Effects of Starch Isolation, Drying, and Grinding Techniques on Its Gelatinization and Retrogradation Properties

L. A. Grant[1]

Cereal Chem. 75(5):590–594

ABSTRACT

The effects of two different methods of starch isolation, drying, and grinding on gelatinization and retrogradation properties were investigated. Starch was isolated from whole wheat and flour of four hard red spring wheat cultivars. Portions of each starch isolate were freeze-dried or air-dried and portions of each dried starch were ground using a mortar and pestle or a Wiley Jr. mill. Less starch damage was obtained for freeze-dried starch regardless of isolation method or grinding technique and for all starches derived from whole wheat. Highest starch damage was obtained for air-dried starch isolates. Wiley-milled starch isolates showed higher water-binding. Whole wheat starch isolates had higher peak, lower trough, and lower final viscosities, as determined by starch paste viscosity analysis, than did starch isolates derived from flour. Major effects of all treatments on differential scanning calorimetry gelatinization properties showed lower onset temperature for flour starch isolates, lower peak temperature for freeze-dried starches, and no effects due to grinding. Endotherms of all starches after refrigerated storage and freeze-thaw cycling were lower than those for gelatinization.

A necessary prerequisite to starch characterization research is the isolation of starch granules from plant tissue without inadvertent modification. Equally important are the subsequent steps of drying and grinding the starch before physical and chemical analysis. In the literature, spanning many decades, two major techniques of starch extraction are often cited. These two procedures are grain-steeping and dough-washing. Grain-steeping, as implied, is the process of soaking whole grain in water (room temperature or cold) with or without the use of chemicals to inhibit enzyme activity. Softening the grain in this manner avoids the starch granule damage normally acquired during dry milling of wheat to flour. Flour is the starting material for the dough-washing method of starch extraction.

Although starch has been isolated for nearly 100 years by one or the other of these two techniques, with many modifications over time, direct comparisons are almost nonexistent. The most recent comparison of starch isolation methods was conducted by Hart and Blanshard (1982). Using one winter wheat, they studied the effects of two starch extraction methods on starch gelatinization behavior. They compared the grain-steeping method of Banks and Greenwood (1975), and the dough-washing method of Wolf (1964), as modified by Bowler et al (1980). The resultant starch samples obtained were air-dried, but no mention of a grinding method was made.

Drying of starch isolates has received more attention in published literature than has grinding of the isolates. Most studies are from the early to late 1960s. Whistler et al (1958, 1959) isolated starch from corn using a wet-milling procedure and studied the effects of three types of drying on the physical properties and chemical reactivity of the granules. The three methods examined were freeze-drying, air-oven drying at 45°C, and alcohol dehydration. The authors suggested that all starch granules dried in air, in particular, but also those dried by certain other means, are physically stressed. They found that air-drying set up strains within the granules and reduced water penetrability of the granule surface due to what they called a “case hardening” effect. Leach (1965) commented that drying starch, even under the gentlest conditions, caused the granules to shrink and crack, and upon rehydration, dried starch did not resume its fully native state. In 1968, Williams and Hlynka reported that some alterations in the properties of starch may take place upon subjecting flour to freeze-drying. They postulated that the sudden addition of water to freeze-dried flour caused enormous stresses in the starch granule and resulted in a significant increase in starch damage.

More recently, Uriyapongson and Rayas-Duarte (1994), using wet and dry-wet milling methods with different levels of abrasion treatments, found starch damage was higher in freeze-dried amaranth starch. However, it was unclear from which milling method the freeze-dried starch was obtained. If the starch was from the dry-wet-milled amaranth, which had received the higher level of abrasion, the higher starch damage may not be from the freeze-drying but rather from the isolation method.

The objective of this study was to compare the two most commonly used starch isolation methods as well as drying and grinding methods used to prepare laboratory-isolated starch for chemical and physical analysis. The major reason for this study was to ascertain the ideal methods for obtaining starch isolates, those that show the least alteration to the starch granules.

MATERIALS AND METHODS

Samples

Four hard red spring wheat cultivars were used for this study. A portion of each wheat was cleaned and tempered to 14% moisture and milled into flour using a Buhler experimental mill according to established laboratory procedures.

Starch Isolation from Whole Wheat

Starch was isolated from the whole grain using the method of Wolf (1964) with minor modifications. Whole wheat (800 g) was steeped in an excess of distilled water at 4°C for 48 hr. During this time, the water was changed every 12 hr to keep the pH within 6.0–6.5 to retard microbial growth and impede enzyme activity. The softened kernels were drained over a sieve and crushed using a Hobart mixer (Hobart Mfg. Co., Troy, OH) equipped with a food grinder using a die with 1/8-in. diameter holes. The crushed wheat kernels were kneaded by hand in distilled water until a gluten ball was formed and the wash water was clear of starch. The starch slurry was passed first through a No. 50 mesh sieve to remove larger particles of bran and bits of gluten and then through six layers of cheese cloth to remove smaller bran contaminants. The sieved and strained slurry was centrifuged at 2,000 × g for 20 min. The solubles were decanted, tailings removed, and starch reslurried. The

---

[1] Research Chemist, USDA-ARS Hard Red Spring and Durum Wheat Quality Laboratory, North Dakota State University, Fargo, ND 58105. E-mail: lgrant@badlands.nodak.edu Fax: 701/239-1377. Names are necessary to report factually on available data; however, the USDA neither guarantees nor warrants the standard of the product, and the use of the name by the USDA implies no approval of the product to the exclusion of others that may also be suitable.

Publication no. C-1998-0803-04R.

starch was purified by repeated centrifugation and resuspension in distilled water.

Starch Isolation from Flour

The dough ball washing method of Walden and McConnell (1955) was used to isolate starch from 800 g of flour from each of the four samples.

Starch Drying

One-half of the amount of each starch isolate (both methods) was spread in glass cake pans (9 × 13 in.) and allowed to air-dry at room temperature (≈25°C) for three days. During the air-drying process, the starch was periodically mashed and turned with a spatula to speed drying and prevent the starch from drying in a hard mass. The remaining one-half of each starch isolate (both methods) was spread in aluminum cake pans, placed in the freezer immediately after isolation, frozen, and then freeze-dried for three days with no heat applied to the freeze-drier shelves.

Starch Grinding

One-half of each of the dried starches was ground with a mortar and pestle and passed through a No. 70 mesh sieve. The other one-half was ground using a Wiley Jr. mill (Thomas Scientific, Swedesboro, NJ) equipped with a No. 60 mesh sieve.

Starch Damage

An enzymatic digestion assay kit (Megazyme International, Wicklow, Ireland) was used to determine starch damage using the method of Gibson et al (1992).

Water-Binding Capacity

The water-binding capacity of the starches was determined using the procedure of Yamazaki (1953) as modified by Medcalf and Gilles (1965).

Starch Pasting Properties

Pasting properties of the starches were examined using a Rapid ViscoAnalyser (RVA) (Newport Scientific, Narrabeen, Australia) interfaced with a personal computer equipped with Thermocline and Thermoview software (Newport Scientific). The method utilized was modeled after that of Walker et al (1988) with minor modifications. Starch (3 g, db) was added to preweighed deionized distilled water in an RVA canister to achieve a total weight of 28 g. The temperature profile consisted of equilibrating the starch slurry at 50°C for 1 min, raising the temperature to 95°C at a rate of 6°/min, holding the temperature at 95°C for 5 min, and lowering the temperature to 50°C (6°/min) for the remainder of the run. Total run time was 25 min.

Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) studies were performed using a DSC 7 (Perkin-Elmer Corp., Norwalk, CT) equipped with a digital DEC-425 thermal analysis data station. The instrument was calibrated using indium and purified, deionized distilled water as standards.

Gelatinization. Starch (3.5 mg, dwb) was weighed directly into aluminum pans, followed by the addition of 8 μL of purified, deionized distilled water. The pans were hermetically sealed and allowed to equilibrate at room temperature overnight. A sealed aluminum pan containing 8 μL of purified, deionized distilled water was used as a reference. Samples were heated from 40 to 130°C at a scanning rate of 10°C/min. Enthalpy (ΔH) and onset (T_o), peak (T_p), and conclusion (T_c) temperatures were computed automatically. The gelatinization temperature range (T_r) was computed as $[2(T_p - T_o)]$, as described by Krueger et al (1987). An average of at least three thermograms was used for each starch.

Refrigerated-storage retrogradation. The method of White et al (1989), briefly described here, was used to measure refrigerated-storage retrogradation. Aluminum pans containing the DSC gelatinized starches were stored at 4°C for seven days. The samples were equilibrated at room temperature (25°C) for 2 hr before being heated

TABLE I
Starch Damage (%) and Water-Binding Capacity (%) of Starch Isolated from Whole Wheat and Flour[a]

	Treatment			
	FD-MP	FD-W	AD-MP	AD-W
Starch damage				
Whole wheat	0.49a	0.58a	0.93b	1.69b
Flour	2.40c	2.51c	2.71d	3.77e
Water-binding capacity				
Whole wheat	73.8a	75.8a	71.2a	74.8a
Flour	82.6b	84.1b	80.2c	85.1b

[a] Mean of four analyses each on duplicate samples expressed on an as-is basis. Comparisons followed by the same letter are not significantly different ($P < 0.05$) within the same row and between whole wheat and flour for each test. FD = freeze-dried, MP = ground with mortar and pestle, AD = air-dried, W = milled with Wiley mill.

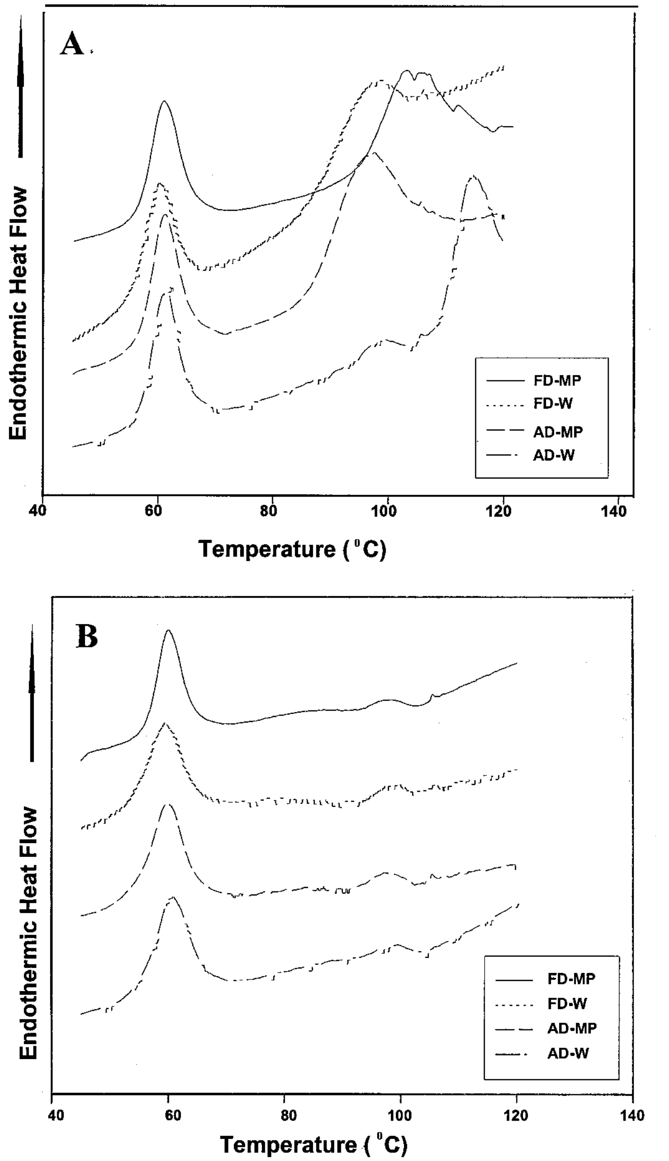

Fig. 1. Differential scanning calorimetry thermograms of whole wheat (**A**) and flour (**B**) freeze-dried and air-dried starch isolates. FD-MP = freeze-dried, mortar and pestle ground, FD-W = freeze-dried, Wiley milled, AD-MP = air-dried, mortar and pestle ground, AD-W = air-dried, Wiley milled.

in the DSC using the same conditions as for gelatinization. The retrogradation peak was analyzed by the data station and the results were compared with those of the gelatinization peak. The enthalpy value, which represents the energy required to break down the retrograded starch, was expressed as a percentage of that required to gelatinize the starch sample, as described by Paton (1987).

Freeze-thaw stability. The method of White et al (1989) was used to measure freeze-thaw stability. Ten freeze-thaw cycles were performed on gelatinized starches before they were heated in the DSC. The peak was analyzed by the data station, and the enthalpy was expressed as a percentage of the original starch gelatinization, as described above.

Statistical Analysis

The two isolation methods, two drying techniques, and two grinding methods on the four wheat cultivars resulted in a total of 32 samples of starch. All analyses were done in at least triplicate and averaged. Data were statistically analyzed using one-way repeated-measures analysis of variance and the pairwise multiple comparison method (Tukey test) (Sigma Stat Statistical Software, version 2.0 and Sigma Plot, version 3, Jandel Scientific, San Rafael, CA).

RESULTS AND DISCUSSION

Analytical Data

The moisture content of the starches reflected the method of drying. The freeze-dried starches were essentially dry when removed from the freeze drier, but the air-dried starches could be dried only to the ambient relative humidity of the room. Because the starches were isolated over a period of several weeks, small differences in moisture content could be the result of daily fluctuations of temperature and humidity of the drying area. Moisture contents ranged from 2.4 to 2.8% and from 12.7 to 13.7% for the freeze-dried and air-dried starches, respectively. Protein, expressed as percent nitrogen, was similar for all starches, indicating that both isolation procedures produced starch of relatively high purity. Protein content ranged from 0.04 to 0.06% for all samples. The repeated centrifugation and resuspension of the starch resulted in very little protein contamination.

Starch Damage

Means of starch damage and water-binding capacity of the samples are shown in Table I. Significant differences in starch damage ($P < 0.05$) were obtained between the freeze-dried and air-dried starches derived from both whole wheat and flour. During the air-drying process, the starch is continually divided into smaller and smaller pieces with a spatula. This continual physical manipulation or crushing may have contributed, in part, to higher starch damage for these samples. But the more likely cause of the high starch damage is the extensive grinding required for air-dried starches, when fully dry, to produce the particle size needed to pass through a No. 70 mesh sieve. Conversely, the freeze-dried starches were loose and light in texture when removed from the freeze-drier and required essentially no energy to grind. As expected, higher starch damage values were obtained for the starches isolated from flour than for those from whole grain, because mechanical damage was imparted during the milling process. Likewise, starches ground using the Wiley mill had high starch damage caused by the aggressive treatment of the starch granules.

TABLE II
Pasting Properties (RVU) of Starch from Whole Wheat and Flour[a]

	Treatment			
	FD-MP	FD-W	AD-MP	AD-W
Peak				
Whole wheat	313.8a	310.3a	347.0b	343.3b
Flour	308.8a	313.0a	336.8c	330.0c
Trough				
Whole wheat	157.8a	155.0a	159.3a	159.8a
Flour	164.8a	164.0a	167.5a	168.3a
End viscosity				
Whole wheat	397.8a	390.5a	393.0a	395.8a
Flour	416.5b	416.3b	419.8b	415.3b

[a] Mean of four analyses each on duplicate samples expressed on a dry basis. Comparisons followed by the same letter are not significantly different ($P < 0.05$) within the same row and between whole wheat and flour for each test. FD = freeze-dried, MP = ground with mortar and pestle, AD = air-dried, W = milled with Wiley mill, RVU = Rapid ViscoAnalyser units.

Water-Binding Capacity

The flour starch isolates, collectively, had statistically greater water-binding capacity than the whole-grain starch isolates (Table I). These data concur with the higher starch damage values for these starches. This relationship would be expected because higher starch damage is conducive to greater amounts of water being bound. Starches ground with a mortar and pestle had lower water-binding capacity values than starches ground using the Wiley mill. These values were not statistically significant except for the air-dried starches obtained from flour and ground with a mortar and pestle. In our laboratory, starch is normally air-dried and ground using a mortar and pestle. We have found that starch treated in this manner generally produces lower water-binding capacity values than have been reported in the literature (Medcalf and Gilles 1965).

Starch Pasting Properties

Means of starch pasting properties, as measured by the RVA, are shown in Table II. Peak viscosity was higher for all air-dried starch, particularly the air-dried starches derived from whole wheat. Statistically significant differences for peak viscosity were found between air-dried and freeze-dried samples from both whole-wheat and flour starch isolates. Concomitantly, significant differences were found between the two grinding methods for air-dried flour and whole-wheat starch isolates. Whistler et al (1959) reported higher peak viscosity for starches with higher moisture content. The average moisture contents of the air-dried and freeze-dried starches for this study were 13.0 and 3.3%, respectively. However, the pasting properties of these samples were determined on a moisture-free basis; therefore the differences observed were probably a result of the

TABLE III
Gelatinization Properties[a] of Starch Isolated from Whole Wheat and Flour as Determined by Differential Scanning Calorimetry[b]

	Treatment			
Properties	FD-MP	FD-W	AD-MP	AD-W
T_o				
Whole wheat	55.4a	55.6a	56.4a	56.2a
Flour	53.9b	53.9b	55.0c	55.1c
T_p				
Whole wheat	60.1a	60.0a	60.8b	60.5b
Flour	59.3ac	59.6ac	60.4bd	60.5bd
T_c				
Whole wheat	71.1a	71.7a	72.4a	72.8a
Flour	72.2a	72.5a	72.5a	73.1a
T_r				
Whole wheat	9.3a	8.9a	8.9a	8.9a
Flour	10.9b	11.4b	10.8b	10.9b
ΔH_g				
Whole wheat	9.62a	9.62a	9.91a	9.91a
Flour	9.41a	9.37a	9.66a	9.41a

[a] T_o, T_p, and T_c = onset, peak and conclusion temperatures (°C), respectively; T_r = gelatinization range, calculated as $[2(T_p - T_o)]$; ΔH_g = enthalpy of gelatinization (J/g).

[b] Mean of four analyses each on duplicate samples expressed on a dry basis. Comparisons followed by the same letter are not significantly different ($P < 0.05$) within the same row and between whole wheat and flour for each test. FD = freeze-dried, MP = ground with mortar and pestle, AD = air-dried, W = milled with Wiley mill.

drying treatment, which may or may not be compounded by starch damage.

Although not statistically significant, the trough viscosity of the whole-wheat starch isolates was lower than for the flour-derived starch isolates (Table II). Trough viscosity indicates the degree of starch breakdown or shear stability, although the viscosity of wheat starch is too low to cause significant shear and breakdown. Final viscosity was similar for all samples isolated from whole wheat and all samples isolated from flour, but the whole-wheat starch isolates had statistically significant lower final viscosities than that of the flour starch isolates. Low final viscosities indicate a slower rate of retrogradation. The method of isolation appeared to have a greater effect on the viscosity of the whole-wheat starch isolates than on that of the flour starch isolates.

DSC

Gelatinization. Means of DSC gelatinization properties of the starches are shown in Table III. For both methods of isolation, the onset of gelatinization (T_o) was slightly higher for the air-dried starches. This difference was statistically significant for the flour starch isolates but not for the whole-wheat starch isolates. T_o was lower by 1–1.5°C for the starches isolated from flour than from starch isolated from whole wheat (Table III). This difference may be due to the higher percentage of damaged starch in the flour starch isolates (Table I). There appeared to be no differences in T_o due to grinding method.

Peak temperature (T_p) was ≈1ºC lower for the freeze-dried starches than for the air-dried starches derived from flour. This difference concurs with the trend observed for the RVA peak viscosity data (Table II). Whistler et al (1959) reported that corn starches with the lowest moisture content gelatinized somewhat more slowly (showed lower peak viscosity) than starches with higher moisture contents. The freeze-dried starches in our study were the more completely dried starches. Consequently, our results agreed with those earlier findings. In another study, Whistler et al (1958) showed that the method and extent of drying corn starch controlled the number of cavitated starch granules obtained, and therefore these granules would differ from solid granules in pasting behavior. Perhaps the process of freeze-drying and subsequent rehydration of the starch with water during the tests caused some physical changes in the starch. Leach (1965) stated that, upon rehydration, starch did not resume its fully native form and that the realigned and reorganized starch was dissimilar from its original form. There appeared to be no statistically significant effect of grinding methods on T_p.

Although not statistically significant, the gelatinization temperature range (T_r) for the whole-wheat starch isolates was lower than that for the flour starch isolates. A lower temperature range $[2(T_p - T_o)]$ gives a sharp DSC peak, and a higher range gives a low, broad peak (Fig. 1). Temperature range is directly affected by onset temperature.

Enthalpy of gelatinization (ΔH, J/g) indicates the amount of energy required to gelatinize the starch. The values, which ranged from 9.37 to 9.91 J/g for all starches regardless of treatment, were typical values obtained for native wheat starch (White et al 1989). Hart and Blanshard (1982) reported a ΔH value of 9.46 J/g for starch isolated from flour and a lower ΔH value (8.07 J/g) for starch isolated from whole wheat. Biliaderis et al (1980) stated that ΔH values were significantly affected by heating rate, amount of water used, and starch damage. Stevens and Elton (1971) attributed differences in ΔH to differences in starch damage. They found that, as starch damage increased, ΔH decreased. In general, these differences were not significant in our study. Our data showed that the ΔH was similar for all starches (Table III) and indicated little, if any, treatment effects.

Refrigerated storage retrogradation. When starch is gelatinized and stored at a low temperature (4°C) for a period of time, the starch molecules reassociate, but in weaker molecular and structural forms than in the native molecules (White et al 1989). Evidence of this is manifest in the markedly higher T_r means and much lower T_o, T_p, and T_c means of all of the starches (Table IV) when compared to the gelatinization means (Table III). White et al (1989) observed similar endothermic differences in native and com-

TABLE IV
Refrigerated Storage Retrogradation Properties[a] of Starch Isolated from Whole Wheat and Flour as Determined by Differential Scanning Calorimetry[b]

Properties	Treatment FD-MP	FD-W	AD-MP	AD-W
T_o				
Whole wheat	42.9a	46.0b	44.9c	41.0d
Flour	42.3a	43.1ba	44.7ca	42.0da
T_p				
Whole wheat	51.4a	54.0b	53.1c	50.4d
Flour	51.3a	51.5a	52.4c	51.0d
T_c				
Whole wheat	62.8a	63.8a	63.7a	63.8a
Flour	62.7a	62.4a	61.3a	63.0a
T_r				
Whole wheat	17.0a	15.9a	16.3a	18.7a
Flour	18.0a	16.8a	15.6a	18.1a
ΔH_r				
Whole wheat	2.63a	3.26a	3.18a	3.26a
Flour	2.97a	2.34a	2.38a	3.22a
$\Delta H_r / \Delta H_g$				
Whole wheat	27.2a	33.7a	32.2a	33.1a
Flour	31.4a	25.0a	24.5a	34.0a

[a] T_o, T_p, and T_c = onset, peak and conclusion temperatures (°C), respectively; T_r = gelatinization range, calculated as $[2(T_p - T_o)]$; ΔH_r = enthalpy of retrogradation (J/g); $\Delta H_r/\Delta H_g$ = difference between enthalpy of retrogradation and enthalpy of gelatinization (%).

[b] Mean of four analyses each on duplicate samples expressed on a dry basis. Comparisons followed by the same letter are not significantly different ($P < 0.05$) within the same row and between whole wheat and flour for each test. FD = freeze-dried, MP = ground with mortar and pestle, AD = air-dried, W = milled with Wiley mill.

TABLE V
Freeze-Thaw Retrogradation Properties[a] of Starch Isolated from Whole Wheat and Flour as Determined by Differential Scanning Calorimetry[b]

Properties	Treatment FD-MP	FD-W	AD-MP	AD-W
T_o				
Whole wheat	44.4a	44.9a	43.9a	44.1a
Flour	43.9a	45.3a	45.2a	44.4a
T_p				
Whole wheat	51.1a	50.9a	51.2a	51.3a
Flour	51.4a	51.2a	51.4a	51.5a
T_c				
Whole wheat	62.4a	64.7a	63.5a	63.2a
Flour	64.6a	63.8a	64.0a	64.0a
T_r				
Whole wheat	13.7a	13.6a	14.7a	14.6a
Flour	14.0a	13.9a	14.2a	14.2a
ΔH_{ft}				
Whole wheat	6.23a	6.19a	6.23a	6.32a
Flour	6.56a	6.44a	6.61a	6.56a
$\Delta H_{ft}/\Delta H_g$				
Whole wheat	64.5a	64.4a	63.3a	63.8a
Flour	69.6a	69.1a	68.6a	69.8a

[a] T_o, T_p, and T_c = onset, peak and conclusion temperatures (°C), respectively; T_r = gelatinization range, calculated as $[2(T_p - T_o)]$; ΔH_{ft} = enthalpy of freeze thaw (J/g); $\Delta H_{ft}/\Delta H_g$ = difference between enthalpy of freeze-thaw and enthalpy of gelatinization (%).

[b] Mean of four analyses each on duplicate samples expressed on a dry basis. Comparisons followed by the same letter are not significantly different ($P < 0.05$) within the same row and between whole wheat and flour for each test. FD = freeze-dried, MP = ground with mortar and pestle, AD = air-dried, W = milled with Wiley mill.

mercially modified starches that had been refrigerated for seven days. The differences between the air-dried and freeze-dried starches, before storage (Table III) are not evident following storage. Statistically, there were significant differences between drying and grinding methods for the whole-wheat starch isolates for DSC onset and peak temperatures but not for the flour starch isolates. Although not statistically significant, slight differences were obtained for the flour starch isolates for temperature range, enthalpy of refrigerated storage gels (ΔH_r), and difference between the enthalpy of refrigerated storage and enthalpy of gelatinization ($\Delta H_r/\Delta H_g$). The lower ΔH_r indicated that the energy required to break down the recrystallized starch molecules was less than the energy required to gelatinize the original starches. The difference (%) between ΔH_r and ΔH_g suggests that certain samples showed more retrogradation than others by their lower percentage values.

Freeze-thaw stability. Table V shows mean freeze-thaw retrogradation properties of the starch samples. All DSC values obtained for all the starches after 10 freeze-thaw cycles were lower than the original gelatinization means (Table III) but slightly higher than the means obtained for the refrigerated-storage gels (Table IV). There was an increase in ΔH for the freeze-thaw starch gels (ΔH_{ft}) compared to the values for ΔH_r, indicating that a higher amount of energy was required to melt the recrystallized starch. This may be due to the insoluble aggregates that form during reassociation of starch gels that have been subjected to freeze-thaw cycling (White et al 1989). There were no statistically significant differences between the whole-wheat and flour starch isolates for ΔH_{ft} and $\Delta H_{ft}/\Delta H_g$.

CONCLUSIONS

Starch damage, measured by enzyme assay, was substantially lower for starch isolated from macerated whole wheat kernels as opposed to starch isolates from dry-milled flour. Starch damage was also lower for freeze-dried starch and starch ground using a mortar and pestle. These observations would be expected because of the less aggressive handling of the starch. Flour starch isolates had greater water-binding capacity as a result of their higher starch damage, which would also be an expected result. Pasting properties (by RVA) showed that all air-dried starches have higher peak viscosities than freeze-dried starches. Freeze-drying appeared to have the greatest overall effect on the starch isolates, particularly those derived from whole wheat. Trough and final viscosity of these starch isolates were most affected by the freeze-drying process.

The major effects of all treatments on DSC gelatinization properties were: lower onset temperature (T_o) by 1–1.5°C for flour starch isolates, slightly lower peak temperature (T_p) for freeze-dried starches, similar enthalpy of gelatinization (ΔH) for all starches, and no effects attributable to grinding. The DSC endotherms of all starches after refrigerated storage were lower than those after gelatinization and freeze-thaw cycling. Significant differences were obtained between drying and grinding methods for the whole-wheat starch isolates for DSC onset and peak temperatures.

No statistically significant effects were observed for all treatments of starch isolates subjected to freeze-thaw cycling.

The results of this investigation demonstrate the importance of isolation and subsequent handling of starch to be used in scientific research. It appears that freeze-drying starch does not increase damage already imparted to the starch through milling but does change some of the pasting and gelatinization characteristics.

ACKNOWLEDGMENT

I gratefully acknowledge the technical assistance of Angela Ostenson.

LITERATURE CITED

Banks, W., and Greenwood, C. T. 1975. Starch and Its Components. Edinburgh University Press: Edinburgh, Scotland.

Biliaderis, C. G., Maurice, T. J., and Vose, J. R. 1980. Starch gelatinization phenomena studied by differential scanning calorimetry. J. Food Sci. 45:1669-1674.

Bowler, P., Williams, M. R., and Angold, R. E. 1980. A hypothesis for the morphological changes which occur on heating lenticular wheat starch in water. Starch/Staerke 32:186-189.

Gibson, T. S., Qalla, H., and McCleary, B. V. 1992. An improved method for the measurement of starch damage in wheat flour. J. Cereal Sci. 15:15-27.

Hart, P. E., and Blanshard, J. M. V. 1982. The effect of extraction method upon the gelatinization behavior of wheat starch granules. Starch/Staerke 34:293-296.

Krueger, B. R., Knutson, C. A., Inglett, G. E., and Walker, C. E. 1987. A differential scanning calorimetry study on the effect of annealing on gelatinization behavior of cornstarch. J. Food Sci. 52:715-718.

Leach, H. W. 1965. Gelatinization of starch. Pages 289-307 in: Starch: Chemistry and Technology, Vol. I. Fundamental Aspects. R. L. Whistler and E. F. Paschall, eds. Academic Press: New York.

Medcalf, D. G., and Gilles, K. A. 1965. Wheat starches. I. Comparison of physicochemical properties. Cereal Chem. 42:558-568.

Paton, D. 1987. Differential scanning calorimetry of oat starch pastes. Cereal Chem. 64:394-399.

Stevens, D. J., and Elton, G. A. H. 1971. Thermal properties of the starch/water system. I. Measurement of heat of gelatinization by differential scanning calorimetry. Staerke 23:8-11.

Uriyapongson, J., and Rayas-Duarte, P. 1994. Comparison of yield and properties of amaranth starches using wet and dry-wet milling processes. Cereal Chem. 71:571-577.

Walden, C. C., and McConnell, W. B. 1955. Studies on the techniques for reconstituting flours. Cereal Chem. 32:227-236.

Walker, C. E., Ross, A. S., Wrigley, C. W., and McMaster, G. J. 1988. Accelerated starch-paste characterization with the Rapid Visco-Analyzer. Cereal Foods World 33:491-494.

Williams, P. C., and Hlynka, I. 1968. A note on the influence of high-vacuum drying on the starch of Canadian wheat flours. Cereal Chem. 45:280-285.

Whistler, R. L., Spencer, W. W., Goatley, J. L., and Nikuni, Z. 1958. Effect of drying on the presence of cavities in corn starch granules. Cereal Chem. 35:331-336.

Whistler, R. L., Goatley, J. L., and Spencer, W. W. 1959. Effect of drying on the physical properties and chemical reactivity of corn starch granules. Cereal Chem. 36:84-90.

White, P. J., Abbas, I. R., and Johnson, L. A. 1989. Freeze-thaw and refrigerated-storage retrogradation of starches. Starch/Staerke 41:176-180.

Wolf, M. J. 1964. Wheat starch. Pages 6-9 in: Methods in Carbohydrate Chemistry, Vol. 4. R. L. Whistler, ed. Academic Press: New York.

Yamazaki, W. T. 1953. An alkaline water retention capacity test for the evaluation of cookie baking potentialities of soft winter wheat flours. Cereal Chem. 30:242-246.

[Received December 22, 1997. Accepted May 21, 1998.]