

Inherent Amylograph Pasting Ability of U.S. Wheat Flours and Starches¹

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ABSTRACT

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Hot-pasting abilities of flours of 68 wheats of U.S. origin were compared with a Brabender amylograph in the absence of amylase activity. Starches prepared from eight of the wheats with minimal granule damage were examined similarly, and their susceptibility to amylase attack was determined. The inherent hot-pasting abilities of flours of U.S. wheat cultivars in the absence of amylase were similar in range to those previously determined for New Zealand wheat cultivars. Flours from soft and club

wheats and one sample of durum wheat had poor pasting ability. A marked seasonal effect on pasting was observed. Part of the variation in pasting can be explained by differences in protein contents and starch damage. Susceptibility of starch granules to attack by fungal amylase during the determination of starch damage was not correlated significantly with susceptibility of starch pasting to attack by sprout amylase.

Wheats from the Australian gene pool and their resulting starches have higher pasting viscosities than do starches from wheats developed elsewhere (Loney et al 1975). The purpose of this investigation was to determine starch-pasting abilities of U.S. wheats, particularly hard winter wheats. Despite the well-known variation among instruments, the Brabender amylograph was used because it is the standard technique accepted by industries using flour and starch as thickeners. The effects of amylase in pasting were obviated by inactivation. Amylase activity and other variables were assayed separately. Susceptibility to amylase attack was examined in some starches.

MATERIALS

A total of 68 samples was used in this study. Forty-one samples of hard winter wheats were obtained in two series. Those included in the first series were from the Research Collection of wheats grown

each year for the Hard Red Winter Wheat Quality Laboratory in Manhattan, KS. The wheats, representing a wide range of bread-making potential and comprising six lines grown in each of four seasons, were Quivira-Tenmarq × Marquillo-Oro, Shawnee, Concho² × Triumph, two Chiefkan-Tenmarq selections, and an Ottawa selection. The protein ranges and averages, all expressed on a 14% moisture basis (mb), were 11.2-13.2% (12.2%), 10.5-14.0% (12.6%), 12.6-14.7% (13.5%), and 17.1-19.4% (18.1%) for wheats from 1975, 1979, 1980, and 1981 crop years, respectively.

In the second series, four cultivars or selections, Centurk, Newton, Scout, and KS 75216, were drawn from cooperative wheat trials of the 1980 harvest in three diverse Kansas locations: Garden City (which included irrigated and nonirrigated cultures for comparison), Hays, and Parsons. The protein ranges and averages, all expressed on a 14% mb, were 9.4-12.6% (10.8%), 11.6-12.0% (11.8%), 8.9-9.7% (9.3%), and 11.8-13.6% (12.9%), for the flours from wheats grown at Hays, Parsons, Garden City dry, and Garden City irrigated, respectively.

Fourteen samples of other wheat types from the 1980 harvest were obtained. Flours from three commercial hard red spring samples contained 12.1, 12.6, and 12.7% protein; flour from a sample of Waldron wheat, 14.4% protein; and flour from a durum sample, 13.3% protein. Soft white wheats Davis, Luke, and McDermid, and club wheats Moro and Paha were obtained from Pullman, WA; protein contents of their flours ranged from 7.8 to 9.3% and averaged 8.4%. Four soft (white and red) winter wheats

¹Trade names and the names of commercial companies are used in this publication solely to provide specific information. Mention of a trade name or manufacturer does not constitute a guarantee or warranty of the product by the U.S. Department of Agriculture or an endorsement by the Department over other products not mentioned.

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were from Michigan: Arthur, Arrow, B4014, and S-78. Flours milled from those wheats contained 9.0–10.3% protein (average 9.7%).

Five other samples of soft white and club wheats, of the 1973 harvest of Pacific Northwest origin, were drawn from cold storage: soft white Luke (grown in Pullman, Walla Walla, and St. John, WA) and club Paha (Walla Walla and St. John). The flours milled from those wheats contained 8.5–11.2% protein (average, 10.1%).

All wheat samples had been held as clean grain in cold storage (about 4°C) and had been freshly milled by a Buhler experimental mill used in a straight-run manner at fixed settings to give about 70% extraction.

Eight aged flours were also drawn from cold storage. They had been milled from hard red winter wheats from the research collection in 1975 or 1979, their years of harvest. The flours ranged in protein content from 11.8 to 13.4% (average, 12.5%).

METHODS

Moisture and protein contents of the flours were determined by AACC methods 44-15A and 46-11 (1979). Protein content was calculated as $N \times 5.7$, and results were expressed on 14% mb. Damaged starch was determined by AACC method 76-30A (1979) using fungal amylase and ferric yanide reduction, but including an added acid-inactivation stage when initially wetting the flour, so that amylase naturally present in high levels in some of the samples would not affect the results. A value for the amount of inherent "flour sugars" was obtained from the blank titrations in the damaged starch determinations. Amylase in the flours was determined by AACC method 22-06 (1979), which involves digesting a covalently dyed amylose substrate and subsequently measuring the color released into solution under specified conditions.

Protein and damaged starch analyses of flours were corrected to 14% mb. Damaged starch was expressed on a dry matter basis. Amylogram peak heights of flours were corrected to 14% mb by assuming a linear relationship with slope 5.5 between log Brabender unit (BU) and log percent solids concentration. Amylase and sugars were expressed on as-is basis.

The Brabender amylograph used in this investigation was a U.S.-manufactured instrument having bowl pins with fivefold symmetry, probe pins with fourfold symmetry, and a center pin. Speed was electrically controlled at 75 rpm; the temperature controller operated from 35 to 95°C during 40 min and then held at 95°C. No cooling coil or bowl cover was used. A 700-cmg spring capsule was used because of a nonstandard pen arm radius and its

sensitivity checked as 615 BU per 125 g of weight added.

The Brabender amylograph used in previous investigations in New Zealand was a German-manufactured instrument having fourfold symmetry of bowl and probe pins and no center pin. Speed was mechanically controlled at 75 rpm. A water-cooled bowl cover was used; the machine was operated with the cooling finger in the raised position with no water flow. The 700-cmg capsule traced at the correct sensitivity.

Flour (60 g, as-is basis) was wetted by shaking with a mixture of 400 ml of water and 20 ml of *N* hydrochloric acid and held at room temperature (27°C) for 20 min with occasional agitation. The mixture was then neutralized by adding 20 ml of *N* sodium hydroxide during vigorous swirling. After 5 min, the mixture was poured into the amylograph bowl and operated as described by Meredith (1970) for inactivation of amylases. The final mixture (flour, water, acid, and alkali) weighed 500 g.

Starches (40 g, dry basis) were treated similarly to flour but with 50 ml of water extract of a sprout-damaged flour included as a source of amylase to determine susceptibility of the starch to amylase attack during pasting. This enzyme solution (containing the solubles of 3 g of flour) was added either to the initial acid-water mixture (inactive) or after sodium hydroxide (active) had been added. It contained 3 dextrinizing units (DU) of enzyme activity according to the AACC method 22-06 (1979). The final mixture (starch, water, acid, alkali, and enzyme extract) was maintained at 450 g.

Starches were prepared from wheats by a process designed to avoid amylase action and mechanical damage (Meredith et al 1978). A mixture of 400 ml of water and 20 ml of *N* hydrochloric acid was placed on 250 g of wheat with repeated applications of vacuum to degas during 2 hr. With vigorous stirring, 20 ml of *N* sodium hydroxide was added, degassing was repeated several times, and the mixture held at 4°C for 40 hr. A longer steeping time is necessary for hard wheats, compared with 16 hr for soft wheats. The steeped wheat was drained, treated with water in a Waring Blendor at low speed for 1 min, and decanted through a 40-mesh sieve. The mash remaining on the sieve was treated with more water in the blender and sieved as before. The process was repeated through three blendings of 1 min each and five blendings of 2 min each. The mash was hand squeezed during each sieving.

The combined extracts were passed through a 200-mesh sieve and then centrifuged. After discarding the supernatant liquid, the solids could be separated by spatula into two fractions, predominantly starch and nonstarch. Each was repeatedly suspended in water, centrifuged, and separated. Appropriate fractions were combined to result in a final yield of well-washed starch with minimal contamination and loss. Wet starch was lyophilized without prior freezing, to reduce potential starch modification by freezing. The possibility that lyophilization may cause some starch damage when the starch mixture is reduced below 4% cannot be excluded. The dry powder was rubbed through a 50-mesh sieve, allowed to equilibrate with air overnight, and stored in closed bottles at 4°C. Moisture content was determined, by oven drying at 110°C for 16 hr. Yield was calculated on a dry basis.

An additional inactivation stage was applied to those samples having a high level of amylase activity by adding acid after they had passed the 200-mesh sieve, with subsequent neutralization.

Susceptibility to attack on starch during pasting, due to the added flour extract, was calculated in arbitrary units that were the difference between the logarithms of the two amylograph maxima, multiplied by 100. This concept is based on the log-log linearity of amylograph response to α -amylase activity.

Two samples of flours made in New Zealand were sent to the United States, and two samples of U.S.-made flours and one sample representing these experimental starches were sent to New Zealand. All samples were stored in air-tight containers at deep-freeze temperatures except during airmail transit. They were analyzed by one operator on both U.S. and New Zealand amylograph instruments (Table I). Flours may be stored at -25°C for extended periods with little change in amylograph peak values (Loney and Meredith 1974).

TABLE I
Cross Calibration of Amylograph

Flour ^a and Description	Peak Value ^b	
	New Zealand	United States
New Zealand Flours		
A		
Active	475	610
Inactive	880	985
B		
Active	563	615
Inactive	1,105	1,160
U.S. Flours		
C		
Active	485	585
Inactive	938	970
D		
Active	453	570
Inactive	695	725
Starch ^c E		
No salt	510	520
With salt	530	565

^a Flours are on "as-is" basis, 60 g in 500 g.

^b In Brabender units.

^c Starch is on dry basis, 40 g in 450 g.

For statistical analyses, we used the pasting data both in original form (Brabender units) and as its inverse (mobility) according to Hlynka (1968). These analyses were made for all data and for three groups of samples (hard winter, aged flours, others).

RESULTS

Replicated analyses of the same flour on the same day showed a 0.5% coefficient of variation for an active flour amylograph peak of mean value 743 BU. Similar analyses for the same flour over a

period of time showed a 1.1% coefficient of variation for a mean value of 748 BU, with no apparent difference between first and last analyses of a day. These figures expressed the precision to be expected for a given flour. The sampling of wheat and its milling into flour, however, might be additional variables.

Cross calibration of the U.S. and New Zealand amylographs was done by exchange of flour and starch samples by a common operator. On average, for all samples including the starch, the New Zealand instrument gave readings about 65 BU lower than the U.S.

TABLE II
Inactivated Amylograph Peak Values for Buhler Flours

Type of Wheat ^a	Mean	Brabender Units		Values			
		1980 Season, Assorted Locations					
Durum	640						
PNW SWW	735	720	745				
PNW Club	770	740	795				
HRS N. Dakota	875	790	870	885	945		
PNW SRW	890						
Michigan	900	885	895	905	905		
Research collection	890	730	785	905	920	935	1,065
HRW (three locations)	915	840	840	850	865	890	890
		900	925	970	980	990	1,055
HWW (three locations)	930	900	920	925	975		
		1973 Season					
PNW Club	855	835	875				
PNW SWW	770	760	760	795			
Season		Research Collection (Six Lines, One Location)					
1975	850	730	810	845	845	910	945
1979	760	605	720	755	795	805	890
1980	890	730	785	905	920	935	1065
1981	745	660	700	725	740	810	825
		Cultivar Luke (One Location)					
1973	795						
1980	720						
Cultivar		Research Collection (Four Seasons, One Location)					
Chiefkan × Tenmarq (404)	680	605	660	730	730		
Chiefkan × Tenmarq (405)	755	700	720	785	810		
Qv-Tm × Mq-Oro	830	755	810	845	905		
Ottawa selection	830	740	795	845	935		
Concho ² × Triumph	860	725	890	910	920		
Shawnee	910	805	825	945	1,065		
		Location Series (One Season, Four Locations)					
Centurk 78	895	840	840	850	1,055		
Newton	905	865	890	890	980		
KS 75216	930	900	920	925	975		
Scout 66	945	900	925	970	990		
		Northwest Series					
Luke (two seasons, One location)	755	720	795				
(one season, three locations)	770	760	760	795			
Paha (one season, two locations)	855	835	875				
Location		Kansas (Four Cultivars, One Season)					
Garden City							
Dry	890	840	890	900	925		
Irrigated	890	850	890	900	925		
Hays	900	840	865	920	970		
Parsons	1,000	975	980	990	1,055		
		Washington State (Two Cultivars, One Season)					
St. John	800	760	835				
Walla Walla	820	760	875				

^a PNW = Pacific Northwest, SWW = soft white winter, HRS = hard red spring, HRW = hard red winter, SRW = soft red winter, HWW = hard white winter.

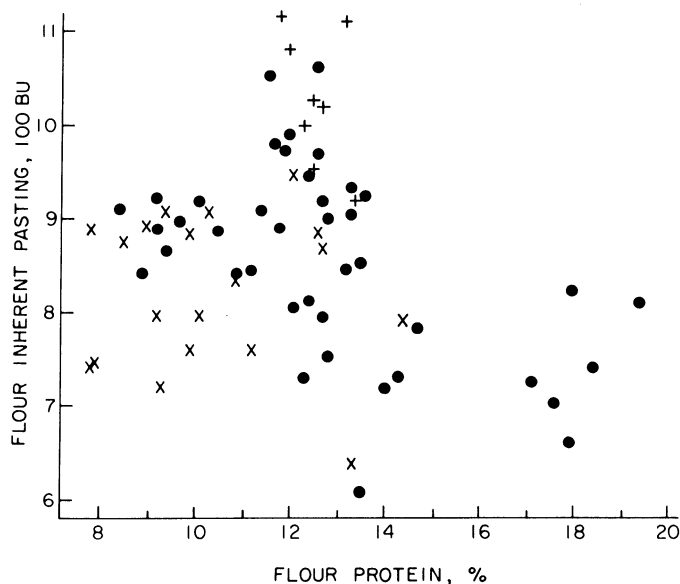


Fig. 1. Relationship between inactive pasting peak value and flour protein content. Both are corrected to 14% moisture basis. ● = hard winter wheat samples, x = other wheat samples, + = aged flours.

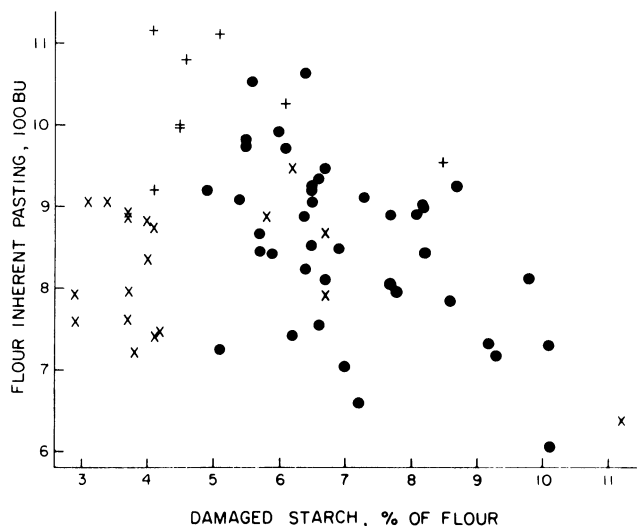


Fig. 2. Relationship between inactive pasting peak value and percentage damaged starch in the flour. ● = hard winter wheat samples, x = other wheat samples, + = aged flours.

TABLE III
Correlation Coefficients Between Inactivated Amylograph Peak Values or Mobilities of Wheat Flours and Protein Content, Starch Damage, or Sugars^a

Samples and Assay	Amylograph Peak Values (BU)	Mobility (1/BU)
All Samples (N = 68)		
Starch damage	-0.304 (1.2) ^a	0.332 (0.6)
Sugars	-0.318 (0.8)	0.316 (0.9)
Hard Red Winter Wheat Flours (N = 41)		
Protein content	-0.505 (0.1)	0.517 (0.1)
Starch damage	-0.491 (0.1)	0.494 (0.1)
Sugars	-0.470 (0.2)	0.430 (0.5)

^a Values in parentheses denote percentage probabilities of nonsignificant correlations.

instrument (Table I). Differences for the active flour averaged about 100 BU and for the inactive flour about 55 BU. Those differences, ranging from 10 BU (for starch E, no salt) to 135 BU (for flour A, active), must be considered when comparisons are made. The comparison also supports previous observations that instruments react differently to different materials, so that two instruments that are in agreement on one sample may differ in their assessment of another sample.

The analyses of the 68 flours are summarized in Table II, in the diagrams of Figs. 1 and 2, and by the statistical analyses presented in Tables III-V.

All amylograph determinations included the addition of flour extract, as a source of enzyme, with the inactivation process applied either to the starch plus flour extract or to the starch only before adding the extract. Adding solubles (including protein) from the flour extract did not appreciably affect the inherent pasting property of these starches in the presence of salt, but salt addition in the inactivation process increased the peak height by 40-50 BU and decreased the apparent effect of amylase on the starch by about 40%.

Analyses of the eight starches and of the corresponding milled flours from the same wheats are given in Table VI.

DISCUSSION

Flour-Pasting Ability

The results for U.S. flours were in the same range as those for New Zealand wheat flours, rather than those of Australian wheat flours. No cultivar, class, or region showed outstanding high pasting ability (Tables I and II). Thus, a major genetic variation of pasting ability, such as appears in the Australian gene pool, is not

TABLE IV
Significant Correlation Coefficients Between Starch Damage or Sugars and Protein or Starch Damage or Amylase

Samples and Assay	Starch Damage (BU) ^a	Sugars (BU)
All Samples (N = 68)		
Protein	0.340 (0.5) ^b	0.274 (2.4)
Starch damage		0.710 (0.01)
Amylase		0.331 (0.6)
Hard Red Winter Wheat Flours (N = 41)		
Starch damage		0.700 (0.01)
Assorted (N = 19)		
Protein	0.699 (0.1)	0.747 (0.02)
Starch damage		0.876 (0.01)
Amylase		0.553 (1.4)

^a BU = Brabender units.

^b Values in parentheses denote percentage probabilities of nonsignificant correlations.

TABLE V
R-Square × 100 Values for Simple or Multiple Correlations Between Inactive Amylograph Peak (or Mobility) Values and One, Two, or Three Other Flour Assays

All Data	Hard Winter Wheat Only		
	Inactive Amylograph Peak		
Sugars	10.1	Protein	25.5
+ Protein	12.0	+ Starch damage	53.9
+ Starch damage	12.7	+ Amylase	56.8
+ Amylase	13.1	+ Sugars	57.9
	Inactive Amylograph Mobility		
Starch damage	11.0	Protein	26.8
+ Protein	13.4	+ Starch damage	55.2
+ Amylase	14.8	+ Amylase	57.2
+ Sugars	15.0	+ Sugars	58.0

evident in U.S. wheats. For the material surveyed, inherent pasting ability of flours varies noticeably with cultivar and with season but little with location.

We examined a limited collection of wheat types. The soft white winter and club wheats of the Pacific Northwest area and the single sample of durum had notably poor pasting ability. The relatively low peak viscosity of the latter sample probably resulted from extensive starch damage during milling. Otherwise, the range of values was not great, with most values falling in the range of 720–1,050 BU.

Cultivar. Two of the Research lines (both Chiefkan × Tenmarq crosses) were poorer in pasting quality than could be accounted for by their higher protein content. The pasting strength of cv. Centurk grown at three locations was highly variable.

Season. Seasons caused the largest variation. For the Research Collection, the seasons 1979 and 1981 gave much poorer pasting ability than did seasons 1975 and 1980. Yields of wheats from the 1979 and 1981 crops were affected adversely by late frost; they were higher in protein content (especially in 1981) than were the 1975 wheats.⁴

Location. Peak viscosities of the samples from Parsons were consistently high, even though their protein levels were higher than those from other locations except Garden City irrigated. Viscosities of the high-protein (average 12.9%) samples from Garden City irrigated were consistently higher than the low protein (average 9.3%) samples from Garden City dry.

Contributions of Protein and Damaged Starch to Variation in Flour Pasting

Correlation coefficients (Table III) showed that both flour starch damage and flour sugars were significantly correlated with the inactive amylograph peak value, whether expressed directly or as mobility (Table III). Similar calculations, restricted to the hard winter wheat flour samples showed significant correlations with flour protein as well as with flour starch damage and flour sugars. All correlation coefficients given in Table III were highly significant (low figures in parentheses denoted percentage probabilities of insignificance). That significance was determined largely by the relatively high numbers of determinations. Still, even the highest correlations, ie, 0.50, explained only 25% of variability ($R\text{-square} \times 100 = 0.5^2 \times 100 = 25\%$).

These general relationships in the flours were to be expected, because protein must inversely affect the amount of starch available for pasting and because mechanical damage to the starch during milling quite likely will affect its pasting properties and

⁴M. E. Johnson. 1981. Personal communication.

depends on hardness and generally on protein content. Flour sugars were probably a reflection of the damaged starch, in that these two were highly correlated (Table IV).

The scatter diagrams (Figs. 1 and 2) show the limited relationships between pasting and protein or damaged starch. Multiple regression analysis (Table V) quantified the extent of that correlation. The values in Table V are squares of simple or multiple correlation coefficients × 100. Those values ($R\text{-square} \times 100$) reflect percent variability explained by simple or multiple correlation coefficients.

For the entire data set, variability of inherent pasting in terms of other analyses was best explained by using all four analyses (starch damage, protein, amylase, sugars, introduced in that order) related to the amylograph mobility. That combination only accounted for 15% ($R\text{-square} \times 100 = 15.0$; Table V) of the variability and for 13.1% of the inactive amylograph peak value itself. Protein was responsible in each instance for less than 2% of the increment of variation above the variability explained by sugars or starch damage alone (12.0–10.1 and 13.4–11.0; Table V).

When, however, we considered only the hard winter class of wheat, and thus more homogenous material, multiple regression analysis showed much more of the variation in pasting character to be explicable by the characteristics for which we had analyses. Protein accounted for 25.5 and 26.8% of the variation. Protein plus damaged starch accounted for 53.9 and 55.2% of the variation, whereas including amylase activity and sugars in the multiple regression explained 57.9 and 58.0% of the variation.

Thus, we suggest that not more than about 40% of the inherent pasting variations of the flour might have been due to variations in the inherent pasting property of the starch itself, the factor we were primarily interested in. Milling damage to the starch was important (as shown by the effects of starch damage), whereas the importance of protein might be readily explained as the quantity of starch available in the flour.

In the more limited but best defined set of material, showing strong genetic and seasonal effects in the analyses (the Research Samples), inherent pasting ability was not related to leaf volume nor to corrected loaf volume (volume expressed on a common protein basis; results not shown).

Contribution of the Inherent Variation of Starch to Flour Pasting

The only clear relationship that could be seen in the prepared starches (Table VI) is that the yields of starches from the wheats were inversely related to the flour protein contents. Their pasting abilities were not clearly related to their amounts obtained from the wheats, and starch pasting ability was only related in a very general

TABLE VI
Analyses of Eight Flours and Their Corresponding Starches

Wheat Cultivar or Selection	Flour (14% mb)				Starch (dry basis)				
	Amylograph Maximum		Amylase (mDU/g) ^b	Protein (%)	Starch Yield (wheat basis, %)	Amylograph Maximum		Susceptibility to Amylase	
	Inactive (BU) ^a	Active (BU)				Inactive (BU)	With Added Amylase (BU)	During Pasting (sprout amylase "units")	In Granule (fungal damaged starch, %)
Shawnee 1980	1,063	810	Neg ^c	12.6	54.2	630	380	22	1.8
Arthur	906	518	Neg	10.3	54.0	610	367	22	1.0
Qv-Tm × Mql-Oro 1975	845	56	288	11.2	54.6	535	340	20	1.6
Waldron	792	51	290	14.4	50.5	576	406	15	2.0
Chiefkan × Tenmarq ^d 1980	732	510	Neg	14.3	47.9	562	322	24	1.8
Chiefkan × Tenmarq ^d 1975	731	288	Neg	12.3	47.9	453	239	28	1.5
Moro	742	38	254	7.8	58.3	448	295	18	0.8
Luke	721	53	114	9.3	58.2	451	247	26	1.1

^aBU = Brabender unit.

^bmDU = milli-Dextrinizing unit.

^cNegligible.

^dNumber 404 in Table II.

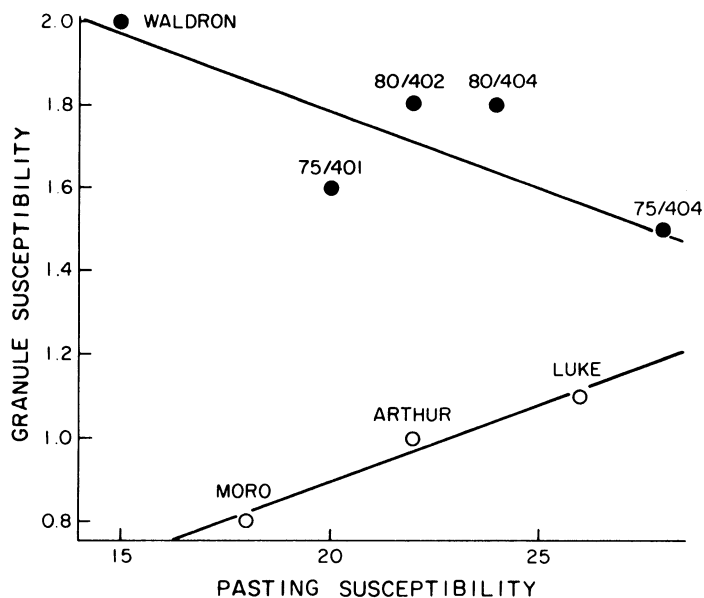


Fig. 3. Relationship between starch granule susceptibility to attack by fungal amylase (percent "damaged starch" in the starch) and susceptibility to sprouted wheat amylase during pasting of the starch in the Brabender amylograph (arbitrary units discussed in the text); 80/402 = Shawnee, 1980; 75/401 = Qv-Tm \times Mq-Oro, 1975; 80/404 and 75/404 = Chiefkan \times Tenmarq from 1980 and 1975, respectively. \bullet = hard wheat, \circ = soft wheat.

manner to flour pasting ability. However, the two soft wheats, cultivars Luke and Moro, contained a high proportion of starch of poor pasting ability.

Kulp (1972), who investigated the pasting of starches prepared from U.S. hard winter and spring wheats and from soft wheats, concluded that, although the starches of soft wheats showed clear maxima in their pasting, those of the hard wheats did not. Our results did not agree with that conclusion. Only two of our eight starches showed a lack of a clear maximum; one was from hard wheat, and one was from soft wheat (indicated by parentheses in Table VI). According to Kulp,⁵ starches in recent years do not reach their peaks at 95°C and require additional heating. Clear peaks are not always observed. Kulp (1982) suggested that this change may be due to changes in wheat cultivars.

⁵K. Kulp. 1982. Personal communication.

Susceptibility to Amylase

The susceptibilities of the carefully prepared starches to attack by amylase during pasting (Table VI) showed no consistent pattern, nor were they correlated with apparent starch damage, unless one assumes the situation of completely different relationships for the starches of hard and soft wheats, as suggested by the lines drawn in Fig. 3. These two measures of susceptibility should not, logically, be related, in that "damaged starch" concerns the susceptibility of the granule to attack by fungal amylase near room temperature, a condition far removed from the pasting situation. That emphasizes that "damaged starch," though a useful measure for partially predicting water absorption of a flour, may have little relevance to the effect of cereal amylase during cooking or baking. Kulp (1972) used a susceptibility method not very different from "damaged starch."

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