

# Starch Characteristics of Waxy and Nonwaxy Tetraploid (*Triticum turgidum* L. var. durum) Wheats

L. A. Grant,<sup>1,2</sup> N. Vignaux,<sup>3</sup> D. C. Doehlert,<sup>1</sup> M. S. McMullen,<sup>3</sup> E. M. Elias,<sup>3</sup> and S. Kianian<sup>3</sup>

## ABSTRACT

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Manufacture of pasta products is paramount for durum wheat (*Triticum turgidum* L. var. durum). The recent development of waxy durum wheat containing starch with essentially 100% amylopectin may provide new food processing applications and present opportunities for value-added crop production. This investigation was conducted to determine differences in some chemical and functional properties of waxy durum starch. Starch was isolated from two waxy endosperm lines and four nonwaxy cultivars of durum wheat. One of the waxy lines (WX-1) was a full waxy durum wheat whereas the other line (WX-0) was heterogeneous, producing both waxy and nonwaxy seed. Effects on starch swelling, solubility, pasting, gelatinization, and retrogradation were examined. The full waxy

starch had four times more swelling power than the nonwaxy durum starches at 95°C, and was also more soluble at three of the four temperatures used. Starch pasting occurred earlier and peak viscosities were greater for starches from both waxy lines than for the nonwaxy starches, but their slurries were less stable with continued stirring and heating. Greater energy was required to melt gelatinized waxy starch gels, but no differences were found in either refrigerated storage or freeze-thaw retrogradation, as determined by differential scanning calorimetry. The results of this investigation showed some significant differences in the starch properties of the waxy durum wheat lines compared to the nonwaxy durum wheats.

Waxy mutants, lacking the functional waxy protein (granule-bound starch synthase [GBSS]), have been identified in several plant species, including maize, rice, barley, sorghum, and amaranth. Waxy hexaploid wheats (*Triticum aestivum* L.) have been developed either through crossbreeding or mutation (Nakamura et al 1995; Yamamori et al 1995; Yasui et al 1997; Kiribuchi-Otobe et al 1997). GBSS has been cited as the key enzyme in the synthesis of amylose (Preiss 1991). The absence of GBSS results in almost complete elimination of amylose. Waxy starch containing 100% amylopectin has different functional and structural properties than normal starch. Waxy hexaploid wheats attain peak viscosities more rapidly and at lower temperature than nonwaxy wheats (Kiribuchi-Otobe et al 1997; Hayakawa et al 1997). X-ray analysis of waxy wheats showed a tightly bound, compact structure due to the higher percentage of amylopectin. Differential scanning calorimetry (DSC) peak temperatures and enthalpies of gelatinization were higher than for nonwaxy wheats (Fujita et al 1998).

Waxy tetraploid wheat (*Triticum turgidum* L. var. durum) development efforts are currently being increased. The objective of this investigation was to examine the starch characteristics of waxy and nonwaxy durum wheats to ascertain differences in starch properties.

## MATERIALS AND METHODS

### Wheat Samples

Four nonwaxy durum wheat cultivars (Ben, Munich, Mountrail, and Lebsock), a full waxy line (WX-1), and a mixed waxy and nonwaxy line (WX-0) were grown at two North Dakota locations (Langdon and Prosper) during the 1999 growing season. A complete randomized block design with four replicates was used in both locations. Planting, field fertility, agronomic data collection, and harvest were in accordance with the North Dakota State University durum breeding program. The waxy durums were derived from a

cross of the partial waxy (Graybosch et al 1998) hard red winter wheat 'Ike' (Martin et al 1994) and the durum 'Ben' (Elias and Miller 1998). Ben was used as the pollen parent. The F1 seed was planted in the greenhouse. Some spikes were allowed to self pollinate to generate F2 seed that was segregating for the full waxy trait. Full waxy seeds were selected from F2 seed by iodine staining of half seeds (Nakamura et al 1995). Half seeds (containing the embryo) expressing the full waxy phenotype were placed in petri plates on top of water-saturated blotter paper and placed in a refrigerator at 4°C for five days to overcome dormancy. After the five-day pre-germination treatment, plates were returned to room temperature. Seed germinated within a day or two and were planted by hand in pots of soil and grown to maturity in the greenhouse. Seed set of F2 plants was poor because of poor pollen fertility. The W-0 line was derived from a particularly productive F2 plant derived from the original Ike×Ben cross. The WX-0 line was originally selected as a full waxy line but analyses in this study indicated that the seed was mixed: half waxy and half nonwaxy. F1 plants from the Ike×Ben cross were used as the female parent and backcrossed to Ben because of the poor pollen fertility of the F1 plants. The F1 seed from the first backcross were grown to maturity. Plants heterozygous for the waxy-null trait in both genomes (A and B) were identified by iodine staining of the pollen. In heterozygous plants, the waxy trait is expressed in one out of four pollen grains, because of the haploid pollen condition. Full waxy F2 seeds from the heterozygous F1 plants were selected, germinated, and cultivated as before. The WX-1 line was selected from other F2-derived lines from the same cross largely because of its yield potential. Each of the six wheat genotypes was milled into semolina using a Quadrumat Jr. mill according to established laboratory procedure. Each wheat sample (≈400 g) was first tempered to 12.5%, then to 15.5% moisture 48 and 24 hr before milling, respectively. The tempered wheat was passed through a Quadrumat Jr. mill equipped with only corrugated rolls and sieved 1 min on a U.S. 35 sieve. The material remaining on the sieve was passed through the mill a second time and sieved again, after which the throughs were combined with the first sievings. The overs were discarded.

### Proximate Analysis

Test weight was determined on cleaned wheat using Approved Method 84-10 (AACC 2000). Kernel size distribution was determined on 100 g of cleaned wheat according to the method of Shuey (1960). Moisture of the semolina samples was determined by Approved Method 44-15A, and protein was measured by Approved Method 46-30 (FP428, Leco Corp., St. Joseph, MI).

<sup>1</sup> USDA-ARS Hard Red Spring and Durum Wheat Quality Laboratory, North Dakota State University, Fargo, ND 58105. 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.

<sup>2</sup> Corresponding author. Fax: 701-239-1377. E-mail: grantl@fargo.ars.usda.gov

<sup>3</sup> Department of Plant Science, North Dakota State University, Fargo, ND 58105.

Enzymatic digestion assay kits (Megazyme Int., Wicklow, Ireland) were used to determine starch damage using the method of Gibson et al (1992) and  $\alpha$ -amylase activity using the method of McCleary and Sheehan (1987). Falling number of the semolina samples was determined by Approved Method 56-81B (AACC 2000).

### Starch Isolation

Starch was isolated from 50 g of each semolina sample unless sample size was limited, in which case 30 g was used. A 1:2 ratio of semolina to distilled water was mixed for 3 min in a Waring blender at low speed. The slurry was transferred to centrifuge bottles and centrifuged at  $2,000 \times g$  for 20 min. The supernatant, which represented the water solubles, was decanted through four layers of cheesecloth into freeze-drier flasks, shell frozen, and freeze-dried. The sediment remaining in the bottles was combined in a 500-mL beaker, distilled water was added, and the starch washed out by hand. The resultant starch slurry was passed through a U.S. 70 sieve. The material remaining on top of the sieve was continuously rinsed with small amounts of distilled water until no starch remained. The starch slurry was centrifuged ( $2,000 \times g$  for 20 min), the supernatant discarded, and the sediment combined and reslurried with distilled water and centrifuged again. The prime starch recovered was air-dried for one day, ground using a mortar and pestle, and sieved through a U.S. 70 sieve. Starch and water soluble materials were obtained from one sample of each semolina and percent recovery recorded.

Percent amylose and amylopectin of the starches was determined using a method developed for HPLC by Grant and Ostenson (*unpublished data*). Starch was initially defatted using Approved Method 30-25 (AACC 2000) with the exception that methanol was used in place of petroleum ether. Defatted starch (20 mg) was solubilized by adding 4.5 mL of 1M KOH and 0.5 mL of 6M urea and heating at  $100^\circ\text{C}$ , under nitrogen, for  $\approx 90$  min according to the method described by Morrison and Laignelet (1983). Laboratory-isolated hard red spring wheat amylose and amylopectin (Montgomery and Senti 1958) were used as standards. After heating, 1 mL of the sample was neutralized with 1 mL of HCl and filtered through a 13-mm diameter,  $0.45\text{-}\mu\text{m}$  hydrophilic polyvinylidene fluoride (PVDF) syringe filter before analysis. Amylose and amylopectin were separated on a Waters ultrahydrogel linear column ( $6\text{--}13\ \mu\text{m}$ ,  $7.8 \times 300\ \text{mm}$ ) using an HPLC (HP1090, Hewlett Packard, Agilent Technologies, Wilmington, DE). All samples were analyzed at  $45^\circ\text{C}$  with filtered, deionized, distilled water as eluent.

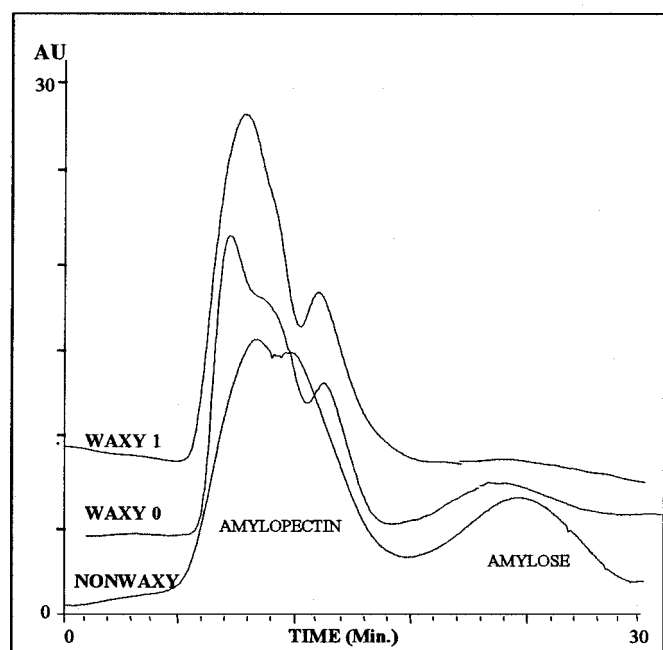


Fig. 1. HPLC chromatograms of waxy and nonwaxy durum wheat starches.

Starch (0.5 g) was dissolved in 1N NaOH as described by Lansky et al (1949). Intrinsic viscosity of the starches was determined according to the method of Leach (1963). A semimicroviscometer (Ubbelohde, Technical Glass Products, Dover, NJ) with a capillary size of 100 (3.0–15 cSt) was equipped with an automatic viscosity timer (Jupiter Instruments, Jupiter, FL). Flow times of the starch diluted to four different concentrations (0.125, 0.167, 0.25, and 0.5% w/v) were measured at  $25^\circ\text{C}$  and used to calculate intrinsic viscosity, which was reported as inherent viscosity versus concentration. The swelling power and solubility of the starches were analyzed using the procedure of Leach et al (1959). The temperatures used were 55, 65, 75, and  $95^\circ\text{C}$ .

### Pasting Properties

Pasting properties of the starch samples were evaluated using a Rapid Visco Analyser (RVA) (Newport Scientific, Narrabeen, Australia) interfaced with a computer equipped with ThermoLine and Thermoview software (Newport Scientific). The method used 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  $50^\circ\text{C}$  for 1 min, raising the temperature to  $95^\circ\text{C}$  at  $6^\circ/\text{min}$ , holding the

TABLE I  
Protein Concentration, Falling Number, Starch Damage, and  $\alpha$ -Amylase Activity of Nonwaxy and Waxy Durum Semolina<sup>a</sup>

Sample	Protein (%) <sup>b</sup>	Falling Number (sec) <sup>b</sup>	Starch Damage (%) <sup>c</sup>	$\alpha$ -Amylase Activity (U/g) <sup>c</sup>
Langdon				
WX-0	13.8c	63b	4.8b	0.29a
WX-1	14.2bc	62b	5.6a	0.25b
Munich	14.8ab	379a	3.9c	0.07c
Ben	15.1a	371a	3.7c	0.09c
Mountrail	14.3bc	367a	3.8c	0.09c
Lebsock	14.6ab	409a	3.8c	0.09c
Prosper				
WX-0	15.9b	62b	3.9b	0.46a
WX-1	15.9b	62b	6.2a	0.53a
Munich	16.7a	319a	3.3c	0.19b
Ben	16.0b	274a	3.6b	0.28b
Mountrail	15.9b	278a	3.6b	0.20b
Lebsock	15.5b	292a	3.6b	0.24b

<sup>a</sup> Values followed by the same letter in the same column are not significantly different ( $P < 0.05$ ).

<sup>b</sup> 14% moisture basis.

<sup>c</sup> As-is basis.

TABLE II  
Recovery of Starch and Water Solubles from Nonwaxy and Waxy Durum Wheats, and Amylose Content and Intrinsic Viscosity of Isolated Starches<sup>a</sup>

Sample	Prime Starch (%) <sup>b</sup>	Water Solubles (%) <sup>b</sup>	Amylose Content (%)	Intrinsic Viscosity (%)
Langdon				
WX-0	45.9b	6.2b	15b	1.62b
WX-1	39.0c	8.2a	6c	0.96c
Munich	50.3a	4.5c	28a	2.66a
Ben	50.3a	4.3c	30a	2.69a
Mountrail	48.1ab	4.9c	27a	2.89a
Lebsock	49.2a	4.6c	28a	3.08a
Prosper				
WX-0	38.7b	6.6b	13b	2.13a
WX-1	37.2b	8.9a	0c	1.26b
Munich	43.4ab	5.4c	24a	2.55a
Ben	45.2ab	5.1c	25a	2.57a
Mountrail	43.2ab	5.2c	24a	2.46a
Lebsock	48.8a	4.9c	26a	2.45a

<sup>a</sup> Values followed by the same letter in the same column are not significantly different ( $P < 0.05$ ).

<sup>b</sup> Dry basis.

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 (DSC 7, Perkin-Elmer Corp., Norwalk, CT) in conjunction with a digital DEC-425 thermal analysis data station. The instrument was calibrated using indium and purified, deionized distilled water as standards.

Starch (3.5 mg, db) 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 of gelatinization ( $\Delta H$ ) and onset ( $T_o$ ) and peak ( $T_p$ ) temperatures were computed automatically. The gelatinization temperature range ( $T_i$ ) 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.

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

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 by 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

Data were statistically analyzed using one-way analysis of variance (ANOVA) and pairwise multiple comparison (Tukey test) (Statistix 7, Analytical Software, Tallahassee, FL). All data were collected in at least triplicate and averaged unless otherwise noted. Because of the poor quality of the wheat from the Prosper location, locations were analyzed separately and no statistical comparisons were made across locations.

## RESULTS AND DISCUSSION

There were differences in the physical appearance of the wheat obtained from the two locations. The wheat grown at Langdon had an average test weight of 58.3 lb/bu, whereas the grain from Prosper averaged 50.5 lb/bu. Test weight has been interpreted as a measure of kernel soundness and as one wheat quality criterion. Kernel size distribution of the wheats from the two locations also were different. Averages of 81.0 and 45.8% large kernels were obtained for wheat from Langdon and Prosper, respectively. The low test weights and small percentage of large kernels obtained

**TABLE III**  
Starch Paste Analysis of Nonwaxy and Waxy Durum Wheat Starches<sup>a</sup>

Starch Sample	Peak Time (min)	Rapid Visco Analyser Units (RVU)				
		Peak Viscosity	Trough	Final Viscosity	Breakdown	Setback
Langdon						
WX-0	6.0b	233d	177b	249b	6b	72b
WX-1	3.2c	351a	133c	163c	219a	30c
Munich	6.4a	258c	189b	305a	69b	116a
Ben	6.7a	271bc	214a	311a	57b	97ab
Mountrail	6.6a	278b	219a	338a	59b	119a
Lebsock	6.4a	284b	217a	334a	66b	117a
Prosper						
WX-0	6.4a	208c	172b	229c	37c	58c
WX-1	3.3b	311a	102c	127d	209a	26d
Munich	6.5a	253b	193a	310ab	61b	117ab
Ben	6.6a	246b	192a	303b	54b	111b
Mountrail	6.6a	250b	200a	323a	50b	124a
Lebsock	6.5a	255b	195a	315ab	60b	121a

<sup>a</sup> Dry basis. Values followed by the same letter in the same column are not significantly different ( $P < 0.05$ ).

**TABLE IV**  
Swelling Power and Solubility of Nonwaxy and Waxy Durum Wheat Starches<sup>a</sup>

Starch Sample	Pasting Temperature (°C)							
	Swelling Power (g/g)				Solubility (%)			
	55	65	75	95	55	65	75	95
Langdon								
WX-0	2.7b	6.6b	12.0b	17.3b	1.6ab	4.1b	7.9b	22.6b
WX-1	2.8b	10.3a	23.6a	44.4a	3.3a	6.8a	13.5a	51.2a
Munich	2.7b	5.8b	6.9c	8.1c	0.7b	3.1c	5.3c	10.2c
Ben	2.7b	5.8b	6.9c	8.0c	1.1ab	2.7c	4.5c	8.7c
Mountrail	3.1a	6.1b	6.9c	8.2c	0.9b	2.6c	4.4c	7.4c
Lebsock	2.8b	5.9b	7.1c	10.3c	1.3ab	3.1c	5.2c	19.6b
Prosper								
WX-0	2.5a	5.4b	11.4b	24.7b	1.4a	4.8b	10.1b	29.6b
WX-1	2.7a	7.7a	21.5a	41.4a	3.3a	7.8a	15.1a	54.0a
Munich	2.6a	5.3b	7.0c	9.1c	1.9a	3.1c	3.3c	12.1c
Ben	2.6a	5.4b	6.8c	9.1c	1.1a	2.0c	4.0c	15.3c
Mountrail	2.7a	5.7b	6.8c	9.8c	1.7a	2.4c	3.3c	14.5c
Lebsock	2.5a	5.4b	6.9c	9.8c	1.2a	2.1c	3.9c	24.1b

<sup>a</sup> Values followed by the same letter in the same column are not significantly different ( $P < 0.05$ ).

for the wheat grown at Prosper indicated poor quality grain. Protein concentration (Table I) was 1–2% lower for the Langdon wheat in comparison with the wheat from Prosper but was within acceptable limits for the manufacture of pasta products. Statistically significant differences in protein concentration were found between WX-0 and three of the nonwaxy wheats (Munich, Ben, and Lebsock) for the wheat grown at the Langdon location. In contrast, the only differences observed for the wheat grown at Prosper was with Munich, which had the highest protein concentration.

In general, the falling number determinations (Table I) suggested some sprout damage, particularly for the wheat grown at the Prosper location. Values <400 sec are generally indicative of some degree of sprouting (Donnelly 1980). The low values obtained for WX-0 and WX-1 are reportedly an inherent trait of waxy wheat that does not necessarily indicate sprout damage (Graybosch et al 2000).

In addition to the lower falling number, the wheat grown at Prosper also had higher  $\alpha$ -amylase activity (Table I), indicating the presence of sprout damage. The wheat grown at the Langdon location showed no statistical differences for amylase activity among the four nonwaxy cultivars but there were significant differences between the four nonwaxy cultivars and the two waxy lines. For the wheat grown at the Prosper location, however, there was statistical difference only between the waxy and nonwaxy wheats. Starch damage was similar for all wheat samples from each location, except the WX-1 wheat, which was  $\approx$ 1–2% higher. There was a statistically significant difference between WX-1 and all other wheat starches for both locations. Bettge et al (2000) reported higher starch damage in full waxy starches. They suggested that the physical granule structure of waxy starch may have less structural integrity under stress than normal starch. Therefore, the actual milling of waxy wheat would impart more damage to the starch granules than it would for nonwaxy wheat.

### Starch Isolation

There were statistically significant differences for starch recovery for the samples derived from the Langdon-grown wheats but not so for those grown at Prosper, except for Lebsock (Table II). The least amount of starch was isolated from the full waxy durum samples. Significantly higher amounts of water-soluble materials were obtained from the waxy lines than from the nonwaxy durum from both locations. This was particularly true for WX-1 wheats. The higher amounts of water-soluble materials obtained from the waxy wheats may be due to higher amounts of small molecular weight nonstarchy polysaccharides or  $\beta$ -glucans present in these wheats, or perhaps higher amounts of damaged starch granules. Concomitantly, the percentage of total starch was similar for all cultivars

and lines from both locations (data not shown). Averages were 71.6 and 70.0% for Langdon and Prosper, respectively. These values were in good agreement with the 63–72% reported for American wheat cultivars (Pomeranz and MacMasters 1968; Cerning and Guilbot 1974). In contrast to this study, Yasui et al (1999) reported lower total starch content for waxy hexaploid wheat lines.

### Amylose to Amylopectin Ratio

The percentage of amylose obtained for the starch samples is shown in Table II. The WX-0 starches from both locations had approximately half the amount of amylose normally present in wheat starch; whereas, the WX-1 starches had little to no amylose present (Fig. 1). From these data, it appeared that WX-0 was heterogeneous for the waxy phenotype. This was confirmed when  $\approx$ 40% of wheat kernels (cut in half, stained blue with iodine solution) indicated the presence of amylose. Typical HPLC chromatograms of WX-0, WX-1, and nonwaxy durum wheat starches are overlaid in Fig. 1 to show differences in the amylose and amylopectin peaks. The double amylopectin peaks, most noticeable for the waxy samples, are due to degradation of amylopectin over time. This event, according to Morrison and Karkalas (1990), is unavoidable and does not change the overall amount of amylopectin in the peak. Our objective was to characterize the starch of the waxy lines and compare them with the nonwaxy cultivars. Therefore, we decided not to eliminate the WX-0 line because it showed some distinct differences from the full waxy line as well as the nonwaxy samples.

Intrinsic viscosities of WX-1 wheat starches (Table II) were significantly lower than for the WX-0 and nonwaxy durum wheat starches. For the starches derived from the Langdon location, WX-0 was significantly lower than the nonwaxy starches from that same location. These differences, however, were not present for the starches derived from the Prosper location. These results agreed with a lower range of intrinsic viscosity ( $\eta$  0.46–1.64) obtained for waxy rice reported by Juliano (1984).

### Pasting Properties

Significant differences in the pasting properties of WX-0 and WX-1 starches and between the waxy and nonwaxy durum wheat starches are shown in Table III. WX-1 starches had the highest peak viscosity and lowest final viscosity of all the starches examined. Peak time for these starch samples occurred 3 min earlier than for the other starches. Similar results have been obtained for waxy hexaploid wheat (*T. aestivum* L.) (Hayakawa et al 1997; Sasaki et al 2000). The earlier peak time of WX-1 starches depicts the first stage of gelatinization, normally not evident on wheat starch profiles unless carboxymethyl cellulose (CMC) is used (Crossland and Favor

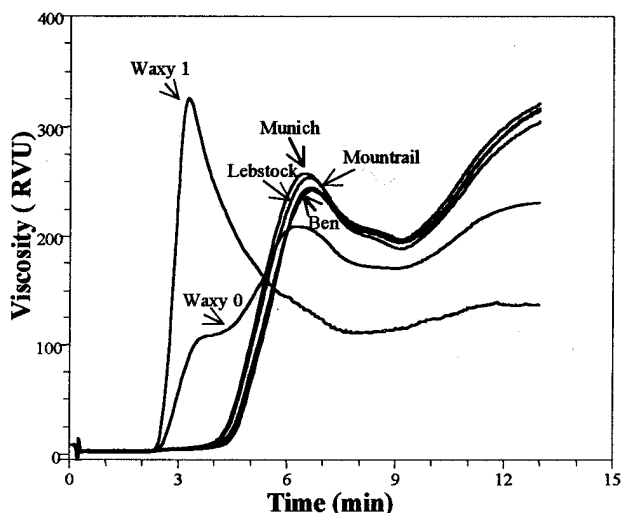


Fig. 2. Pasting properties of starch isolated from waxy and nonwaxy durum wheats.

TABLE V  
Gelatinization Properties of Starch Isolated from Nonwaxy and Waxy Durum Wheats<sup>a,b</sup>

Starch Sample	$T_o$ (°C)	$T_p$ (°C)	$T_r$ (°C)	$\Delta H_g$ (J/g)
Langdon				
WX-0	54.3b	60.8b	12.9a	10.8b
WX-1	56.2a	62.5a	12.4a	13.0a
Munich	53.3bc	59.6c	10.6b	10.7b
Ben	53.7bc	58.9cd	10.4b	10.5b
Mountrail	53.0c	58.0d	9.9b	10.5b
Lebsock	53.3bc	58.6d	10.5b	10.4b
Prosper				
WX-0	55.7b	62.2b	12.9a	12.1b
WX-1	56.9a	63.6a	13.2a	13.5a
Munich	55.9b	61.0c	10.2b	10.5c
Ben	55.1bc	60.4d	10.7b	10.4c
Mountrail	54.4c	59.4e	9.9b	10.5c
Lebsock	55.4bc	60.4d	10.0b	10.5c

<sup>a</sup> Dry basis. Values followed by the same letter in the same column are not significantly different ( $P < 0.05$ ).

<sup>b</sup>  $T_o$  and  $T_p$  = onset and peak temperatures, respectively;  $T_r$  = gelatinization range, calculated as  $2(T_p - T_o)$ ;  $\Delta H_g$  = enthalpy of gelatinization.

1948). The higher peak viscosity may be due to the absence of amylose because amylose suppresses swelling and maintains starch granule integrity (Hermansson and Svegmak 1996). The WX-0 starch with intermediate levels of amylose showed the first stage of gelatinization (Fig. 2). Leach et al (1959) reported two-stage swelling for waxy sorghum, which he attributed to two sets of bonding forces within the starch granule. The rapid swelling of waxy starch is a property of amylopectin. Starch granules composed only of amylopectin degrade quickly at lower temperatures because they cannot maintain the stability of paste viscosity (Tester and Morrison 1990). This was evident with the dramatic drop in viscosity (breakdown) and low final viscosity of these starch samples. The WX-0 starches had peak times similar to those of the nonwaxy starches but had lower peak and final viscosities. This was obviously due to the intermediate levels of amylose. The low setback of the WX-1 starches is due to lack of amylose, which recrystallizes irreversibly during cooling of normal starches and creates a gel.

### Swelling Power and Solubility

Swelling power of all starch samples increased as temperature increased (Table IV). All starches from both locations were similar at the lowest temperature (55°C) and, in general, similar at 65°C, with the exception of the WX-1 starches, which were significantly different from the other starches. Starch swelling power is attributed to the strength and character of the micellar network within the starch granule (Stone and Lorenz 1984). Beyond 65°C, the waxy character of the WX-0 and WX-1 starches became more pronounced. Tester and Morrison (1990) reported that the amylopectin fraction of starch was responsible for swelling power. Swelling power doubled and then quadrupled at 75 and 95°C for the WX-1 starches, indicating a loosely bonded micellar structure due to the absence of amylose. The absence of the amylose-lipid complex found in normal wheat starch, which acts as an inhibitor of starch swelling, may be another factor for increased swelling of waxy wheat starches. The WX-0 starches had higher swelling power than the nonwaxy starches due to higher amounts of amylopectin and less amylose in these starches. Waxy starches appeared to swell unrestrictedly and were much more soluble than nonwaxy wheat starches.

Similar to swelling power, solubility of the starches (Table IV) increased as temperature increased. Significant differences were generally found between WX-0 and WX-1 and between the waxy and nonwaxy starches at the higher temperatures (65, 75, and 95°C) for samples derived from both locations. Of the nonwaxy starches, Lebsack showed unusually high values at 95°C compared with the other nonwaxy starches. The values for Lebsack were similar to those for the WX-0 starches for both locations.

### Thermal Properties

DSC gelatinization properties of the starch samples are shown in Table V. Higher onset and peak temperatures and enthalpy of gelatinization were obtained for the WX-0 and WX-1 starches from both locations. All measurements showed statistically significant differences between WX-0 and WX-1 for except temperature range ( $T_i$ ) for each location. Similar results were reported by Sasaki et al (1999) and Gudmundsson and Elliasson (1992) for waxy hexaploid wheat and waxy barley starches, respectively. Amylopectin plays a major role in starch crystallinity (Flipse et al 1996). Starch containing high amounts of amylopectin has less amorphous and more crystalline regions, which in turn raises gelatinization temperature and endothermic enthalpy. Enthalpy of gelatinization reflects the loss of molecular order. Krueger et al (1987) stated that without amylose-rich amorphous regions, more energy is needed to initiate melting. Waxy starches, therefore, exhibit higher enthalpy of gelatinization. There was a significant difference between the WX-1 starch and the nonwaxy starches for  $\Delta H$  but no significant differences between WX-0 and the nonwaxy starches for the Langdon. For samples from Prosper, there were significant differences between both the WX-0 and WX-1 starches and the nonwaxy starches.

In general, the values obtained for refrigerated storage and freeze-thaw retrogradation (data not shown) indicated little to no effect on the retrogradation of the starch gels. Schoch (1967) reported superior freeze-thaw stability for waxy rice but not for waxy sorghum or waxy corn. In contrast, our study did not show any effect of the waxy trait on freeze-thaw retrogradation.

## CONCLUSIONS

Semolina derived from waxy tetraploid wheats had low falling numbers when compared with the nonwaxy semolina samples. This was not attributed to sprout damage, even though  $\alpha$ -amylase values indicated some sprout damage was present for all samples. Less prime starch and more water-soluble materials were isolated from waxy wheat semolinas. This may be due in part to reportedly higher  $\beta$ -glucans and nonstarchy polysaccharides. Starch pasting properties (by RVA) showed higher peak viscosities, earlier peak times, and lower stabilities and final viscosities for the full waxy starches. These properties have been reported for other waxy cereal starches as well. High starch swelling power and solubility were characteristic of the WX-0 and WX-1 starches. DSC results indicated that waxy durum wheat starches gelatinized at higher  $T_o$  and  $T_p$  temperatures and required more energy (J/g) to melt the gelatinized starch gels ( $\Delta H$ ), but no differences between the waxy and nonwaxy durum starches were observed for refrigerated storage and freeze-thaw retrogradation.

The results of this investigation demonstrate that waxy durum starch possess some dramatically different starch properties that may provide potential new uses for this specialty crop.

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