Journal of Food Engineering 152 (2015) 65–71

Contents lists available at ScienceDirect

Journal of Food Engineering

journal homepage: www.elsevier.com/locate/jfoodeng

Loss of birefringence and swelling behavior in native starch granules: Microstructural and thermal properties

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ARTICLE INFO

Article history:
Received 11 July 2014
Received in revised form 28 October 2014
Accepted 2 November 2014
Available online 10 December 2014

Keywords:
Starch gelatinization
Degree of gelatinization
Birefringence

ABSTRACT

Starch granules imbibe water and swell when exposed to abundant water and temperatures above their gelatinization point. The degree of gelatinization of four native starches, wheat, potato, cassava and corn, was determined by the enthalpic transitions and simultaneous events between loss of birefringence and swelling to quantify the process *in situ* and in real time. In all cases, the following three stages were identified: low granular swelling, with little water absorption and 100% birefringence; gradual leading to complete loss of birefringence with the absorption of a large amount of water (approximately 50%); and complete granular swelling to equilibrium. A clear gap between the beginning and end of the loss of birefringence and swelling was observed. When the birefringence reached zero value, 50% swelling was reached at 55.7, 62.0, 68.7 and 70.6 °C for wheat, potato, cassava and corn, respectively. A good correlation between the degree of gelatinization measured by differential scanning calorimeter and microscopy was found for potato, corn and cassava with r^2 values of 0.98, 0.99 and 0.98, respectively, and an r^2 of 0.70 for wheat. Therefore, the loss of birefringence and swelling does not characterize gelatinization in equivalent ways.

1. Introduction

Starch represents between 70% and 80% of the calories consumed by people worldwide (Bertolini, 2010b), and it is one of the primary components in several foods because it is responsible for many desirable functional and nutritional properties in the food and other industries. Starches from different botanical sources vary in composition, morphology, molecular structure as well as in arrangement and content of amylose and amylopectin (Hoover, 2010). The content of amylopectin has been suggested as responsible for granule swelling while the presence of an amylose–lipid complex may retard it (Schirmer et al., 2013). Starches from cereals are known to contain higher quantities of lipids complexed with amylose, while most starches from tubers have little or no lipids (Tester et al., 2004). Most of cereal starches show A-type crystallinity, whereas tuber starches shows B-type X-ray diffraction patterns (Tester et al., 2004). Starch is a widely studied food material, and the characterization of its granules and gelatinization has been studied with atomic force microscopy (Simao and Cordenunsi, 2010). Its functional properties have been assessed through thermal rheological analysis (Kim et al., 2012; Mishra and Rai, 2006) and scanning electron microscopy (Jane et al., 1994). The swelling and water absorption properties of starch have also been evaluated (Choi and Kerr, 2004; Hellman et al., 1952). Starch has many roles in the food industry. For example, it has been used as adhesive, binder, emulsion stabilizer, encapsulation agent, expansion agent, fat replacement, foam stabilizer and thickening agent (Mason, 2009). The gelatinization process is a primary issue in food processing. The starch gelatinization process has been studied in different food matrices using modern techniques, such as enthalpic transitions (Ratnayake et al., 2009), crystallinity disruption patterns (Cai and Wei, 2013) and behavior with isolated components (Beleia et al., 1996; Biliaderis, 1991; Kaur et al., 2008; Zhou et al., 2008). The gelatinization process occurs when starch is heated

Abbreviations: T_{gel}, gelatinization temperature; W_s, weight of sediment; W_l, weight of the dried supernatant; WSI, water soluble index; SP, swelling power; SEM, scanning electron microscopy; $T_{o\text{-}m}$, onset temperature measured by hot-stage and image analysis; $T_{p\text{-}m}$, peak temperature by hot-stage and image analysis; DG_m, degree of gelatinization by microscopy; DSC, differential scanning calorimeter; $T_{o\text{-}D}$, onset temperature measured by DSC; $T_{p\text{-}D}$, peak temperature measured by DSC; ΔH, heat of gelatinization; DG_D, degree of gelatinization by DSC; r^2, correlation coefficients; SPP, swelling power progression; T_o, onset temperature; T_p, peak temperature; T_e, endset temperature; T/T_p, relationship between a determined temperature and peak temperature.

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http://dx.doi.org/10.1016/j.jfoodeng.2014.11.017
0260-8774/

above its gelatinization point in the presence of excess water, which disrupts the native crystalline structure (Parker and Ring, 2001). Biliaderis (2009) reported that when the granules are heated progressively to higher temperatures in the presence of excess of water, there is a point at which the Maltese cross of the native granules disappears and the granules begin to irreversibly swell. Colonna and Buleon (2010) describe the gelatinization as several events that occur simultaneously, and one event is the predominant swelling of the granule after the loss of birefringence. Singh et al. (2004) report that starch swelling occurs concomitantly with loss of birefringence and precedes solubilization. Ratnayake et al. (2009) identified the following three distinct stages in the gelatinization process: (i) granular swelling by slow water absorption; (ii) followed by a rapid loss of birefringence via the absorption of large amounts of water by the granules; and (iii) finally, leaching of the soluble portion into the solution, transforming the granules into formless sacs.

The gelatinization process has been characterized by many chemical, physical and enzymatic methods, but there is not clear consensus as to when the process starts and ends. Currently, several physical properties, such as the loss of birefringence and swelling, are described as separate events. Therefore, it is necessary to implement methodologies that allow for *in situ* and real time observation to monitor the gelatinization process under controlled conditions that allow for the understanding of the importance of the degree of gelatinization. The objective of this study was to identify the morphological and thermal changes during gelatinization of four of the most important starches in foods (two from tubers and two from grains), all of them widely used in the food industry.

2. Materials and methods

2.1. Materials

Four native food grade starches were used in this study. Wheat, potato, corn and cassava native starch were purchased from the local market.

2.2. Swelling power progression (%)

Swelling power progression was determined in triplicate using the modified method proposed by Li and Yeh (2001). Nine samples of 0.5 g each starch were weighed into a centrifuge tube with a screw cap. Distilled water (10 ml) was added and the tubes were heated at 55, 65, 75, 85 and 95 °C in a shaking water bath. At 10, 20 and 30 min, each set of three tubes was cooled in an ice water bath until they reached room temperature (20 °C) and then centrifuged at 8000*g* for 20 min. The supernatant was decanted from each tube and the weight of the sediment was recorded (W_s). The weight of the dried supernatant at constant weight was also measured (W_l). The water soluble index (WSI) and swelling power (SP) were calculated using the following equations:

$$\mathrm{WSI} = \frac{W_l}{0.5} \times 100 \quad (1)$$

$$\mathrm{SP} = \frac{W_s}{0.5(100 - \mathrm{WSI})} \quad (2)$$

2.3. Morphology and particle size distribution

The morphology of the four native starches was qualitatively assessed using scanning electron microscopy (SEM). Corn, potato, wheat and cassava native starches were sputter coated with gold and examined with a LEO 1420 VP scanning electron microscope (Cambridge, UK), operated at an acceleration voltage of 25 kV. The images were analyzed with the software LEO 32 (Soft Imaging System GmbH 1986–2003, Münster, Germany).

The particle size distribution was measured in sextuple using a Malvern Mastersizer 2000 laser diffraction particle-size analyzer (Malvern, Worcestershire, UK) with distilled water at 18 °C as a solvent according to the method of Stoddard (1999). The particle size distribution and average granule diameter were calculated using the software Hydro Application 5.60 (Malvern, Worcestershire, UK.).

2.4. Hot-stage light microscopy and the progression of the gelatinization process

The progression of the gelatinization process was determined for the four starches as parallel events of the loss of birefringence and swelling using image analysis. Starch granule suspensions were prepared prior to observation by resuspending 1 mg each starch in 1 ml distilled water using a vortex mixer. The suspensions were equilibrated for 2 h, and several drops were deposited in a concave slide and covered with a coverslip. Each specimen was observed under normal and polarized light. To determine the progression of the process, a Hot-stage Linkam (model THMS350V, Linkam Scientific Instruments, Surrey, UK) adapted to an Olympus BX61 light microscope (Olympus Optical Corporation, Tokyo, Japan) equipped with polarized filter and analyzer was used. The hot-stage is also equipped with a temperature controller (PE95/T95 System controller), which allows for heating from 20 to 80 °C at rate of 2 °C/min. A sequence of images were captured with a digital camera (Cool Snap Pro Colour, Photometrics Roper Division, Inc., Tucson, AZ, USA) every 2 min between the temperatures T_{gel} −10 °C and T_{gel} +10 °C.

Based on Ovalle et al. (2013), after image acquisition each image was processed by enhancement and filtering, where the contrast and brightness were adjusted. Then the noise was reduced by applying a median filter and the borders were enhanced using a sharpening filter with the Image Pro Plus 4.5 Program (Media Cybernetics, Inc.). Subsequently, the images were segmented selecting the threshold manually in order to binarize (black and white) the images. Thereafter, to each binarized granule the Feret diameter was measured in order to identify and count each one. Finally, the area of the granules was obtained. The onset temperature ($T_{o\text{-}m}$) at which the Maltese cross starts to disappear, and the peak temperature ($T_{p\text{-}m}$) at which the granule stops swelling and remains in equilibrium were determined. Next, the birefringence area and the total area of the granules in pixels were calculated. The degree of gelatinization by microscopy (DG_m) was determined as the percentage of birefringence in pixels reached from T_o to a specific temperature.

2.5. Thermal properties

The thermal characteristics of each starch were studied in triplicate using a Mettler Toledo Star System 821e differential scanning calorimeter (DSC) at heating rate of 25–90 °C at 2 °C/min to determine the thermal transitions. Approximately 10 mg of each starch solution (10% starch and 90% distilled water) was loaded into a 160 μL capacity aluminum pan. The samples were hermetically sealed and each specimen was equilibrated for 2 h at 20 °C for prior analysis. An empty crucible was used as the reference. The analyses were performed under a nitrogen atmosphere. The onset temperature ($T_{o\text{-}D}$), peak temperature ($T_{p\text{-}D}$) and heat of gelatinization (ΔH) were determined using the data analysis system software $Star_e$. The degree of gelatinization by DSC (DG_D) was

calculated according to the method of Li et al. (2013) as the percentage of area integrated from T_o a specific temperature (3).

$$DG_D(\%) = \int_{t_0}^{t} f(x)dx \quad (3)$$

2.6. Statistical analysis

Regression analysis was used to establish the relationship between the degree of gelatinization determined by DSC and degree of gelatinization determined by image analysis determining their correlation coefficients r^2. The results were calculated and analyzed by Excel software and each curve was fitted by Sigma Plot 11.0 software.

3. Results and discussion

3.1. Swelling power progression SPP (%)

The onset of swelling is an indicator of the water absorption index of the granules during heating. Native starch granules are insoluble in cold water, but when the granules are heated in the presence of excess water, the granules absorb water and swell (Cai and Wei, 2013). This behavior has been described as the loss of radial organization of amylopectin and amylose chains (Colonna and Buleon, 2010), in which an irreversible disruption of the molecular order within starch granules occurs. As described in the literature, the swelling power of starches of different origins increases with increasing temperature (Li and Yeh, 2001), and the differences in the swelling of native starches is primarily attributed to the granule size, crystallinity and amylose–lipid complex content in presence of sufficient water (Bertolini, 2010a). Fig. 1 shows the swelling power of the native starches at different constant temperatures. To quantify the water uptake, the starches were subjected to temperatures between $T_{gel} \pm 10$ °C. All starches continue absorbing water until equilibrium is reached after passing through the gelatinization temperature and reaching the peak gelatinization temperature. This behavior was described by Colonna and Buleon (2010), and it includes the predominant swelling of the granule after the loss of birefringence. Consistent with Singh et al. (2003) and Li and Yeh (2001), potato starch has greater swelling power compared with corn, wheat and cassava starch, but all continue absorbing water above the T_p. For the four starches evaluated here, maximum swelling is observed closer to the gelatinization temperature, and this behavior was also reported by French et al. (1950), who reported that this is the stage at which the absorption of large quantities of water occurs, but that swelling does not stop at this point. Rather, it continues until equilibrium.

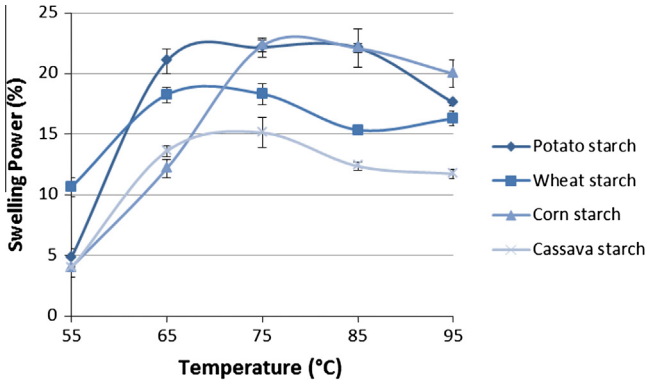


Fig. 1. Swelling power progression of different native starches.

This behavior correlates well with T/T_p for the four starches, but only between the $T_{gel} \pm 10$ °C. Above this temperature, the relationship between swelling and T/T_p does not characterize this behavior. As described by Li and Yeh (2001) for the temperature effect, T_p is a good reference point because T_p is a critical point for the disruption of starch granules.

3.2. Morphology and particle size distribution

Fig. 2 shows the SEM images of the size distribution and the average granule size for the four native starches. As shown in Fig. 2a, corn starch has a polyhedral shape, and cassava (Fig. 2d) is spherical with some shortened granules as previously reported by Mishra and Rai (2006). Fig. 2b is the potato starch, which has two shapes, spherical and oval, as reported by Jane (2009). Consistent with Patel and Seetharaman (2006), the wheat native starch has two types of granules and a bimodal size distribution, a large lenticular shape and small spherical shape (Fig. 2c). This behavior is also shown in the particle size distribution graph. The average diameters of the starch granules range between 15 and 45 μm, with potato starch as the largest and corn and cassava as the smallest, which is consistent with Jane (2009). As reported by Li and Yeh (2001), the gelatinization behavior of starch is not necessarily a function of granule size. In this study, cassava had the smallest size, but in proportion to its size, cassava has the highest increase in area during the gelatinization process. Apparently, the granule size and morphology do not correlate with the increase in size and the changes in morphology during swelling.

3.3. Hot-stage light microscopy – gelatinization process progression

Changes in the starch granule morphology (granular swelling and crystallinity disappearance) and size during heating were analyzed. During gelatinization, the physical changes that occur in the granules are mainly microstructural. The mechanism by which this occurs has been reported by many authors. During gelatinization, the birefringence gradually disappear when the temperature is increased to the $T_{p\text{-}m}$ (Biliaderis, 2009; Colonna and Buleon, 2010), and the total area increases until equilibrium and uniform granule size is reached (Patel and Seetharaman, 2006). With increasing temperature in excess water, the native granules begin swelling almost simultaneously with the gradual disappearance of birefringence, the disruption of the crystalline structure and swelling of disrupted areas can be evidently seen (Cai and Wei (2013). Fig. 3 represents the gelatinization process expressed in the area of the loss of birefringence and the increase in the total area of the native granules (area birefringence/total area). Both phenomena, the loss of birefringence and the increase in total area begin almost simultaneously in all the starches, but the onset temperatures and the slope for the starches is different, while to cassava starch the slope for increase in area is the higher, to corn is the lower. The same behavior was observed to the slope for loss of birefringence; therefore at the same heating rate cassava starch gelatinize before than the others. This behavior has been suggested by Cai and Wei (2013) as an event during gelatinization at which point the loss of birefringence occurs and swelling is accompanied by rupture of the granule. Furthermore, Biliaderis (2009) suggested that swelling usually begins at a temperature corresponding to the onset temperature of the DSC endothermic transition. This is consistent for each starch (Table 1), and the temperatures T_o that are measured using DSC are close to the T_o measured using the hot-stage to the initiation of swelling. In this study, for the four native starches, a clear gap between the start and end of the loss of birefringence and swelling was observed. In the first period of gelatinization process the area of birefringence of the native granules decreased more rapidly than swelling and disappears completely

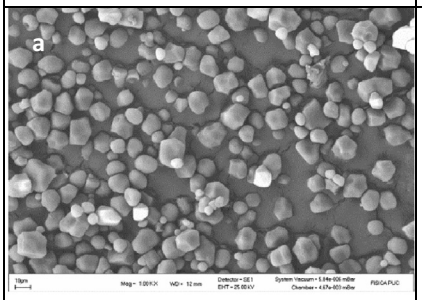
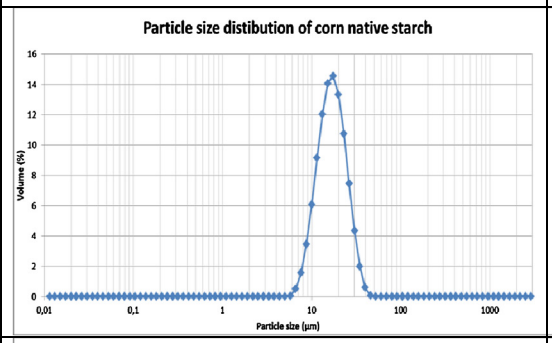
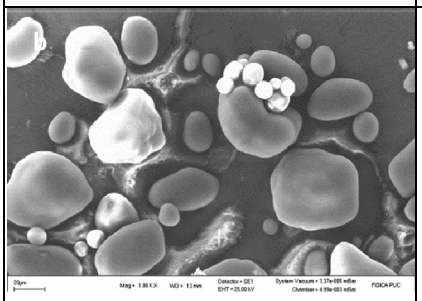
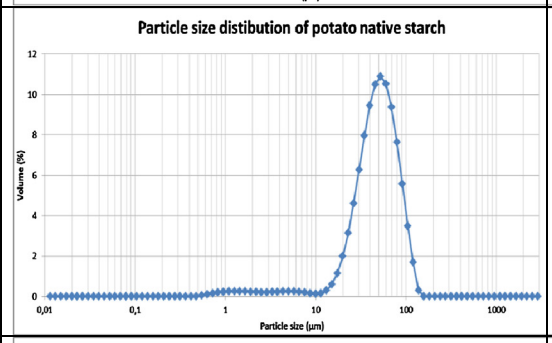
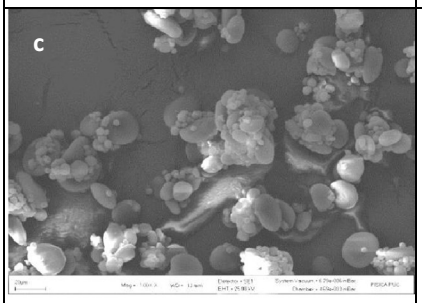
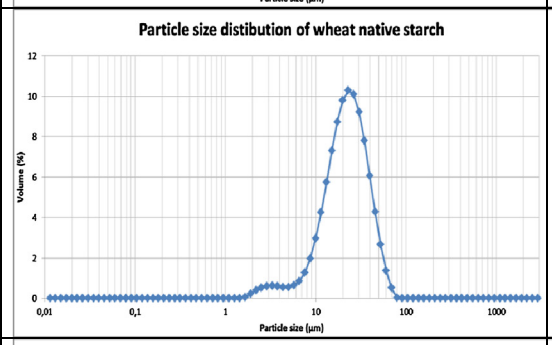
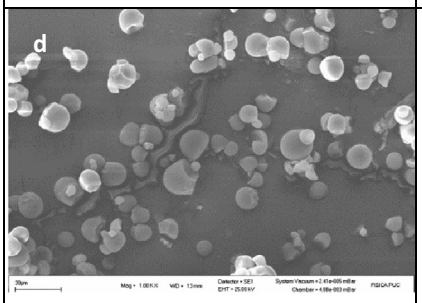
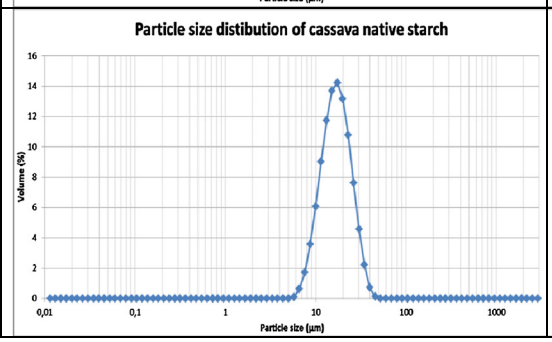
SEM Images	Particle size distribution	Granule size (μm)		
		d(0.1)	d(0.5)	d(0.9)
		8.35	15.44	24.72
		21.15	44.98	80.68
		4.44	20.07	39.08
		9.49	15.42	24.59

Fig. 2. Morphology and particle size distribution.

while swelling continues. During a second period of gelatinization, specific events occur slightly after the gelatinization temperature, the swelling and birefringence have different behaviors. The birefringence area set to zero after a few seconds, while swelling (total area of granule) continues increasing for several seconds until reach the equilibrium (Fig. 3).

Therefore, loss of birefringence and swelling characterize gelatinization differently. The birefringence end point is the temperature at which 98% of the granules have lost their birefringence (Biliaderis, 2009). The $T_{o\text{-}m}$ observed using this methodology were slightly different than the $T_{o\text{-}D}$ observed by DSC. These differences were explained by Biliaderis (2009) and occur because within a given starch granule population, the T_o of individual granules may span 10–15 °C.

Parker and Ring (2001) suggested that in starch suspensions <10% w/w granules swell irreversibly to many times their original size when heated above T_{gel}. Comparatively, and expressed in increase in area, the total increase was 5.86, 4.41, 4.55 and 4.82 times its original size for cassava, corn, potato and wheat starch, respectively. Consistent with Ratnayake et al. (2009), the following three observations were done during heating of the four types of starch:

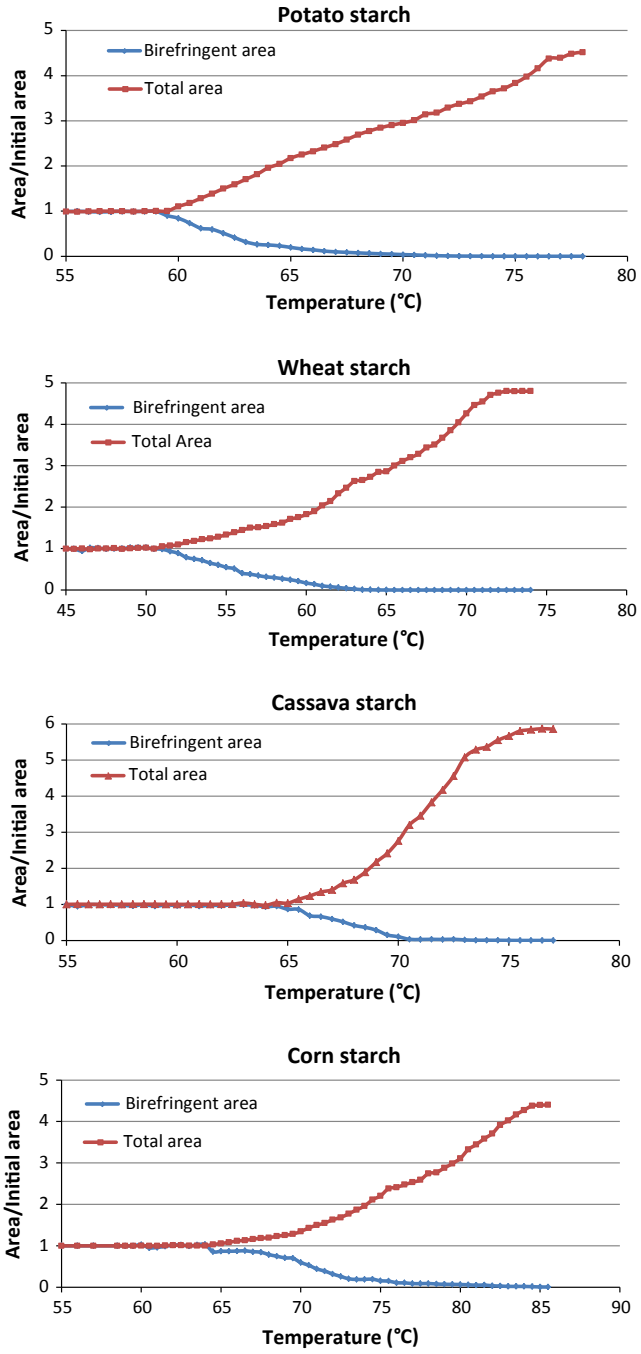


Fig. 3. Gelatinization process expressed in area.

1. Both, loss of birefringence and swelling started at the same temperature (e.g., T_{gel} onset) that was different for each starch source (see Table 1). It is clear that the birefringence and swelling take different paths, it mean, the Maltesse disappear gradually and birefringence goes to 0, while swelling begin to increase.
2. From that point on the rate of loss of birefringence varied between the sources of starch. This point it characterize by the disappearance of Maltesse cross and total loss of birefringence. In this phase apparently about of 50% swelling is reached in all of the starches such as increase of area and still increasing while birefringence set to 0.
3. Even when birefringence had been completely lost, the granules continuing absorbing water to its full capacity until a characteristic asymptotic or equilibrium value is reached for each type of starch. Immediately after reaching equilibrium, the granules leach the soluble portion into the solution and transform into formless sacs.

This study shows that both events, the loss of birefringence and swelling (expressed as increase in area) begin almost simultaneously, with only a small difference in temperature (0.5–3 °C). However, after several seconds, they show different behaviors. This is consistent with Goering et al. (1974) who showed that the birefringence measurement did not fully reveal starch gelatinization-related structural changes.

Notably, using this method, the 50% water absorption measured as the percentage of the increase of total area was reached at 68.5, 77.55, 65.1 and 70.9 °C for potato, corn, wheat and cassava, respectively. Surprisingly, this is close to the temperature at which the birefringence is zero. Based in these observations, potato starch has the highest swelling power in combination with the lower temperature at which the 50% water absorption is reached; therefore, this starch may eventually be used in food models as a water absorber.

The degree of gelatinization by microscopy (DG_m) was calculated as the percentage of loss of birefringence in pixels from the T_o to a specific temperature, consistent with the modified method proposed by Li et al. (2013). As shown in Fig. 4a, the 100% DG_m was reached at 69.5, 76.5, 62.5 and 70.5 °C for potato, corn, wheat and cassava, respectively, as measured by image analysis.

3.4. Thermal properties

The thermograms, enthalpies and transition temperatures were obtained using DSC. The gelatinization of the starches begins at onset temperatures of 61.88, 66.18, 56.37 and 63.70 °C for potato, corn, wheat and cassava, respectively. Integration of the endothermic peaks was used to obtain the respective enthalpies of gelatinization (Table 1). The degree of gelatinization (DG_D) values were determined as the percentage of area integrated from an initial temperature to a specific temperature with respect to the total area of the endothermic peak for each starch sample (Fig. 4b). At 60 °C, the potato, cassava and corn starch have not begun to gelatinize, whereas the wheat starch shows 32.5% gelatinization. Additionally, the 50% gelatinization was reached at 61.6, 65.2, 67.7 and 70.3 °C for wheat, potato, cassava and corn, respectively. Wheat

Table 1
Enthalpy of gelatinization and transition temperatures of different starch samples.

Starch	Gelatinization enthalpy (J/g)	T_o (°C)	T_p (°C)	T_e (°C)	T_o (°C)	T_p (°C)[a]	T_e (°C)[b]
	Differential scanning calorimetric				Hot-stage and image analysis		
Corn	4.28	66.18	70.31	77.12	64.50	76.50	84.5
Wheat	4.55	56.37	61.40	67.25	52.50	62.50	72.5
Potato	9.04	61.88	64.95	68.84	59.50	69.50	76.5
Cassava	10.68	63.70	68.69	73.84	64.50	70.50	75.5

[a] Temperature measured considering 98% loss of birefringence.
[b] Temperature measured considering the first temperature when the equilibrium was reached in increase total area.

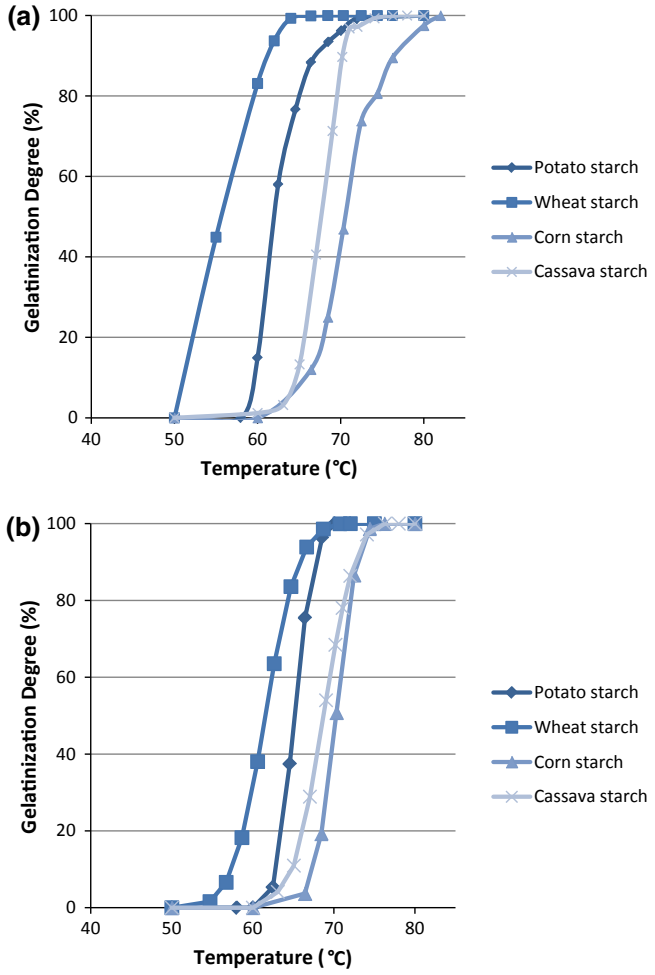


Fig. 4. Gelatinization degree by image analysis and DSC. Gelatinization degree (a) measured by image analysis (microscopy) and (b) measured by DSC.

starch starts to gelatinize before the others starches, requires a lower temperature, while corn starch requires a higher temperature to reach 50% gelatinization. For the DG_m, the criteria used to define the end of gelatinization was 98% loss of birefringence. Therefore, the temperatures to reach 50% of gelatinization measured by image analysis were slightly lower to potato (62.0 °C) and wheat (55.7 °C) and slightly higher to cassava (68.7 °C) and corn (70.6 °C) compared with the temperatures measured by DSC. Different endothermic peak produced from starch gelatinization were observed at 70.31, 68.69, 64.95 and 61.40 °C for corn, cassava, potato and wheat starch, respectively. Each endothermic peak represents the gelatinization peak temperature.

The transition temperatures T_o, T_p and T_e determined by DSC and image analysis are summarized in Table 1. The values of enthalpy range between 4.28 and 10.68 J/g and are independent of the granule size as determined by light scattering. A similar behavior has been reported for wheat starch (Eliasson and Larsson, 1993). Using DSC, the temperature onset and peak obtained are slightly higher than the same temperatures obtained using hot-stage. This has been described by Doublier (1987) and Tester and Morrison (1990) as good consistency between the DSC transition temperatures and the birefringence end point temperatures for the granular starches of different botanical origins (Doublier, 1987). These small differences were defined by Li et al. (2013) and include various degrees of expansion before the DSC curve shows the gelatinization initiation temperature.

The T_o and T_p obtained by DSC and hot-stage correlated well (e.g., followed the same order from highest to lowest).This phenomenon was reported by Biliaderis (2009). However, the absolute values observed for the end set temperatures as well as their relative positions in terms of highest to lowest were not that clear. Evidently, heat transfer in the DSC is different than in the hot-stage experiment and several factors may influence the heat flow: geometry of the system, contact between heat source and sample holder, material supporting the sample, etc. The correlation of the gelatinization degree measured by DSC and hot-stage (image analysis) showed a good relationship between DG_D and DG_m for potato, corn and cassava, with r^2 of 0.98, 0.99 and 0.98, respectively, and 0.70 for wheat. This confirms that these two methods used to measure the degree of gelatinization characterize the process in equivalent ways and predict water absorption at specific temperatures.

4. Conclusions

The results of this study identified three clear phases in the gelatinization process and suggest new indices to correlate the thermal and microstructural changes. Also, degree of gelatinization at specific temperature can be a useful tool to predict water absorption in a specific point as well determine as the granule microstructure is changing as a function of the temperature. There is little evidence of the divergence of these two phenomena; therefore, this research may provide new information about starch gelatinization. However, additional studies are necessary to describe this behavior and determine the exact points at gelatinization starts and finishes.

Acknowledgment

The authors acknowledge the financial support of FONDECYT Project no. 3130448.

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