

Temperature of Liquid Contents in RVA Cans During Operation^{1,2}

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ABSTRACT

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The Rapid Visco-Analyser (RVA) normally does not indicate the actual temperature inside the sample can during operation, but rather that of the heated block. Sample cans were modified by incorporating a thermocouple that was immersed in the contents, and its temperature could then be monitored during operation. Three different starch types and four temperature profiles of differing ramping rates were used. It

was found that the actual paste temperature lagged behind the block temperature, and the lag was a function of the heating/cooling rate and the apparent peak viscosity of the starch sample. A second-order multiple-regression equation ($r = 0.94$) was developed to predict that lag. The results can be used to predict the actual liquid temperature for any given point on the pasting curve.

Initially, the Australians developed the Rapid Visco-Analyser (RVA) (Newport Scientific Pty. Ltd., Warriewood, NSW, Australia) as a tool to measure the extent of sprout damage for a wheat crop (Ross et al 1987). The first RVAs did not have computer-controlled temperature. The temperature programs were limited to three set points, each for a limited choice of times. When the set point was changed, the heating or cooling operated at maximum until the desired temperature was approached. No linear ramping was available, as it is today. Early experiments with ramping required the operator to manually increase the temperature control by one degree for every time unit (i.e., every 40 sec for a rate of 1.5°C/min). This was satisfactory for studying the instrument and potential applications, but not for routine use. Unlike the Brabender Visco/Amylograph, the operating temperature of the RVA is based on feedback from the block temperature of the machine, not from the temperature of the sample. The temperature of the liquid was known to lag behind that of the heating block, but the amount of difference between the two was not known.

Early on, the RVA developers recognized this problem, complicated by the fact that the block temperatures were not accurately known. An attempt was made to measure the temperature of the contents during the machine's operation. Through a small hole drilled into the heating block, a hypodermic needle, containing a thermocouple, was used to pierce the sample container, thus positioning the thermocouple just above the paddle yet still immersed in the contents. Temperature was recorded manually on the same strip chart that was recording the viscosity change. Although this crude method was not entirely satisfactory, it did permit making estimates of an actual sample's temperature at critical points, such as at peak viscosity.

With the incorporation of computer technology, the RVA has evolved into a very reproducible, versatile, rapid-pasting device (Walker et al 1988). However, there is still concern as to the correct temperature of the sample, especially by those accustomed to using the amylograph. The objectives of this study were to measure the actual temperature of the liquid contents inside the RVA can during operation and then to compare it with the block temperature of the machine as a function of heating rate and fluid viscosity. These results would then be applied to the development

of an equation that could predict the sample's actual peak temperature at a given viscosity and heating rate.

MATERIALS AND METHODS

A model 3-CR RVA (Newport Scientific Pty. Ltd.) was used. Four temperature profiles were utilized. One was the 3-min rapid-pasting or sprout-damage test. Its temperature profile was set to maintain a constant block temperature of 95°C for 3 min (Anonymous 1993). The other tests were 13-, 23-, and 130-min gelatinization, pasting, and setback profiles, which used different ramping rates (12.2, 6.0, and 1.5°C/min, respectively). These profiles began at 50°C. Block temperature was increased to 95°C, at which it was held for a time and then cooled to 50°C and held again. The actual profiles are outlined in Table I.

Measuring Actual Temperature

The RVA developers had devoted considerable effort in the design of the cans and paddles to ensure a homogenous slurry and optimal heat transfer. The paddle was at least 10 mm below the slurry's surface, and the paddle-to-can clearance was 2.4 and 4.5 mm from the sides and bottom, respectively (field measured). Preliminary trials, with the thermocouples located at several different points in the can, showed that the thermocouple location had no significant effect on temperature data. Therefore, thermocouple location was based on the physical ease in modifying the cans.

New, disposable sample cans were modified by drilling a small hole through the lower edge and inserting a 30-gauge "type K" thermocouple (TT-K-30, Omega Engineering, Inc., Stamford, CT) (Fig. 1). The wire was placed along the bottom of the can, half-way from the center, where it was glued in place using epoxy. To measure the liquid's temperature and not the can's temperature, the exposed end of the thermocouple was bent up. Before each

TABLE I
RVA Gelatinization, Pasting, and Setback Heating Profiles

	Profiles		
	13-min	23-min	130-min
50°C idle temperature			
50°C	1.0 min	1.0 min	10.0 min
95°C	4.7 min	8.5 min	40.0 min
95°C	7.2 min	13.0 min	70.0 min
50°C	11.0 min	21.0 min	100.0 min
50°C	13.0 min	23.0 min	130.0 min
End	13.0 min	23.0 min	130.0 min
Ramping rate	12.2°C/min	6.0°C/min	1.5°C/min

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run, the remaining portion of the thermocouple wire was threaded down through the bottom of the split copper block and wired into a connector plug. Actual sample-temperature data were collected during the RVA test cycle using a model 50 datalogger (Electronic Controls Design, Inc., Milwaukie, OR). Temperatures were recorded at 4-sec intervals to the nearest 0.1°C.

Starch Description and Procedure

Three different starch types were selected, based on preliminary studies of a group of starches that vary in viscosity. They included an unmodified wheat starch (unmodified) (Midsol-50, Midwest Grain Products Inc., Atchison, KS); a waxy corn starch (waxy) (7350 Waxy No. 1, A. E. Staley Mfg., Co., Decatur, IL); and a cross-linked, acetylated tapioca starch (modified) (Tenderfil 473A, A. E. Staley Mfg., Co.).

Twenty-five milliliters of distilled water (25°C) were dispensed into a sample can. Three grams of starch (on a 12% moisture basis) were placed on the surface of the water and, using the paddle, the mixture was jogged for 10 sec to produce a lump-free slurry. The can and paddle were inserted carefully into the machine, so as not to tangle the thermocouple wire. With the paddle firmly seated in the drive motor clutch, the test cycle was activated by depressing the motor tower. After initial 10-sec, high-speed (900 rpm) stirring, the test was continued at a constant stirring speed of 160 rpm. The proprietary RVA software (Booth 1992) controlled the block temperature and collected the viscosity data.

Temperature was recorded in °C; time was recorded in minutes; and apparent viscosity was expressed in rapid visco units (RVUs, in which 1 RVU \cong 10 cp). Viscosity, temperature, and times for peak and end-time were determined for the 3-min, sprout-damage test. For the pasting tests, viscosities, temperatures, and times for peak (maximum), breakdown (minimum), and end-time (setback) were identified from the completed pasting curves.

Experimental Design

Each of the three starches was run in triplicate for each of the four RVA profiles (3-, 13-, 23-, and 130-min tests). One-way analyses of variance (ANOVA) were done to find any differences

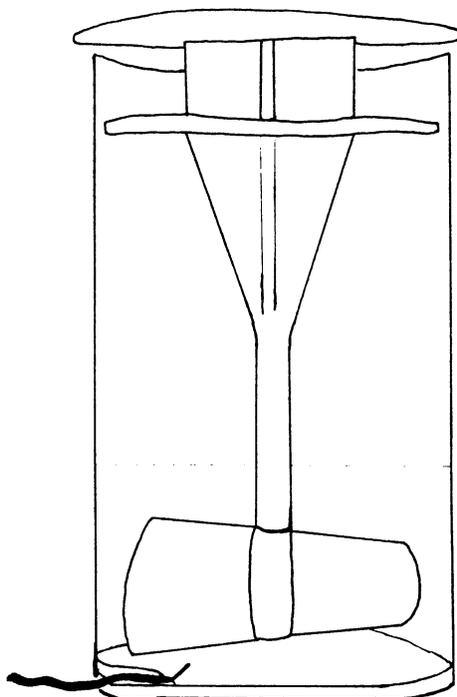


Fig. 1. Cut-away view of the modified liquid-temperature-sensing sample can. The dotted line represents the contents level. Note the thermocouple inserted through the bottom edge of the can.

among block and actual temperatures for the 3-min profile. Regression analysis was performed on both block and actual temperatures of the pasting profiles and on viscosity at several critical points (peak, breakdown, and end-time), according to ramping rate. The response surface methodology (RSM) statistical technique (RSMPLUS, AEW Consulting, Lincoln, NE) was used to generate a second-order multiple-regression equation, including a correlation coefficient and standard error, that would predict temperature lag (block minus actual) as the dependent variable (Walker and Parkhurst 1984).

RESULTS AND DISCUSSION

3-Min, Sprout-Damage Test

This quick test, developed for a specific purpose, has a high degree of correlation with other methods used for sprout-damage analysis. Although this test is not used normally for pure starch samples, it was included because of its rapid heating and short duration. The actual liquid temperature during the test is not normally a concern. The test starts with the block preheated to 95°C and attempts to maintain that temperature during the entire test

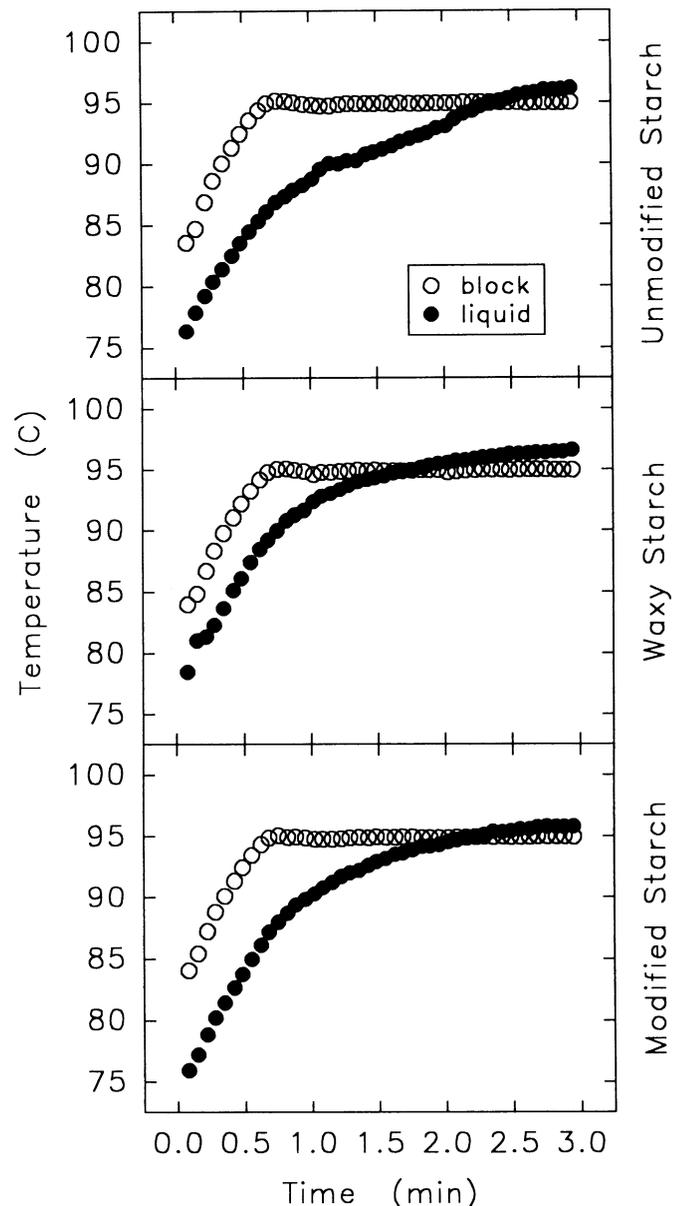


Fig. 2. Comparison of the block versus actual liquid temperature for three starches using the 3-min profile.

time. Figure 2 shows that, in fact, the block temperature did drop substantially as the copper heat sink attempted to rapidly bring the sample up to temperature. As predicted, a significant difference occurred between the reported block temperature and the actual liquid temperature inside the can ($P > 0.01$). The block temperature did recover fairly rapidly, returning to the 95°C set point in approximately 30 sec. However, the liquid temperatures did not quite stabilize and were still increasing slightly even at the end of the 3-min test cycle.

Although the temperature lag (block minus actual) was not quite the same for the different starches, the test seemed to work satisfactorily for that purpose and should not be changed. It should be noted, however, that Figure 2 illustrates the danger in omitting or shortening the preconditioning 50°C hold temperature used in most approved analytical temperature profiles, especially those that have very rapid ramping rates (Deffenbaugh and Walker 1989).

Gelatinization, Pasting, and Setback Tests

General characteristic appearance of the pasting curves. Figure 3 illustrates the differences in characteristic RVA viscosity curves for the three starches tested. Note that the modified tapioca starch had the highest viscosity and was the earliest to peak. The waxy corn starch peaked slightly later, but still well before the block temperature reached 95°C. Also, note that both the modi-

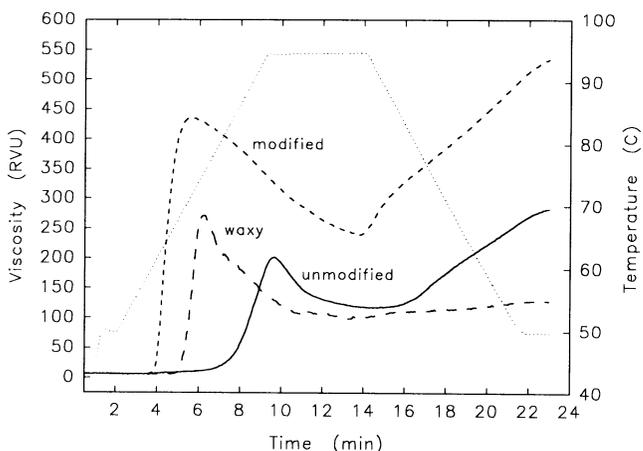


Fig. 3. Viscosity curves showing the different pasting characteristics of the three starches used.

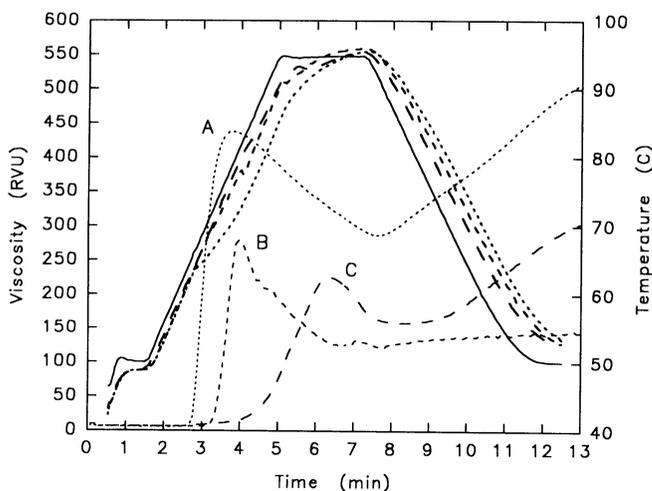


Fig. 4. For the 13-min profile, viscosity curves (lower set; A = modified, B = waxy, and C = unmodified) for three starches showing block (solid line) and liquid temperatures (upper set). Average of three trials.

fied and unmodified starches exhibited substantial setbacks when they were cooled to 50°C, but the waxy starch did not, as is characteristic of starches high in amylopectin.

When the three starches were analyzed by the 13-, 23-, and 130-min profiles, each representing a different temperature ramping rate (12.2, 6.0, and 1.5°C/min, respectively), two interesting things occurred (Figs. 4–6, respectively). First, a temperature lag (sample behind block indicated) occurred, which was rather large for the short test with the higher ramping rate, measurably less for the 23-min test, and almost nonexistent for the long, 130-min test. This was because of the content's inability to keep up with the change in block temperature. Second, although not as obvious from these graphs, the amount of lag was not the same for the three starch samples, but appeared to be larger for the more viscous samples. This was especially apparent in the 13-min test for the modified starch. The temperature lag became especially noticeable at about 2.5 min, just as the starch began its very rapid, viscosity-building phase (Fig. 4).

It is also interesting to note that the most viscous starch (modified starch) was the only one in the longer test that appeared to have rather sharp breaks at the ends of its breakdown section, before the cooling-induced setback began (Fig. 6). During the shorter profiles, all curves for the three starches appeared to have

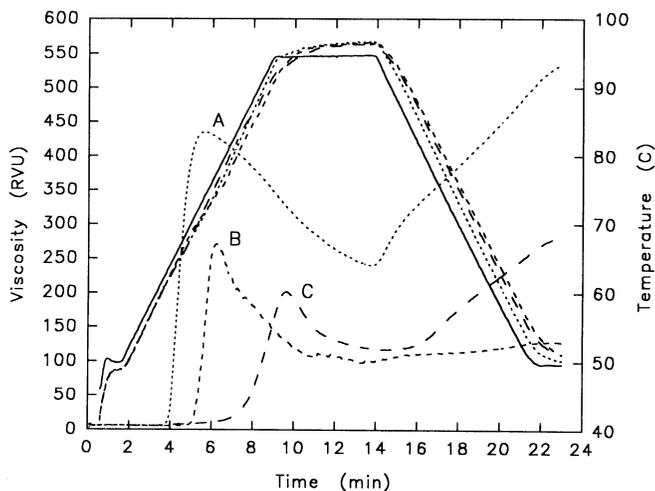


Fig. 5. For the 23-min profile, viscosity curves (lower set; A = modified, B = waxy, and C = unmodified) for three starches showing block (solid line) and liquid temperatures (upper set). Average of three trials.

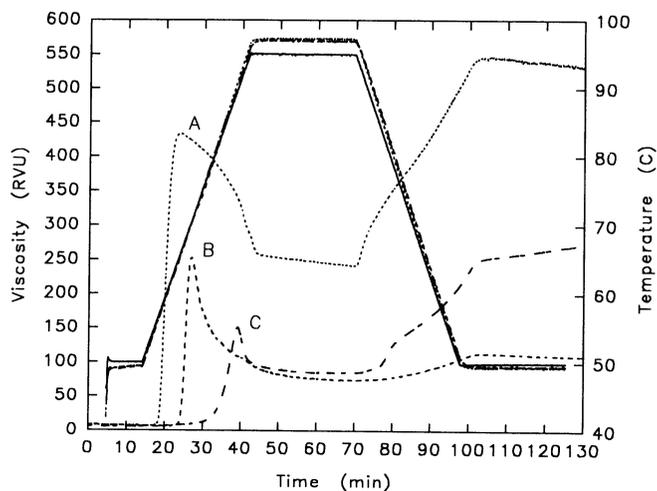


Fig. 6. For the 130-min profile, viscosity curves (lower set; A = modified, B = waxy, and C = unmodified) for three starches showing block (solid line) and liquid temperatures (upper set). Average of three trials.

not yet reached their minimum breakdown before setback occurred, and, especially for the 13-min test, this appeared to result in a higher viscosity at the end of the setback period (Figs. 4 and 5). The viscosity was still rising during this time, perhaps a reflection that the measured sample temperature was still falling, not having yet reached 50°C.

When the measured sample temperature for the three starches for each heating rate was graphed, the temperature lag was indeed larger for the shorter heating profiles and for the more viscous starches (Fig. 7). These curves exhibited the characteristic hysteresis pattern, with the liquid temperature lagging the block temperature during both the heating and cooling phases. That is, the sample temperature was cooler than the indicated block temperature during heating and warmer during the cooling stage. Also, the lag appeared to be larger during the cooling stage because of decreased heat transfer from the thickened starch. At the ramping rate of 1.5°C/min, the hysteresis effect was still present but of little significance.

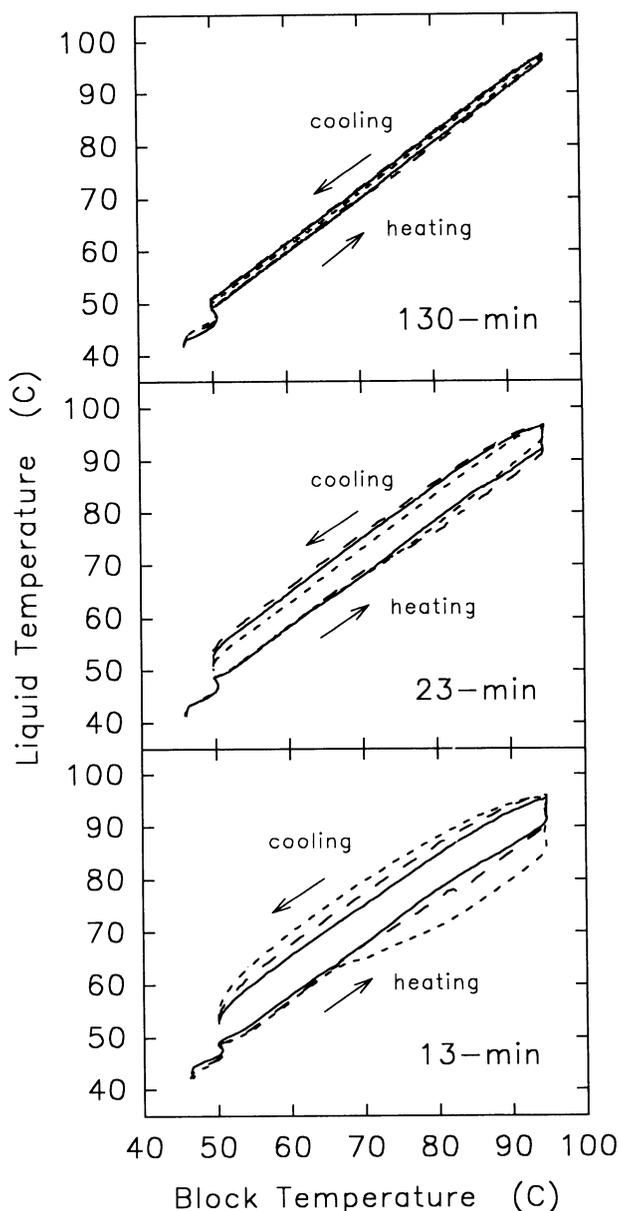


Fig. 7. Liquid versus block temperatures of three starches (modified = short dashes, waxy = long dashes, and unmodified = solid line) for three profiles. Note the characteristic hysteresis pattern.

Block and sample temperatures at certain points as a function of temperature ramping rate. The temperature at which peak viscosity occurs is often requested by users. Remember that this is a function of the prior treatment and the test procedure followed; therefore, it must be interpreted carefully. However, it is useful for comparing different starches or different treatments with the same starch.

Notice that, for all three starches, the indicated block temperature at which peak occurred appeared to rise as the ramping rate increased (Fig. 8). This was misleading, however, because the apparent increase in temperature with ramping rate for the actual sample was not as much as indicated by the block temperatures. For example, the waxy starch showed an apparent increase of only four degrees Celsius in temperature at peak viscosity instead of the eight degrees Celsius indicated by the block temperature (Fig. 8). This four degrees Celsius is still important, indicating differences in behavior under the more rapid heating conditions.

Finally, although Figures 4–6 indicates that the apparent peak viscosity seemed to change somewhat with ramping rate, the pat-

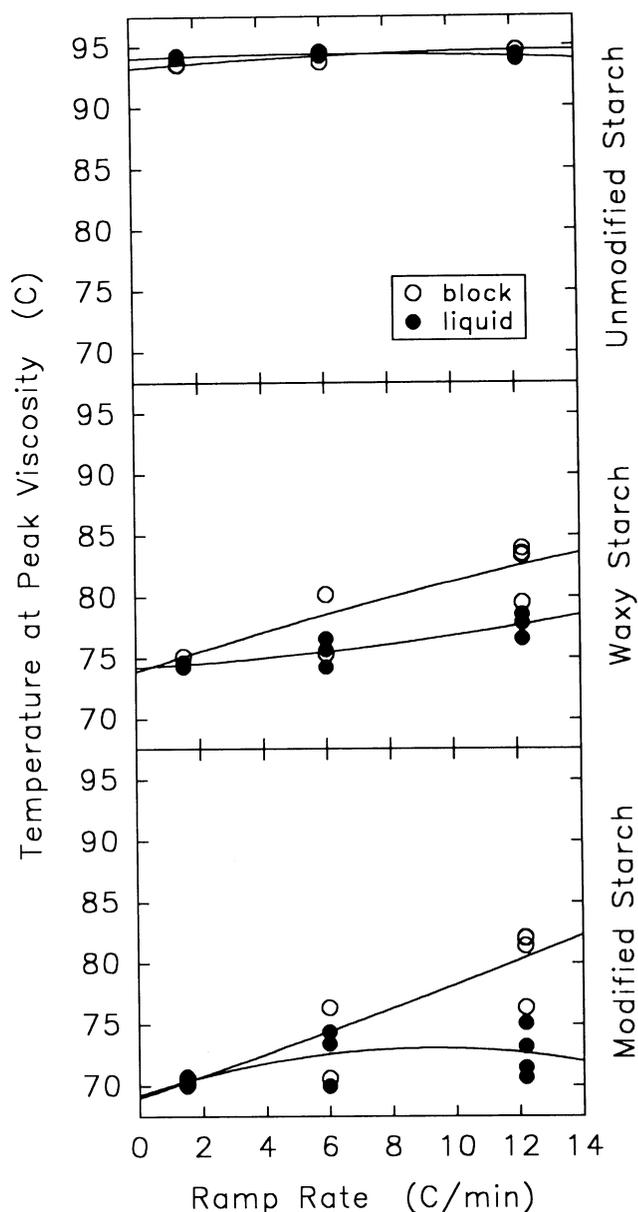


Fig. 8. Liquid and block temperatures at peak viscosity versus ramping rate for three starches.

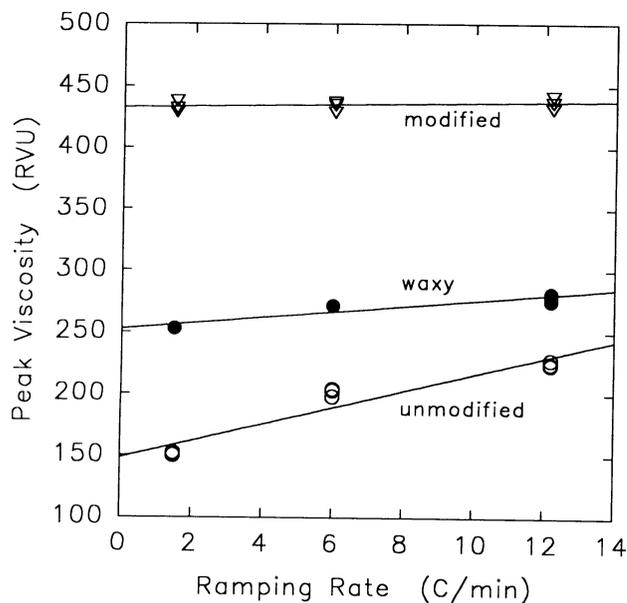


Fig. 9. Apparent peak viscosity versus ramping rate for three starches.

tern may be seen more clearly in Figure 9. The lowest viscosity sample (unmodified starch) tended to greatly "overshoot" the value determined at the slower ramping rate, and the highest viscosity sample (modified starch) showed the least difference with accelerated heating. Why this happened is not clear, but possibly the viscosity of the modified tapioca starch was less affected by the heating rate for a constant shear rate than the viscosity of native wheat starch. It is for such properties that starches are modified in the first place.

The breakdown viscosity did not show quite as definite a pattern; although the trends were the same (Fig. 10). However, for the end-time or setback viscosity, the pattern was more evident. Although the block temperatures apparently had equilibrated, the sample temperatures were still falling (Fig. 11). As a result, because this endpoint was determined by time rather than by temperature, as was peak viscosity, the setback viscosity was being underestimated. Setback viscosity would be expected to continue to rise by three to five degrees as the sample cooled further.

RSM. By applying RSM to the data for peak viscosity and temperature lag for the three different starches and three different ramping rates, taken in triplicate, we were able to generate a second-order multiple-regression equation to predict temperature lag as the dependent variable:

$$TL = -9.28 - 0.93 (RR) + 0.079 (PV) + 0.0043 (RR) (PV) - 0.0017 (RR)^2 - 0.00014 (PV)^2 \quad (1)$$

in which TL = temperature lag, RR = ramping rate, and PV = peak viscosity. The regression equation fits the 27 data points well, with a coefficient of multiple correlation of 0.94 and a standard error of 1.18°C.

The equation was then used to predict a series of points used to prepare Figure 12, which illustrates how one may correct the indicated block temperature to the predicted, actual sample temperature at the time of peak viscosity. For example, if the peak viscosity was 350 RVUs and the ramping rate was 6°C/min, then the probable sample temperature lag behind the block temperature would be about 4°C. This value could then be subtracted from the block temperature, thus correcting it to a value closer to the actual sample temperature at the time when peak viscosity occurred. Note that, below a ramping rate of about 3°C/min, the correction may not be significant. Only one RVA was tested in this manner. Each machine may need its own calibration.

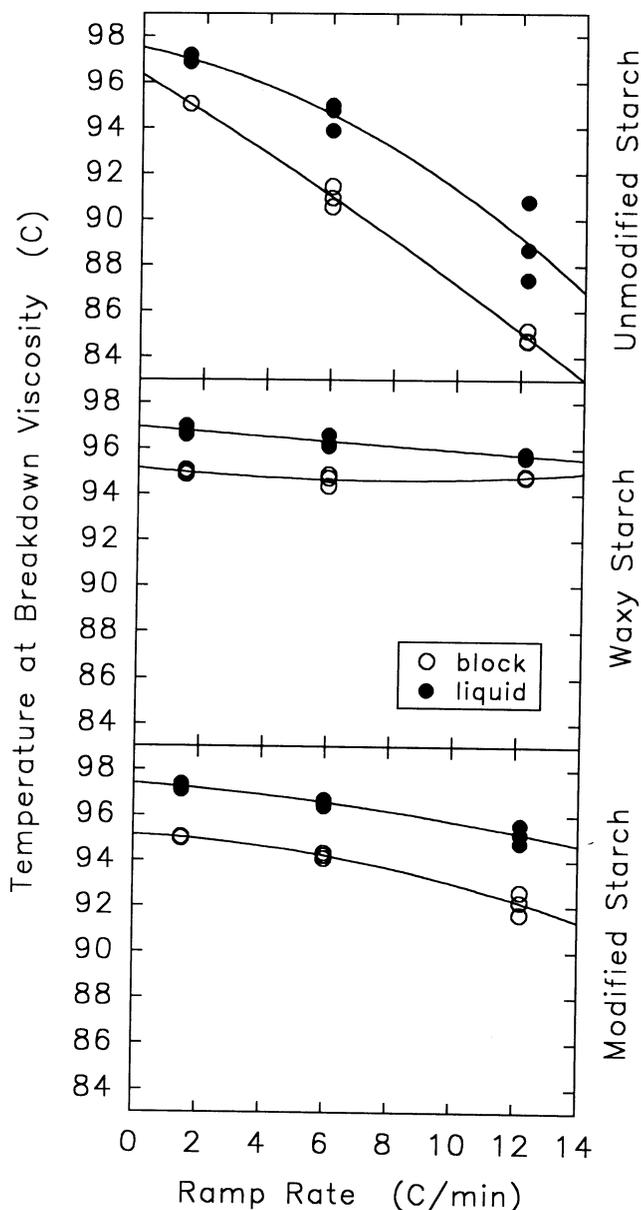


Fig. 10. Liquid and block temperatures at breakdown viscosity versus ramping rate for three starches.

CONCLUSIONS

The actual liquid temperature within the RVA sample can lag behind the indicated block temperature of the machine, giving an inaccurate indication of the true temperatures at which certain curve features such as peak viscosity occur. The lag is a function of the rate at which the temperature is being changed and the apparent viscosity of the starch sample. By use of appropriate calibration curves, adjustments can be made in the indicated temperatures to predict the actual temperatures at which the event would occur.

A comprehensive study needs to be performed at different temperature ramping rates, using a series of liquids that have a range in viscosities with more nearly Newtonian properties and that do not demonstrate phase changes or other dramatic changes in apparent viscosity during use. Construction of a series of tables, a graphical conversion chart, or a multiple-correlation equation would allow the temperature corrections for events (peak, breakdown, etc.) to be made manually or to be incorporated into the computer software that operates the RVA during routine use. This

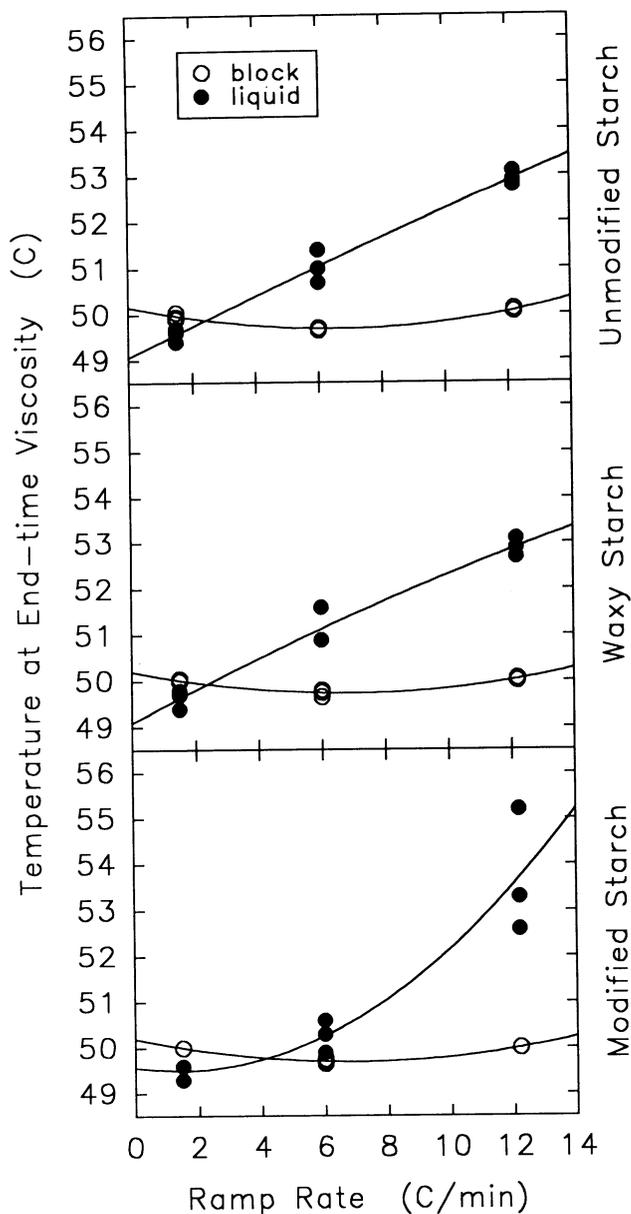


Fig. 11. Liquid and block temperatures at end-time viscosity versus ramping rate for three starches.

would remove one of the concerns expressed by users who wish to know the actual temperature at which certain events occur.

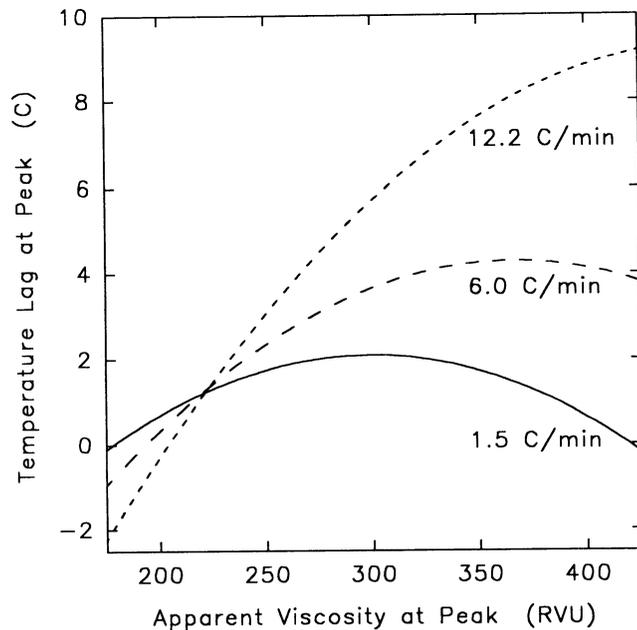


Fig. 12. Illustration of the response surface methodology-generated equation for predicting temperature lag, given the peak viscosity and ramping rate. RVU = rapid visco-unit.

This procedure could be used until suitable devices are incorporated into the RVA can or paddle to actually measure the temperature of the liquid contents during operation.

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