

Physicochemical and Structural Characteristics of Flours and Starches from Waxy and Nonwaxy Wheats¹

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ABSTRACT

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A waxy spring wheat (*Triticum aestivum* L.) genotype was fractionated into flour and starch by roller and wet-milling, respectively. The resultant flour and starch were evaluated for end-use properties and compared with their counterparts from hard and soft wheats and with commercial waxy and nonwaxy corn (*Zea mays* L.) starches. The waxy wheat flour had exceptionally high levels of water absorption and peak viscosity compared with hard or soft wheat flour. The flour formed an intermediate-strength dough that developed rapidly and was relatively susceptible to mixing. Analysis by differential scanning calorimetry and X-ray diffractometry

showed waxy wheat starch had higher gelatinization temperatures, a greater degree of crystallization, and an absence of an amylose-lipid complex compared with nonwaxy wheat. Waxy wheat and corn starches showed greater refrigeration and freeze-thaw stabilities than did nonwaxy starches as demonstrated by syneresis tests. They were also similar in pasting properties, but waxy wheat starch required lower temperature and enthalpy to gelatinize. The results show analogies between waxy wheat and waxy corn starches, but waxy wheat flour was distinct from hard or soft wheat flour in pasting and mixing properties.

Starch, the major component in the wheat (*Triticum aestivum* L.) kernel, affects quality and staling properties of wheat-based products. Starch granules are composed of two types of glucose polymers, the essentially linear amylose and the highly branched amylopectin, and other minor components such as proteins and lipids. The structure of amylose and amylopectin and their contents in starch granules determine pasting, gelation, and retrogradation properties of starch, and hence product quality and stability. Eating quality of Japanese and Australian noodles, for example, was negatively correlated with amylose content of wheat flour (Oda et al 1980; Miura and Tanii 1994; Wang and Seib 1996). The low content of amylose is associated with high noodle quality and can be predicted from alleles at genetic loci encoding the granule-bound starch synthase (waxy protein), the key enzyme in amylose biosynthesis (Miura and Tanii 1994). A reduced level of amylose also is associated with high values of swelling power in wheat flours or starches (Wang and Seib 1996; Sasaki and Matsuki 1998). Swelling power is a simple test that measures changes in starch granule volume due to swelling when starch is heated with excess water, and is a recognized indicator of noodle quality (Crosbie 1991). Staling of bread (Krog et al 1989) and expansion of extruded snack foods (Wang 1997) were also dependent on the content of amylose and amylopectin.

Several studies have shown that waxy wheat starches behave differently from wild-type wheat starch when heated in excess water and during cooling and storage. Waxy (<5% amylose, null three waxy alleles) and partially waxy (≈12% amylose, null two waxy alleles) wheat starches demonstrated higher onset and peak gelatinization temperatures, enthalpy of gelatinization, and degree of crystallization than did nonwaxy wheat starch (Yasui et al 1996; Demeke et al 1999). Waxy and partially waxy wheat starches also exhibited greater retrogradation resistance and freeze-thaw stability compared with nonwaxy wheat starches (Hayakawa et al 1997; Yuan and Thompson 1998; Sasaki et al 2000). These characteristics are important quality attributes for chilled and frozen starchy foods. Nevertheless, little information is available with regard to physicochemical properties of waxy wheat flours. Such information is needed to assist in identifying end-uses for waxy wheat. A study showed waxy flours had lower pasting temperature, peak viscosity, and α -

amylase activity than their nonwaxy parent (Yasui et al 1999). The present study was undertaken to investigate physicochemical properties of waxy wheat flour in relation to its starch characteristics. The properties of waxy wheat flour were compared with those obtained from hard and soft wheat that were grown in the same environment. Starches were isolated and purified from waxy and nonwaxy wheat, and their pasting and thermal properties were evaluated and related to starch structure as determined by X-ray diffractometry and α -amylolysis. Commercial waxy and nonwaxy corn (*Zea mays* L.) starches were also included for comparison.

MATERIALS AND METHODS

Wheat Flours and Starches

A waxy, hard-kerneled, red-grained spring wheat genotype (CDC W2) selected from the cross Bai Huo/Kanto 107, was grown at Saskatoon, SK, Canada, during the summer of 1997 and was used in the present study. The hard red spring (HRS) (cv. Neepawa) and soft white spring (cv. AC Reed) wheats were grown in plots adjacent to the waxy wheat. The wheat grains were milled into flours on a Brabender Quadrumat Jr. flour mill (Brabender Co., South Hackensack, NJ) at flour yield averaging 69, 70, and 71% for waxy, hard, and soft wheat, respectively. The amylose content, determined by concanavalin-A precipitation of amylopectin (Gibson et al 1997), of waxy wheat grains was 3.6% of the total starch versus 27.3% in HRS wheat.

Starches were isolated from waxy and hard wheat grains by wet-milling. The grains were steeped in 0.5% sodium bisulfite solution at room temperature for ≈16 hr. The softened grains were drained and rinsed with distilled water and homogenized in a Waring blender for 2 min. The homogenate was passed through a 210- μ m polypropylene screen. The residue was homogenized two more times and the combined extracts passed through a 70- μ m polypropylene screen. The filtrate was allowed to sediment at 4°C and the supernatant was discarded. Starch was purified by suspending the granules in 0.05N NaOH solution for 1 hr with continuous agitation and centrifuged to remove the soluble proteins. The light brownish proteinaceous layer was scraped off. The starch was resuspended in distilled water and neutralized to pH 7.0 with 0.5N HCl and centrifuged. The starch was washed thoroughly on a Buchner funnel with distilled water, followed by 95% ethanol. The filter cake was dried overnight at 40°C. Native waxy and nonwaxy corn starches were purchased from Sigma Chemical Co. (St. Louis, MO).

Physicochemical Tests of Flour

Mixograph characteristics of wheat flours were measured on a mixograph interfaced with an IBM computer operated by Mixsmart

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software (National Mfg., Lincoln, NE) using 10.0 g of flour (14% moisture basis) and constant water absorption of 65%. The mixograph data is analyzed by the Mixsmart to obtain time to peak (maximum mixing resistance, min); peak height as a percentage of full scale (peak value, % torque); mixograph curve slope, an indication for mixing stability or dough tolerance (% torque/min); bandwidth, an indication of dough strength or weakness (% torque); and area under curve, representing the work required to develop dough (% torque × min). Farinograph, sedimentation, and Falling Number tests were determined by the Approved Methods 54-21, 56-60, and 56-81B, respectively (AACC 2000). The farinograph parameters included water absorption (%); arrival time (time required to reach 500 BU, min); peak or dough development time (min); dough stability (difference between departure and arrival time, min); and mixing tolerance index (MTI) (difference between top of the curve at peak and top of the curve measured 5 min after peak is reached, BU).

Analytical Tests of Starch

The granule shape and size of wheat and corn starches was examined by a scanning electron microscope (Phillips SEM 505) and image analyzer (BioQuant System IV, Image Technology, New York, NY) as described by Abdel-Aal et al (1997). Total starch, damaged starch, amylose, and α -amylase were determined using assay kits (Megazyme, Wicklow, Ireland). Crude protein (N × 5.7), crude fat, total ash, and moisture were analyzed by Approved Methods 46-13, 30-25, 08-01, and 44-15A, respectively (AACC 2000). Swelling power was based on the method of Sasaki and Matsuki (1998). Pasting properties of flours (12%, w/v, 14% moisture basis), with and without mercuric acetate (200 mg/500 mL) and silver nitrate (85 mg/500 mL) as amylase inhibitors, and starches (8%, w/v, dry basis) were measured using a viscoamylograph (model VA-1A, Brabender) equipped with a 700-cmg cartridge. The gel samples, obtained after cooling the cooked starch slurry to 50°C in the amylograph bowl, were analyzed for gel stability (degree of syneresis) under cold and frozen storage according to the method of Zheng and Sosulski (1998). Three types of exuded water were determined. Free water is the water separated by centrifugation from a freshly cooked starch gel, expelled water is the amount of water released from a gel after refrigeration or freeze-thaw, and absorbed water is the amount of water removed by centrifugation after removal of expelled water by decantation. Net syneresis was calculated as the difference between expelled and absorbed water content and free water content. The transition temperatures and gelatinization enthalpy of starches were measured using differential scanning calorimetry (DSC) (Mettler TA 3000, TA Instruments, New Castle, DE) as outlined previously (Abdel-Aal et al 1997). Amylolytic activity was determined as described by Singh et al (1982). The X-ray diffraction pattern of starch was recorded on a diffractometer (model 42273, Philips Analytical, Eindhoven, The Netherlands). Reagent-grade alumina powder (Anachemia, Lachine, QC) was used as an internal reference standard (reference peak at 38.5°, 2 θ) for measuring the relative intensity of starch diffraction peaks and providing an absolute diffraction angle (2 θ) reference position. The area under the peak of crystalline and amorphous regions was measured using a compensating polar planimeter and degree of relative crystallization was calculated according to Fujita et al (1998):

$$\text{Degree of crystallization (\%)} = I_c / (I_a + I_c) \times 100$$

where I_c = crystalline area on the X-ray diffractogram; I_a = amorphous area on the X-ray diffractogram.

The percent of relative intensity for the major peaks was calculated as:

$$\text{Percent relative intensity} = \text{starch peak height} / \text{alumina peak height} \times 100$$

The d -spacing was calculated from 2 θ according to Bragg's equation ($n\lambda = 2d \sin\theta$; where d = intercrystalline spacing, $n = 1$, and $\lambda = 1.54178\text{\AA}$).

Statistical Analysis

All samples were analyzed at least in duplicate. Data were subjected to analysis of variance using statistical software (v. 12, Minitab Inc, State College, PA). Means were compared by computation of least significant difference (LSD) values.

RESULTS AND DISCUSSION

Physicochemical Properties of Flours

Compared with a high-quality bread flour, waxy wheat flours formed intermediate strength doughs that require significantly less time and work to develop, as indicated by the mixograph and farinograph tests (Table I). The dough showed less stability and was sensitive to mechanical mixing. The mild strength of waxy flour also was indicated by an intermediate sedimentation volume when compared with hard wheat (Table I). The waxy wheat flours, however, absorbed significantly more water than did HRS or soft wheat flour. Several investigations have shown negative correlations between amylose content and water uptake or swelling power (Kovacs et al 1997; Sasaki and Matsuki 1998).

The viscoamylograph characteristics of waxy and nonwaxy wheat flours were measured on 12% slurries, with and without mercuric acetate as an amylase inhibitor (Table I). The pasting and peak temperatures of waxy flour were lower than those of HRS or soft wheat. Both temperatures were unaffected by the enzyme inhibitor except for peak temperature of waxy wheat, which decreased on addition of mercuric acetate. Jane et al (1999) and Hayakawa et al (1997) reported that waxy wheat starch had lower pasting temperatures, higher peak viscosity, and lower setback viscosity than non-waxy wheat. Waxy wheat flour exhibited the highest peak viscosity, followed by soft wheat, and finally HRS wheat. Higher peak viscosities in wheat starch are commonly associated with lower amylose contents (Zeng et al 1997). Peak viscosities of the three flours increased significantly when mercuric acetate was added to inactivate α -amylase. The effect of the enzyme inhibitor on peak viscosity of waxy flour was greater than with nonwaxy flours. For example, in the presence of inhibitor, the peak viscosity increased by 2.2× in waxy wheat compared with 1.7 and 1.5× in HRS and soft wheat, respectively. Another inhibitor (silver nitrate) was used to verify this finding (the enormous increase in peak viscosity of waxy wheat in the presence of mercuric acetate). Silver nitrate produced a similar effect on peak viscosity of waxy wheat. The peak viscosity of waxy wheat increased from 730 to 1,700 BU. When α -amylase activity was measured in the flours, slight differences were observed between waxy (0.16 Ceralpha units/g) and HRS wheat (0.14 Ceralpha units/g).

TABLE I
Physicochemical Properties of Waxy and Nonwaxy Wheat Flours^a

| Property | CDC W2 | Neepawa | AC Reed | LSD ₀₅ |
|-------------------------|-------------|-------------|-------------|-------------------|
| Mixograph | | | | |
| Peak time (min) | 1.4 | 2.3 | 1.8 | 0.5 |
| Peak value (%) | 59.9 | 61.7 | 50.4 | 5.5 |
| Slope (%/min) | -10.2 | -1.9 | -7.5 | 0.2 |
| Band width (%) | 14.4 | 22.7 | 9.4 | 0.4 |
| Area (%Tq × min) | 313 | 374 | 271 | 27 |
| Farinograph | | | | |
| Water absorption (%) | 76 | 67 | 60 | 8.8 |
| Arrival time (min) | 1.1 | 1.8 | 0.5 | 0.5 |
| Peak time (min) | 1.6 | 3.2 | 1.1 | 0.6 |
| Stability (min) | 1.2 | 9.4 | 2.0 | 0.2 |
| MTI (BU) | 150 | 30 | 120 | 3.6 |
| Sedimentation vol. (mL) | 31.9 | 56.7 | 17.1 | 1.1 |
| Viscoamylograph | | | | |
| Pasting temp. (°C) | 70.5 (70.5) | 72.0 (72.0) | 72.0 (73.5) | 1.5 (3.3) |
| Peak temp. (°C) | 82.5 (79.5) | 95.0 (95.0) | 95.0 (95.0) | 1.8 (1.8) |
| Peak viscosity (BU) | 730 (1600) | 420 (720) | 550 (810) | 80 (127) |
| Falling Number (sec) | 72 | 635 | 536 | 10 |

^a Values in parentheses indicate samples with enzyme inhibitor mercuric acetate. MTI = mixing tolerance index.

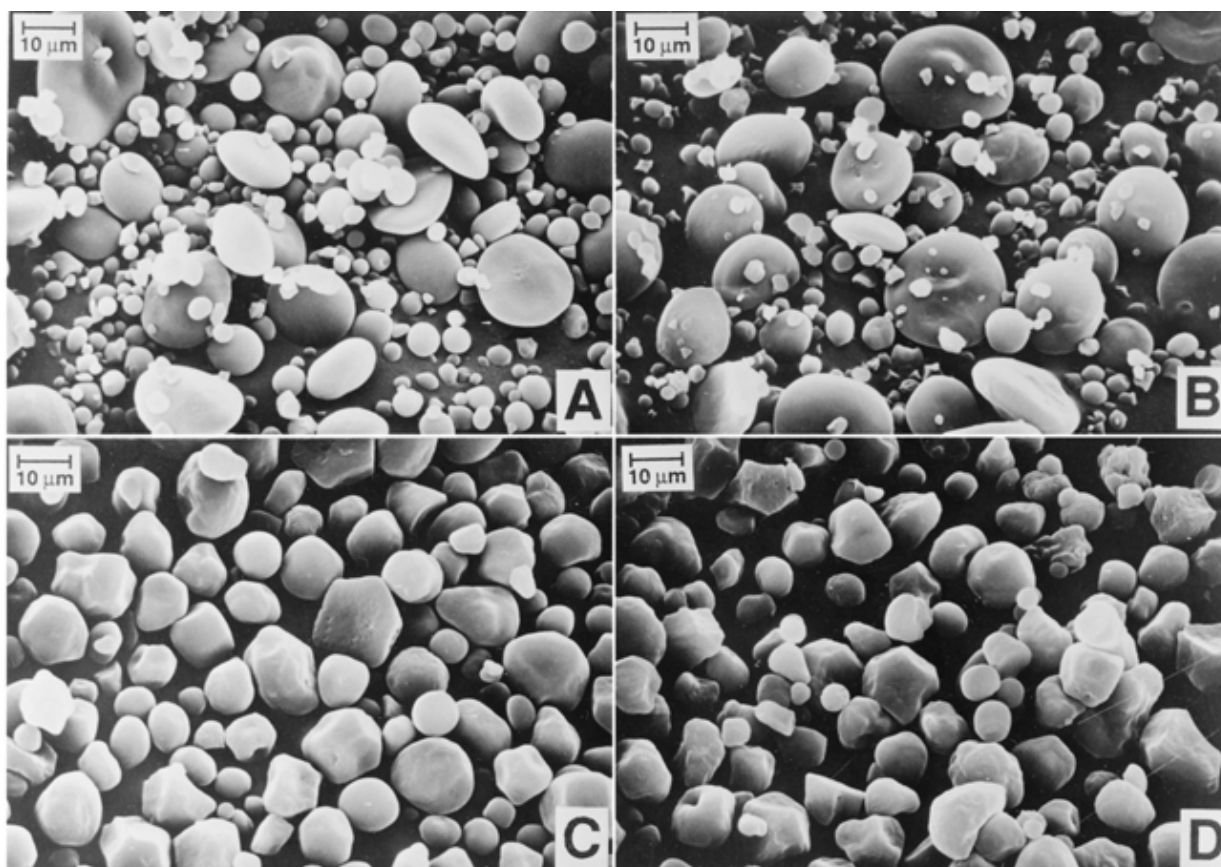


Fig. 1. Scanning electron micrographs of starches: (A) nonwaxy wheat, cv. Neepawa; (B) waxy wheat, line CDC W2; (C) nonwaxy corn; and (D) waxy corn.

TABLE II
Starch Extraction Efficiency and Starch Damage Content of Waxy and Nonwaxy Wheats (dry basis)^{a,b}

| Starch Type | Starch in Grain (%) | Starch Yield (g/100 g of grain) | Starch Extraction Efficiency (%) | Starch Damage (%) |
|-------------|---------------------|---------------------------------|----------------------------------|----------------------------|
| CDC W2 | 56.3 ± 1.84 | 53.8 ± 1.98 | 88.2 ± 2.12 | 0.79 ± 0.028 (4.24 ± 0.16) |
| Neepawa | 60.5 ± 1.70 | 56.1 ± 2.69 | 87.4 ± 2.47 | 0.44 ± 0.014 (3.97 ± 0.13) |

^a Mean ± standard deviation.

^b Values in parentheses indicate starch damage in flours.

TABLE III
Chemical Composition of Waxy and Nonwaxy Wheat and Corn Starches (dry basis)^a

| Starch Source | Starch (%) | Amylose (% of starch) | Protein (%) | Fat (%) | Ash (%) |
|---------------|-------------|-----------------------|-------------|--------------|--------------|
| Wheat | | | | | |
| CDC W2 | 92.4 ± 2.26 | 3.2 ± 0.14 | 0.33 ± 0.01 | 0.07 ± 0.01 | 0.04 ± 0.006 |
| Neepawa | 94.3 ± 2.69 | 26.9 ± 1.27 | 0.29 ± 0.01 | 0.09 ± 0.01 | 0.11 ± 0.01 |
| Corn | | | | | |
| Waxy | 92.3 ± 1.56 | 2.9 ± 0.14 | 0.34 ± 0.02 | 0.01 ± 0.001 | 0.07 ± 0.002 |
| Nonwaxy | 94.8 ± 2.69 | 21.0 ± 0.08 | 0.32 ± 0.02 | 0.01 ± 0.001 | 0.08 ± 0.003 |

^a Mean ± standard deviation.

TABLE IV
Free Water of Freshly Cooked Pastes and Syneresis of Expelled and Absorbed Water from Waxy and Nonwaxy Wheat and Corn Starch Gels After Seven Days of Cold and Frozen Storage (% gel wt)

| Starch Source | Free Water | Expelled Water | Absorbed Water | Net Syneresis |
|-----------------------------------|------------|----------------|----------------|---------------|
| After seven days storage at 4°C | | | | |
| CDC W2 | 4.39 | 0.00 | 4.52 | 0.14 |
| Neepawa | 2.81 | 1.03 | 3.95 | 2.17 |
| Waxy corn | 3.41 | 0.00 | 3.54 | 0.13 |
| Nonwaxy corn | 3.47 | 6.26 | 12.81 | 15.60 |
| LSD ₀₅ | 0.44 | 0.43 | 0.68 | 1.11 |
| After seven days storage at -15°C | | | | |
| CDC W2 | 4.39 | 0.00 | 4.98 | 0.56 |
| Neepawa | 2.88 | 5.48 | 54.95 | 57.55 |
| Waxy corn | 3.41 | 0.00 | 3.42 | 0.01 |
| Nonwaxy corn | 3.47 | 4.01 | 57.50 | 58.04 |
| LSD ₀₅ | 0.59 | 0.43 | 6.75 | 5.42 |

TABLE V
Differential Scanning Calorimetry (DSC) Properties of Waxy and Nonwaxy Wheat and Corn Starches

| Starch Type | Endothermic Transition Temperatures (°C) | | | | Enthalpy of Gelatinization (ΔH , J/g) |
|-------------------|--|----------------|----------------------|-----------------------|--|
| | Onset (T_o) | Peak (T_p) | Completion (T_c) | Range ($T_c - T_o$) | |
| Wheat | | | | | |
| CDC W2 | 55.6 | 66.0 | 79.6 | 24.0 | 11.8 |
| Neepawa | 54.6 | 62.5 | 73.4 | 18.8 | 11.5 |
| Corn | | | | | |
| Waxy | 59.8 | 71.7 | 83.5 | 23.7 | 16.7 |
| Nonwaxy | 57.1 | 70.0 | 80.5 | 23.4 | 12.7 |
| LSD ₀₅ | 1.8 | 1.0 | 2.1 | 0.6 | 0.5 |

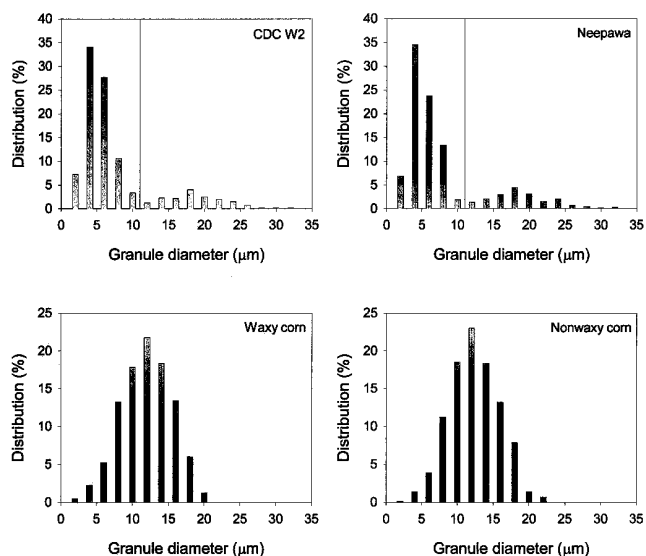


Fig. 2. Granule size distribution of waxy and nonwaxy wheat and corn starches.

This indicates that the dramatic increase in peak viscosity of waxy wheat is attributed to the waxy trait and not to sprout damage where the levels of α -amylase in waxy and nonwaxy wheat flours were somewhat similar. This finding warrants further investigation to study effects of these chemicals on pasting properties of waxy starches.

Falling Number (FN) is a measure of viscosity of flour slurries in seconds, with high values predictive of low α -amylase activity. The FN of waxy wheat flour averaged 72 sec compared with 635 and 536 sec for HRS and soft wheat flour, respectively (Table I). Similar results were reported by Graybosch et al (2000), who found an average falling number of 71 sec for 40 waxy wheat samples. When the test was run on 127 waxy wheat breeding lines, similar results were obtained. The absence of amylose, which becomes readily soluble and viscous at 95°C, and sensitivity of amylopectin to shearing action at higher temperatures (95°C), in addition to short time of heating (1 min), results in a low-consistency flour slurry in the FN test. This indicates that the FN test is not applicable to waxy wheat as an indirect measure of α -amylase activity, and other methods must be used. This finding was further confirmed when α -amylase activity was directly measured in waxy and nonwaxy wheat flours.

Starch Extraction, Composition, and Granule Size

Waxy wheat gave lower starch yield compared with HRS wheat due to a lower starch content (Table II). However, the starch extraction efficiency of waxy wheat was not significantly different from that of nonwaxy wheat, indicating similar recovery of starch. Isolation of starch from whole wheat grains resulted in lower levels of starch damage compared with starch damage in wheat flours. Waxy materials (starch and flour) had higher levels of starch damage compared with those of nonwaxy wheat. Waxy starch granules were

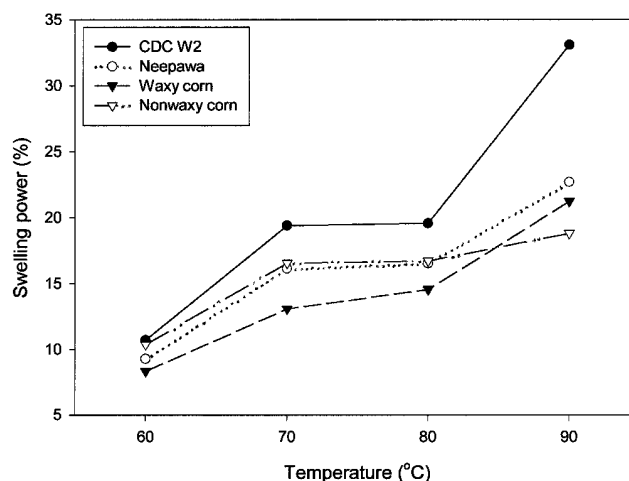


Fig. 3. Swelling power of waxy and nonwaxy wheat and corn starches.

less resistant to mechanical damage than normal starch granules (Bettge et al 2000). Wheat starches (laboratory prepared) were characterized by lower starch damage levels than those of commercial corn starches (0.79 vs. 1.87% in waxy starch and 0.44 vs. 1.78% in nonwaxy starch). Proximate composition of wheat starches and commercial corn starches indicated that they were somewhat similar in starch and protein contents, but wheat starches had higher levels of fat (Table III). Waxy wheat and waxy corn starches contained similar amount of amylose ($\approx 3\%$). Ash content of nonwaxy wheat starch was higher than that of waxy wheat. In general, composition of wheat starch was comparable to that reported by Abdel-Aal et al (1997) and Swinkels (1985).

There were no differences in granule shape between waxy and nonwaxy wheat starch or between waxy and nonwaxy corn starch (Fig. 1). Waxy and nonwaxy wheat starches contained two types of starch granules, type A-granule ($>10 \mu\text{m}$ in diameter) and type B-granule ($1\text{--}10 \mu\text{m}$ in diameter) as found in wheat species (Stoddard 1999). Waxy and nonwaxy corn starch granules exhibited one size distribution range of $3\text{--}22 \mu\text{m}$ (Fig. 2). The size distribution curve of corn starch granules showed a unimodal peak at $\approx 12 \mu\text{m}$, while wheat had two peaks, the first peak at $\approx 4 \mu\text{m}$ and the second peak at $\approx 18 \mu\text{m}$. The size distribution of wheat starch granules in both waxy and nonwaxy lines was very similar to that reported by Peng et al (1999).

Swelling and Pasting Properties

Nonwaxy wheat starch was similar to nonwaxy corn starch in swelling power at 60–80°C, but it had higher swelling power at 90°C (Fig. 3). Waxy corn starch showed unusually low swelling power compared with other starches. The swelling power of starches increased with temperature, with a sharp increase between 60 and 70°C for all starches and between 80 and 90°C for waxy wheat starch. This indicates a negative association between swelling power and amylose content that agrees with data reported by Sasaki and Matsuki (1998). It is noteworthy that waxy wheat starch had much

greater swelling power than nonwaxy wheat, particularly at high temperature (90°C). This property reflects the lack of amylose in waxy starch and could be used to identify waxy wheat lines in breeding programs.

Pasting characteristics of waxy and nonwaxy wheat and corn starches during cycles of heating and cooling are presented in Fig. 4. Both waxy starches swelled rapidly at a low temperature to peak viscosities between 1,100 and 1,200 BU, but then the pastes disintegrated quickly and there was low gel consistency due to the lack of amylose. In other words, waxy starches showed greater degrees of pasting and shear thinning compared with nonwaxy starches. Waxy starches also exhibited greater resistance to retrogradation as indicated by lower setback viscosities. These results agree well

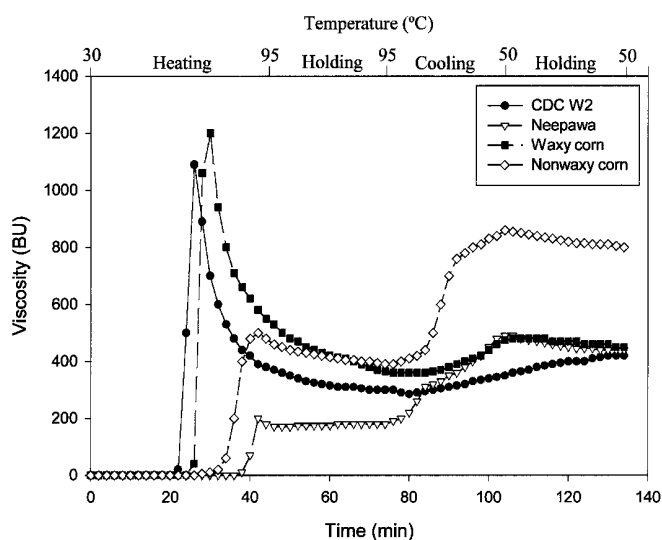


Fig. 4. Viscoamylograms of waxy and nonwaxy wheat and corn starches.

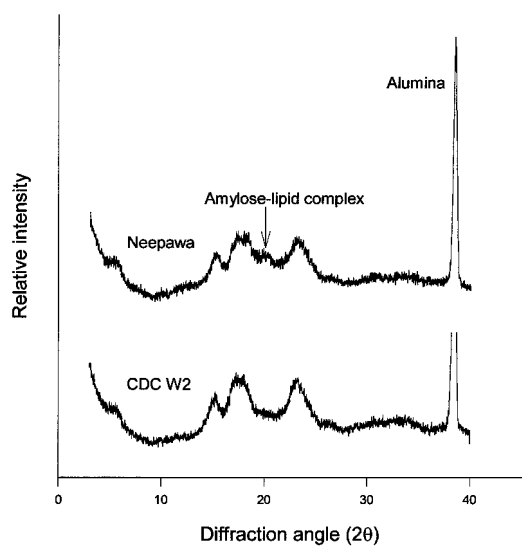


Fig. 5. X-ray diffractograms of waxy and nonwaxy wheat starches.

with other findings (Zeng et al 1997; Jane, et al 1999; Sasaki et al 2000). Slight differences were observed between waxy wheat starch and waxy corn starch in pasting properties, whereas there were marked differences between nonwaxy wheat and corn starch.

The stability of starch gels under cold and frozen storage was determined (Table IV). Free water separated from freshly prepared waxy starch pastes was significantly higher than from other starch pastes, with nonwaxy wheat fresh paste exuding the least amount of water. Zheng and Sosulski (1998), however, found that wheat flour pastes had a higher level of free water than nonwaxy corn starch. The total water separation of expelled and absorbed water from waxy starch gels after one week of refrigeration or one freeze-thaw cycle was lower than from nonwaxy starch gels. Waxy starch gels showed greater refrigeration and freeze-thaw stability than did nonwaxy gels, as indicated by significantly lower values of net syneresis. In other words, waxy starch gels were more stable than nonwaxy starch gels when stored at 4°C or at -15°C. The refrigeration and freeze-thaw stability of waxy wheat starch gels was similar to that of waxy corn starch gels in the test period (seven days). The greater retrogradation resistance and freeze-thaw stability of waxy wheat starch would be useful in chilled and frozen products.

Thermal Characteristics

The gelatinization transition temperatures (onset, peak, and completion) and gelatinization temperature range of waxy starches were significantly higher than those of nonwaxy starches, except for onset temperature of wheat, which was only slightly higher (Table V). The peak gelatinization temperature of waxy wheat starch averaged 66°C compared with 63°C for nonwaxy wheat, whereas corn starch had gelatinization temperatures of 72 and 70°C, respectively. Yasui et al (1996) reported similar gelatinization temperatures for waxy wheat (64–66°C) and nonwaxy wheat (61–63°C) starches. The enthalpy of gelatinization was significantly higher in waxy corn compared with nonwaxy corn, but was only slightly higher in waxy wheat starch compared with nonwaxy wheat starch. Other investigators have found significantly higher gelatinization enthalpy for waxy wheat starch compared with nonwaxy wheat starch (Yasui et

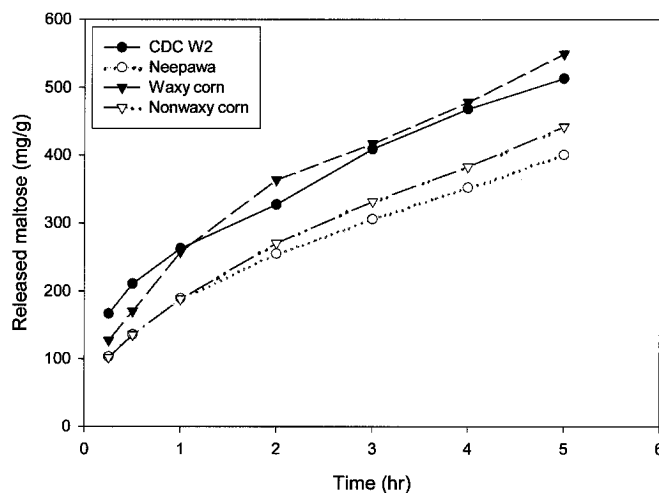


Fig. 6. Amylolysis of waxy and nonwaxy wheat and corn starches.

TABLE VI
Degree of Crystallization and %Relative Intensity of Major Peaks in X-ray Diffraction Patterns of Waxy and Nonwaxy Wheat Starches^{a,b}

| Starch Source | Crystallization (%) | X-ray Diffraction Angle (2θ) | | | | |
|---------------|---------------------|------------------------------|-------------|-------------|-------------|-------------|
| | | 15.2 (5.8) | 17.2 (5.2) | 18.4 (4.8) | 20.0 (4.4) | 23.2 (3.8) |
| CDC W2 | 21.2 ± 0.71 | 23.8 ± 0.82 | 29.6 ± 0.85 | 29.6 ± 0.85 | · · · | 28.8 ± 0.73 |
| Neepawa | 15.7 ± 0.57 | 22.5 ± 0.56 | 29.2 ± 0.57 | 29.2 ± 0.57 | 24.2 ± 0.42 | 26.7 ± 0.56 |

^a Values in parentheses indicate starch crystal *d*-spacing (Å).

^b Mean ± standard deviation.

al 1996; Hayakawa et al 1997). The high temperature and energy required to gelatinize waxy starches could reflect a more compact structure of amylopectin molecules. Among waxy starches, waxy wheat starch had lower gelatinization temperatures and enthalpies than waxy corn starch.

Starch Structure

The X-ray diffraction A-type pattern was observed for both waxy and nonwaxy wheat starch, showing major peaks near *d*-spacings of 5.8, 5.2, 4.8, and 3.8 Å that are characteristic of most cereal starches (Table VI). The *d*-spacing 4.4 Å, which is characteristic of the amylose-lipid complex (Zobel 1988), was absent in waxy starch but was observed in nonwaxy starch (Fig. 5). Despite the similarity in X-ray diffraction pattern, significant differences in the degree of crystallization between waxy and nonwaxy wheat starch were observed. The degree of crystallization of waxy wheat starch was estimated at 21.2% compared with 15.7% for nonwaxy starch. Waxy starch also showed slightly higher percent relative intensities of the major peaks particularly at 2θ 23.2°. The higher degree of crystallization in waxy wheat starch probably reflects more closely packed crystal structures due to a higher amylopectin content. Several studies have shown that waxy wheat starches attain higher degrees of crystallinity than nonwaxy wheat (Hayakawa et al 1997; Fujita et al 1998; Demeke, et al 1999). Despite the high degree of crystallization in waxy starches, they exhibited higher rates of α -amylolysis (Fig. 6). This observation was obtained for both waxy wheat and waxy corn starches. The higher rate of α -amylolysis in waxy starches could reflect greater accessible positions for α -amylase in the branched starch molecules. In other words, the absence of bound lipids such as amylose-lipid complexes in waxy starches could make it more susceptible toward hydrolysis by α -amylase. Hydrolysis of four types of maize starches by porcine pancreatic α -amylase for 72 hr was in the order dull waxy > waxy > nonwaxy > amylo maize (Hoover and Manuel 1996).

CONCLUSIONS

The high level of amylopectin in waxy wheat had major effects on flour and starch properties. The waxy wheat flour absorbed more water and required less time and energy for dough development. The waxy dough was more susceptible to overmixing. In agreement with the findings of Bettge et al (2000), waxy wheat starch granules were less resistance to mechanical damage than nonwaxy starch. These properties should be considered when using waxy wheat in composite flour for baking applications. Due to the susceptibility of amylopectin to shearing particularly at high temperature, and the absence of amylose, the FN test was not applicable to measurement of α -amylase activity in waxy wheat. Graybosch et al (2000) also reported low falling number for waxy wheats. If waxy wheats find commercial applications, other means of indirect or direct measurements of α -amylase activity will be required or a scale applicable to waxy cultivars will need to be developed. Another interesting finding is the enormous increase in peak viscosity of waxy wheat flour in the presence of mercuric acetate or silver nitrate in spite of ordinary levels of α -amylase. This finding warrants further investigation to study effects of these chemicals on pasting properties of waxy starches.

There were no differences in granule shape and size between waxy and nonwaxy starch from wheat or corn, but major differences were observed in pasting, gelation, retrogradation, and structural properties. Waxy wheat starches showed greater pasting viscosity and refrigeration and freeze-thaw stabilities than did nonwaxy starch. The waxy starch was characterized by the absence of *d*-spacing 4.4 Å, which is characteristic of the amylose-lipid complex. Waxy wheat starch was similar to waxy corn starch, particularly in pasting and syneresis properties. Waxy wheat flour was distinct from HRS or soft wheat flour in terms of pasting and mixing properties.

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