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Pasting viscosity and *in vitro* digestibility of retrograded waxy and normal corn starch powders

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ABSTRACT

Pasting viscosity and *in vitro* digestibility of oven-dried powders of waxy and normal corn starch gels (40% solids) retrograded under an isothermal (4 °C) or temperature cycled (4/30 °C) storage were investigated. Temperature cycling induced higher onset temperature for melting of amylopectin crystals than isothermal storage under a differential scanning calorimeter whereas little difference in crystalline type was observed under X-ray diffraction analysis. Temperature cycling caused higher pasting temperature and viscosity for the retrograded starches than isothermal storage. The retrograded waxy corn starch powders exhibited pasting behaviors similar to that of native waxy corn starch. However, the retrograded normal corn starch powders showed very much different pasting patterns with lower pasting viscosity but higher pasting temperature than native starch counterpart. The retrogradation increased slowly digestible starch content without changing resistant starch content, more effectively by the temperature cycling than the isothermal storage.

1. Introduction

Starch is one of the primary carbohydrates in human diet and often used as a food additive as thickener or stabilizer (Lehmann & Robin, 2007). For nutritional purposes, starch is classified into rapidly digestible starch (RDS), slowly digestible starch (SDS) and resistant starch (RS), based on the rate and extent of digestion (Englyst, Kingman, & Cummings, 1992). SDS and RS are considered to slower glucose release in digestive tract resulting in low glycemic response (Shu, Jia, Ye, Li, & Wu, 2009). A continuous intake of the foods of high glycemic index (GI) may lead to obesity, diabetes and cardiovascular disease (Ludwig, 2000). Thus, low GI foods have been suggested to be beneficial in preventing diseases associated with various metabolic disorders (Brand-Miller, Hayne, Petocz, & Colagiuri, 2003).

Substantial efforts have been made to modify the starch to reduce its digestibility, including chemical modification by cross linking (Woo & Seib, 2002), enzymatic modification by debranching (Berry, 1986; Miao, Jiang, & Zhang, 2009; Shin et al., 2004) or by elongating side chains (Ryu et al., 2010; Shin, Choi, Park, & Moon, 2010), and physical modification by irradiation (Chung, Lee, et al., 2010), thermal treatments (Chung, Liu, & Hoover, 2009) and starch retrogradation (Haralampu, 2000; Park, Baik, & Lim, 2009). Among these techniques, reduction of starch digestibility by controlling starch retrogradation is relatively simple and requires no chemical reagents, and the retrograded starch is safer than those from chemical modification.

Retrogradation of starch naturally occurs during cooling and storage of the cooked starch or starchy foods. Starch retrogradation includes short term retrogradation mainly by amylose and long term retrogradation by amylopectin. The RS in retrograded starch is mainly attributed to retrograded amylose (Haralampu, 2000). Park et al. (2009) reported that retrograded amylopectin in waxy corn starch gels stored at cycled temperatures reduced GI and induced an increased amount of RS. However, no RS was found in retrograded amylopectin powders prepared by drying and milling waxy starch gels, whereas hydrolysis rate by α-amylase was reduced to some degree (Fredriksson et al., 2000). Starch pastes retrograde continously during storage with changes in rheological properties and crystallinity. These changes depend on the storage conditions. In our previous study (Zhou, Baik, Wang, & Lim, 2010), amylopectin recrystallization with higher thermal stability was achieved by a temperature cycled storage (4 and 30 °C) compared to an isothermal storage (4 °C).

Waxy rice starch had been retrograded and dried to powders for a potential utilization as food ingredient (Jung, Han, & Lim, 2010). The hydrogel prepared with 30% retrograded waxy rice starch powders at 4 °C displayed a thick creamy texture with retrograded starch crystals that could melt at a temperature range of 35–51 °C. In this study, waxy and normal corn starch gels (40% solids) were retrograded under isothermal or temperature cycled storage, and then dried in a convection oven (40 °C) and milled to powders. For

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potential applications of these starch powders, pasting viscosity and *in vitro* digestibility of the retrograded starch powder were investigated.

2. Materials and methods

2.1. Materials

Waxy and normal corn starches were provided by Samyang Genex Company (Seoul, Korea). Porcine pancreatic α-amylase, and amyloglucosidase were purchased from the Sigma–Aldrich Company (St. Louis, MO).

2.2. Retrograded starch powders

Waxy or normal corn starch (100 g, dry solids) was suspended in distilled water (150 ml) in a capped container, and the mixture was preheated in a water bath from 65 to 90 °C (1 °C/min) with constant shaking (160 rpm). For gelatinization, the starch dispersion was heated in a boiling water bath for 30 min. The starch paste was then retrograded by storing for up to 8 days under two different conditions: at cycled temperatures of 4 and 30 °C each for 1 day, and at a constant temperature of 4 °C. Before the storage, silicon oil (2 ml) was applied to the surface of the starch pastes to prevent moisture loss during storage. The starch pastes rapidly became gels by retrogradation. The gels were cut into pieces (less than 1 cm in length and thickness) and dried in a convection oven (MOV-112F, Sanyo, Japan) at 40 °C overnight. The dried gels were then ground in a cyclone mill (Cyclotec 1093 sample mill, Foss, USA) and passed through a sieve (1 mm).

2.3. DSC analysis

A differential scanning calorimeter (DSC6100, Seiko Instruments Inc., Chiba, Japan) was used to determine the thermal transition characteristics of the powders of retrograded starch gels. The starch (2 mg, dry solids) was weighed into an aluminum DSC pan and distilled water was added until the starch was fully wet. The excess moisture was allowed to evaporate in a balance until the total weight of starch and water reached 5 mg to achieve a starch concentration of 40% (w/w). Then the pan was hermetically sealed, and equilibrated for 24 h at 4 °C. After equilibration, the pans were scanned from 20 to 120 °C at a rate of 5 °C/min. Indium and mercury were used for temperature calibration, and sapphire was used for heat capacity calibration, and an empty pan was used as a reference. All measurements were performed in triplicates.

2.4. X-ray diffraction analysis

The starch powders were equilibrated in a temperature humidity chamber (85% RH, 25 °C, 48 h). The final moisture content of the gel powder was around 20%. The diffraction patterns were determined by using an X-ray diffractometer (MO3XHF22, MAC Science Co., Japan), which was operated at 30 mA and 40 kV with the diffraction angles of 3–30° (2θ).

2.5. Pasting viscosity

Pasting viscosity of the starch powders was measured using a Rapid Visco Analyser (RVA-3D, Newport Scientific, Warriewood, Australia) at a solid concentration of 10.0% (3 g starch solids in 30 g total). Starch slurries were held at 50 °C for 1 min, heated to 95 °C at a rate of 12.2 °C/min, held at 95 °C for 2.5 min, cooled to 50 °C at 12.2 °C/min, and held at 50 °C for 2 min.

2.6. In vitro digestibility

The digestibility of the starch powders was determined by using the Englyst method (Englyst et al., 1992) with modifications. Starch (100 mg, dry solids) was hydrolyzed by a mixed solution of α-amylase and amyloglucosidase. Pancreatic α-amylase (No.7545, Sigma–Aldrich Company, St Louis, MO) (1 g) was dispersed in water (33 ml) by stirring for 10 min. The enzyme dispersion was then centrifuged for 10 min at 1500 × *g*, and a portion of the supernatant (24 ml) was transferred to a conical tube and mixed with amyloglucosidase (No.9913, Sigma–Aldrich Company, St Louis, MO) (0.6 ml). The enzyme solution was freshly prepared for the digestion analysis. Sodium acetate buffer (pH 5.2, 0.1 M, 20 ml) and five glass balls (10 mm in diameter) were added to each of the conical tubes containing homogenized starch powders (100 mg, starch solids). Then the enzyme solution (5 ml) was immediately added to the sample tube. The tube was screw capped firmly and immersed horizontally in the shaking water bath (170 rpm, 37 °C). Aliquots (0.5 ml) were taken at 10, 20, 30, 60, 120 and 180 min time intervals, mixed with 4 ml of 80% ethanol and centrifuged at 1500 × *g* for 10 min. The glucose content in the supernatant was determined using a glucose oxidase-peroxidase assay kit (GAGO-20, Sigma). RDS was calculated as the starch that was hydrolyzed within 20 min of incubation, SDS as the starch digested during the period between 20 and 120 min, RS as the starch that was not hydrolyzed within 120 min.

GI was calculated based on the equation GI = 39.71 + 0.549 × HI given by Goni, Garcia-Alonso, and Saura-Calixto (1997). The hydrolysis index (HI) was obtained by dividing the area under the hydrolysis curve of the sample by the area obtained for a standard material which was glucose.

2.7. Statistical analysis

All numerical results were the means of triplicates. Data were analyzed by one-way analysis of variance (ANOVA) using ORIGIN 8.0 (Origin Lab Inc., USA). The means comparison were determined by Turkey's test ($P < 0.05$).

3. Results and discussion

3.1. Thermal transition

The oven-dried starch powders of freshly prepared starch pastes showed no melting endotherm in DSC thermograms (data not shown), indicating that no recrystallization had occurred during drying. The starch powders prepared after retrogradation with either normal or waxy corn starch, however, exhibited broad endotherms for the melting of amylopectin crystals (Table 1). Normal corn starch displayed an additional endotherm above 100 °C which was responsible for the melting of amylose–lipid complex (data not shown). The endotherm for amylose–lipid complex was little affected by the retrogradation conditions.

Compared to the isothermal storage at 4 °C, the temperature cycling (4/30 °C) resulted in higher onset temperatures (T_o) but similar conclusion temperatures (T_c) for the melting of amylopectin crystals, resulting in narrower endotherms. It may indicate that the amylopectin crystals formed by the temperature cycling were more uniform and more thermally stable than those formed by the isothermal storage. The melting enthalpies (ΔH) of the starch powders retrograded at cycled temperatures, however, were smaller (Table 1). Some starch crystals of low stability might melt during the storage at 30 °C (Baik, Kim, Cheon, Ha, & Kim, 1997; Durrani & Donald, 1995; Elfstrand et al., 2004; Silverio, Fredriksson, Andersson, Eliasson, & Aman, 2000).

The oven-dried starch gel powders melted in slightly broader temperature range with smaller ΔH values in comparison with

Table 1
Thermal transition properties of oven-dried powders of native and retrograded normal and waxy corn starch gels (40% solids).[A]

Starches	T_o (°C)	T_p (°C)	T_c (°C)	T_r (°C)	ΔH (J/g)
Normal corn					
Native	66.0[a]	70.3[a]	76.5[b]	10.5[g]	14.4[b]
4 °C 2d	35.4[c]	55.2[f]	69.6[d]	34.2[c]	6.3[f]
4 °C 8d	34.8[c]	57.5[d]	70.0[d]	35.2[b]	8.0[e]
4/30 °C 2d	49.6[b]	58.9[bc]	69.2[d]	19.6[f]	3.7[h]
4/30 °C 8d	50.3[b]	60.0[b]	70.1[d]	19.8[f]	5.1[g]
Waxy corn					
Native	65.2[a]	70.7[a]	86.2[a]	21.0[e]	18.7[a]
4 °C 2d	36.5[c]	58.3[cd]	71.5[c]	35.0[b]	10.3[d]
4 °C 8d	35.9[c]	56.5[e]	72.2[c]	36.3[a]	11.7[c]
4/30 °C 2d	49.4[b]	59.7[b]	72.4[c]	23.0[d]	6.9[f]
4/30 °C 8d	49.6[b]	60.3[b]	72.8[c]	23.2[d]	8.2[e]

T_o: onset melting temperature; T_p: peak melting temperature; T_c: conclusion melting temperature; T_r: melting temperature range; ΔH: melting enthalpy.

[A] Means of triplicates. Values followed by different superscripts in each column are significantly different ($P < 0.05$).

the melting of the counterpart starch gels (data not shown). Rehydration was additionally required for the melting of dried starch powders whereas starch gels were directly analyzed. It was supposed that the rehydration could not fully recover the initial starch gel structure. It was also expected that starch chain association occurred during drying, mainly in the amorphous regions of starch because no melting endotherm had been found in the freshly gelatinized starch. Although drying the retrograded starch gels proceeded at 40 °C, the onset for crystal melting of the dried powders for the starches isothermally stored occurred at lower temperatures (34.8–36.5 °C) (Table 1). It indicates that the drying (40 °C) did not damage the crystals formed by retrogradation.

3.2. X-ray diffraction pattern

Fig. 1 shows the X-ray diffraction patterns of the retrograded starch powders either under the isothermal or under temperature cycled conditions. The diffraction peaks at 5°, 15°, 17°, 20°, 22° and 23° of 2θ (Gidley & Bulpin, 1987) revealed that the crystals in retrograded normal and waxy corn starches had B-type configuration, which was typical for retrograded starches (Miles, Morris, Orford, & Ring, 1985). Compared with waxy corn starch, normal corn starch showed stronger diffraction intensity at 20° (2θ), indicating that the starch crystals additionally contained V-type crystals (Cheetham & Tao, 1998) which were formed by amylose–lipid complex. It was observed that the starch gels stored under the isothermal condition had slightly higher relative crystallinity (peak intensity in X-ray diffraction pattern) than those stored at cycled temperatures. However, little difference in crystallinity was observed when increasing the storage period from 2 to 8 days.

3.3. Pasting viscosity

Pasting viscosities of the retrograded starch powders are shown in Fig. 2. In native granular form, waxy corn starch showed lower pasting temperature (75.0 °C) and higher peak viscosity (4315 cP) than normal corn starch (77.5 °C and 3223 cP, respectively). Viscosity increase during pasting is usually initiated by the swelling of starch granules, which results from the ability of starch to entrap water. Tester and Morrison (1990) reported that amylopectin in starch was mainly responsible for starch swelling, whereas amylose suppressed swelling maintaining the integrity of starch granules. The waxy corn starch granules exclusively consist of amylopectin, and thus swelled more readily than normal corn starch resulting in a lower pasting temperature and higher peak viscosity.

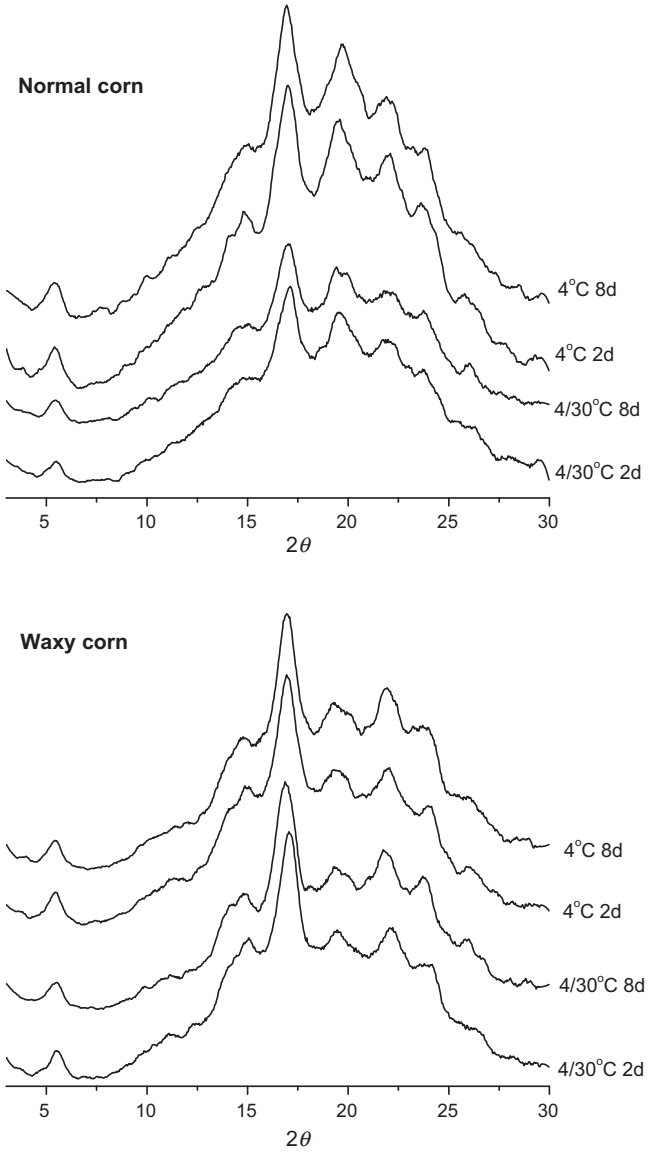

Fig. 1. X-ray diffraction patterns of oven-dried powders of waxy and normal corn starch gels retrograded under an isothermal condition at 4 °C or temperature cycling of 4 °C and 30 °C (1 day each, 4/30 °C) for 2 and 8 days.

Pasting viscosity of the oven-dried powders of retrograded waxy corn starch gels showed similar patterns to that of native waxy corn starch: high peak viscosity, high breakdown and low set back. The dry powders of freshly gelatinized waxy corn starch showed a rapid swelling and broad peak of paste viscosity as soon as the starch dispersion was heated. The rapid increase in viscosity at the early stage of pasting for the gelatinized waxy corn starch powders resulted from the rapid hydration and swelling of the amorphous starch powders. During retrogradation, the amylopectin chains spontaneously reassociated forming the gel matrices of increased thermal stability. It was evidenced by the increased pasting temperature with the increase of retrogradation period (Fig. 2). The pasting temperature of all the retrograded waxy corn starch powders tested (51–60 °C), however, was still lower than that of native starch (75 °C). The peak viscosity of the powders of retrograded waxy corn starches was relatively comparable to that of the native waxy corn starch. It indicates that the swollen particles of the starch could retain the integrity until the viscosity reached the peak. The regeneration of the pasting behavior of the native starch was possible because of the reassociation of amylopectin during

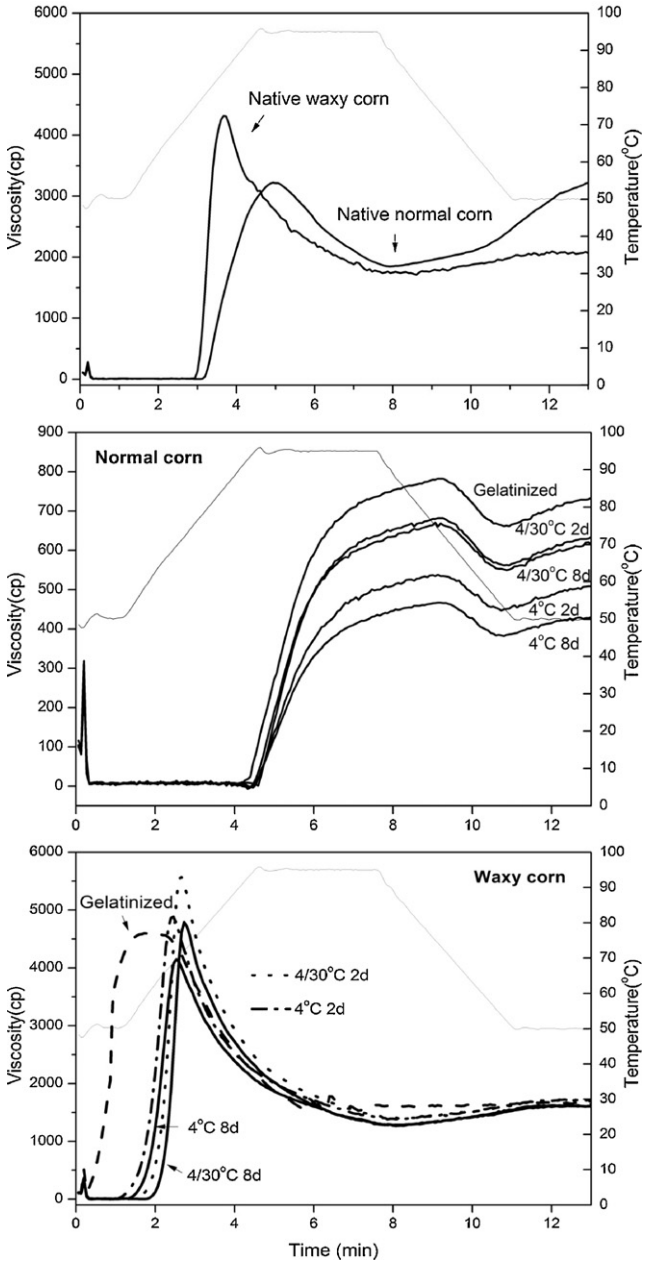

Fig. 2. Pasting viscosity of native waxy and normal corn starches, and oven-dried powders of gelatinized and retrograded waxy and normal corn starch gels.

retrogradation. The pasting viscosity data revealed that the waxy corn starch powders produced after retrogradation could be pasted with less thermal energy and providing the pasting viscosity as did native waxy corn starch. The B-type amylopectin crystals formed by retrogradation are thermally less stable than the A-type crystals in native starch as shown in DSC data (Table 1). It could be one of the reasons for the lower pasting temperature of the retrograded waxy starch powders than that of native starch counterpart.

The starch powders prepared after temperature cycling retrogradation formed pastes at higher temperatures than did the starch powders isothermally retrograded, for both waxy and normal corn starches (Fig. 2). The starch powders after temperature cycling retrogradation also exhibited higher peak viscosity than those after isothermal retrogradation. The amylopectin crystals formed by temperature cycling had higher perfection than those from the isothermal storage as shown in DSC data (Table 1), and thus temperature cycling produced the retrograded starch powders

Table 2
Nutritional starch fractions and GI of oven-dried powders of gelatinized and retrograded normal and waxy corn starch gels (40% solids).[A]

Starches	RDS (%)	SDS (%)	RS (%)	GI
Normal corn				
Gelatinized	88.9^{b}	6.3^{h}	4.7^{a}	90.0^{b}
4 °C 2d	81.0^{e}	14.6^{e}	4.4^{a}	83.8^{e}
4 °C 8d	79.7^{f}	15.3^{d}	5.0^{a}	81.3^{g}
4/30 °C 2d	78.3^{g}	16.9^{c}	4.9^{a}	83.0^{f}
4/30 °C 8d	73.7^{h}	21.8^{a}	4.6^{a}	76.7^{h}
Waxy corn				
Gelatinized	98.8 ^{a}	0.6^{i}	0.5^{b}	99.2^{a}
4 °C 2d	88.6 ^{b}	10.5^{g}	0.8^{b}	89.2^{c}
4 °C 8d	87.8 ^{c}	10.9^{g}	1.2^{b}	85.8^{d}
4/30 °C 2d	85.2^{d}	13.1^{f}	1.0^{b}	88.5^{c}
4/30 °C 8d	79.9^{f}	18.8^{b}	1.2^{b}	80.8^{g}

[A] Means of triplicates. Values followed by different superscripts in each column are significantly different ($P < 0.05$).

of greater thermal stability against pasting. It was noteworthy that the increased thermal stability for the temperature cycled starches resulted in increased peak viscosity for both normal and waxy corn starch samples (Fig. 2). It indicates that the swollen starch powders of temperature cycling were more resistant to heating and mechanical shearing. However, it was observed that the peak viscosity decreased as the storage period was increased from 2 to 8 days regardless of starch types and storage conditions. The increased level of recrystallization by extending the storage might give the adverse effect of hindering the swelling of starch powders.

In contrast to waxy corn starch powders, normal corn starch powders exhibited the pasting viscosity patterns significantly different from that of native normal corn starch. Normal corn starch powders showed increased pasting temperature and reduced viscosity by retrogradation. Amylose exists mainly in amorphous entity in native starch and some of which may associate with residual lipids (Zobel, 1988). During gelatinization, amylose may leach out of swollen starch granules whereas most of amylopectin remains inside the granules. The swollen granules are physically weak and readily disintegrated by mechanical stirring/shaking afterwards (Tester & Morrison, 1990). The pastes prepared in this study were very thick because starch content was high (40%). In this circumstance, even after complete gelatinization, the swollen granules might be not much disintegrated so that most amylopectin chains remained inside the granules. It was also expected that the amylose chains leached out of the swollen granules could effectively protect the swollen granules from being destructed by the mechanical shaking because of the limited amount of water content. Upon cooling, the amylose might contribute to form the gel matrix with increased mechanical rigidity. Therefore the lack of pasting ability of the retrograded normal corn starch powders was attributed to the amylose which had leached from starch granules and restricted the water-uptake and swelling of the dry powders. Consequently, the retrograded normal corn starch powders exhibited the pasting behaviors opposite to those of retrograded waxy corn starch powders, as results of the presence of amylose.

3.4. *In vitro digestibility*

The dry powders of gelatinized normal and waxy corn starches which had not been retrograded were readily digested with high RDS values (88.9 and 98.8%, respectively) and glycemic indexes (GI 90.0 and 99.2, respectively, Table 2). The starch molecules in the freshly gelatinized starches are amorphous and susceptible to digestive enzymes. The normal corn starch powders, however, were less digestible than the waxy corn starch powders. The gelatinized normal corn starch contained RS near 5%, whereas the

gelatinized waxy corn starch contained almost no RS. The presence of amylose (Chung, Liu, Wang, Yin, & Li, 2010) and amylose–lipid complex (Tufvesson, Skrabanja, Bjorck, Elmstahl, & Eliasson, 2001) is known to retard starch digestibility.

By retrogradation, RDS content was decreased and SDS was increased. However, no significant change in RS was observed for both retrograded waxy and normal corn starches. Chung, Liu, et al. (2010) found that retrogradation had little effect on RS content in starch. Fredriksson et al. (2000) reported that retrogradation of starch gels at cycled temperatures had no effects on RS content, compared to that under isothermal conditions. However, Park et al. (2009) reported that temperature cycled storage induced the formation of greater amount of RS and a much lower GI for waxy corn starch gels. For their study, the gels were directly used to assess the *in vitro* digestibility. Although RS is primarily correlated to the amylose content in starch (Haralampu, 2000), amylopectin recrystallization may contribute in the formation of RS. The controversy in the effect of amylopectin recrystallization on RS might be from the differences in physical status of starch samples and analysis procedure.

In this study, the GI decrease and increase in SDS content were more pronounced when the starch gels were treated under the storage of temperature cycling compared to those of isothermal storage. The SDS therefore appeared to be related with the differences in thermal transition of the amylopectin crystals and in GI of the retrograded starches tested in this study. With either starch gels or dry powders of the gels, temperature cycling for retrogradation appeared to be more effective in reducing GI compared to isothermal storage. The drying process usually helps the chain association and thus rigidifies starch gel matrix as water is removed. Therefore, the drying itself may cause the reduction of GI.

4. Conclusions

Pasting viscosity of retrograded corn starch powders was affected by the presence of amylose and retrogradation conditions. The amylose leaches from starch granules during pasting and rigidifies starch gel matrices, and thus the retrograded normal corn starch powders showed higher pasting temperature and lower viscosity than native starch counterpart. In contrast, the dry powders of retrograded waxy corn starch exhibited the pasting viscosity very similar to that of native starch. Starches retrograded under a temperature cycled storage (4/30 °C) showed higher pasting viscosities and temperatures than those under an isothermal storage (4 °C). Unlike commercially pregelatinized starch powders, the retrograded starch powders may be utilized with typical pasting behaviors of native granular starches and additional health benefits of high SDS content and low GI.

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