New Viscograph for Rheological Analysis of a Small Quantity of Wheat Flour

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ABSTRACT

A cone-plate viscometer was developed to analyze viscosity changes during heating of a small quantity of wheat flour suspension. The instrument consists of a cone, a plate, a temperature-control heater, and a water trap to prevent water evaporation from a sample. With this apparatus it is possible to record a viscogram using only ≈100 mg of wheat flour. Although the flour concentration was low, the temperature at which the viscosity began to rise in this cone-plate viscometer was closer to the gelatinization temperature determined by differential scanning calorimetry than that determined with other viscographs. It suggests that the new apparatus was more sensitive to initial swelling of gelatinized starch. The maximum viscosity in viscograms of wheat starch measured by the new instrument decreased with the α-amylase activity. Depending on the heating rate, viscogram parameters obtained with this instrument were well correlated with those obtained with other viscometers.

In various foods containing wheat flour, the properties of gelatinized starch are related to the rheological properties of those foods. Therefore, it is important to evaluate the behavior of gelatinized wheat starch to control the quality of wheat grain and the texture of wheat food products. One instrument used to analyze the pasting properties of wheat starch is the Brabender Amylograph (Brabender, Duisburg, Germany), which detects the changes in torque induced by a wheat flour or starch suspension in a rotating mixing vessel that is heated at a constant rate (Anker and Geddes 1944, Brown and Harrel 1944, Crossland and Favor 1947, Mazurs et al 1957, Leelavathi et al 1987).

However, measurements with the Brabender Amylograph generally require a large quantity of sample (65 g). At times, this requirement cannot be met, as is the case for wheat breeders. Chemical analysis of wheat flour properties can be done on a small amount of sample (Sulaiman and Morrison 1990). However, because of problems associated with sensitivity and sample preparation, the Brabender Amylograph is not suitable for rheological measurements on a small quantity of wheat flour. Therefore, it is necessary to develop an apparatus that enables the recording of a viscogram using a small amount of wheat flour.

Attempts to reduce the sample quantity needed to record a viscogram have been reported (Kumagai et al 1968, Kemph and Berlin 1972, Voisey et al 1977, Dengate and Meredith 1984, Imai et al 1988). New instruments with principles of measurement similar to those of the Brabender Amylograph have been marketed. The TS Viscograph (TSV) produced by Toyoseiki Seisakusho Ltd., Tokyo, Japan (Imai et al 1988) and the Rapid Visco Analyser (RVA) produced by Newport Scientific Pty. Ltd., Narrabeen, Australia (Rosset al 1987; Deffenbaugh and Walker 1989a,b; Panozzo and McCormick 1993). The smallest amount of sample required by those instruments is ≈1.0 g of wheat starch reported for the TSV or 3.0 g for the RVA.

In this investigation, we developed a viscometer capable of recording a viscogram on ≈100 mg of flour, a quantity of wheat flour that corresponds to the weight of the endosperm of several wheat kernels.

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The Brabender Amylograph and other similar instruments are equipped with agitators for mixing flour with water. However, the agitators do not produce a constant shear rate throughout the sample. Though the torque value recorded by the Brabender Amylograph is related to the sample viscosity, it is difficult to calculate viscosity. We designed a cone-plate viscometer capable of producing a constant shear rate, yet requiring only a small quantity of sample. The shallow geometry of the cone-plate viscometer eliminated settling of the solids during measurement and a humidified enclosure prevented evaporation of moisture. The constant shear rate makes it possible to calculate viscosity.

Several test conditions with the cone-plate viscometer were evaluated, and measurements at a high heating rate were also investigated to determine whether the apparatus could be used for rapid analysis. Finally, the parameters obtained with the cone-plate viscometer were compared with those obtained with other instruments.

MATERIALS AND METHODS

Six samples of wheat flour obtained from Zen-Nippon Seifun Kyodo-kumiai Rengo-kai, Tokyo, Japan, and commercially available wheat flour and starch (Sanwa-Denpun Co. Ltd., Kashihara, Japan) were used without further purification. The six flours gave different maximum viscosities (MV) in amylograms. Moisture content of flour samples was 14% (w/w). Barley-derived α-amylase reagent was purchased from Sigma Chemical Co., St. Louis, MO.

Structure of the New Viscograph

Figure 1 shows the cone-plate rotary viscometer used for this investigation. This apparatus was a prototype of Toyoseiki Seisakusho Ltd., Tokyo, and will be referred to here as the Mini-Visco.

Both the cone and plate of the Mini-Visco were made of stainless steel. The angle of the cone to the plate was 3°, and the cone was truncated (Whorlow 1980). The gap at the center of the plate was adjusted to 100 μm using a dial gauge. The radii of the cone and plate were 20.0 and 23.5 mm, respectively. It was possible to modify the sensitivity of the instrument by using a torsion spring with a different value for the spring constant (k) and by exchanging the cone with a different radius (R) or angle θ. In this investigation, a relatively flexible torsion spring with a maximum torque of 1.2×10^{-4} Nm was used to achieve high sensitivity. Overloading the torsion spring was prevented by a limiter beside the torsion spring.

To avoid frictional drag caused by rotation, the shaft attached to the center of the cone was connected to an air bearing, which was driven by dry air from a compressor. When the lower plate was

rotated at a constant rate by a motor, flow of the sample induced the torque in the upper part, then the shaft with an attached mirror rotated. The torsion angle of the upper part was calculated from the displacement, detected by a position sensor, of the mirror-reflected light generated by a light-emission diode. Because the torque (T) is proportional to the torsion angle $a(T = ka)$, the constant k was determined using several standard oils (2.5, 5, 10, and 50 cP). The viscosity η is:

$$\eta = (3T/2\pi R^3) / (\Omega/\theta)$$

where Ω is the angular velocity of the plate (Whorlow1980).

A space heater was used to heat the sample at a constant rate. Temperature during the measurements was controlled with a personal computer. An outer moat surrounding the sample plate was constructed in the heated zone, and water was put into the moat to prevent evaporation of water from the sample during measurement. A thermocouple was inserted into the water in the outer moat to monitor the temperature.

Experimental conditions were recorded on a personal computer (PC-9801, NEC, Japan) connected with the viscometer by an RS-232C port, to control the rotation of the plate, temperature, heating rate, and time of sampling. Temperature and viscosity data for a sample were output to the computer at every sampling time (1–3,600 sec) and stored on a floppy disk.

Viscosity Measurements

Wheat flour (80–140 mg) plus distilled water (total weight 2.0 g) were weighed directly on the plate of the Mini-Visco and mixed. Water (3.0 ml) was put into the outer moat, and measurement conditions were set up. The flour suspension was heated from 40 to 95°C at a constant rate (1–5°C/min), and held at 95°C for 5 min. Rotation rates varied from 10 to 30 rpm. The changes in the viscosity of the suspension were recorded every 5 sec.

As shown in Figure 2, four parameters were recorded: 1) pasting temperature (PT), temperature at which the viscogram first ascends from the baseline; 2) maximum viscosity (MV); 3) maximum viscosity temperature (MVT); 4) viscosity decrease (VD), between the MV and the minimum viscosity after the MV was recorded. The values for these parameters obtained from the Mini-Visco were compared with those obtained from the Brabender Amylograph, TSV, and RVA. Measurement conditions of each viscometer are shown in Table I.

Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) measurements were made (SSC 5200 with DSC-220 module, Seiko, Tokyo). Wheat flour and distilled water were weighed and mixed to obtain a 4.4–12.8% (w/w) suspension (total weight ≈50 mg) in a 70 µl silver pan. The pan was sealed hermetically and reweighed to determine the amount of water added. A reference pan contained a quantity of water equal to that in the sample pan.

Samples were heated from 20 to 120°C at a heating rate of 1.5°C/min, conditions similar to those used in measurements with a Brabender Amylograph. Onset temperature was determined as the intersection between the extrapolated baseline and the low temperature side of the endotherm.

α-Amylase Assay

α-Amylase activity was measured using a commercial α-amylase assay kit (Ceralpha, Megazyme, Sydney, Australia) with p-nitrophenyl maltoheptaoside as the substrate (McCleary and Sheehan 1987).

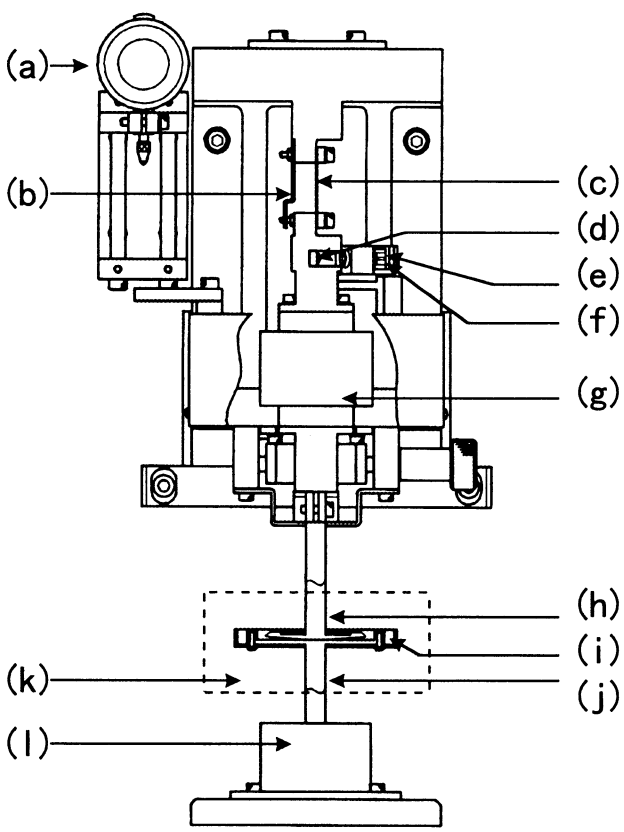

Fig. 1. Structure of the Mini-Visco: dial gauge (a), limiter (b), torsion spring (c), mirror (d), position-sensor (e), light-emission diode (f), air-bearing (g), cone (h), outer moat (i), plate (j), heating chamber (k), and motor (l).

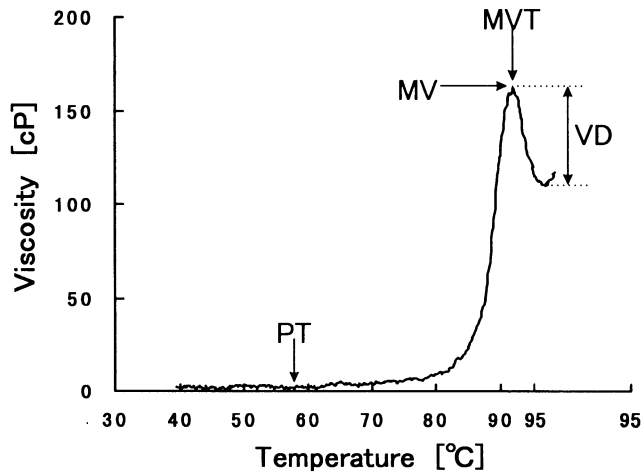

Fig. 2. Typical viscogram obtained with the Mini-Visco for a 7.0% (w/w) wheat flour suspension heated at 1.5°C/min from 40 to 95°C and held for 5.0 min. Rotation rate was 20 rpm. PT = pasting temperature, MV = maximum viscosity, MVT = maximum viscosity temperature, VD = viscosity decrease.

TABLE I
Standard Measurement Conditions for Four Viscographs[a]

	Mini-V	Amylo	RVA	TSV
Flour quantity (g)	0.1	65	4	1.5
Water quantity (g)	1.9	450	25	10
Rotation rate (rpm)	20	75	160	75
Temperature range (°C)	40–98	30–95	40–95	40–95
Heating rate (°C/min)	1.5/5.0	1.5	5.0	1.5

[a] Mini-V = Mini-Visco, Amylo = Brabender Amylograph, RVA = Rapid Visco Analyser, TSV = TS Viscograph.

RESULTS AND DISCUSSION

Temperature Difference Between Water in the Outer Moat and Sample on the Plate

Because of the shallow gap, it is difficult for a cone-plate viscometer to measure the sample temperature directly. If a thermosensor comes into direct contact with the sample suspension, it may affect the viscosity value. Therefore, a thermocouple was inserted into the outer moat of the Mini-Visco. Then the difference in temperature between the solvent in the outer moat and the sample on the inner plate was measured to determine whether the water temperature could be substituted for the temperature of the sample.

When the heating rate was set at 1.5°C/min or 5.0°C/min, the difference in temperature between water in the outer moat and sample on the inner plate was <1.0°C over a wide range of temperatures (30–95°C). Therefore, the water temperature in the outer moat was recorded for viscograph measurements in this investigation.

When water (3.0 ml) was put into the outer moat, the water loss of the sample after a standard measurement was <1.0%. The water in the outer moat prevented moisture evaporation in the sample.

Viscosity Measurements with the Mini-Visco

Figure 2 shows a typical viscosity curve obtained with the Mini-Visco for a wheat flour suspension heated at a constant rate. Its shape was similar to that obtained with the Brabender Amylograph. Because the Mini-Visco was constructed with a cone-plate geometry, the viscosity value could be calculated in cP units. Generally, food products including starch suspensions are non-Newtonian fluids, and since the viscosity depends on the shear rate, the rheological behavior is complex (Dickinson 1992). The Mini-Visco, with its cone-plate geometry, facilitates theoretical treatments of viscosity.

Similar viscograms were obtained under various conditions: wheat flour concentrations of 4.0–7.0% (w/w); heating rates of 1.0–5.0°C/min; plate rotation rates of 10–30 rpm; and corresponding shear rates of 20–60 sec^{-1}. The Mini-Visco could record a viscogram using 80–140 mg of wheat flour suspended in <2.0 ml of water.

Table II shows the means and coefficients of variation (CV) of the PT, MV, MVT, and VD parameters in the viscograms for a 5.0% (w/w) wheat flour suspension measured six times with the Mini-Visco. The reproducibility of the parameters was high although the amount of sample (100 mg of wheat flour) was small.

TABLE II
Reproducibility of the Viscogram Parameters[a] Measured by a Mini-Visco[b]

Parameter	Mean	CV (%)[c]
PT (°C)	65.5	2.5
MV (cP)	44.6	4.7
MVT (°C)	93.0	0
VD (cP)	5.0	11.3

[a] PT = pasting temperature, MV = maximum viscosity, MVT = maximum viscosity temperature, VD = viscosity decrease.
[b] Wheat flour suspension (5.0%, w/w) measured six times at 20 rpm and 5.0°C/min.
[c] Coefficient of variation calculated for each parameter.

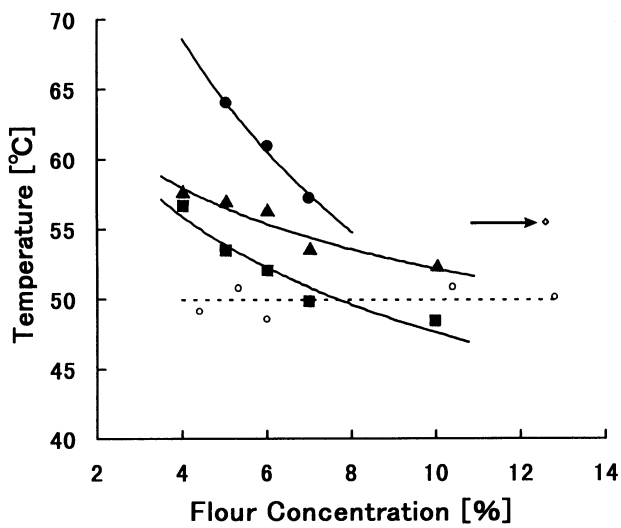

Fig. 3. Relationship between flour concentration and pasting temperature in viscograms and the onset temperature (○ - - -) measured by differential scanning calorimetry. Heating rate 1.5°C/min. Plate rotation rates: 10 (●), 20 (▲), and 30 rpm (■). Arrow indicates pasting temperature obtained with the Brabender Amylograph under the conditions given in Table I.

Dependence of Viscograms on Wheat Flour Concentrations or Rotation Rates

Figure 3 shows the effect of the wheat flour concentration and rotation rate of the plate on the PT obtained with the Mini-Visco. The effect of the wheat flour concentration on the onset temperature measured by DSC is also shown. PT decreased as the wheat flour concentration increased. This was also observed in Brabender Amylograph measurements (Anker and Geddes 1944, Crossland and Favor 1948). On the other hand, the onset temperature recorded by DSC corresponding to the gelatinization of starch was independent of flour concentrations at 4.4–12.8%. Similar observations were previously reported for starch (Biliaderis et al 1986, Kohyama and Nishinari 1991).

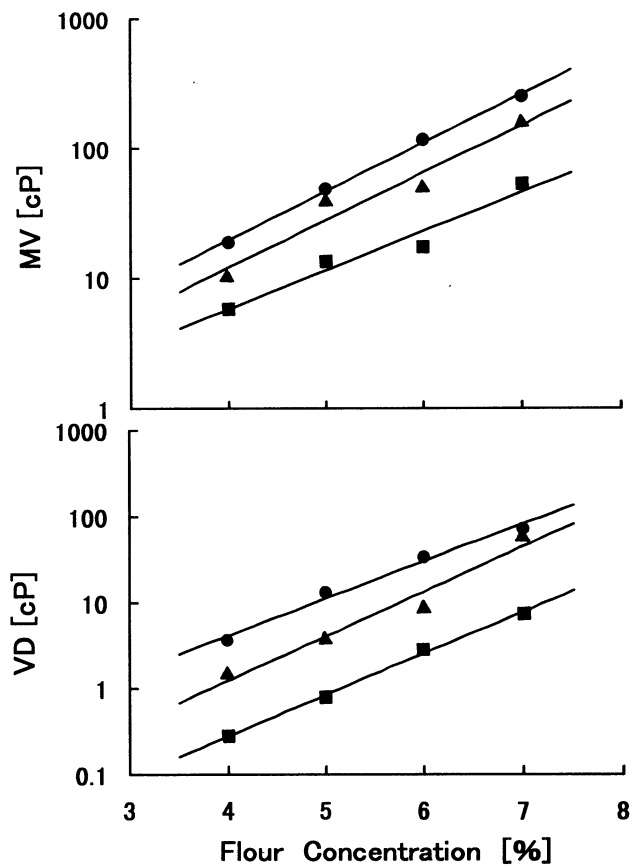

Fig. 4. Relationship between flour concentration, maximum viscosity (MV), and viscosity decrease (VD) in viscograms. Heating rate: 1.5°C/min. Rotation rates: 10 (●), 20 (▲), and 30 rpm (■).

The PT measured with the Mini-Visco was near the gelatinization temperature determined by DSC because of the sensitivity of the Mini-Visco, which detected a slight change in viscosity as soon as starch began to gelatinize. PT decreased with as the rotation rate of plate increased, suggesting that a slight change in viscosity could be detected better at 30 rpm than it could at 10–20 rpm.

The amount of sample required in the Mini-Visco measurements was small (Table I). The PT value recorded by the Mini-Visco at a rotation rate of 20 rpm and at flour concentration of >7% or at a rotation rate of 30 rpm and at flour concentration of >5% was lower than that of the Brabender Amylograph and was closer to the gelatinization temperature.

Figure 4 shows relationship between flour concentration and MV or VD values in viscograms. MV and VD values increased linearly as wheat flour concentration increased, as previously reported by Leelavathi et al (1987) and Imai et al (1988). MV and VD values decreased as shear rate (rotation rate of the plate) increased, indicating that the wheat flour suspension behaved as a pseudoplastic fluid (Doublier et al 1987, Ellis et al 1989).

The ratio of VD to MV obtained with the Mini-Visco (11.2%) was relatively lower than that recorded by other viscometers (Brabender Amylograph, 32.1%; RVA, 67.2%; TSV, 59.6%). Generally, a VD at ≈90°C is considered to be the result of the disintegration of gelatinized and swollen starch granules (Smith 1964). In Mini-Visco measurements, it appears that gelatinized and swollen starch granules did not disintegrate as readily as they did in other instruments because of the reduced shear rate and the low sample concentration (Table I).

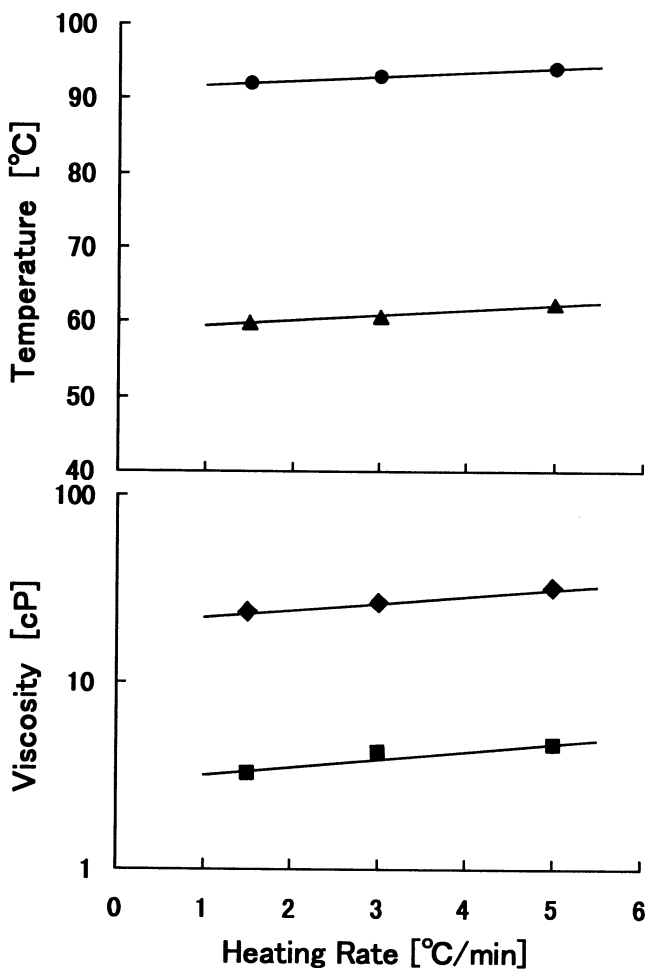

Fig. 5. Relationship between heating rate and viscogram parameters (● = maximum viscosity temperature; ▲ = pasting temperature; ♦ = maximum viscosity; ■ = viscosity decrease). Concentration of flour suspension: 5.0%, w/w. Rotation rate: 20 rpm.

Effect of Heating Rate on Viscogram Parameters

With the Mini-Visco it was possible to raise the temperature linearly up to 5.0°C/min. Figure 5 shows the effect of the heating rate on the viscogram parameters. PT increased as heating rate increased (Bloksma 1980). As starch swelling in a heated flour suspension is a nonequilibrium change, the parameters derived from a viscogram are likely to change with both temperature and time. Starch granules in flour-water mixtures heated at a slow rate are exposed to a given temperature longer than those heated at a rapid rate. Thus, the pasting temperature increases somewhat with heating rate increases (Fig. 5).

MV and VD values also increased as the heating rate increased (Deffenbaugh and Walker 1987a, Ushiyama et al 1994). Eliasson (1986) observed that the changes in the rheological properties during gelatinization of starch depended on the presence of swollen starch granules as a dispersed phase, an amylose-amylopectin matrix as a continuous phase, and the interactions between both phases. Moreover, Doublier et al (1987) reported that with the increase of the heating rate, swelling of wheat starch granules and solubility of amylose were enhanced, and consequently, the viscosity of the paste increased. Ellis et al (1989) also demonstrated wheat starch pastes gave increased viscosity when heated at a high rate.

Correlation Between α-Amylase Activity and MV

The main purpose of obtaining the viscogram parameter values was to determine the α-amylase activity induced by sprouting of

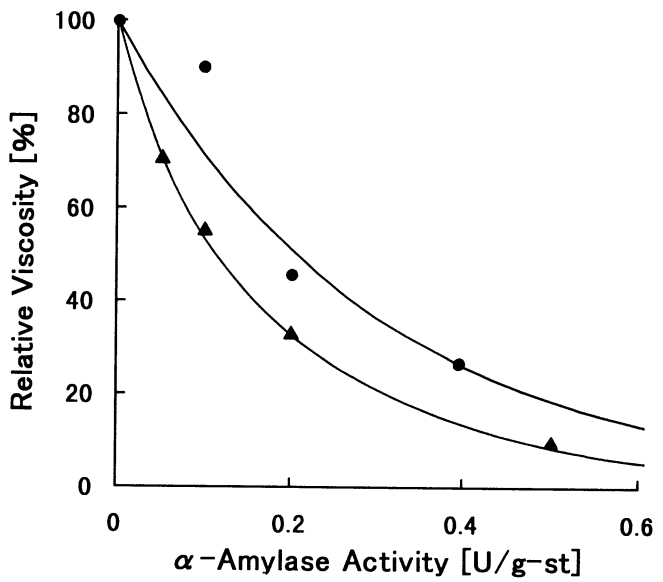

Fig. 6. Relationship between α-amylase added to wheat starch and the maximum viscosity of the paste. Viscosity value expressed as relative to the standard. ● = Mini-Visco (6.0%, w/w, wheat starch suspension, 1.5°C/min, 30 rpm). ▲ = Brabender Amylograph (12.6%, w/w, wheat starch suspension, 1.5°C/min, 75 rpm).

TABLE III
Correlation Coefficients for Viscogram Parameters[a] Using Three Viscographs[b] Compared to the Brabender Amylograph

	Heating rate (°C/min)			
	Mini-V		RVA	TSV
	1.5	5.0	5.0	1.5
PT	0.801	0.191	0.854*[c]	0.437
MV	0.874*	0.823*	0.942**	0.885*
VD	0.464	0.885*	0.872*	0.850*

[a] PT = pasting temperature, MV = maximum viscosity, VD = viscosity decrease.
[b] Mini-V = Mini-Visco, RVA = Rapid Visco Analyser, TSV = TS Viscograph.
[c] * = $P < 0.05$, ** = $P < 0.01$.

wheat grain. When the MV decreases because of α-amylase activity, the commercial value of the wheat flour decreases. Therefore, it is important to determine the α-amylase activity of wheat grain or flour simply and rapidly.

Figure 6 shows the effect of added α-amylase on the MV of wheat starch measured with the Mini-Visco and the Brabender-Amylograph. The viscosity was expressed relative to that for the control containing no added α-amylase. The MV values recorded with the Mini-Visco and the Brabender Amylograph both decreased with an increase of in α-amylase activity. This observation suggested that the α-amylase activity in flour could be deduced from the MV value when using the Mini-Visco. However, the slope of the curve in which MV was plotted against the α-amylase activity was slightly lower as determined with the Mini-Visco than with the Brabender Amylograph. This finding suggested that the Mini-Visco was less sensitive to the α-amylase activity than was the Brabender Amylograph. The difference in sensitivity may be caused by the higher level of solids (12.6%, w/w) used in the Brabender Amylograph compared to 6.0% (w/w) in the Mini-Visco, or by the different stirring mechanisms used in the two instruments. However, the Mini-Visco was able to evaluate the α-amylase activity with a small amount of sample.

Correlation of Results from the Mini-Visco with Other Instruments

Six samples of flour were tested on commercial instruments (Brabender Amylograph, RVA, and TS Viscograph) to compare the results with those obtained using the Mini-Visco (Table I). Table III shows the simple correlation coefficients for PT, MV, and VD values obtained by the various instruments compared with those obtained from the Brabender Amylograph. Since variation in MVT was small (<1.0), correlation for MVT values was not reported. Although the RVA and TSV were not originally designed to evaluate PT values, those values are included for comparison.

The PT and MV values measured with the Mini-Visco at a heating rate of 1.5°C/min showed a relatively high correlation with those measured by the Brabender Amylograph. At 1.5°C/min, the correlation coefficient for VD values obtained with the Mini-Visco and with the Brabender Amylograph was not significant because the VD value recorded with the Mini-Visco was lower when compared with that measured with the Brabender Amylograph.

When the heating rate was 5.0°C/min for rapid analysis, the MV and VD values measured with the Mini-Visco were well correlated with those of the Brabender Amylograph.

These observations suggest that the optimum measurement conditions for the Mini-Visco are 1.5°C/min for PT and MV and at 5.0°C/min for MV and VD.

CONCLUSIONS

The Mini-Visco, a cone-plate viscometer, was developed. The characteristics of the new instrument are: 1) it is possible to record a viscogram using a small amount of wheat flour suspension (2.0 g), containing 80–140 mg of wheat flour. 2) it is possible to deduce the level of α-amylase activity with starch; 3) viscogram parameters were well correlated with those obtained with Brabender Amylograph. Optimum measurement conditions on the Mini-Visco are a heating rate of 1.5°C/min to determine PT and MV and 5.0°C/min to determine MV and VD.

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