

กลไกการเกิดข้าวตอก



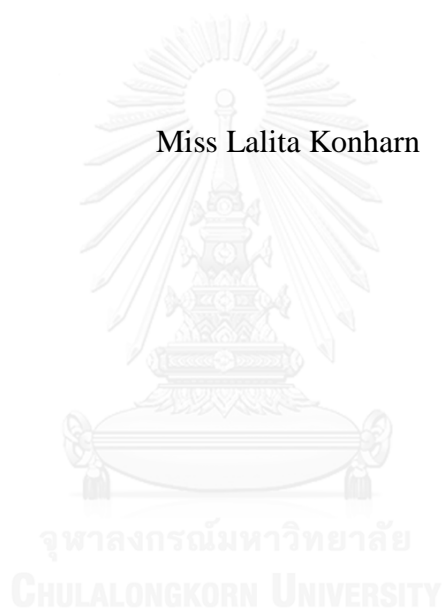
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Popping mechanism of popped rice

Miss Lalita Konharn



A Thesis Submitted in Partial Fulfillment of the Requirements  
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วิทยานิพนธ์นี้นำเสนอกลไกการเกิดข้างตอก และอิทธิพลของอุณหภูมิที่ส่งผลต่อ  
 โครงสร้างของข้าวตอกเปรียบเทียบกับข้าวโพดคั่ว กระบวนการทำข้าวตอกเกิดจากการให้ความ  
 ร้อนแก่ข้าวเปลือกจนกระทั่งเมล็ดข้าวระเบิดและพองตัวกลายเป็นข้าวตอกพร้อมกับปลดปล่อย  
 เสียงป๊อป เนื่องจากเปลือกของข้าวประกอบด้วย 2 ส่วนคือ lemma และ palea เกาะเกี่ยวกันบริเวณ  
 ที่เรียกว่า จุดเชื่อมประสาน ซึ่งเป็น โครงสร้างที่ปิดแน่น จึงทำให้เปลือกข้าวทนทานต่ออุณหภูมิสูง  
 กว่าเปลือกของข้าวโพด การที่ข้าวตอกระเบิดที่อุณหภูมิสูงกว่าข้าวโพดคั่วส่งผลให้มีการสะสมของ  
 แรงดันไอน้ำที่เกิดจากความชื้นที่สะสมอยู่ภายในเมล็ดที่มีความร้อนยวดยิ่ง มีค่าสูงกว่าในข้าวโพด  
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 อุณหภูมิสูงกว่าข้าวโพดคั่วทำให้ข้าวตอกกระโดดได้สูงกว่าข้าวโพดคั่ว ความรุนแรงในการระเบิด  
 ทำให้เมล็ดและเปลือกข้าวกระเด็นแยกหลุดออกจากกัน ข้าวตอกบางเมล็ดกระโดดได้สูงมากกว่า  
 100 cm ขณะที่ความสูงในการกระโดดของข้าวโพดคั่วไม่เกิน 10 cm

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This thesis presents the popping mechanism and the influence of temperature on structure of popped rice. We compared the popping process of popped rice and popped corn. When a rice grain/corn kernel was hot air heated, they exploded into popped products with an emitted characteristic “pop” sound. The thermally stable rice husk with an air-tight interlocking of lemma and palea made rice grain popped at a higher temperature as compared to that of popped corn. The higher popping temperature generated a superheated water of higher pressure and a greater mass of water involving in the popping process. The high pressure of superheated steam ruptured the rice husk. At this moment, the superheated steam exploded the microscopic starch granules into inter-connected starch balloons with micrometer-thin starch walls. SEM images confirmed a larger pore size at greater popping temperature. The popped rice expanded ~21 times the grain volume while the popped corn expanded ~13 times volume of kernel. As the popping process, the transformation of rice and corn did not undergo chemical changes while they only transformed their structures. Due to a higher popping temperature and a greater mass of water involving in the popping process, the popped rice jumped higher into the air with an instantaneous separation of popped rice and its husk. The popped rice jump as high as 100 cm compared to less than 10 cm of popped corn.

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**LIST OF SYMBOLS AND ABBREVIATIONS**

SEM	: Scanning electron microscope
TGA	: Thermal gravitation analysis
DSC	: Differential scanning calorimeter
FT-IR	: Fourier-transform infrared spectrometer
SLO-MO app	: SLO-MO application
AOAC	: Association of official analytical chemists
fps	: Frame per second
$\mu\text{m}$	: Micrometer
mm	: Millimeter
cm	: Centimeter
$\text{cm}^3$	: Cubic centimeter
$\text{cm}^{-1}$	: Reciprocal centimeter
$\text{m}^3$	: Cubic meter
L	: Liter
mg	: Milligram
g	: Gram
kg	: Kilogram
ms	: Millisecond
$\text{ms}^{-1}$	: Meter per second
s	: Second
min	: Minute
hr	: Hour
$^{\circ}\text{C}$	: Degree Celsius
K	: Kelvin
kV	: Kilovolt

kPa	: Kilopascal
mol	: Mole
atm	: Atmosphere
N <sub>2</sub>	: Nitrogen
3D	: Three dimension
NA	: Not available
Avg.	: Average
n	: Mole
R	: Gas constant
T	: Temperature
T <sub>p</sub>	: Peak temperature
T <sub>pop</sub>	: Popping temperature
P	: Pressure
P <sub>ss</sub>	: Pressure of saturated steam
M <sub>g</sub>	: Mass of a grain
M <sub>k</sub>	: Mass of a kernel
M <sub>p</sub>	: Mass of a popped product
M <sub>w</sub>	: Mass of water
V	: Volume
V <sub>g</sub>	: Volume of a grain
V <sub>k</sub>	: Volume of a kernel
V <sub>p</sub>	: Volume of a popped product
ρ <sub>g</sub>	: Density of a grain
ρ <sub>p</sub>	: Density of a popped product
ρ <sub>sw</sub>	: Density of superheated water
ρ <sub>s</sub>	: Density of steam

## CHAPTER I

### INTRODUCTION

Rice is one of wild grasses originated in southern India 10,000-14,000 years ago. Indian people employ rice as main food in their meals since 2000 B.C. Rice spreads to Asia, Europe, Africa, America and New South Wales. In 2007, approximately 660 million tons of rice were produced [1-3]. Corn, on the other hand, was originated in Mexico far back to 8000 B.C. It spreads to Canada and the South of Argentina [4]. It is consumed as the staple food crop since 1,000 years ago. Nowadays, rice and corn are the most common staple food. Both of them can be made into several kinds of foods. The whole grain brings numerous beneficial nutrients [5].

Some varieties of rices and corns have been employed for making popped products by heating un-milled rice and corn kernel by hot air for 45 s [6]. The popped rice and popped corn are ready to eat. The process of making popped rice has been revealed since a hundred years ago. In India, the country of popped rice production, popped rice accounted for 10% of all rice production [2]. It is also a cereal for breakfast and infant food in Southeast Asia [5, 7]. Although the popping process of popped rice is similar to that of popped corn, surprisingly, popped corn was developed in the U.S. since 1930s. In 1998, the economic value of popped corn was estimated at 43 billion U.S. dollars in snack-food industry [4]. The improvement of corn varieties and development of popping protocol were continuously improved for plentiful available corns and satisfying demand. However, popped rice did not reach the large scale production of economic value product. Popped rice is normally assumed as a symbol of cultural heritage relating to the way of life, especially in South and Southeast Asia. Popped rice is only used in religious ceremony and as local snack [5, 7].

Popped corn was systematically studied almost 100 years ago. In the fundamental study of popped corn, the study of popping mechanism leads to an

improvement of the quality of popped corn [8-13]. For the popping process of popcorn, many researchers have revealed that the popping process composed of the gelatinization and expansion of moisture content within the starch granule upon heating [13, 14]. The expansion of moisture generates a high pressure (110-120 psi) that ruptures the corn hull at the critical temperature (~170-190 °C). The steam pressure in corn kernel directly affected the physical properties of popped corn [6, 15-17]. In addition, the reduction of pressure surrounding the corn kernel before popping also increase the expansion volume of popped corn [18]. Recently, the popping mechanism and jumping behavior of popped corn were elucidated. Upon heating of corn kernel on a hotplate, the protruding gelatinized starch or 'leg' compresses on the plate. After that, a jumping of the popped corn a few millimeters to several centimeters high with an emission of 'pop' sound is observed. The popped corn turned somersault with a rotational angle of ~490°. From the measurement, the researchers obtained 96% of popcorn which popped at 180 °C [19]. From the results of popping studies of popped corn, the moisture content, amount of amylose and amylopectin in the starch granule, and the physical strength of hull are the crucial parameters in the popping process. The systematic study led popped corn into the development of business plan and global market which resulted in an increase of marketing value of popped corn greater than popped rice for a billion dollars in annually snack market.

However, there are few reports focused on the fundamental understanding of popped rice. The investigation of popped rice was only studied among Asian researchers. The physical and chemical properties of popped rice which was popped in a microwave oven have been reported. Waxy rice which has very low amylose content expands more than that of non-waxy rice which contains high amylose [5]. Nevertheless, the effect of chemical composition in rice grain on popping ability is less important than physical parameters such as husk thickness, the locking efficiency of lemma and palea and the hardness or breaking strength of rice grain. These parameters

affect the husk ability on holding the high pressure generated by the superheated water in rice grain upon heating [20]. Moisture content of 10-17% within the grain initiates the popping after heating at 225-240 °C for 45 s. The optimum condition for best yield is 14% moisture content with 16 times volume expansion while the poor condition is 10% moisture content with 6 times volume expansion [6, 20-22].

Although popped rice has been produced for a long time in Asia, there were no systematic studies on the pressure and popping temperature of popped rice derived from water content. In addition, the influence of popping temperature on the microstructure of popped rice has not been reported. Interestingly, the popped rice has not been introduced in the large-scale snack industry as compared to that of popped corn.

In this research, a comparative study of popped rice and popped corn was performed. This is the first time that the popping temperature was revealed to have an influence on popped rice and popped corn morphologies. A detail of information on pore size, wall thickness, size distribution, thermal properties were investigated in order to gain an insight understanding of the popping mechanism. The fundamental study of popped rice can induce an increase of available popped product of rice besides original rice grain. It is expected that the knowledge can be applied as encapsulation and adsorbent materials by porous morphology. These applications show the valuable commercial products in the future.

### **1.1 Objective of the research**

This contribution performs a comparative study of popped rice and popped corn. This is the first time that popping temperature is revealed to have influence on popped rice and popped corn morphology. In addition, this work also investigates the effect of physical parameters which induced the difference in the popping process of popped rice and popped corn. The objectives of this research are shown as follows:

- 1.1.1 To study and compare popping mechanism of popped rice with popped corn.
- 1.1.2 To investigate the influence of popping temperature on the popped rice morphology, i.e., pore size, wall thickness, and size distribution.
- 1.1.3 To investigate the influence of parameters influencing the popping process of popped rice, i.e., defects on the rice husk, thermal properties, and crystallinity.

## **1.2 Scope of the research**

- 1.2.1 To study the popping mechanism and influence of popping temperature on morphologies of waxy rice (Laow-taek) and corn (McGarrett, American popcorn).
- 1.2.2 To analyze popping mechanism of popped rice with popped corn from slow motion VDO clips, i.e., trajectory profiles, jumping height, and popping temperature.
- 1.2.3 To calculate mass of water involved in the popping process based on the ideal gas equation of state ( $PV=nRT$ ), where water and its steam are in equilibrium.
- 1.2.4 To compare the physical properties of un-popped and popped products of rice and corn, i.e., morphology, mass, volume, density, and expansion ratio.
- 1.2.5 To compare the microscopic images of popped rice with popped corn, i.e., pore size, wall thickness, and size distribution.

### **1.3 Expected outcome of the research**

- 1.3.1 Protocol of popping process with highly expanded volume of popped rice.
- 1.3.2 To apply popped rice into several innovations with adding the value of rice, i.e., fragrance encapsulation and slow releasing from porous morphology of popped rice.



## CHAPTER II

### THEORETICAL BACKGROUND

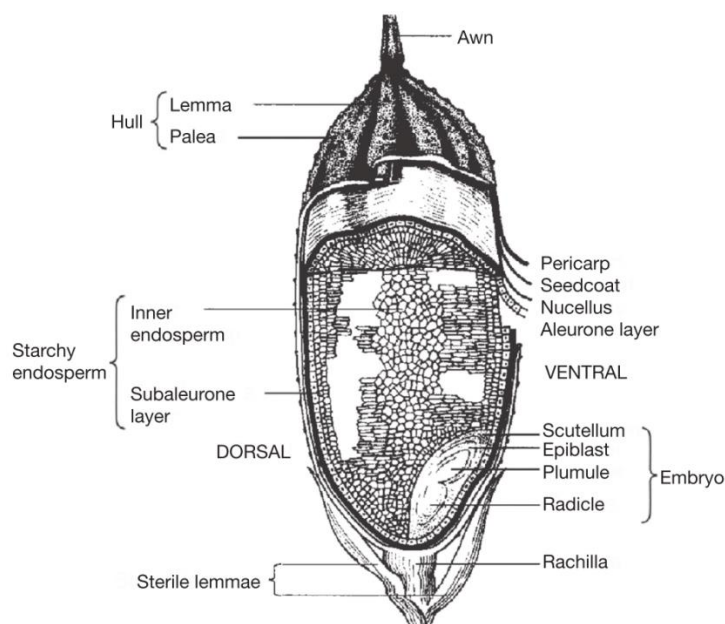
#### 2.1 Cereal grains

##### 2.1.1 Rice

Rice is a kind of wild grasses that may originate in South India 10,000-14,000 years ago. The grains are served as main cereal of meals. In 2000 B.C., the rice had spread out the north of India (the foothills of the Himalayas) and China. A thousand years later, it overspread to Korea, Philippines, Indonesia, Sri Lanka, and Japan. The spreading of rice was not only in Asia, but also in Europe, Africa, America, and New South Wales. Although, there are about 100,000 varieties of rice species having been recorded, only some of them can serve as grain sources. Nowadays, rice is life-sustaining of the world's populations as their primary source of food [1, 3].

Asian rice (*Oryza Sativa* L.) is commonly cultivated in Asia. The genus *Oryza* was divided from *Oryzaceae* which is under the sub-family *Pooideae* in the grass family. *Sativa* name is one of *Sativae* section [23]. The un-milled rice grains consist of several parts (Fig. 2.1). The outermost part is the husk which is divided into several parts. Lemma and palea are the most important part of the husk. They function as the tight enclosure of the rice grain (brown rice) by interlocking structures for maintaining rice seed. The area size of lemma is bigger than palea. The other parts of the husk include awn, sterile lemmas, and rachilla [22, 23]. Recently, Chungcharoen *et al.* constructed a geometry model of un-milled rice for studying the characteristics of germinated, un-milled rice. The model represented the lengths of un-milled rice ( $1.15 \times 4.91 \times 0.925$  mm) and rice grain ( $1.05 \times 3.85 \times 0.85$  mm). The germinated rice grain and their husk were assumed to the point of attachment in the center while the gaps appear in both of the end points of them. As a result, the rice grain is smaller than its husk [24].





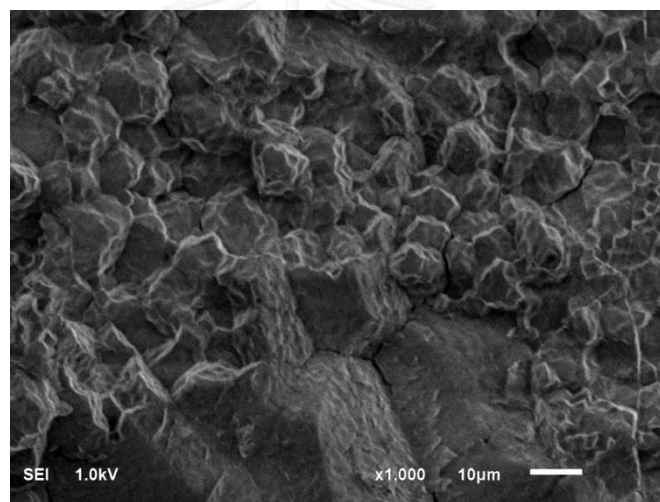
**Figure 2.1** The important parts of an un-milled rice grain [22]. Copyright 2016 Elsevier Ltd.

### 1. Morphology of rice grains

The rice grain locates inside its husk. The morphology of outer rice grain comprises of two parts, i.e., dorsal and ventral side. A thin film layer (1-2% of un-milled rice) coated on the grain, called pericarp, act as the husk-attachment layer. The other compositions of rice grain (4-6%) contain the combination of aleurone, nucellus, and seed coat layers. About 90% of rice grain is endosperm while at least 3% of embryo (germ) lies on the ventral side of a grain [23]. In general, the weight of a grain is about 12-44 mg while the size (such as length, width, and thickness) depends on their varieties [22].

The endosperm of rice grain consists of numerous starch granules which were produced by amyloplasts. Several researches reported that the morphology of starch granules is the polygonal shape [15, 25-28]. The close-packing structure appears in starch granule of rice with a diameter of 2-15  $\mu\text{m}$  (Fig. 2.2) which is the smallest of all cereal grains [26, 29].

Starch granule consists of two types of polymer chains: amylose and amylopectin. The physical structure of amylopectin chains entirely is crystalline, while amorphous is the chain arrangement of amylose. Normally, the amylose content of starch granules were classified into three types: waxy or low amylose (less than 20%), intermediate amylose (20%-25%), and high amylose (higher than 25%) [4, 30]. In the waxy rice grains, the amylopectin is entirely contained in the starch granule. In the case of non-waxy (normal) varieties, the amylose is highly contained in the starchy endosperm. Furthermore, the sugars, fats, crude fiber, and inorganic materials are also contained in the starch granule [23].



**Figure 2.2** SEM images of starch granules in a rice grain showing the polygonal shape with closed packing structure. The granule size is ~ 2-15 µm [31].

## 2. Morphology of rice husks

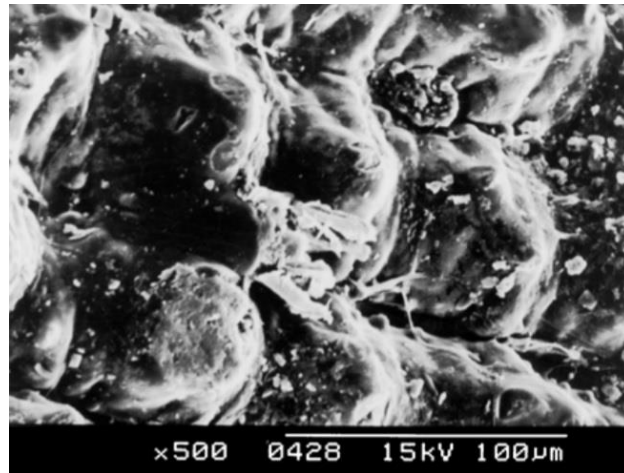
Rice husk is the outermost part of the un-milled rice grain that functions as the tight enclosure of the grain (Fig. 2.3). The un-milled rice grain contains the proportion of husk about 20% by weight. The chemical components of rice husk are reported as fibrous materials which are mainly composed of silica and organic materials. According to previous studies, the chemical compositions of dry rice husk from Western World

and Asia were evaluated as 20% of ash, 22% of lignin, 38% of cellulose, 18% of pentosans, and 2% of other organic matters. The ash of rice husk initially decomposes at over 1600 °C because of its high silica content. Silica within rice husk is in the form of hydrated amorphous portion while the husk on burning is obtained from ~95% of ash with the crystalline form [32, 33]. Fig. 2.4 shows the texture of the husk, which the high silica content present on protuberances and hairs (trichomes) in the outer region. In addition, the white materials that disperse on the epidermis of rice husk were also identified as silica. According to the silica accumulation above the epidermis, what were called “silicified cells” [34, 35]. On the other hand, the inner region mostly concentrates cellulose and lignin. The beginning of the decomposition of organic materials is in the range of 280-350 °C. Therefore, the temperatures of 200 °C do not decompose the husk. The heat just separates lemma-palea interlocking [33, 35].



**Figure 2.3** SEM image of rice husk showing the outer (rough) and inner (smooth) texture with the separated portion of lemma-palea interlocking [33].

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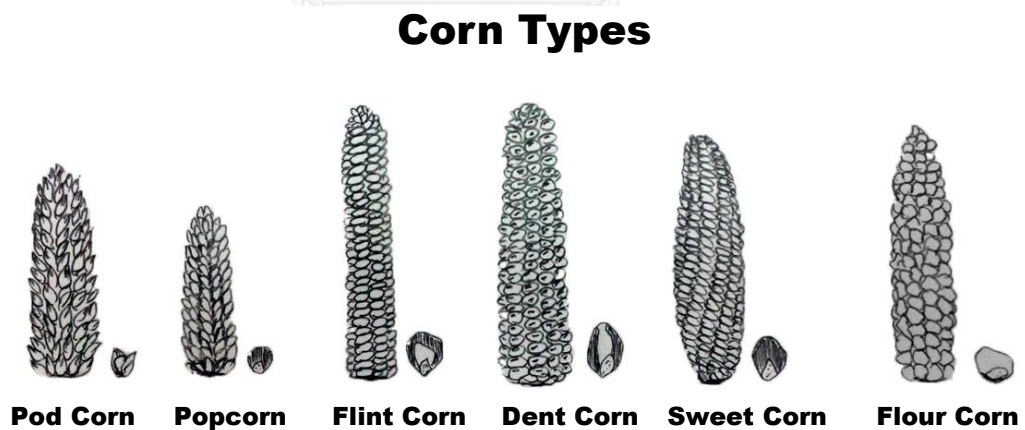
**Figure 2.4** SEM image of the outer texture of rice husk showing the protuberances and hairs (trichomes) region. These positions mostly contain silica. The accumulation of white materials on the epidermis was identified as silicified cells [33]. Copyright 2003 Kluwer Academic Publishers.

### 2.1.2 Corn

People have first known corn as a wild grasses. The story of corn has apparently originated in Mexico and America. The oldest progenitor of corn was explored for long time ago at least 8000 B.C., which are derived from native Mexicans and Central Americans. Mayas, the native people from Mesoamerican civilization, cultivated some corn varieties and used corn for making staple food. It overspread to Canada and Argentina. In 1930s, corn has been cultivated on 70-80 million acres in the U.S. Corn is developed into large scale of highly economic production especially in U.S. [4, 36-38].

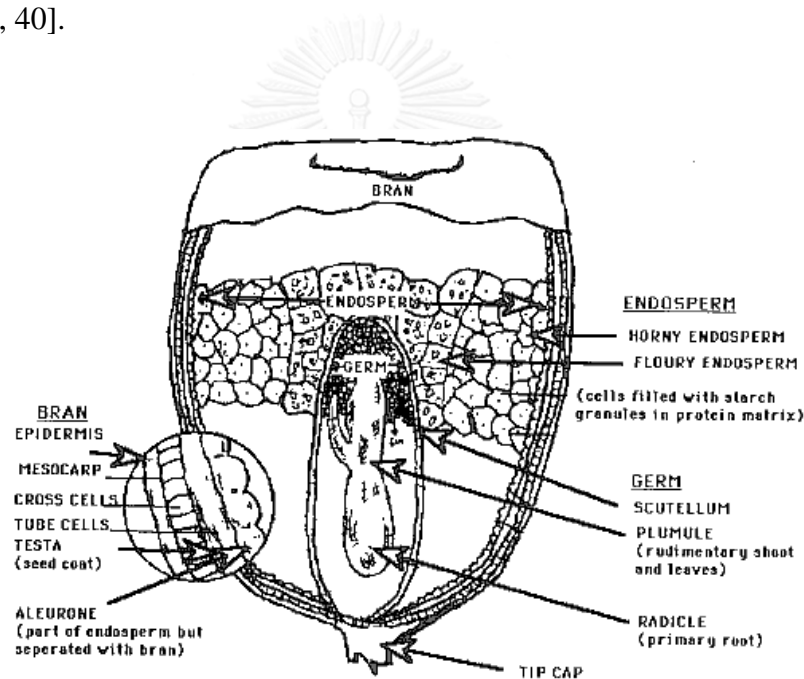
The ancient corn was reported as the original corn, called “grandfather corn” or “pod corn”. The morphology of pod corn is different from other types as each kernel of them is covered by an outer small hull. Although pod corn is a progenitor of corn, however, it is not used as commercial cereal product. In the United States, all corn varieties were classified into five main types, namely popcorn, flint corn, dent corn, sweet corn, and flour corn. Their classified corn varieties were divided by their

morphological characters of kernel and starchy proportion in the endosperm, i.e., horny (hard) starch and soft starch. Each type of corn varieties is used in several ways. First of all, popcorn, the endosperm within a kernel contains high percentage of horny starch while soft starch was least filled near the center of kernel. People mostly heat popcorn into popped corn, which is one of snack foods. Popped corn industry is a large scale of commercial product in the world making from raw materials of popcorn. Secondly, the horny starch covers mainly the endosperm of flint corn with a small portion of soft starch above a germ. Flint corn is used in foods in the form of hominy corn. Next, dent corn contains horny starch on both sides of a haft-kernel and soft starch throughout the top. Domestic animals are usually fed by yellow dent corn. In the case of sweet corn, the entire kernel is horny starch. This type accumulates high sugar content which is produced for cooking the desserts. Finally, in flour corn or soft corn, the soft starch is fully confined in the endosperm of kernel as making the finely powdered flour (Fig. 2.5) [8, 36-38].



**Figure 2.5** Corn types classified from the morphologies into six different types: pod corn or grandfather corn, popcorn, flint corn, dent corn, sweet corn, and soft or flour corn.

Corn (*Zea mays* L.), also known as maize, is one of the oldest cultivated cereal grains. The scientific name of corn was derived from the genus “*Zea*” while its specific name is “*mays*” in biological taxonomy [38]. One fruit consists of a shank and silk at its end. This is covered by the husk leaves that show many corn kernels. The important parts of corn kernel consist of four major components [Fig. 2.6]. A pericarp or hull is the outer part of corn kernel appearing as a single sheet. It functions as an entirely covered film of kernel, which protects the inner parts, i.e., the endosperm and the germ (embryo) what attaching with tip cap. Normally, a part of tip cap closely attaches with its core [39, 40].



**Figure 2.6** Important part of corn kernel [41]. Copyright 2015 Food and Agriculture Organization Publishers.

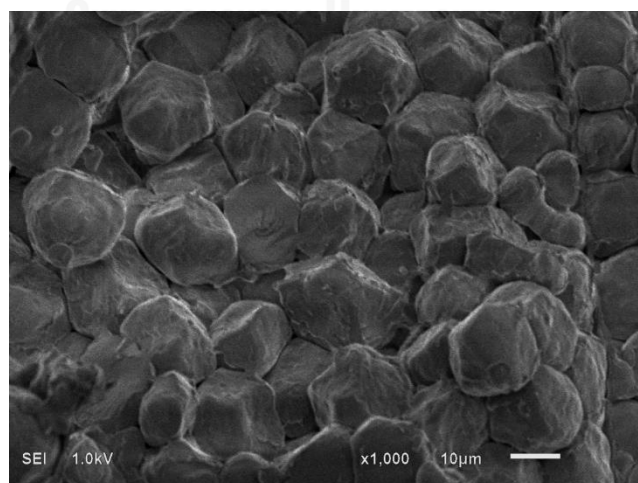
### 1. Morphology of corn kernels

The relative amounts of main components in a corn kernel depend on the genotypes and cultivating environments of corn. Some layers of corn kernel are similar to that of rice grain, but the starch position within corn kernel is contrast distinctively

as relate to corn types. In general, the dry weight of kernel constitutes about 80-85% of endosperm and 8-10% of germ (embryo) [38, 39].

Relating to rice grain, the whole endosperm of a corn kernel consists of the carbohydrates, which are amylose and amylopectin, for ~80%. Especially, the starch within the endosperm is classified into the horny or hard starch as polygonal shape with the closed packing and soft or flour starch as quite spherical shape with the loose packing. The distorted structures of starch were formed by starch synthesis and the influence of protein bodies which press on top of the starch granule. According to the dispersion of numerous starch granules within the kernel, the SEM images reveal that the normal size of starch granules is approximately 6-30  $\mu\text{m}$  (Fig. 2.7) [40, 42, 43].

In the case of starchy endosperm, corn kernels were divided into two major types that referred to waxy and normal corn. The amount of amylopectin through 100% was called “waxy corn” while the increasing of amylose content more than about 25% referred to normal corn. Each characteristic of starch is applied into the different industrial products. Other components within corn kernels were described to consist of protein (8-10%) and oil (4-5%). [4, 36, 38, 44, 45].



**Figure 2.7** A SEM image of starch granules in a corn kernel showing the polygonal shape with closed packing structure. The granule size is 2-15  $\mu\text{m}$ . [31]

## **2. Morphology of corn hull**

Corn hull perform as a protecting part of endosperm and embryo within a kernel. The structure of corn hull is a brittle, thin film converged on a tip. Regarding to their chemical composition, the thin layer of corn hull consists of 70% of cellulose, 2-14% of lignin, 10-18% of extractives, and the low content of ash. According to the high content of cellulose within the hull, the thermal stability of hull cannot endure when it contacts the heating instrument at above 187 °C [46]. That heating point makes the ripped cellulose and creates an evaporating superheated-water spot of moisture content within the starch granule. The major weight loss occurred at about 280-340 °C as continuously weight loss goes up to 340-450 °C. [47, 48].

## **3. Popped product of corn (Popped corn)**

Popcorn is one of the five types in the classification of corn varieties. Although popcorn has a small kernel, compared with the other types of corn, it has high expanding ability due to its endosperm contains high horny starch content. There are three methods for popping popcorn (i.e., heated air, heated oil, and microwave). As the corn kernels are heated by hot air for 40-60 s, their starch granule will be exploded due to the expanding of superheated water within the granule. The kernel can highly expand into a sponge-like structure which was commonly known as “popped corn” [15, 16, 19]. Popped corn is one of the most important foods for surviving of world population. People consume popped corn in the nutritious snacks for hundreds years. Nowadays, popped corn consumption continuously spread worldwide with the economic value estimated to be about 43 billion dollars in the U.S. snack food industry in 1998 [4]. The special harvesting and suitable storage are an importance in the popping quality. Popping volume, which relate to the highly horny starch content, is effect on the economic value of popped corn product. In general, the commercial products of popped corn are white and yellow pop corns with butterfly and mushroom shapes of popped



corn. A butterfly shape shows many wings-like structure which was produced from freedom bursting at all directions of the corn hull. In this research, the butterfly shape is only one type for systematic investigation [4, 13, 36, 45].

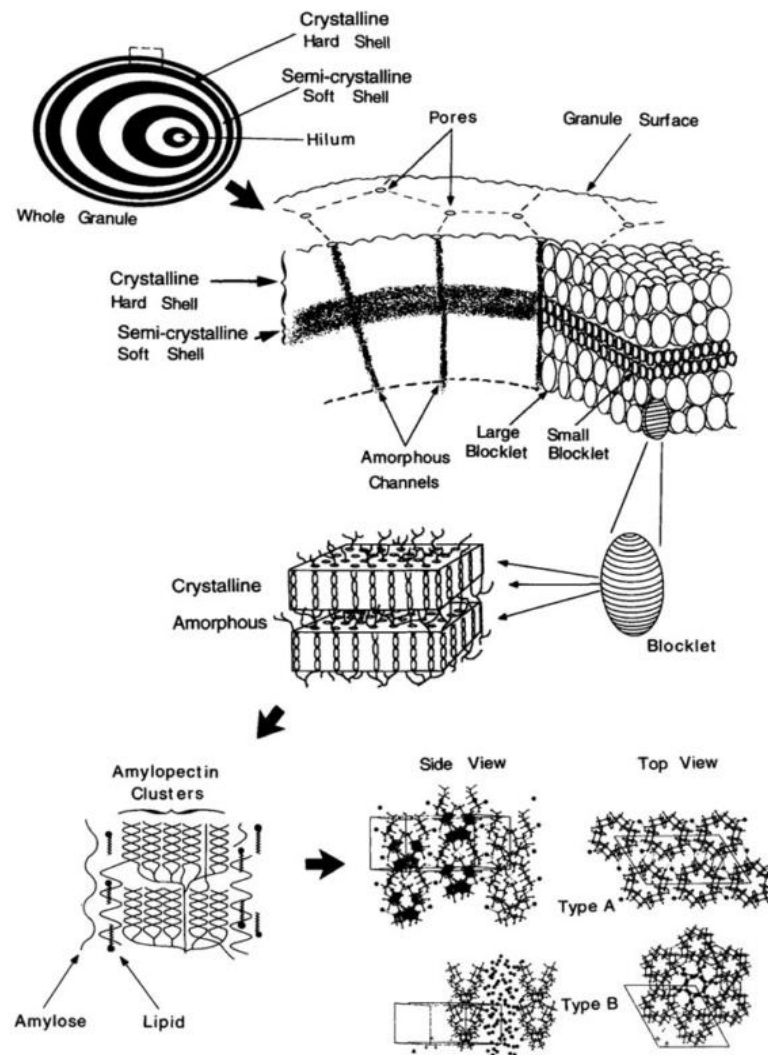
### 2.1.3 Starch granule

The endosperm within amyloplasts generally includes numerous polygonal-shape starch granules. Those starch granules are entirely contained in a homopolymer of glucose in the form of amylose and amylopectin. Typically, the starch granule contains amylose:amylopectin in the ratio of 1:3 with  $10^9$  amylose and  $10^7$  amylopectin molecules [38]. Fig. 2.8 shows the arrangement of blocklet within a granule which contain both of crystalline and amorphous lamellae. The large blocklets refer to the crystalline shells while the small blocklets involve an amorphous lamella that combine with crystalline region. The degree of crystallinity or amorphous form depends on the ratio of amylose and amylopectin.

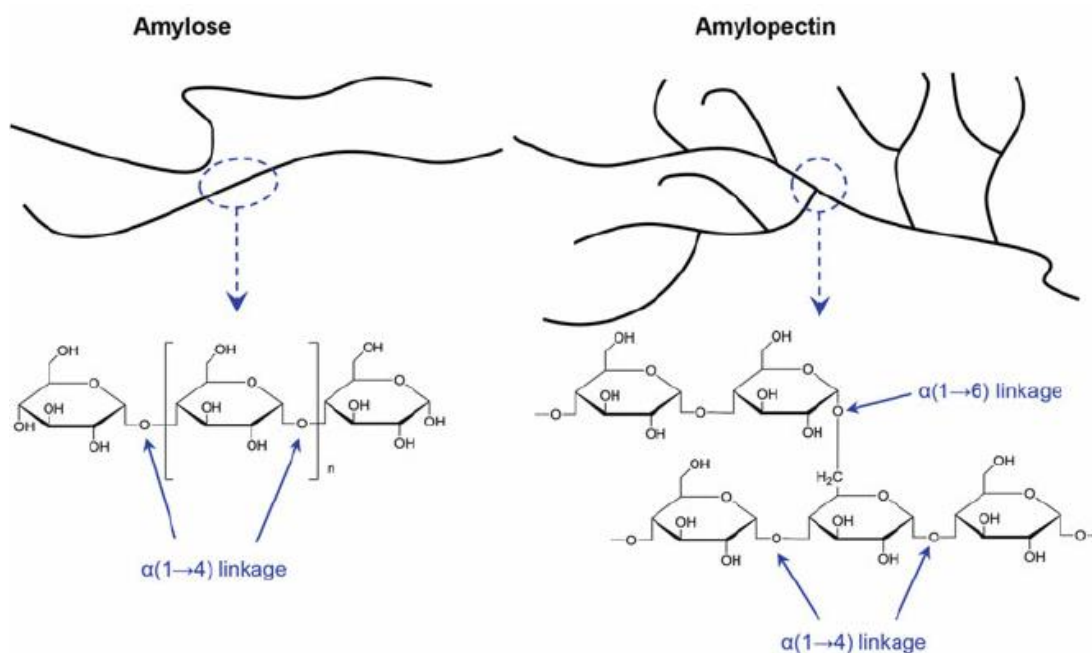
Amylose consists of  $\alpha$ -1,4-glycosidic linkage showing the linear molecules without branched chains (Fig. 2.9). Because of much linear chain in amylose, the starch granule still appears amorphous region and crystalline region when amylose co-arrangement with amylopectin molecules. The amylose fraction can form complex structure with iodine. The amylose-iodine complex was occurred from the iodide ion within the gap segments of the helix in amylose chain. This colorimetric method was commonly used to characterize the amylose content in the starch with the appearing of the blue color.

Amylopectin is the major component which found in the starch granule of waxy rice and popcorn. The chemical structure of amylopectin is  $\alpha$ -1,4-glycosidic linkage with high branching of  $\alpha$ -1,6-bonds forming into the macromolecule (Fig. 2.9). According to the chemical structure of amylopectin, the parts of double helices in linear chains constitute the crystalline areas while branch points of amylopectin were reported

in amorphous starch. The ratio of both components affect the structural and physicochemical properties of rice starches and corn starches [36, 38, 44, 49, 50].



**Figure 2.8** Diagram of starch granule structure showing the arrangements of blocklets within a granule and their starch component (amylose and amylopectin). The arrangements of amylose and amylopectin chains induce the crystallinity and amorphous form of starch [51]. Copyright 2014 Springer International Publishing Switzerland.



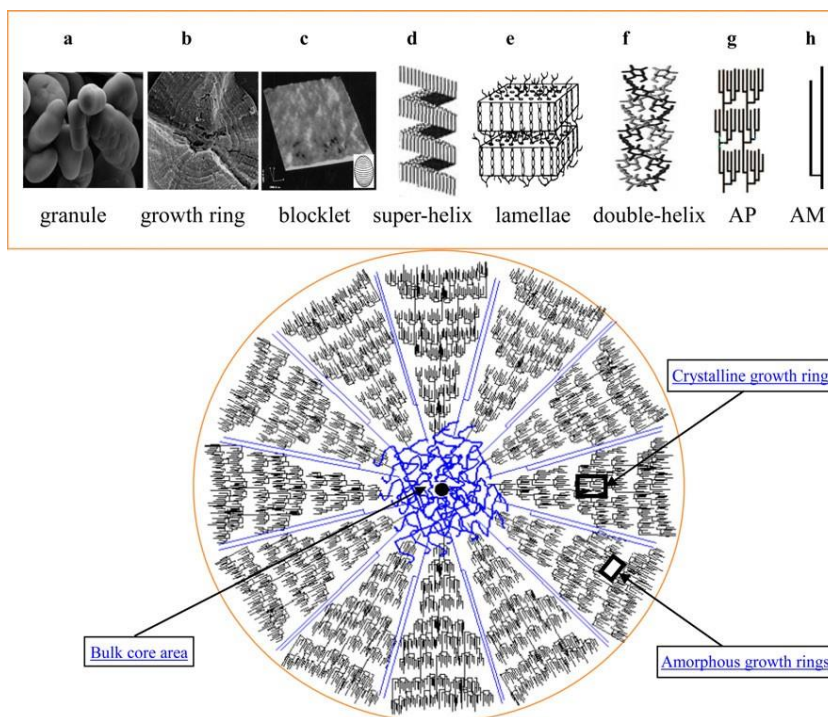
**Figure 2.9** Chemical structures of amylose and amylopectin molecules of starch. The structures include a homopolymer of glucose in form of  $\alpha$ -1,4-glycosidic linkage showing the linear chains in amylose and main area of amylopectin while the branched point of amylopectin were formed from  $\alpha$ -1,6-glycosidic linkage [51]. Copyright 2014 Springer International Publishing Switzerland.

จุฬาลงกรณ์มหาวิทยาลัย  
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## 2.2 Gelatinization and retrogradation processes of starch

In the starch granule, the closed packing structure is semi-crystalline areas where composed of crystalline and amorphous regions. Their regions were created by a copolymer of linear chain from amylose and partial amylopectin and branched chain from amylopectin forming in the growth rings of the hierarchical structure (Fig 2.10). From the atomic force microscopy (AFM) analysis in rice starch, the size of growth rings is approximately 400 nm which contain the alternating crystalline and amorphous region forming into semi-crystalline growth rings. Amorphous regions are disordered from amylose and the branched chain of amylopectin, while the crystalline parts are

linear chain of amylopectin as involving in the gelatinization process of starch when they were heated [44].



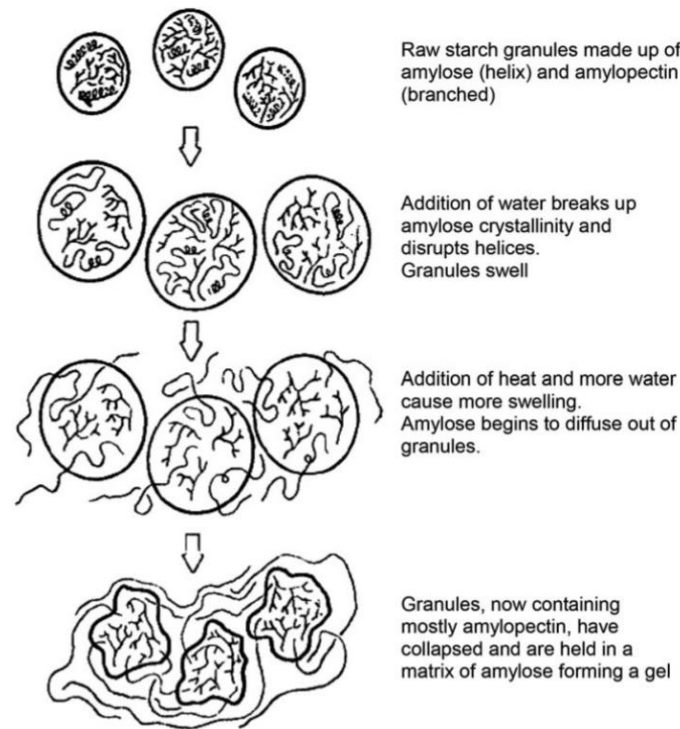
**Figure 2.10** Schematic representation of the hierarchical structure of starch granules. SEM image of (a) native starch granules of pea, (b) growth rings, (c) AFM image of blocklet structures. Drawing representations of (d) super helix, (e) lamellar, (f) structure of double-helix, (g) amylopectin, and (h) amylose molecules. The diagram represents the long chain of amylose molecule (blue line) and the arrangement of high branched amylopectin (black line) forming into growth ring [44]. Copyright 2015 John Wiley & Sons, Inc.

### 2.2.1 Starch gelatinization

The gelatinization (molecular disordered process) of starch was occurred by heating of native starch. The gelatinized temperature depends on the starch components and physiochemical properties of starch granules which relative to the genotypes of

starch. When the native starches (i.e., rice and corn) are heated with the presence of water. The amorphous region will be transformed in a 2-step process. First, water in the system breaks down the hydrogen bonds in the starch granule and induce the starch granule into swelling. Next, remained water acts as a plasticizer in the hydration and swelling process. The parts of crystalline structure, the process of gelatinization will be differently occurred under the water concentration in the system. In the high concentration of water like the cook rice process, the moisture within the starch granules is boiled making the swelling starch granule. Amylose chains are leaked out by swelling at low heating temperature. After that, the amylopectin is condensed in the granule matrix of gel-formed amylose, as shown in Fig 2.11.

In this research, we do not add the water in the system. The only water in the granule (i.e., rice grain, corn kernel) was focus. Then, the difficult performance to destroy the strong crystallinity at a limited water concentration. The starch will not be completely gelatinized by heat at the same temperature range. Therefore, the heating temperature should be increased for more movement of starch molecules and destroy the crystalline areas. At the low water content with high temperature heating, the starch granule will be melt, which is called “melting” of starch with observation via several techniques such as texture profile analysis, NMR, XRD, DSC, Raman and FT-IR spectroscopy. However, DSC technique is suitable for characterization of gelatinized starch. DSC thermogram reported the enthalpy, gelatinization onset ( $T_o$ ), peak ( $T_p$ ), and conclusion ( $T_c$ ) temperatures. Especially, the peak temperature ( $T_p$ ) and enthalpy refer to crystallinity of gelatinized starch. As the results, the semi-crystalline structure is damaged into disordered proportions in starch with an appearance of birefringence loss under polarized light microscope [44, 50-52].

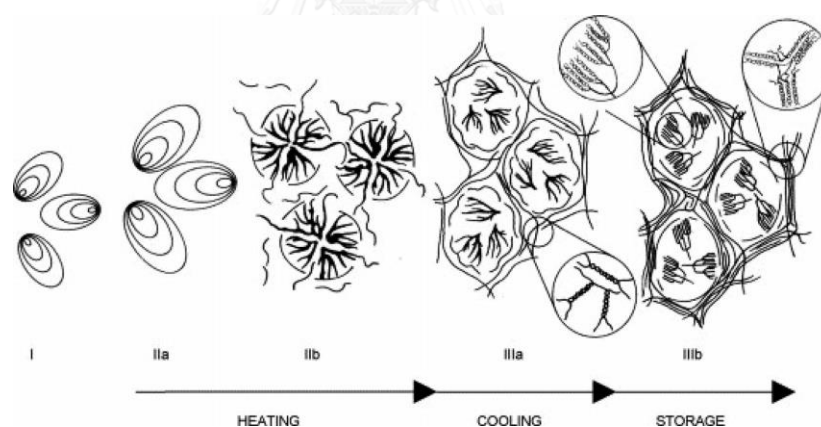


**Figure 2.11** Shows the mechanism of starch gelatinization. The native starch contains the rigid amylose and amylopectin molecules. After heating, the moisture within a starch granule continuously boils and the heat disrupts the hydrogen bonds of starch granule. Amylose starts to diffuse out from granule and transform into a gel, while amylopectin will be collapsed within a granule with there is held by gelatinous amylose [51]. Copyright 2014 Springer International Publishing Switzerland

### 2.2.2 Starch retrogradation

After the termination of gelatinization process, the temperature of gelatinized starch starts to cool down. At this moment, some parts of water in the copolymer structure are loose from evaporated steam within the gelatinized starch, reproducing the hydrogen bond between molecules. The hydrogen bond reformation will induce the aggregation of double helices in the polymer chains of amylose and amylopectin into different rearrangements before appearance of the partially recrystallized starch. The crystallinity of recrystallization process is less than a native starch, which is known

basically as “retrogradation”. The retrograded starch is not flexible texture with gradual changing to the firmness starch as represented in Fig. 2.12. The obvious instance is stayed cooked-rice at refrigerated condition making the rice uneatable. The rearrangement of amylose and amylopectin chains mainly concern to retrogradation process of their starch. The amylose molecules transform into inelasticity at short-time retrogradation (over minutes to hours). In contrast, amylopectin retrogradation slowly arise which long time more than that of amylose (over hours to days). The retrograded process depends on the crystalline content of amylopectin with loose polymer chain. Basically, the retrogradation of storage gelatinized starch refers to the amylopectin recrystallization. The retrogradation process of starch is characterized by DSC thermogram. The enthalpy change and retrograded temperature were reported a parabolic shape depending on the water content within the starch gel [44, 50, 52].



**Figure 2.12** Schematic representation of the native starch transformation during heating, cooling, and storage. (I) Native starch granules, (II) the gelatinization process during heating in form of [a] the swelling starch, [b] the occurring of amylose diffuse out of granule, (III) the retrogradation process shows [a] formation of an amylose network out of starch during cooling process and [b] formation of amylopectin rearrangement into crystalline structure during storage [44]. Copyright 2015 John Wiley & Sons, Inc.

### 2.3 Volume expansion of the moisture in corn under heating

The popping process was occurred after heating of corn kernel. The moisture, in form of liquid water within the starch granule, evaporates and traps within corn hull which function as the pressure vessel. The water vapor and the liquid water are contained in equilibrium system in starch granule. A continuous heating transforms the rigid starch granule into softly gelatinized starch which simultaneously generate the vapor pressure until the water will be superheated [15, 16]. As the increasing of temperature, the vessel cannot endure the high accumulated pressure. A catastrophic pressure rapidly ruptures the vessels and turned the superheated water into steam with a released characteristic 'pop' sound. This is basically called "popping process". Water within the starch granule is in the equilibrium between liquid and vapor at only one component with two phase of water. In this study, the temperature is only one value which was directly measured from the experiment for a systemic definition. A temperature and a pressure are interrelated each equilibrium as that moment gives only one value of temperature and pressure. Therefore, a pressure at popping temperature in equilibrium is obtained from the saturated steam table of the closed system (available in Physical handbook). The pressure values as shown in the table were derived from the temperature values where water and its steam are in equilibrium. As a result, the temperature and pressure are two parameters used in the calculation of water that is converted to vapor in the popping process. Based on the moisture in equilibrium within starch granule which were assumed in the close system, an ideal gas equation ( $PV=nRT$ ) was used for the calculation the mole of water. The actual pressure, which makes the corn kernel ruptures, is a steam pressure without the atmospheric pressure (14.7 psi). Previous researches reported the popping temperature of popped corn at about 170-190 °C, generated the high pressure at 110-120 psi for rupturing of corn hull. The pressure ruptures the corn hull and creates the driving force for the expansion of starch granule into a popped corn [6, 15-17]. Hosney et al. reported their corn, which



contained 14% moisture content, popped at about 177 °C with 120.3 psi of popping pressure. The mass of water involved in the popping process of corn kernel was calculated by ideal gas equation. Only small value of water within the granule would be superheated and created a pressure at the popping moment [15]. The water still retains as liquid at about 4.5% within the kernel after the popping process. As the popping of popped corn study in 1991, the mass of water involved in the popping process was calculated from ideal gas equation. These water were reported only 2.3% converting water into steam [16]. After popping of popcorn, a kernel will be expanded into a spongy-like structure of starch called “popped corn”.



## CHAPTER III

### EXPERIMENTAL SECTION

#### 3.1 Materials and Chemicals

1. A local waxy rice variety, called ‘Laow-taek’ (*Oryza sativa* L.) obtained from Yasothon province, Thailand
2. A popcorn variety (*Zea mays* L.) (McGarrett, American popcorn)
3. Water soluble yellow dye (Printing Ink-Yellow from EPSON)
4. Red organic dye (Firefly colored lamp oil Dye-Red from Andersen Academy Miami)

#### 3.2 Instruments and Equipment

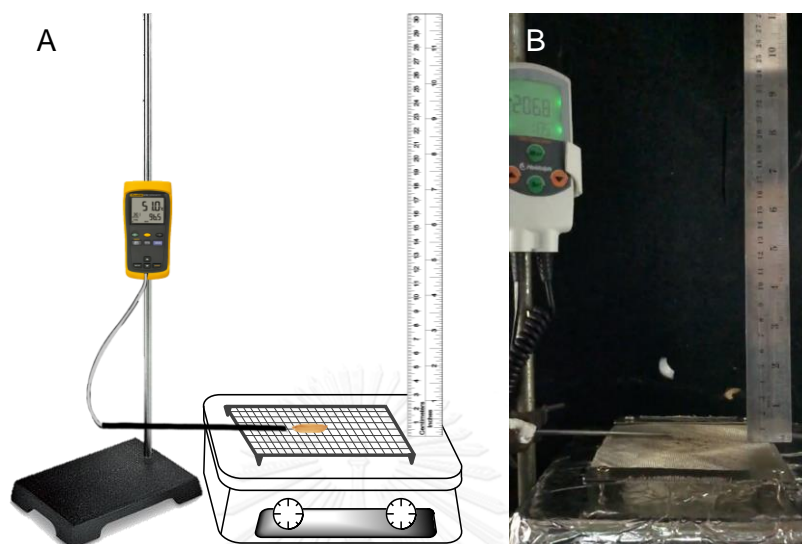
1. iPhone 6s Plus with SLO-MO app
2. Nikon D90 digital camera, with Macro lenses
3. Thermocouple, Heidolph with EKT Hei-Con temperature sensor
4. Optical microscope (OM), Carl Zeiss Axio Scope. A1 with a CCD camera (Carl Zeiss, AxioCam HRc)
5. Scanning Electron Microscope (SEM), JEOL JSM-6510A
6. Thermal gravitation analysis (TGA), Perkin Elmer, Pyris 1TGA
7. Differential scanning calorimeter (DSC), *Mettler Toledo* DSC1
8. Fourier-transform infrared (FT-IR) spectrometer, Nicolet 6700
9. Image processing software (ImageJ 1.43r, Rasband, W. National institutes of health, USA)
10. Glass beads with diameter of 500  $\mu\text{m}$  (Obtained from Blast Master company, <http://www.thaisandblast.com>)
11. Steel ball with diameter of 0.95 cm (volume of 0.14  $\text{cm}^3$ )

12. Burette (50 mL)
13. Stainless steel sieve (400 mesh)
14. Stand and clamp
15. Ruler (100 cm)
16. Glass cover
17. Razor blade
18. Needle (diameter of 0.6 mm)
19. Petri dish
20. Dropper

### 3.3 Popping Process

The jumping trajectory of the grain (rice grain or corn kernel) were recorded in a slow-motion by the SLO-MO app installed on iPhone 6s Plus. The frame rate was set at 240 frames per second (fps). Under this frame rate, two consecutive images are separated by 4.2 ms. The slow-motion videos of the popping process were recorded under the setup shown in Fig. 3.1. The smartphone was placed at 30 cm above the hotplate. To avoid a rice grain/corn kernel burning due to an overheating by a direct contact with the hotplate and to facilitate an accurate temperature measurement of a thermocouple, a rice grain/corn kernel was placed on a square stainless steel sieve (400 mesh). The sieve was placed ~5 mm above the hotplate via four stainless steel screws at each corner. The thermocouple was positioned in the vicinity of the rice grain/corn kernel without touching the sieve. A 100-cm stainless steel ruler was positioned beside the rice grain/corn kernel by a stand and clamp. We used a matt dark background with a right angle spot-light illumination to enhance the image contrast as the popped rice traveled with a relatively high speed. The hotplate was set at the maximum temperature of limitation of device. The popping process was completed within 40–60 s. We retrieved an individual frame from the slow-motion video in order to get the time and

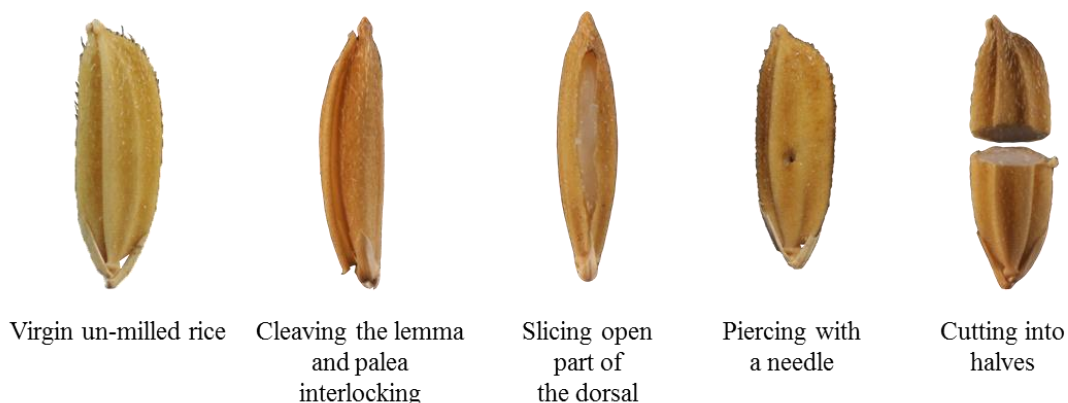
jumping height of the popped product. To obtain reliable statistical data, a set of measurement were repeated with 100 grains were employed in our study.



**Figure 3.1** (A) Experimental setup for recording the trajectory profile of popped rice and popped corn. A stainless steel ruler was used for measuring the jumping height of the popped grain. (B) An image extracted from a slow-motion VDO clip shows the jumping height of a rice grain (4.8 cm) and a rice husk (3.2 cm) at 42 ms after bursting.

### 3.4 Defects on the rice husk

The defects on un-milled rice grains were made on rice husks. These can be categorized into 4 types as shown in Fig. 3.2. For Type 1, the lemma and palea interlocking of rice husks were cleaved by razor blade at one side. In Type 2, the dorsal parts of un-milled rice grains were sliced open exposing the rice grain. For Type 3, a needle was used to pierce at the middle of the grains. For Type 4, the un-milled rice grain was cut into halves by razor blade. All types of the defected rice husks were photographed via Nikon D90 digital camera with a macro lens. Each defected rice grain was placed on a flat glass (petri dish) and heated by hot air until popping process was observed. A set of 5 grains in each type was employed in our study.



**Figure 3.2** The virgin rice grain with 4 types of defects: (1) cleaving the lemma and palea interlocking, (2) slicing dorsal into open part, (3) piercing with a needle, and (4) cutting into halves

### 3.5 Moisture content measurement of grains

The moisture contents (i.e., un-milled rice and corn kernel) were determined using a standard Association of Official Analytical Chemists (AOAC 925.10-1925) method [53-55]. The weight of grains (~2 g) was recorded before placing in air convection oven held at 130 °C for 1 hr. After baked for 1 hr, the grains were removed from the oven and cooled down to room temperature in a desiccator. Then, the dried grains were weighed again. The process of moisture content measurement was repeated several times until the weight did not change. The percentages of moisture contents were calculated from the mass losses of the grains.

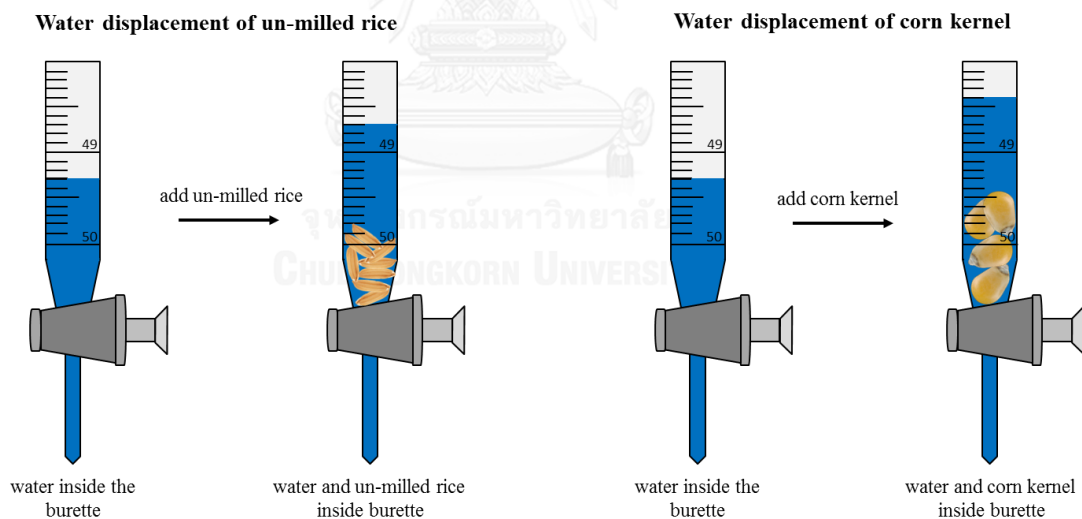
### 3.6 Moisture content measurement of popped products

For popped rice and popped corn, their moisture contents were determined using thermal gravimetric analysis (TGA, Perkin Elmer Model: pyris 1TGA). The process was carried out in the presence of nitrogen (N<sub>2</sub>). A specimen (1–10 mg) was placed into TGA pans. The samples were heated from 30.00 °C to 850.00 °C at the heating rate

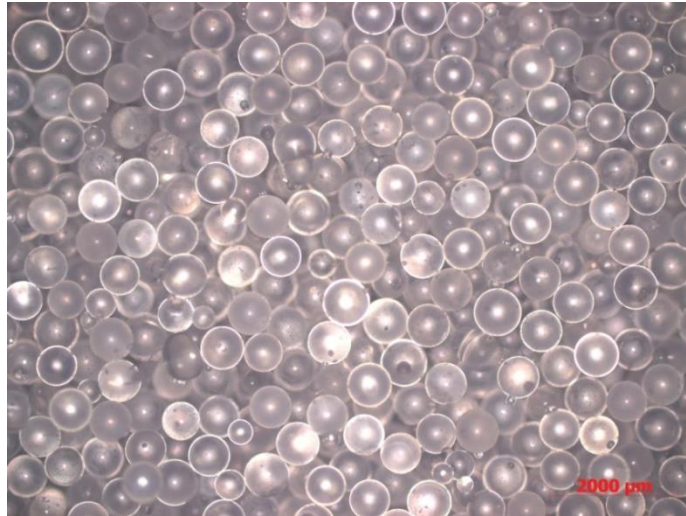
of 20.00 °C/min. The initial mass loss was shown at about 100 °C due to the evaporated moisture from the grains.

### 3.7 Volume measurements

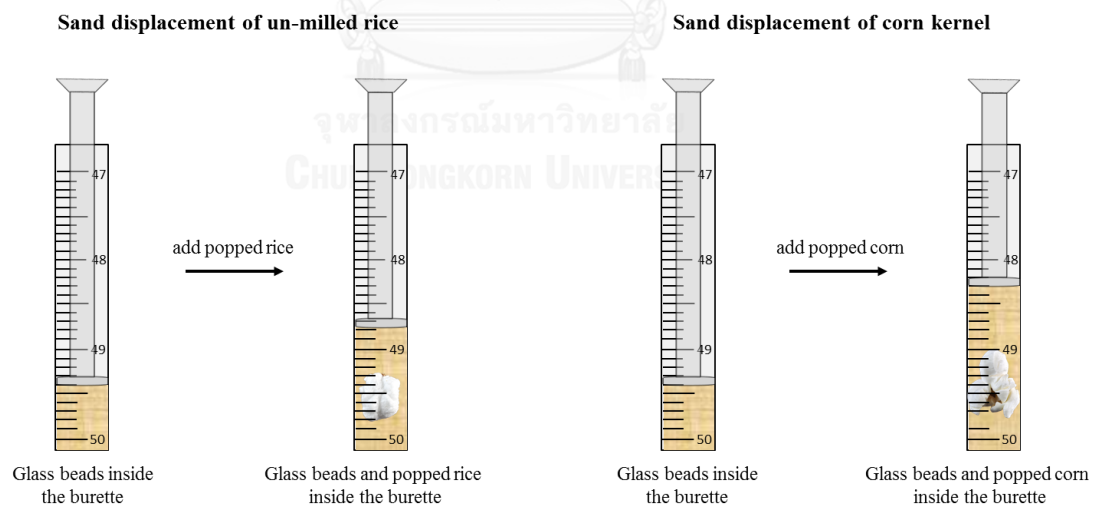
Volumes of rice grain and corn kernel were measured by water displacement method in burette [56, 57] (see Fig. 3.3). Briefly, a set 20 grains/kernels was employed in each sample. Water was filled in graduated burette. Then, rice grain or corn kernel sample was deposited into the burette. The volume of rice grain or corn kernel was measured as the increased volume. The volume of popped grain was measured by the sand displacement method in graduated burette using the glass bead (average particle size of 500  $\mu\text{m}$ , as shown in Fig. 3.4 [57, 58]). This method was calibrated with steel ball (diameter of 0.95 cm, volume of 0.14  $\text{cm}^3$ , as shown in Fig. 3.5).



**Figure 3.3** Schematic drawings of volume measurement of un-milled rice and corn kernel via water displacement using a burette.



**Figure 3.4** An optical microscope image of glass beads (Blast Master, <http://www.Thaisandblast.com>). The spherical glass beads have an average particle size of 500 μm. The glass beads were employed in sand displacement experiment for the measurement of volumes of popped rice and popped corn.



**Figure 3.5** Schematic drawings of volume measurement of popped rice and popped corn via sand displacement using a burette.

### **3.8 Grains/popped product morphology**

The morphology of grains/popped products were investigated using scanning electron microscope (SEM), JEOL JSM-6510 at an accelerating voltage of 1.0 kV. This technique was applied to investigate the three dimensional (3D) structure of popped products. The samples (i.e., rice grain, corn kernel, popped rice, and popped corn) were attached on aluminum stub by a conductive carbon tape before characterization. An imaging software (ImageJ 1.43r, Rasband, W. National institutes of health, USA) was used to measure the sizes of starch balloon and wall thicknesses of popped rice and popped corn.

### **3.9 Structural investigation**

#### **3.9.1 Investigation of the weakest spot of rice husks**

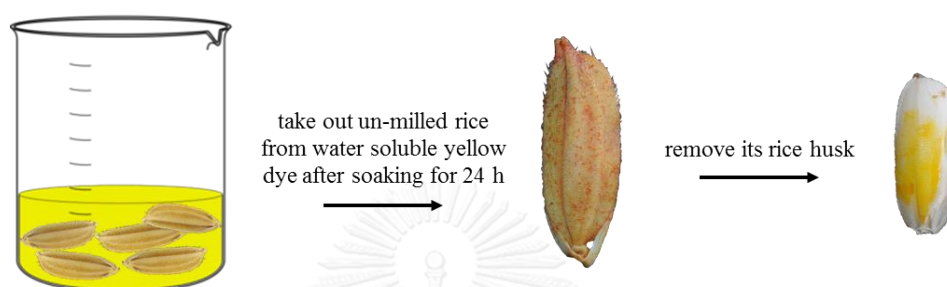
The dye adsorption method was used for investigated weakest spot of rice husks. After soaking un-milled rice grains in water soluble yellow dye for 24 hrs., grains were taken out from dye solution. The rice husk was removed, as shown in Fig. 3.6. These photos of rice grains were taken by Nikon D90 digital camera with Macro lenses

#### **3.9.2 Investigation of the open pore morphology of popped rice**

The open pore morphology of popped rice was characterized by the dye flow into the inner grain. Briefly, a grain of popped rice was employed in this study. In the first experiment, once the red organic dye was dropped on the top of popped rice, the dropping time was started. After dropping time of 1 min, the flowing process was recorded by Nikon D90 digital camera with Macro lenses. The recording of dropping time were followed every 1 min for 10 min, as shown in Fig. 3.7. In addition, the flowing distances of dye in the vertical direction were investigated to repeatedly confirm the existence of open pore in the structure of popped rice by cutting a cross section with a razor blade. A set of 10 grains of popped rice were used to study. For



more detailed investigation, the diffusion process of dye on popped rice was characterized by optical microscope. To monitor the diffusion rate, the dye was dropped on 10 popped rices. In every minute, a popped rice was cut to capture a cross section image. Therefore, the 10 popped rice was responded for 10 minute of the diffusion rate of dye.



**Figure 3.6** Schematic drawing of 5 un-milled rice grains after soaking in water soluble yellow dye for 24 hrs. and their husks removed.



**Figure 3.7** Schematic drawing of a drop of red organic dye on top of popped rice.

### 3.10 FT-IR measurement

FT-IR spectra of the grains and popped products (i.e., rice grain, corn kernel, popped rice, and popped corn) were recorded using a ATR FT-IR spectroscopy via a Nicolet 6700 FT-IR spectrometer. The samples were scanned in the spectral region of  $4000\text{ cm}^{-1}$  to  $750\text{ cm}^{-1}$ , representing the average of 64 scans. [59].

### 3.11 Thermal properties

Crystallinity of rice grain/corn kernel and their popped products were investigated by DSC measurements performed using a Mettler Toledo DSC1. The instrument calibration was done with indium standard. A sample (1–10 mg) was placed into a stainless steel pan which was later hermetically sealed. An empty pan was used as a reference. The samples were scanned from 30 to 250 °C with a heating rate of 10 °C/min [60].

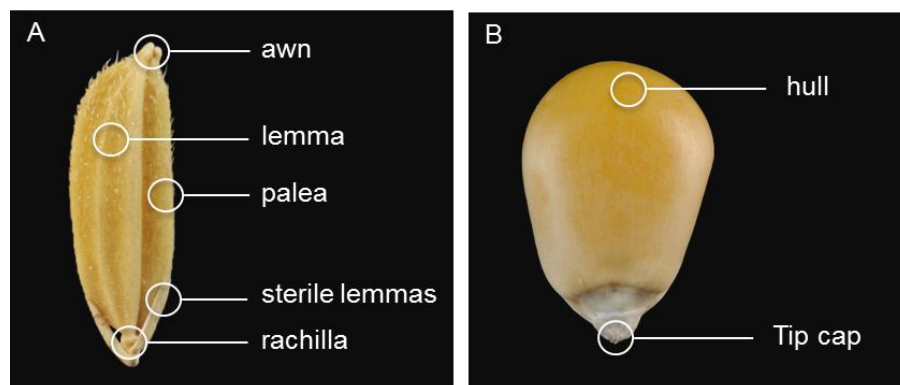


## CHAPTER IV

### RESULTS AND DISSCUSION

#### 4.1 The popping process

Popped rice has been a part of the Asian cultures for a long time and it relates to the way of their life. The previous studies of popped rice were reported less than 30 years ago [6]. On the contrary, a systematic study on the popping mechanism and the control of its popping behavior was not well studied as compared to those of popped corn [4, 15, 61, 62]. To gain an insight understanding on the popping mechanism of popped rice, we performed systematic studies of popped rice and popped corn. The popped grains were transformed from waxy rice and popcorn. External characteristics of un-milled rice grain compost of lemma, palea, awn, sterile lemmas, and rachilla which enclose the rice grain. The commercialized popcorn (McGarrett, American popcorn) was used for making popped corn. The main parts of corn consist of a single sheet of hull and tip cap which cover the starch within corn endosperm as shown in Fig 4.1



**Figure 4.1** The important parts of (A) un-milled rice and (B) corn kernel.

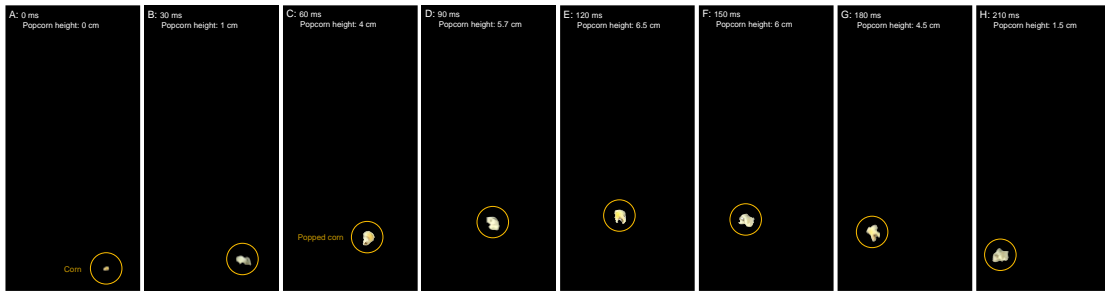
A single grain of rice or corn was hot-air heated as shown in the experimental setup (Fig. 3.1). The popping temperatures and jumping trajectories of the popped products were slow motion recorded with a smartphone (Apple iPhone 6s Plus) at 240 frame per second (fps). A total of 100 grains were experimentally conducted for statistical analyses.

For popped rice, after a hot-air heating for 40-60 s, the rice grain burst with an emitted characteristic 'pop' sound and suddenly jumped off in this heating stage [19]. Although the popping time, popping temperature, and the jumping trajectory cannot be accurately predicted, we managed to record a hundred of slow motion video clips of the bursts and the corresponding popped rice trajectories. A series of still images with 30 ms interval was extracted from the video (Fig. 4.2). The jumping heights of the popped rice and the husk were measured directly from the imbedded ruler. The ruler was positioned nearby the grain and during the recording process. This particular rice grain burst at 209.5 °C with a jumping height of popped rice greater than 100 cm. The burst was so violent that the popped rice and its husk were immediately separated. The violent burst of popped rice made the travel speed so high that the image became elongated and the jumping height was greater than the viewing area of the camera.

In the case of popped corn, the images in Fig. 4.3 were still images extracted from slow motion VDO clips which imbedded both of traveling distance and time. As a result, the popped corn and its hull were still attached after burst. We observed the normal characteristic 'pop' sound as the corn burst. In all cases, the corn kernels popped at relatively lower temperatures and jumped lower than those of popped rice. We have performed a hundred popping of single corn kernel while recording slow motion images.



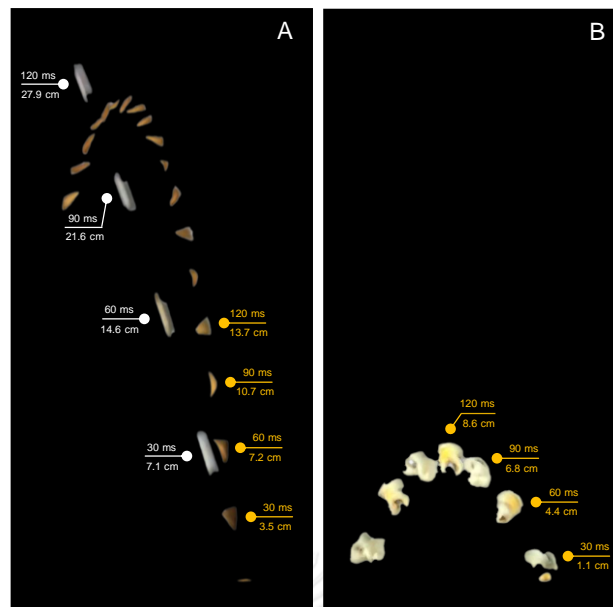
**Figure 4.2** A series of still images with 30 ms interval extracting from a slow-motion VDO clip shows trajectory of popped rice and its husk as the grain popped upon heating. This particular rice grain popped at 209.5 °C as indicated by the thermocouple. With Photoshop software, the positions of popped rice and its husk were overlaid on the same image.



**Figure 4.3** A series of still images with 30 ms interval extracting from a slow-motion VDO clip shows trajectory of popped corn as the kernel popped upon heating. This particular corn kernel popped at 189.7 °C. With Photoshop software, the position of popped corn was overlaid on the same image.

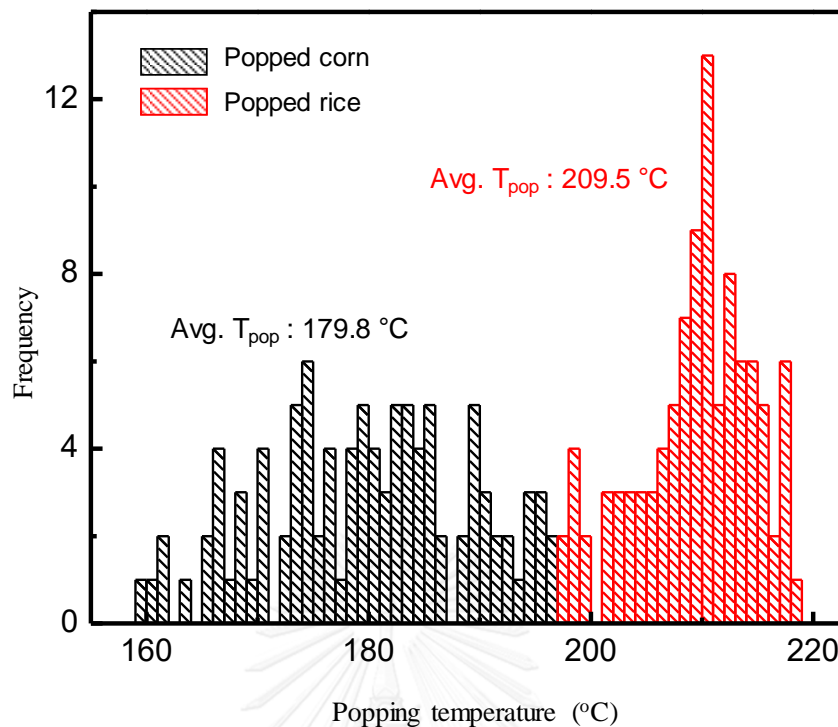
From a series of still images, the positions of popped rice and its husk were overlaid on the same image for a clear graphical representation by Photoshop software as shown in Fig. 4.4A. Based on the measured heights and times, the averaged travel speeds of popped rice and rice husk were  $2.43 \text{ ms}^{-1}$  and  $1.17 \text{ ms}^{-1}$ , respectively, in the first 30 ms and  $2.23 \text{ ms}^{-1}$  and  $1.27 \text{ ms}^{-1}$  in the second 30 ms. Later, the speeds were reduced due to air friction and gravity. The popped rice traveled out of the viewing frame after 120 ms with the height of the viewing frame was 28 cm.

Fig. 4.4B showing overlaid images displays the trajectory of popped corn which was reconstructed from still images in Fig. 4.3. For the particular kernel of popped corn (shown in Fig. 4.4B), the kernel burst at 179.8 °C with a maximum jumping height of 6.5 cm. The travel speeds of popped corn were  $0.33 \text{ ms}^{-1}$  and  $1.00 \text{ ms}^{-1}$  in the first and second 30 ms, respectively. Similar to that of popped rice, the speed became slower after 60 ms.



**Figure 4.4** Overlaid images display trajectories of (A) popped rice and (B) popped corn as they burst from the rice husk and corn hull, respectively. The images were reconstructed from still images extracted from slow motion VDO clips. To make a clear graphical representation, some times and traveling distance during the trajectory are indicated in the images. It should be noted that some of the popped rice jump higher than 100 cm while the maximum jump height of the popped corn was only 6.5 cm. The still images of popped rice are available in Fig. 4.2 while those of popped corn are available in Fig. 4.3.

We have performed the popping operation on 100 waxy rice grains and 100 corn kernels. However, those grains popped at different temperatures. Fig. 4.5 summarizes the distribution of popping temperatures of rice grains and corn kernels. The average popping of rice grains ( $209.5 \pm 9.23$  °C) was greater than that of corn kernels ( $179.8 \pm 5.19$  °C).



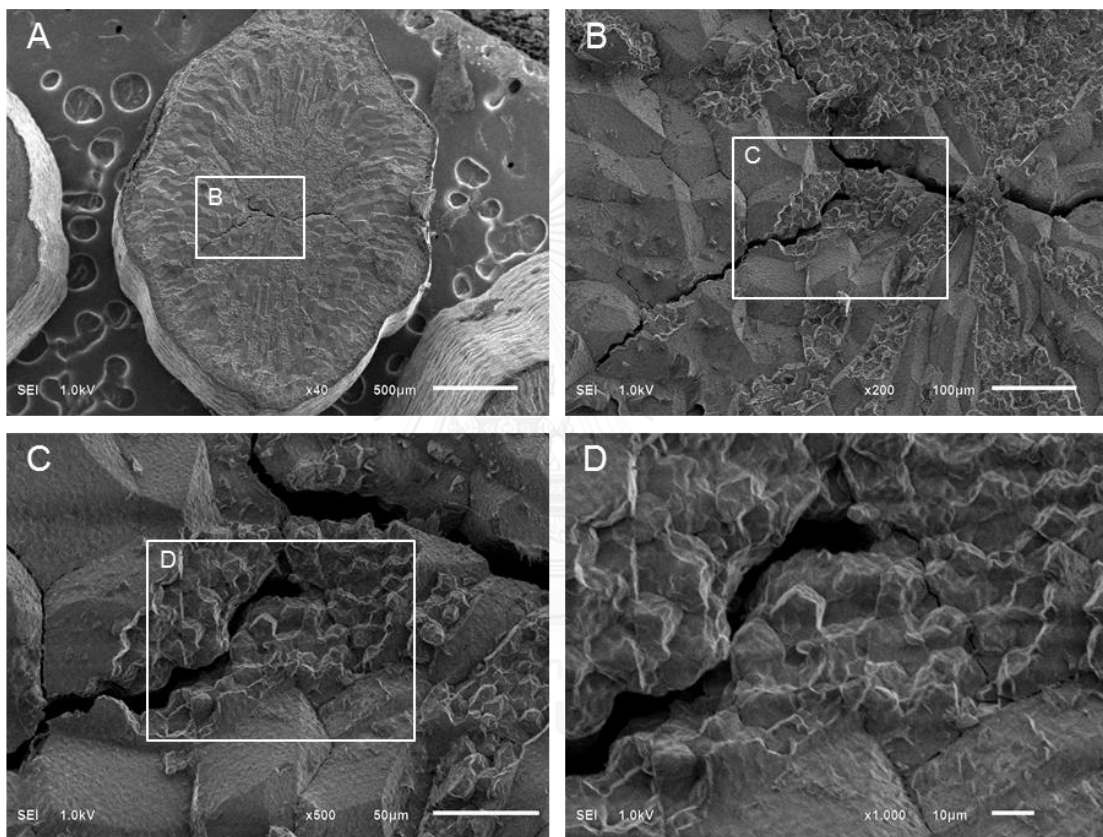
**Figure 4.5** Distributions of popping temperature of popped rice and popped corn. A set of 100 grains were analyzed. Based on the experimental results, the rice grain popped at a relatively higher temperature than the corn kernel.

The starch granule of waxy rice grain and popcorn kernel involved in the popping process in term of moisture expansion within the starch granule. The morphological details of their starch granules were recorded by SEM technique. For rice grain, the starch granules show the compact packing structure with polygonal shapes. The shapes are similar to the previous reports of rice starch characterization [6, 26]. A grain of waxy rice contains the numerous starch granules with diameter of 5-15  $\mu\text{m}$  (Fig. 4.6)

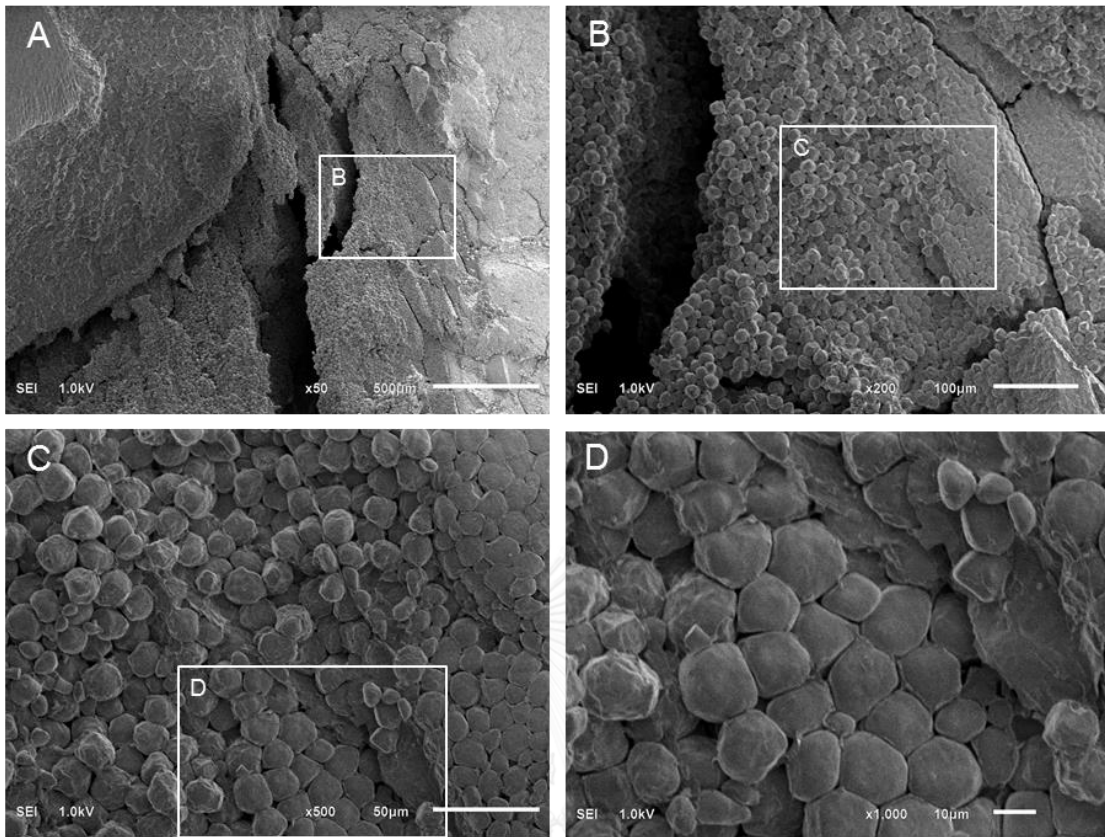
In case of popcorn kernel, although the arrangement of starch granules is similar to that of rice grain, the starch granule of corn is classified to the horny (hard) and soft (flour) starch [8, 37]. The shape of horny starch is polygonal structure while the soft starch is spherical shape. The horny starch is contained in the endosperm in form of



closed packing, but the loose packing is shown in the soft starch. From SEM images, the size of starch granule is approximately about 10-20  $\mu\text{m}$ , as shown in Fig. 4.7. Therefore, the size of native starch granule of rice grain is smaller than that of corn kernel; nevertheless, the packing of starch granule in rice grain is more compact than that of corn kernel.



**Figure 4.6** SEM images show morphology of starch granules in rice grain (5-15  $\mu\text{m}$ ) with difference magnifications at (A) 40X, (B) 200X, (C) 500X, and (D) 1000X.



**Figure 4.7** SEM images show morphology of starch granules in corn kernel (10-20  $\mu\text{m}$ ) with difference magnifications at (A) 50X, (B) 200X, (C) 500X, and (D) 1000X.

The popping process of un-milled rice grain and corn kernel was occurred upon the hot-air condition. When a grain was heated, parts of the liquid water inside the starch granules evaporated and trapped within the vessels. The rice husk and corn hull functioned as the pressure vessels trapping the evaporated steam [15]. A continuous heating increased vapor pressure in equilibrium with the liquid water in starch granules. A pressure buildup at high temperature turned liquid water into a superheated water capable of gelatinizing the hard and rigid starch granules [15, 16]. As the temperature was continuously increased, a catastrophic pressure ruptured the vessels (i.e., rice husk or corn hull) and turned the superheated water into steam with a released characteristic ‘pop’ sound. As the steam expanded more than a thousand times of its original volume

as liquid water, the rapid expansion exploded the microscopic starch granules into interconnected micro-balloons with micrometer-thin starch wall. As a result, the starch granules transformed into sponge-like structures with interconnected micro-balloons upon popping. The malleable sponge was rapidly solidified and dried with sufficient cooling by the vaporization of the superheated steam.

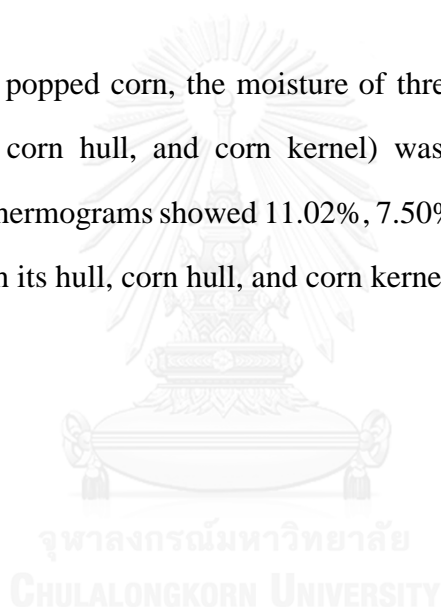
In the case of popped rice, as the husk failed at 209.5 °C (the average temperature), the superheated water expanded 1427 times its original volume as liquid water at 100 °C. A grain of waxy rice containing starch granules with diameter of 5-15  $\mu\text{m}$  (Fig. 4.6) was transformed into a sponge-like structure with 21 times volume increment, Table 4.1. Based on the ideal gas equation of state ( $PV=nRT$ ) and the steam-water equilibrium from the saturated steam table, the pressure that caused the rice husk to rupture at 209.5 °C was 1886.5 kPa [16, 63]. For a rice grain (22.4 mg in weight) with moisture content of 13.08%, only  $8.0 \times 10^{-6}$  mol (4.8%) of water was turned into steam and was involved with the popping process (a detailed calculation is available in the Appendix). The vaporization of steam induced a subsequent cooling while liberating additional superheated water from the popped rice. Finally, the cooled, popped rice contained only 5.07% moisture as measured by TGA (Fig. 4.8A).

In the case of popped corn, the hull failed at lower temperature of 179.8 °C with a superheated steam expansion 1484 times the original volume of water at 100 °C. The superheated water at this temperature blew up the corn kernel containing starch granules (10-20  $\mu\text{m}$  diameters as shown in Fig. 4.7) into a porous popped corn with 13.2 times volume increment. Similar to that of popped rice, the pressure that ruptures the corn hull was 989.9 kPa. Although the corn kernel and rice grain contained almost the same moisture content (~13%), only 2.70% of water involved in the popping process of popped corn while 4.8% of that in popped rice. This is due to its lower popping temperature compared to that of popped rice. Finally, the cooled, popped corn contained only 4.36% moisture measured by TGA (see Fig. 4.8B). Our experimental

results and theoretical calculations are in good agreement with those of previous reports [15, 16, 19].

TGA technique was not only used for characterizing of the moisture content in popped rice, but also used for characterization of the moisture contents in the other parts of rice (i.e., un-milled rice, rice husk, and rice grain). As a result, the mass loss of water within the un-milled rice grain was 10.72% which was similar to the moisture content from AOAC 925.10-1925 measurement (~13%). The majority of moisture was in the rice grain (about 10.53%) while only 2.38% moisture was found within the rice husk (see Fig. 4.8A).

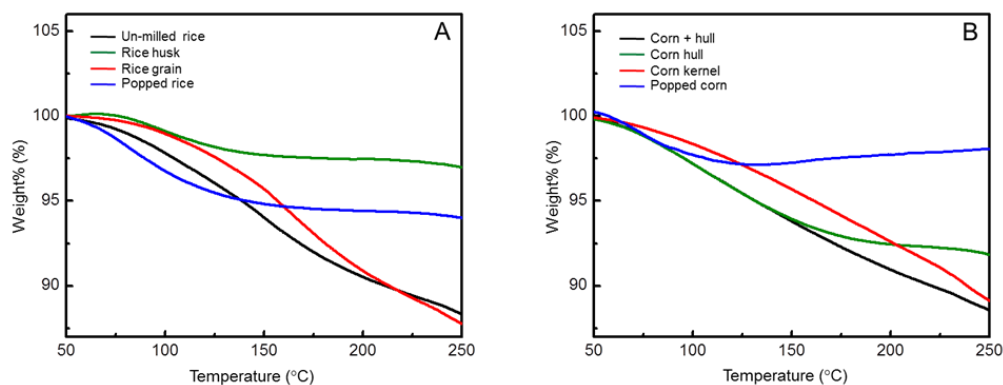
In the case of popped corn, the moisture of three parts of corn (i.e., the corn kernel with its hull, corn hull, and corn kernel) was also characterized by TGA technique. The TGA thermograms showed 11.02%, 7.50%, and 9.29% moisture content in the corn kernel with its hull, corn hull, and corn kernel, respectively (Fig. 4.8B).



**Table 4.1** Data summary of popped rice and popped corn compared to those from Hunt R. G.,1991 [16].

Grain/kernel properties	This research		Ref.16 <sup>f</sup>
	Rice	Corn	Corn
Avg. mass of a grain/kernel (g)	0.0224±0.0020	0.1570±0.0066	54.4 <sup>f</sup>
Avg. volume of a grain/kernel (cm <sup>3</sup> ) <sup>a</sup>	0.017±0.001	0.118±0.006	39.0 <sup>f</sup>
Avg. density of a grain/kernel (g/cm <sup>3</sup> )	1.32±0.03	1.33±0.01	1.39 <sup>f</sup>
Avg. mass of water inside a grain/kernel (g)	2.93x10 <sup>-3</sup>	2.066x10 <sup>-2</sup>	7.07 <sup>f</sup>
Avg. moisture content of a grain/kernel (%) <sup>b</sup>	13.08	13.16	13 <sup>f</sup>
<b>Popping condition</b>			
Avg. Popping temperature (T <sub>pop</sub> , K)	482.6±9.23	453.0±5.19	448
Pressure of saturated steam at T <sub>pop</sub> (kPa)	1886.7	989.9	889
Density of superheated water at T <sub>pop</sub> (kg/m <sup>3</sup> )	853.33	887.21	891
Density of steam at 373 K (kg/m <sup>3</sup> )	0.598	0.598	0.598
Mass of water involved in the popping process (g) <sup>c</sup>	1.4x10 <sup>-4</sup>	5.58x10 <sup>-4</sup>	0.167
Mass of water involved in the popping process (%)	4.8	2.70	2.3
Volume ratio of steam at T <sub>pop</sub> to that at 373 K	1427	1484	1490
<b>Popped rice/ Popped corn properties</b>			
Avg. mass of a popped rice/popped corn (g)	0.0236±0.0027	0.1509±0.0187	NA
Avg. volume of a popped rice/popped corn (cm <sup>3</sup> ) <sup>d</sup>	0.35±0.13	1.56±0.26	NA
Avg. density of a popped rice/popped corn (g/cm <sup>3</sup> )	0.078±0.031	0.0976±0.0115	NA
Avg. mass of water inside a popped rice/popped corn (g)	1.197x10 <sup>-3</sup>	6.576x10 <sup>-3</sup>	NA
Moisture content of a popped rice/popped corn (%) <sup>e</sup>	5.072	4.358	1-2 <sup>f</sup>
Expansion ratio of popped rice/popped corn	21	13.2	30 <sup>f</sup>

Note: The pressure at the popping temperature was obtained from the saturated steam table of the closed system where water and its steam are in equilibrium [63]. <sup>a</sup> measured by water displacement, <sup>b</sup> determined according to AOAC 925.10-1925, <sup>c</sup> mass and number of mole of water was calculated by the ideal gas equation of state (PV=nRT). Detailed calculations are available in the Appendix. <sup>d</sup> measured by sand displacement, <sup>e</sup> measured by TGA technique, <sup>f</sup> data from 54.4 g of corn kernel. The relatively high expansion ratio of 30 was due to voids between popped corns as the value was measured from popped corn derived from 54.4 g of corn kernels instead of a single popped corn. The butterfly-shaped popped corn is relative bulky with large voids (Fig. 4.9A).



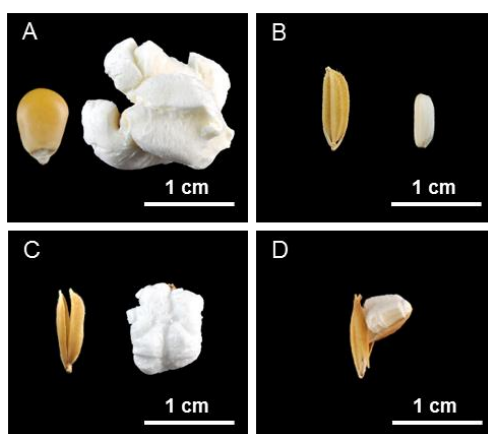
**Figure 4.8** TGA thermograms of (A) un-milled rice, rice husk, rice grain, and popped rice and (B) corn kernel with its hull, corn hull, corn kernel, and popped corn.

The calculations in Table 4.1 corroborate the more violent burst and higher jumping height of popped rice compared to popped corn as the high popping temperature led to higher catastrophic pressure. The pressure of steam at the popping temperature of popped rice (1886.7 kPa, 209.5 °C) was 1.9 times that of popped corn (989.9 kPa, 179.8 °C). The mass of water involved in the popping process was greater in the case of popped rice (4.8%) comparison to that of popped corn (2.7%). The higher popping temperature of popped rice was due to the thermal stability of rice husk. The husk with high silica content (~20%) [33, 35] decomposes at a relatively high temperature. The corn hull, on the other hand, yields at a certain critical temperature (177-187 °C) [46].

According to Table 4.1, the saturated steam pressures depend strongly on the popping temperatures. The masses of water involved in the popping process per a gram of starch were  $1.4 \times 10^{-4}$  g for popped rice and  $5.58 \times 10^{-4}$  g for popped corn. By popping at a higher temperature (i.e., 209.5 °C vs. 179.8 °C), the mass of water per gram of starch involved in the popping process was increased by 1.76 times. Since their volume expansions of steam at different popping temperatures are very similar (i.e., 1427 at 209.5 °C vs. 1484 at 179.8 °C), the mass of water per gram of starch involved in the

popping process controls the expansion volume of popped products. The volume expansion ratio of popped rice was 1.59 times that of popped corn. This value is in good agreement with the mass of water involved in the popping process.

The thermal decomposition of corn hull at the position which contacted with the heating plate created a releasing spot for superheated steam and the gelatinized starch [12, 19]. As confirmed by E. Virot and A. Ponomarenko, the protruding gelatinized starch or 'leg' compresses on the plate and induces a jumping of the popped corn. Our averaged popping temperature and pressure were in good agreement with those previous reports [15, 16, 19]. An expansion of gelatinized starch then creates the butterfly-shaped popped corn as shown in Fig. 4.9A [4]. Rice husk, on the other hand, did not thermally decompose but it breaks down at the interlocking between lemma and palea (Figs. 4.9B and 4.9C). The thermally stable rice husk promoted the pressure build-up of the superheated steam to the point where the interlocking cannot withstand. The rapid rupture of the husk with a release of superheated steam detached the popped rice and emptied rice husk (Fig. 4.9C). In the case of imperfect rice husk, the husk was prematurely ruptured at a relatively lower temperature. In this case, the husk and the popped rice are still intact (Fig. 4.9D).

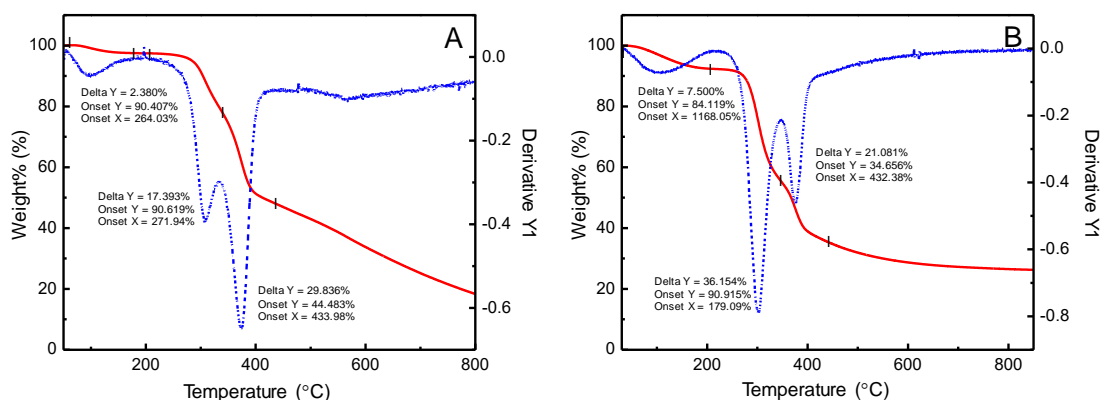


**Figure 4.9** Photographic images of (A) corn kernel and popped corn, (B) un-milled rice and a rice grain, (C) rice husk and a popped rice, and (D) an incomplete popped rice. The scale bars indicate 1 cm.

The temperature at the popping moment of both popped rice and popped corn were measured at the ruptured husk/hull. Therefore, thermal decompositions, which were analyzed by TGA technique, cannot explain the ruptured moment of rice husk and corn hull. From the TGA thermogram of rice in Fig. 4.10A, the initial mass loss was evaporated water (only 2.38%) while the major mass loss related to the decomposition of organic compounds. Most of cellulose started to burned at 250 to 350 °C while the lignin decomposition was occurred after 350 °C which are similar to the study of M. G. A. Vieira et al. [35]. Furthermore, the rice husk could withstand at high temperature, which a slow decomposition up to 800 °C confirmed the remained inorganic compounds. The main component of rice husk is silicon dioxide which is decomposed at high temperature (>1000 °C) [64].

For the TGA analysis of corn hull, the loss of water (7.50%) was observed in the initial curve. The rapid decomposition of major mass loss began at 250 °C and extended up to 340 °C which was due to the combustion of small molecules (i.e., sugar, protein, wax, etc.) and the depolymerization of hemicellulose. The third decomposition was related to lignin which was slightly decomposed between 340 and 450 °C. The compositions of corn hull were completely decomposed after 600 °C as shown in Fig. 4.10B.





**Figure 4.10** TGA thermograms of (A) rice husk and (B) corn hull. The corn hulls completely decomposed at  $\sim 400$  °C. The rice husk could withstand at high temperature as a slow decomposition up to 800 °C was observed.

According to the rupture behavior of corn hull from previous study, the hull was burnt at the position of which contacted with the heating plate. The gelatinized starch protruded from a burnt spot and began to expand in a few milliseconds later. For rice husk, on the other hand, the rupture of its husk was occurred by the superheated steam. The steam created the driving force of buildup pressure and breaking down the interlocking between lemma and palea.

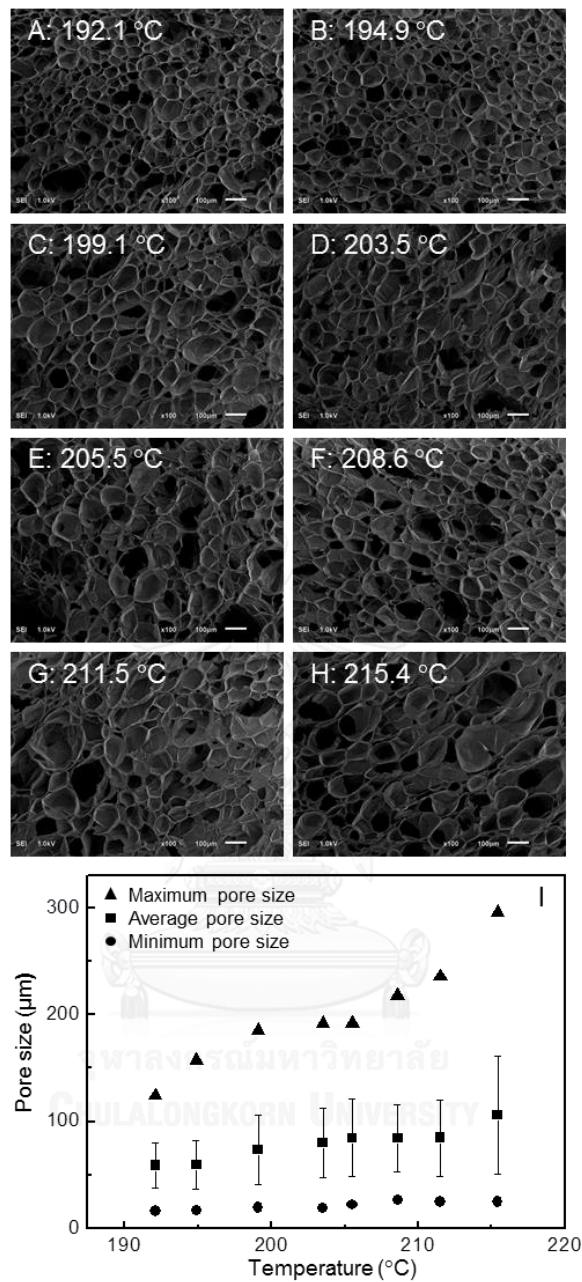
The weakest spot of rice husks was confirmed by the dye adsorption method. The five un-milled rice grains were taken out from water soluble yellow dye after soaking for 24 hrs (see Fig. 4.11A). The results showed that the dye preferentially deposited along the lemma-palea interlocking which can be clearly observed in the rice grains after removing the rice husks in Fig. 4.11B. The stained patterns suggest that dye diffused through only one side of the lemma-palea interlocking while no stain was observed on the other side/ as shown in Fig. 4.11C. The results suggested that only one side of interlocking is the weakest spot of rice husk.



**Figure 4.11** (A) Photographic images of un-milled rice grains after soaking in water-soluble yellow dye for 24 hrs. The dye preferentially deposited along the lemma-palea interlocking. (B) and (C) are the grains after removing the rice husk. The stained patterns occurred at only one side of the lemma-palea interlocking.

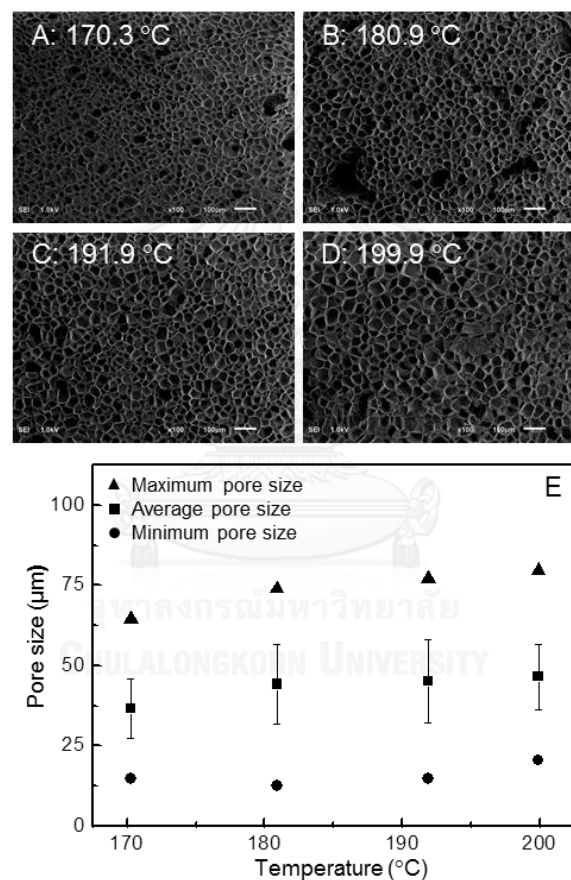
#### 4.2 Pore morphology characterizations of popped grains

The SEM images in Fig. 4.12 show cross sections of popped rice burst at various temperatures. The images display the morphologies of starch balloons developed from starch granules. The inter-connected starch balloons made the popped products a sponge-like structure with three-dimensional network of pores. An increase in the maximum pore size confirmed an increment of the mass of water involved in the popping process as the popping temperature increased. An increase of water mass per gram of starch involved in the popping process of 4.64, 4.91, 5.27, 5.76, 5.98, 6.29, 6.70, and 7.14 mg together with an increasing in the popping temperatures of 192.1, 194.9, 199.1, 203.5, 205.5, 208.6, 211.5, and 215.4 °C resulted in an increase in the maximum pore sizes of 124, 157, 185, 191, 192, 218, 235, and 296  $\mu\text{m}$ , respectively. Note: The maximum pore size represented the largest possible diameter of starch balloons achieved at a particular popping temperature.



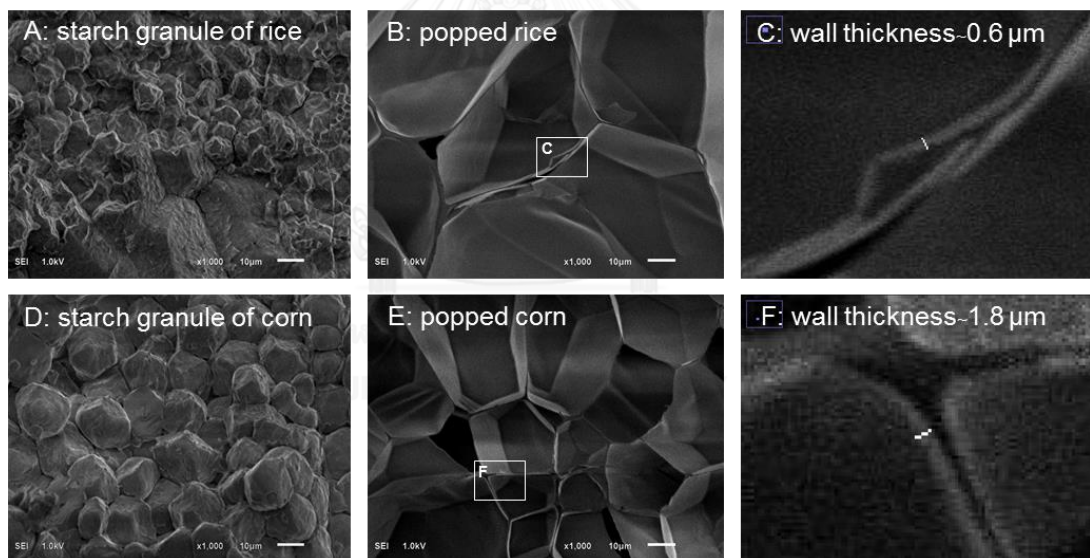
**Figure 4.12** SEM images show pore morphologies of popped rice burst at (A) 192.1, (B) 194.9, (C) 199.1, (D) 203.5, (E) 205.5, (F) 208.6, (G) 211.5, and (H) 215.4  $^{\circ}\text{C}$ . (I) A plot shows the maximum, minimum, and average pore sizes with error bars at various burst temperatures. A slight shift of the average pore size towards the minimum pore size suggested a non-uniform pore size distribution. The statistical data are calculated from 300 pores.

The popping temperature of popped corn was generally lower than that of popped rice. As a result, the mass of water involved in the popping process per gram of starch and volume expansion ratio were also smaller (Table 4.1). As shown in Fig. 4.13, popped corns popping at 170.3, 180.9, 191.9, and 199.9 °C with the maxima pore sizes of 64, 74, 77, and 79  $\mu\text{m}$ , respectively, were generated by  $2.90 \times 10^{-3}$ ,  $3.26 \times 10^{-3}$ ,  $4.55 \times 10^{-3}$ , and  $5.35 \times 10^{-3}$  g of water per gram of starch (i.e., as calculated by the ideal gas law model).



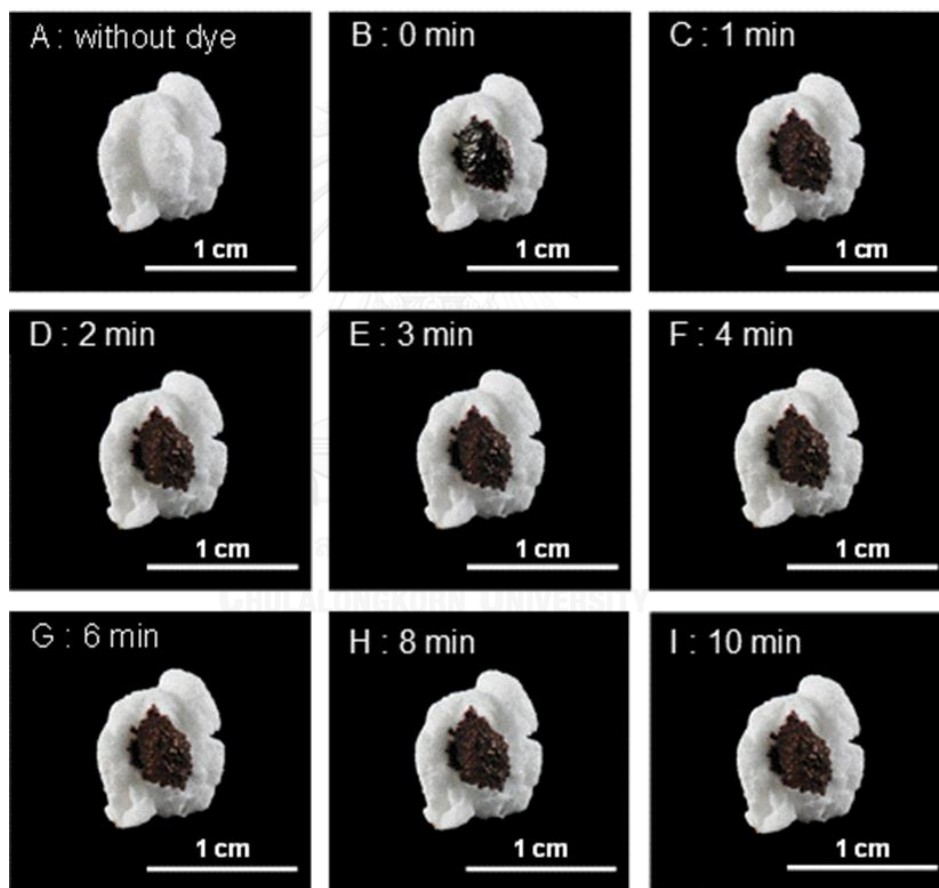
**Figure 4.13** SEM images show pore morphologies of popped corn burst at (A) 170.3 °C, (B) 180.9 °C, (C) 191.9 °C, and (D) 199.9°C. (E) A plot shows maximum, minimum, and average pore sizes with error bars at various burst temperatures. The statistical analysis was made from 300 pores.

According to the SEM images shown in Fig. 4.14A and 4.14D, the sizes of polygonal starch granules in rice grain and corn kernel were 5-15  $\mu\text{m}$  and 10-20  $\mu\text{m}$ , respectively [15, 16, 24, 26]. The rice starch granules that exploded into starch balloons at 208.6  $^{\circ}\text{C}$  had a maximum size of 218  $\mu\text{m}$  (Fig. 4.14B) and a wall thickness of  $\sim 0.6$   $\mu\text{m}$  (Fig 4.14C). The corn starch granules, on the other hand, exploded at 180.9  $^{\circ}\text{C}$  into starch balloons with a maximum size of 74  $\mu\text{m}$  in Fig. 4.14E and wall thickness of  $\sim 1.8$   $\mu\text{m}$ , Fig. 4.14F. The superheated water at higher temperature in popped rice turned starch granules into a softer gelatinized starch. The catastrophic pressure then exploded rice grain on turned the smaller rice starch granules into a bigger starch balloons with thinner walls.



**Figure 4.14** SEM images of (A) starch granules in a rice grain and the corresponding, (B) starch balloons popped at 208.6  $^{\circ}\text{C}$ , with (C) the wall thickness of  $\sim 0.6$   $\mu\text{m}$ . SEM images of (D) starch granules in a corn kernel and the corresponding, (E) starch balloons popped at 180.9  $^{\circ}\text{C}$ , with (F) the wall thickness of  $\sim 1.8$   $\mu\text{m}$ . (The scale bars of Fig. A-B and D-E indicate 10  $\mu\text{m}$ .)

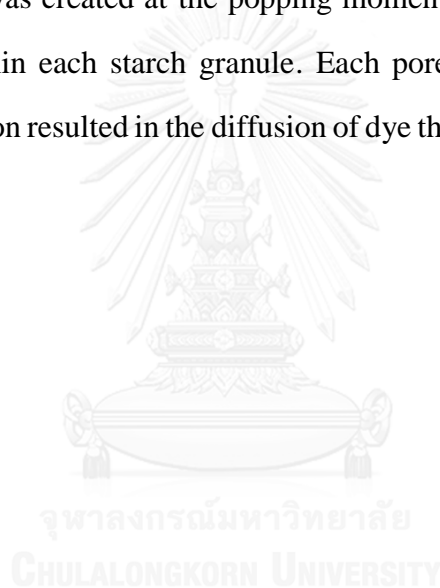
After the popping of un-milled rice grains, the morphology of popped rice appears sponge-like. To confirm the open-pore characteristic of popped rice, a drop of red organic dye was dropped on the top of popped rice. After dropping process in Fig. 4.15B, a wet organic dye was observed on to the top of popped rice. After that the toluene soluble dye suddenly diffused through the inner part of popped rice, as shown in Fig.4.15C. The results showed that after 1 min dye dropping, popped rice was dried up without spreading of organic dye in the radial direction (Fig. 4.15C-I).



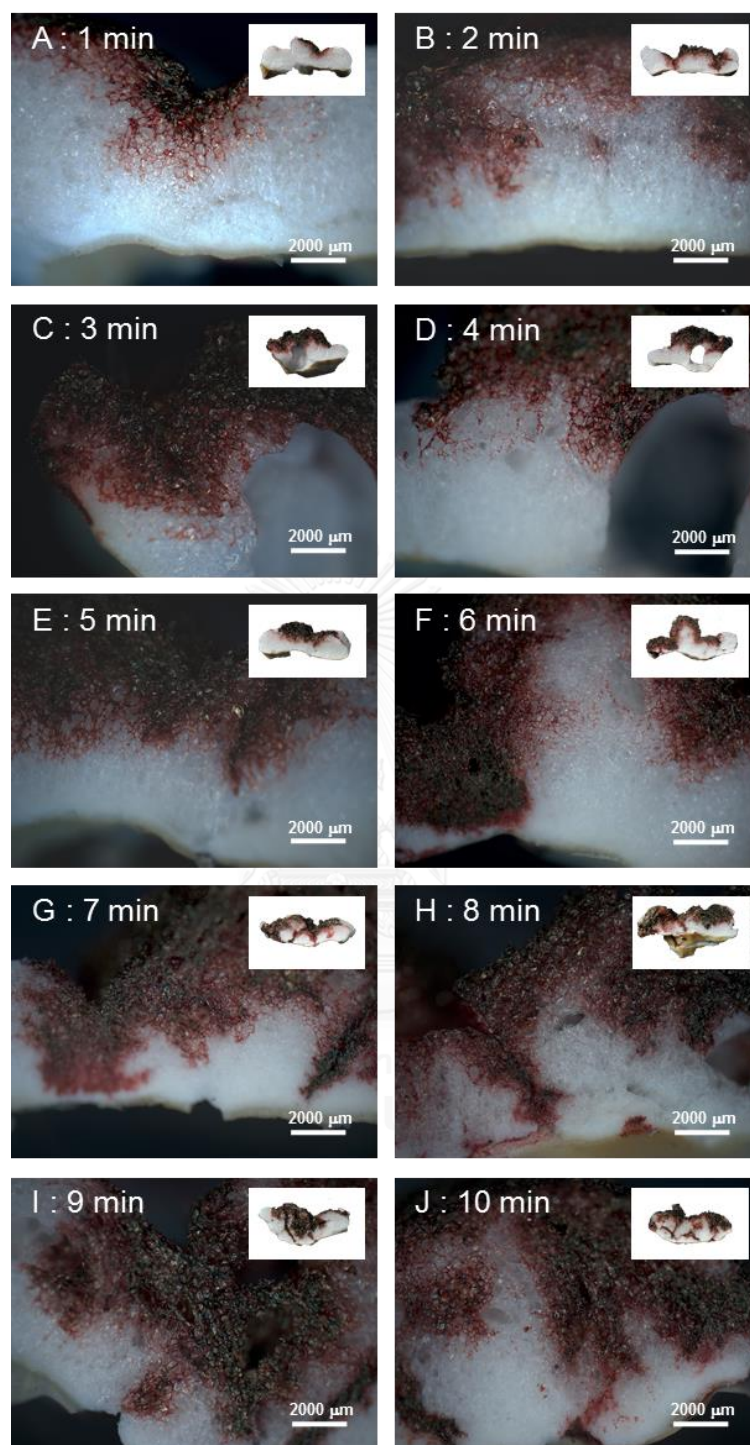
**Figure 4.15** Photographic images of a drop of red organic dye deposited on popped rice. A dye did not spread in the radial direction after 1 min dropping.

According to the result in Fig. 4.15, a drop of red organic dye did not spread in the radial direction which was observed on the top view. The result suggested that the

dye diffused through the open pores of the popped rice. Moreover, the open pore morphology of the popped rice was also confirmed by following the diffusion distance of dye at various passed times after dropping, as shown in Fig. 4.16. Each sample had 1 min interval dropping time until 10 min for the next samples. The popped rice was cut in a cross section after desired dye dropping time. In the cross section of popped rice, a drop of dye slightly diffused down after 1 min. The diffusion pattern of dye with 1 min interval dropping time showed the increase of diffusion distance as the increasing passed time. This phenomenon confirms the open pore morphology of popped rice. The open pore structure was created at the popping moment by releasing the superheated steam contained within each starch granule. Each pore connected with neighboring pores, this phenomenon resulted in the diffusion of dye through the interconnected pore.







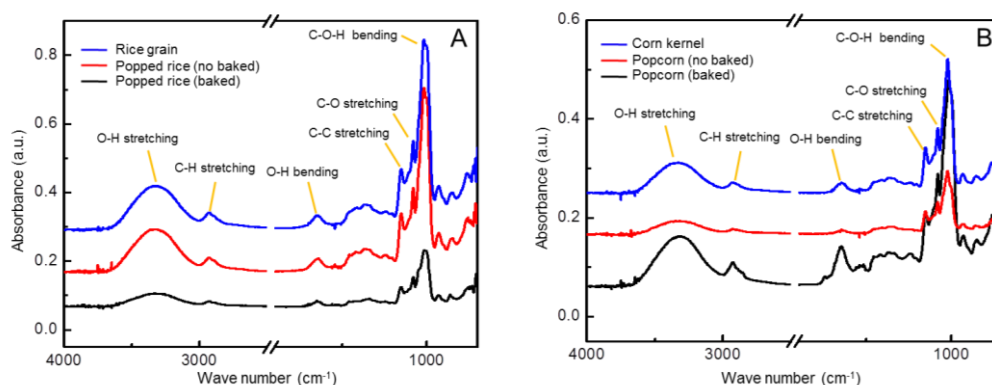
**Figure 4.16** Optical microscope images show cross sections of popped rice which were dropped by red organic dye on the top of popped rice, at various dropping time. The increase in the diffusion distance of organic dye resulted in an increase of diffusion times after dropping. This phenomenon confirms the open pore morphology of the popped rice.



The morphology of rice and corn starch was transformed from the numerous starch granules with polygonal shape into sponge-like structures with interconnected micro-balloons after popping. The FT-IR technique was employed to characterize functional groups of raw grain and popped products (i.e., unbaked and baked). FT-IR spectra for the rice grains and popped rice are shown in Fig. 4.17A. The functional groups of all samples (i.e., rice grain, unbaked popped rice, and baked pepped rice) were similar. They showed the extremely broad bands in the O–H stretching region at  $3400\text{ cm}^{-1}$  due to the hydrogen bond of water. The characteristic peaks at  $2918\text{ cm}^{-1}$  are the C–H stretching. The peak at  $1635\text{ cm}^{-1}$  indicate the O–H bending due to the absorbed water in the amorphous region of starch. The peaks at  $1248\text{ cm}^{-1}$  and  $1160\text{ cm}^{-1}$  are probably due to the coupling mode of C–C and C–O stretching, respectively, which represent the anhydroglucose ring. The strong absorption bands at  $1080\text{ cm}^{-1}$  represent C–O–H bending [65-67].

The FT-IR spectra of corn kernel, no baked popped corn, and baked popped corn are shown in Fig. 4.17B. These spectra show the same functional groups and the profiles are also similar. Moreover, the spectra of corn kernel and its popped products are not significantly different compare to those of rice grain and its popped product.

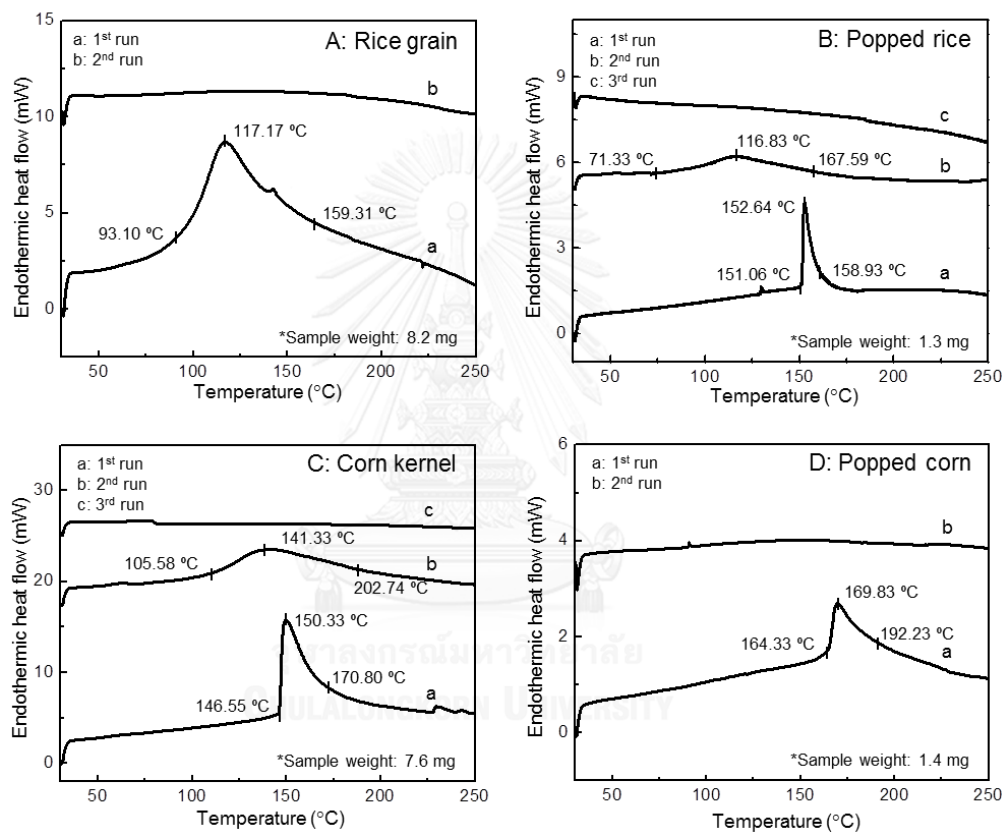
The results of FT-IR spectra indicate that they do not have any characteristic peaks related to the developed functional groups after popping process. These results confirm that the transformation of rice and corn do not produce new functional groups or involve chemical changes, and the transformation was purely a structural change.



**Figure 4.17** FT-IR spectra of (A) rice gain and popped rice and (B) corn kernel and popped corn.

The structural changes of rice/corn with their arrangements of polymer chains upon popping process were studied. The changes in crystalline domains of rice grain/popped rice and corn kernel/popped corn due to the hot air popping process are realized from DSC thermograms shown in Fig. 4.18. The rapid cooling of gelatinized starch is accompanying by the retrogradation process in popped products. Amylose and amylopectin chains rearrange and reassociate into ordered structures with increased crystallinity. In case of popped rice, an increase of crystalline temperature ( $T_p$ ) from 117 °C (rice grain, Fig. 4.18A) to 152 °C (popped rice, Fig. 4.18B) with a significant narrowing of the crystalline band suggested a drastic change in the semi-crystalline domain upon popping. An extremely rapid cooling made the semi-crystalline domain uniform. However, a slow cooling of the DSC measurements inhibits the retrogradation process as a sharp crystalline band was not observed in the 2<sup>nd</sup> run. A broad and small crystalline band centered at 116.83 °C was observed in the 2<sup>nd</sup> run. The disappearance of crystalline band in the 3<sup>rd</sup> consecutive run suggested the formation of amorphous domain of starch in popped rice after a slow cooling in DSC machine. In the case of popped corn, an increase of  $T_p$  from 150 °C (corn kernel, Fig. 4.18C) to 170 °C (popped corn, Fig. 4.18D) suggested a drastic change in the semi-crystalline domain upon popping. However, the crystalline band disappeared in the 2<sup>nd</sup> run (Fig. 4.18D). The

disappearance of the crystalline band after a subsequent DSC measurement may associate with water content in the samples. There are only 5% and 4.4% moisture content within popped rice and popped corn, respectively (Table 4.1). During the heating and subsequent slow cooling in the 1<sup>st</sup> DSC run, most of the water evaporated out of the starch structures. The retrogradation process with a rearrangement of disordered amylose and amylopectin chains cannot accomplish.



**Figure 4.18** DSC thermograms show thermal characteristics of (A) rice grain, (B) popped rice, (C) corn kernel, and (D) popped corn.

### 4.3 Influence of defects on the rice husk

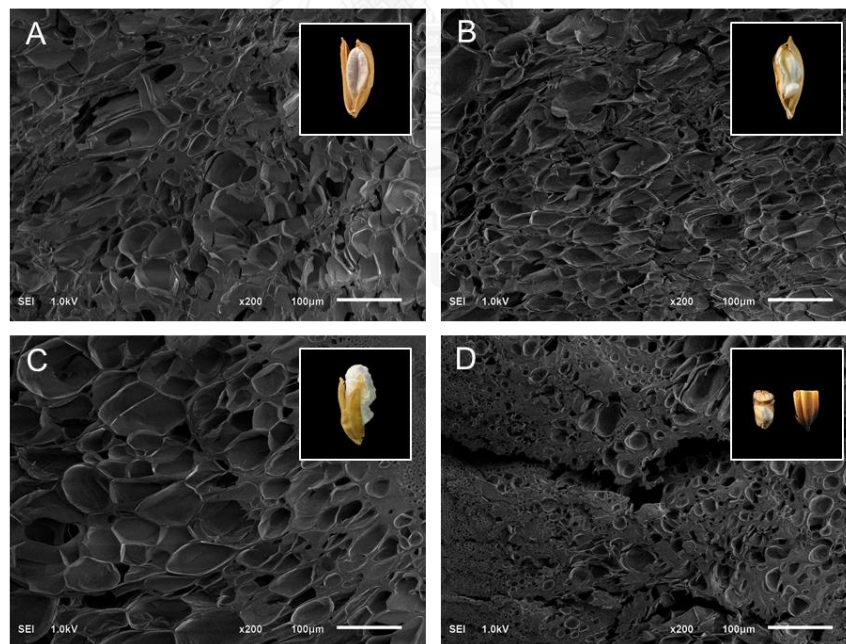
To confirm the existence of gelatinized starch prior to the popping of starch granules. The un-milled rice grains were intentionally defects on rice husks into 4 types (e.g., cleaving the lemma and occurred palea interlocking, slicing open part of the

dorsal, piercing with a needle, and cutting into halves) as shown in Fig. 4.19A1-4.19D1. The defected rice husks were heated by hot air until popping. Interestingly, although the husk was deliberately destroyed, the popping was still occurred. However, the popping was incomplete with minor expansion of the rice grain. The grain and the rice husk were still intact with part of the grain retained its original structure (Fig. 4.19A2-4.19D2). As a result, an incomplete popping was due to the loss of holding ability in the high pressure from superheated water at the popping moment. The expansion ability of rice grain was decreased when the defect of rice husk was increased.

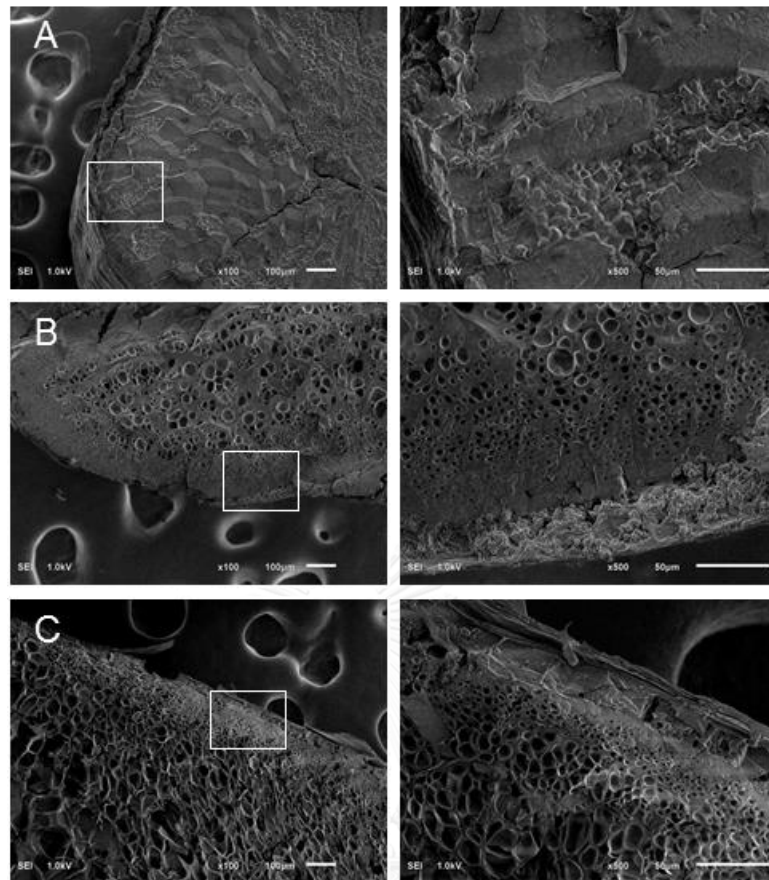


**Figure 4.19** Photographic images of incompletely un-milled rice and their popped products due to manmade defects. We deliberately made defects by (A) cleaving the lemma and palea interlocking, (B) slicing open part of the dorsal, (C) piercing with a needle, and (D) cutting into halves. Note: (A1-D1) incomplete un-milled rice grains and (A2-D2) popped products of incomplete un-milled rice grains.

From the results of the incompletely popped rice which were deliberately made defects, the SEM images in Fig. 4.20 indicated the small starch balloons from incomplete expansion of starch granules. The incompletely popped rice appeared with the small starch balloons and thick starch walls. The focus on popping pattern of popped rice in Fig. 4.21, the separated starch granules (Fig. 4.21A) were gelatinized and were fused together as the polyhedral boundaries were disappeared upon heating. The small starch balloons developed in the inner region of the grain. The pore morphology in Fig. 4.21B suggested that the outer part of the rice grain functioned as the vessel capable of withstanding superheated steam of lower pressures. It failed at much lower pressures compared to that of rice husk. As a result, smaller starch balloons with fused starch granules were obtained (Fig. 4.21B) as compared to big starch balloons with thin starch walls of the completely popped rice (Fig. 4.21C).



**Figure 4.20** SEM images of starch balloons of incompletely popped rice due to manmade defects made by (A) cleaving the lemma and palea interlocking, (B) slicing open part of the dorsal, (C) piercing with a needle, and (D) cutting into halves.



**Figure 4.21** SEM images show morphologies of (A) starch granules within rice grain, (B) starch balloons of incomplete popped rice via slicing open part of the husk (Fig. 4.19B), and (C) starch balloons of a complete popping.

Herein, our fundamental knowledge reviews the great pore size, high volume, and open pore characteristic of popped rice. We find the application of the unique phenomena of popped rice for adding the value of them. Therefore, we have demonstrated prototyping products of fragrance encapsulation from porous morphology of popped rice for slow-releasing air freshener with naturally biodegradable properties. This is one of several innovations for applying popped rice with increasing the value added of rice.

## CHAPTER V

### CONCLUSIONS

We have demonstrated that local, waxy rice variety, called 'Laow-taek' with high content of amylopectin (93.26% amylopectin and 6.74% amylose) can be popped by hot-air heating in the same manner as popcorn with extremely high amylopectin content. The averaged popping temperature of popped rice was 209.5 °C, which is ~30 °C greater than that of popped corn (179.8 °C). When compared between popped rice vs popped corn, the higher popping temperature led to a higher popping pressure (186.8 kPa for rice, 98.9 kPa for corn), greater maximum pore size of starch balloons (218 μm for popped rice, 74 μm for popped corn), and greater expansion ratio from the original grains (21 times for rice, 13.2 times for corn). The water involved in the popping process was also greater in the case of popped rice (4.8% vs 2.7%). Although their moisture contents were similar (~13%), the differences of their physical properties were due to the greater thermal stability and pressure tolerance of rice husks compared to those of corn hulls. The lemma-palea interlocking, which was the weakest spot of the air-tight rice husk, broke down at a relatively greater pressure. The high popping temperature of popped rice also led to a softer gelatinized starch under the superheated steam. As the catastrophic failure occurred at a higher temperature, the popped rice jumped higher. The influences of the thermally stable rice husk on pore morphologies were realized as an incomplete popping was observed when defects were made on the husk. Although the starch granules of rice and corn were transformed into sponge-like structure, the popping process did not create a new functional group. The retrogradation process was took place when a rapid cooling of gelatinized starch occurred after popping. The disrupted amylose and amylopectin chains rearranged and reassociated into ordered structures with increased crystallinity.

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**APPENDIX**

จุฬาลงกรณ์มหาวิทยาลัย  
CHULALONGKORN UNIVERSITY



## DATA CALCULATIONS OF POPPED RICE

### Primary information from experiment

Average mass of a grain (Avg. $M_g$ )	= 0.0224 g
Average volume of a grain (Avg. $V_g$ )	= 0.017 cm <sup>3</sup>
Percent average moisture content of a grain	= 13.08%
Average mass of a popped rice (Avg. $M_p$ )	= 0.0236 g
Average volume of a popped rice (Avg. $V_p$ )	= 0.35 cm <sup>3</sup>
Moisture content of a popped rice	= 5.072%
Average popping temperature (Avg. $T_{pop}$ )	= 482.6 K

### Calculation of physical parameters

Average density of a grain (Avg. $\rho_g$ )	$= \frac{\text{Avg. } M_g}{\text{Avg. } V_g}$ $= \frac{0.0224 \text{ g}}{0.017 \text{ cm}^3}$ $= 1.32 \text{ g/cm}^3$
Mass of water ( $M_w$ ) inside a grain	$= \frac{(\text{Avg. } M_g)(\% \text{Avg. moisture content})}{100}$ $= \frac{(0.0224 \text{ g})(13.08)}{100}$ $= 2.9299 \times 10^{-3} \text{ g}$ $\approx 2.93 \times 10^{-3} \text{ g}$
Average density of a popped rice (Avg. $\rho_p$ )	$= \frac{\text{Avg. } M_p}{\text{Avg. } V_p}$ $= \frac{0.0236 \text{ g}}{0.35 \text{ cm}^3}$ $= 0.078 \text{ g/cm}^3$

### The pressure of steam at the popping temperature

$$\begin{aligned}
 \text{Average popping temperature (Avg. } T_{\text{pop}}) &= 482.6 \text{ K} \\
 \text{Pressure of saturated steam (} P_{\text{ss}}) \text{ at } T_{\text{pop}} &= 1886.7 \text{ kPa (18.62 atm)} \\
 \text{Density of superheated water (} \rho_{\text{sw}}) \text{ at } T_{\text{pop}} &= 853.33 \text{ kg/m}^3 \\
 \text{Density of steam (} \rho_{\text{s}}) \text{ at 373 K} &= 0.598 \text{ kg/m}^3 \\
 \text{Mole of water involved in the popping process (n)} &= \frac{(P_{\text{ss}})(\text{Avg. } V_{\text{g}})}{(R)(\text{Avg. } T_{\text{pop}})} \\
 &= \frac{(18.62 \text{ atm})(0.017 \times 10^{-3} \text{ L})}{(0.0821 \text{ L} \cdot \text{atm} \cdot \text{K}^{-1} \cdot \text{mol}^{-1})(482.6 \text{ K})} \\
 &= 7.9992 \times 10^{-6} \text{ mol} \\
 &\approx 8.0 \times 10^{-6} \text{ mol} \\
 \text{M}_w \text{ involved in the popping process (g)} &= \text{number of mole (n) x MW of water (g)} \\
 &= (8.0 \times 10^{-6} \text{ mol})(18 \text{ g} \cdot \text{mol}^{-1}) \\
 &= 1.4 \times 10^{-4} \text{ g} \\
 \text{\% of water involved in the popping process} &= \frac{\text{M}_w \text{ involved in the popping process x 100}}{\text{M}_w \text{ inside a grain}} \\
 &= \frac{(1.4 \times 10^{-4} \text{ g})(100)}{2.93 \times 10^{-3} \text{ g}} \\
 &= 4.8\% \\
 \text{Volume ratio of steam at } T_{\text{pop}} \text{ to that at 373 K} &= \frac{\rho_{\text{sw}} \text{ at } T_{\text{pop}}}{\rho_{\text{s}} \text{ at 373 K}} \\
 &= \frac{853.33 \text{ kg} \cdot \text{m}^{-3}}{0.598 \text{ kg} \cdot \text{m}^{-3}} \\
 &= 1427 \\
 \text{Average } M_w \text{ inside a popped rice} &= \frac{(\text{Avg. } M_p)(\% \text{ Avg. moisture content})}{100} \\
 &= \frac{(0.0236 \text{ g})(5.072)}{100} \\
 &= 1.197 \times 10^{-3} \text{ g}
 \end{aligned}$$

Expansion ratio of popped rice

$$\begin{aligned} &= \frac{\text{Avg. } V_p}{\text{Avg. } V_g} \\ &= \frac{0.35 \text{ cm}^3}{0.017 \text{ cm}^3} \\ &= 21 \end{aligned}$$



## DATA CALCULATIONS OF POPPED CORN

### Primary information from experiment

Average mass of a kernel (Avg. $M_k$ )	= 0.1570 g
Average volume of a kernel (Avg. $V_k$ )	= 0.118 cm <sup>3</sup>
Percent average moisture content of a kernel	= 13.16%
Average mass of a popped corn (Avg. $M_p$ )	= 0.1509 g
Average volume of a popped corn (Avg. $V_p$ )	= 1.56 cm <sup>3</sup>
Moisture content of a popped corn	= 4.358%
Average popping temperature (Avg. $T_{pop}$ )	= 453.0 K

### Calculation of physical parameters

Average density of a kernel (Avg. $\rho_g$ )	$= \frac{\text{Avg. } M_k}{\text{Avg. } V_k}$ $= \frac{0.1570 \text{ g}}{0.118 \text{ cm}^3}$ $= 1.33 \text{ g/cm}^3$
Mass of water ( $M_w$ ) inside a kernel	$= \frac{(\text{Avg. } M_k)(\% \text{Avg. moisture content})}{100}$ $= \frac{(0.1570 \text{ g})(13.16)}{100}$ $= 0.02066 \text{ g}$ $\approx 2.066 \times 10^{-2} \text{ g}$
Average density of a popped corn (Avg. $\rho_p$ )	$= \frac{\text{Avg. } M_p}{\text{Avg. } V_p}$ $= \frac{0.1509 \text{ g}}{1.56 \text{ cm}^3}$ $= 0.0976 \text{ g/cm}^3$

### The pressure of steam at the popping temperature

Average popping temperature (Avg. $T_{pop}$ )	= 453.0 K
Pressure of saturated steam ( $P_{ss}$ ) at $T_{pop}$	= 989.9 kPa (9.7696 atm)
Density of superheated water ( $\rho_{sw}$ ) at $T_{pop}$	= 887.21 kg/m <sup>3</sup>
Density of steam ( $\rho_s$ ) at 373 K	= 0.598 kg/m <sup>3</sup>
$M_w$ involved in the popping process (n)	= $\frac{(P_{ss})(Avg. V_k)}{(R)(Avg. T_{pop})}$
	= $\frac{(9.7696 \text{ atm})(0.118 \times 10^{-3} \text{ L})}{(0.0821 \text{ L} \cdot \text{atm} \cdot \text{K}^{-1} \cdot \text{mol}^{-1})(453.0 \text{ K})}$
	= $3.10 \times 10^{-5} \text{ mol}$
$M_w$ involved in the popping process (g)	= number of mole (n) x MW of water (g)
	= $(3.10 \times 10^{-5} \text{ mol})(18 \text{ g} \cdot \text{mol}^{-1})$
	= $5.58 \times 10^{-4} \text{ g}$
% of water involved in the popping process (g)	= $\frac{M_w \text{ involved in the popping process}}{Avg. M_g}$
	= $\frac{(5.58 \times 10^{-4} \text{ g})(100)}{2.066 \times 10^{-2} \text{ g}}$
	= 2.70%
Volume ratio of steam at $T_{pop}$ to that at 373 K	= $\frac{\rho_{sw} \text{ at } T_{pop}}{\rho_s \text{ at } 373 \text{ K}}$
	= $\frac{887.21 \text{ kg} \cdot \text{m}^{-3}}{0.598 \text{ kg} \cdot \text{m}^{-3}}$
	= 1484
Average $M_w$ inside popped corn	= $\frac{(Avg. M_p)(\% Avg. \text{ moisture content})}{100}$
	= $\frac{(0.1509 \text{ g})(4.358)}{100}$

Expansion ratio of popped corn

$$\begin{aligned} &= 6.576 \times 10^{-3} \text{ g} \\ &= \frac{\text{Avg. } V_p}{\text{Avg. } V_k} \\ &= \frac{1.56 \text{ cm}^3}{0.118 \text{ cm}^3} \\ &= 13.2 \end{aligned}$$



## RESEARCH ACHIEVEMENTS

I have successfully studied the popping mechanism of popped rice and applied the scientific knowledge into commercial potentiality. I found many opportunities for practicing presentation in research field such as instructor of technology workshops, innovations and technology exhibitor, and contest participant. I have received three awards from the scientific society and national organization. Moreover, my work was publicized in radio and national newspaper. The list of research achievements is given below.

Honors and Awards:

**2016**

**Award:** The second prize of the 9<sup>th</sup> Science and Technology Initiative and Sustainability Awards (STISA) in concept of “Creative Processes and Materials for Social Sustainability”, 1 August 2016

**Organized by:** Thai Institute of Chemical Engineering and Applied Chemistry cooperated with Dow Chemical Company and SCG Chemical Company

**Invention:** ‘Scented Popped Rice’ Innovation for Fragrance Encapsulation and Slow Releasing (team: MONTAROP)

**2016**

**Award:** The second prize of the Invention and Innovation Contest 2016 of Graduate Student, 18-20 August 2016

**Organized by:** National Research Council of Thailand (NRCT)

**Invention:** Innovative Products from Popped Rice

**2017**

**Award:** Preliminary Final Round in The Three Minute Thesis (3MT®) at Chulalongkorn University, 24 February 2017

**Organized by:** Faculty of Engineering, Chulalongkorn University with Total Access Communication PLC. (DTAC)

**Invention:** Popping Mechanism of Popped rice

**Instructor of Workshops on Scented Popped Rice Technology**

1. Workshop on scented popped rice production  
Technology transferred group: Agriculturist Group in Yasothon Province, Thailand, 25-26 May 2015
2. Workshop on scented popped rice production  
Technology transferred group: Agriculturist Group in Saraburi Province, Thailand, 7 November 2015
3. Workshop on scented popped rice production  
Technology transferred group: Agriculturist Group in Raychaburi Province, Thailand, 17 May 2016

**Innovations and Technology Exhibitions**

1. Innovation and technology exhibition on scented popped rice product, Inventor's Day 2015, 2-5 February 2015, IMPACT Arena, Exhibition and Convention Center, Bangkok, Thailand
2. Innovation and technology exhibition on scented popped rice product, Smart SME EXPO 2015, 2-5 July 2015, IMPACT Arena, Exhibition and Convention Center, Bangkok, Thailand



3. Innovation and technology exhibition on scented popped rice product, Thailand Research Expo 2015, 16-20 August 2015, Centara Grand Hotel and Convention Center at CentralWorld, Bangkok, Thailand
4. Innovation and technology exhibition on scented popped rice product, Medical Innovation of Thailand Fair, 18-20 August 2015, Hall 9 IMPACT Forum, Exhibition and Convention Center, Bangkok, Thailand
5. Innovation and technology exhibition on scented popped rice product, Thailand Innovation and Design EXPO 2015, 17-20 September 2015, Queen Sirikit National Convention Center, Bangkok, Thailand
6. Innovation and technology exhibition on scented popped rice product, Public Announcement of Research for Community, 9 December 2015, National Research Council of Thailand (NRCT), Bangkok, Thailand
7. Innovation and technology exhibition on scented popped rice product, Public Announcement of 1<sup>st</sup> year Anniversary of Government Achievements, 23-25 December 2015, Government House of Thailand, Bangkok, Thailand
8. Innovation and technology exhibition on scented popped rice product, Progressive Thinking project, 2-4 February 2016, Thai Army Club Viphavadi, Bangkok, Thailand
9. Innovation and technology exhibition on scented popped rice product, Public Announcement of Technology Show 1/2016, 3 February 2016, Ministry of Science and Technology, Bangkok, Thailand
10. Innovation and technology exhibition on scented popped rice product, Technology Show 1/2016, 23-24 February 2016, Queen Sirikit National Convention Center, Bangkok, Thailand
11. Innovation and technology exhibition on scented popped rice product, Salable Researches for Thailand Sustainability, 5-27 March 2016,

- Khlong Phadung Krung Kasem, Government House of Thailand, Bangkok, Thailand
12. Innovation and technology exhibition on scented popped rice product and contest participation, The 9<sup>th</sup> Science and Technology Initiative and Sustainability Awards (STISA) in concept of “Creative Processes and Materials for Social Sustainability”, 1 August 2016, Faculty of Engineering, King Mongkut's Institute of Technology, Ladkrabang, Bangkok, Thailand (The second prize)
  13. Innovation and technology exhibition on scented popped rice product and contest participation, The Invention and Innovation Contest 2016 of Graduate Student, 17-21 August 2016, Centara Grand Hotel and Convention Center at CentralWorld, Bangkok, Thailand (The second prize)
  14. Innovation and technology exhibition on scented popped rice product and contest participation, Inventor's Day 2016, 26-27 September 2016, National Research Council of Thailand (NRCT), Bangkok, Thailand

### **Publicities**

1. News publicity of scented popped rice product, Dailynews Newspaper, 11 February 2016
2. Guest speakers, The Scientific World on CU Radio FM 101.5 MHz, Chulalongkorn University Broadcasting Station, On air in 1 October 2016, 10-10.30 am

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Chulalongkorn University, Bangkok, Thailand

Scholarships:

2010-2015 The Science Achievement Scholarship of Thailand (SAST)

2015-2017 The National Research University Project, the Office of  
Higher Education Commission (WCU-58-008-FW).

Conferences:

1. Popping Mechanism of Popped Rice, Pure and Applied Chemistry  
International Conference (PACCON) 2016, 9-11 February 2016,  
BITEC, Bangkok, Thailand
2. Influence of Popping Temperature on Pore Size of Popped rice and  
Popcorn, The 42nd Congress on Science and Technology of  
Thailand (STT42), 30 November- 2 December, 2016, Centara Grand  
at Central Plaza Ladprao, Bangkok, Thailand

Publication:

Influence of Popping Temperature on the Structure of Popped Rice

