Thermal and Viscous Properties of Rye Starch Extracted from Different Varieties

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ABSTRACT

Thermal and viscous properties of rye starch extracted from different cultivars were studied and compared with gelatinization parameters and retrogradation of wheat and maize starches. Gelatinization parameters for the rye starches were shown to be lower than those for wheat starch but similar to those for oat starches (literature value). The transition of the amylose-lipid complex was low, which indicated a low lipid content in the starch. Retrogradation of the rye starch was quite low compared with that of wheat and maize starches despite the low lipid content. The viscous properties of the rye starches were investigated in the 60–90°C temperature interval. The rye starches increased in viscosity very differently with increasing temperature and differed in their end-viscosity values after 400 sec holding time at 90°C. The gelatinization parameters, retrogradation (enthalpy value after 14 days of storage), and viscosity (end value after 400 sec at 90°C) were correlated with other measured factors, such as falling number, gel volume, amylose leaching, and percentage of fissures on the surface of starch granules. Moderate to high correlation was found between gelatinization enthalpy and falling number, end viscosity, and percentage of fissures (0.64, −0.72, −0.95, respectively). Gelatinization temperature correlated moderately with falling number, end viscosity, and gel volume (0.61, −0.85, and −0.70, respectively). Falling number also had a moderately high-correlation with percentage of fissures and amylose leaching (−0.84 and 0.78). Retrogradation (enthalpy value after 14 days of storage) correlated highly with amylose leaching (0.90). End viscosity correlated highly with percentage of fissures and gel volume (0.91 and 0.79). These correlations helped considerably to explain the thermal and viscous properties of the rye starches investigated.

The main interest in rye has been in relation to bread production (Sollars and Rubenthaler 1971, Roewe et al 1982) or its pentosan content (Casier et al 1973, Jankiewicz and Michniewicz 1987). The physicochemical properties of rye starch itself have gotten very little attention. Studies have been performed on chemical composition, water-binding capacity, and amylose content (Berry et al 1971); on swelling properties (Williams and Bowler 1982); and on Brabender amylograms, average chain length of amylopectin, and gelatinization temperature measured as loss of birefringence (Lii and Lineback 1977).

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The aim of the present investigation was to study the variation in viscous and thermal properties (gelatinization and retrogradation) of some varieties of rye starch. The thermal properties of rye starches were compared with those of wheat starch.

MATERIALS AND METHODS

Materials

Nine different rye samples were supplied by Wasabröd, Filipstad, Sweden: Otello, Ernte '86, Danko, Ernte '88, Petkus II '88, Petkus II '89, LPH 11, LPH 12, and Epos ('86, '88, and '89 indicate the year of cultivation). The samples thus represented different years as well as varieties. Wheat starch (from the variety Amy) was prepared in the same way as the rye starches were (described below). Maize starch (Lyckeby National, Kristianstad, Sweden) was also used.

Methods

Starch extraction. Starch was extracted from the rye and wheat grains as described by Meredith et al (1978), except that a Sorvall omnimixer (Ivan Sorvall Inc., Norwalk, CT) was used at low speed to crush the grains.

Microscopic examination. Rye starch suspensions were stained with Congo red (0.1%) and viewed under the microscope (magnifications 10 × 40 and 10 × 100) under both ordinary and polarized light to see whether the granules were damaged (Moss 1976). Colored granules and granules with visible fissures on the surface were counted.

Starch granule size measurements. Starch granules were measured using a Coulter Counter (Perkin-Elmer DSC-2); size distribution was calculated as weight percentage versus particle diameter. The method has been described elsewhere (Eliasson and Karlsson 1983).

Falling number. To estimate the enzymatic activity of the rye flour, the falling-number test was used according to the method described by Perten (1964).

Gel volume and amylose leaching. Modifications of the methods of Schoch (1964) were used for measuring swelling power and solubility. Around 40–50 mg of starch (in 3 ml of water) was used. The suspensions were heated in test tubes and were shaken thoroughly during heating. The test tubes were kept in a water bath at 90°C for 30 min and centrifuged at 1,000 × *g* for 15 min; the gel volume was then measured. The amount of released amylose was obtained by measuring absorbances according to the blue-value method of Gilbert and Spragg (1964) on an aliquot of the supernatant. Pure amylose from potato (Sigma, St. Louis, MO) was used for the calibration curve.

Measurements of viscous properties. The viscosity of the starch suspensions was investigated in the 60–90°C temperature interval using the Bohlin Rheometer System (Bohlin Reologi, Lund, Sweden). Concentric cylinders were used with 10 ml of suspension. The starch suspensions (3.5% w/w) were preheated in a water bath at 60°C for 5 min and then were transferred to the rheometer. Measurements were taken at a constant shear rate (184.6 sec^{-1}), and the temperature was increased at 6°C/min. When the temperature of the starch suspension reached 90°C, measurements were continued for 400 sec at this temperature. The average of three measurements for each rye starch variety was taken; the standard deviation was always within 10% of the mean.

Differential scanning calorimetry measurements. A Perkin-Elmer DSC-2 calorimeter was used for measuring thermal properties. Gelatinization and retrogradation were studied for starch suspensions with 60% (w/w) water content (i.e., water was added to the starch on a dry basis). Samples (10–20 mg) of the test suspensions were transferred to weighed sample pans (DuPont coated pans), which were then sealed and reweighed. The samples were heated from 17 to 127°C at a rate of 10°C/min, with an empty sample pan as a reference. Water content was determined by puncturing the pans, drying them in an oven at 105°C for 24 hr, and then reweighing them. In the retrogradation study, the sample pans were stored at room temperature (23°C) after the first heating during the DSC measurement and then were reheated after 1, 2, 3, 7, or 14 days of storage. The temperatures reported are those of the maximum of the endotherm peaks. The results reported are the mean and the standard deviation of 10 scans for the gelatinization parameters and the mean of two scans for the retrogradation.

Correlation coefficients and their significance were calculated using the procedure described by Woodward (1988).

RESULTS

Microscopy

The microscopic investigation showed that the rye starch granules were mostly undamaged. Less than 1% (based on counting) of the granules took on any color; however, some granules had fissures or scars (Table I). The difference in the percentage of granules showing fissures, based on counting 1,000 granules) among the rye starches was considerable. The percentage of fissures correlated negatively with gelatinization enthalpy and positively with end-viscosity values (i.e., viscosity after 400 sec at 90°C) (Table II). A high, negative correlation coefficient was evident with falling-number values.

Falling Number

The falling-number measurements differed among the rye samples (Table I), indicating a considerable difference in enzymatic activity in the rye flour. Falling-number values correlated moderately and significantly (0.1 level) with gelatinization enthalpy and temperature, percentage of fissures (0.01 level), and amylose leaching (0.01 level).

Viscosity Measurements

Results of the viscosity measurements (Fig. 1) are shown as viscosity versus temperature, with a 400-sec holding period at 90°C. The variety Otello increased the most in viscosity with increasing temperature and reached the highest end-viscosity value of all the rye starches investigated. Epos and Petkus II '88 showed somewhat lower values. Variety LPH 11 had the least increase; LPH 12 was only marginally higher. The other varieties (Ernte '86, Danko, Ernte '88, and Petkus II '89) also had rather small

TABLE I
Rye Starch Granules with Fissures on the Surface and the Falling Number Values

Variety	Percentage with Fissures[a]	Falling Number
Otello	12.6	120
Ernte '86	12.5	135
Danko	10.2	165
Ernte '88	9.8	95
Petkus II '88	9.6	110
Petkus II '89	9.2	255
LPH 11	8.9	245
LPH 12	8.9	280
Epos	8.8	260

[a] Based on counting, per 1,000 granules.

TABLE II
Correlation Coefficients for Several Parameters in Samples from Rye Starches[a]

	ΔH_G	T_G	Falling Number	ΔH_c	Viscosity After 400 sec at 90°C	Percentage of Fissures	Gel Volume	Amylose Leaching
ΔH_G	...	0.51 n.s.	0.64*[b]	0.47 n.s.	−0.72**	−0.95***	−0.47 n.s.	0.40 n.s.
T_G		...	0.61*	0.17 n.s.	−0.85***	−0.36 n.s.	−0.70**	0.32 n.s.
Falling number			...	0.57 n.s.	−0.41 n.s.	−0.84***	−0.48 n.s.	0.78***
ΔH_C[c]				...	−0.21 n.s.	−0.43 n.s.	−0.43 n.s.	0.90***
Viscosity after 400 sec at 90°C					...	0.91***	0.79***	−0.19 n.s.
Percentage of fissures						...	0.44 n.s.	−0.42 n.s.
Gel volume							...	−0.50 n.s.
Amylose leaching								...

[a] ΔH_G = gelatinization enthalpy, T_G = gelatinization temperature, ΔH_C = melting enthalpy of recrystallized starch.
[b] *** = significant at 0.01 level, ** = significant at 0.05 level, * = significant at 0.1 level, n.s. = not significant.
[c] Enthalpy values measured after 14 days of storage.

increases in viscosity with increasing temperature. The viscosity of the variety with the highest value was more than double the value of the variety with the lowest viscosity. End-viscosity values correlated moderately (negatively) with gelatinization enthalpy and temperature, and positively with percentage of fissures and gel volume (Table II).

Starch Granule Size Distribution

The results of the Coulter-Counter measurements for three varieties of rye starch (Otello, Epos, and LPH 11) are shown in Figure 2. These varieties were chosen on the basis of viscosity measurements. Otello and LPH 11 were two extremes, with Otello showing the highest end viscosity and LPH 11 the lowest. Epos showed an intermediate viscosity value. The difference in granule size distribution was very small, as these three varieties have nearly the same mean granule size and the same distribution range. The measurements also showed that during the preparation of rye starch, the small granules (those below 4 μ) were lost.

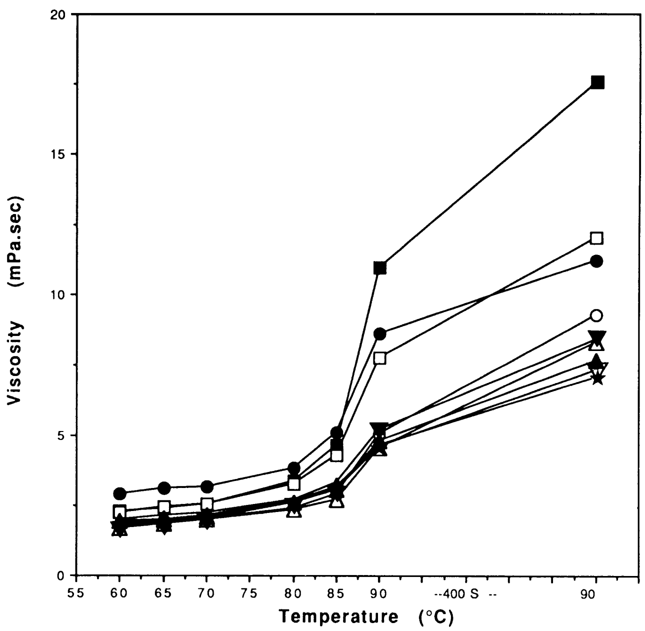

Fig. 1. Viscosity as a function of temperature for nine varieties of rye starch. ■ = Otello; □ = Epos, ● = Ernte '86, ○ = Petkus II '88, ▼ = Danko, △ = Ernte '88, ▲ = Petkus II' 89, ▽ = LPH 12, ★ = LPH 11.

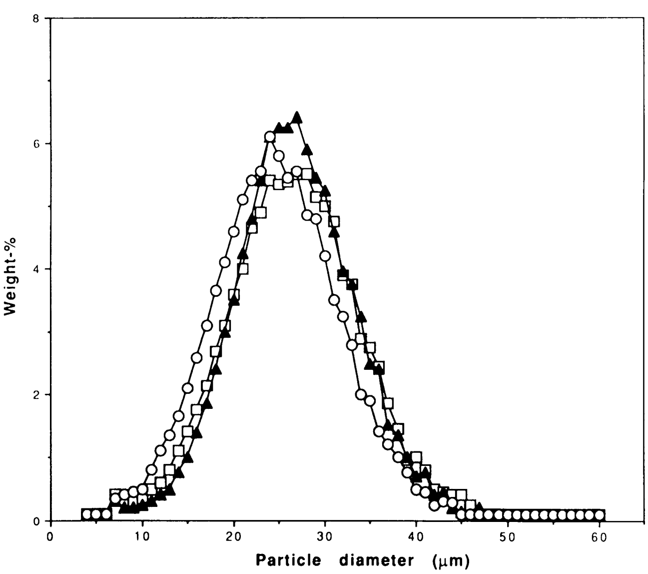

Fig. 2. Particle weight percentage versus particle diameter for three varieties of rye starch. □ = Epos, ▲ = Otello, ○ = LPH 11.

Gel Volume and Amylose Leaching

The rye starches varied greatly in gel volume and amylose leaching (Table III), which were correlated with other measured parameters (Table II). Gel volume correlated positively with end-viscosity values and negatively with gelatinization temperature. Amylose leaching correlated moderately with falling number and highly with retrogradation (crystallization enthalpy on the 14th day of storage). Surprisingly, amylose leaching correlated negatively with fissures, although not significantly.

DSC Measurements

The DSC techchnique measures the enthalpy and temperature of gelatinization, the enthalpy and temperature of the transition of the amylose-lipid complex, and the enthalpy and temperature of melting of the recrystallized starch. The DSC parameters of gelatinization and of the transition of the amylose-lipid complex are shown in Table IV. All the starches compared with the rye starches had the same water content. Lineback (1984) reported a gelatinization temperature range for rye starch of 57–70°C without mentioning what methods were used. The gelatinization temperature of the rye starch varieties in the present work was between 54.8 and 60.3°C. The gelatinization enthalpies did not differ much among the rye starches. The lowest value was 10.6 J/g, and the highest was 12.0 J/g, which are somewhat lower than that of wheat starch.

Rye starches differed from the wheat starch in that they showed low values of the transition enthalpy of the amylose-lipid complex (Table IV). The transition enthalpy of the amylose-lipid complex for wheat starch was up to three times that of the rye starches, and compared with oat starches, the values for rye starches were up to 10 times lower (Gudmundsson and Eliasson 1989). This indicates that the rye starches have less complexed lipids than most other cereal starches. Low amounts of extracted lipids from rye starch compared with other cereals were reported by Acker and Becker (1971). The gelatinization parameters correlated significantly only with end viscosity and percentage of fissures (Table II). Rye starches retrograded to a lesser extent than maize starch or wheat starch (Fig. 3). The difference in retrogradation

TABLE III
Gel Volume and Amylose Leaching at 90°C

Varieties	Gel Volume (ml/g,db)	Amylose Leaching (mg/g,db)
Otello	19.7	145
Epos	19.7	144
Ernte '88	18.4	120
Ernte '86	17.7	130
Petkus II '88	17.2	138
Danko	16.2	142
LPH 11	15.5	159
LPH 12	15.5	172
Petkus II '89	14.8	146

TABLE IV
Gelatinization Parameters for Nine Rye Starch Samples Compared with Wheat (Average ± Standard Deviation)[a]

Variety	ΔH_G (J/g,db)	T_G (°C)	ΔH_{Cx} (J/g,db)	T_{Cx} (°C)
LPH 12	12.0 ± 0.3	60.3 ± 0.5	0.3 ± 0.1	105.0 ± 0.5
LPH 11	12.0 ± 0.3	59.8 ± 0.5	0.3 ± 0.1	100.0 ± 0.4
Epos	11.9 ± 0.3	57.7 ± 0.3	0.5 ± 0.2	104.9 ± 0.5
Danko	11.7 ± 0.3	57.9 ± 0.3	0.3 ± 0.2	105.0 ± 0.4
Ernte '88	11.7 ± 0.3	58.1 ± 0.4	0.6 ± 0.1	105.8 ± 0.3
Petkus '89	11.6 ± 0.4	60.3 ± 0.4	0.4 ± 0.2	104.4 ± 0.5
Petkus '88	11.6 ± 0.8	55.9 ± 0.2	0.6 ± 0.1	104.9 ± 0.4
Ernte '86	11.1 ± 0.6	60.0 ± 0.9	0.4 ± 0.2	106.6 ± 0.4
Otello	10.6 ± 0.1	54.8 ± 0.3	0.4 ± 0.2	103.4 ± 0.4
Wheat[b]	13.6 ± 0.3	61.1 ± 0.5	1.6 ± 0.3	104.5 ± 0.3

[a] ΔH_G = gelatinization enthalpy, T_G = gelatinization temperature, ΔH_{cx} = transition enthalpy of the amylose-lipid cmplex, T_{Cx} = transition temperature of the amylose-lipid complex.
[b] From Gudmundsson and Eliasson (1989).

was considerable between the rye starches with the lowest and the highest levels of retrogradation (Ernte '86 and LPH 12, respectively) (Fig. 4). These results indicate that the low level of retrogradation of the rye starches is not dependent on rye lipids.

It is well established that adding lipids to starch or starch-based foods lowers the level of retrogradation (Lagendijk and Pennings 1970, Kulp and Ponte 1981, Russell 1983), and defatting the starch increases the level of retrogradation (Gudmundsson and Eliasson 1989, Hibi et al 1990). Other than lipids, another factor that has a great effect on retrogradation of starch is the water content of the sample. Maximum retrogradation is obtained with the water content between 40 and 50%, with a rather flat peak. Zero enthalpy is obtained with the water content below 20% and above 90% (Longton and LeGrys 1981, Zeleznak and Hoseney 1986). In the present research, the water content is higher, so maximum retrogradation is probably not reached, but the results should be suitable for comparison.

DISCUSSION

The gelatinization properties of the rye starches as measured by DSC showed some differences in gelatinization enthalpy and temperature. All the rye starches had a lower gelatinization enthalpy and temperature than wheat starch. The gelatinization temperatures of the rye starches were higher than the midpoint values of the birefringence measurements reported by Lii and Lineback (1977). This could be the result of the different methods or varieties used. The rye starches had gelatinization parameters very similar to those of oat starches (Gudmundsson and Eliasson 1989). The correlation between falling-number values and gelatinization parameters was moderately positive and significant at the 0.1 level. Thus, there is a slight tendency for starch from rye with a high falling number to have a high gelatinization enthalpy. Low falling-number values indicate low gelatinization enthalpy and high enzymatic activity like that of α- and β-amylases. These enzymes hydrolyze starch polymers, probably in the amorphous region (Kulp and Ponte 1981), even though one cannot rule out the possibility that they also influence the crystallites and therefore give rise to a lower gelatinization enthalpy.

Also, a relationship seems to exist between the falling number and the gelatinization temperature, as starch from rye with a high falling number tends to have a higher gelatinization temperature. This could mean that rye starch with more perfect crystallites either has lower enzymatic activity in the rye, or the starch is less affected by the enzymes. The latter relationship, however, is not in accordance with the findings of Zobel and Senti (1959) and Dragsdorf and Varriano-Marston (1980). They found that aged bread and starches with added α-amylases showed increased X-ray crystallinity compared with control samples. On the other hand, high enzymatic activity in native rye grain does not necessarily mean that the starch granules are affected, especially without treatment that causes the granules to be vulnerable to enzymatic attack, such as heat treatment. The correlation between falling number and percentage of fissures was quite high (−0.84, Table II); thus, the fissures might result from enzymatic activity. Since all granules with fissures were not stained with Congo red, they were evidently not heavily damaged. Similar fissures on the surface of rye starch granules were shown on micrographs published by Berry et al (1971).

As two varieties from different years were included in the investigation (1986 and 1988 for Ernte, 1988 and 1989 for Petkus), it should be possible to obtain some indication about the extent to which the results depend on environmental conditions. The two samples of Ernte were compared and were found to be similar in gel volume and amylose leaching (Table III), gelatinization enthalpy (Table IV), viscosity (Fig. 1), and retrogradation behavior (Fig. 4). A small difference was found in gelatinization temperature, with 60.0 and 58.1°C for Ernte '86 and Ernte '88, respectively (Table IV). Both of these samples had low falling numbers: 95 for Ernte '86 and 135 for Ernte '88. The two Petkus samples differed from each other in several properties, such as gel volume (Table III), gelatinization temperature (Table IV), end viscosity (Fig. 1), and retrogradation behavior (Fig. 4). The falling number differed considerably: 110 for Petkus II '88 and 255 for Petkus II '89. Amylose leaching and percentage of fissures showed moderately high correlations with the falling number (Table II); thus, it can be expected that these properties will depend, for example, on harvest conditions. The low falling numbers of the Ernte samples might be too low to reveal any differences.

Inherited differences should be more predominant when two varieties with the same falling number are compared. At least environmental conditions resulting in different falling numbers have been eliminated. Petkus II '89 and Epos had almost identical falling numbers (255 and 260, respectively), but they differed greatly in gel volume (Table III), gelatinization temperature (Table IV), and end viscosity (Fig. 1). Their retrogradation behavior, on the other hand, was quite similar (Fig. 4).

Compared with maize or wheat starches, the retrogradation

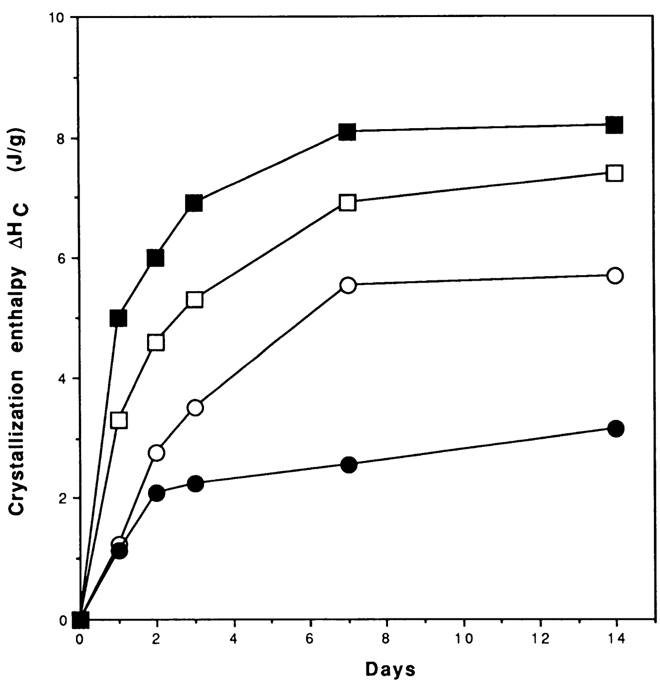

Fig. 3. Retrogradation of rye starch varieties LPH 12 and Ernte '86 compared with maize and wheat starches. ■ = maize starch, □ = wheat starch, ○ = LPH 12, ● = Ernte '86.

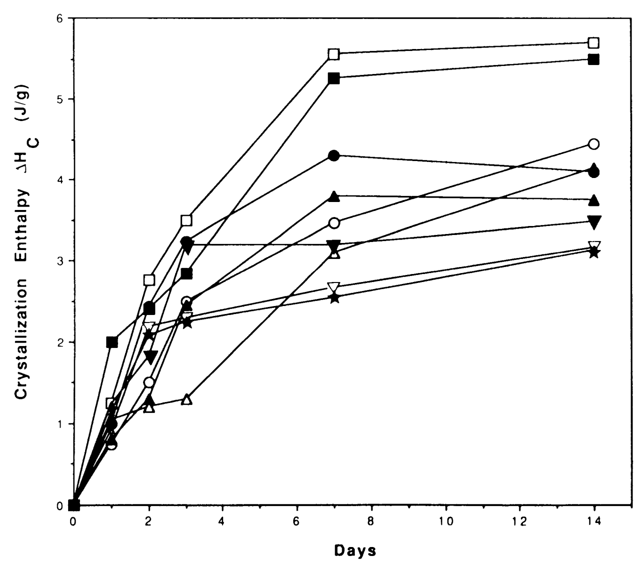

Fig. 4. Comparison of the retrogradation of rye starches. □ = LPH 12, ■ = LPH 11, ○ = Danko, △ = Otello, ● = Petkus II '88, ▲ = Epos, ▼ = Petkus II '89, ▽ = Ernte '88, ☆ = Ernte '86.

of rye starches was low, but not as low as that of oat starches (Gudmundsson and Eliasson 1989). This tendency toward low retrogradation was quite remarkable, as the rye starches had a low lipid content, indicated by the low value of the transition enthalpy of the amylose-lipid complex. Thus, the structure of the rye amylopectin could be somewhat different from that of wheat or maize, which could help the rye starch retrograde more slowly. The tendency toward retrogradation among the rye starches varied somewhat, but retrogradation was not significantly related to any other factor except amylose leaching. This could indicate that the amylopectin is more easily recrystallized when more amylose is leached out of the starch granule.

The viscosity measurements showed that the rye starches increased in viscosity very differently with increasing temperature. The variety Otello, which had the greatest increase and the greatest end value of viscosity, also had the lowest gelatinization enthalpy and temperature. In addition, it was very low in falling-number value. LPH 11 and LPH 12 showed high falling-number values and increased the least in viscosity. They had the lowest end value in viscosity and the highest gelatinization enthalpies and temperatures. The end viscosity had a moderately high negative correlation with gelatinization enthalpy and temperature, which seems to indicate that more stable rye crystallites retard the swelling of rye starch and therefore low-end viscosity values result. Thus, regarding rye starches, gel volume (i.e., the swelling capacity of granules) is a much better indicator of viscosity properties than amylose leaching. Williams and Bowler (1982) showed that the swelling pattern of rye starch granules is very similar to that of wheat and barley but different from that of oat, potato, and field bean granules. Therefore, a similar relationship between gel volume and viscosity could exist for wheat and barley starches. Water-binding capacity is also very similar in wheat and rye starches (Berry et al 1971), which further supports the likelihood of similarities in viscosity properties.

The damage to starch granules was small in all rye varieties judged from Congo red coloring, but the number of granules with visible fissures correlated highly with the end-viscosity values of the rye starches. The difference in viscosity can be explained to some extent by the percentage of fissures on the surface of the granules. As the granule size distribution of three different rye varieties with high, low, and intermediate viscosity values, were very similar, it cannot contribute to the difference in viscosity.

The loss of small granules in the starch preparation could affect the results of this research in several ways. However, previous studies do not agree unanimously on what the difference is, if any, between large and small granules. Bimodal size distribution of starch granules has been found for wheat barley and rye (Kulp 1973, Meredith et al 1978, Meredith 1981). The lipid content is higher in small wheat starch granules (Meredith et al 1978), but as rye starch has a lower lipid content, the difference in lipid content between large and small granules is probably minimal. Some reports claim that the amylose content is higher in the large granules (Kulp 1973, Duffus and Murdoch 1979), whereas others have found the same amylose content in both small and large granules (Evers et al 1974). Concerning the thermal properties, it has been reported that small wheat granules have higher gelatinization temperatures measured with DSC (Stevens and Elton 1971) and a broader gelatinization peak (Eliasson and Karlsson 1983). However, these differences are rather small and depend on the water content of the starch suspension, as lower water content decreases the difference (Eliasson and Karlsson 1983).

Similar gelatinization enthalpies have been reported for wheat starch granules of different size classes (Stevens and Elton 1971, Eliasson and Karlsson 1983), but Wong and Lelievre (1982) observed greater enthalpies of small wheat starch granules. The exclusion of small granules in the rye starch preparation could then have some effect on gelatinization temperature, but it is difficult to predict any specific effects on other properties, compared with bimodal distributed starch. The loss of the small granules could be due to the viscous properties of the pentosans, as they make it harder to filter the small granules. Small granules could also be more tightly bound to proteins and membranes, as in wheat starch (Evers 1973).

ACKNOWLEDGMENT

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