18)	58.54de (0.49)	63.75gh (0.14)	1.39de (0.05)	75.46a (0.52)	81.39ab (0.11)	87.69bc (0.13)	5.67b (0.14)	94.39c (0.49)	102.21d (0.04)	108.07e (0.22)	1.38ab (0.21)	111.99ab (0.06)	115.57b (0.07)	118.67c (0.12)	0.34a (0.06)
0.12	48	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
4.00	48	47.85e (0.17)	58.99cd (0.47)	71.40a (0.47)	2.74c (0.15)	-	-	-	-	-	-	-	-	-	-	-	-
9.00	48	48.38de (0.50)	59.68c (0.36)	69.55b (0.18)	5.98a (0.29)	-	-	-	-	93.63cd (0.25)	103.36b (0.15)	111.45b (0.33)	0.60e (0.05)	-	-	-	-
14.00	48	47.74e (0.25)	57.12g (0.12)	64.84ef (0.12)	1.16e (0.02)	69.45d (0.13)	81.38ab (0.37)	89.38a (0.37)	4.90d (0.08)	96.64a (0.40)	103.36b (0.33)	108.71d (0.35)	1.25bc (0.01)	112.46a (0.28)	115.65b (0.14)	118.56c (0.08)	0.26ab (0.11)
19.00	48	49.02cd (0.46)	57.03g (0.13)	64.35f (0.19)	1.45de (0.25)	72.85c (0.03)	81.27ab (0.35)	87.50c (1.46)	5.78b (0.07)	93.38d (0.36)	102.51cd (0.18)	109.33c (0.04)	1.38abc (0.21)	110.27c (0.23)	115.57b (0.08)	120.16b (0.01)	0.24ab (0.07)
0.12	72	49.52c (0.29)	57.47fg (0.42)	63.15h (0.03)	0.95e (0.01)	-	-	-	-	-	-	-	-	-	-	-	-
4.00	72	48.39de (0.49)	59.46c (0.64)	69.22b (0.25)	5.23b (0.31)	-	-	-	-	-	-	-	-	-	-	-	-
9.00	72	54.09a (0.11)	62.30a (0.27)	71.41a (0.28)	6.50a (0.58)	-	-	-	-	95.80b(0.21)	104.61a (0.25)	112.45a (0.43)	0.82d (0.08)	-	-	-	-
14.00	72	49.61c (0.20)	58.25def (0.23)	64.44f (0.23)	1.03e (0.47)	73.11c (0.06)	81.24ab (0.13)	88.72abc (0.38)	5.37c (0.06)	94.39c (0.52)	102.69c (0.13)	109.61c (0.2)	1.18c (0.16)	111.64b (0.22)	116.27a (0.32)	120.57b (0.17)	0.23ab (0.08)
19.00	72	48.83cd (0.18)	57.09g (0.11)	64.48f (0.44)	1.36de (0.40)	74.71b (0.47)	80.77b (0.14)	87.41c (0.11)	6.22a (0.17)	96.26ab ((0.35)	103.59b (0.07)	108.62de (0.13)	1.50a (0.00)	112.44a (0.01)	115.31b (0.30)	116.78d (0.52)	0.15b (0.09)

Means followed by the same superscript in each column are not significantly different ($p>0.05$).
Values shown in parentheses are standard deviation.

Table 4.5 Thermal properties of pellet from starch gel cooled at 1 ºC/min

Amylose Content (%)	Aging Time (h)	Endotherm I				Endotherm III			
		T_o (ºC)	T_p (ºC)	T_c (ºC)	ΔH_r (J/g)	T_o (ºC)	T_p (ºC)	T_c (ºC)	ΔH_{A-L} (J/g)
0.12	24	-	-	-	-	-	-	-	-
4.00	24	57.50f (0.61)	63.72cd (0.33)	68.36d (0.38)	1.34j (0.05)	-	-	-	-
9.00	24	59.44d (0.37)	64.48b (0.36)	70.47b (0.57)	1.91h (0.01)	87.54e (0.63)	106.87e (0.05)	118.72e (0.52)	1.36a (0.05)
14.00	24	56.59g (0.28)	63.63cde (0.02)	69.14cd(0.16)	2.85e (0.04)	92.20c (0.36)	110.19d (0.93)	120.72c (0.23)	1.28a (0.01)
19.00	24	58.45e (0.62)	61.38f (0.50)	66.36f (0.35)	3.16c (0.01)	93.66b (0.54)	116.84a (0.35)	122.92a (0.04)	1.57a (0.01)
0.12	48	-	-	-	-	-	-	-	-
4.00	48	57.71ef (0.24)	63.58cde (0.40)	67.46e (0.64)	1.66i (0.04)	-	-	-	-
9.00	48	50.48j (0.58)	59.39g (0.54)	64.43g (0.08)	2.05g (0.06)	89.73d (0.23)	110.59d (0.23)	120.74c (0.28)	1.38a (0.00)
14.00	48	60.70c (0.02)	62.97de (0.04)	66.18f (0.10)	2.93d (0.04)	91.66c (0.17)	111.26d (0.99)	121.71b (0.08)	1.38a (0.00)
19	48	63.25b (0.27)	64.33bc (0.45)	65.58f (0.25)	3.26b (0.03)	95.18a (1.12)	115.21b (0.17)	121.50bc (0.09)	1.68a (0.00)
0.12	72	52.63h (0.37)	64.54b (0.50)	68.44d (0.46)	0.48k (0.01)	-	-	-	-
4	72	51.39i (0.42)	58.53h (0.02)	64.65g (0.47)	1.70i (0.01)	-	-	-	-
9	72	58.25ef (0.29)	63.56cde (0.33)	69.29c (0.24)	2.26f (0.02)	89.86d (0.14)	106.10e (0.64)	119.59d (0.71)	1.45a (0.04)
14	72	60.46c (0.33)	62.89e (0.08)	66.35f (0.16)	3.11c (0.02)	92.12c (1.17)	113.35c (0.81)	122.16b (0.25)	1.45a (0.04)
19	72	65.37a (0.06)	70.71a (0.39)	76.52a (0.65)	3.93a (0.04)	95.17a (0.08)	115.42b (0.50)	122.05b (0.03)	1.23a (0.09)

Means followed by the same superscript in each column are not significantly different ($p>0.05$).

Values shown in parentheses are standard deviation.

Table 4.6 Thermal properties of pellet from starch gel cooled at 3 ºC/min

Amylose Content (%)	Aging Time (h)	Endotherm I (51-70 ºC)				Endotherm III (87-123 ºC)			
		T_o (ºC)	T_p (ºC)	T_c (ºC)	ΔH_r (J/g)	T_o (ºC)	T_p (ºC)	T_c (ºC)	ΔH_{A-L} (J/g)
0.12	24	-	-	-	-	-	-	-	-
4.00	24	55.42h (0.33)	63.53c (0.56)	69.75ab(0.02)	1.39i (0.02)	-	-	-	-
9.00	24	57.73ef (0.22)	59.88e (0.09)	61.92f (0.11)	1.79f (0.04)	87.10g (0.00)	105.57e (0.74)	118.00e (0.13)	1.31a (0.00)
14.00	24	55.78gh(0.30)	63.53c (0.08)	70.39a (0.49)	2.74d (0.07)	92.39cd (0.78)	110.40c (0.63)	120.72bcd (0.21)	1.29a (0.01)
19.00	24	57.38f (0.50)	61.50d (0.44)	66.59d (0.01)	3.18a (0.06)	94.70ab (0.21)	115.35a (0.17)	121.71abc (0.05)	1.56a (0.04)
0.12	48	-	-	-	-	-	-	-	-
4.00	48	59.43bc (0.57)	64.62b (0.18)	70.22ab (0.15)	1.57h (0.01)	-	-	-	-
9.00	48	49.35j (0.45)	58.60f (0.17)	67.50c (0.40)	2.09e (0.02)	89.71ef (0.21)	108.85d (1.41)	119.66d (0.25)	1.39a (0.01)
14.00	48	59.50bc (0.05)	63.52c (0.36)	67.19cd (0.09)	2.89c (0.02)	92.37cd (0.83)	112.59b (0.71)	121.21bc (0.16)	1.38a (0.00)
19	48	58.52de (0.66)	61.66d (0.47)	66.58d (0.41)	3.23a (0.05)	93.28bc (0.37)	115.49a (0.23)	121.74abc (0.98)	1.55a (0.05)
0.12	72	56.40g (0.40)	64.44b (0.33)	70.48a (0.54)	0.42j (0.11)	-	-	-	-
4	72	51.40i (0.54)	58.60f (0.12)	64.51e (0.24)	1.69g (0.02)	-	-	-	-
9	72	59.64b (0.06)	64.49b (0.38)	70.46a (0.27)	2.08e (0.02)	88.98f (1.25)	106.86e (0.11)	120.59cd (0.08)	1.67a (0.38)
14	72	58.73cd (0.09)	63.68c (0.13)	67.49c (0.68)	3.09b (0.00)	91.21de (0.63)	111.88bc (0.78)	122.03ab (1.04)	1.39a (0.01)
19	72	61.55a (0.45)	65.36a (0.44)	61.51b (0.57)	3.93c (0.05)	96.45a (1.41)	115.40a (0.08)	122.99a (0.63)	1.56a (0.01)

Means followed by the same superscript in each column are not significantly different ($p>0.05$).

Values shown in parentheses are standard deviation.

Table 4.7 Thermal properties of pellet from starch gel cooled at 5 °C/min

Amylose Content (%)	Aging Time (h)	Endotherm I				Endotherm III			
		T_o (°C)	T_p (°C)	T_c (°C)	ΔH_r (J/g)	T_o (°C)	T_p (°C)	T_c (°C)	ΔH_{A-L} (J/g)
0.12	24	-	-	-	-	-	-	-	-
4.00	24	55.50d (0.69)	63.25bc (0.33)	76.45a (0.14)	1.06j (0.01)	-	-	-	-
9.00	24	53.47e (0.45)	64.41a (0.37)	69.04d (0.06)	1.79g (0.02)	85.99f (0.14)	104.12e (0.39)	116.57e (0.74)	1.32bc (0.11)
14.00	24	57.55c (0.61)	63.44b (0.16)	69.58c (0.25)	2.42d (0.04)	89.88e (1.11)	108.26c (0.98)	120.45bc (0.57)	1.28c (0.01)
19.00	24	60.57a (0.43)	62.55c (0.51)	65.67g (0.31)	3.13b (0.03)	94.44b (0.17)	116.31a (0.66)	121.74ab (0.42)	1.58a (0.03)
0.12	48	-	-	-	-	-	-	-	-
4.00	48	59.46b (0.43)	64.64a (0.21)	70.11b (0.04)	1.41i (0.01)	-	-	-	-
9.00	48	50.60g (0.45)	59.41e (0.50)	64.40i (0.10)	1.92f (0.03)	89.04e (1.34)	106.96cd (1.23)	118.34d (0.42)	1.39b (0.01)
14.00	48	60.63a (0.40)	62.84bc (0.22)	66.36f (0.37)	2.92c (0.04)	92.16cd (0.69)	110.86b (0.28)	121.58ab (0.69)	1.38b (0.01)
19	48	57.83c (0.14)	61.53d (0.51)	66.64f (0.17)	3.23a (0.06)	93.92bc (0.88)	115.06a (0.01)	121.87ab (0.01)	1.59a (0.01)
0.12	72	59.47b (0.33)	63.26bc (0.17)	65.13h (0.18)	0.25k (0.00)	-	-	-	-
4	72	57.61c (0.30)	61.49d (0.35)	64.70hi (0.21)	1.68h (0.01)	-	-	-	-
9	72	51.46f (0.31)	58.42f (0.43)	63.72j (0.22)	2.05e (0.05)	89.38e (0.41)	105.79d (0.15)	119.75c (0.30)	1.39b (0.00)
14	72	58.40c (0.37)	63.52b (0.28)	68.35e (0.36)	2.96c (0.07)	91.95d (1.02)	112.06b (1.01)	122.44a (0.64)	1.38b (0.01)
19	72	57.52c (0.18)	59.94e (0.08)	62.53k (0.47)	3.39i (0.01)	98.31a (0.64)	116.27a (0.26)	122.74a (0.98)	1.61a (0.04)

Means followed by the same superscript in each column are not significantly different ($p>0.05$).

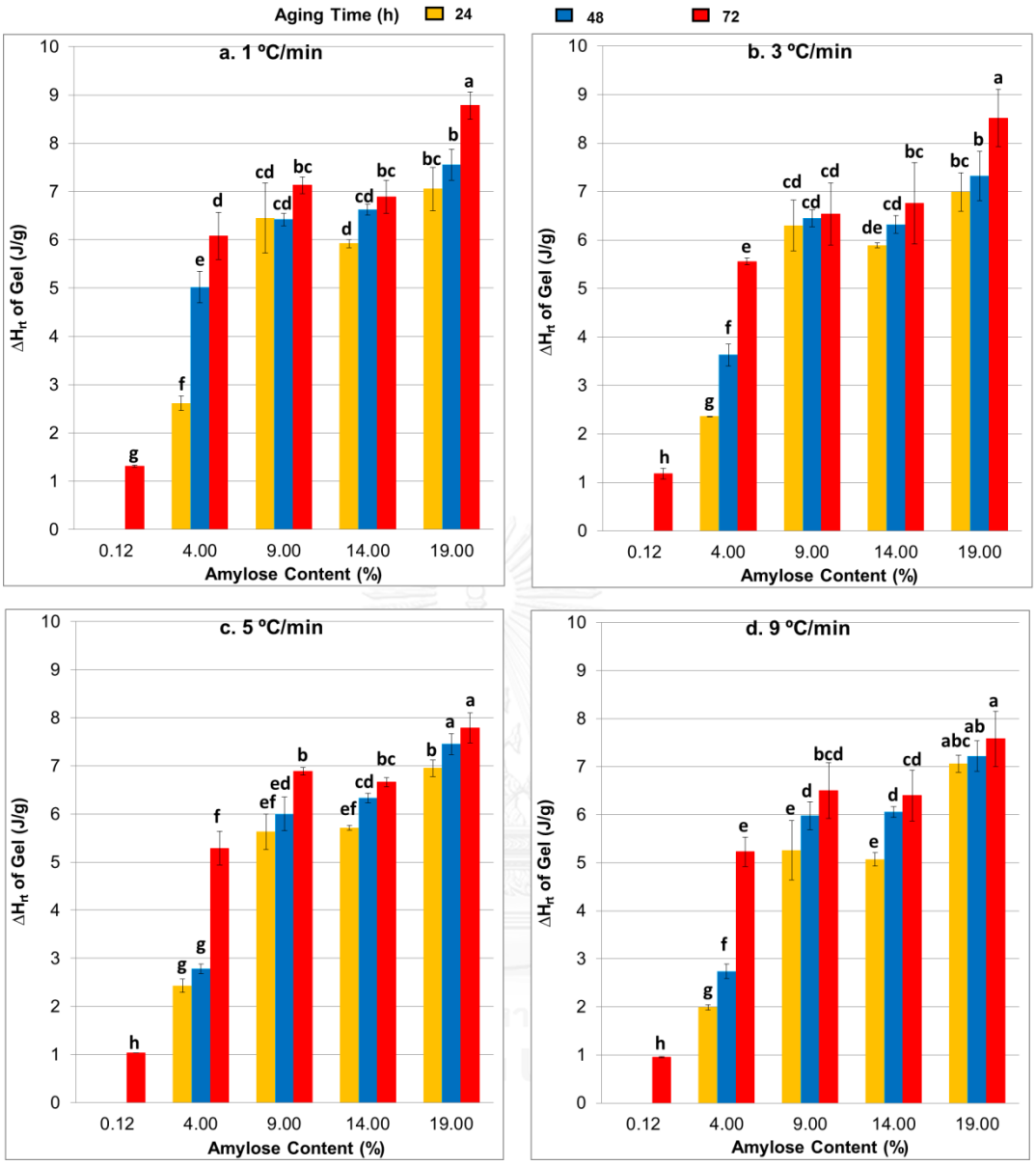
Values shown in parentheses are standard deviation.

Table 4.8 Thermal properties of pellet from starch gel cooled at 9 °C/min

Amylose Content (%)	Aging Time (h)	Endotherm I				Endotherm III			
		T_o (°C)	T_p (°C)	T_c (°C)	ΔH_r (J/g)	T_o (°C)	T_p (°C)	T_c (°C)	ΔH_{A-L} (J/g)
0.12	24	-	-	-	-	-	-	-	-
4.00	24	53.81f (0.14)	63.90cd (0.13)	77.47a (0.28)	0.90h (0.01)	-	-	-	-
9.00	24	58.49bc (0.11)	64.35bc (0.16)	69.60d (0.52)	1.65fg (0.09)	85.82f (0.18)	103.40d (0.78)	116.16e (0.46)	1.33de (0.02)
14.00	24	56.79e (0.12)	63.52de (0.17)	69.52d (0.40)	2.54d (0.07)	89.84cd (0.36)	106.94c (0.06)	120.78bc (0.16)	1.30e (0.00)
19.00	24	59.39b (0.38)	62.62f (0.38)	66.50g (0.10)	3.17b (0.05)	94.38a (0.78)	115.66a (0.33)	121.90ab (0.08)	1.58b (0.02)
0.12	48	-	-	-	-	-	-	-	-
4.00	48	58.52bc (0.66)	64.86b (0.19)	71.29c (0.25)	1.56g (0.02)	-	-	-	-
9.00	48	50.58h (0.35)	59.30h (0.35)	64.38i (0.15)	1.91e (0.01)	88.10e (1.40)	106.57c (0.67)	118.77d (0.96)	1.35cde (0.05)
14.00	48	58.48bc (0.01)	63.56de (0.12)	68.14e (0.10)	2.94c (0.06)	91.54b (0.45)	110.70b (0.08)	121.21ab (0.76)	1.39c (0.01)
19	48	57.50de (0.68)	61.80g (0.11)	67.62ef (0.04)	3.24b (0.05)	95.49a (0.01)	116.60a (1.49)	122.10ab (0.78)	1.58b (0.00)
0.12	72	58.58bc (0.45)	63.21e (0.27)	67.31f (0.12)	0.21i (0.02)	-	-	-	-
4	72	57.67cd (0.36)	61.54g (0.42)	64.60i (0.33)	1.66f (0.01)	-	-	-	-
9	72	51.54g (0.64)	58.45i (0.62)	65.56h (0.29)	1.96e (0.06)	89.09de (0.00)	107.95c (2.69)	119.66cd (0.13)	1.37cd (0.03)
14	72	58.53bc (0.28)	63.58de (0.13)	68.11e (0.10)	3.01c (0.01)	91.17bc (0.55)	110.71b (0.05)	121.27ab (0.41)	1.39c (0.01)
19	72	64.57a (0.47)	70.68a (0.22)	76.48b (0.40)	3.43a (0.05)	95.34a (0.42)	115.34a (0.00)	122.30a (0.34)	1.66a (0.02)

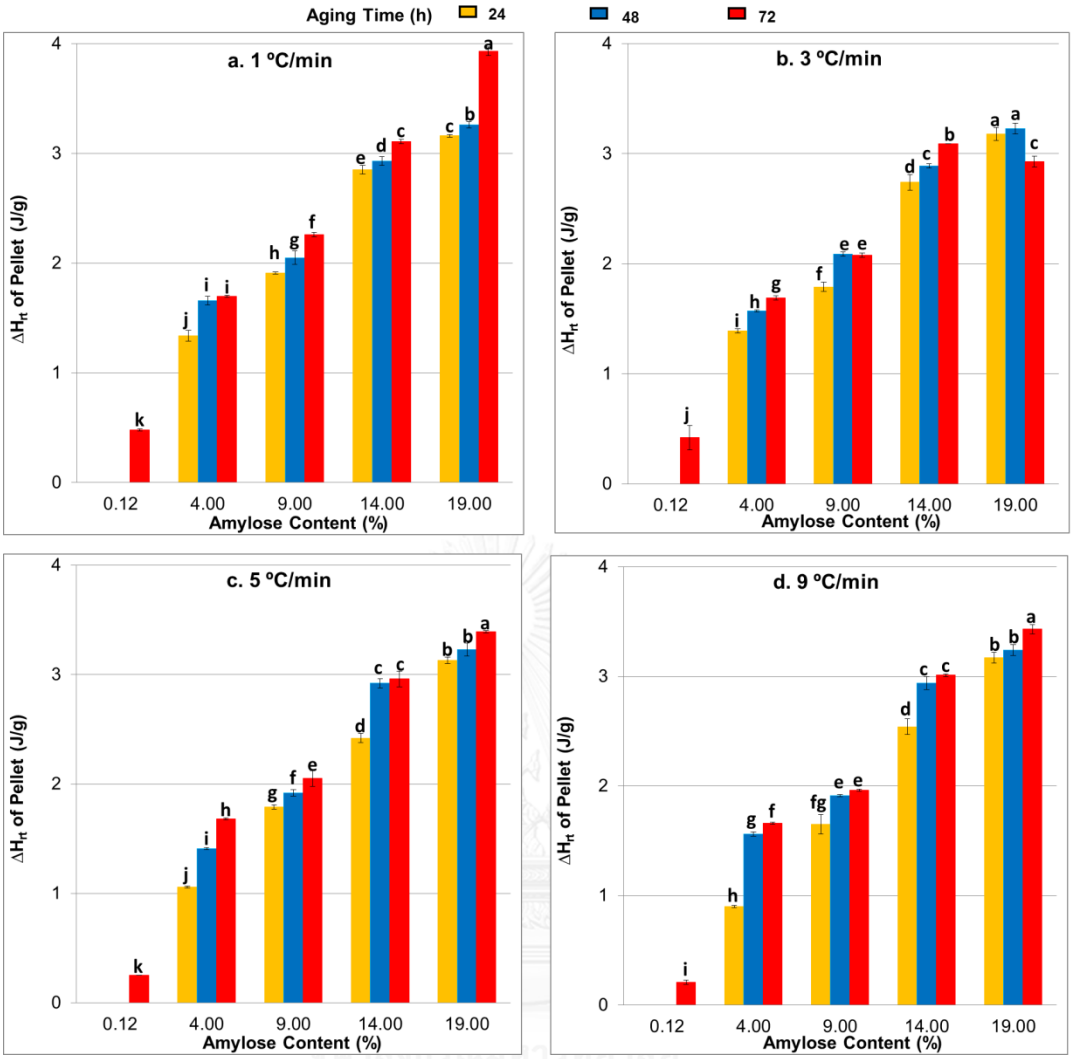
Means followed by the same superscript in each column are not significantly different ($p>0.05$).

Values shown in parentheses are standard deviation.



The same letters are not significantly different ($p>0.05$) in each cooling rate.

Figure 4.14 Effect of amylose content and aging time at each cooling rate on retrogradation enthalpy of starch gel.



The same letters are not significantly different ($p>0.05$) in each cooling rate.

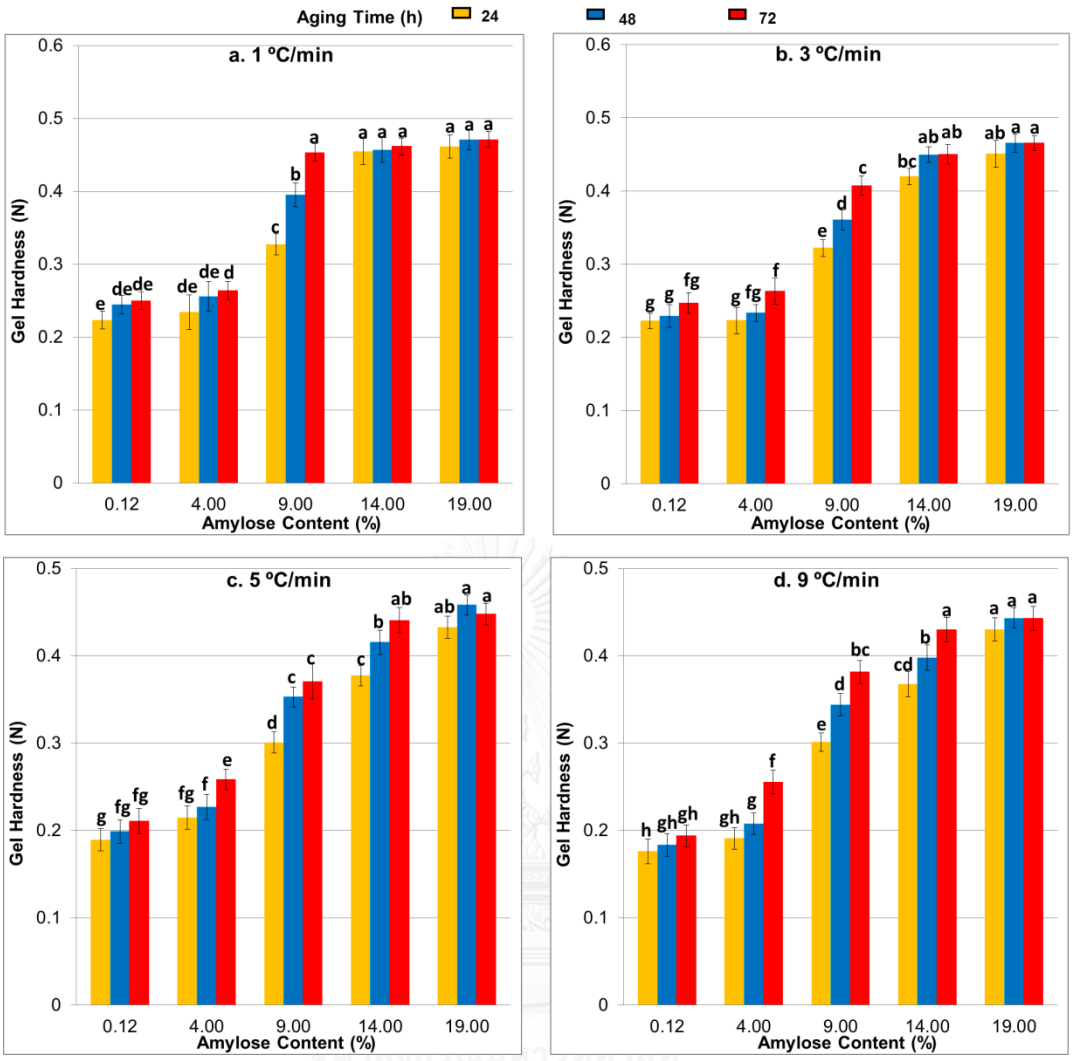
Figure 4.15 Effects of amylose content and aging time at each cooling rate on retrogradation enthalpy of pellets.

4.2.3 Hardness

Statistical analysis as shown in Appendix C indicated that the order of significant factors affected hardness of gels and pellets was AC > aging time > cooling rate. Therefore, Figure 4.16 and 4.17 show interaction between AC and aging time at each cooling rate on the hardness of gel and pellet, respectively. Figures 4.16 and Figure 4.17 show that higher AC and aging time significantly increased in both hardness of gel and pellet at constant cooling rate ($p<0.05$). However, for hardness of

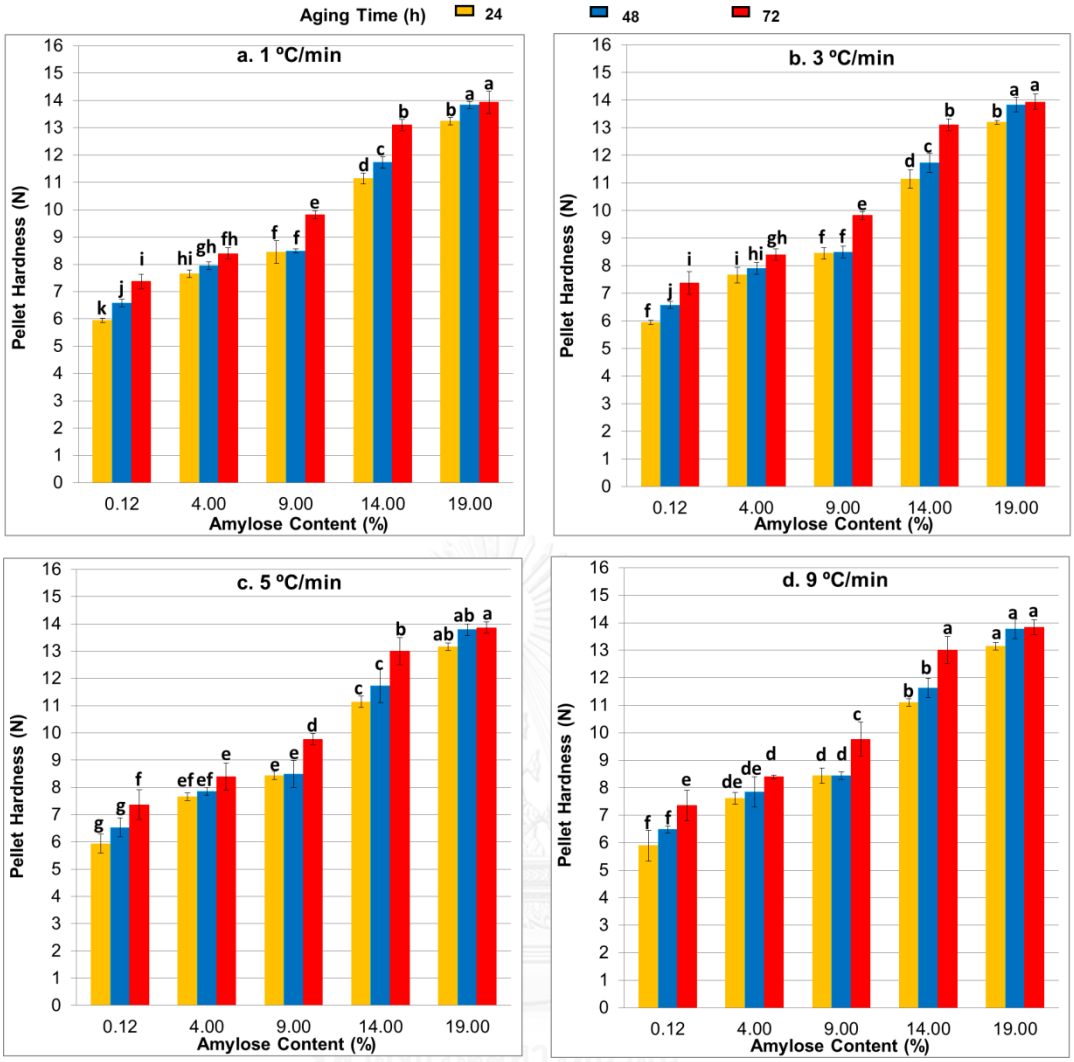
gel with 19.00%AC was not affected by aging time. The decrease in cooling rate significantly increased hardness of gel, but did not affect hardness of pellet. The interaction between AC and aging time ($p<0.05$) on hardness of gel. For 9.00% and 14.00% AC, pellet hardness did not change as aging time increased from 24 h to 48 h. For 0.12%, 4.00%,and 19.00% AC, pellet hardness did not change as aging time increased from 48 to 72 h.

An increase in the hardness of gel and pellet was resulted from of an increase in starch retrogradation. An increase in starch retrogradation with AC and aging time was detected by XRD and DSC reported in Section 4.1.1 and 4.1.2. The comparison between hardness of gel and pellet showed that hardness of pellet was about 29 folds higher than that of gel. This was because the crystallites of pellet were packed tighter after drying. Compared to XRD and DSC, the texture analysis might be a better method for detection the effect of cooling rate on starch retrogradation.



The same letters are not significantly different ($p>0.05$) in each cooling rate.

Figure 4.16 Effects of amylose content and aging time at each cooling rate on gel hardness.



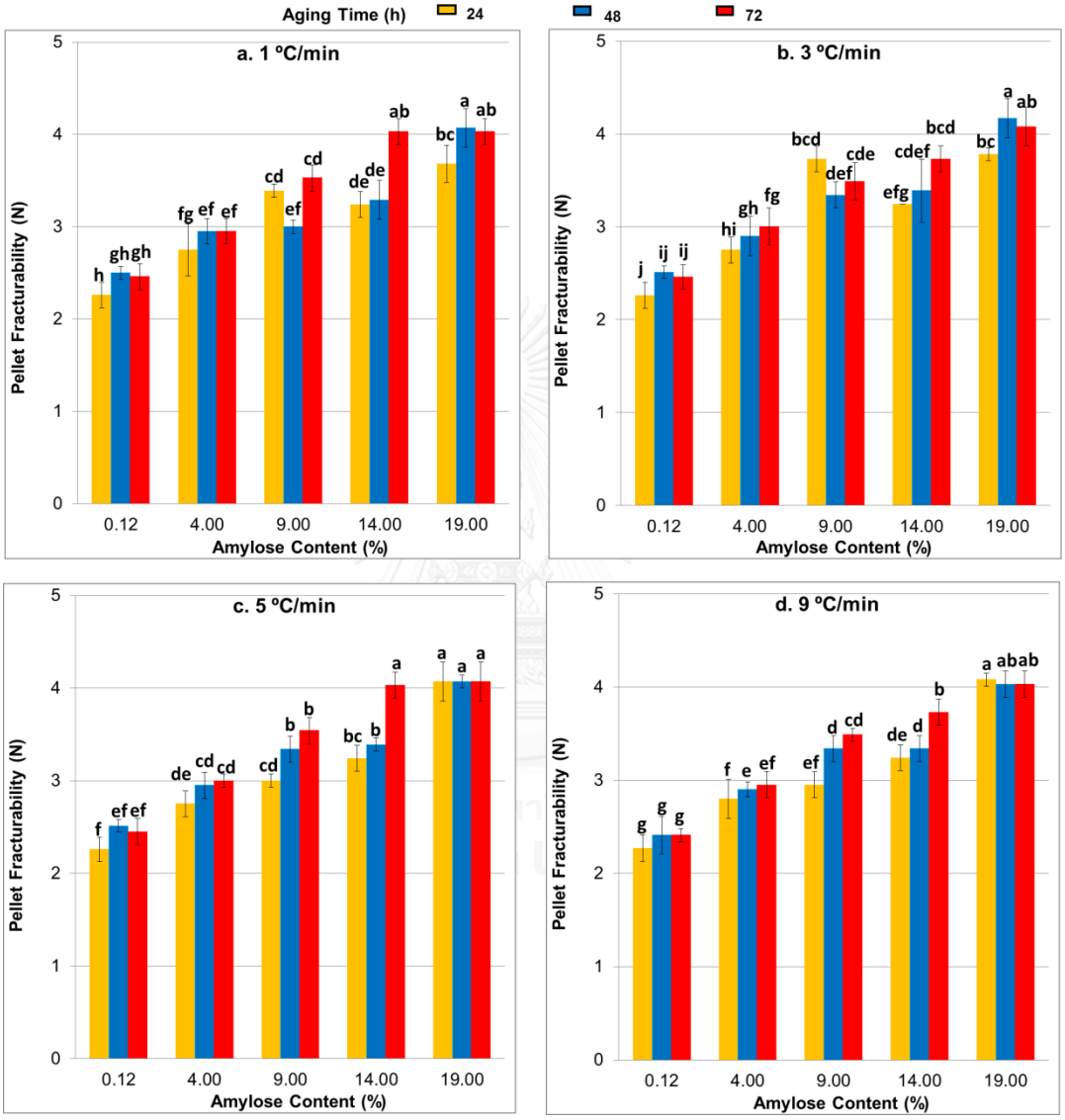
The same letters are not significantly different ($p>0.05$) in each amylose content.

Figure 4.17 Effects of amylose content, cooling rate and aging time on hardness of pellet.

4.2.4 Fracturability

There were the main effects of AC, and aging time and its interaction on fracturability of pellet (≤ 0.05) but fracturability of pellet was not affected by cooling rate ($p>0.05$) as shown in Appendix C. Effect of AC was the highest follow by aging time. The interaction was showed in Figure 4.18. Fracturability of pellet increased as AC and aging time increased except for fracturability of pellet with 0.12, 4.00, and

19.00% AC which was not affected by aging time in the range of 24 to 72 h. The effects of AC and aging time on fracturability and hardness can be explained with the same reasons in Section 4.2.3.



The same letters are not significantly different ($p>0.05$) in each cooling rate.

Figure 4.18 Effects of amylose content and aging time at each cooling rate on fracturability of pellet.

4.2.5 SEM image of starch gel

SEM images of starch gel in Figure 4.19 show that starch gel with higher AC gave a more dense and uniform network than starch gel with lower AC. Pore size decreased with higher AC and aging time. The higher extent of starch retrogradation with the higher AC and the longer aging time resulted in an increase in network continuity and a smaller pore size (Utrilla-Coello et al., 2013).

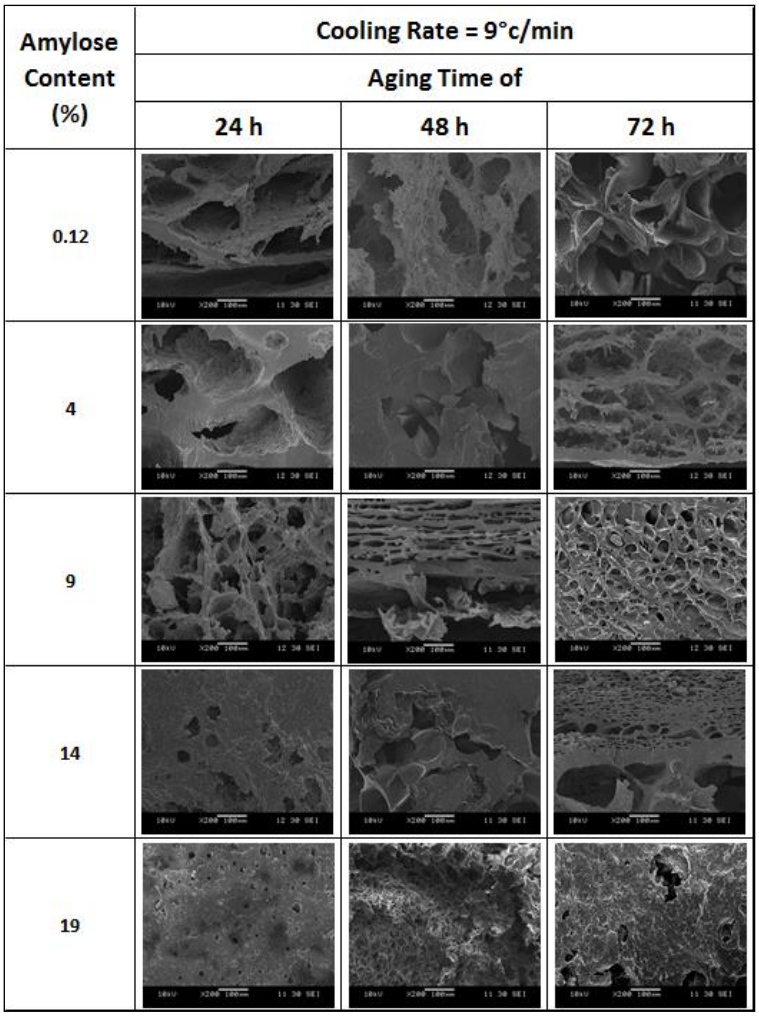


Figure 4.19 SEM images (200x) of rice starch gels.

4.3 Puffed product properties

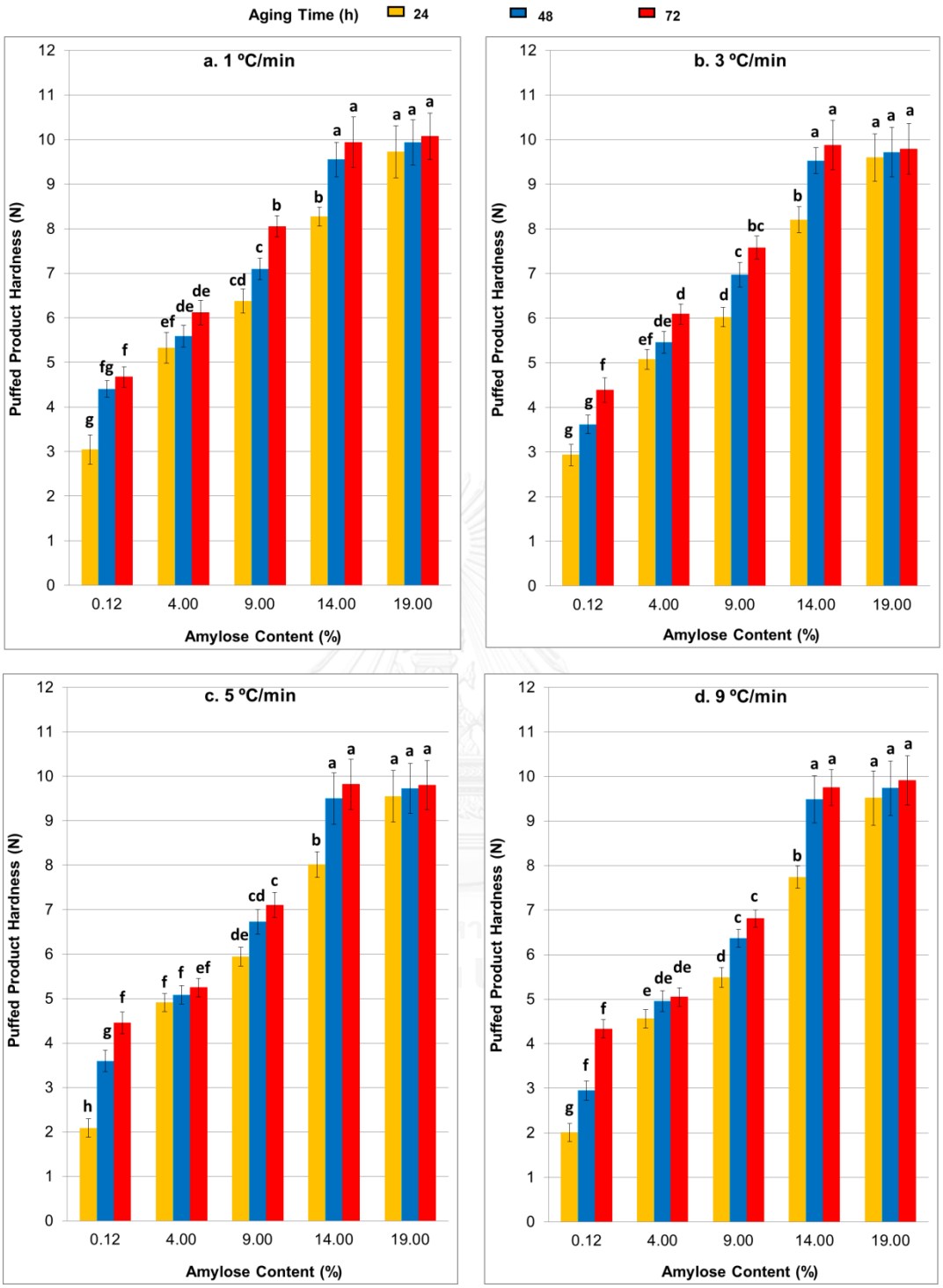
4.3.1 Physical properties

There were the effects of three factor (AC, aging time and cooling rate) and interaction between AC and aging time on hardness, fracturability, bulk density and expansion ratio ($p \leq 0.05$) as shown in Appendix C. For hardness and fracturability, effect of AC was the highest follow by aging time and cooling rate. For bulk density and expansion ratio, effect of AC was the highest follow by cooling rate and aging time. From the interaction, Figures 4.20, 4.21 and 4.22 show that hardness, fracturability, and bulk density significantly increased with AC for every cooling rates at 24 h aging time. AC in the range of 14.00-19.00% did not affect hardness at 48 and 72 h aging and fracturability at 72 aging. On the other hand, the expansion ratio significantly decreased as the AC increased (Figure 4.23).

For products obtained from AC ≤14.00%, hardness increased as aging time increased from 24 to 48 h, while fracturability increased as aging time increased from 48 to 72 h. For products obtained from 0.12% and 14.00%, bulk density increased as aging time increased from 24to 48 h. Expansion ratio decreased as aging time increased from 48 to 72 h for all AC.

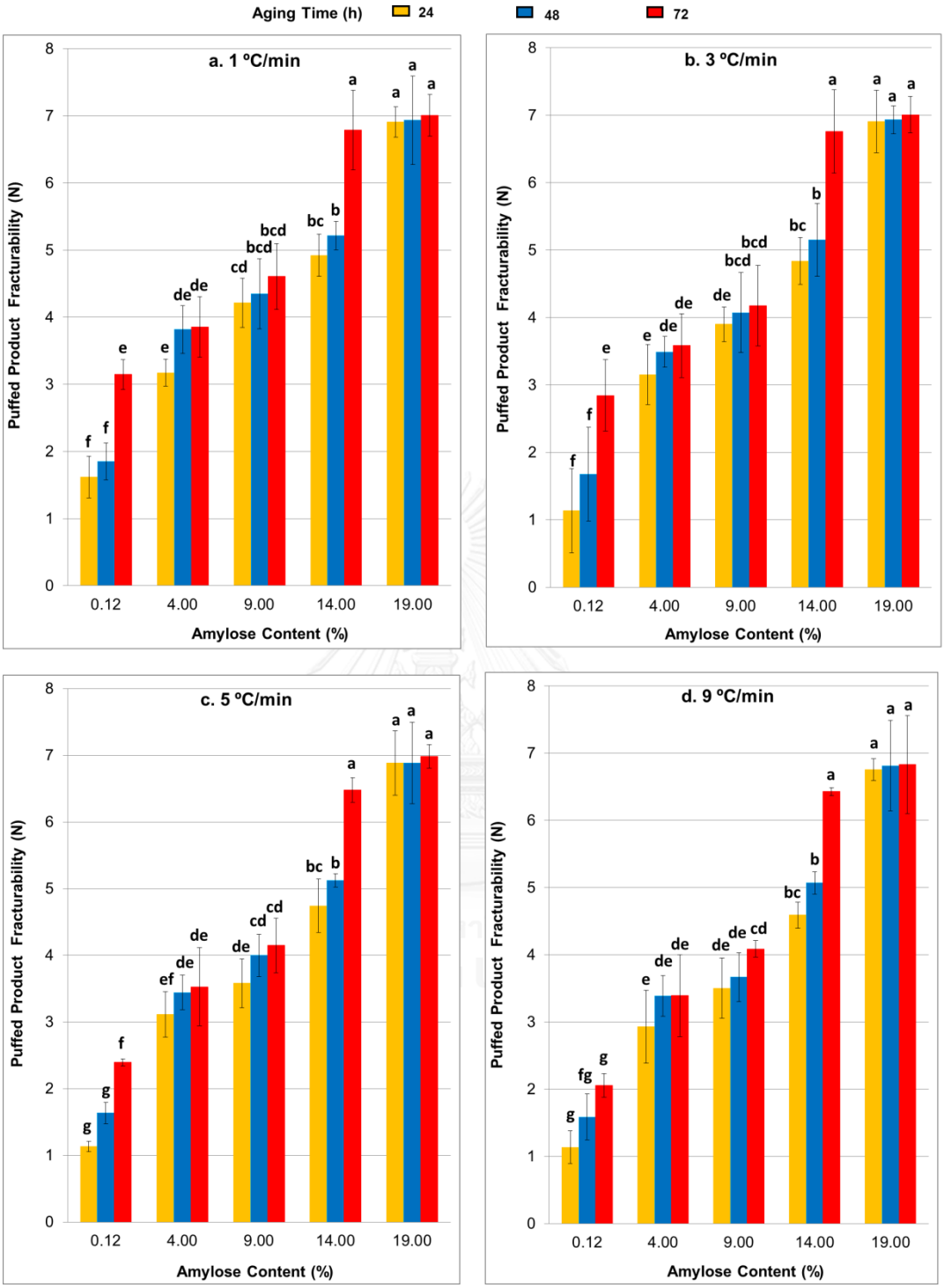
The water vaporized from pellet upon frying resulting in the expansion of network and porous structure (Norton et al., 2011; Schwartzberg et al., 1995). The linear chain structure of amylose forms a strong network which resists the expansion resulting in a hard texture, while amylopectin, having a highly branched chain structure, forms a weaker network, contributing an expanded texture and a lighter product (Chen and Yeh, 2001; Wang, 1997). Furthermore, as aforementioned, the higher AC promoted retrogradation resulting in a stronger gel, thus resisting

expansion. Chen and Yeh (2001) and Tongdang et al. (2008) reported that a higher retrogradation of starch gel resulted in a higher hardness, and bulk density of puffed products but lower expansion ratio because a strong gel from high retrogradation might resist bubble formation during frying process, while a weak gel may not provide the bubbles sufficient integrity.



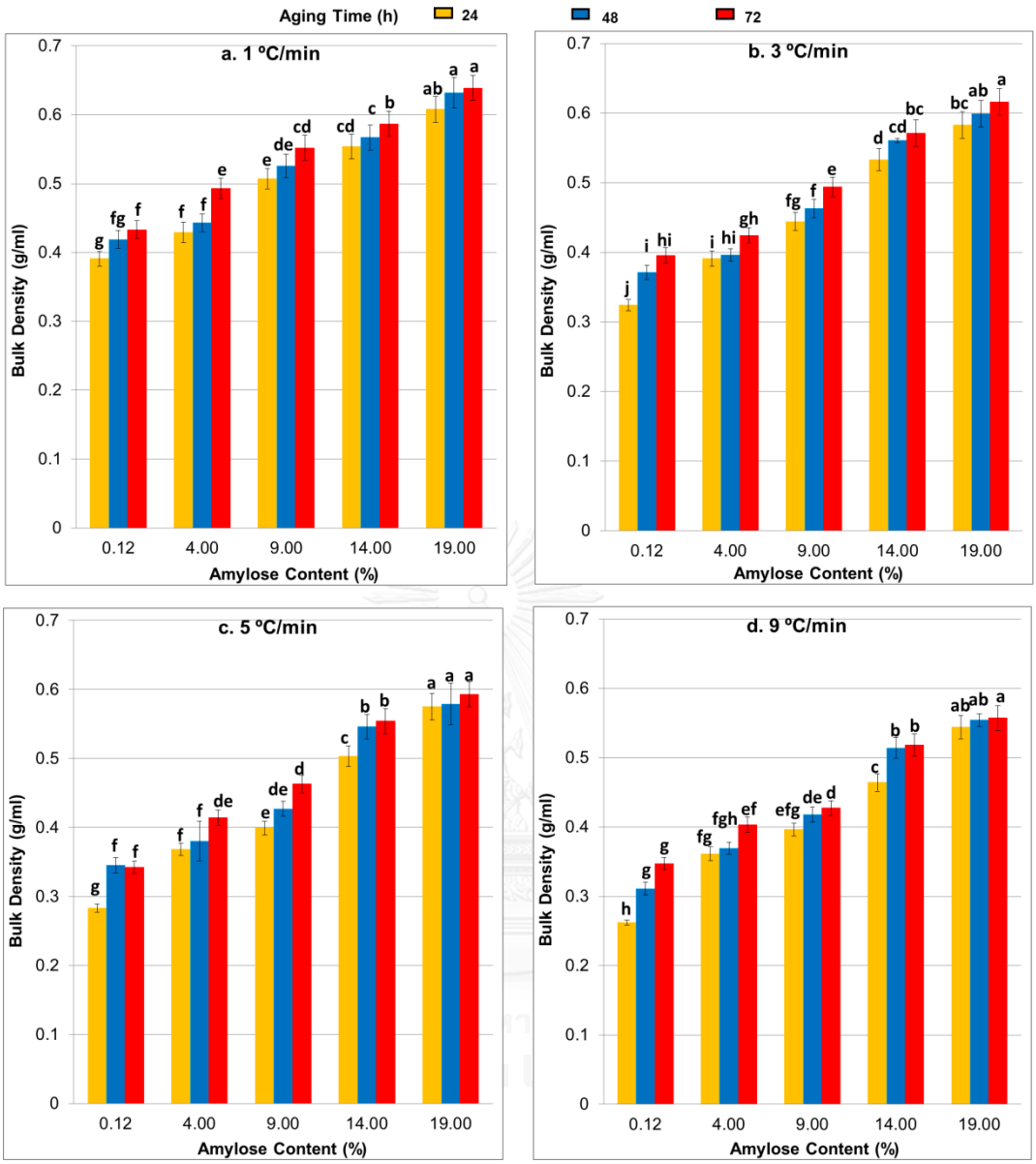
The same letters are not significantly different ($p>0.05$) in each cooling rate.

Figure 4.20 Effects of amylose content and aging time at each cooling rate on hardness of puffed product.



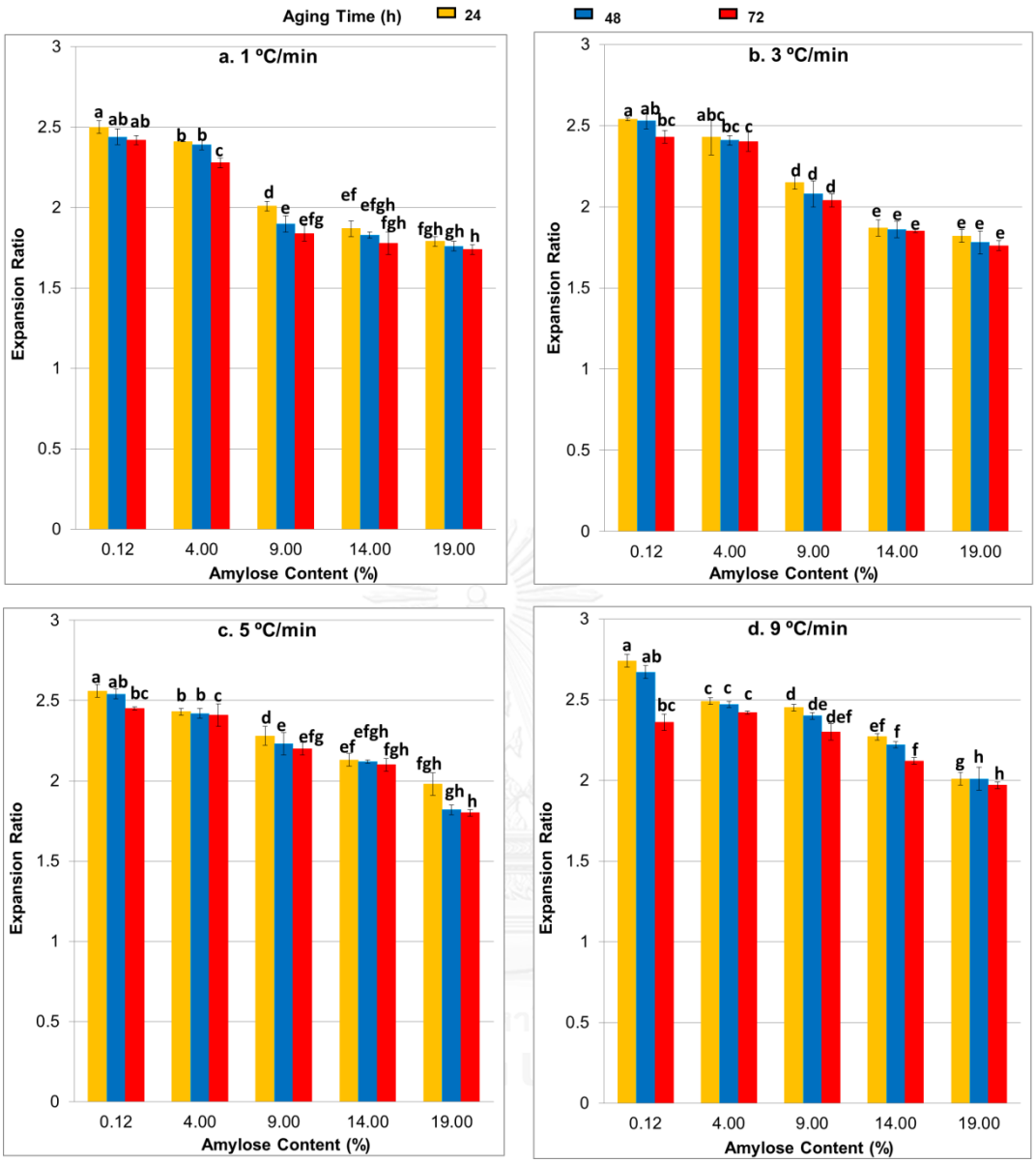
The same letters are not significantly different ($p>0.05$) in each cooling rate.

Figure 4.21 Effects of amylose content and aging time at each cooling rate on fracturability of puffed product.



The same letters are not significantly different ($p>0.05$) in each cooling rate.

Figure 4.22 Effects of amylose content and aging time at each cooling rate on bulk density of pellet.



The same letters are not significantly different ($p>0.05$) in each cooling rate.

Figure 4.23 Effects of amylose content and aging time at each cooling rate on expansion ratio of puffed product.

4.3.2 Images of puffed products

The images of puffed products were taken by the image analyzer (Figure 4.24) and camera (Figure 4.25). These images show that the higher AC and aging time gave less expansion and smaller air cell size than the lower AC and aging time.

Prior to puffing, drying of starch gels caused moisture loss and starch network was dense and formed crust. A pellet having strong starch network provided a sufficient integrity of air cell wall during expansion.

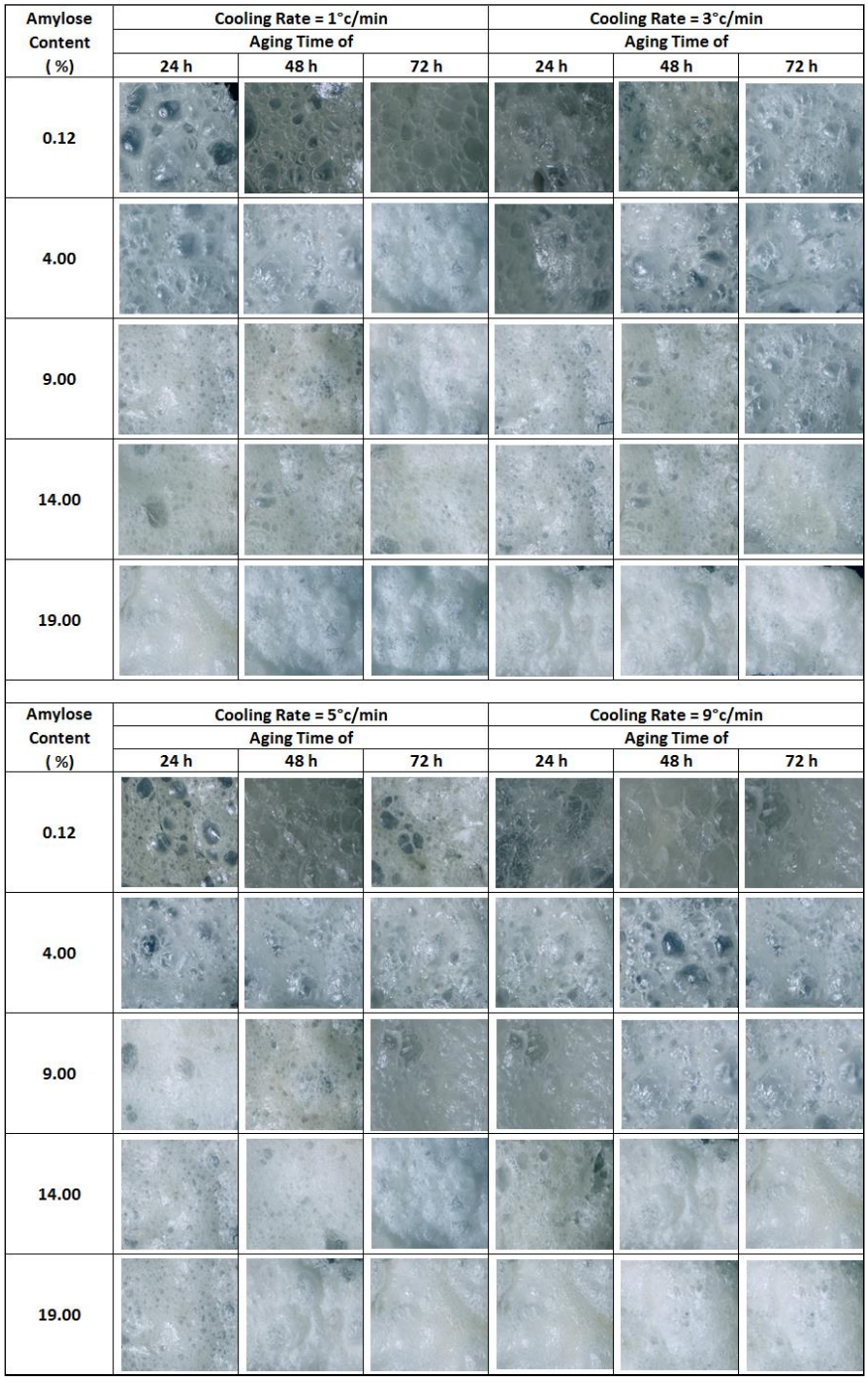
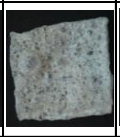
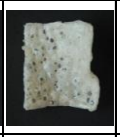
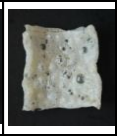
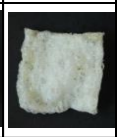


Figure 4.24 Image analyzer images of puffed products.

Amylose Content (%)	Cooling Rate = 1°c/min Aging Time of			Cooling Rate = 3°c/min Aging Time of		
	24 h	48 h	72 h	24 h	48 h	72 h
0.12						
4.00						
9.00						
14.00						
19.00						

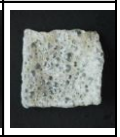
Amylose Content (%)	Cooling Rate = 5°c/min Aging Time of			Cooling Rate = 9°c/min Aging Time of		
	24 h	48 h	72 h	24 h	48 h	72 h
0.12						
4.00						
9.00						
14.00						
19.00						

Figure 4.25 Puffed product images from camera.

4.3.3 Thermogram of puffed products

Thermograms of puffed products were obtained after storing at 25 ˚C for 1 week. Endotherm at 100-120 ºC was found for all products obtained from cooling rate of 5 ºC/min and aged for 48 h (Figure 4.26). The thermal properties were not affected by AC. During deep-frying at 110 °C, amylopectin crystallites and aggregates were destroyed, while formation of amylose-lipid complex was promoted. For AC ≥9.00%, very small peak, having ΔH_r of 0.5 J/g, was detected at 51-68 ˚C indicating the melting of amylopectin retrogradation. However, the enthalpies for both melting of amylopectin retrogradation and amylose-lipid complex were not significantly affected by AC.

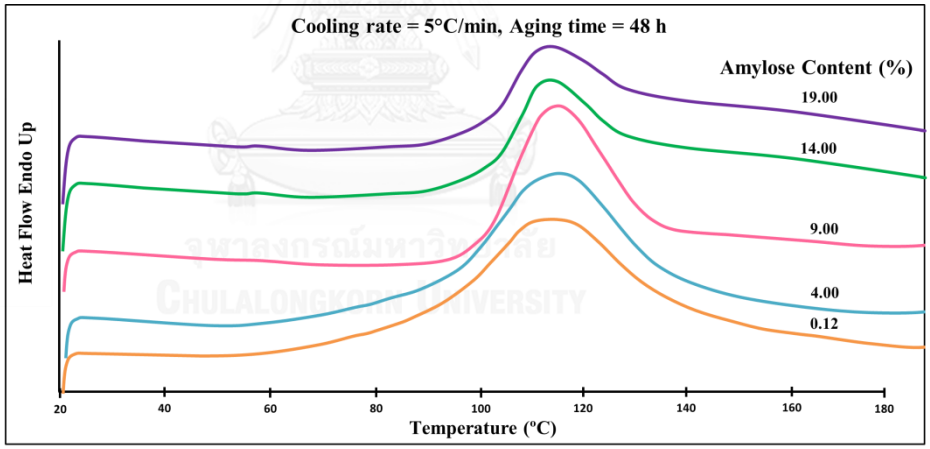


Figure 4.26 Thermograms of puffed product.

Table 4.9 Thermograms of puffed product

Amylose content (%)	Endotherm I				Endotherm II			
	T_o (ºC)	T_p (ºC)	T_c (ºC)	ΔH_r (J/g)	T_o (ºC)	T_p (ºC)	T_c^{ns} (ºC)	ΔH_{A-L}^{ns} (J/g)
0.12	-	-	-	-	77.94[b] (1.36)	116.06[b] (0.40)	153.43 (3.61)	230.73 (5.37)
4.00	-	-	-	-	82.54[b] (4.87)	119.71[a] (1.03)	150.99 (0.63)	222.62 (17.24)
9.00	51.06 (0.86)	60.30 (0.06)	68.66[a] (0.45)	0.51[a] (0.06)	92.87[a] (1.26)	117.48[b] (0.71)	152.09 (2.83)	226.04 (7.62)
14.00	54.16 (0.85)	61.40 (0.23)	68.27[a] (0.25)	0.53[a] (0.01)	96.07[a] (2.79)	113.46[c] (0.68)	130.04 (1.34)	222.42 (12.22)
19.00	53.94 (1.00)	61.45 (0.57)	68.25[a] (0.25)	0.53[a] (0.03)	99.21[a] (2.20)	110.76[d] (0.16)	158.43 (2.04)	227.02 (5.71)

Means followed by the same superscript in each column are not significantly different ($p>0.05$).

4.4 Sensory textural profiles of puffed products

The sensory characteristic of 27 puffed products, prepared from AC in range of 4.00, 9.00, and 14.00%; aging time for 24, 48, and 72 h; cooling rate at 3, 5, 9 ºC/min, were profiled following the lexicon developed during training sessions. The lexicon of 11 sensory attributes (in 3 main categories) with definitions and references was shown in Table 4.10 (including Table 3.1 and Figure 3.2).

Table 4.10 Lexicon for the descriptive sensory analysis of rice starch puffed products**.**

Terms	Definition	Evaluation Technique	Reference (Intensity)*
Appearance			
Expansion	Volume of product by increasing horizontally expands.	Visual	D (2) A (6) B (2)
Puffiness	Volume of products by increasing lifted up vertically resulting in thickness.	Visual	A (2) B (7) C (14)
Air cell density	Degree of compression of the air cell inside the product.	Cross-section breaking to view the air cell denseness.	A (2) B (7) C (14)
Air cell size	The average size of the air cell (pore) inside the product.	Visual	E (1) B (7) A (14)
Texture			
Crispness	Attribute associated with high pitched sounds and are likely to melt in the mouth.	Measure the level of noise by biting through the sample with incisor teeth.	CornPuff (2) Poppo (7) Tawan (11)
Hardness	Force required for compressing a substance between molar teeth.	Place sample between molar teeth and bite evenly, evaluate the force required to compress the product.	Roller (3) CornPuff (6) Poppo (9) Bourbon (15)
Brittleness	The degree of ease in breaking, fracturing or splintering in the mouth as the force to bite through with incisors.	Measure the level of force to bite through with incisors.	Bourbon (2) Roller (6) Poppo (8) Pringle (14)
Cohesiveness of mass	Degree to which the chewed sample holds together.	Place sample between molars and compress fully (can be done with incisors), chew five times, and evaluate the amount the samples hold together or form.	Bourbon (3) Poppo (6) CornPuff (13)
Oiliness	The amount of oil perceived within the mouth while chewing.	Chew sample and evaluate the amount of oil in the mouth.	Roller (1) Masita (8) Entrée (15)
Tooth Stickiness	Attribute associated with degree that the sample becomes attached to the teeth after mastication	Measure amount of product adhering to the teeth after mastication	Tawan (1) Nalie (6) CornPuff (14)
After-taste			
Oil Coating	Perception of oil remains in the mouth after swallowing	Measure amount of oil after swallow.	Roller (1) Masita (8) Entrée (15)

* Intensity from a 15-point numerical scale

For each attribute, intensity scores of all 27 products were compared. The results showed that the AC and the aging time are key factors affecting the puffed characteristics, while cooling rate has less effect.

The profiles of 27 samples at each cooling rate were shown in Figure 4.27 (a, b, and c). There were significant differences among samples in all 12 sensory characteristics ($p<0.05$). The texture profile from the 3 cooling rates shows similarly patterns. The result suggests that the AC is the key factor affecting textural properties of the puffed product. The scores of sensory attributes including, puffiness, air cell density, crispiness, hardness, and brittleness increased with increasing AC. However, expansion, air cell size, oiliness cohesiveness of mass, tooth stickiness and oil coating tended to decrease with increasing AC. At the same level of AC, the samples with longer aging time also show similar results. The result also indicated that linear expansion decreased as AC increased (Boischot et al., 2003; Cheow et al., 2004; Saeleaw and Schleining, 2010; Tongdang et al., 2008). A decrease in oiliness was due to higher air cell density and less porosity with higher AC and longer aging time. The higher oil absorption level was obtained from lower AC (Mohamed and Abd Hamid, 1994).

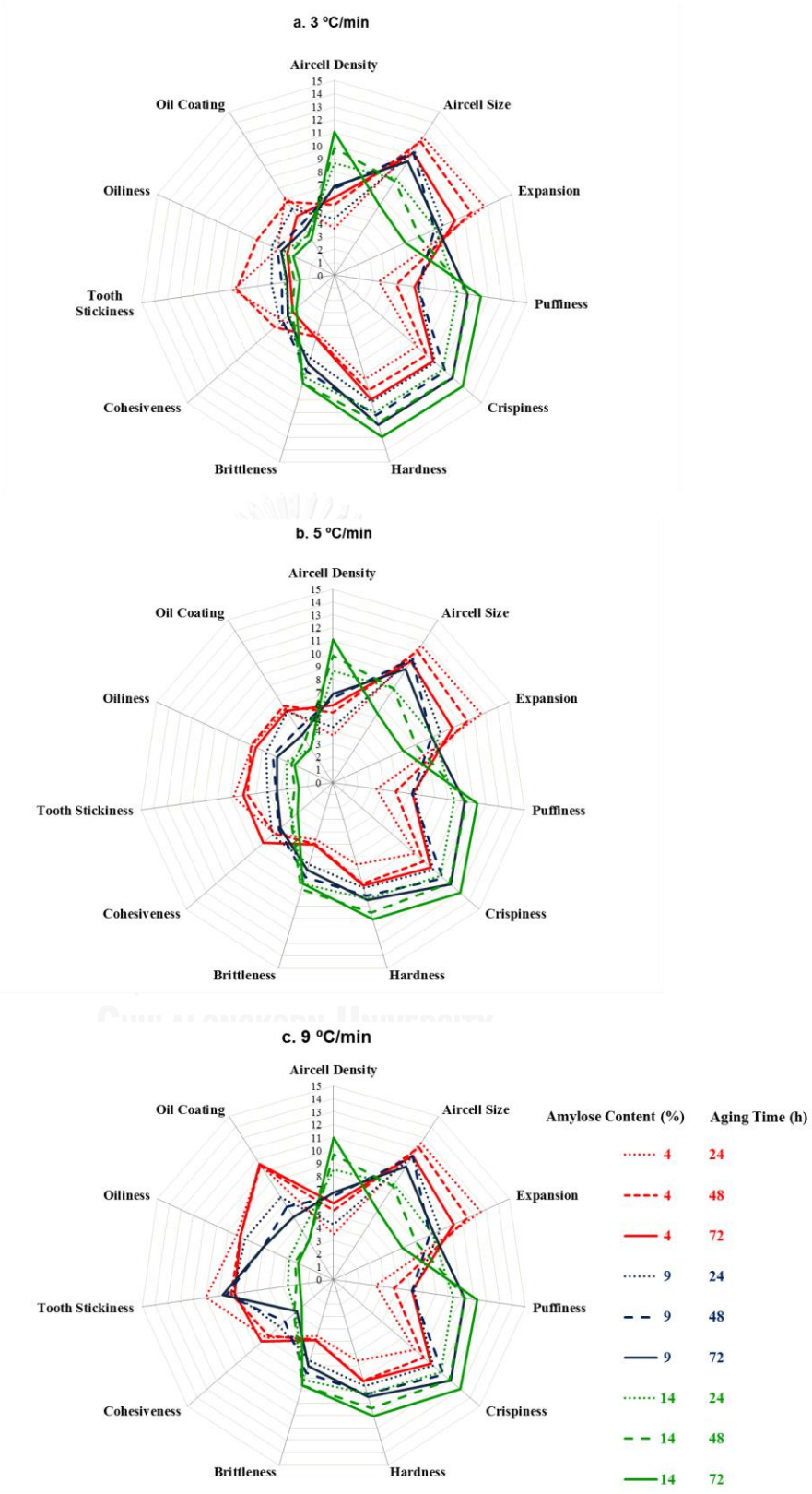


Figure 4.27 Sensory profile of test puffed samples.

4.5 Correlations of attributes

Table 4.11 shows correlation coefficients of some pairs of sensory attributes. High correlations was observed between expansion and puffiness (r = -0.88), expansion and crispness (r = 0.96), hardness and puffiness (r = 0.91), hardness and crispness (r = 0.95), crispness and brittleness (r = 0.90), oiliness and oil coating (r = 0.89).

The term crispness, developed from these panels, was defined as a noise after biting which differs from other languages that might have defined it as the force during chewing a sample (Faller and Heymann, 1996; Zampini and Spence, 2004). Crispness was related to brittleness (fracture) which is defined as the degree of force needed to break the sample (Faller and Heymann, 1996). High crispness scores indicated that the sample was fractured more easily. Moreover, the crispness correlated to appearance attributes as brittleness (r = 0.90). It was concluded that crispness is a multidimentional attribute according to Lazou et al. (2010) who reported that crunchiness of extruded snacks correlated with appearance, porosity and diameter (Faller and Heymann, 1996).

Table 4.11 The pearson correlation (r) of sensory attributes of puffed product

Attributes	Puffiness	Crispness	Oiliness	Air Cell Size
Expansion	**-0.88**	**0.96**	**0.69**	**0.86**
Hardness	**0.91**	**0.95**	**0.73**	**-0.88**
Brittleness	**0.88**	**0.90**	**0.79**	**-0.85**
Oil Coating	**0.75**	**0.71**	**0.89**	**0.73**
Air Cell Density	**0.86**	**0.80**	**-0.76**	**-0.95**

Intensity of expansion was visually evaluated as degree of horizontally increasing in the volume (size) while intensity of puffiness was visually evaluated as increasing in thickness. The data was showed in Figure 4.28-4.29, as the highly negative correlation found. There are main effects of AC, aging time and the interaction of these factors on the expansion and the puffiness ($p<0.05$) as showed in Figure 4.28-4.29. The higher AC significantly decreased the expansion, but increase in puffiness. Increasing aging time also significantly affected these 2 attributes with the interaction between AC and aging time was also found ($p\leq0.05$). This was corresponding to the expansion ratio of puffed product which depend on the AC and aging time vice versa cooling rate.

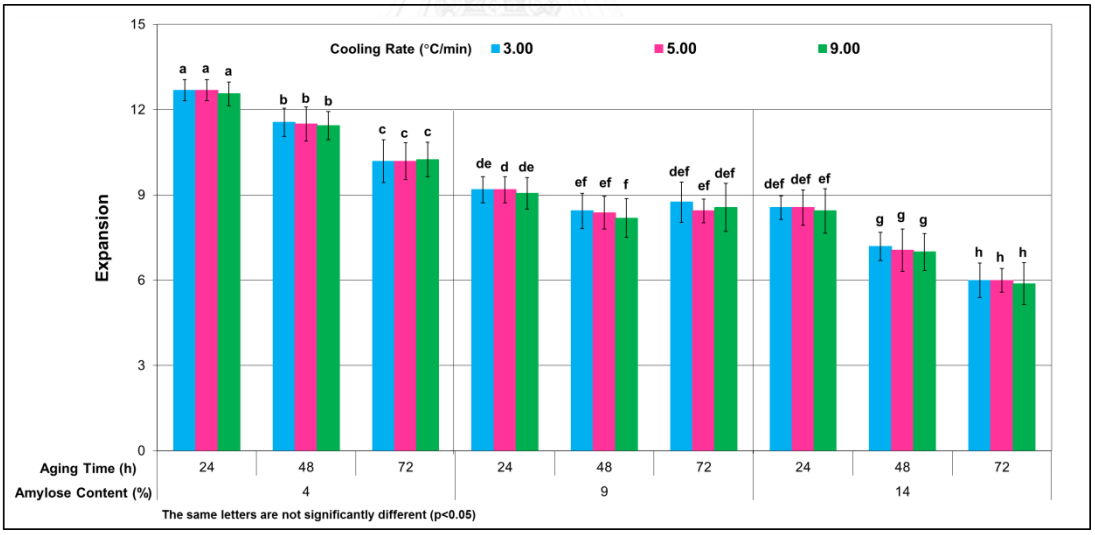


Figure 4.28 Expansion of puffed products from rice starch gels

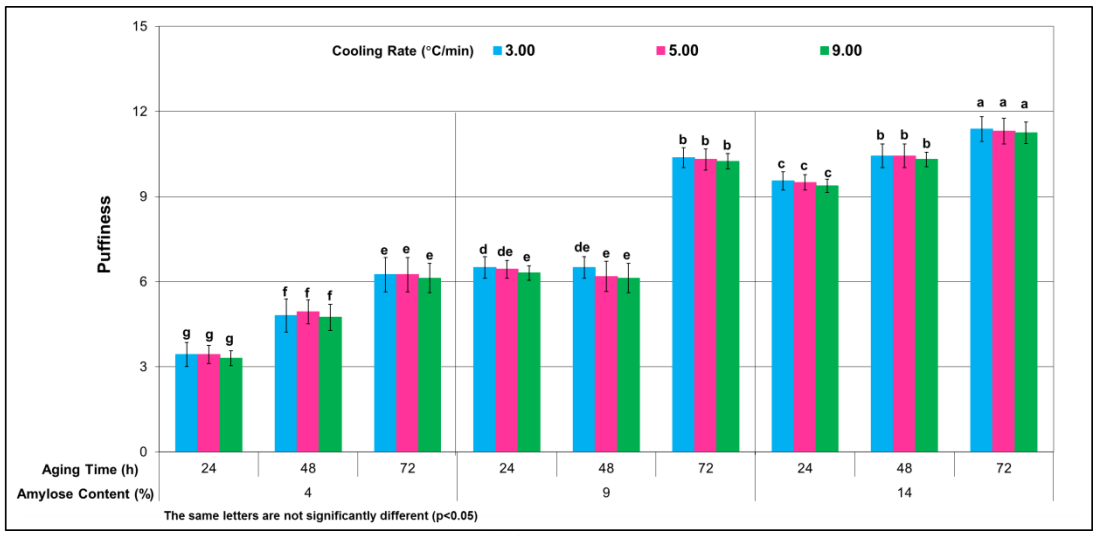


Figure 4.29 Puffiness of puffed products from rice starch gels.

Characteristics of air cell were visually evaluated as density and size. Scores were shown in Figure 4.30 – 4.31. The AC played major role on air cell density and size ($p \leq 0.05$). The 14% AC significantly highest in the air cell density ($p \leq 0.05$). There was an interaction between AC and aging time ($p \leq 0.05$). As expected from images of starch gel and puffed product, network of puffed products with more density obtained from high AC and aging time. Air cell density was defined as degree of compression of the air cell inside the product, while air cell size was defined as the average size of the air cell (pore) inside the product. The higher AC significantly increased the air cell density but lower in air cell size ($p \leq 0.05$) with $r = -0.95$. The interaction between AC and aging time was also found for both attributes ($p \leq 0.05$). At the same AC, the cooling rate did not affect both attributes. As expected from images of starch gel and puffed product, more opacity indicated higher density of the network due to higher starch retrogradation at higher AC and longer aging time. The higher

extent of starch retrogradation at higher AC and with longer aging time resulted in more continuity of the network and the smaller size of the air cells.

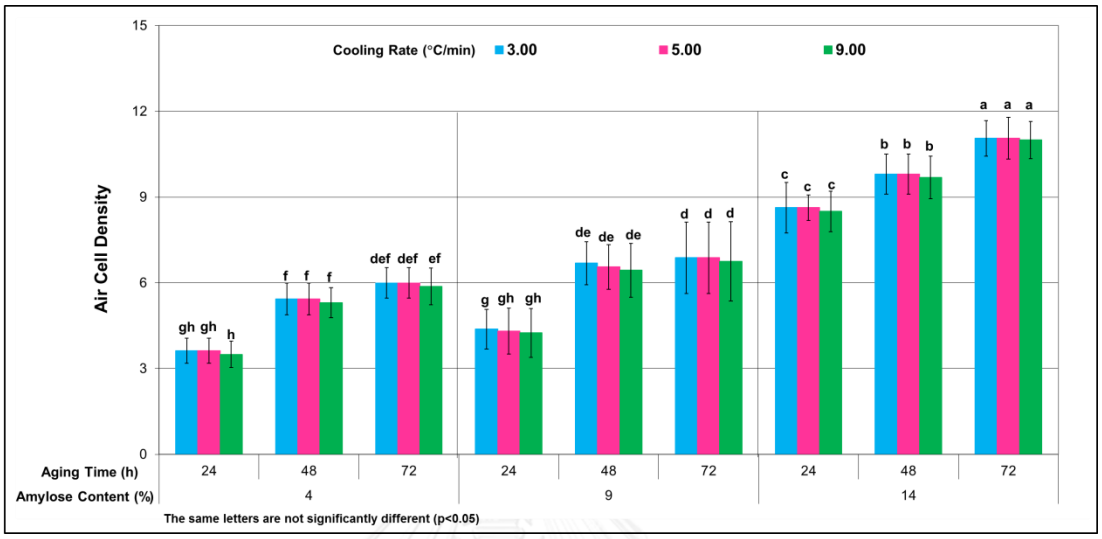


Figure 4.30 Air cell density of puffed products from rice starch gels.

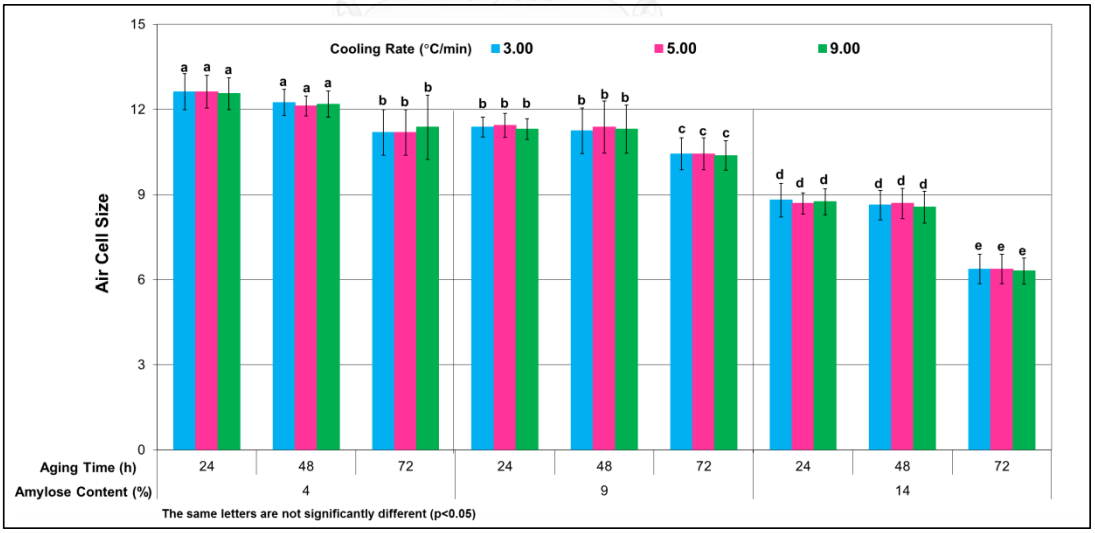


Figure 4.31 Air cell size of puffed products from rice starch gels.

Crispness was evaluated the high pitched sounds when biting the product. Hardness was evaluated the force required for compressing product. Brittleness or

fracturability was evaluated the degree of ease in breaking, fracturing or splintering in the mouth as the force to bite. At a constant cooling rate, as the result shown in Figure 4.32-4.34, the product with higher AC had significantly more crispness, hardness, and brittleness. The longer aging time provided more crispness and hardness while no significantly brittleness when amylose content at 4% and 14%. An interaction between AC and the aging time was also found for these attributes ($p \leq 0.05$). The aging time affected the brittleness score only at the 9.00% AC, as the interaction between these factors was found ($p \leq 0.05$). The content of amylose and time for aging interacted causing the brittleness. The cooling rate did not affect crispness, hardness, and brittleness intensity. The interaction between cooling rate and AC or aging time were not found ($p > 0.05$). The AC is the key factor affecting the puffed product sensory quality with a lower impact from the aging time. This was due to the linear chain structure of amylose formed a strong network resisting expansion, while amylopectin, a highly branched chain structure, formed a weaker network giving expanded texture and lighter product (Chen and Yeh, 2001). The longer aging time resulted in higher RC and retrogradation which was responsible for higher hardness, crispness, and brittleness of the puffed products. A lower retrogradation and crystallinity gave an expanded texture and a lighter and more brittle product which results in lower crispness.

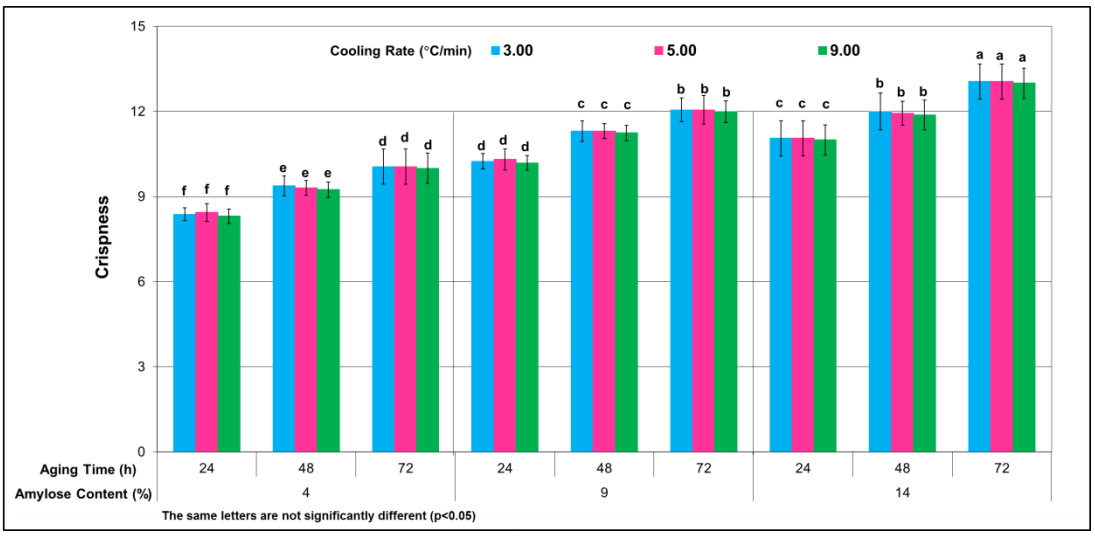


Figure 4.32 Crispness of puffed products from rice starch gels.

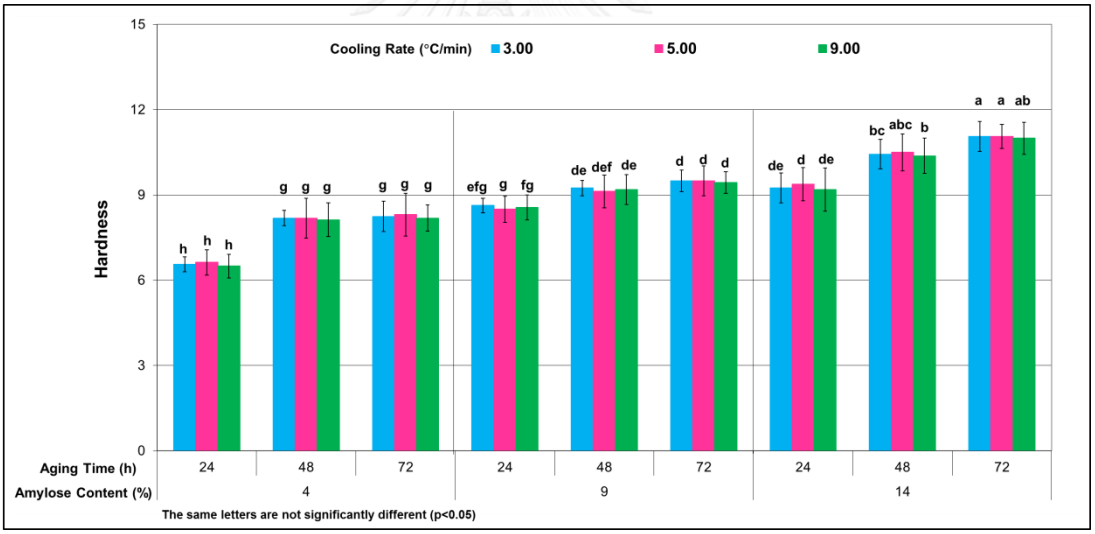


Figure 4.33 Hardness of puffed products from rice starch gels.

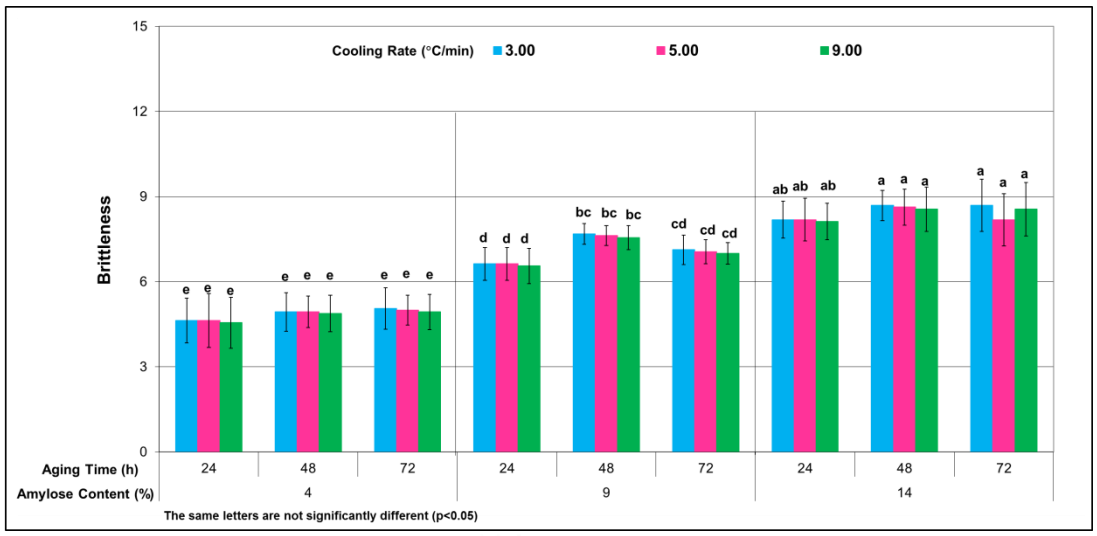


Figure 4.34 Brittleness of puffed products from rice starch gels.

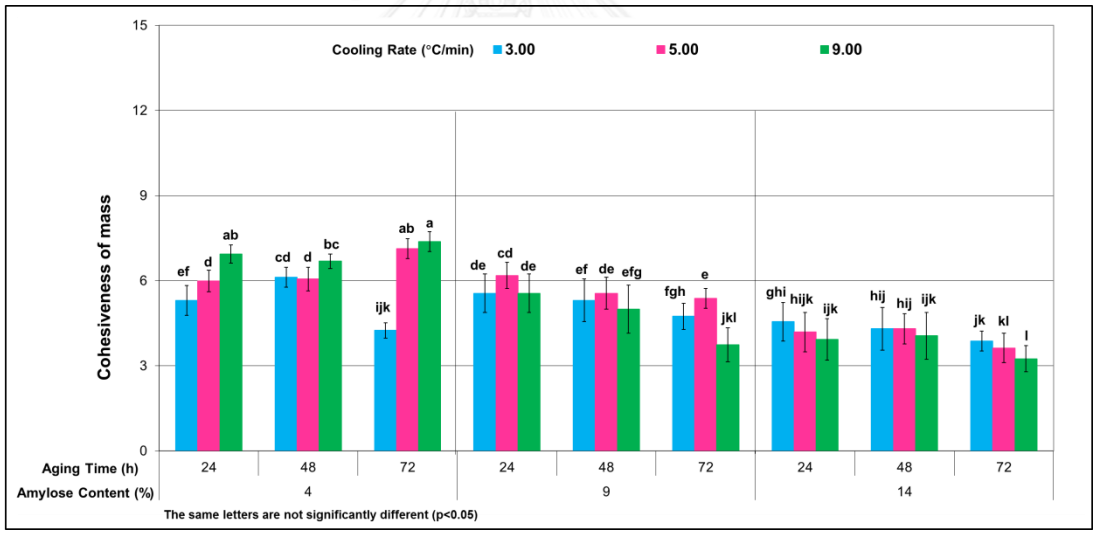


Figure 4.35 Cohesiveness of mass of puffed products from rice starch gels.

Cohesiveness of mass was evaluated degree to which the chewed sample holds together. Tooth stickiness was evaluated the degree that the sample becomes attached to the teeth after mastication. The AC, aging time and cooling rate affected both attributes ($p \leq 0.05$) as shown in Figure 4.35-4.36. There was an interaction of these factors. Therefore, at AC of 9.00% and 14.00%, lower aging time decreased

cohesiveness of mass. Both attributes decreased with increasing AC and aging time. This was due to the fact that low AC (high amylopectin) starch provided sticky starch gel which could combine and hold more mass of starch during chewing. Thus, higher AC and longer aging time thus resulted in a less stickiness and mass holding than the lower one.

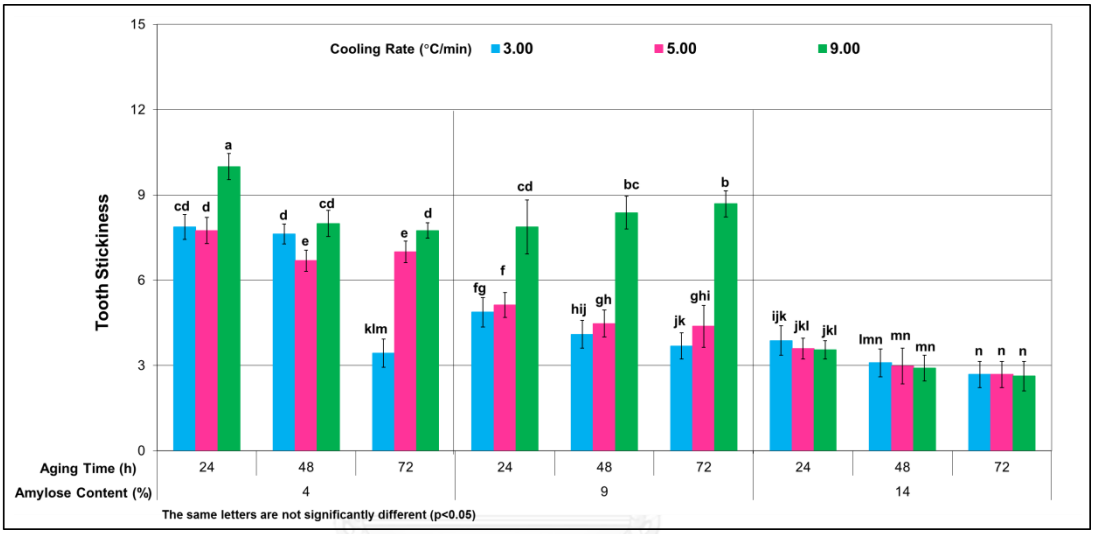


Figure 4.36 Tooth stickiness of puffed products from rice starch gels.

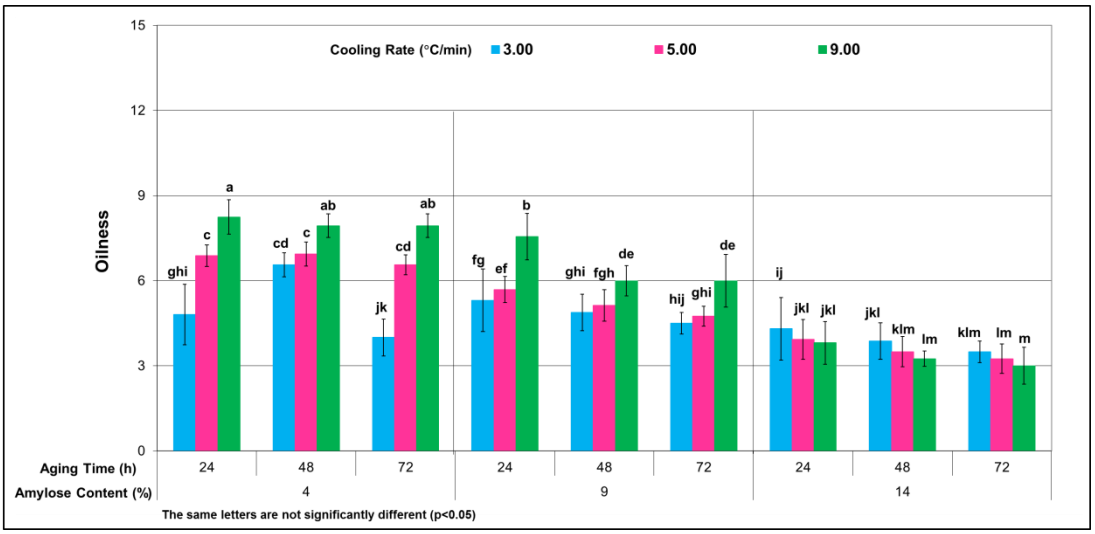


Figure 4.37 Oiliness of puffed products from rice starch gels.

Oiliness was evaluated the amount of oil perceived within the mouth while chewing. Oil coating was evaluated the degree of oil remains in the mouth after swallowing. Both attributes tended to decrease with increasing AC (Figure 4.37-4.38). Because of the interaction between AC and aging time ($p \leq 0.05$), the 4.00% AC resulted in highest oiliness score but aging time had no effect on oiliness. In contrast, for 9.00% AC and 14.00% AC, increasing aging time tended to significantly decrease oiliness. For AC of 4.00% and cooling rate at 9 ºC/min, the aging time had no effect on oil coating. In contrast, for 9.00% AC and 14.00% AC, increasing aging time tended to significantly decrease in oil coating. A decrease in the oiliness and oil coating results from the starch gel network of pellets characterized by compactness and lesser porosity at higher AC and longer aging time. Therefore, the oil absorption of higher AC and longer aging time of the puffed product decreased as a consequence of oiliness and oil coating. This result agreed with Mohamed and Abd Hamid (1994) who reported that there is a high correlation between oil absorption and amylopectin and AC of the rice flour.

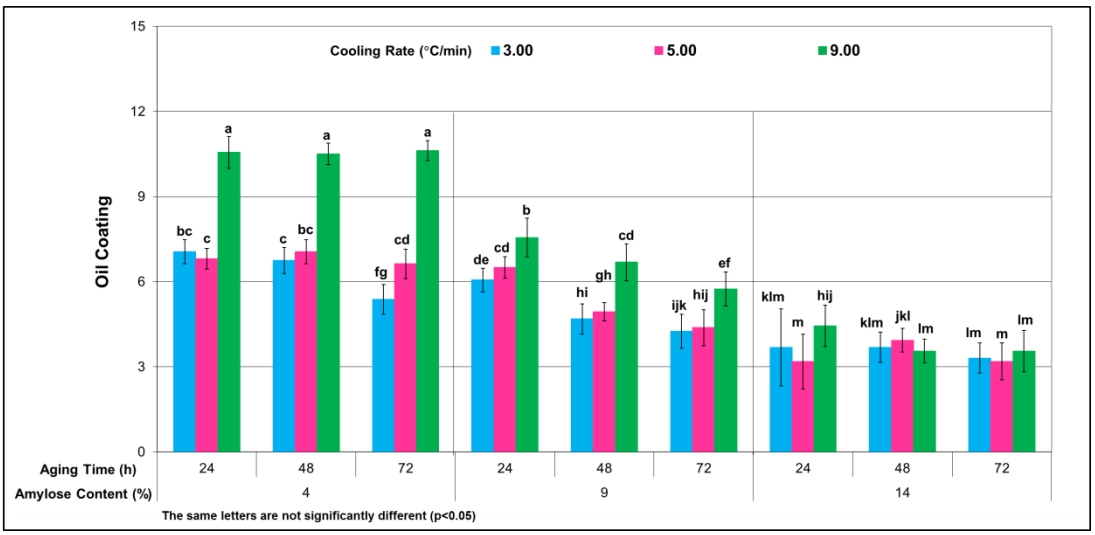


Figure 4.38 Oil Coating of puffed products from rice starch gels.

4.6 Prediction models between crystallinity and pellet properties

Figure 4.39 shows the relationship between the RC and hardness and fracturability of the pellet samples with a coefficient of determination (R^2) for regression equations 3 and 4. Pellets with higher RC had greater hardness and fracturability. Starch re-crystallization developed during aging affecting the properties of dried starch gel or pellets. Upon cooling, a three-dimensional network develops in the continuous phase as a result of leached amylose re-association (Tukomane and Varavinit, 2008) which increases rigidity and crystallinity of starch gels. Drying of starch gels causes moisture loss, a dense starch network and crust formation. As a result, the starch gel puffs when the RC within the network is suitable to expand the network matrix. The retrogradation of starch gel progress resulting in the higher RC of the pellet and hence increase gel strength. The physical properties, as hardness and fracturability of the puffed product were also related to pellet properties (Figure 4.40) which can be determined as in equations 5 and 6. The hardness and fracturability of puffed product increased as pellet hardness and fracturability increased. The determination of puffed product properties from RC of pellet may be difficult in practicing as routine work in the industrial level. Therefore, the hardness and fracturability of pellet could be also used for determine puffed product properties.

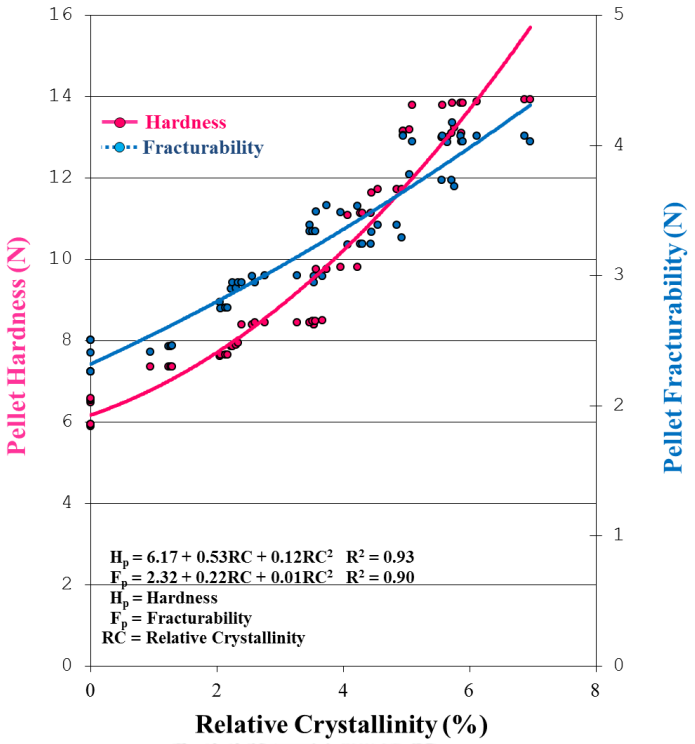


Figure 4.39 Correlation between relative crystallinity of pellets and pellet properties

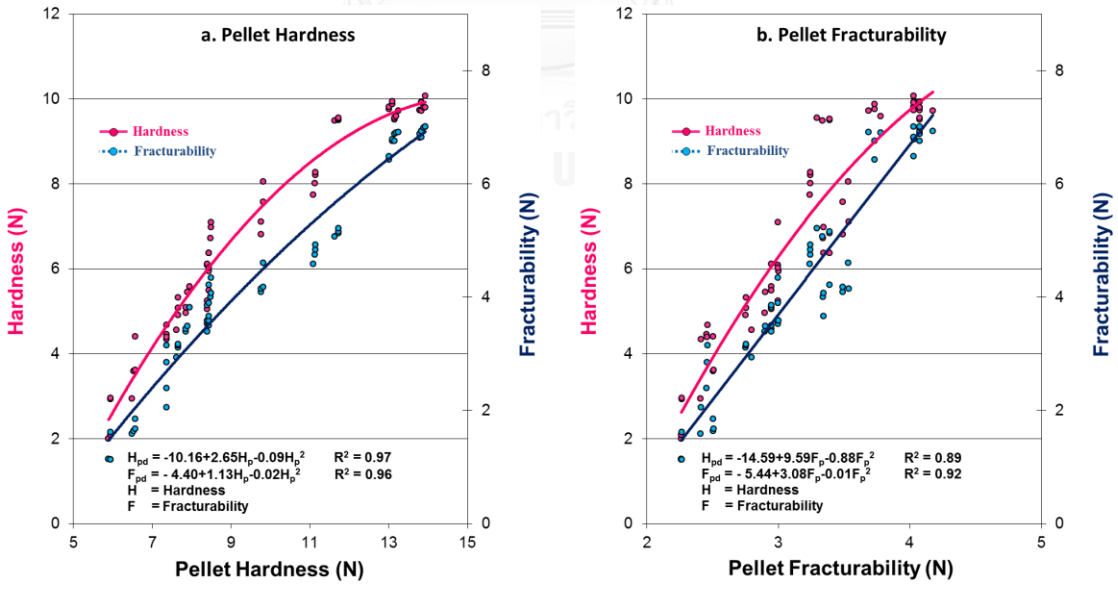


Figure 4.40 Determination of puffed product hardness and fracturability by pellet hardness and fracturability.

$$H_p = 6.17 + 0.53RC + 0.12RC^2 \quad R^2 = 0.93 \quad [3]$$

$$F_p = 2.32 + 0.22RC + 0.01RC^2 \quad R^2 = 0.90 \quad [4]$$

$$H_{pd} = -10.16 + 2.65H_p - 0.09H_p^{\,2} \quad R^2 = 0.97 \quad [5]$$

$$F_{pd} = -5.44 + 3.08F_p - 0.01F_p^{\,2} \quad R^2 = 0.92 \quad [6]$$

(H = hardness; F = fracturability. Subscript p or pd = properties of pellet or puffed product)

4.7 Pellet crystallinity and fried-puffed product properties

Pellets with higher RC were characterized by greater hardness, fracturability and bulk density but also a lower expansion ratio of puffed product as show in Figure 4.41 and Figure 4.42. The hardness and fracturability of puffed products could be determined by pellet RC as shown in the equations 7 and 8. Bulk density and expansion ratio also displayed good correlations with RC as shown in equations 9 and 10. A higher level of RC forms a strong network which resists expansion, while lower RC starch gel forms a weaker network producing an expanded texture and a lighter product. Therefore, a certain degree of RC is required to obtain a suitable gel strength which will result in desirable properties for puffed products.

$$H_{pd} = 2.82 + 1.20RC \quad R^2 = 0.94 \quad [7]$$

$$F_{pd} = 1.31 + 0.89RC \quad R^2 = 0.94 \quad [8]$$

$$BD = 0.33 + 0.03RC \quad R^2 = 0.87 \quad [9]$$

$$ER = 2.62 - 0.13RC \quad R^2 = 0.81 \quad [10]$$

(BD = bulk density; ER = expansion ratio)

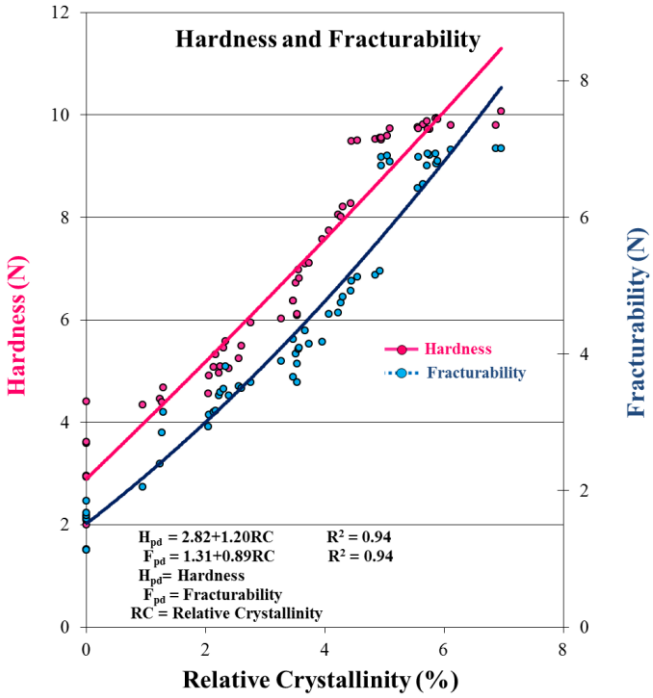


Figure 4.41 Correlation between relative crystallinity of pellets and hardness and fracturability of puffed product.

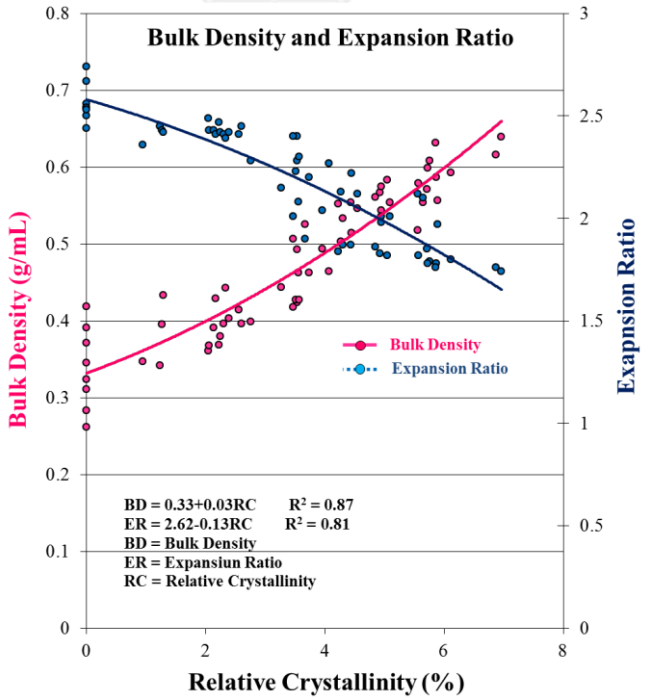


Figure 4.42 Correlation between relative crystallinity of pellets and bulk density and expansion ratio of puffed product.

4.8 Prediction models between pellet crystallinity and puffed product sensory attributes

Figure 4.43 shows the pellets with higher RC resulted in a higher air cell density, puffiness, crispness, hardness and brittleness but lower air cell size, expansion, and oiliness of puffed product. Figure 4.43 also shows the correlation between pellet RC and some sensory attributes of puffed products. Regression models with high R^2 are shown in equations 11, 12, 13, 14, 15, 16 and 17. With retrogradation and crystallinity of starch gel, the linear chain structure of amylose results in more retrogradation and crystallinity which resists expansion thus, resulting in a hard texture. However, the cohesiveness of mass and tooth stickiness showed regression models with poor R^2 shown in Figure 4.40.

$$AD = 2.54 + 0.36RC + 0.22RC^2 \quad R^2 = 0.86 \quad [11]$$

$$AS = 12.96 + 0.17RC - 0.23RC^2 \quad R^2 = 0.93 \quad [12]$$

$$E = 17.64 - 3.29RC + 0.22RC^2 \quad R^2 = 0.86 \quad [13]$$

$$P = -3.61 + 4.30RC - 0.29RC^2 \quad R^2 = 0.85 \quad [14]$$

$$C = 4.69 + 2.41RC - 0.18RC^2 \quad R^2 = 0.86 \quad [15]$$

$$Hs = 4.49 + 1.54RC - 0.06RC^2 \quad R^2 = 0.85 \quad [16]$$

$$B = -0.73 + 3.14RC - 0.26RC^2 \quad R^2 = 0.90 \quad [17]$$

$$O = 11.68 - 52.49RC + 0.17RC^2 \quad R^2 = 0.74 \quad [18]$$

(AD = air cell density; AS = air cell size; E = expansion; P = puffiness; C = crispness; H_s= hardness from sensory evaluation; B = brittleness; O = oiliness)

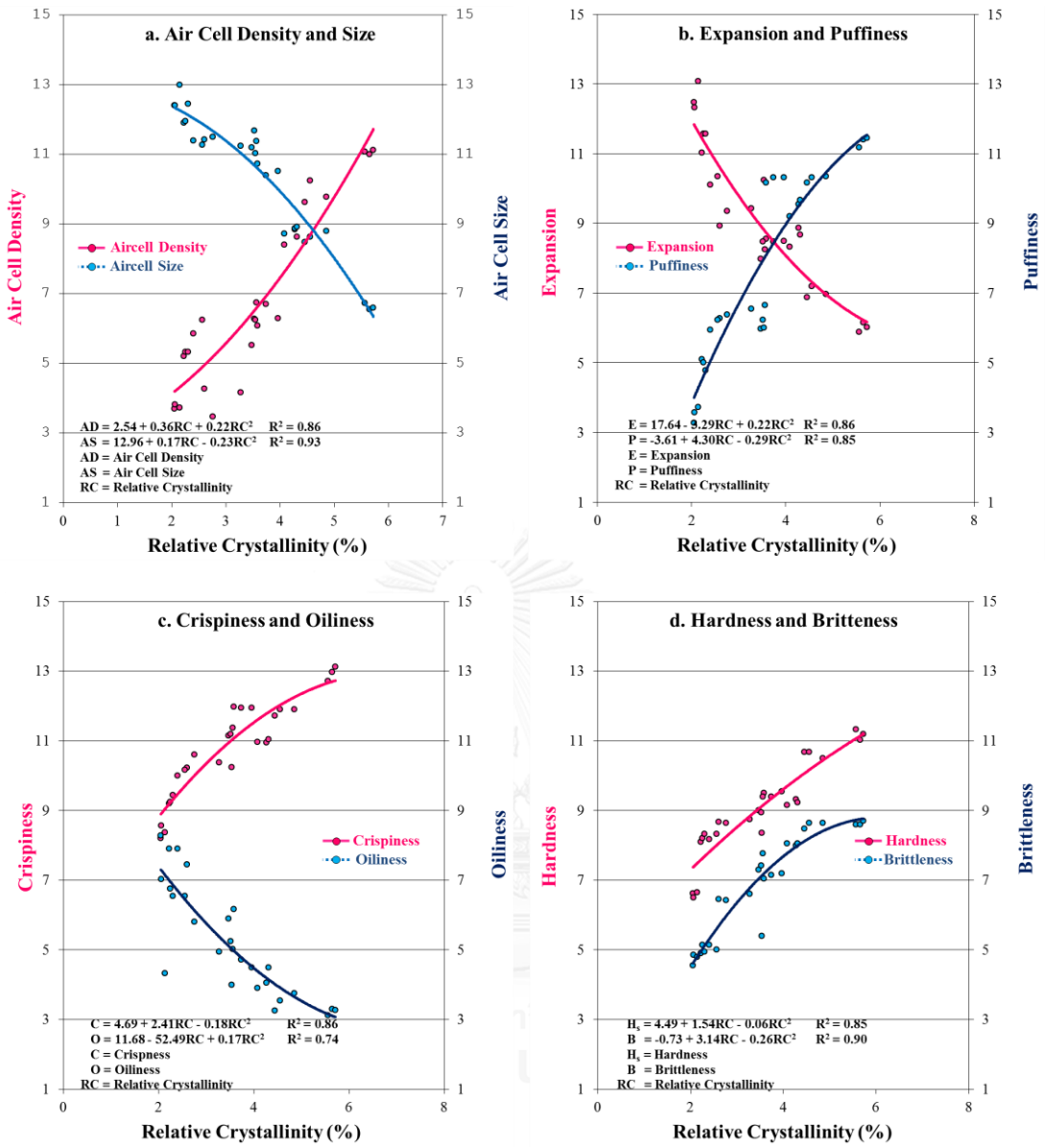


Figure 4.43 Correlation between relative crystallinity of pellet and puffed product characteristics from sensory evaluation.

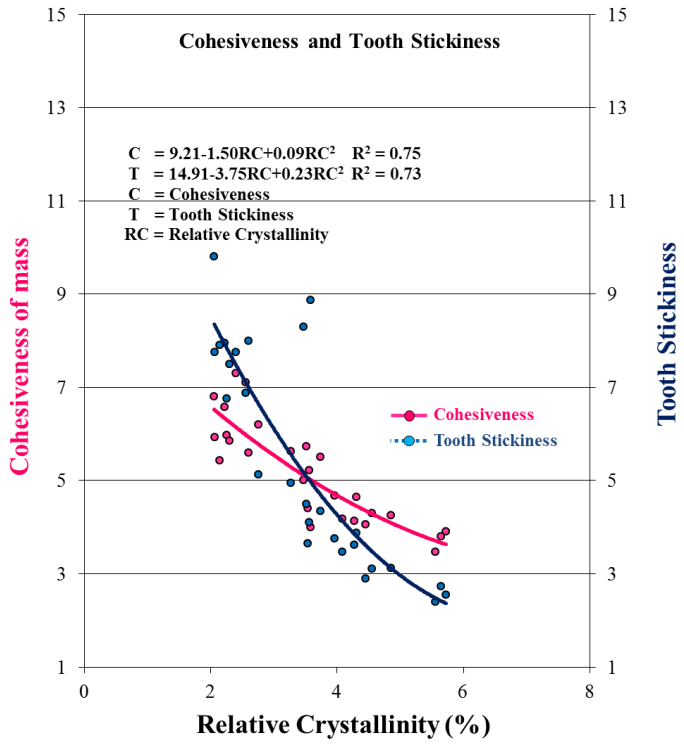


Figure 4.44 Correlation between relative crystallinity of pellet and cohesiveness of mass and tooth stickiness.

Table 4.12 shows the correlations between sensory and instrumental data. This is in line with previous studies which reported that crispness related directly to percentage (or ratio) of expansion (Kyaw et al., 2001; Siaw et al., 1985). An important textural attribute of puffed products is fracturability which often is described using several sensory terms such as crispy. Faller and Heymann (1996) reported that the breaking strength of puffed products was also highly positively correlated with sensory crispness. Therefore, sensory attributes could be reasonably supported by the corresponding physical characteristics to explain characteristics of puffed products containing various RC. Daget and Collyer (1984) also reported that physically and mechanically related sensory attributes could be evaluated effectively using physical measurements.

Table 4.12 The pearson correlation (r) of properties from instrumental analysis and sensory evaluation

Instrument	**Sensory attributes**				
value	Hardness	Brittleness	Puffiness	Crispness	Oiliness
Hardness	**0.93**	**0.94**	**0.91**	**0.88**	**0.86**
Fracturability	**0.90**	**0.85**	**0.87**	**0.90**	**0.78**
Puffiness	**0.85**	**0.87**	**0.83**	**0.90**	**0.74**
Lipid	**0.91**	**0.94**	**0.93**	**0.91**	**0.77**

4.9 Validation of models

The predicted values from models and experimental values from physical properties of the 9 puffed products were compared (Table 4.13) and no significant differences ($p>0.05$) were found between them. The models of pellet RC were reliable for predicting the hardness, fracturability, bulk density, and expansion ratio of puffed products.

Table 4.13 Comparison of predicted and experimental values

Amylose content (%)	Aging Time (h)	Relative crystallinity (%)	Hardness[ns]		Fracturability[ns]		Bulk Density[ns]		Expansion Ratio[ns]	
			Predicted[1]	Experimental	Predicted[2]	Experimental	Predicted[3]	Experimental	Predicted[4]	Experimental
4	24	2.06	4.88	4.47±0.64	3.14	3.14±0.32	0.39	0.39±0.01	2.35	2.43±0.03
4	48	2.25	5.07	5.24±0.37	3.31	3.20±0.37	0.40	0.40±0.00	2.33	2.37±0.03
4	72	2.56	5.38	5.40±0.57	3.59	3.25±0.43	0.41	0.42±0.01	2.29	2.23±0.03
9	24	2.76	5.58	5.65±0.35	3.76	3.36±0.57	0.41	0.41±0.01	2.26	2.26±0.07
9	48	3.52	6.34	6.09±0.16	4.44	4.47±0.47	0.44	0.44±0.03	2.16	2.23±0.03
9	72	3.74	6.56	6.76±0.36	4.63	4.72±0.28	0.44	0.47±0.01	2.13	2.14±0.07
14	24	4.27	7.09	7.23±0.39	5.11	4.83±0.43	0.46	0.45±0.00	2.07	2.07±0.06
14	48	4.55	7.37	7.25±0.26	5.36	5.42±0.56	0.47	0.47±0.01	2.03	2.17±0.08
14	72	5.65	8.47	8.55±0.42	6.34	6.25±0.44	0.50	0.50±0.01	1.89	1.89±0.02

[1] from equation 5: $H_{pd} = 2.82 + 1.20RC$

[2] from equation 6: $F_{pd} = 1.31 + 0.89RC$

[3] from equation 7: $BD = 0.33 + 0.03RC$

[4] from equation 8: $ER = 2.62 - 0.13RC$

H is hardness, F is fracturability, BD is bulk density, and ER is expansion ratio. Subscript pd indicates property of puffedproduct.

Mean±SD is reported and ns indicated the data in the same row and each property are not significantly different ($p>0.05$)

An acceptance test was conducted with 60 assessors (Figure 4.41). From descriptive analysis of 27 samples, the cooling rate had no effect on crispness, hardness, and brittleness which were attributes to test acceptability. Therefore, the cooling rate was the factor would not be considered. Among the 9 puffed samples, the puffed product with 9% AC and 48 h of aging time (pellet RC = 3.5%) had the highest score for liking of appearance. The 3 samples with 9% AC (2.79-3.74% RC) had the highest scores for texture. Samples with higher AC had higher crispiness, air cell opacity, and density but lower hardness, fracturability, and expansion than the samples with lower AC. The panelists preferred the puffed product with specific characteristics which were neither too low nor too high in crispness and hardness. This was consistent with the test from the just-about-right scales which found that puffed products with an AC of 9% and aging time for 48 h were found to have just the right intensity of crispness and hardness by 78.3 and 76.3% of assessors as shown in Table 4.14 and 4.15, respectively. Therefore, a certain degree of RC is required to obtain a suitable pellet yielding desirable properties for puffed products.

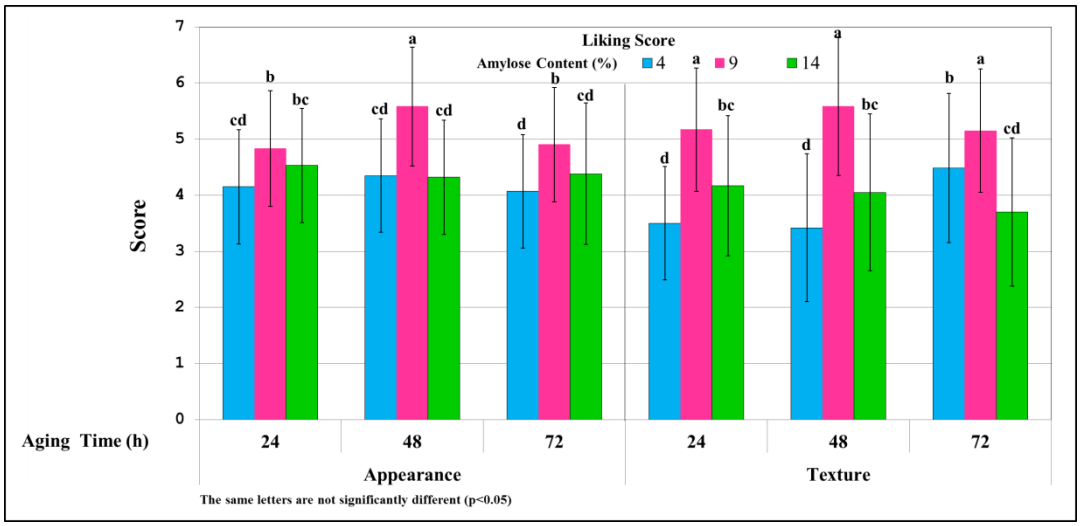


Figure 4.45 Liking scores from acceptance test

Table 4.14 Just about right for crispness of puffed product

Amylose Content (%)	Percentage of consumer (N=60)					Aging Time (h)
	Much too weak	Little too weak	Just right	Little too strong	Much too strong	
4	36.1	37.7	18.0	3.3	4.9	24
	15.0	43.3	31.7	5.0	5.0	48
	5.0	38.3	40.0	11.7	5.0	72
9	3.3	20.0	58.3	16.7	1.7	24
	0.0	6.7	78.3	13.3	1.7	48
	1.6	4.9	55.7	36.1	1.6	72
14	0.0	1.7	41.7	48.3	8.3	24
	0.0	0.0	21.7	63.3	15.0	48
	0.0	0.0	13.3	73.3	13.3	72

Table 4.15 Just about right for hardness of puffed product

Amylose Content (%)	Percentage of consumer (N=60)					Aging Time (h)
	Much too weak	Little too weak	Just right	Little too strong	Much too strong	
4	36.7	43.3	20.0	0.0	0.0	24
	21.7	58.3	20.0	0.0	0.0	48
	15.0	45.0	33.3	6.7	0.0	72
9	5.0	28.3	55.0	10.0	1.7	24
	0.0	10.2	76.3	11.9	1.7	48
	1.7	1.7	68.3	26.7	1.7	72
14	0.0	1.7	46.7	41.7	10.0	24
	0.0	0.0	13.3	65.0	21.7	48
	0.0	0.0	16.7	66.7	16.7	72

CHAPTER V
CONCLUSION

Puffed product characteristics (namely hardness, fracturability, bulk density, expansion ratio, crispness and oiliness) were strongly affected by relative crystallinity of retrograded starch gel and pellets. The relative crystallinity was influenced by amylose content, aging time and cooling rate. The higher amylose content and aging time resulted in higher relative crystallinity. The higher relative crystallinity provided a higher hardness and fracturability of gel, pellets, and puffed product, crispness and bulk density, but also resulted in a lower expansion ratio and less oiliness. Regression models showed that the relative crystallinity of pellets can be an excellent predictor of properties (hardness, fracturability, bulk density, and expansion ratio) and the sensory characteristics (air cell size, hardness, brittleness, crispness, and oiliness) of puffed products. The instrumental value (hardness, fracturability and puffiness) correlated well to the sensory attributes (hardness, brittleness, and crispness). Products which were made from a 9% amylose content rice starch mixture and which had a relative crystallinity of 2.8-3.7% were most accepted. The main attributes affecting the liking decision of consumers were crispness and hardness.

Recommendations for future research

Nowadays an important criterion for consumer's choice is healthy food especially low fat food. Therefore, to puff the product, baking is preferable to frying. The effects of baking time and temperature on product properties should be studied. Furthermore, the use of freeze drying has increased for commercialized products. Freeze-drying may be used in the drying step. Freeze-drying is expected to preserve the crystalline structure formed during the aging process. Hence, it will alter the physical properties and sensory attributes of puffed products, as well as the acceptance of consumers.

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APPENDIX

APPENDIX A

Amperometric method

Procedure (Williams et al., 1970)

The galvanometer was set to zero, with the use of water, a reagent blank, and amylopectin blank. Starch was dispersed in 10 ml. of 0.5N KOH solution for 5 min with a magnetic stirrer. Distilled water (75 ml) was added, followed by 10 ml of 1.0 N HCl and 5 ml of 0.4N KI solution, making the total volume up to 100 ml. The solution was titrated against 0.005N KIO_3 solution by means of the circuit illustrated in Figure A.1., and was stirred continuously with a magnetic stirrer working in the opposite direction to the rotating platinum electrode. This assisted in maintaining the sample in complete dispersion, and also stabilized reaction time. Potassium iodate solution was added in 0.25 ml increments. Galvanometer readings were taken at zero time and 1.5 min after the start of each addition of KIO_3. The rotating electrode was driven at 600 rpm by a suitable motor. After each analysis, the bridge, electrode, and burette tip were rinsed with demineralized distilled water and immersed in a vial of distilled water.

Typical titration curves are illustrated in Figure A.2. The end point was determined by extrapolation. A perpendicular was dropped from the point of intersection of the two arms of the curve to determine the precise location of the end point. To check precision of the test a sample of 6 mg pure amylose was analyzed each day during the course of the experiments. The amount of KIO_3 solution required ranged from 1.70-1.72 ml, which represented a variation from 19.06-19.28% iodine absorbed by the amylose with a mean value of 19.24%. The literature value for the

iodine absorption of potato amylose is 19.5, which gives the amylose used in the work a purity of 98.7%

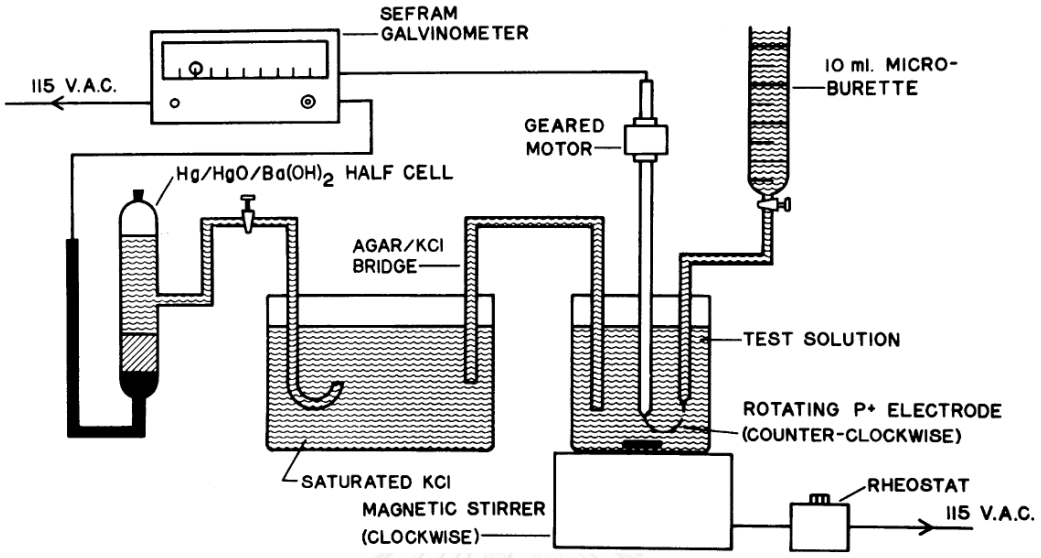


Figure A.1 Schematic diagram of circuit used for amperometric determination of amylose.

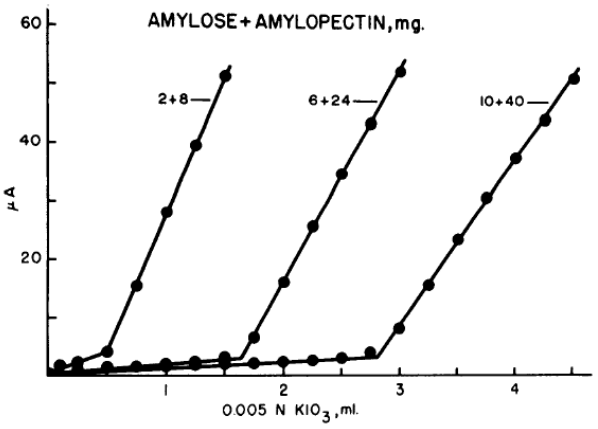


Figure A.2 Titration curves obtained on titration of increasing amounts of amylose at constant amylose-amylopectin ratio.

Calculation

The amount of iodine absorbed by a given weight of amylose can be calculated as follows:

$$(63.46/W)V_a = \%\ \text{iodine absorbed}$$

where W is the sample weight in mg and V_a is the tritration value in ml 0.005 N KIO_3 obtained for the amylose sample.

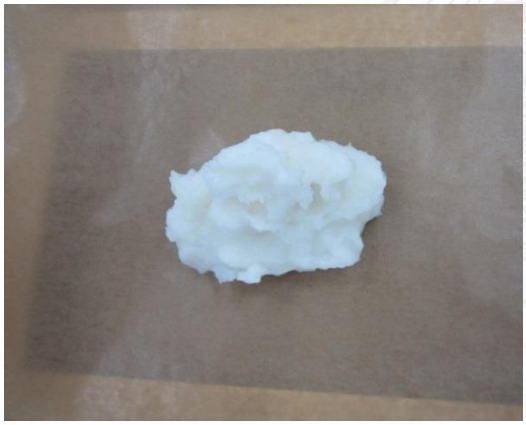
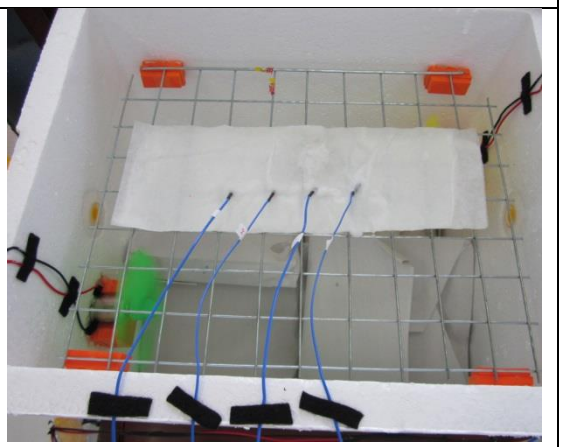
The amount of amylose present in a starch sample is calculated from

$$X = (63.46V_s)/19.24,$$

where V_s = titration value in ml 0.0005 N KIO_3 obtained for the starch sample, 19.24 is average value obtained experimentally for the percentage of iodine absorbed by the standard amylose, and X is the weight of amylose present in the sample. The percentage of amylose in a starch sample is given by 100(X/W), where W is the weight of starch ample on moisture-free basis.

APPENDIX B

Puffed product making process

1. Stirring starch mixture	**2. Heating at 110 ºC for 20 min**
	
3. Forming starch paste/gel	**4. Spreading starch paste/gel by rolling**
	
5. Cutting the starch paste	**6. Cooling in cooling box**
	

7. Recording the data by data logger 	**8. Aging for 24, 48 , and 72 h** 
9. Cutting and drying the starch gel 	**10. Frying dried starch pellet** 

Figure B.1 Puffed product making process

APPENDIX C

Statistical analysis

C.1 Statistical analysis of gel properties

Table C.1.1 Statistical analysis: Relative crystallinity of gel

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	2084.031[a]	59	35.323	228.048	.000
Intercept	7125.430	1	7125.430	46002.916	.000
Amylose	120.500	4	30.125	194.491	.000
AgingTime	10.123	2	5.061	32.677	.000
CoolingRate	.572	3	.191	1.231	.306
Amylose * AgingTime	825.829	8	103.229	666.461	.000
Amylose * CoolingRate	291.534	12	24.295	156.849	.000
AgingTime * CoolingRate	150.824	6	25.137	162.290	.000
Amylose * AgingTime * CoolingRate	684.650	24	28.527	184.175	.000
Error	9.293	60	.155		
Total	9218.755	120			
Corrected Total	2093.325	119			

Table C.1.2 Statistical analysis: ΔH_{rt} of gel

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	175.285[a]	59	2.971	978.466	.000
Intercept	467.780	1	467.780	154061.356	.000
Amylose	162.053	4	40.513	13342.905	.000
AgingTime	4.711	2	2.355	775.742	.000
CoolingRate	1.548	3	.516	169.937	.000
Amylose * AgingTime	1.625	8	.203	66.893	.000
Amylose * CoolingRate	1.064	12	.089	29.199	.000
AgingTime * CoolingRate	.845	6	.141	46.399	.000
Amylose * AgingTime * CoolingRate	3.439	24	.143	47.194	.000
Error	.182	60	.003		
Total	643.247	120			
Corrected Total	175.468	119			

Table C.1.3 Statistical analysis: Gel hardness

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	12886.446[a]	59	218.414	107.728	.000
Intercept	142481.606	1	142481.606	70276.074	.000
Amylose	11857.198	4	2964.299	1462.079	.000
Rate	388.438	3	129.479	63.863	.000
AgingTime	373.224	2	186.612	92.042	.000
Amylose * AgingTime	155.890	8	19.486	9.611	.000
Amylose * Rate	49.975	12	4.165	2.054	.034
Rate * AgingTime	5.247	6	.875	.431	.855
Amylose * Rate * AgingTime	56.475	24	2.353	1.161	.313
Error	121.647	60	2.027		
Total	155489.699	120			
Corrected Total	13008.093	119			

C.2 Statistical analysis of pellet properties

Table C.2.1 Statistical analysis: Relative crystallinity of pellet

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	457.578[a]	35	13.074	643.887	.000
Intercept	1380.873	1	1380.873	68009.021	.000
Amylose	421.618	4	105.404	5191.245	.000
AgingTime	27.436	2	13.718	675.629	.000
CoolingRate	4.269	3	1.423	70.090	.000
Amylose * AgingTime	2.277	8	.285	14.020	.000
Amylose * CoolingRate	1.230	12	.103	5.049	.000
AgingTime * CoolingRate	.747	6	.124	6.131	.000
Error	1.706	84	.020		
Total	1840.156	120			
Corrected Total	459.283	119			

a. R Squared = .996 (Adjusted R Squared = .995)

Table C.2.2 Statistical analysis: ΔH_r of pellet

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	845.036[a]	35	24.144	201.611	.000
Intercept	2815.466	1	2815.466	23510.266	.000
Amylose	773.889	4	193.472	1615.571	.000
AgingTime	44.473	2	22.236	185.683	.000
CoolingRate	6.936	3	2.312	19.306	.000
Amylose * AgingTime	16.679	8	2.085	17.410	.000
Amylose * CoolingRate	2.685	12	.224	1.869	.050
AgingTime * CoolingRate	.374	6	.062	.520	.791
Error	10.059	84	.120		
Total	3670.561	120			
Corrected Total	855.096	119			

a. R Squared = .988 (Adjusted R Squared = .983)

Table C.2.3 Statistical analysis: Pellet hardness

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	863.197[a]	59	14.630	156.735	.000
Intercept	11747.835	1	11747.835	125853.844	.000
Amylose_Content	793.708	4	198.427	2125.736	.000
AgingTime	22.724	2	11.362	121.722	.000
Cooling_Rate	2.550	3	.850	9.105	.000
Amylose_Content * AgingTime	14.111	8	1.764	18.897	.000
Amylose_Content * Cooling_Rate	8.588	12	.716	7.667	.000
AgingTime * Cooling_Rate	4.199	6	.700	7.496	.000
Amylose_Content * AgingTime * Cooling_Rate	17.317	24	.722	7.730	.000
Error	5.601	60	.093		
Total	12616.633	120			
Corrected Total	868.798	119			

a. R Squared = .994 (Adjusted R Squared = .987)

Table C.2.4 Statistical analysis: Pellet fracturability

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	40.480[a]	59	.686	30.313	.000
Intercept	1249.412	1	1249.412	55201.755	.000
Amylose_Content	36.235	4	9.059	400.235	.000
AgingTime	1.624	2	.812	35.884	.000
Cooling_Rate	.071	3	.024	1.041	.381
Amylose_Content * AgingTime	1.139	8	.142	6.288	.000
Amylose_Content * Cooling_Rate	.323	12	.027	1.188	.312
AgingTime * Cooling_Rate	.109	6	.018	.806	.569
Amylose_Content * AgingTime * Cooling_Rate	.979	24	.041	1.803	.034
Error	1.358	60	.023		
Total	1291.250	120			
Corrected Total	41.838	119			

C.3 Statistical analysis of puffed product properties

Table C.3.1 Statistical analysis: Puffed product hardness

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	7.295E6	59	123643.084	88.511	.000
Intercept	5.920E7	1	5.920E7	42382.024	.000
Amylose	6765219.295	4	1691304.824	1210.737	.000
Rate	69877.818	3	23292.606	16.674	.000
AgingTime	319282.697	2	159641.348	114.281	.000
Amylose * AgingTime	97582.063	8	12197.758	8.732	.000
Amylose * Rate	22897.820	12	1908.152	1.366	.208
Rate * AgingTime	1198.579	6	199.763	.143	.990
Amylose * Rate * AgingTime	18883.700	24	786.821	.563	.939
Error	83815.285	60	1396.921		
Total	6.658E7	120			
Corrected Total	7378757.256	119			

Table C.3.2 Statistical analysis: Fracturability of puffed product

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	4.074E6	59	69056.570	39.643	.000
Intercept	2.349E7	1	2.349E7	13486.802	.000
Amylose	3771183.954	4	942795.988	541.233	.000
Rate	28431.555	3	9477.185	5.441	.002
AgingTime	154256.312	2	77128.156	44.277	.000
Amylose * AgingTime	104869.438	8	13108.680	7.525	.000
Amylose * Rate	7072.874	12	589.406	.338	.979
Rate * AgingTime	1398.608	6	233.101	.134	.991
Amylose * Rate * AgingTime	7124.862	24	296.869	.170	1.000
Error	104516.425	60	1741.940		
Total	2.767E7	120			
Corrected Total	4178854.027	119			

a. R Squared = .975 (Adjusted R Squared = .950)

Table C.3.3 Statistical analysis: Bulk Density of puffed product

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1.092[a]	59	.019	83.816	.000
Intercept	26.439	1	26.439	119722.274	.000
Amylose	.897	4	.224	1015.416	.000
Rate	.132	3	.044	199.471	.000
AgingTime	.041	2	.020	92.772	.000
Amylose * AgingTime	.006	8	.001	3.347	.003
Amylose * Rate	.012	12	.001	4.540	.000
Rate * AgingTime	.000	6	5.777E-5	.262	.953
Amylose * Rate * AgingTime	.004	24	.000	.696	.835
Error	.013	60	.000		
Total	27.545	120			
Corrected Total	1.105	119			

a. R Squared = .988 (Adjusted R Squared = .976)

Table C.3.4 Statistical analysis: Expansion Ratio of puffed product

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	9.362[a]	59	.159	82.578	.000
Intercept	574.487	1	574.487	298968.093	.000
Amylose	7.339	4	1.835	954.808	.000
Rate	1.209	3	.403	209.709	.000
AgingTime	.215	2	.107	55.900	.000
Amylose * AgingTime	.047	8	.006	3.057	.006
Amylose * Rate	.431	12	.036	18.707	.000
Rate * AgingTime	.034	6	.006	2.978	.013
Amylose * Rate * AgingTime	.087	24	.004	1.880	.025
Error	.115	60	.002		
Total	583.965	120			
Corrected Total	9.477	119			

C.4 Statistical analysis of sensory characteristics

Table C.4.1 Statistical analysis: Expansion from sensory evaluation

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	839.719[a]	25	33.589	98.221	.000
Intercept	17921.557	1	17921.557	52406.619	.000
Amylose	673.676	2	336.838	984.989	.000
Time	125.780	2	62.890	183.904	.000
Rate	.627	2	.314	.917	.401
Block	6.212	7	.887	2.595	.014
Amylose * Time	33.102	4	8.275	24.199	.000
Amylose * Rate	.130	4	.032	.095	.984
Time * Rate	.192	4	.048	.140	.967
Error	64.975	190	.342		
Total	18826.250	216			
Corrected Total	904.693	215			

Table C.4.2 Statistical analysis: Puffiness from sensory evaluation

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1514.523[a]	25	60.581	395.520	.000
Intercept	12558.375	1	12558.375	81991.045	.000
Amylose	1122.437	2	561.219	3664.082	.000
Time	314.528	2	157.264	1026.744	.000
Rate	.965	2	.483	3.151	.045
Block	3.329	7	.476	3.105	.004
Amylose * Time	73.014	4	18.253	119.173	.000
Amylose * Rate	.222	4	.056	.363	.835
Time * Rate	.028	4	.007	.045	.996
Error	29.102	190	.153		
Total	14102.000	216			
Corrected Total	1543.625	215			

a. R Squared = .981 (Adjusted R Squared = .979)

Table C.4.3 Statistical analysis: Air cell opacity from sensory evaluation

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	3027.060[a]	25	121.082	352.475	.000
Intercept	12027.311	1	12027.311	35011.871	.000
Amylose	2955.100	2	1477.550	4301.194	.000
Time	46.946	2	23.473	68.331	.000
Rate	.768	2	.384	1.118	.329
Block	8.383	7	1.198	3.486	.002
Amylose * Time	15.102	4	3.776	10.991	.000
Amylose * Rate	.267	4	.067	.195	.941
Time * Rate	.492	4	.123	.358	.838
Error	65.269	190	.344		
Total	15119.640	216			
Corrected Total	3092.329	215			

a. R Squared = .979 (Adjusted R Squared = .976)

Table C.4.4 Statistical analysis: Air cell density from sensory evaluation

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1193.303[a]	25	47.732	91.841	.000
Intercept	10264.449	1	10264.449	19749.783	.000
Amylose	942.183	2	471.091	906.425	.000
Time	229.530	2	114.765	220.819	.000
Rate	.725	2	.362	.697	.499
Block	11.921	7	1.703	3.277	.003
Amylose * Time	8.880	4	2.220	4.271	.002
Amylose * Rate	.039	4	.010	.019	.999
Time * Rate	.025	4	.006	.012	1.000
Error	98.748	190	.520		
Total	11556.500	216			
Corrected Total	1292.051	215			

a. R Squared = .924 (Adjusted R Squared = .914)

Table C.4.5 Statistical analysis: Air cell size from sensory evaluation

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	797.442[a]	25	31.898	102.693	.000
Intercept	23002.042	1	23002.042	74054.033	.000
Amylose	661.646	2	330.823	1065.069	.000
Time	105.007	2	52.503	169.033	.000
Rate	.021	2	.010	.034	.967
Block	11.366	7	1.624	5.227	.000
Amylose * Time	19.181	4	4.795	15.438	.000
Amylose * Rate	.167	4	.042	.134	.970
Time * Rate	.056	4	.014	.045	.996
Error	59.016	190	.311		
Total	23858.500	216			
Corrected Total	856.458	215			

a. R Squared = .931 (Adjusted R Squared = .922)

Table C.4.6 Statistical analysis: Crispness from sensory evaluation

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	418.025[a]	25	16.721	96.077	.000
Intercept	25263.407	1	25263.407	145160.692	.000
Amylose	290.572	2	145.286	834.796	.000
Time	119.287	2	59.644	342.705	.000
Rate	.280	2	.140	.805	.449
Block	6.574	7	.939	5.396	.000
Amylose * Time	1.255	4	.314	1.802	.130
Amylose * Rate	.012	4	.003	.017	.999
Time * Rate	.046	4	.012	.067	.992
Error	33.067	190	.174		
Total	25714.500	216			
Corrected Total	451.093	215			

a. R Squared = .927 (Adjusted R Squared = .917)

Table C.4.7 Statistical analysis: Hardness from sensory evaluation

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	340.134[a]	25	13.605	46.694	.000
Intercept	17478.005	1	17478.005	59984.722	.000
Amylose	242.252	2	121.126	415.706	.000
Time	84.350	2	42.175	144.744	.000
Rate	.211	2	.105	.361	.697
Block	4.606	7	.658	2.258	.031
Amylose * Time	8.498	4	2.124	7.291	.000
Amylose * Rate	.199	4	.050	.171	.953
Time * Rate	.019	4	.005	.016	1.000
Error	55.361	190	.291		
Total	17873.500	216			
Corrected Total	395.495	215			

a. R Squared = .860 (Adjusted R Squared = .842)

Table C.4.8 Statistical analysis: Fracturability from sensory evaluation

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	509.966[a]	25	20.399	50.060	.000
Intercept	9997.362	1	9997.362	24534.578	.000
Amylose	484.211	2	242.105	594.152	.000
Time	13.766	2	6.883	16.892	.000
Rate	.350	2	.175	.429	.652
Block	7.369	7	1.053	2.584	.014
Amylose * Time	4.234	4	1.058	2.598	.038
Amylose * Rate	.005	4	.001	.003	1.000
Time * Rate	.032	4	.008	.020	.999
Error	77.421	190	.407		
Total	10584.750	216			
Corrected Total	587.388	215			

a. R Squared = .868 (Adjusted R Squared = .851)

Table C.4.9 Statistical analysis: Cohesiveness of mass from sensory evaluation

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	265.348[a]	25	10.614	29.652	.000
Intercept	5729.890	1	5729.890	16007.262	.000
Amylose	174.030	2	87.015	243.089	.000
Time	12.127	2	6.064	16.940	.000
Rate	8.565	2	4.282	11.964	.000
Block	8.397	7	1.200	3.351	.002
Amylose * Time	11.025	4	2.756	7.700	.000
Amylose * Rate	43.921	4	10.980	30.675	.000
Time * Rate	7.282	4	1.821	5.086	.001
Error	68.012	190	.358		
Total	6063.250	216			
Corrected Total	333.360	215			

a. R Squared = .796 (Adjusted R Squared = .769)

Table C.4.10 Statistical analysis: Oiliness from sensory evaluation

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	538.199[a]	25	21.528	53.288	.000
Intercept	5985.042	1	5985.042	14814.603	.000
Amylose	342.507	2	171.253	423.899	.000
Time	22.799	2	11.399	28.216	.000
Rate	64.750	2	32.375	80.137	.000
Block	8.755	7	1.251	3.096	.004
Amylose * Time	12.069	4	3.017	7.469	.000
Amylose * Rate	77.639	4	19.410	48.044	.000
Time * Rate	9.681	4	2.420	5.991	.000
Error	76.759	190	.404		
Total	6600.000	216			
Corrected Total	614.958	215			

a. R Squared = .875 (Adjusted R Squared = .859)

Table C.4.11 Statistical analysis: Tooth stickiness from sensory evaluation

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1084.454[a]	25	43.378	104.852	.000
Intercept	6292.082	1	6292.082	15208.971	.000
Amylose	657.160	2	328.580	794.231	.000
Time	59.945	2	29.973	72.449	.000
Rate	172.803	2	86.401	208.846	.000
Block	6.720	7	.960	2.320	.027
Amylose * Time	28.828	4	7.207	17.421	.000
Amylose * Rate	133.273	4	33.318	80.536	.000
Time * Rate	25.725	4	6.431	15.545	.000
Error	78.605	190	.414		
Total	7455.140	216			
Corrected Total	1163.058	215			

a. R Squared = .932 (Adjusted R Squared = .924)

Table C.4.12 Statistical analysis: Oil coating from sensory evaluation

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1021.312[a]	25	40.852	100.008	.000
Intercept	7095.574	1	7095.574	17370.179	.000
Amylose	670.308	2	335.154	820.467	.000
Time	34.586	2	17.293	42.333	.000
Rate	182.843	2	91.421	223.802	.000
Block	2.167	7	.310	.758	.623
Amylose * Time	19.053	4	4.763	11.661	.000
Amylose * Rate	109.421	4	27.355	66.967	.000
Time * Rate	2.935	4	.734	1.796	.131
Error	77.613	190	.408		
Total	8194.500	216			
Corrected Total	1098.926	215			

R Squared = .929 (Adjusted R Squared = .920)

APPENDIX D

Thermograms from Differential Scanning Calorimeter

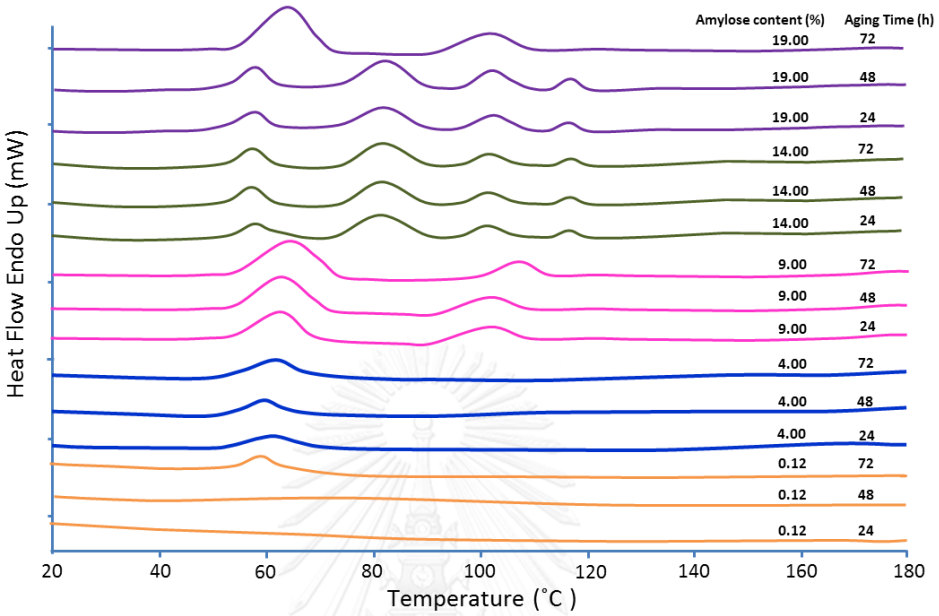


Figure D.1 Starch gel cooled at 1 ºC/min.

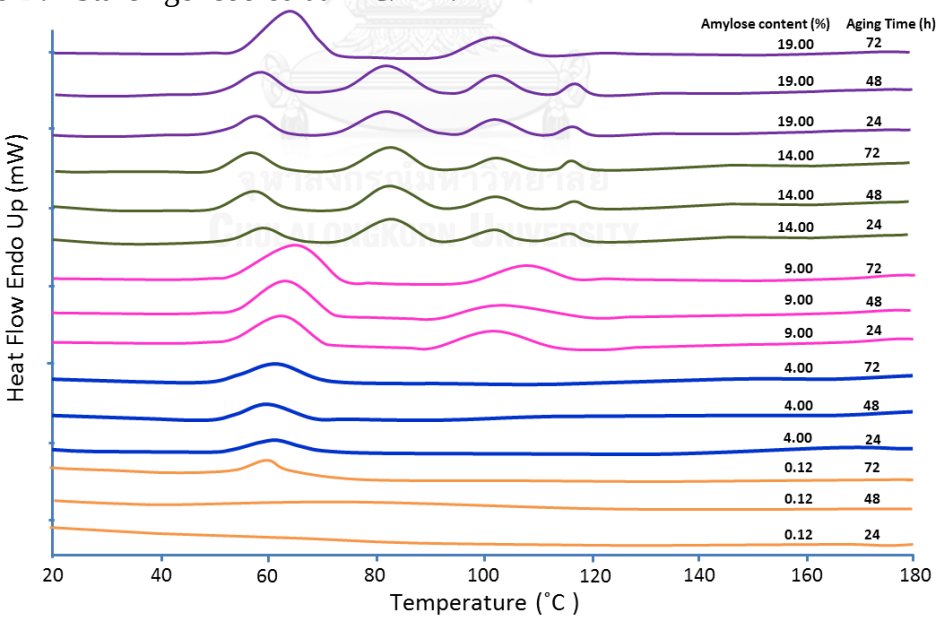


Figure D.2 Starch gel cooled at 3 ºC/min.

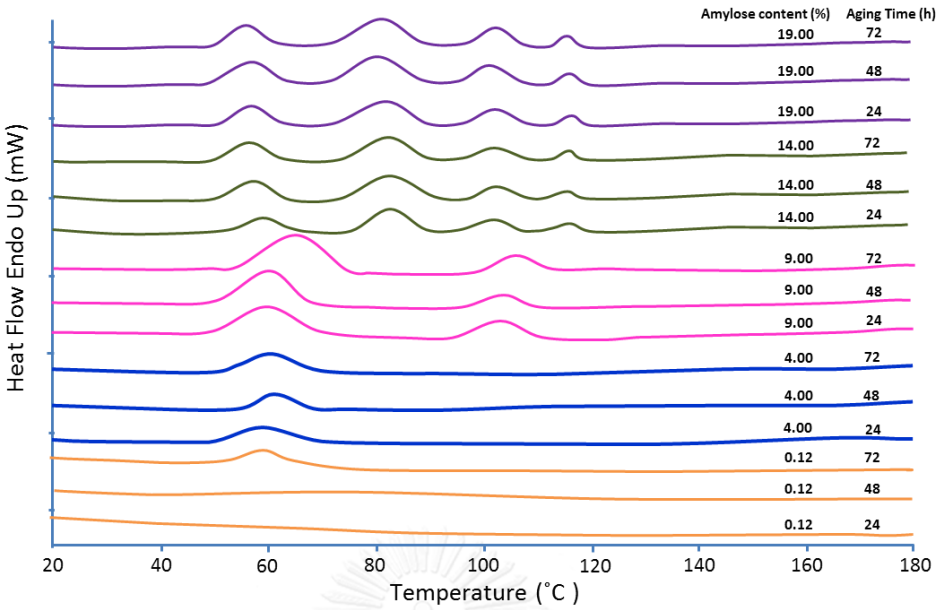


Figure D.3 Starch gel cooled at 5 ºC/min.

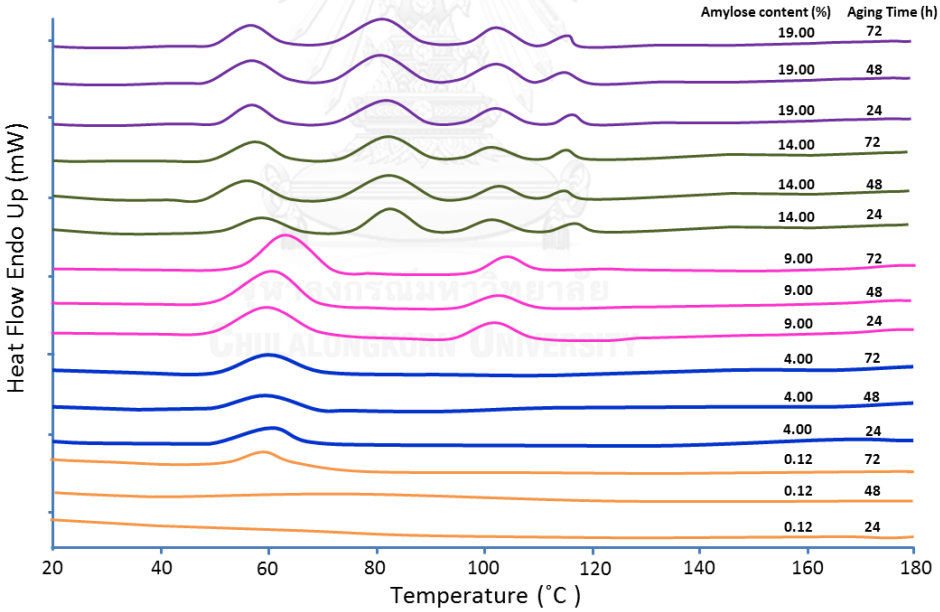


Figure D.4 Starch gel cooled at 9 ºC/min.

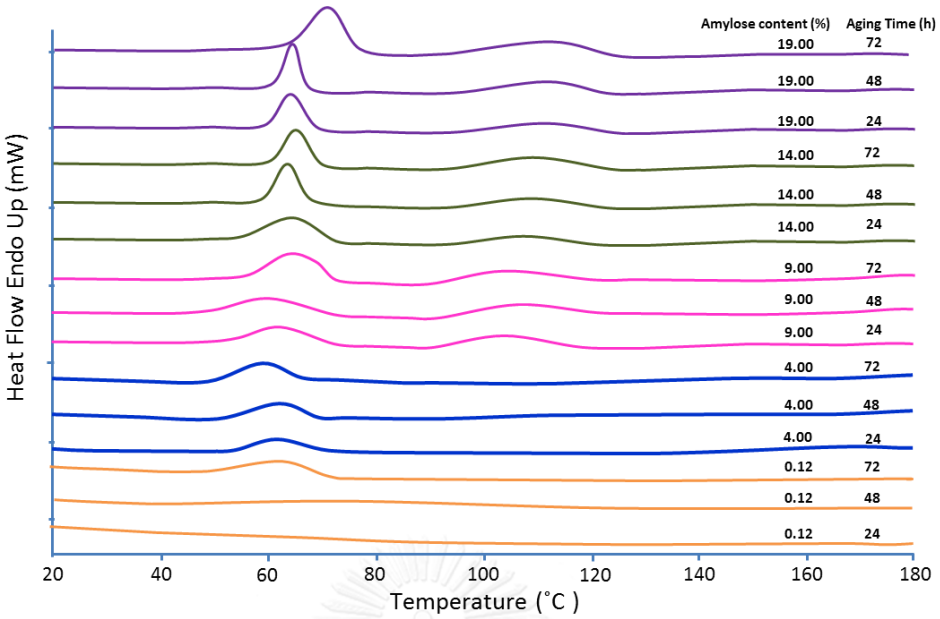


Figure D.5 Pellet from starch gel cooled at 1 ºC/min.

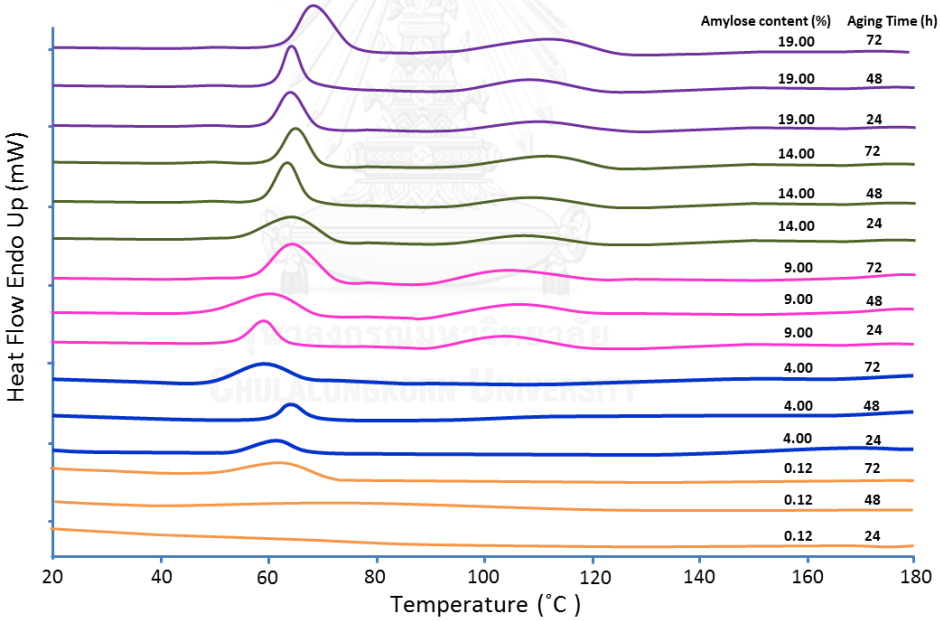


Figure D.6 Pellet from starch gel cooled at 3 ºC/min.

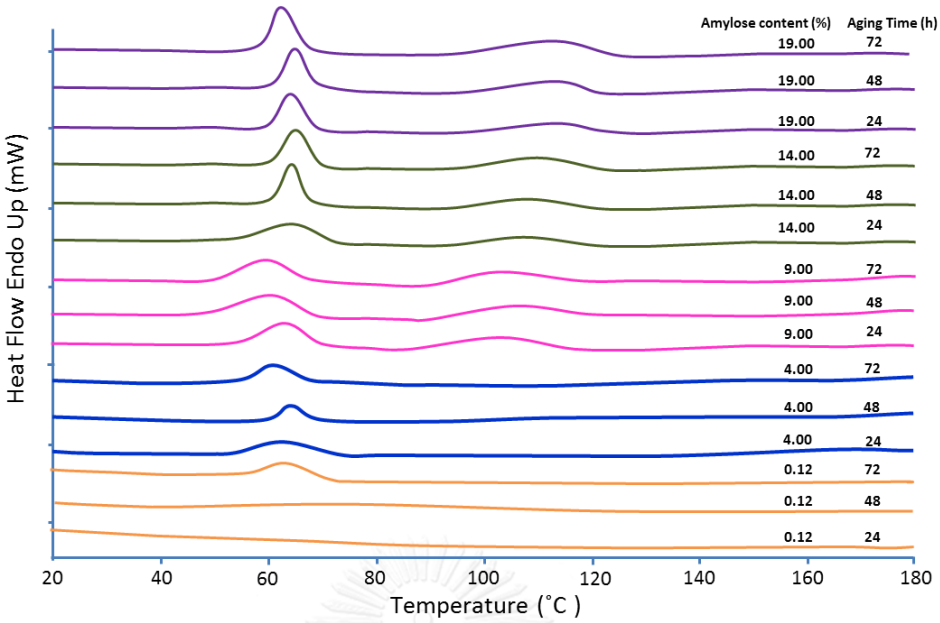


Figure D.7 Pellet from starch gel cooled at 5 ºC/min.

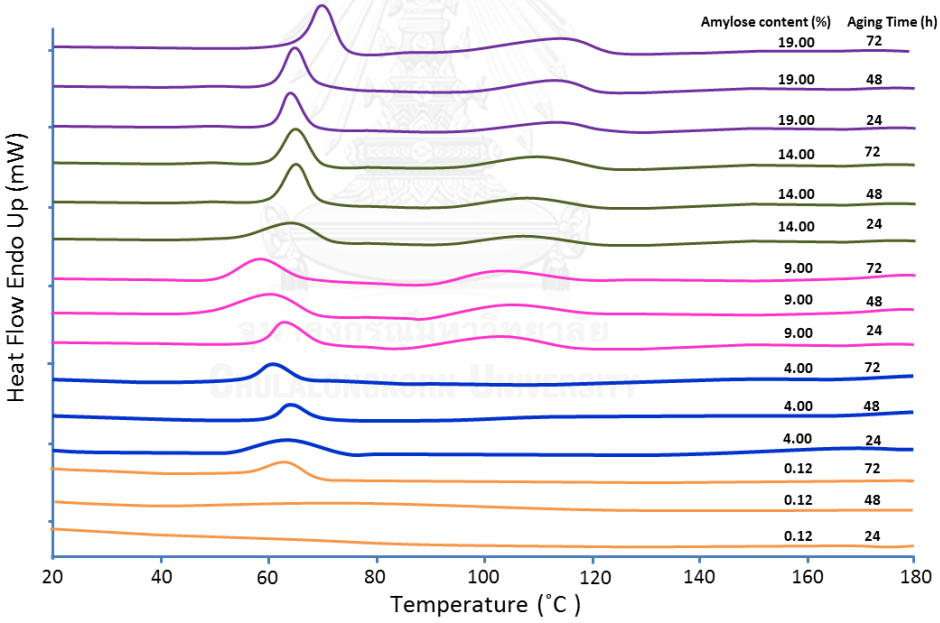


Figure D.8 Pellet from starch gel cooled at 9 ºC/min.

VITA

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