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Effects of partial gelatinization on structure and thermal properties of corn starch after spray drying

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ABSTRACT

Partially gelatinized starch was prepared by gelatinizing corn starch at 64–72 °C followed by spray drying (inlet temperature, feed flow rate, air flow rate of 200 °C, 7.2 ml/min and 0.375 m^3/h, respectively). The morphology, granule size, crystalline fraction, swelling power and gelatinizing properties of partially gelatinized starch were investigated and compared with those of native starch. The surface morphology of partially gelatinized starch granules was shriveled with multiple surface folds. The granule size of partially gelatinized starch was smaller than that of native corn starch. There was either complete absence of crystalline fraction in partially gelatinized starch or the extent of crystalline fraction was greatly reduced. The swelling power of partially gelatinized starch was higher than that of the native starch below 60 °C, while it was lower than that of native starch above 60 °C. There was an increase in gelatinization temperature and a decrease in gelatinization enthalpy in partially gelatinized starch compared to that of native starch.

1. Introduction

Starch has been widely used in food, chemical, textile, papermaking, medicine and many other industries because it is biodegradable, comes from renewable sources and is relatively cheap (Hoover, Hughes, Chung, & Liu, 2010). However, native starch has limited application because of its instability under processing conditions that involve variation in temperature, shear and pH. Physical and chemical modification is commonly applied to develop desirable functional properties such as solubility, adhesion and heat tolerance (Bhosale & Singhal, 2007).

Starch has been considered as a semi-crystalline material. Structurally intact starch granules are known to exhibit three characteristic diffraction patterns designated as the A-, B- and C-types (Singh, Ali, Somashelar, & Mukherjee, 2006). The crystallinity of native starch granules varies from 15 to 45%. Because of this reason, the crystallinity plays an important role in the starch granule architecture and physicochemical characteristics, such as the susceptibility to enzymatic degradation and the poor solubility in cold water (Tang, Mitsunaga, & Kawamura, 2006).

There are many studies which have investigated the ways in which the degree of crystallinity in starch granules is reduced or completely eliminated, such as high-pressure treatment (Błaszczak et al., 2007; Li et al., 2011), oxidation (Fiedorowicz & Para, 2006), ball milling (Tamaki, Hisamatsu, Teranishi, & Yamada, 1997) and spray drying (Laovachirasuwan, Peerapattana, Srijesdaruk, Chitropas, & Otsuka, 2010). The ordered structure of starch granules can be disrupted by these treatments and amorphous starch can be produced. However, the destruction of granule integrity is severely affected when starch granules are subjected to high-pressure treatment. The degree of oxidation is not easy to control and the chemical oxidation process is far more complicated compared to methods involving physical modifications. Similarly, it requires very long milling time if the crystalline fraction of starch granules has to be removed by ball milling.

Spray drying is a very common technique used for preparing starch-based materials because of its low cost and available equipment (Gharsallaoui, Roudaut, Chambin, Voilley, & Saurel, 2007). It is very rapid drying process which converts liquid solution droplets mostly into amorphous or semi-amorphous particulates. Due to these reasons, pregelatinized starch without losing granular integrity has been prepared by spray drying since a long time ago (Pitchon, O'Rourke, & Joseph, 1981; Rubens, 1992). Native corn starch is slurried in water at room temperature. The slurry is pumped into nozzle with a centrifugal pump. Saturated steam, as the heating and atomizing medium, is injected into nozzle providing a suitable ratio of steam and slurry. The spray from the nozzle appears uniform, narrow, long and travels down the dryer in waves. The finely atomized gelatinized starch dries easily without any significant build-up of hard or gelatinous material. Pregelatinized starch prepared by spray drying has significantly better properties

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than a drum-dried sample which set quickly to a grainy, chunky consistency.

Pregelatinized starches are used widely for their attributes such as dispersibility in hot or cold water, high viscosity and smooth texture. In many starchy foods, a portion of residual starch is not fully gelatinized during processing, usually due to limited water content or insufficient heating (Chung, Lim, & Lim, 2006). Studies of starch ghosts have described the preparation of gelatinized granular starch (Atkin, Abeysekera, & Robards, 1998; Debet & Gidley, 2007). However, there is surprisingly little background information available in the literature on the effect of pre-drying treatments on the structure and properties of dried materials. When starch is heated in excess water above gelatinization temperature, starch granules will swell and ultimately lose their birefringence. Partially gelatinized starch can be prepared by heating the starch slurry at a suitable temperature over a certain period of time and drying it using spray drying. By eliminating or greatly removing the crystalline fraction in starch, the granule size and size distribution, crystalline/amorphous nature, swelling power and the gelatinization temperature and other physic-chemical characteristics of partially gelatinized starch can be greatly altered (Liu, Zhang, Shen, Hu, & Li, 2010).

The objectives of the current work were to prepare partially gelatinized starch from corn through pre-heating followed by spray drying and to study the properties of partially gelatinized starch obtained. The morphology, granule size and size distribution, crystalline/amorphous nature and swelling power of partially gelatinized starch were studied and compared to those of native corn starch.

2. Materials and methods

2.1. Materials

Corn starch was obtained from Hebei Zhangjiakou Yujin Food Co., Ltd. (Hebei, China). Its moisture content was 10% (w/w) during the entire tests period.

2.2. Preparation of partially gelatinized starch

Starch dispersions at a solid concentration of 10.0% (w/w) were prepared by adding 20.0 g of pre-dried corn starch into deionized water at 24 ± 1 °C. Each batch of dispersion was thoroughly stirred at 300 rpm (in beakers) for 15 min using a thermostated water bath at 64 °C, 66 °C, 68 °C, 69 °C, 70 °C and 72 °C. The evaporation of water during the gelatinization stage was minimized by covering the beakers with six layers of preservative films.

The partially gelatinized starch dispersions obtained as described above were spray dried using a bench-top spray drier (GPW120-II, Shandong Tianli Drying Equipment Inc., China). The inlet temperature and the exhaust aspiration lever were set at 200 °C and 95%, respectively. The flow rate of the air was maintained at 0.375 m^3/h. The feed rate was 7.2 ml/min throughout the experiments. The corresponding dried granular starches (*S64*, *S66*, *S68*, *S69*, *S70*, and *S72*) were collected at the bottom of the cyclone.

2.3. Morphology examination

The morphology of sample was examined on the scanning electron microscope (S-3400N, Hitachi Instruments Ltd., Japan). Sample particles were fixed on the silicon wafer and sputtered with gold. The shape and the surface characteristics of the samples were observed and recorded under S-3400N.

2.4. Granule size distribution

The granule size distribution of sample was determined using laser light diffraction technique with a He–Ne laser (Mastersizer 2000, Malvern Instruments, UK). The sample was dispersed in anhydrous ethanol which was used as dispersing reagent using a sonicator attached with the Mastersizer 2000. Volume size distribution of the particles was obtained using a computer program supplied by the manufacturer and the average particle size was expressed as volume mean diameter ($D[4, 3]$) in micrometer. Measurements were carried out in triplicate and average values are reported here.

2.5. X-ray diffraction (XRD) analysis

The amorphous/crystalline structure of the sample was characterized using a XD-2 X-ray diffractometer (Beijing Purkinje General Instrument Co., Ltd., China) under the following conditions: Nickel filtered Cu Kα radiation (λ = 0.15406 nm) at a voltage of 36 kV and current of 20 mA. Samples were scanned from 10° to 40° (2θ) with a scanning rate of 0.5°/min and sampling interval of 0.02°.

2.6. Swelling power (SP)

SP was determined by using the method proposed by Li and Yeh (2001) with some modification. About 0.4 g of spray dried sample was weighted into a centrifuge tube with coated screw cap. Deionized water was added to maintain the solid concentration of the dispersion at 2.0% (w/w). The tube and its content was gently shaken by a planetary shaker and was heated at 30 °C, 50 °C, 60 °C, 70 °C, 80 °C and 90 °C in a water bath for 30 min. The tube was cooled to room temperature in a second water bath. Finally, the heated starch particle suspension was centrifuged at 3000 rpm for 15 min. The supernatant was poured out from the tube carefully. The weight of the residue (W_1) was recorded. Then the residue was dried to constant weight (W_2) in an air oven at 105 °C. The swelling power of the starch granules (SP) was calculated using Eq. (1), given below.

$$\mathrm{SP} = \frac{W_1}{W_2} \tag{1}$$

2.7. The gelatinization properties

The gelatinization properties of the sample in the presence of excess water was measured using a differential scanning calorimeter (DSC-Q10, TA Instruments, New Castle, USA) equipped with a thermal analysis data station. The DSC analyzer was calibrated using indium (melting point = 156.6 °C, melting enthalpy = 26.59 J/g) and an empty aluminum pan was used as a reference. Distilled water (7.5 μL) was added with a microsyringe into starch (2.5 mg) in an aluminum pan. This mixture of starch and water was then immediately sealed with an aluminum lid and hermetically sealed. Each sealed sample was equilibrated for 3 h at ambient temperature before thermally scanning in the DSC. The samples were then heated from 20 °C to 120 °C at 10 °C/min. The onset, peak, and the end temperatures (T_o, T_p, and T_e) together with gelatinization enthalpy (ΔH_{gel}) were quantified.

The degree of gelatinization (GD) was calculated using Eq. (2), given below (Błaszczak et al., 2007).

$$\mathrm{GD} = \{(\Delta H_{ns} - \Delta H_{ts})\Delta H_{ns}^{-1}\} \times 100\% \tag{2}$$

where ΔH_{ns} and ΔH_{ts} are the melting enthalpies of native and modified starches, respectively.

2.8. Statistical analyses

All experiments were carried out at least triplicate and results are reported as the mean and standard derivation of these measurements excluding the X-ray diffractograms. Duncan's multiple comparison tests were conducted to determine the significant effect of heating processes on the properties of starch samples at 95% ($p < 0.05$) confidence level using the SPSS statistical package (LEAD Technologies, USA).

3. Results and discussion

3.1. Morphology examination

The change in morphology of spray dried corn starch particles as affected by the gelatinization process are shown in Fig. 1. As can be seen from these SEM micrographs the shape as well as the surface morphology of the spray dried starch granules changed remarkably as the gelatinization temperature increased. The shape and morphology of particle whose precursor slurry was gelatinized at 72 °C (*S72*) were similar to those of the particle whose precursor slurry was gelatinized at 70 °C (*S70*) and the micrograph of former is not presented in Fig. 1.

As can be seen in Fig. 1A, native corn starch granules have a characteristic truncated shape of native starch which is in agreement with the previous publications reporting the shape of starch granules (Nagano, Tamaki & Funami, 2008; Van Velde, Van Riel & Tromp, 2002). When the starch was heated at 64 °C, the shape of the subsequently spray dried (*S64*) granules was almost similar to that of the native starch. A notable difference is that some degree of surface folds or troughs appeared on the surface of *S64* starch granules (Fig. 1B). When the starch was heated at 66 °C, the shape of the spray dried starch granules became elliptical and shrinkage of volume occurred in some large granules (Fig. 1C). As the heating temperature increased from 68 °C to 72 °C, surface of the spray dried granules became more and more concave and shriveled, which is typical of particles of macromolecules produced by spray drying (Bertolini, Siani, & Grosso, 2001; Tonon, Grosso, & Hubinger, 2011) (Fig. 1D, 68 °C; Fig. 1E, 69 °C; Fig. 1F, 70 °C).

Liu and Zhao (1990) studied the gelatinization of starch granules in excess water and found that granules lost their native structure and formed a molecular network at 64 °C. In contrast, Ratnayake and Jackson (2006) reported that complete granular disruption and the formation of a gelatinized solution did not occur below 70 °C. They suggested that the gelatinization temperature of starch depends on the concentration of starch in the solution and the source of plant from which it is derived. As shown in Fig. 1, there was no obvious difference on the surface morphology and shape between native corn starch and *S64*. This might be due to the fact that water was unable to penetrate into starch granules and only the outmost layer of the granules was hydrated. When only partially hydrated granules were spray dried, the water absorbed at the outmost layers was rapidly vaporized which made the surface of granules become concave and slightly wrinkled. As the heating temperature increased during gelatinization, the starch granules began to absorb large amount of water which allowed the granules to expand to a greater extent. As a result, the internal structure of starch granules was disintegrated and the granules were swollen considerably. When these greatly expanded (in size) or subsequently ruptured starch granules were spray dried, the volumetric shrinkage in the subsequently spray dried granules was quite remarkable.

Furthermore, the development in the surface morphology in the gelatinized starch particles follows the mechanisms of morphology development in solutions of polymeric macromolecules such as maltodextrins and whey proteins as explained by Adhikari, Howes, Bhandari, and Troung (2003). They suggested that the effective moisture diffusivity in the solution of polymeric macromolecules is very slow which results into to very high moisture gradients between the drying air and the interior of the droplets/particles of such polymeric macromolecules. In order to reduce the diffusion path and to provide greater interfacial area for moisture evaporation (ultimately to enhance the outward diffusion of moisture) the drying droplets/particles try various ways to increase the surface available for moisture evaporation and reduce the distance

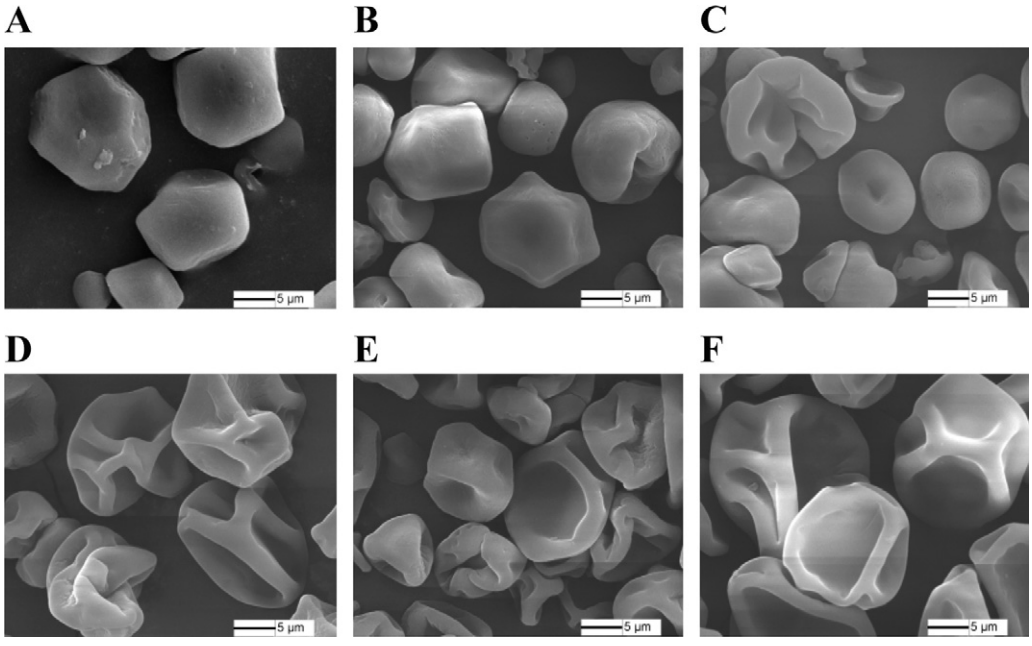

Fig. 1. SEM microphotographs of partially gelatinized starch as affected by the gelatinization process (3000×): (A) native corn starch; (B) *S64*; (C) *S66*; (D) *S68*; (E) *S69*; and (F) *S70*.

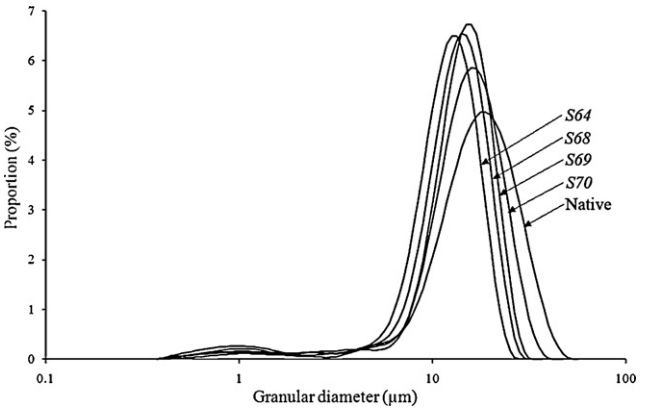

Fig. 2. Granular size distribution of partially gelatinized starch and native corn starch.

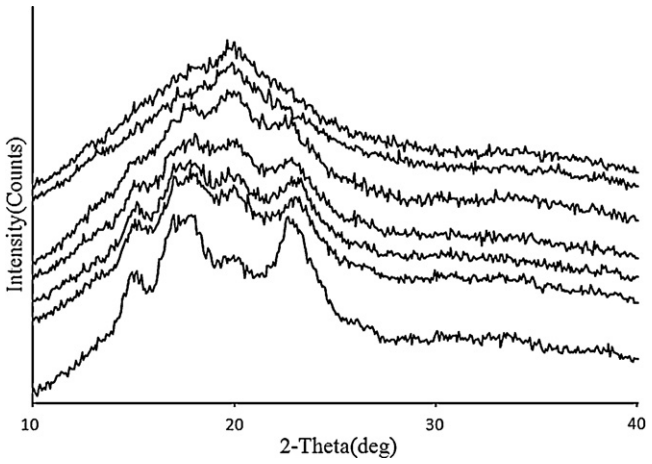

Fig. 3. X-ray diffraction patterns of partially gelatinized starch and native corn starch (native corn starch, *S64*, *S66*, *S68*, *S69*, *S70* and *S72* from bottom to up).

between particle surface and its core. This leads to formation of various morphological shapes such as deviation from sphericity to formation of surface folds and troughs that greatly facilitates the outward diffusion of water. This explains why the formation of surface folds and wrinkles takes place in spray dried particles of fully gelatinized starch solutions.

3.2. Granule size distribution

The size distribution of corn starch granules are shown in Fig. 2 and Table 1. The size distribution plots of *S66* and *S72* were similar to *S64* and *S70*, respectively and are not presented in Fig. 2.

As can be seen in Table 1, the median diameter of native corn starch is larger than the median diameter of gelatinized and subsequently spray dried corn starch samples. And, the size distribution of native corn starch is broader than that of spray dried samples (Fig. 2). As can be seen from Table 1, the median diameter of gelatinized and subsequently spray dried starch has increased with increase in gelatinization temperature from 64 °C to 70 °C and decreased slightly as the gelatinization temperature increased from 70 °C to 72 °C. The broadening of the particle size distribution of gelatinized starch granules increased as the gelatinization temperature increased (Fig. 2).

There are two reasons which might have caused the decrease in the median diameter of spray dried corn starch granules as a function of gelatinization temperature of the feed. Firstly, the native corn starch used in these tests contained 10% (w/w) moisture and this level of moisture content helped maintaining the integrity of starch granules. When such starch slurry was spray dried, the residue moisture content of starch granules transferred out which made the granules shrink that led to the decrease in the size of the granules. Secondly, the starch granules swelled and lost their birefringence when the starch was heated in excess water. This was generally followed by a release of amylose from the granule structure in the surrounding solution (Koganti, Mitchell, Ibbett, & Foster, 2011). The mass of starch granules decreased because of the release of amylose which resulted in the reduction of median diameter when the gelatinized starch solution was spray dried. It is generally established that large starch granules swell first during the gelatinization process. The small granules do not start swelling until the temperature is higher or heating time is longer. Increasingly large number of granules started to swell and their size of swollen granules become larger when the gelatinization temperature was increased from 64 °C to 70 °C. However, the structure of starch granules becomes frangible and large granules disintegrate when the heating temperature is increased to 72 °C. This explains why the median diameter of starch granules increased first and then decreased when the temperature increased from 64 °C to 72 °C.

3.3. X-ray diffraction (XRD) analysis

X-ray diffracograms of spray dried starch particles as a function of gelatinization temperature are presented in Fig. 3. The native starches possess two types of crystallinity, an A-type, prevailing in cereal starches (except high amylose variety), and B-type prevailing in root and tuber starches (Aggarwal & Dollimore, 1998). The occurrences of peaks at $2\theta = 15°, 17°, 18°$ and $23°$ suggested that the crystalline structure of native corn starch follows A-type pattern (Ferrini, Rocha, Demiate, & Franco, 2008). As shown in Fig. 3, this crystalline pattern was not altered and only the peak values were gradually decreased as gelatinization temperature was increased. When the gelatinization temperature was above 70 °C, the peaks occurring at $2\theta = 15°, 17°, 18°$ and $23°$ disappeared. This observation suggested that the crystalline native corn starch was converted largely into non-crystalline form.

A native starch is a mixture of amylose (a linear structure of α-1, 4 linked glucose units) and amylopectin (a highly branched structure of short α-1, 4 chains linked with by α-1, 6 bonds). Starch granules have a layered organization with alternating amorphous and semi-crystalline regions. This layered organization manifests itself in a hierarchical structural periodicity in native starch granules (Blazek et al., 2009). For amylopectin-rich starches it is understood that the origin of crystallinity is due to the intertwining of the outer chains of amylopectin (exterior or external chains, representing A- and B_1 type) in the form of double helices. These associate together to form more or less ordered arrays where the ordered structures are crystalline entities (Tester, Karkalas & Qi, 2004). When starch is heated in excess water, the order-to-disorder transition is definitely not a sudden or "quick" process that takes place within a narrow temperature range. During the first portion of the phase transition, water absorbed by starch granules increases the mobility of starch polymers (Ratnayake & Jackson, 2007). Upon further heating, at increased temperature, starch polymers become more mobile, reduce or lose their intermolecular interactions which induce the disintegration of ordered structure. When the gelatinized starch solution is dried using a very rapid drying process such as spray drying in which the drying time is much shorter than the time required for crystallization, particles or granules with little crystal are obtained. Due to these reasons, the peak value of crystalline pattern decreased progressively from *S64* to *S70*.

Table 1
Granule size (diameter) of partially gelatinized starch and native corn starch as a function of gelatinization temperature.

Starch	Granule size distribution (%)							Median diameter (μm)
	0–2 μm	2–5 μm	5–10 μm	10–15 μm	15–20 μm	20–30 μm	30–60 μm	
Native	3.70	1.59	8.78	26.59	24.42	26.93	7.99	17.25
S64	5.05	2.14	22.04	48.17	19.40	3.20	–	12.01
S66	4.22	2.15	19.69	47.90	21.61	4.43	–	12.45
S68	3.34	2.27	15.30	44.87	25.94	8.28	–	13.41
S69	2.99	2.13	10.75	41.52	29.84	12.75	0.02	14.48
S70	2.10	2.50	9.76	35.57	27.96	20.45	1.66	15.43
S72	2.03	2.62	11.43	36.33	27.24	19.06	1.29	15.05

Values are the means of duplicate. "–" mean no data.

Table 2
Swelling power (SP) of partially gelatinized starch and native corn starch.

Starch	Temperature (°C)					
	30	50	60	70	80	90
Native	$2.19 \pm 0.14^{A,a}$	$2.09 \pm 0.03^{A,a}$	$2.54 \pm 0.04^{A,a}$	$10.17 \pm 0.40^{B,b}$	$12.47 \pm 0.19^{C,c}$	$15.04 \pm 0.67^{D,c}$
S64	$3.69 \pm 0.04^{A,b}$	$3.82 \pm 0.01^{A,b}$	$4.27 \pm 0.01^{B,b}$	$9.13 \pm 0.50^{C,a}$	$10.80 \pm 0.16^{D,ab}$	$11.91 \pm 0.20^{E,ab}$
S66	$5.31 \pm 0.06^{A,c}$	$5.43 \pm 0.04^{A,c}$	$5.61 \pm 0.05^{A,c}$	$9.08 \pm 0.55^{B,a}$	$10.37 \pm 0.19^{C,ab}$	$12.01 \pm 0.09^{D,ab}$
S68	$5.36 \pm 0.03^{A,c}$	$5.56 \pm 0.07^{AB,d}$	$5.87 \pm 0.11^{B,c}$	$9.11 \pm 0.43^{C,a}$	$10.52 \pm 0.42^{D,ab}$	$11.98 \pm 0.07^{E,ab}$
S69	$6.20 \pm 0.04^{A,d}$	$6.29 \pm 0.06^{A,e}$	$6.60 \pm 0.07^{B,d}$	$9.23 \pm 0.24^{C,a}$	$10.64 \pm 0.11^{D,a}$	$11.80 \pm 0.13^{E,a}$
S70	$7.41 \pm 0.09^{A,e}$	$7.38 \pm 0.06^{A,f}$	$7.65 \pm 0.09^{A,e}$	$9.12 \pm 0.09^{B,a}$	$11.08 \pm 0.48^{C,ab}$	$12.00 \pm 0.33^{D,ab}$
S72	$8.94 \pm 0.21^{A,f}$	$9.12 \pm 0.03^{A,g}$	$9.70 \pm 0.46^{B,f}$	$10.20 \pm 0.04^{C,b}$	$11.12 \pm 0.02^{D,b}$	$12.43 \pm 0.16^{E,b}$

Values are mean ± standard deviation ($n = 3$). Values in the same line followed by different capital letters differ significantly ($p < 0.05$); values in the same column followed by different lowercase letters differ significantly ($p < 0.05$).

3.4. Swelling power (SP)

The data on the SP of starch granules are presented in Table 2. When the heating temperature was increased from 30 °C to 50 °C, there was no significant difference ($p > 0.05$) on the SP of each sample. When the heating temperature was above 60 °C, the SP of each sample increased significantly ($p < 0.05$). It can be seen from Table 2 that the variation in SP values among the samples is quite remarkable when the heating temperature was below 60 °C. But when the heating temperature ranged from 70 °C to 90 °C, there was no significant difference ($p > 0.05$) in SP values among the samples except native corn starch and *S72*. In addition, it can be seen that the SP of gelatinized samples was higher than that of native corn starch at low temperature (30 °C). However, the SP of native corn starch increased very rapidly when the heating temperature was increased from 60 °C to 70 °C. At 90 °C, the SP value of native corn starch was found to be 15.04 ± 0.67 which is much higher than the SP values of gelatinized samples.

When starch is heated in excess water, its crystalline structure is disrupted due to breakage of intra-molecular and inter-molecular hydrogen bonds. Subsequently the water molecules form hydrogen bonding with exposed hydroxyl groups of amylose and amylopectin which results into increase in granule swelling. Swelling power is significantly influenced by the magnitude of interaction between starch chains within the amorphous and crystalline domains. The extent of this interaction is determined by the amylose/amylopectin ratio and the characteristics of amylose and amylopectin in terms of molecular weight/distribution, degree and length of branching, and conformation (Hoover, 2001). The strength of bonding within the starch granules plays an important role in determining the extent to which the starch granules are capable of swelling (Lee, Kumar, Rozman, & Azemi, 2005). The ordered structure and the rigidity of native starch granules are disrupted by the gelatinization process. The water molecules can diffuse into previously gelatinized starch granules easier than native starch. This is the main reason why the SP of previously gelatinized starch granules was higher at low temperature. The gelatinized starch granules have gone through both the gelatinization and spray drying processes. As a consequence, their structure is weaker because of the disappearance of crystalline domain. When heated at high temperatures (60 °C and above), solid content from previously gelatinized starch granules leach out easily. In addition, the large granules of previously gelatinized starch tend to break into small pieces with relative ease. From particle size distribution data, we also observe that the median diameter of previously gelatinized starch granules is smaller than that of native starch. Smaller granules could not hold more water which also contributed to the decrease in SP value.

3.5. Gelatinization behavior of partially gelatinized starch

The gelatinization behavior of all partially gelatinized starch and native corn starch obtained through DSC thermograms is presented in Fig. 4. The gelatinization temperatures (onset, T_o; peak, T_p; and end, T_e), enthalpy of gelatinization (ΔH_{gel}) and the degree of gelatinization (GD) are presented in Table 3. The T_o, T_p and T_e of the partially gelatinized starch increased significantly as the temperature used in the gelatinization process increased with the highest value observed at 72 °C. The ΔH_{gel} of the partially gelatinized starch decreased from 12.97 ± 0.28 to 0.51 ± 0.08 J/g when the temperatures used for the gelatinization process was increased up to 70 °C. However, there is no significant difference on the T_o, T_p, T_e and ΔH_{gel} between *S70* and *S72*. The degree of gelatinization (GD) of

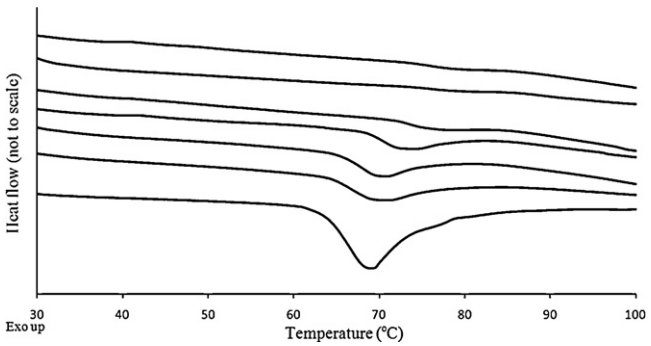

Fig. 4. DSC thermograms of partially gelatinized starch and native corn starch (native corn starch, *S64*, *S66*, *S68*, *S69*, *S70* and *S72* from bottom to up).

Table 3
The gelatinization properties of partially gelatinized starch and native corn starch measured by DSC.

Sample	T_o (°C)	T_p (°C)	T_e (°C)	ΔH_{gel} (J/g)	GD (%)
Native	63.82 ± 0.20[a]	68.76 ± 0.11[a]	73.86 ± 0.78[a]	12.97 ± 0.28[e]	0
S64	64.23 ± 0.21[b]	69.71 ± 0.09[c]	78.67 ± 0.05[b]	6.77 ± 0.11[d]	47.75 ± 1.04[a]
S66	64.45 ± 0.07[b]	69.28 ± 0.21[b]	78.63 ± 0.16[b]	6.92 ± 0.09[d]	46.59 ± 1.43[a]
S68	68.64 ± 0.22[c]	72.53 ± 0.40[d]	80.96 ± 0.22[c]	3.96 ± 0.16[c]	69.40 ± 0.38[b]
S69	71.71 ± 0.13[d]	76.94 ± 0.08[e]	85.09 ± 0.16[d]	1.29 ± 0.11[b]	90.01 ± 0.23[c]
S70	73.84 ± 0.06[e]	78.76 ± 0.21[f]	86.27 ± 0.16[e]	0.42 ± 0.05[a]	96.78 ± 0.09[d]
S72	73.70 ± 0.27[e]	78.82 ± 0.14[f]	86.77 ± 0.12[e]	0.51 ± 0.08[a]	96.08 ± 0.13[d]

Values are mean ± standard deviation ($n = 3$). Different letters within the same column differ significantly ($p < 0.05$).

starch treated at 70 °C was 96.78 ± 0.09% which indicated that a complete gelatinization of the starch sample used in this study takes place at 70 °C.

The melting temperature of starch granules depends on structural organization of the amylopectin clusters. Thickness of crystals, their polymorphic structure and free energy of surface of face side significantly affect the melting temperature of starch granules (Błaszczak et al., 2007). In this study, the melting temperature of partially gelatinized corn starch was higher than that of native corn starch. Similar observations have been reported by Laovachirasuwan et al. (2010). These authors suggested that the increase in melting temperature could be due to the colloidal molecular structure of starch granules, amylopectin chain length and reordering of the crystalline structure after hydrolysis. In this study, the gelatinized starch granules were obtained through spray drying using relative high temperatures (inlet temperature = 200 °C, outlet temperature about 110 °C) which could also have affected the melting temperature of the partially gelatinized starch (Altay & Gunasekaran, 2006). The gelatinization process of starch acts to destabilize the amylopectin crystals within the crystalline lamellae, which are ripped apart (smaller crystallites are destroyed first). Hence, the ΔH_{gel} of starch is attributed to disordering of amylopectin crystallites (Tester & Morrison, 1990). From the results obtained from X-ray diffraction, it can be observed that the extent of crystallinity decreased with increasing the gelatinization temperature (Fig. 3). As a result, the ΔH_{gel} of gelatinized and subsequently spray dried partially gelatinized starch is less than that of native corn starch.

4. Conclusions

In this study, the physical properties of partially gelatinized starch obtained by gelatinization followed by spray drying were investigated. The shape of the partially gelatinized starch was truncated with concave surface troughs and the surface morphology of the granules was shriveled with many surface folds. The median diameter of the partially gelatinized starch was smaller than that of its corresponding native form. The partially gelatinized starch gelatinized at 70–72 °C had almost no crystalline domain in their structure. The swelling power (SP) of native starch was higher than that of partially gelatinized starch above 60 °C. However, the SP of partially gelatinized starch was much higher compared to that of the native corn starch below 60 °C suggesting the partially gelatinized starch had better hydration at the low temperature. The differential scanning calorimetric analysis of partially gelatinized starch showed a distinct increase in gelatinization temperatures (T_o, T_p and T_c) and a decrease in ΔH_{gel}. The granule size distribution of partially gelatinized starch increased with increase in the gelatinization temperature within 64–72 °C.

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