Changes in Carbohydrate Components during Wheat Maturation. II. Changes in Sugars, Pentosans, and Starch[1]

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ABSTRACT

Work was continued to investigate the changes in carbohydrate components during wheat maturation. A study of the changes in free simple sugars present in the bran during the different stages of maturity revealed that fructose and glucose decreased with maturation, while raffinose, which appeared at the later stages, showed an increase with maturity. Sucrose content appeared to fluctuate during the period investigated. Maltose was present in small amounts and was relatively constant during the different stages of maturity. Ribose was detected in the bran in minor concentrations throughout all stages of maturity. The presence of ribose in bran had not been reported previously. The percent total free simple sugars present in bran was considerably higher than that present in flour or semolina. A study of the changes in pentosan and starch content as related to the changes in total reducing and nonreducing sugars and in mono-, di-, and trisaccharides during maturation of two hard red spring and durum wheat varieties was conducted. Results indicated that wheat maturation was characterized by an increase in the pentosan and the starch content of the kernel at the expense of the available sugars. Amylose and amylopectin components of starch both increased as the kernel matured. Results showed variations in starch pasting properties and birefringence during the stages of maturity.

Regular hard red spring (HRS) wheat bran was reported to contain sucrose, raffinose, neokestose, stachyose, fructans, and fructosyl raffinose (1). Undetermined small amounts of glycerol, xylose, arabinose, glucose, and fructose were also present. It has been suggested that in the testa-pericarp, reducing sugars and sucrose were predominantly precursors of pentosans (2).

The changes in carbohydrate composition of maturing wheat have been studied by several workers (2,3,4). Pentosans in the endosperm were reported to increase throughout kernel development (2). This increase was attributed to the synthesis of new cell walls for the accommodation of the newly synthesized storage materials. The detailed studies by Jennings and Morton (2) showed that reducing sugars disappeared almost completely during the maturation phase. They concluded that reducing sugars and sucrose make a significant contribution to the dry weight of the endosperm, and that starch synthesis utilizes the pool of precursor compounds. The cytological observations of Sandstedt (5) revealed that starch synthesis in wheat endosperm starts at about 5 days after pollination.

Bice et al. (6) reported that the amylose content of wheat starch increased with maturity, and that the amylose-amylopectin ratio increased during the same period. Bice et al. (6) and Briones et al. (7) reported that the gelatinization temperature range of rice and wheat starches remained constant during the early stages of development, then slightly decreased towards maturity. Kestler et al. (8), however, reported that the stage of maturity of rice grain did not influence the gelatinization temperature as measured by the amylograph. Wheat starch exhibited birefringence 2 days after pollination (6). Brown et al. (9) reported that certain differences exist

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in the birefringence end point temperature of corn starch granules at the different stages of kernel development. No changes were observed in wheat starch relative viscosity as measured by a Stormer Viscometer (6).

Because only limited information is available on the effect of stage of maturity on the carbohydrate composition of HRS and durum wheats grown in North Dakota, the present study was undertaken.

Part I of this study dealt with the changes in free sugars in the wheat endosperm during maturation. The object of this part of the investigation was to study the changes in free sugars present in the bran of the maturing wheat, and also to study the changes in pentosan and starch during the maturation period.

MATERIALS AND METHODS

Samples

The preparation of samples of whole wheat, flour, semolina, and bran, and the analytical procedures used for the determination of their total reducing and nonreducing sugars and free simple sugars content, were described in a previous report (10).

Extraction of Free Sugars from Bran

Free simple sugars in bran were extracted using the procedure described by Saunders and Walker (1).

Ion-Exchange Chromatography

Free simple sugars in the bran extract were analyzed quantitatively by ion-exchange chromatography. Details of the procedure were described in a previous paper (10).

Pentosan

Pentosan content in flour was determined by the volumetric bromine procedure (11).

Starch

Starch content in the whole wheat flour and semolina was determined by either the polarimetric procedure (11) or the enzymatic procedure described by Banks et al. (12) and MacGregor et al. (13).

Starch Isolation

The flour or semolina sample (50.0 g.) was mixed in a Waring Blendor in a ratio of 1 part flour to 2 parts distilled water for 2 min. at low speed. The suspension was centrifuged 15 min. at 2,000 × g. The water-soluble supernatant was decanted and the material above the prime starch layer was removed with a flat-tip spatula. The starch layer was reslurried and centrifuged at the same speed as above. The purified prime starch was allowed to air-dry for 2 days, passed through a 70-mesh sieve, and stored for analysis.

Amylose

The percentage of amylose in starch was determined according to the colorimetric procedure described by Williams et al. (14).

Gelatinization Properties of Starch

Starch-gelatinization properties were examined using the Brabender Viscoamylograph. The carboxymethyl cellulose (CMC) technique as described by Medcalf and Gilles (15) was used.

Measurement of Loss of Birefringence

The Kofler hot stage as described by Schoch and Maywald (16) was used to determine the gelatinization end point temperature of starch when suspended in water. Three temperatures were recorded. These were when 10, 50, and 98% of the granules, respectively, had lost their birefringence.

Intrinsic Viscosity

Intrinsic viscosity was determined at 25°C. using an Ubbelohde Viscometer as described by Leach (17).

RESULTS AND DISCUSSION

Changes in Free Sugars of Wheat Bran during Maturation

The group of sugars studied during this investigation were the mono-, di-, and trisaccharides, which were extracted with 70% ethanol and included raffinose, sucrose, maltose, glucose, fructose, and ribose. During the chromatographic analysis of the extracts, other sugars appeared as two distinct peaks on the chromatogram and were the first to be eluted from the column. They decreased progressively during maturation. No attempts were made to investigate this group; however, it is

TABLE I. FREE SUGARS IN THE BRAN OF MATURING HRS AND DURUM WHEATS[a]

Original Moisture %	Fructose %	Sucrose %	Glucose %	Raffinose %	Maltose %	Ribose %
Justin (1969 Crop)						
70.0	2.86	1.56	1.36	0.00	0.08	0.04
59.9	2.37	0.78	0.99	0.00	0.10	0.03
58.0	1.72	0.73	0.89	0.00	0.13	0.03
50.7	0.99	2.03	0.77	0.00	0.22	0.02
49.3	0.58	0.94	0.42	0.29	0.17	0.02
43.7	0.47	0.77	0.44	0.54	0.08	0.04
28.0	0.18	1.02	0.37	0.97	0.08	0.02
12.5	0.14	1.42	0.31	1.53	0.15	0.03
9.9	0.06	1.22	0.27	1.11	0.10	0.01
Leeds (1969 Crop)						
64.0	1.82	1.01	0.87	0.00	0.80	0.04
61.4	1.21	0.87	0.71	0.00	0.10	0.03
54.4	0.74	0.82	0.57	trace	0.17	0.01
50.4	0.99	0.93	0.63	0.32	0.11	0.02
44.1	0.42	0.87	0.43	0.49	0.10	0.02
32.2	0.15	0.94	0.28	1.23	0.09	0.02
13.8	0.09	0.93	0.14	0.89	0.06	0.01
10.2	0.07	0.80	0.17	1.12	0.06	0.02
9.7	0.09	0.88	0.18	0.83	0.12	0.03

[a]At 14.0% moisture basis.

suggested that they are glucofructans of the types identified by Saunders and Walker (1).

The changes in individual free sugar content during maturation in the bran of HRS and durum wheat varieties grown in the 1969 crop year are shown in Table I. In the early stages fructose, sucrose, and glucose were the principal simple sugars in the bran extract.

HRS Wheat Bran. In the Justin variety, fructose declined sharply from 2.86% at 70.0% original moisture to 0.99% at 50.7% original moisture, then decreased uniformly to a minimum of 0.06% at the final stage of maturity. Sucrose reached its highest level suddenly as the original moisture reached 50.7% in contrast with the trend observed for fructose during the same period. This was followed by a decrease with the values fluctuating to the end of the maturation period. The observed trend, particularly at the early stages of maturity, agrees with the previous findings of Jennings and Morton (2), who reported a decrease in sucrose concentration in the testa pericarp during kernel development. At the final stage of maturity, sucrose was the major simple sugar present in the bran, followed by raffinose. Glucose decreased uniformly during maturation. The concentration decreased from 1.36% at 70.0% original moisture to 0.27% at 9.9% original moisture.

Raffinose was not detected at the initial stages of maturity. It appeared at a relatively high concentration, 0.29% at 49.3% original moisture. Raffinose content at the final stage of maturity was 1.11%. Raffinose in Justin wheat bran was detected at an earlier stage than that noted for the flour extracted from the same wheat (10), and also appeared in much higher amounts.

Slight differences were noted in maltose at the different stages of maturity. Ribose was detected in the bran extract in very small concentrations throughout the stages of maturity.

Durum Wheat Bran. Similar trends were observed for the changes in free sugars of the bran from the durum variety Leeds for the 1969 crop year (Table I), with the exception of sucrose and raffinose.

Sucrose concentration initially decreased from 1.01% at 64% original moisture to 0.82% at 54.4%. Values for sucrose thereafter remained essentially constant.

Unlike the semolina (10), raffinose in bran was detected at an earlier stage of maturity. The concentration increased considerably from 0.32% at 50.4% original moisture to 1.23% at 32.2% original moisture. The content of raffinose then decreased with some fluctuation toward the end of maturation. Raffinose concentration at the final stage of maturity was 0.83%.

The presence of ribose in the bran of either HRS or durum wheat as well as individual values for glucose, fructose, and ribose have not been reported previously.

Changes in Pentosan Content during Maturation

The pentosan contents of Justin flour and Leeds semolina for the 1969 crop year at the different stages of maturity are presented in Table II.

Cerning (18) reported a slightly increasing trend in wheat, barley, and maize pentosans with maturation. In this study, only small differences were noted in the amount of pentosans at the different stages of maturity. However, if the pentosan content of the flour or semolina is expressed on a per kernel basis, the amount of

TABLE II. PENTOSAN CONTENT OF MATURING WHEAT FLOUR AND SEMOLINA (1969 CROP)

Days Pre-Ripe	Original Moisture %	Pentosan[a] %	Pentosan in Flour or Semolina per kernel mg.
HRS (Justin)			
21	70.0	3.7	0.14
18	59.9	4.0	0.23
16	58.0	3.9	0.28
13	50.7	3.1	0.29
11	49.3	3.2	0.42
9	43.7	3.0	0.33
4	28.0	3.5	0.45
2	12.5	3.7	0.53
0	9.9	3.3	0.54
Durum (Leeds)			
20	64.0	3.5	0.41
18	61.4	3.2	0.46
15	54.4	3.7	0.67
13	50.3	3.1	0.67
11	44.1	3.4	0.72
6	32.2	3.2	0.64
4	13.8	3.6	0.85
2	10.3	3.2	0.93
0	9.7	3.2	0.95

[a]At 14.0% moisture basis.

pentosans in both varieties increased throughout the maturation period, which agrees with the findings of Jennings and Morton (2). These workers reported that the increase in pentosan content in the endosperm per kernel 14 days after flowering was due to the increase in the new cell-wall synthesis that accompanied the enlargement of endosperm cells for the accommodation of the newly synthesized storage materials like starch. When the amount of pentosan in flour or semolina was correlated with total reducing and nonreducing sugars per kernel, highly significant correlations were obtained, suggesting that the pentosans in the kernel are synthesized at the expense of total reducing and nonreducing sugars and would confirm the early work reported by Jennings and Morton (2).

Changes in Starch Content during Maturation

The changes in starch content as related to the changes in total reducing and nonreducing sugars (10) in the flour and semolina of the maturing HRS Justin variety and the durum Leeds variety for the 1969 crop year are shown in Figs. 1 and 2. Note the inverse relationship between starch content and reducing and nonreducing sugars at the different stages of maturity. A similar trend was observed for the 1970 crop year (Table III); however, the early samples harvested for both Justin and Leeds varieties were more immature than the initial samples harvested for the same varieties in the 1969 crop, which resulted in a much lower starch content. The results thus obtained confirmed previous reports (2,13) that most of the starch accumulation takes place at the initial stages after pollination when new cell walls are being formed, and continues to be synthesized during the maturation

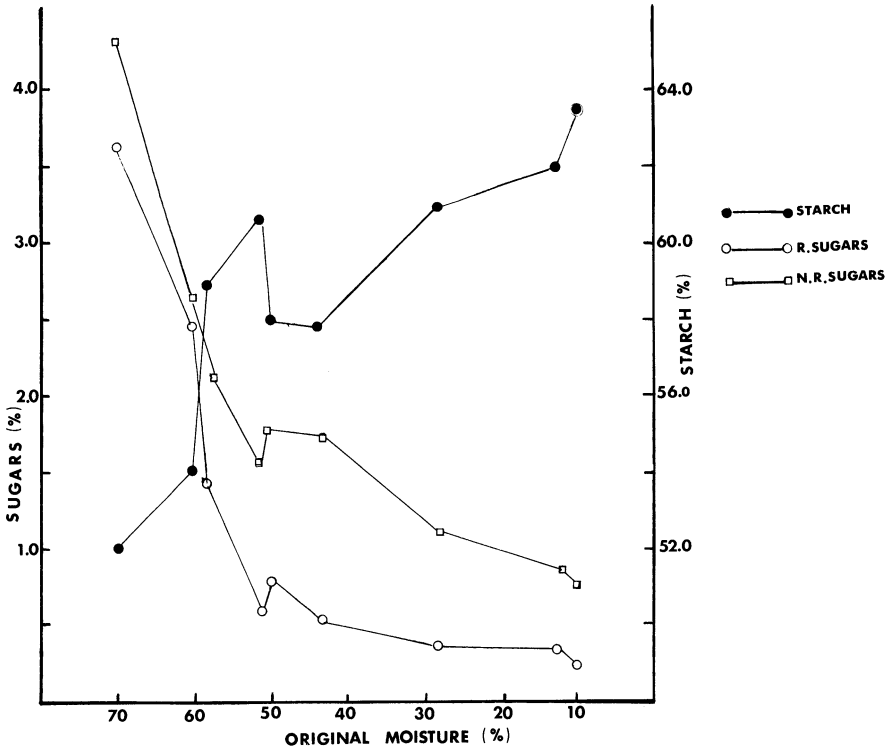

Fig. 1. Changes in starch content in flour of Justin wheat as related to the changes in both reducing and nonreducing sugars (1969 crop).

stages. When percent reducing and nonreducing sugars in wheat flour or semolina were correlated with starch content in wheat flour or semolina, highly significant correlation coefficients were obtained. The same was true when starch content was correlated with 1,000 kernel weight. This suggests that starch synthesis utilizes the pool of precursor compounds during the maturation process, as suggested by Jennings and Morton (2). Also, it has been reported that although active starch synthesis starts early after pollination, it continues to be synthesized during the maturation stages (2,13,19).

Similar significant correlations were obtained in the 1970 crop samples between starch content in the flour or semolina and either reducing or nonreducing sugars and between starch content and 1,000 kernel weight (10). The results of the

The results of the present work agree with the results of Jennings and Morton (2), and suggest that the total sugars in the kernel, which arise by both translocation from the plant and by photosynthesis within the kernel, are utilized in the rapid process of starch synthesis. The total sugar content present in the kernel at any particular stage of maturity theoretically cannot account for the total amount of starch synthesized at the following stage, indicating that translocation and photosynthesis of sugars both are essential for starch accumulation in the kernel.

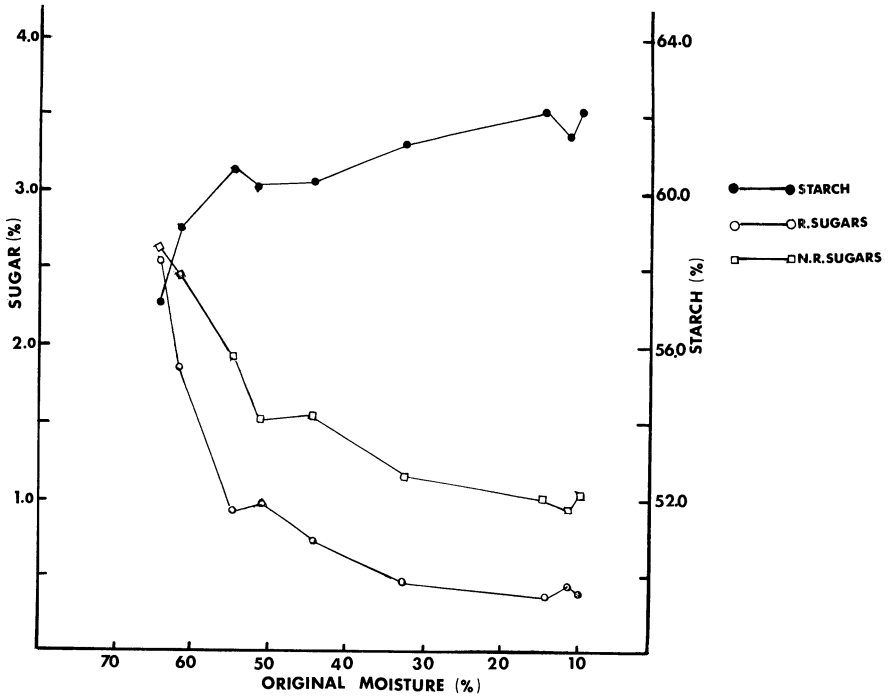

Fig. 2. Changes in starch content in semolina of Leeds wheat as related to the changes in both reducing and nonreducing sugars (1969 crop).

TABLE III. STARCH CONTENT OF MATURING WHEAT FLOUR AND SEMOLINA (1970 CROP)

Days Pre-Ripe	Original Moisture %	Starch[a,b] %
HRS (Justin)		
29	76.0	31.2
23	72.5	45.4
20	66.9	51.7
16	58.4	66.8
13	52.8	66.3
9	45.0	67.0
6	43.2	70.6
3	31.1	68.4
0	17.9	70.9
Durum (Leeds)		
...[c]	74.0	29.9
...	67.0	49.7
...	61.4	56.8
...	51.9	61.3
...	46.1	62.4
...	39.9	63.2

[a]Determined by enzymatic procedure (12,13).

[b]At 14.0% moisture basis.

[c]Undetermined because of environmental conditions.

TABLE IV. CHANGES IN AMYLOSE AND AMYLOPECTIN CONTENT DURING MATURATION OF HRS AND DURUM WHEAT (1969 CROP)

Original Moisture %	Amylose[a] %	Amylose in Starch per kernel mg.	Amylopectin[a,b], %	Amylopectin in Starch per kernel mg.	Amylose: Amylopectin Ratio per kernel
HRS (Justin)					
70.0	18.78	1.29	81.22	5.59	1:4.3
59.9	18.78	1.61	81.22	6.96	1:4.3
58.0	21.33	2.35	78.67	8.67	1:3.7
50.6	23.04	2.98	76.96	9.94	1:3.3
49.3	23.04	2.96	76.96	9.88	1:3.3
43.7	23.46	2.92	76.54	9.53	1:3.3
28.0	24.74	3.65	75.26	11.11	1:3.0
12.5	22.46	3.97	77.54	13.71	1:3.4
9.9	25.17	4.59	74.83	13.65	1:3.0
Durum (Leeds)					
64.0	15.37	0.31	84.63	1.69	1:5.5
61.4	16.64	0.52	83.36	2.58	1:5.0
54.4	16.64	0.71	83.36	3.55	1:5.0
50.3	20.90	1.18	79.10	4.45	1:3.8
44.1	21.33	1.65	78.67	6.08	1:3.7
32.2	23.04	1.45	76.96	4.85	1:3.3
13.8	23.89	1.89	76.11	6.04	1:3.2
10.3	22.61	2.03	77.39	6.93	1:3.4
9.7	24.31	2.54	75.69	7.91	1:3.1

[a]At 14.0% moisture basis.

[b]Amylopectin percent = 100 − percent amylose.

Effect of Stage of Maturity on the Chemical and Physical Properties of Starch

Effect on Starch Chemical Composition. Table IV presents the changes in amylose and amylopectin content in starch during maturation. Examination of the data reveals that amylose content in starch increased steadily during maturation for both wheat varieties. These results confirm the findings of MacGregor et al. (13) and Harris and MacWilliam (20). The ratio of amylose to amylopectin in Justin wheat increased from 1:4.3 at the first stage of maturity to 1:3.0 at the final stage of maturity. Similar trends were observed for the durum variety. At the final stages of maturity slightly different ratios of amylose to amylopectin were obtained for the different varieties harvested at approximately the same moisture level which suggests that the amylose-amylopectin ratio is a varietal characteristic. The results of this work also indicate that during the early stages of development the amylopectin fraction was synthesized at a relatively faster rate than the amylose fraction.

The results of the sugar and starch analysis at the different stages of maturity provided data in agreement with the proposed pathways found in the literature (21,22,23). The changes in fructose, glucose, sucrose, and raffinose parallel the changes in the reducing and nonreducing sugars, which in turn parallel the changes in the moisture content of the endosperm during the stages of maturity. These relationships are consistent with the rate of starch synthesis. According to Whelan

(24), the formation of amylose and amylopectin may proceed via two independent pathways. Sucrose, which is synthesized within the kernel or translocated from the plant to the kernel, is phosphorylated forming glucose-1-phosphate (G-1-P). G-1-P is converted to uridine diphosphate glucose, which is involved in amylose synthesis by glucosyl transfer to a suitable acceptor. Amylopectin is associated with the activities of phosphorylase and Q-enzyme. The pool of glucose is maintained at a suitable level for starch synthesis through the hydrolysis of oligosaccharides to yield glucose and fructose. Fructose is converted by the action of hexokinase to fructose-6-phosphate (F-6-P), which is tranformed by isomerase to glucose-6-phosphate (G-6-P). G-6-P is converted to G-1-P by the mutase enzyme action (23).

Effect on Starch-Pasting Properties. Data from the amylograph studies on selected samples from two wheat varieties of the 1970 crop indicated that in the starch of the Justin variety, no apparent increase in pasting temperature was observed as the original moisture decreased from 58.4 to 17.9%. A progressive increase in peak height was observed during the period investigated (Fig. 3). Medcalf and Gilles (25) report that the increase in peak height is due to the increase in the integrity of the starch granules. According to this view, there is a progressive increase in the integrity of starch granules with maturation. This in turn could be due to the increase in the molecular association within the granules during maturation (16). A slight increase in peak temperature was observed at the final stage of maturity. The 15-min. height, similar to the peak height, increased with the starch isolated from the more mature samples. In the case of Leeds starch, pasting

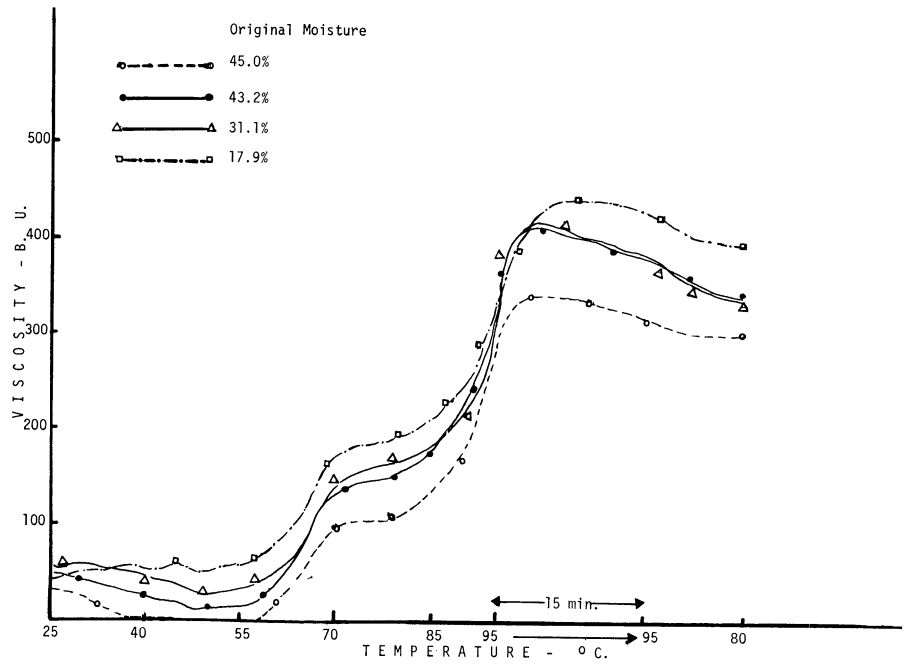

Fig. 3. HRS Justin starch CMC-amylograph curves (corrected for viscosity of CMC).

temperature decreased from 59.5°C. at 61.4% original moisture to 53.0°C. at 51.9% original moisture. The same pasting temperature was observed at 39.9% original moisture.

Peak height (Fig. 4) increased with maturation during the period investigated. The changes in both peak temperature and the 15-min. height were not consistent. It was not possible to draw any conclusions about the starch from the durum variety as it approached complete maturation because of the absence of wheat samples at the final stages of maturity.

Effect on Starch Birefringence. Birefringence end point temperature (BEPT) is defined as the temperature at which 98% of the granules have lost their polarization crosses. The results in this study indicated that all starch samples exhibited birefringence at the first stage of maturity. This confirms the early observations noted by Bice et al. (6) and by Sandstedt (5).

In the starch from the Justin variety, there were little differences in the gelatinization temperature during maturation; however, a slight decrease in BEPT was observed from the initial stage at 70.0% original moisture to the stage having 50.7% original moisture.

In the Leeds starch (Table V) no extreme differences were noted in the initial temperature of gelatinization or BEPT.

Brown et al. (9) reported that among the starch properties, BEPT is one measurement that can be affected genetically.

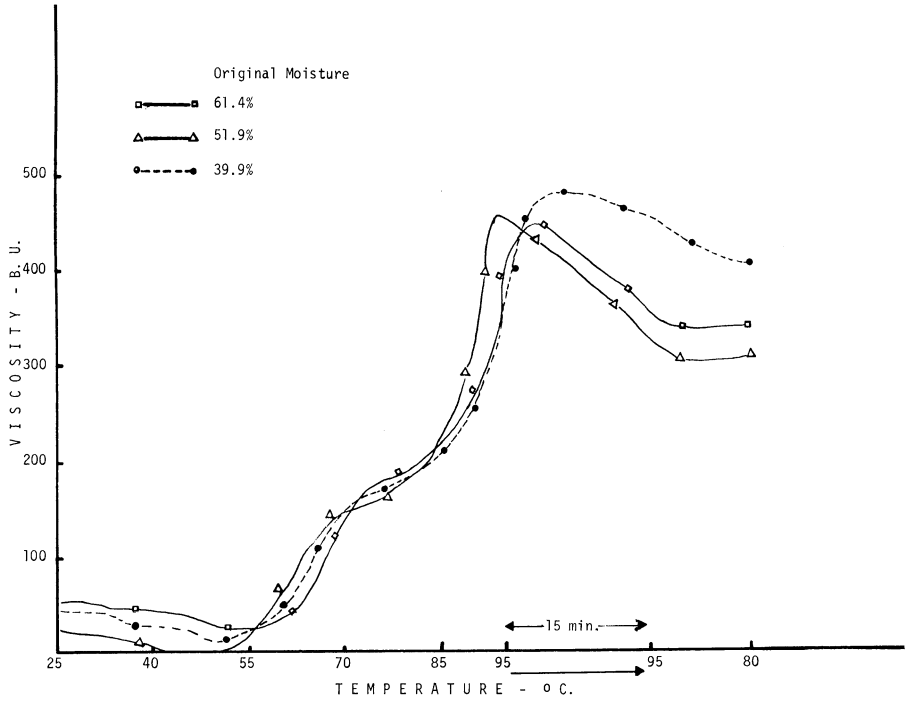

Fig. 4. Durum Leeds starch CMC-amylograph curves (corrected for viscosity of CMC).

TABLE V. STARCH INTRINSIC VISCOSITY VALUES AT DIFFERENT MATURATION STAGES (1969 CROP)

Original Moisture %	Intrinsic Viscosity [η]
HRS (Justin)	
70.0	1.65
49.3	1.90
9.9	1.80
Durum (Leeds)	
64.0	1.50
44.1	1.75
9.7	1.75

Effect of Stage of Maturity on Starch Intrinsic Viscosity. The intrinsic viscosity values of starch isolated from Justin flour and Leeds semolina at different maturation stages are shown in Table V. The results indicate the starch intrinsic viscosity values for the Justin variety increased from 1.65 to 1.90 as the original moisture decreased from 70.0 to 40.3%, then slightly decreased to 1.80 at the final stage of maturity. In the Leeds variety, likewise, an increase in intrinsic viscosity was observed as the original moisture decreased from 64.0 to 44.0%, with the same higher value being obtained at the final stage of maturity (Table V).

Bice et al. (6), who used a Stormer Viscometer, reported that no change in wheat starch viscosity occurred during maturation, while Briones et al. (7) noted an increase in relative viscosity of a rice starch dissolved in dimethyl sulfoxide during maturation. Medcalf and Gilles (15) concluded that intrinsic viscosity may give evidence concerning the relative molecular size of the various starches, and that this is controlled largely by the environmental conditions with little relation to wheat class or variety.

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