THE OTTAWA STARCH VISCOMETER—A NEW INSTRUMENT FOR RESEARCH AND QUALITY CONTROL APPLICATIONS[1]

P. W. VOISEY, D. PATON, and G. E. TIMBERS, Engineering Research Service and Food Research Institute, Research Branch, Agriculture Canada, Ottawa, Ontario K1A 0C6

ABSTRACT   Cereal Chem. 54(3): 534-557

A new versatile viscometer for measuring the properties of starch slurries during cooking is described and demonstrated. The effects of sample size, rotational speed, shearing gap size, and starch concentration on the characteristics of the viscosity, time, and temperature relationships were examined to establish optimum test conditions. The instrument records precise viscosity curves and indicates the slurry temperature in the region where it is undergoing shear. The viscometer is simple to calibrate and gives repeatable readings with 70 g of slurry. Essential starch characteristics can be obtained quicker than with existing instruments and more information is available about the initial swelling behavior. These advantages are gained in an instrument that simulates the conditions used in commercial processing. It was concluded that the Ottawa starch viscometer is useful for both research and quality control.

The rheological properties of starch slurries are important to the food industry. A popular instrument used to predict this starch behavior during processing is the amylograph (1) (C. W. Brabender Instruments, South Hackensack, N. J.). It is our opinion that, from the food processing viewpoint, this instrument does not adequately simulate commercial conditions or achieve precise measurements because: a) the starch is subjected to several shear rates by the pins; b) the shearing action is intermittent; c) the shear rates are not constant; d) the radiant electric heating and water cooling do not achieve comparable heat transfer rates; and e) the starch temperature is measured and controlled at an arbitrary point and not at the location where the maximum temperature occurs. Also, the test procedures are time-consuming. The spring cartridge indicating torque was replaced with a strain gage transducer to achieve constant shearing conditions (2) and to increase the precision of measurement. A similar transducer was used in a new viscometer (3) where the starch was mixed by a flat paddle rotating at constant speed in a stationary bowl. Cooking was accomplished by immersing the bowl in hot water. Experience with this instrument (4) indicated potential further development for research and quality control applications, to further reduce the time taken to obtain the essential behavioral characteristics of starch. The purpose here is to describe the new Ottawa starch viscometer, its performance, and the development of standardized test procedures, and to make a comparison with the amylograph.

DESCRIPTION OF APPARATUS

The viscometer (Fig. 1) comprises a brass bowl (J) with a spherical bottom and constant wall thickness. This gives uniform heat transfer and simulates the shape of a processing kettle. The bowl is held in a cradle (Fig. 2, A) mounted on a rotating shaft (P, Fig. 1) in a constant-temperature hot bath (H). The shaft (P) is driven by a belt (Q) from a variable speed 0.125 h.p., 1725-rpm motor (R), so that

the bowl-speed ranges from 30 to 300 rpm. Bowl-speed is sensed by a 60-tooth gear on the shaft and an electro-magnetic transducer connected to a digital tachometer reading to 4 digits to select and control speed within 0.1 rpm.

The starch slurry is stirred by a stationary flat paddle (K, Fig. 1) suspended on a shaft (G) (Fig. 2, B and C). A disk on this shaft covers (but clears) the bowl during operation to prevent sample evaporation. Rotation of the shaft (G) is prevented by a transducer to detect the torque generated by shearing the starch slurry. The torque transducer and head assembly move up and down vertically,
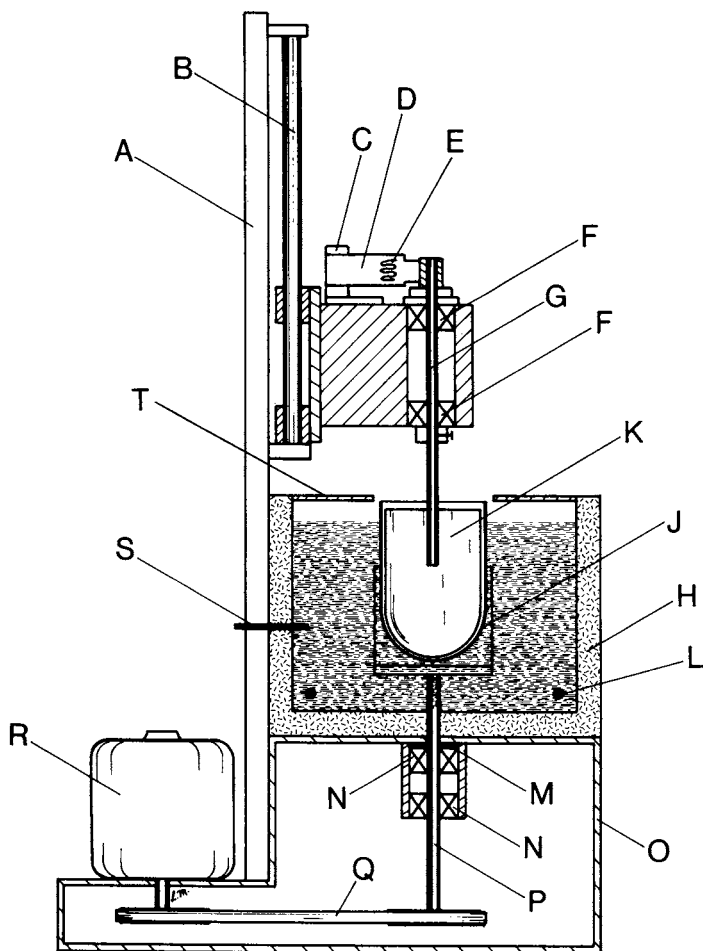

Fig. 1. Diagram of viscometer. A, Plate attached to base; B, rods; C, clamp; D, steel beam; E, strain gages; F, ball bearings; G, shaft; H, insulated water bath; J, bowl with spherical bottom; K, flat blade stirring paddle; L, immersion water heater; M, magnetic seal; N, ball bearings; O, base frame; P, bowl drive shaft; Q, knotched belt and pulley drive; R, variable speed motor; S, water bath temperature control sensor; and T, insulated cover over water bath.
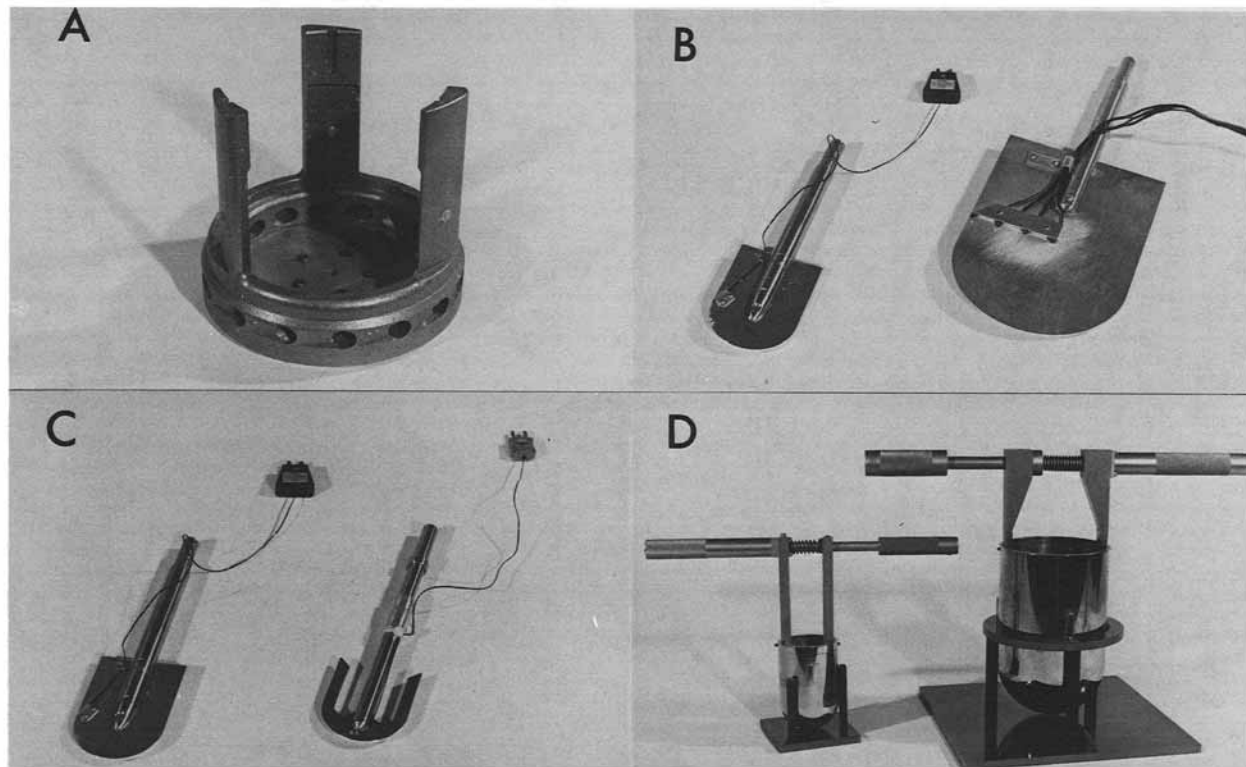
Fig. 2. Components of viscometer. A, Bowl cradle—note holes that circulate water as the cradle rotates; B, flat paddle stirring blades—on the left for the 100-cc bowl and on the right for the 1000-cc bowl, which also has four thermocouples installed to measure temperature distribution in slurry; C, flat stirring paddles for 100-cc bowl with the single thermocouple installed at the shearing edge—on the left the original solid design, and on the right the final design used; and D, 100- and 1000-cc bowls with lifting calipers (the bowls are mounted in their storage stands).

for sample loading, on two rods (B, Fig. 1) and are counterbalanced by a constant-force spring motor (Fig. 3, A). The operating position is accurately maintained by a rigid stop and locking mechanism so that the clearances between the paddle and bowl are repeatable.

The torque transducer is connected to an amplifier which is connected to a recorder to monitor slurry viscosity. Several different recorders were used to give curves of torque (*i.e.*, viscosity) against time or temperature at the paddle edge.
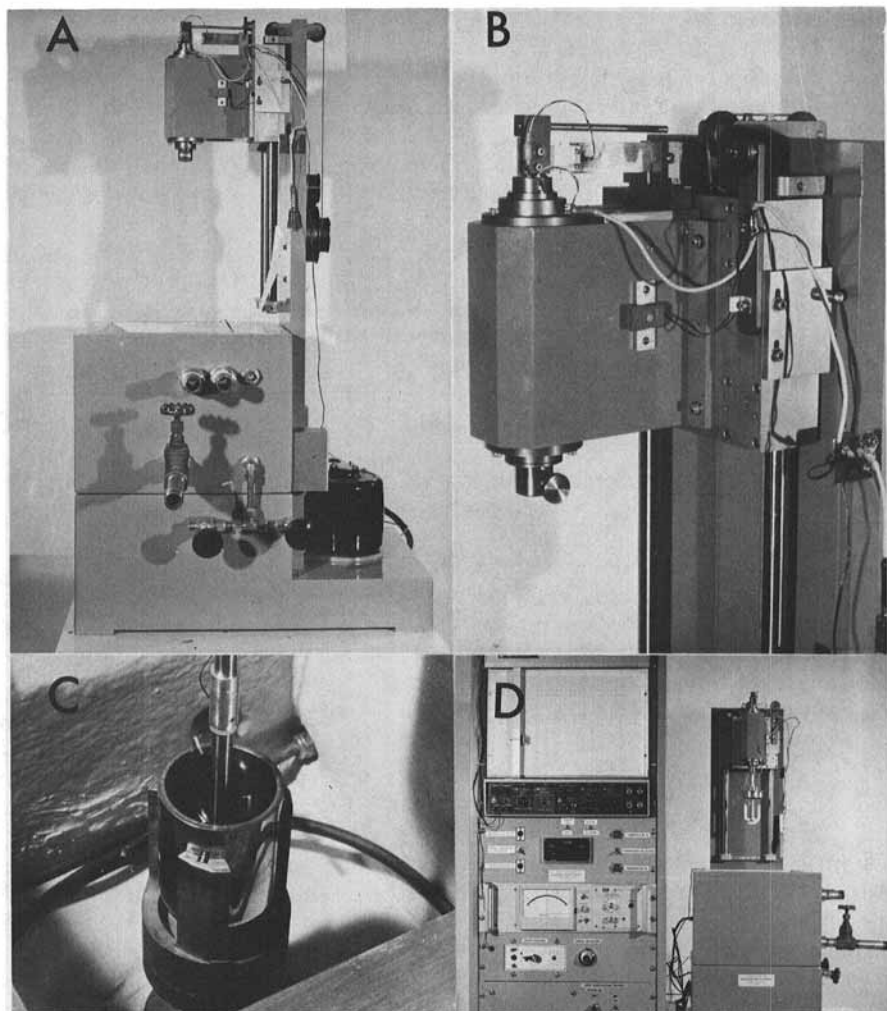

Fig. 3. A, Side view of viscometer—note the drains and overflow pipes for transferring water into and out of the bath and the sensing head counterbalance mechanism at the right hand side; B, close-up of sensing head; C, close-up of 100-cc bowl installed in its cradle in the water bath with the paddle partially lowered; and D, the viscometer and recording apparatus using an X-Y recorder to record torque directly against temperature.

The transducer is calibrated by applying a selected weight to a bar, at a 10-cm radius from the paddle center line, by means of a cord and pulley (5) (Fig. 3, B). The temperature of the starch at the paddle shearing edge is measured by a thermocouple clamped to the paddle with its tip level with the edge (Fig. 2, B and C). To measure spatial temperature distribution within the starch during preliminary tests, thermocouples were mounted at intervals across the blade (Fig. 2, B) and connected to an automatic data logging system to record temperature within 0.1°C and operating time within 1 sec. The thermocouples were calibrated from 25° to 100°C by immersion in water, using a precision thermometer as the reference.

The bowl size can be selected to suit the application. For research, only small quantities of starch may be available, whereas in quality control, large samples are desirable and generally available. Two bowl sizes are shown (Fig. 2, D), with capacities of 100 and 1000 cc, and it is estimated that sizes ranging from 25 to 4000 cc are possible. The work reported here was performed with the 100-cc bowl because, as with any physical test, reduction of sample size compounds design and operational problems.

Interchangeable blades, 1.63 mm thick, were manufactured (Fig. 2, B) to provide shearing gaps between the blade edge and bowl surface ranging from 0.5 to 3.0 mm in 0.5-mm increments. Initially, a solid flat blade was selected for ease of manufacture (3) (Fig. 2, B and C). However, the slurry built up on one side of the blade and the peak viscosity was not linearly related to the shearing gap size, as predicted by analysis. The blade was cut away on each side of the spindle (Figs. 2, C, and 3, C), which also better simulated the paddle shape used in commercial mixers.

Spring-loaded calipers engaged holes at the top of the bowl to handle the bowls when they were hot (Fig. 2, D). To cool the samples after cooking, a large drain rapidly drained the hot water into a storage bath to keep it heated until it was pumped back. The instrument bath was then filled and continuously flushed with cold tap water which exited via an overflow (Fig. 3, A and D).

The new design provides operational flexibility. The sample size can be varied within bowls and by choosing the bowl size and the shear rates selected by rotational speed or gap size between the bowl and blade edge. Heat transfer (*i.e.*, cooking) rates can be varied by selecting the temperature of the water bath and cooling water.

MATERIALS AND METHODS

Four commercial starches were used to provide a range of behavior for the tests: corn, waxy maize, wheat, and oat; six other starches were also tested, but are not fully reported here. A "quick" test cycle was used to evaluate the effect of various parameters. The starch slurry was heated until the peak viscosity was reached and then held for a selected time. The following were noted: a) the time taken and temperature to reach the onset of gelation (*i.e.*, the point where the torque starts to increase from zero, indicating that starch granule swelling has just begun—called the gelation point here); b) the time taken to reach the peak viscosity, starch temperature, and the torque at this point; and c) the torque after the selected holding time.

Slurries were prepared by mixing starch on a dry weight basis (d.w.b.) with 25°

± 1°C water. Standard test conditions were arbitrarily selected and then one parameter varied at a time. The conditions were: a) speed, 200 rpm; b) sample size, 100 g (100-cc bowl); c) starch slurry concentration, 9% (d.w.b.); d) shearing gap size, 1 mm; e) initial slurry temperature, 25° ± 1.5° C; and f) hot water bath temperature, 97° ± 1.5° C.

The effect of shearing gap size was evaluated by testing samples of the four starches using gap sizes ranging from 0.5 to 3.0 mm in 0.5-mm increments. Similarly, the effects of speeds ranging from 50 to 300 rpm in 50-rpm increments, concentration ranging from 3 to 10% in 1% increments, and sample sizes ranging from 50 to 100 g in 10-g increments were also determined. The samples were tested for a 7-min holding time after the peak viscosity. The repeatability of readings within a starch was evaluated by testing 10 replicates of each of the four starches under the arbitrary test conditions, except that 70-g samples and a 3-min holding time after the peak viscosity were used.

RESULTS

Instrument Performance

With the bowl cradle stationary, the water bath temperature varied by ± 1.5° C, which reduced to ± 0.6° C when the cradle was rotated. The maximum variation of water bath temperature was ± 1.4° C at the start of 99 sample test cycles, and at the end of the cycles, it was ± 0.5° C when the water bath temperature averaged 97.1° C (Table I). This was caused by inserting the 25° C sample and bowl into the 97° C bath. It was concluded that the temperature of the water surrounding the bowl was 97° ± 1.5° C for the duration of each test.

The relation between torque applied to the transducer (by increments of weights) and the recorder reading was linear within 1% for selected torques to

TABLE I
Variation of Initial and Final Bath and Starch Temperatures—Means
of N Samples and SD in °C

Test	A (gap)	B (speed)	C (size)	D (concentration)
No. of samples	24	23	24	28
Initial temperature				
Bath	98.2 1.3	97.5 0.7	97.6 1.1	97.9 1.4
Starch	26.0 0.8	24.9 1.7	25.5 1.1	26.8 1.2
Final temperature				
Bath	97.0 0.4	97.1 0.4	97.1 0.5	97.1 0.4
Starch	95.7 0.7	95.8 0.7	95.9 0.7	96.2 0.4
Difference[a]	1.3	1.3	1.2	0.9

[a]Difference between water bath temperature and starch temperature at the edge of the mixing blade.

give a full-scale chart reading ranging from 100 to 2000 cm g. Thus, the torque (*i.e.*, viscosity) was recorded with an accuracy of at least ± 0.5%.

Typically, the starch slurry temperature at the shearing edge increased uniformly and rapidly for about the first 1.7 min, and then increased at a
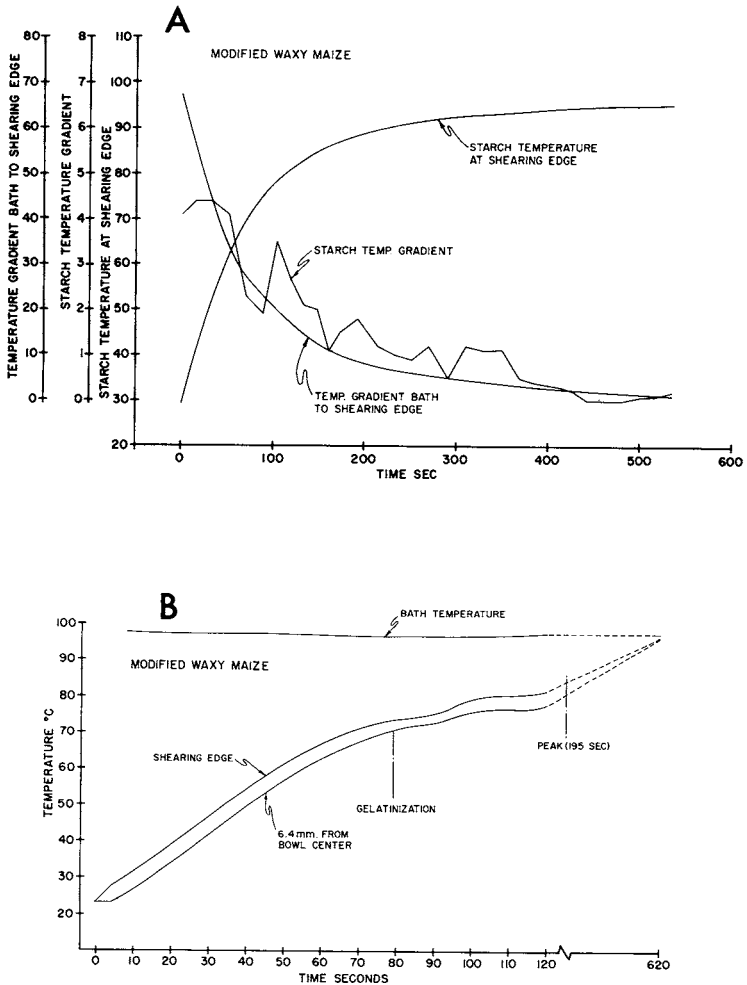

Fig. 4. Typical results, showing starch temperature during cooking, using modified waxy maize as an example. A, Records of the same temperature at the blade shearing edge, temperature gradient across the bowl radius within the slurry, and temperature difference between the water bath and slurry at the blade shearing edge; and B, plot of envelope formed by the minimum slurry temperature near the bowl center and the maximum slurry temperature at the blade shearing edge as starch is heated to the water bath temperature.

gradually decreasing rate with time until, at the end of the test, it approached the water bath temperature asymptotically (Fig. 4, A and B). The temperature difference between the starch at the blade shearing edge and the water bath 7 min after the peak viscosity ranged from 0.9° to 1.3° C (Table I). Slurry temperature maximized at the shearing edge. The temperature difference within the slurry between the center and edge of the blade was a gradient that fluctuated during the test (Fig. 4, A) but, on the average, the difference was uniform for most of the test cycle (Fig. 4, B), indicating uniform heat transfer through the sample. The maximum temperature difference in the slurry between the center and edge of the blade did not exceed 8° C for any of the 10 test starches at the start of the test cycle (average 6.3° C) and, within 3.3 min, this gradient averaged 2.2° C. At the end of the test cycle, the difference reduced to almost zero (Fig. 4, A and B). The vigorous stirring action of the blade rapidly reduced temperature variations in the slurry to negligible proportions, and the mass was subjected to uniform heating conditions. These results support a theoretical analysis that indicated a high heat transfer rate was achieved with the rotating bowl.

Effect of Shearing Gap Size

Decreasing the shearing gap size from 3.0 to 0.5 mm changed the characteristic shape of the viscosity-time curve and increased the peak torque (Fig. 5, A and B). The time to reach the gelation point changed nonlinearly with shearing gap size (Fig. 6, B), whereas the time to reach the peak viscosity was directly proportional to the gap size (Fig. 6, A) for the four starches tested. These times increased with
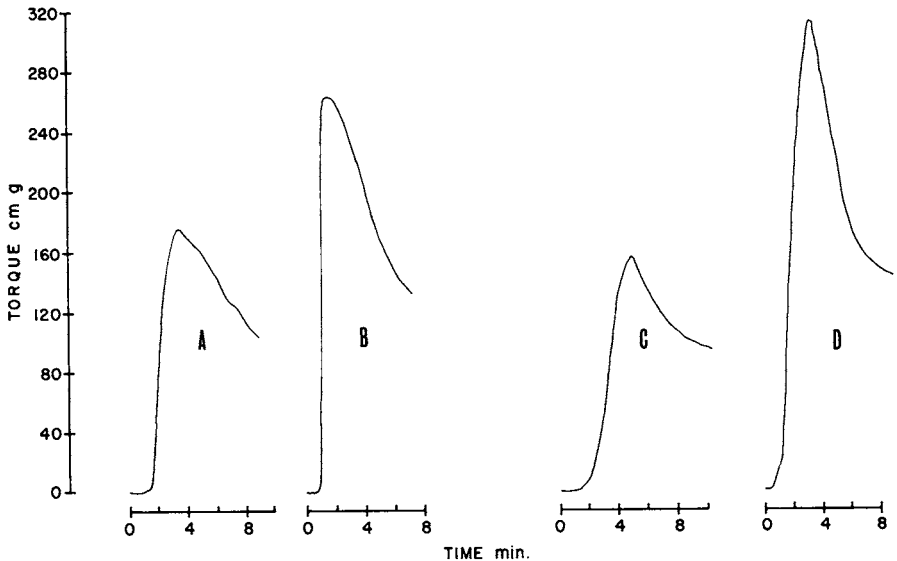

Fig. 5. Effect of variables on characteristic shape of viscometer torque-time records. A and B, corn starch using A, 3-mm, and B, 0.5-mm gap between blade and bowl; C and D, wheat starch tested at C, 50 rpm, and D, 300 rpm. All samples are 9% concentrations (d.w.b.).
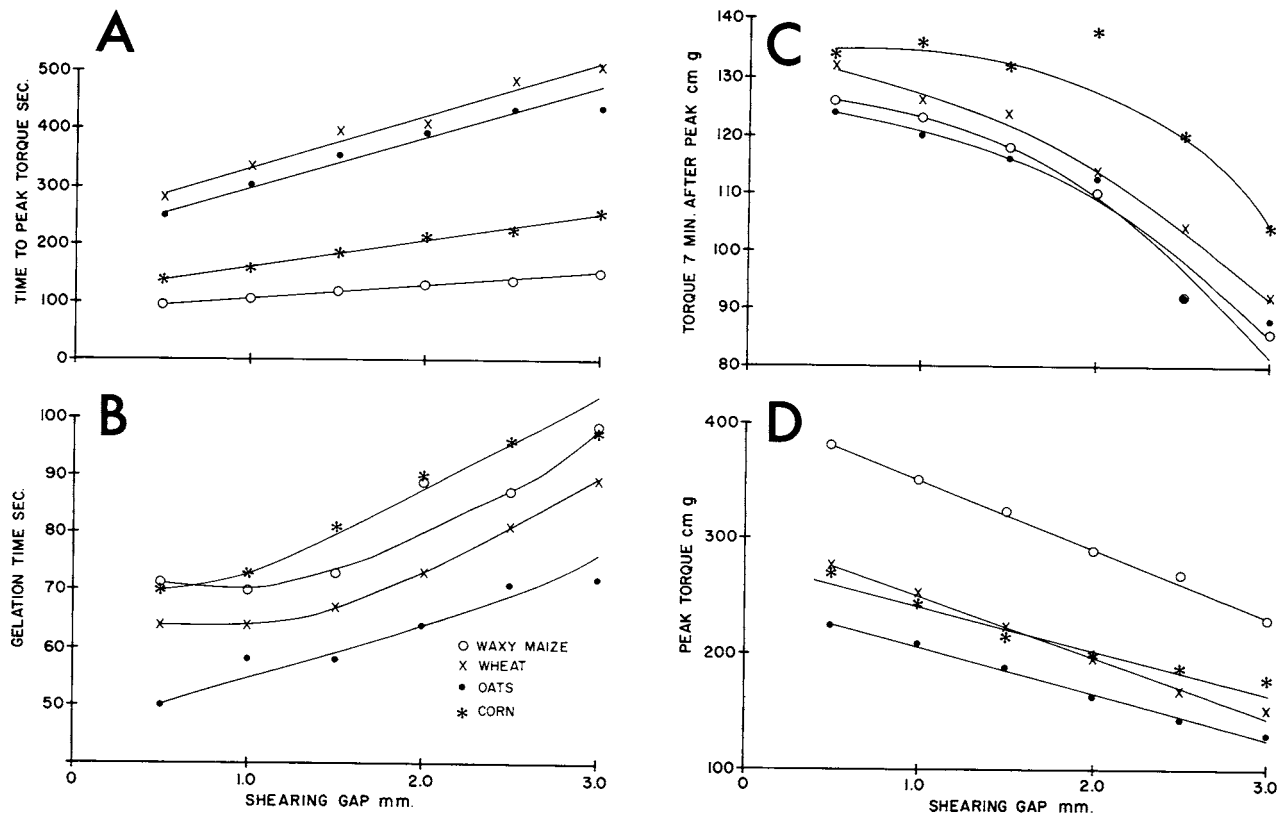
Fig. 6. Effect of changing the gap between the blade edge and bowl on: A, time to reach maximum viscosity; B, time to reach the gelation point; C, torque 7 min after the peak viscosity; and D, the peak viscosity. All samples are 9% concentrations (d.w.b.) and recorded at 200 rpm.

increasing gap size. The gelation point was reached within 1.5 min for any condition, and the peak viscosity was reached within less than 8.3 min. Varying the shear gap size did not affect the slurry temperature at the gelation point by more than $\pm 1.4°$ C or the temperature at the peak viscosity by more than $\pm 1.6°$ C (Table II).

The peak torque decreased linearly with increasing shear gap size (Fig. 6, D). The torque 7 min after the peak viscosity also decreased with increasing shear gap size, but the relationship was not linear (Fig. 6, C). The effect of decreasing the shear gap size was to speed up the process of cooking the starch due to the increased shear applied. Because the peak torque and time to reach this condition were linarly related to gap size, it is possible to predict results from one gap size to another, whereas with time to reach the gelation point and torque after a 7-min holding period, it is inconvenient due to the nonlinear relationship.

Effect of Rotational Speed

Increasing the rotational speed of the bowl from 50 to 300 rpm had an effect similar to reducing the shear gap size; *i.e.*, the characteristic shape of the viscosity time curves changed and the torque readings increased (Fig. 5, C and D). The times to reach the gelation point and the peak viscosity decreased nonlinearly with speed (Fig. 7, A and B). The maximum torque and the torque at the end of the 7-min holding period increased nonlinearly with speed (Fig. 7, C and D),

TABLE II
Effect of Changing Four Parameters on the Temperature Required to
Initiate Gelation and the Temperature at the Maximum Viscosity

Parameter Varied	No. of Increments in the Parameter	Starch Tested	Gelation Temperature °C Mean[a]	S.D.[a]	Temperature at Maximum Viscosity, °C Mean[a]	S.D.[a]
Shear gap size	6	Wheat	65.1	0.8	94.0	0.6
		Oat	61.8	0.9	93.9	0.6
		Corn	70.8	1.2	87.0	1.2
		Waxy maize	68.5	1.4	77.8	1.6
Speed	6	Wheat	67.5	3.9	93.5	0.5
		Oat	60.8	1.1	93.1	0.5
		Corn	70.5	0.4	88.0	0.8
		Waxy maize	70.3	0.6	77.7	0.8
Concentration	8	Wheat	...	...	94.1	0.8
		Oat	...	...	93.6	0.7
		Corn	...	...	90.4	2.2
		Waxy maize	...	...	81.8	3.7
Sample size	6	Wheat	65.3	0.7	93.2	0.3
		Oat	60.0	0.8	92.9	0.6
		Corn	71.4	1.0	86.3	0.5
		Waxy maize	68.4	1.0	77.6	0.8

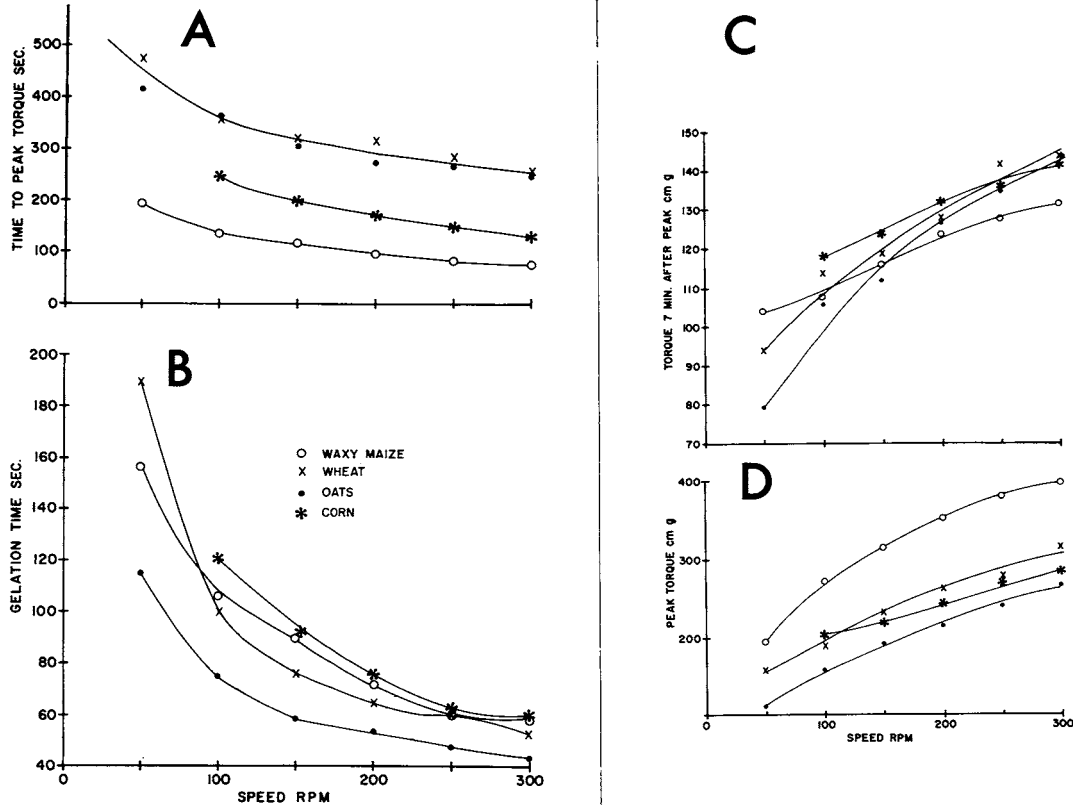[a]Mean and S.D. for one sample at each increment of the test parameter varied.

Fig. 7. Effect of changing the rotational speed of the bowl on: A, time to reach peak viscosity; B, time to reach the gelation point; C, torque 7 min after the peak viscosity; and D, maximum viscosity. All samples are 9% concentration (d.w.b.) and recorded with a 1-mm shear gap size.
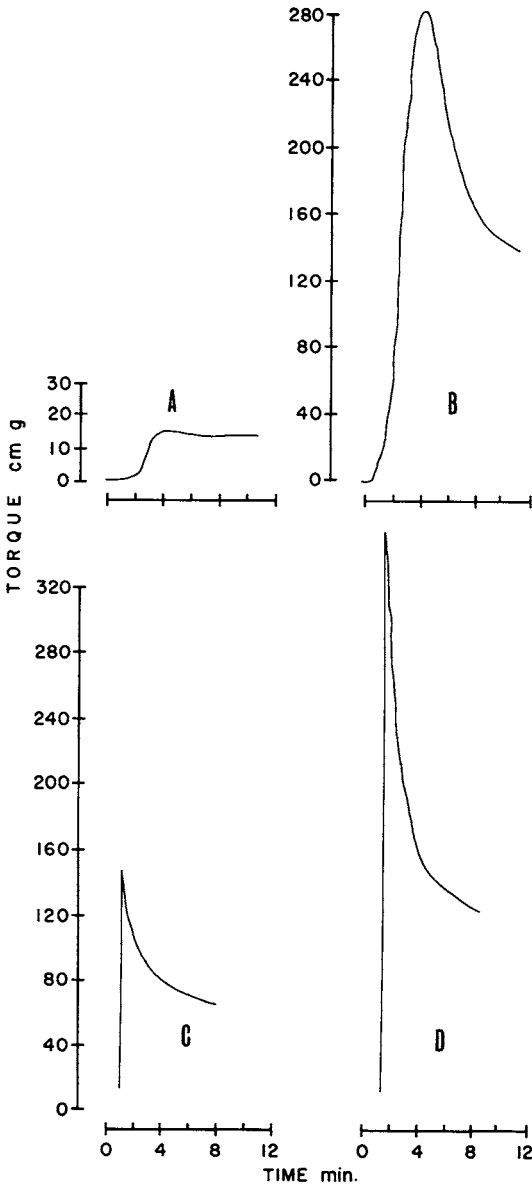
Fig. 8. Effect of variables on characteristic shape of viscometer torque-time records. A and B, oat starch using A, 4%, and B, 10% concentration (d.w.b.), 200 rpm and 1-mm shear gap size; C and D, waxy maize using C, a 50-g, and D, a 100-g sample size at 9% concentration, 200 rpm, and 1-mm shear gap size.
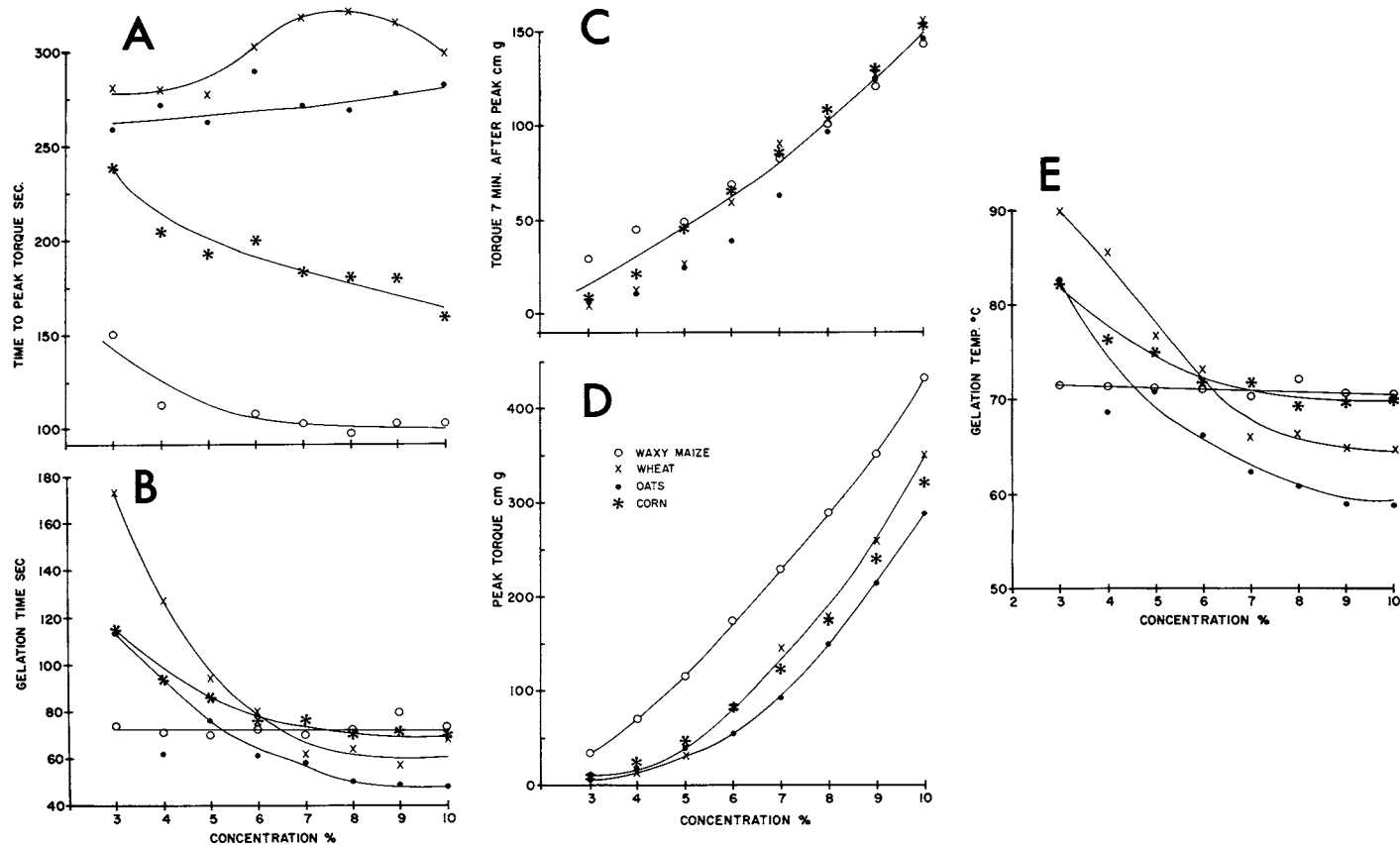
Fig. 9. Effect of changing the starch concentration on: A, time to reach maximum viscosity; B, time to reach the gelation point; C, viscosity 7 min after the maximum viscosity; D, maximum viscosity; and E, temperature to initiate gelation. Recorded at 200 rpm with a 1-mm shear gap size.

which was expected since starch slurries are known to behave in a non-Newtonian manner. Changing the speed did not affect the temperature required to reach the gelation point by more than $\pm$ 3.9° C, or the temperature at peak viscosity by more than $\pm$ 0.8° C (Table II).

Effect of Starch Concentration

Concentration had a marked effect on the characteristic shape of the viscosity curves (Fig. 8, A and B); torque increased with increased concentration. The relation between concentration and the curve parameters was generally nonlinear and complex, depending on the starch (Fig. 9). The time to reach the maximum viscosity increased with increasing concentration of wheat and oat starch, but decreased for waxy maize and corn (Fig. 9, A). The time to reach the gelation point decreased with increasing concentration for three of the starches, but remained virtually constant for waxy maize (Fig. 9, B). The maximum torque and the torque 7 min after the peak viscosity increased nonlinearly with increasing concentration (Fig. 9, C and D). Increasing the concentration did not change the slurry temperature at the peak viscosity by more than $\pm$ 3.7° C (Table II). For three of the starches (wheat, oats, and corn), the temperature at the gelation point decreased with increasing concentration (Fig. 9, E). Waxy maize appeared to have a constant temperature at the gelation point over the range of concentrations tested. A consistent relation did not exist between the time to reach the maximum viscosity and concentration (Fig. 9, A). This effect was small, however, since for the range of concentrations tested, the average times to reach the peak viscosity were: wheat, 5.0 ± 0.3 min; oat, 4.55 ± 0.16 min; corn, 3.18 ± 0.4 min; and waxy maize, 1.75 ± 0.3 min.

Effect of Sample Size

Sample size had a marked effect on the characteristic shape of the viscosity curves (Fig. 8, C and D). The times to reach the gelation point and the maximum torque increased linearly with sample size (Fig. 10, A and B). The maximum torque (Fig. 10, C) and torque 7 min later (Fig. 10, D) increased linearly with sample size up to 80 g for the four starches. This was expected, since theoretical considerations indicate that the increase should be linear. However, as the sample size was further increased, a nonlinear relationship was evident (above 80 g for waxy maize and above 90 g for wheat, oat, and corn starch). This was attributed to an "end effect" between the top of the stirring blade and upper surface of the starch. It was observed that at the maximum sample size the starch spilled over the top of the blade, which introduced additional shear, therefore increasing torque at the peak viscosity disproportionately. This caused the sudden increase in rate of change of peak torque with sample size (Fig. 10, C).

Thus, for the four starches tested, to eliminate or be able to predict variations due to sample size, the size should not exceed 80 g. To ensure that the "end effects" do not influence the results, a 70-g sample size would be a safer choice.

The temperature required to reach the gelation point did not change by more than $\pm$ 1° C within the range of test sample sizes (Table II). The temperature at the peak viscosity was similarly unaffected, varying less than $\pm$ 0.8° C.

Repeatability of Readings

Results for 10 replicates of four starches showed that the repeatability of all the readings taken from the viscosity curves was improved when the sample size was
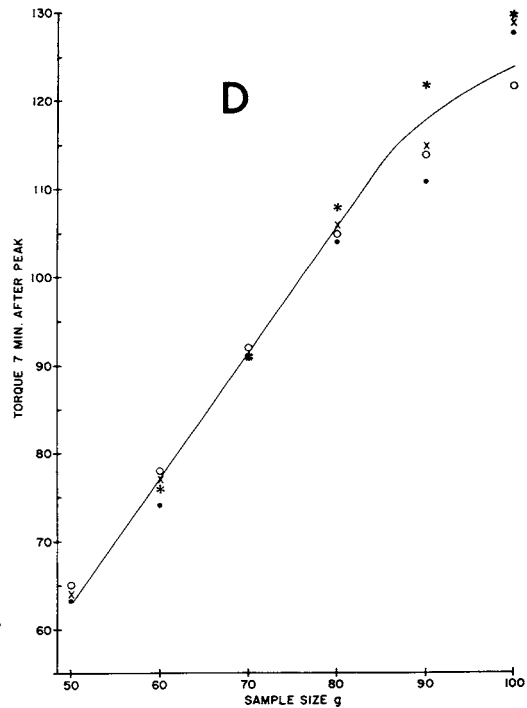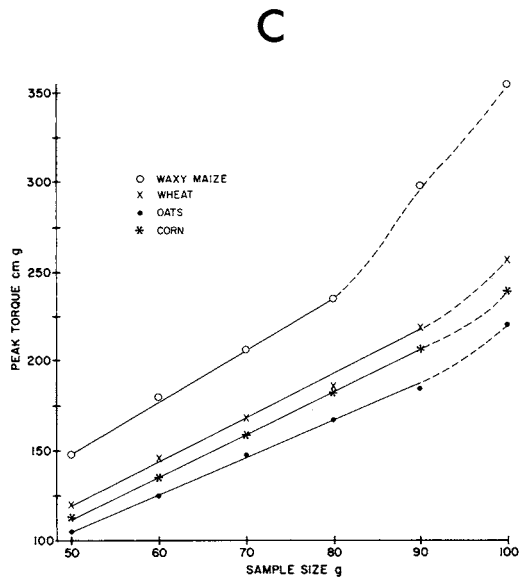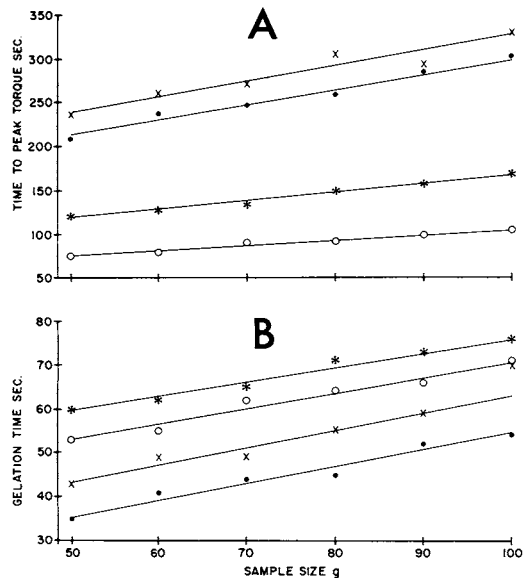
Fig. 10. Effect of changing the sample size on: A, time to reach the maximum viscosity; B, time to reach the gelation point; C, maximum viscosity; and D, viscosity 7 min after the peak viscosity. All samples are 9% concentration (d.w.b.) recorded at 200 rpm with a 1-mm shear gap size.

reduced from 100 to 70 g (*e.g.*, for wheat, Table III). Variations of all readings within each starch were of a low order, indicating that the instrument gave repeatable readings for critical temperatures, times, and viscosities in the test cycle (Table III). The results also indicated that during routine operation the initial starch temperature within 10 replicates did not vary by more than ± 2.3° C. The maximum variation in water bath temperature was ± 1.5° C at the start and ±

TABLE III
Summary of Data Showing Means and Variation for Ten Replicates of Four
Starches Tested Using a 1-mm Shear Gap at 200 rpm and a 9%
Concentration (d.w.b.) for the Readings Taken from the Viscosity Curves

Quantity of Slurry	g	100	70	70	70	70
Starch		Wheat	Wheat	Oat	Corn	Waxy maize
Time to gelation point, sec	Mean	63	51	45	61	63
	S.D.	4	1	2	3	3
	C.V. %	6	3	5	6	4
Temperature at gelation point, °C	Mean	65.0	65.6	61.6	70.9	71.8
	S.D.	1.0	0.8	0.9	0.5	1.1
	C.V. %	2.0	1.2	1.4	0.7	1.5
Time to peak torque, sec	Mean	317	269	245	138	83
	S.D.	15	6	14	6	3
	C.V. %	5	2	6	4	4
Peak temperature, °C	Mean	93.7	93.1	92.4	86.8	78.6
	S.D.	0.5	0.2	0.3	0.5	1.4
	C.V. %	0.5	0.2	0.4	0.5	1.8
Peak torque, cm g	Mean	252	177	147	159	203
	S.D.	5	2	4	2	2
	C.V. %	2	1	2	1	1
Torque 3 min after peak, cm g	Mean	165	120	105	125	113
	S.D.	6	1	3	1	1
	C.V. %	3	1	3	1	1

TABLE IV
Maximum Differences in Temperature at the Gelation Point and the Temperature
at the Peak Viscosity Observed within Five Different Tests in °C, Excluding the
Effect of Concentration on the Temperature at the Gelation Point[a]

Starch	Maximum Difference in Temperature at Gelation Point	Maximum Difference in Temperature at Peak Viscosity
Wheat	2.4	1.0
Oat	1.8	1.5
Corn	0.9	4.1
Waxy maize	3.4	4.2

[a]Tests: effects of gap size, speed, sample size, concentration, and repeatability.

0.6° C at the completion of the test cycles. The maximum variation in starch temperature at the end of the test cycle was ± 0.4° C for 10 replicates of any of the four starches.

Excluding the effect of concentration on the temperature required to reach the gelation point, examination of the results from the 5 different tests (gap size, speed, concentration, sample size, and repeatability) showed an excellent agreement between the gelation point and peak viscosity temperatures indicated for each starch by the results within each test. The difference depended on the starch and ranged from 0.9° to 4.2° C (Table IV). This was a further indication of the precision with which these temperatures can be determined and compared under different conditions.

CALIBRATION OF INSTRUMENT IN UNITS OF VISCOSITY

Because it was too complex to convert the torque readings to viscosity units by theoretical analysis, an empirical technique was required. A solution was to use oils of known viscosities as the reference. Therefore, a series of oils (Brookfield Engineering Laboratories Inc.) was tested with the water bath at 25° C. A replicate 70-g sample of each oil was used for each test condition. One series was
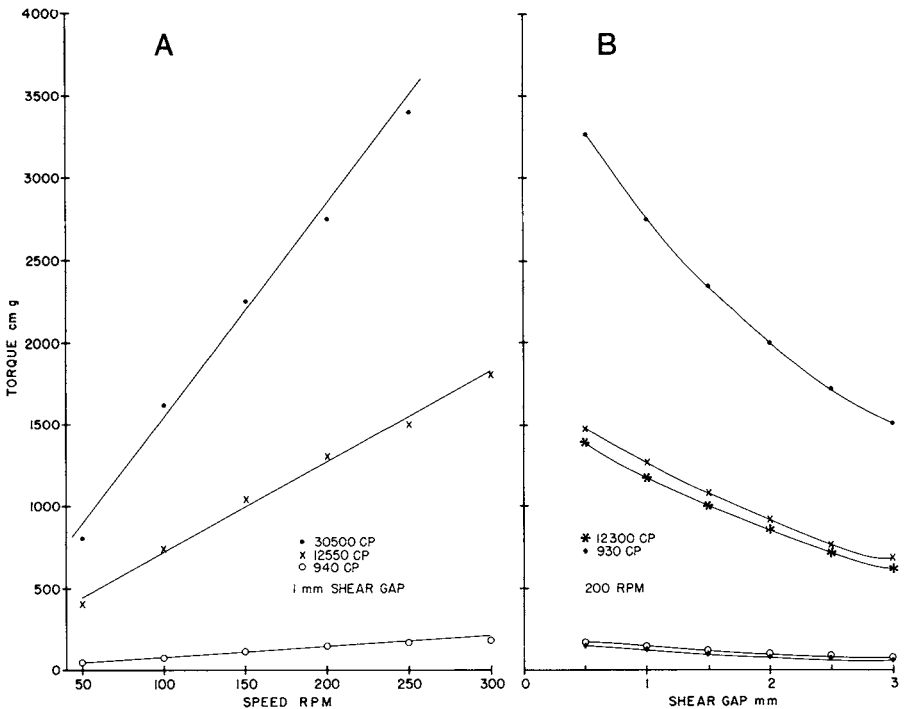

Fig. 11. Calibration curves from standard viscosity oils: A, at a fixed shear gap size of 1 mm and varying the speed; and B, at a fixed speed of 200 rpm and varying the shear gap size.

tested with a constant 1-mm shear gap size and torque readings obtained at speeds ranging from 50 to 300 rpm in 50-rpm increments. A second series was run at a constant speed of 200 rpm and shear gap sizes ranging from 0.5 to 3.0 mm in 0.5-mm increments.

These tests demonstrated that it was practical to calibrate the instrument torque scale in viscosity units by this method. The torque was linearly related to the rotational speed of the bowl (Fig. 11, A), which was expected since the oils are known to behave in a Newtonian manner. However, the torque and shearing gap size showed a slight nonlinearity in the relationship (Fig. 11, B), indicating that the shear rates imposed were not directly proportional to the shear gap size. This was attributed to the complex flows that occurred within the sample during mixing. It was observed that, providing undisturbed oil samples were tested each time, the torque readings were repeatable with a high order of accuracy. Once tested, the oils contained entrapped air and required at least a day to return to their standard condition. The instrument was sensitive to small changes in sample viscosity. For example, the difference between oil samples with a viscosity of 97 and 100 cP could be discriminated and represented a change in torque reading of 2 cm g which, in this case, was 2% of the recorder scale. A similar resolution was observed in comparing oils with viscosities of 12550 and 12300 cP and 940 cP (Fig. 11, B). The data also showed that torque and viscosity were linearly related under any given set of operating conditions for the range of oils tested.

If it is necessary to express the viscometer readings in viscosity units, a family of curves such as the one shown (Fig. 11) can be plotted. The apparent viscosity of a sample can then be read off the appropriate curve or interpolated between curves.

A DEMONSTRATION OF THE INSTRUMENT

Based on the preceding work, test conditions were adopted using a speed of 200 rpm, a 1-mm shearing gap, a 70-g sample size, and a 9% concentration (d.w.b.) to demonstrate potential applications of the instrument. The torque (*i.e.*, viscosity) was recorded directly against temperature at the blade edge on an X-Y recorder. The temperature axis was calibrated over the 25° to 100° C range and found to be precise within ± 0.3° C and repeatable within ± 0.1° C. Time was recorded with a stopwatch. The samples were cooked to peak viscosity, held for 3 min, and then cooled to 25° C.

Typical viscosity-temperature curves for four starches (Fig. 12) demonstrate the clear picture presented of the starch slurry behavior during the process. For example, it was readily apparent that minor deviations from a smooth increase in viscosity during initial cooking stages were attributable to slight variations in the water bath temperature, erratic changes in starch temperature at the shearing edge (Fig. 4), and periodic fluctuations in sample mixing. The degree of slurry homogeneity and its cohesiveness were indicated by small torque fluctuations, particularly near the peak viscosity and during the cooling period (Fig. 12, C *cf.* Fig. 12, D). The fluctuations provide subjective indexes of starch behavior.

The viscosity-temperature curve for corn starch (Fig. 12, B) is to be contrasted with the viscosity-time curve obtained from the same starch using an amylograph with electronic torque sensing (2) and a programmed heating rate of 1.5° C/min
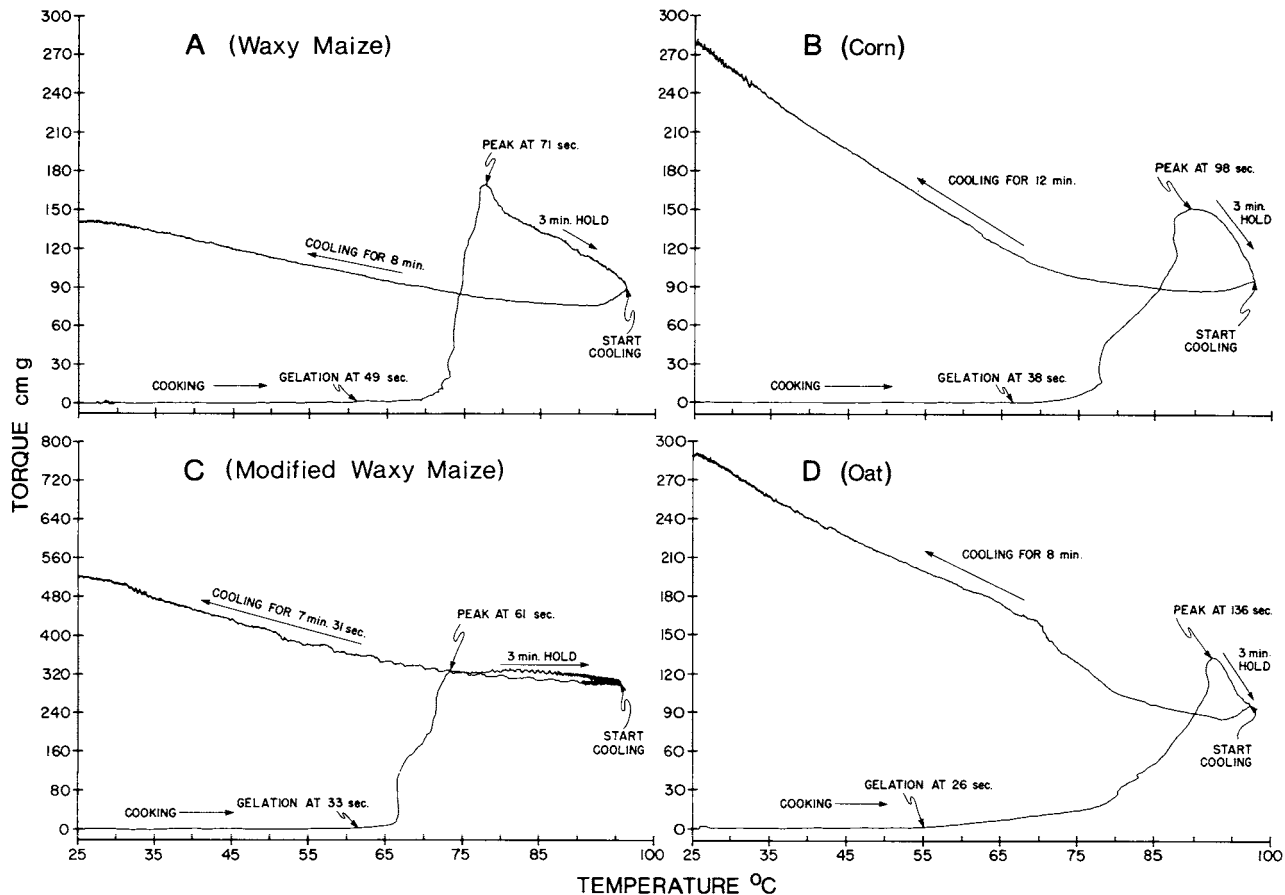
Fig. 12. Typical torque (viscosity)-temperature records for 70-g samples of 9% starch slurries tested at 200 rpm with a 1-mm shear gap size (cooked to the peak viscosity, held for 3 min, and then cooled). The times required to reach the gelation point, peak viscosity, and return to 25°C are noted. A, waxy maize; B, corn; C, modified waxy maize; and D, oat starch.

(Fig. 13, A). It was observed that the amylograph cooking curve was smooth and suggested abrupt and rapid swelling to peak viscosity with time (and therefore temperature), as opposed to the initial gradual swelling in the O.S.V. A viscosity curve (Fig. 13, B) was also obtained with the amylograph thermoregulator set at 95° C to more closely simulate heating conditions in the O.S.V. This reduced the cooking time from 45 to 20 min; however, use of the same chart speed here as was used with the programmed heating rate apparently results in a much sharper viscosity response with time and temperature.

The same corn starch cooked with the O.S.V. (but recording viscosity against time) produced a smooth curve, but it was traced in a much shorter time (Fig. 13, C). The fact that curves A, B, and C (Fig. 13) have different shapes was a function of the recorder sensitivity, the chart speed relative to the rise in viscosity, and the influence of the rate of heat transfer. A curve very similar in shape to C can be traced with the modified amylograph (2) by using the 20-min rapid heat cycle and a chart speed of the same magnitude as that used with the O.S.V. The initial
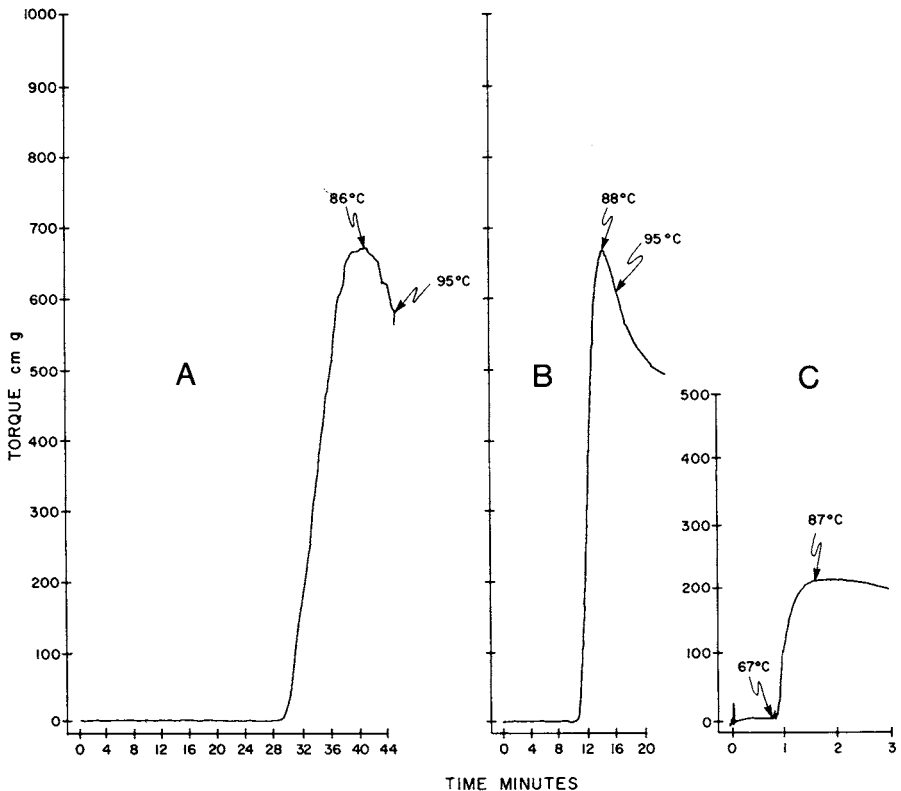

Fig. 13. A and B. Torque-time records obtained for corn starch with a modified amylograph (2) (with the same recorder as used to obtain Figs. 5 and 8), with the chart running at the same speed in each case: A, using a programmed heating rate of 1.5° C/min; B, with the thermostat set at 95° C to achieve the maximum heating rate; and C, obtained with the Ottawa starch viscometer and a different chart speed.

swelling phase can be better observed on the O.S.V. temperature viscosity curves, because the resolution is due to a combination of torque sensing mechanism sensitivity and direct temperature recording, which is not influenced by the recorder chart speed.

The viscosity-temperature curve for wheat starch using the O.S.V. (Fig. 14, A) is contrasted with the viscosity-time curve for wheat starch obtained with a conventional amylograph (Fig. 14, A) and employing the carboxymethyl
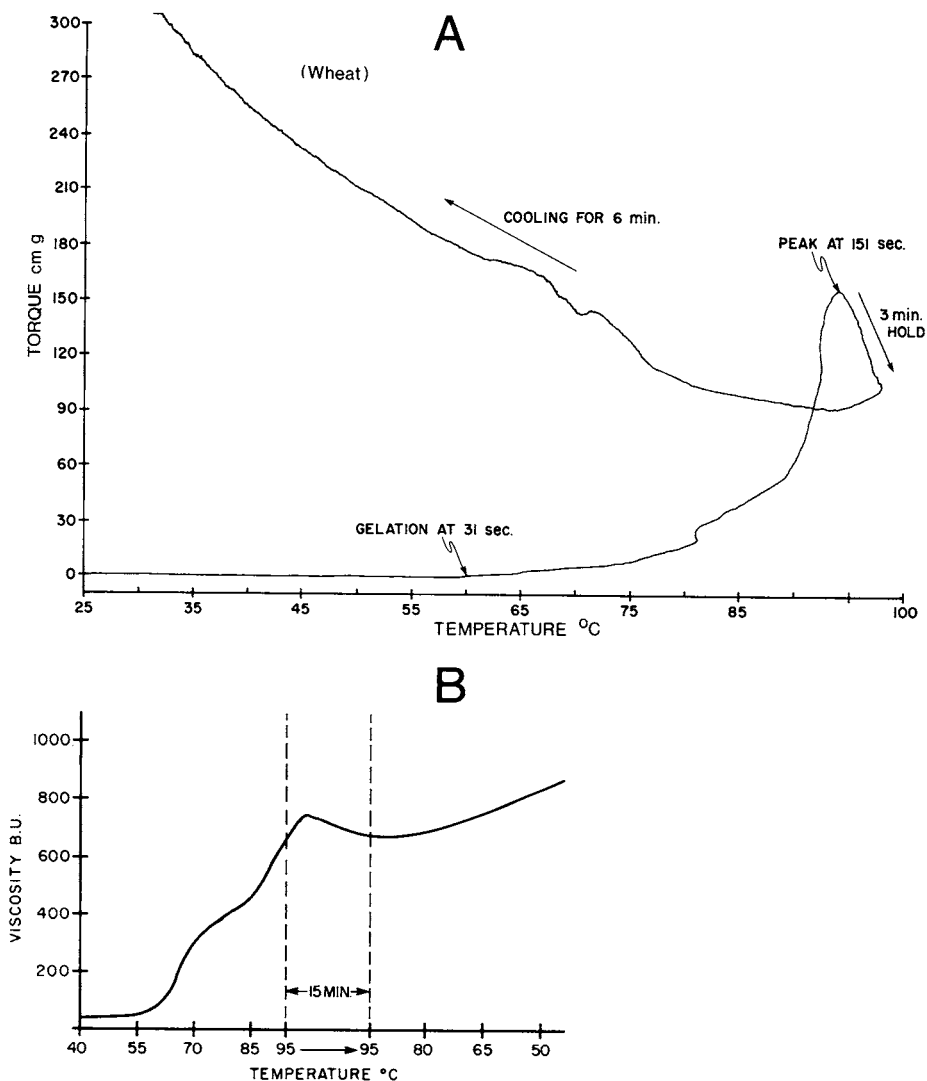

Fig. 14. Viscosity-temperature curves for wheat starch: A, from the Ottawa starch viscometer; B, adapted from Medcalf and Gilles (6), obtained with a standard amylograph and the carboxymethyl cellulose procedure.

cellulose modification (6). Both showed initial baseline viscosity deflections at approximately the same temperature (*i.e.*, the gelation point). The next stage was a steady increase in viscosity until a temperature of about 85° C. In the final stage, there was a further rapid viscosity increase until the peak was reached. This type of O.S.V. temperature-viscosity curve for wheat (Fig. 14, A), corn (Fig. 12, B), and oat (Fig. 12, D) starch was found to be repeatable. These viscosity increases are in contrast with that for waxy maize starch (Fig. 12, A), which showed a rapid constant swelling to predominate, which was more in keeping with the behavior of tuber starches. This demonstrates that the O.S.V. is capable of giving the same kind of information about the changes in swelling rate during cooking as the amylograph (6), but in a much shorter time.

An interesting point in the O.S.V. temperature-viscosity curves was the time taken for each event to occur (*e.g.*, corn and oat, Fig. 12, B and D). Although corn and oat starch differ in their peak viscosity temperatures by only $4°-5°$ C, the time to reach this point differed by 38 sec. Following the 3-min hold period, the oat starch cooled to 25° C in $2/3$ of the time taken for corn. These differences are not observed with a conventional amylograph because it is programmed at 1.5° C/min.

For efficient testing, several bowls are an advantage. Four bowls were manufactured to the same dimensions within practical manufacturing tolerances (± 0.025 mm). Tests showed that each bowl replicated the results from the others within the variation expected from the differences between samples of the same starch in a single bowl (Table III).

DISCUSSION

The principal advantages of the Ottawa viscometer system are: a) a reduction in time of 80 to 90% to observe critical events in the starch cooking process; and b) a reduction in slurry sample size of 84% (from 450 to 70 g) for research applications. The system is so sensitive, when operating with a torque-temperature recorder to plot viscosity directly against sample temperature, that essential information on the starch swelling can be obtained within 1 to 3 min. Rapid pasting curves can be equally well obtained with the Ottawa viscometer connected to a torque-time recorder. Curves of the same shape and size as those produced by the amylograph can also be obtained by using a torque-time recorder and selecting an appropriate chart speed and torque transducer sensitivity. More information is presented than with conventional instruments, particularly during the early cooking stages as the swelling of the starch granules begins. For example, the exact temperature of the starch actually undergoing shear at this point can be determined. This phenomenon is obscured in the amylograph by the friction and hysteresis in the mechanical torque indicating system (2), as well as by the programmed heating rate and location of the temperature-sensing element.

The Ottawa starch viscometer is a precise versatile instrument that is easy to calibrate by independent standards (weights and oil). The variable test conditions (rpm, shear gap size, sample size, and slurry concentration) offer a means of accelerating the testing process which is already shorter than with conventional instruments. Changes of these conditions do not affect two essential characteristics, namely, the temperature to initiate gelation and the

temperature to reach the maximum viscosity, with the exception of the concentration effect on the temperature at the gelation point. Changes in readings due to varying the test conditions are generally predictable. The operational flexibility of the instrument can be used with confidence as the need arises. Interchangeable bowls are easily made and they increase testing efficiency.

Satisfactory operating conditions for a range of starches are 200 rpm, a 1-mm shearing gap size, and a 70-g slurry sample. These can be changed to advantage, for example, to increase the torque readings (by speed increase and/or shear gap size reduction), thereby increasing the resolution of measurement when testing low starch concentrations. Maximum sample size must be limited to less than that which introduces blade end effects. Because a large range of sample sizes can be tested in different capacity bowls, the same instrument can be applied to research or quality control.

The new instrument subjects the starch to different shear rates in the spherical section at the bottom of the bowl, because a velocity gradient occurs. However, this gradient is constant and the shear rate at each point in the gradient is also constant. Thus, the starch is subjected to consistent shearing throughout the process while simulating a typical commercial process.

An important aspect in applying the new instrument is the correlation between the readings obtained and the readings from the amylograph, because extensive data are already on hand from the amylograph. Tests have shown that peak viscosity readings from the two instruments are highly correlated ($r^2 = 0.98$). Also, there is the question of how well the O.S.V. predicts starch behavior in a full-scale process. Preliminary experiments comparing the O.S.V. results, from a 70-g slurry sample, with a steam-heated kettle instrumented to serve as a viscometer with 68-kg samples indicated a close agreement.

CONCLUSIONS

The Ottawa starch viscometer provides an accurate, repeatable means of obtaining essential characteristics of starch slurries during cooking more rapidly than with the amylograph. Additional information, particularly during the initial phases of starch granule swelling, is readily observed because of the precision and resolution of the viscosity- and temperature-recording techniques. Because temperature is recorded where most of the shearing takes place and where the slurry temperature is the maximum, the viscosity-temperature relationships are more meaningful in relation to starch processing. The uniformity of cooking and heat transfer is a further advantage.

Instrument versatility is enhanced by a number of variable test conditions: a) bowl and sample size, and b) shear rate by rotational speed and shear gap size. These can be used to overcome testing problems that may be encountered in a wide range of starches and different applications. The work reported serves to demonstrate the potential of the new instrument, whose results appear to agree with those of the amylograph and a larger viscometer.

Literature Cited

1. MYERS, R. R., and KNAUSS, C. J. Mechanical properties of starch pastes, p. 393. In: Starch: Chemistry and technology, Vol. I, ed. by R. L. Whistler and E. F. Paschall. Academic Press: New York (1965).
2. VOISEY, P. W., and NUNES, A. An electronic recording Amylograph. J. Can. Inst. Food Technol. 4: 128 (1968).
3. VOISEY, P. W., MURRAY, R., and KEIGHTLY, G. A viscometer for studying starch slurry behaviour during cooking. J. Can. Inst. Food Sci. Technol. 5: 129 (1972).
4. KRUGER, L., and MURRAY, R. Starch texture. In: Rheology and texture in food quality, ed. by J. M. deMan, P. W. Voisey, D. W. Stanley, and V. F. Rasper. Avi Pub. Co.: Westport, Conn. (1976).
5. VOISEY, P. W., MILLER, H., and KLOEK, M. An electronic recording dough mixer. 1. The apparatus. Cereal Chem. 43: 408 (1966).
6. MEDCALF, D. G., and GILLIS, K. A. Wheat starches. 1. Comparison of physicochemical properties. Cereal Chem. 42: 558 (1965).