WHEAT STARCHES

I. Comparison of Physicochemical Properties[1]

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ABSTRACT

Starches for seventeen varieties of wheat were isolated with a minimum of granule damage. These varieties represented hard red spring, hard red winter, durum, and soft wheat classes. Amylose and amylopectin were isolated from both HRS and durum wheat starches and characterized by intrinsic viscosity and amount of iodine absorbed. No significant differences were observed between the polymers from the two wheat types. Starches were characterized by various physicochemical properties including percent amylose, intrinsic viscosity, water-binding capacity, gelatinization curves, and rate of iodine absorption. Percent amylose in the starches studied ranged from 23.4 to 27.6%. Durum starches tended to be on the high end of the range. Intrinsic viscosities varied for starches within the same wheat class and even for different samples of the same variety; however, no significant differences between classes were observed. Starches from durum wheat generally had larger water-binding capacities, greater rates of iodine absorption, and slightly lower temperatures of initial pasting than those from other wheat classes. These data suggest that solvents can penetrate durum starches more easily than other wheat starches. Starches from several spring wheat varieties had markedly different gelatinization characteristics.

Starch is the major constituent of wheat endosperm, and its importance to the properties of wheat products is recognized (1). Starch is also an important industrial raw material. Most proposals for increased utilization of wheat have involved use of the properties of the starch, either separately or as a part of flour (2,3).

Properties of wheat starch have been studied with the use of commercial products primarily. Relatively little work has been done on comparison of the properties of starches from various wheat classes and varieties. It is recognized that starches differ in baking quality (4,5), and some work has been done on differences in gelatinization characteristics among starches from various wheats (6–9). The amount of amylose in several wheat starches has been investigated (10), but the method used was not precise and only gross differences could be observed.

Much of the information on class and varietal differences among wheat starches was done prior to our present knowledge of the structure of starch and before many of the present techniques for starch

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study were developed. Further investigation of varietal and class differences among wheat starches seems desirable.

The purpose of the present work was to characterize a series of wheat starches using various physicochemical properties. Primary interest was in differences among hard red spring (HRS) wheat varieties and between durum and HRS wheat classes. Hard red winter (HRW) and soft wheat starches, as well as a sample of commercial wheat starch, were included for comparative purposes.

Materials and Methods

Wheat Samples. Twenty-one samples of wheat from 17 different varieties were used in this investigation. They represented HRS, HRW, durum, and soft wheat classes. Pertinent data on these samples are summarized in Table I.

Starch Isolation. Wheat (400 g.) was steeped 3 to 4 days in distilled water at 4°C. and then thoroughly crushed with a rolling pin. The crushed grain was mixed with 50 ml. distilled water, made into balls,

TABLE I
DATA ON WHEAT SAMPLES

WHEAT VARIETY	CROP YEAR	LOCATION WHERE GROWN	PROTEIN (14% m.b.)
			%
Hard red spring			
Selkirk	1962	Langdon, N.D.	14.4
Selkirk	1963	Williston, N.D.	15.5
Selkirk	1963	Fargo, N.D.	12.2
Selkirk	1964	Carrington, N.D.	13.4
Justin	1963	Minot, N.D.	17.2
Justin	1964	Fargo, N.D.	14.8
Lee	1963	Williston, N.D.	15.9
Pembina	1963	Minot, N.D.	15.9
Crim	1963	Minot, N.D.	15.6
Mida	1963	Dickinson, N.D.	16.0
Canthatch	1963	Williston, N.D.	15.3
Thatcher	1963	Minot, N.D.	15.0
Willet	1963	Fargo, N.D.	17.4
Durum			
Mindum	1962	Langdon, N.D.	12.6
Sentry	1962	Langdon, N.D.	13.9
Lakota	1962	Langdon, N.D.	14.1
Wells	1963	Langdon, N.D.	15.0
Hard red winter			
Super Triumph	1961	Stillwater, Okla.	14.0
Comanche	1963	Stillwater, Okla.	17.8
Soft			
Monon	1963	Wooster, Ohio	13.2
Seneca	1963	Wooster, Ohio	12.2

and stored in the cold for 1 hr. Starch was washed from the dough balls with 2 liters of cold distilled water, and the starch milk was passed through No. 12 bolting silk to remove particles of bran. After centrifugation the supernatant was decanted, and the brown, upper layer of "sludge" scraped off. The starch was resuspended in water and centrifuged, and the sludge was again removed. This process was repeated several times until no sludge layer could be detected. Starches were air-dried, passed through a 70-mesh sieve, defatted with absolute methanol for 3 days in a Soxhlet extractor, and again air-dried. It should be recognized that some loss of small-granule starch probably occurred during the isolation procedure.

In addition to the samples isolated from whole wheat, two other starches were used in this work. These were a composite starch sample isolated from a mixture of HRS wheat flours and a commercial wheat starch.[3] They were defatted as described above.

All samples were analyzed for nitrogen using a micro-Kjeldahl procedure (11) and for moisture using a vacuum-oven technique (12). Nitrogen contents varied from 0.03 to 0.06% and moistures from 6.0 to 9.7%.

Starch Fractionation. A portion of the starches from the wheat varieties Mindum, Selkirk, and Mida was separated into amylose and amylopectin fractions according to the procedure of Montgomery and Senti (13). Amyloses were recrystallized once from 1-butanol, isolated by precipitation with methanol, and vacuum-dried at 40°C. Amylopectin was isolated only from Mindum and Selkirk starches. The gel after amylose extraction was mixed with methanol in a Waring Blendor and the resulting precipitate of amylopectin was collected and vacuum-dried at 40°C.

Amylose Determination. The amount of iodine absorbed by the various starches and starch fractions was determined amperometrically according to the procedure of Larson *et al.* (14). Absorbed iodine was determined from a direct plot of current *vs.* amount of iodine added by extrapolating the linear ascending portion of the curve back to the level of residual current (about 0.1 μa.). A description of the electrodes and titration assembly is given elsewhere (15). Each sample was titrated four times and the results were averaged. Duplicate determinations always agreed within ±0.2 g. I/100 g. sample. Percent amylose in each starch was then calculated from the ratio of iodine absorbed by the starch to that absorbed by pure amylose.

Intrinsic Viscosities. Starches and starch fractions were dissolved in 1*N* potassium hydroxide as described by Lansky *et al.* (16). Intrinsic

[3] "Starbake," obtained from Hercules Powder Co., Harbor Beach, Mich.

viscosities (17) were determined at 25°C. using an Ostwald viscometer having a flow time of approximately 100 sec. with distilled water.

Water-Binding Capacity. The procedure described by Yamazaki (18) for the determination of alkaline water retention capacity of soft wheat flours was used with slight modification. Starch (5.0 g., dry basis) was added to 75 ml. distilled water in a tared 100-ml. centrifuge bottle. The bottle was stoppered and agitated on a wrist-action shaker for 1 hr. It was then centrifuged for 10 min. at 2,200 × *g*, the water was decanted, and the bottle was tipped up and allowed to drain for 10 min. more. The bottle was weighed and the amount of water held by the starch was determined. The water-binding capacity was calculated from the formula, g. bound water × 100/5.0. Each determination was done four times and the results were averaged. Duplicate determinations always agreed within ± 0.2 g. of bound water and usually within ± 0.1 g.

Rate of Iodine Absorption. The apparatus and technique for determination of the rate of iodine absorption by a starch suspension is described elsewhere (15). Iodine Absorption Values (IAV) were obtained in duplicate for all starches and the results were averaged. Duplicate results agreed within ± 4 IAV units.

Granule Density. Absolute densities of various starch samples were determined by the xylene displacement method as described by Schoch and Leach (19), except that determinations were made at 20° instead of 30°C. All determinations were done in duplicate and the results were averaged.

Gelatinization Curves. Starch gelatinization curves were obtained using the carboxymethyl cellulose(CMC)-amylograph technique as described by Sandstedt and Abbott (20). Starch (20 g., d.b.) was suspended in 350 ml. distilled water in a Waring Blendor. CMC[4] (3.6 g.) was added with gentle stirring over 30 sec.; the mixture was stirred for 1 min. additional and then poured into the amylograph bowl. The blender was rinsed with 100 ml. of additional water. The temperature was raised from 25° to 95°C., held at 95° for 15 min., and lowered uniformly to 50°C. Values for a blank curve using only CMC were subtracted from the starch-CMC curves to give the corrected starch curves.

Temperature of initial pasting was defined for this work as the temperature at which the initial increase in viscosity reached 10 B.U. Peak height was the maximum viscosity obtained, which in all cases occurred shortly after the temperature reached 95°C. The 15-min. height was the viscosity of the sample after 15-min. holding at 95°C.,

[4] Cellulose Gum 7 HP, Hercules Powder Co., Wilmington, Dela.

and the 50° height was the viscosity of the sample after it had cooled to 50°C.

Results and Discussion

Starch Fractions. Iodine absorption and intrinsic viscosity data for the starch fractions are shown in Table II.

The amylose fractions absorbed essentially the same amount of iodine. No significant differences were observed between the HRS (Selkirk and Mida) and durum (Mindum) varieties.

TABLE II
INTRINSIC VISCOSITY AND IODINE ABSORPTION OF WHEAT STARCH FRACTIONS

Starch Sample	Iodine Absorption (g. I/100 g. Sample)		Intrinsic Viscosity (1N KOH)	
	Amylose	Amylopectin	Amylose	Amylopectin
	g.	g.		
Selkirk (Langdon)	18.5	0.66	2.90	2.02
Mida	18.4	...	2.46	...
Mindum	18.6	0.75	2.55	1.84

Wheat amylose is generally reported to absorb 20 g. I/100 g. amylose when determined potentiometrically (13). The values obtained in this work are somewhat lower. This appears to be a general characteristic of the amperometric method (14). However, this does not affect the results when this value is used to calculate percent amylose in starch, since the IAV of the starch is lower also. In the present work, the amounts of iodine absorbed by the individual amylose fractions were averaged, and this value (18.50 g. I/100 g. amylose) was used for calculation of percent amylose in starch.[5]

Amylopectin samples absorbed very little iodine; this indicated that they were relatively free from amylose.

Some variations were observed in the intrinsic viscosities of the amylose and amylopectin fractions. However, these variations were relatively small, and the differences between HRS and durum wheats do not appear to be significant. The values obtained agreed well with those reported by Aspinal and Greenwood (21) for wheat starch fractions but are somewhat higher than those reported by other workers (13,16).

Amylose Determinations. Percent amylose in the various starches is shown in Table III. Values ranged from 23.4 to 27.6%. This agrees well with the value of 25% generally given as the average percent amylose in wheat starches (22). Variation in amylose content

[5] It should be recognized that the percent amylose determined in this way is only an approximation. To determine the exact percent amylose, the iodine absorption of each individual amylose would have to be determined.

TABLE III
PHYSICOCHEMICAL PROPERTIES OF WHEAT STARCHES

STARCH SAMPLE [a]	AMYLOSE	INTRINSIC VISCOSITY (1N KOH)	WATER-BINDING CAPACITY	IODINE ABSORPTION VALUE
	%		%	
Hard red spring wheats				
Selkirk (Langdon)	24.3	2.33	91	35
Selkirk (Williston)	24.0	2.02	84	40
Selkirk (Fargo)	24.7	2.10	86	38
Selkirk (Carrington)	23.5	2.01	87	43
Justin (Minot)	26.2	2.13	89	41
Justin (Fargo)	25.4	1.73	86	54
Lee	26.3	2.01	88	42
Pembina	26.4	2.12	88	42
Crim	26.9	2.45	83	40
Mida	27.6	2.18	87	39
Canthatch	25.7	2.08	86	40
Thatcher	24.7	2.13	86	43
Willet	24.7	1.68	88	42
Durum wheats				
Mindum	27.5	2.06	93	54
Sentry	26.6	2.15	91	51
Lakota	26.4	1.94	92	51
Wells	26.4	1.86	88	54
Hard red winter wheats				
Super Triumph	25.9	1.88	83	44
Comanche	23.4	1.55	83	38
Soft wheats				
Monon	24.3	1.49	84	36
Seneca	23.9	1.83	83	38
Others				
Composite	25.2	2.19	87	48
Commercial	24.4	2.07	104	79

[a] Different samples from the same variety are distinguished by the location where the wheat was grown.

among the HRS wheat starches was relatively small, although definite differences were evident. However, no samples with very high or very low amylose contents were found. The four durum wheat starches were among the highest six samples tested, although the highest percent amylose found was from Mida, a HRS variety. Both soft wheat varieties tested had relatively low amylose contents.

In rice and barley, percent amylose has been shown to be largely a varietal characteristic and not appreciably affected by environment (23,24). That this is also the case for wheat was indicated by the data for the four samples of Selkirk wheat grown at four different locations. All had similar amylose contents.

Intrinsic Viscosities. Viscosities of the various starches in 1*N* potassium hydroxide are shown in Table III. Values ranged from 2.33 to 1.49. Starches within each class varied in intrinsic viscosity, but no significant differences among wheat classes were detected. Differences among samples from the same variety also were relatively large.

Intrinsic viscosity studies may give evidence concerning the relative molecular size of the various starches. From the data obtained in this work it appears that this factor is controlled largely by environment with little relation to wheat class or variety.

Water-Binding Capacities. Results of experiments on water-binding capacity are shown in Table III. The amount of bound water determined in this way was quite reproducible for each starch sample. Bound water was undoubtedly both absorbed by the granules and adsorbed on their surface. Values obtained agreed quite well with that reported by Larsen (25) for prime wheat starch using a similar technique. In general, starches within each class had similar water-binding capacity. Durum wheat starches had generally higher values than those from HRS wheats, whereas HRW and soft wheat starches had generally lower values.

Iodine Absorption Values. The IAV is a measure of the rate at which iodine is absorbed by the starch granules. It is affected by the physical state of the granule and can be used to measure starch damage quantitatively (15). Starches used in this study were isolated in such a way that essentially no mechanical starch damage could have occurred.

Differences in IAV among the various starches doubtless are related to the basic structure of the granules themselves. Results are shown in Table III. Starches from HRS, HRW, and soft wheat varieties generally had similar IAV's. Durum wheat starches had significantly higher values. These results suggest that solvents can penetrate durum starch more easily than starches from other wheat classes. This was also indicated by the generally higher water-binding capacity of durum starches. One explanation for these observations is that durum starch may have a less compact granule structure than other wheat starches. In this case, durum starches should have lower

TABLE IV

DENSITIES OF WHEAT STARCHES

Starch Sample	Density	Starch Sample	Density
Hard red spring wheats		Durum wheats	
Justin (raw)[a]	1.491	Wells (raw)[a]	1.486
Selkirk	1.473	Mindum	1.469
Justin	1.476	Sentry-Lakota[b]	1.467
Crim	1.475	Wells	1.464

[a] Sample was not defatted.
[b] This sample was a mixture of the two varieties, because of lack of sufficient amounts of the individual starches.

granule densities than HRS wheat starches. Densities of representative samples of both durum and HRS wheat starches are shown in Table IV. Differences between starches from the two wheat classes were small. However, durum starches did have lower granule densities in all samples studied. This may explain why durum starch is reported to be more susceptible to enzyme attack and to have greater swelling capacity than other wheat starches (26).

Composite starch had a higher IAV than the pure HRS wheat starches. This can be attributed to the fact that this sample was isolated from flour and probably reflects damage to the granules during milling. The commercial wheat starch had a very high IAV, which indicates extensive granule damage.

Gelatinization Studies. The CMC-amylograph method was used because it shows the two-step gelatinization of wheat starch. Gelatinization curves of all samples tested had the same general shape. Typical examples are shown in Fig. 1. These are plots of the amylograph data corrected for the viscosity of the CMC itself. Gelatinization data are summarized in Table V. All data are from plots of corrected amylograph viscosities. These values summarize the pertinent data which can be obtained from the amylograph curves. Temperature of initial pasting indicates the temperature at which swelling begins. Peak height shows the maximum viscosity obtained and, when compared with the 15-min. height, gives an indication of the relative

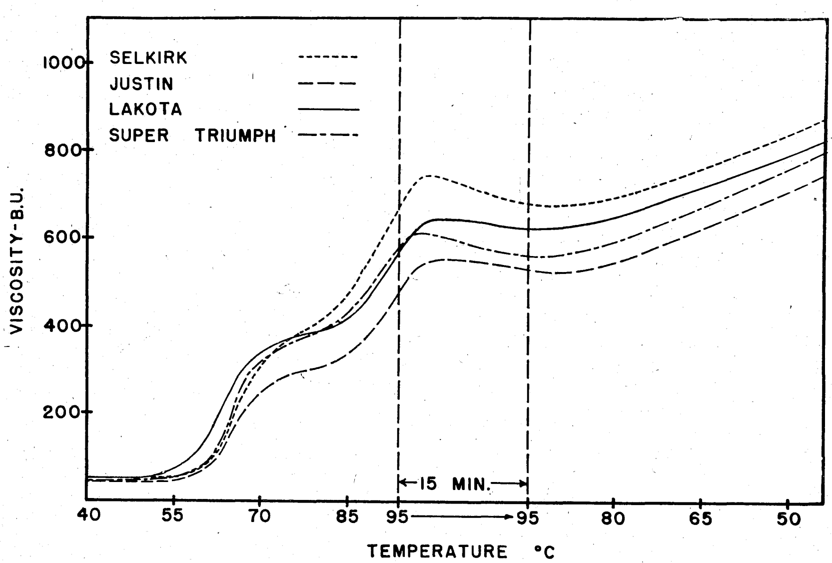

Fig. 1. Plots of starch CMC-amylograph data (corrected for viscosity of CMC).

TABLE V
SUMMARY OF WHEAT STARCH AMYLOGRAMS

STARCH SAMPLE [a]	TEMPERATURE OF INITIAL PASTING [b]	PEAK HEIGHT [c]	15-MINUTE HEIGHT [d]	50° HEIGHT [e]
	°C.	*B.U.*	*B.U.*	*B.U.*
Hard red spring wheats				
Selkirk (Carrington)	56	760	690	840
Selkirk (Williston)	58	745	690	820
Selkirk (Fargo)	58	785	740	895
Selkirk (Langdon)	56	710	710	845
Justin (Minot)	57	570	570	715
Justin (Fargo)	56	550	525	700
Lee	58	670	650	805
Pembina	59	620	615	785
Crim	58	610	600	780
Mida	55	570	560	730
Canthatch	59	645	640	780
Thatcher	57	630	615	800
Willet	57	560	530	700
Durum wheats				
Mindum	53	600	590	785
Sentry	54	625	615	780
Lakota	53	645	625	790
Wells	54	615	580	760
Hard red winter wheats				
Super Triumph	57	620	560	770
Comanche	56	710	640	810
Soft wheats				
Monon	56	675	615	805
Seneca	55	650	595	780
Others				
Composite	57	630	630	775
Commercial	55	555	490	670

[a] Different samples from the same variety are distinguished by the location where the wheat was grown.
[b] Temperature at which the corrected starch curve showed a rise of 10 B.U.
[c] Maximum viscosity of the corrected starch curve.
[d] Viscosity of the corrected starch curve at the end of the 15-min. period of holding at 95°C.
[e] Viscosity at 50°C. during the cooling cycle.

stability of the starch paste. The 50° height indicates the relative set-back of the cooled paste.

These data indicate that durum starch begins to swell at a slightly lower temperature than starch from other wheat classes. This observation may be related to the suggested less-compact granule structure of durum starch. With the exception of Selkirk starch, the maximum viscosities of durum and HRS starches were relatively stable during stirring at 95°C. Selkirk starch, as well as the soft and HRW starches, showed the usual drop in viscosity after the maximum had been reached. This viscosity drop is considered typical of wheat starches (20). The commercial wheat starch showed this characteristic also, whereas the composite HRS starch did not.

Durum starches and the majority of HRS starches had similar maximum viscosities and viscosities at 50°C. Several notable exceptions to this trend occurred among the HRS starches. All samples of Selkirk starch studied had peak heights and 50° heights markedly greater than any of the other spring or durum wheat starches. Justin (two samples), Mida, and Willet starches had peak heights and 50° heights markedly lower than any of the other starches. Representative samples of each grouping are shown in Fig. 1.

No explanation for the observed differences in gelatinization characteristics between the various wheat starches can be given at this time. Further work in this area is in progress.

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