Physical Behavior of Durum Wheat Starch (*Triticum durum*) during Industrial Pasta Processing

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Starch was isolated and yields and granule size distributions were determined at different steps during industrial pasta production and subsequent high-temperature drying (88 °C). The first drying steps rendered the starch granules in general and the small ones in particular less extractable, possibly due to increased physical inclusion or interaction between starch and gluten components. On the basis of analytical results, starches were divided in a first group containing those withdrawn before the pasta drier and a second group of starches that have undergone partial or complete drying. The latter starches showed higher gelatinization temperatures and viscosities and lower swelling powers and solubilities during gelatinization. It is suggested that the high-temperature pasta drying results in less permeable and thus more rigid starch granules.

Keywords: *Triticum durum; starch; interactions; pasta production*

INTRODUCTION

A lot of research has dealt with the physicochemical and technological behavior of starch, as it is the main constituent in many vegetable foods. Interactions between starch and other wheat components (mainly proteins) have been studied (D'Egidio et al., 1984; Lindahl and Eliasson, 1986; Eliasson and Tjerneld, 1990; Chedid and Kokini, 1992). Little work has been done, however, on the behavior of the granular starch and its interactions during durum wheat pasta processing.

Lintas and D'Appolonia (1973) determined the effects of pasta processing on carbohydrates. Pasting properties, water-binding capacity, and starch damage values were all affected by processing. Some starch damage took place during mixing and extruding, but particularly during low-temperature (LT) drying. It was suggested that starch, mechanically damaged during milling and extrusion, provides a suitable substrate for amylolytic enzymes during the drying step (Lintas and D'Appolonia, 1973).

Viscoamylograph setback and maximal peak viscosity were higher for starches isolated from high-temperature (75 °C, 90% RH (relative humidity)) than from ultra-high-temperature (90 °C, 90% RH) dried pasta, probably because the latter causes a higher resistance of the starch to disintegration (Cubbada et al., 1987, 1988) as exemplified by the lower total organic matter contents in wash water for pasta dried at high temperatures. High-temperature drying induces changes in the physicochemical properties in general and the permeability of the starches (for water) in particular (Cubbada et al., 1987, 1988).

More recently, Cunin (1995) observed no change in birefringence even when very high temperatures (maximum 90 °C, 85 to 60% RH) were applied at a high moisture content (VHT-HM) of the pasta. The X-ray diffraction patterns basically did not differ from the original A type pattern of intact native wheat starch, except for the more intense 20° 2θ peak, possibly because of complexation of amylose with endogenous lipids in the starch granules. The mixing, extrusion, and high-temperature (HT) drying altered neither the X-ray diffraction pattern nor the differential scanning calorimetry (DSC) characteristics of the samples. However, the DSC temperature of gelatinization (T_g) shifted to higher values with more intensive drying conditions. At the same time, the temperature range of gelatinization ΔT decreased by 6 °C and the gelatinization enthalpy ΔH was reduced with 3.9 J/g of dry pasta when drying was shifted from LT to VHT-HM conditions. It was suggested that the observed change in gelatinization characteristics resulted from a heat moisture treatment, i.e., a hydrothermal treatment at low moisture levels. Such treatment can indeed change the T_g, ΔT, and ΔH values of starch (Eerlingen et al., 1996). However, heat moisture treatment is generally believed to induce a broadening instead of a narrowing of the gelatinization temperature range. Also possible is that the pasta ultrastructure changes during high-temperature drying and that such change leads to slower water penetration (Resmini and Pagani, 1983; Pagani et al., 1986) during cooking or DSC analysis. Furthermore, the changes in starch permeability, as described above (Cubbada et al., 1987, 1988), can also be in play.

Fortini (1988) emphasized that a study of the interactions between components that are qualitatively and functionally important is necessary in understanding the pasta quality determining factors. Indeed, knowledge of the chemical composition is not sufficient for understanding the interactions between semolina components during pasta processing and the subsequent formation of aggregates with properties different from those of the individual components. An improvement in pasta quality can probably be explained better on the basis of protein−starch interactions than on the basis of the protein content itself (D'Egidio et al., 1984) because the interactions affect pasta texture and con-

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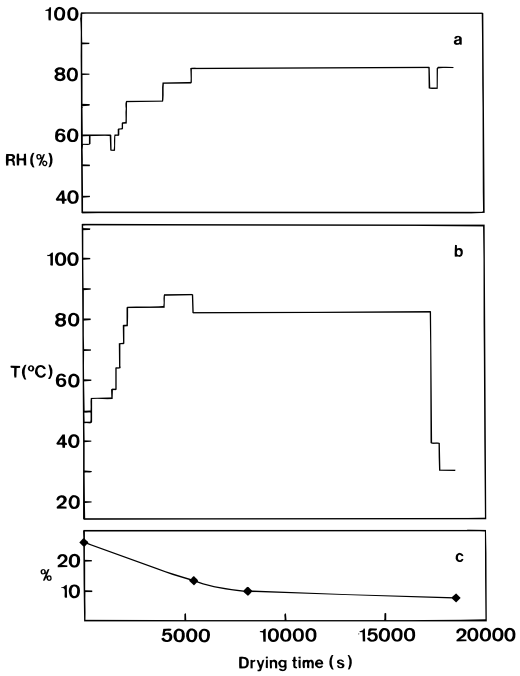

Figure 1. High-temperature drying profile: (a) relative humidity (%); (b) temperature (°C); (c) moisture content of the samples (%) against time (min) spent in drier.

sequently firmness, stickiness, and cooking losses. With freeze-fracturing micrographs (Pagani et al., 1986) it was found that, in very high-temperature dried spaghetti of high cooking quality, proteins take up the area where, in spaghetti dried at low temperature, a water coat (at least 30 nm wide) surrounds the starch granules. It was thought that starch−protein interactions are present at this interlayer.

In the work presented here, we describe the behavior of starch and its interactions during industrial pasta processing as part of a systematic effort to elucidate the role of starch and interactions between pasta components in pasta quality.

EXPERIMENTAL PROCEDURES

Samples. Samples were withdrawn at several stages during the industrial production of long good, high-quality pasta (fine spaghetti, 1.45 mm diameter) from semolina (SA0), obtained by milling a commercial blend (1995 harvest) of durum wheats. After a first rapid (75 s) premixing of the semolina and tap water (final moisture content 31%), the dough (SA1) was further mixed in a blender during 15 min to homogenize the water distribution in the dough particles. The resulting dough (SA2) was fed into the extruder via a vacuum lock chamber. It was extruded at 100−110 bar and 47−48 °C. The extruded product (SA4) was dried using a high-temperature drying profile, as illustrated in Figure 1. Samples were withdrawn from the drier after 90 (SA5) and 135 min (SA6) drying time, i.e., the points where we could easily access the drier. At the beginning of the drying process, the product adopted the wet bulb temperature (ca. 30 °C). After 36 min (and later), the product temperature equalled the drier temperature. Also the end product (SA7) was sampled. Additionally, semolina and end product of the same pasta production line were sampled independently twice more and confirmed the changes discussed below.

Moisture Contents. Moisture contents were determined at least in duplicate as weight loss of ca. 3 g accurately weighed sample or starch after 90 min at 130 °C.

Starch Isolation (Hoseney, 1994). Samples (500 g) were hand-kneaded to a firm dough during several minutes after addition of a suitable amount of deionized water. Starch was washed out of the dough balls by a trickle of deionized water (ca. 1500 mL in total). The resulting suspensions were sieved twice (250 and 112 μm). Residues contained mainly protein but also considerable amounts of starch. Filtrates that passed both sieves were centrifuged (10 min, 1800 g, room temperature). The supernatants were decanted, and the yellow-brown upper layers of "sludge" (of which the main constituent was gluten) were scraped off. The starch layers were suspended in water again and centrifuged as above, and the sludge layers were removed again. This process was repeated until the starch was sludge free. Starches were air-dried overnight and then stored at room temperature.

Samples withdrawn after extrusion were processed using a batter method. The batter was prepared by mixing the samples in excess water (300 mL/100 g of product). The obtained suspensions were treated as described above.

Analytical Methods. For *protein determination*, starch samples (ca. 100 mg) were accurately weighed. Exactly 10.0 mL of 0.5 N sodium hydroxide was added, and the starch was stirred magnetically until fully dispersed. An aliquot of this suspension (1.0 mL) was diluted with 4.0 mL of deionized water. A 1 mL aliquot of the obtained mixture was analyzed in duplicate with the Lowry et al. (1951) method with bovine serum albumin as standard.

For *lipid content*, starch (10.0 g) was hydrolyzed in 300 mL of 3.0 N hydrochloric acid for 30 min (100 °C). Samples were filtered and washed with deionized water until the filtrate was neutral. The filters were dried at 65 °C overnight, and fat was extracted with 50 mL of petroleum ether using a Soxtec extraction apparatus (120 min). Crude fat was weighed after drying for 90 min at 100 °C. Analyses were performed at least in duplicate.

For *apparent amylose content*, a colorimetric procedure (Williams et al., 1970) was used with measurement of the blue color after 20 min. Potato amylose (type III, Sigma) served as standard.

Granule Size Distribution. Granule size distribution was analyzed using a Coulter Multisizer II equipped with a 140 μm aperture tube and measuring in 256 channels, resulting in measurement range between 2.8 and 84 μm. Calibration was with polystyrene divinyl benzene latex. Starch samples were dispersed in 0.5% sodium chloride.

Starch Damage. Starch damage (Hoseney, 1994) was measured at least in duplicate with a Chopin SD4 measurement apparatus, based on an iodine absorption working principle described earlier (Medcalf and Gilles, 1965).

Differential Scanning Calorimetry. DSC experiments were with a Seiko DSC-120. Indium and tin were used as standards. Approximately 5 mg of starch was accurately weighed in an aluminum sample pan. Water was added to obtain a ratio of 1:2 (w/w). The sample pan was hermetically closed and then heated from 20 to 150 °C at a rate of 4 °C/min. An empty pan was used as reference. The transition temperatures (°C) reported are for onset (T_o), peak (T_p), and completion (T_c). The enthalpy (ΔH, mJ/mg) of gelatinization was determined by integration of the endotherm. Each sample was analyzed at least in triplicate.

Rapid Visco Analysis. For measurements with the rapid visco analyzer (RVA), we used 25.0 g of either a 6.6% or a 9.9% starch-in-water suspension (on weight base). The temperature profile included a 2 min isothermal step at 50 °C, a linear temperature increase to 95 °C in 7 min, a holding step (8 min at 95 °C), a cooling step (7 min) with a linear temperature decrease to 50 °C, and a final isothermal step at 50 °C. Duplicate measurements always agreed within 5 RVU over the whole profile.

Swelling Power (SP), Amylose (S_{amy}), and Total (S) Solubility. For swelling power, the weight (g) occupied by 1 g of dry starch after gelatinization in excess water, determination, we gelatinized 200 g of starch suspension (1% (w/w)) for 30 min in a water bath at 95 °C. The suspensions were centrifuged (10 min at 3500*g*). Sediment was weighed and SP (g/g) was calculated. Supernatant was used to determine the amylose solubility (blue value procedure (Gilbert and

Table 1. Starch Codes and Description, Isolation Yields (Dry Starch on Total Dry Matter), Swelling Power (SP), Total Solubility (S), Amylose Solubility (S_{amy}), and Starch Damage (Standard Deviations Given in Parentheses)

starch	isolated out of	isolation yield[a] (%)	SP (g/g)	S (%)	S_{amy} (%)	starch damage (UCD)
SA0	semolina	40	11.8 (0.2)	12.4 (1.0)	15.8 (1.7)	25.2 (0.5)
SA1	dough after premixer	50	12.0 (0.2)	12.6 (0.6)	17.6 (1.1)	nd[b]
SA2	dough after mixer	45	11.7 (0.5)	12.1 (1.3)	17.0 (1.8)	nd[b]
SA3	dough after vacuum lock chamber	56	12.1 (0.3)	13.7 (1.3)	17.5 (0.6)	25.7 (0.8)
SA4	extruded pasta	48	11.5 (0.1)	12.4 (0.5)	17.2 (1.0)	29.4 (1.3)
SA5	pasta dried for 90 min	27	10.6 (0.2)	11.3 (0.9)	16.5 (1.9)	31.8 (1.1)
SA6	pasta dried for 135 min	27	10.7 (0.2)	11.6 (1.1)	16.8 (2.1)	nd[b]
SA7	end product	28	10.5 (0.2)	12.2 (0.5)	17.0 (0.2)	31.6 (0.3)

[a] %: percentage dry starch isolated out of total dry matter. [b] nd: not determined.

Spragg, 1964)) and the total solubility (phenol−sulfuric acid method (Dubois et al., 1956)) of the starches during gelatinization.

Susceptibility of Starches to Annealing. The samples SA1 and SA7 were incubated at a temperature 3.3% lower than their peak gelatinization temperature (expressed in kelvin, as measured with DSC) for 24 h in excess water. After this treatment, starches were Buchner-filtered and air-dried.

Statistical Data Analyses. We used the (double sided) two sample *t*-test, assuming unequal variances (Wonnacott and Wonnacott, 1990).

RESULTS AND DISCUSSION

Chemical Analyses. All starch samples had similar protein and lipid contents. These averaged 0.46 and 0.55%, respectively. The amylose contents of all starches were also similar (fluctuations were less than 1% and hence within the experimental error). This finding concurs with earlier work (De Stefanis and Sgruletta, 1990), where no differences in analytical amylose contents of starch before and after spaghetti production were found.

Yields of the Starch Isolation. The starch yields are represented in Table 1. Two isolation procedures were used. Samples SA0 through SA3 were washed from doughs; the other samples were obtained with the batter method. Additional experiments proved that the isolation method had no real influence on the yields. Also, starches SA3 and SA4, both isolated in a different way, were obtained in similar yields.

The samples can be divided in two distinct groups. SA0 through SA4 were obtained in higher yields than the starches obtained from partially or totally dried products (SA5−SA7). Apparently, the high-temperature drying renders the starch less extractable, possibly due to increased physical starch inclusion or increased interactions between starch and gluten components. In that way, more starch may have been removed with the gluten fraction (as residue on the sieves and sludge layer after centrifugation) during the isolation procedure. This concurs with the earlier finding that, during high-temperature drying, proteins coagulate and may envelop the starch granules within a continuous network (Resmini and Pagani, 1983).

Granule Size Distribution. Preliminary experiments indicated that the two isolation methods generate starch with practically the same granule size distribution. Also, the granule size distributions of SA4 and SA5 are quasi identical. Microscopic observations taught that all isolated starches were birefringent and not aggregated.

Figure 2 clearly illustrates that relatively less small starch granules can be isolated out of samples that were dried to a variable extent (SA5−SA7). This also may indicate stronger starch−gluten interactions as a result of drying. As a consequence, not only less starch (cf. supra) but also relatively less small granules can be isolated from the dried samples. This seems logical because the smaller granules have a larger surface/volume ratio than the larger granules and hence have more interaction surface per weight unit than the larger granules. It also seems likely that smaller granules are more easily entrapped in the gluten matrix (physical inclusion) during processing. Hence, the smaller granules are removed in larger amounts with the protein fraction during isolation.

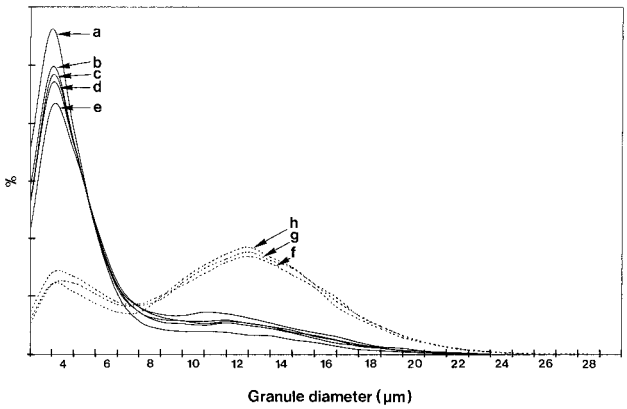

Figure 2. Granule size number distribution of the starches SA0−SA4 (straight curves) and the starches SA5−SA7 (dotted curves): a = SA0; b = SA1; c = SA2; d = SA3; e = SA4; f = SA5; g = SA6; h = SA7.

Starch Damage. Mixing (SA0−SA3) and the final drying stages (SA5−SA7) clearly do not induce starch damage (Table 1). Extrusion (SA3−SA4) and the first drying stages (SA4−SA5) on the other hand lead to an increase in the starch damage levels of 3.7 and 2.4 UCD, respectively. It was found earlier (Lintas and D'Appolonia, 1973) that the change in starch damage during the (low-temperature) drying step is higher than that during the extrusion step.

Differential Scanning Calorimetry. Starches isolated from dried samples (SA5−SA7) had slightly but significantly ($\alpha = 0.05$) higher onset (T_o) and peak (T_p) temperatures of gelatinization and a more narrow gelatinization range ($\Delta T = T_c - T_o$) than their undried counterparts (see Table 2). Earlier data by Cunin (1995) suggest that, under the experimental conditions used by the author, pasta mixing, extrusion, and high-temperature drying (below 76 °C) do not alter any DSC characteristics of the starch fraction. The observed increase in T_o and T_p and subsequent narrowing of ΔT in our work cannot be considered to result from annealing (Gough and Pybus, 1971; Slade and Levine, 1987; Krueger et al., 1987a,b; Knutson, 1990; Larsson and Eliasson, 1991; Stute, 1992; Hoover and Vasanthan, 1994) since it implies an incubation in excess water at a temperature close to the gelatinization temperature. We further can ascribe our DSC findings neither to

Table 2. Onset (T_o), Peak (T_p), and Conclusion (T_c) Temperatures of Gelatinization, Gelatinization Enthalpy (ΔH), and Gelatinization Range ($\Delta T = T_c - T_o$) As Determined with DSC (Standard Deviations Given in Parentheses)

starch	SA0[a]	SA1[a]	SA2[a]	SA3[a]	SA4[a]	SA5[a]	SA6[a]	SA7[a]	SA1T[b]	SA7T[b]
T_o (°C)	48.3 (0.1)	49.9 (0.8)	49.1 (0.3)	48.3 (0.9)	48.6 (1.0)	51.3 (0.0)	51.6 (0.2)	51.3 (0.3)	59.7 (0.1)	60.5 (0.2)
T_p (°C)	55.8 (0.3)	56.2 (0.0)	56.1 (0.1)	56.2 (0.2)	55.8 (0.3)	57.3 (0.0)	57.5 (0.0)	57.2 (0.1)	62.0 (0.2)	62.9 (0.2)
T_c (°C)	63.4 (0.1)	65.0 (0.6)	64.4 (0.6)	66.7 (0.2)	63.5 (1.5)	64.5 (0.3)	64.6 (0.3)	64.6 (0.4)	65.8 (0.5)	66.4 (0.3)
ΔH (J/g)	11.2 (0.3)	10.7 (2.1)	12.0 (0.7)	10.2 (0.9)	10.4 (1.2)	10.2 (0.4)	10.2 (1.0)	11.5 (0.1)	10.3 (0.6)	10.2 (0.1)
ΔT (°C)	15.1 (0.1)	15.1 (2.2)	15.3 (0.8)	18.4 (1.3)	14.9 (1.2)	13.2 (0.4)	12.9 (1.0)	13.3 (0.3)	6.1 (0.6)	5.9 (0.2)

[a] Starch codes as in Table 1. [b] Annealed starches; see text for full explanation.

Table 3. Peak Viscosity (V_p), Viscosity after Shear Thinning (V_{st}), and End Viscosity (V_e) of the Starches As Measured with RVA with Concentrations of 6.6 and 9.9%

	SA0[a]	SA1[a]	SA2[a]	SA3[a]	SA4[a]	SA5[a]	SA6[a]	SA7[a]
6.6%								
V_p (RVU)	29	29	32	32	37	43	44	41
V_{st} (RVU)	25	25	25	27	29	36	36	33
V_e (RVU)	70	70	70	70	75	79	80	74
9.9%								
V_p (RVU)	226	244	253	240	255	295	294	288
V_{st} (RVU)	153	162	150	156	159	184	176	176
V_e (RVU)	358	370	356	365	368	417	400	400

[a] Starch codes as in Table 1.

differences in contents of minor constituents of the starches nor to the differences in granule size distribution (cf. supra) because the starches with relatively less small granules had the higher gelatinization temperatures. This contrasts with the general belief that smaller granules gelatinize at higher temperatures than the large ones (Vasanthan and Bhatty, 1996; Kulp, 1973). Also, starch damage (cf. supra) cannot explain the DSC results since it is expected to lower ΔH values (Eliasson and Larsson, 1993) and gelatinization temperatures (Morrison et al., 1994). Cubbada et al. (1988) earlier suggested a change in physicochemical character, perhaps permeability, during high-temperature drying of starch. It seems logical that a decrease in starch permeability can increase the gelatinization temperatures as measured in the DSC.

Rapid Visco Analysis. The RVA results are presented in Table 3. Also here, the starches can be grouped in two subsets. A first set contains the starches SA0−SA4 and shows lower viscosities than the second set (SA5−SA7). No supernatant could be detected after centrifuging (10 min, 3500 g) the starch paste obtained after the first 17 min of the RVA run (6.6%), indicating that all RVA analyses were made in the concentrated regime where viscosity is mainly governed by the rigidity of the starch granules (Steeneken, 1989). Thus, the observed higher viscosity for the dried starches implies that the starch granules become more rigid during the drying process. As already mentioned, Cubbada et al. (1988) earlier suggested a possible change in permeability, during high-temperature drying of starch. It seems logical to assume that lower permeability renders starch more rigid in the warm water RVA surroundings.

Swelling Power (SP), Total (S), and Amylose Solubility (S_{amy}) during Gelatinization. The same two groups of samples can again be discerned (Table 1). The first group, containing the starches SA0−SA4, had mean SP, S, and S_{amy} of 11.8 g/g, 12.6%, and 17.0%, respectively. The first two parameters are (95% probability level) significantly lower for the second group (values were respectively 10.6 g/g, 11.7%, and 16.8%). These results indicate again a change in the physicochemical character (permeability, rigidity) of the starch granules during high-temperature drying. Indeed, it can be assumed that a lower permeability diminishes the starch swelling capacity and renders the starch more rigid and less soluble.

Susceptibility of Starches to Annealing. Annealing increased the gelatinization temperature for both SA1 and SA7 with almost 6 °C. The gelatinization range became smaller for both starches (with 9.0 and 7.4 °C, respectively). We also noticed a small decrease in gelatinization enthalpy for both annealed starches. These results illustrate that the susceptibility to annealing of the starch at the beginning and the end of the pasta production line is equal, indicating that starch does not anneal during pasta drying.

CONCLUSIONS

Starch isolation yields and granule size distributions provided indications about the interaction behavior of starch with other semolina components in general and proteins in particular. The first drying steps of an industrial pasta production render the starch granules in general and the small ones in particular less extractable, possibly due to increased physical inclusion or interaction between starch and gluten components. Other production steps had much less impact on the interaction behavior.

Also, clear differences exist in gelatinization characteristics of the starches from an industrial high-temperature pasta production line, as clearly shown by the DSC and RVA gelatinization characteristics, swelling powers, and total solubilities. In each case, the results split the starches in two groups. A first group contains the starches from samples withdrawn before the pasta drier. The second group contains starches that have undergone partial or complete drying (SA5−SA7). Pasta processing, and more specifically the drying, affects the starch. The observed differences cannot be explained on the basis of apparent amylose contents or amounts of minor constituents as no clear differences or trends in these data were found. The observed narrowing of the gelatinization range rejects heat moisture treatment (Eerlingen et al., 1996) as an explanation. Also, annealing does not occur during the first drying stages since the temperature/humidity circumstances of the samples in the drier do not even approximate annealing conditions and the susceptibility of the starch to annealing remains the same after pasta-making. Starch damage is not in play either, since increased starch damage is known, contrarily to our results, to lower peak viscosity (Holas and Tipples, 1978), gelatinization temperature (Morrison et al., 1994), and gelatinization enthalpy (Eliasson and Larsson, 1993) and to increase swelling power and starch solubility (Tester and Morrison, 1994). Besides, it would not be clear why most of the observed changes occur during the first drying stages while starch damage occurs also during extrusion.

Cubbada et al. (1987, 1988) suggested a change in the physicochemical character (such as the change in permeability of starch during high-temperature drying of pasta). Our results indicate a more rigid starch granule

with lower solubility and swelling capacity after high-temperature pasta drying. They thus point to a loss of permeability as already suggested by Cubbada et al. (1987, 1988). However, more research is needed to further reveal the properties of this drying associated change in the starch physicochemical character.

ABBREVIATIONS USED

DSC, differential scanning calorimetry; ΔH, gelatinization enthalpy; ΔT, gelatinization range; HT, high temperature; LT, low temperature; RH, relative humidity; RVA, rapid visco analyzer; RVU, rapid visco units; S_{amy}, amylose solubility; S, total solubility; SP: swelling power; T_c, conclusion temperature of gelatinization; T_g, gelatinization temperature; T_o, onset temperature of gelatinization; T_p, peak temperature of gelatinization; UCD, Chopin Dubois units; VHT-HM, very high temperature applied at high moisture content.

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