

Rapid Testing Method for Characterizing the Rheological Behavior of Gelatinizing Corn Starch Slurries

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ABSTRACT

Cereal Chem. 66(1):65-68

A mixer viscometer system was developed to evaluate the flow behavior of corn starch slurries during gelatinization. An instrumental system involving a Brookfield RVT-D viscometer and a modified Brookfield small sample adapter equipped with a flag impeller and a thermocouple was constructed. A double fluid bath system allowed the samples to be heated

and cooled over the gelatinization temperature range. Results, collected on a strip chart recorder, gave sample temperature and torque response as a function of time. Instrument sensitivity and data reproducibility were excellent, indicating that the instrument will provide a valuable rheological tool for the starch industry.

The project reported in this paper is the response to a need, expressed by the wet corn milling industry, for new instrumentation to evaluate the rheological behavior of gelatinizing corn starch slurries. Concerns that have been expressed regarding the acceptability of using the traditional methodology, i.e., the Brabender Visco/Amylo/Graph (C.W. Brabender Instruments, Inc., Hackensack, NJ) include long testing times, lack of instrument-to-instrument reproducibility, lack of sensitivity, and nonuniform sample temperature. The Visco/Amylo/Graph will continue to find valuable use in the starch industry; however, the availability of alternative instrumentation and analytical techniques will enhance technological advancement.

There have been earlier attempts to develop alternatives to the Visco/Amylo/Graph. The C.W. Brabender Company developed the Rapid Amylogram in an effort to reduce testing time. A similar idea was presented by Sandstedt and Abbott (1961) in which a small bowl was developed for the Visco/Amylo/Graph. Voisey et al (1977) described the Ottawa Starch Viscometer, which uses a brass bowl with a spherical bottom and a matching flat blade stirring paddle. This device was effective in characterizing starch during gelatinization; however, D. Paton and P. W. Voisey (*personal communication*, 1988) noted that a commercial version of the unit is not available (efforts are underway). More recent efforts have been reported by Walker et al (1988) to evaluate pasting behavior as a quick test for sprout damage in the Australian wheat industry.

The purpose of the present study was to develop a rapid, low-cost rheological instrument and methodology for characterizing the gelatinization behavior of raw corn starch slurries. The new instrumental method was tested for sensitivity and instrument-to-instrument reproducibility.

MATERIALS AND METHODS

Experimental Equipment

The overall experimental system (Fig. 1) was an assemblage of standard, modified, and specially constructed pieces of equipment: two constant-temperature baths, a bath flow selector, a Brookfield RVT-D viscometer (Brookfield Engineering Lab., Stoughton, MA), mixer apparatus, standard tubing and fittings, and a standard dual pen strip chart recorder. The flow selector consists of four solenoid valves (model no. 2A197, Dayton Mfg. Co.,

Chicago, IL) and solenoid coils (model no. 6XS43, Dayton Mfg. Co., Chicago, IL) controlled with a potentiometer, in conjunction with a time-delay relay (Macromatic control circuit, SS series off delay timer, stock no. SS 3562-15), used to set the length of time for heating and cooling cycles. Data collection was accomplished by continuously recording the torque and thermocouple output signals as a function of time on the strip chart recorder. If desirable, these electrical signals could easily be collected using sophisticated data acquisition equipment or a simpler system such as the one developed by Castell-Perez et al (1987).

The mixer apparatus (Fig. 2) was constructed by modifying a Brookfield small sample adapter to accept a copper-constantan thermocouple (Omega hypodermic probe model HYP-2, Type T, Omega Engineering, Stamford, CT). The temperature sensor was put in the sample cup through a small hole drilled in the bottom of the cup. The thermocouple was held and sealed in place using a threaded bolt in combination with a rubber grommet. The small sample adapter mounting bracket was also modified by cutting a long slot over the length of the part that allowed vertical movement of the mixer apparatus. The flag impeller (Fig. 3) was constructed from a standard Brookfield spindle shaft that was shortened to total length of 8.30 cm. The impeller blade (Fig. 3) was ground from a solid piece of stainless steel that had been drilled to allow a press fit on to the shaft.

Equipment Design Considerations

The experimental equipment described above is the result of numerous preliminary investigations. Major considerations centered on the mixer apparatus. Many construction methods and flag sizes were designed, constructed, and pretested. Impeller wobble due to unbalanced flags or distorted shafts and problems of manufacturing duplicate impellers were initial issues. The use of

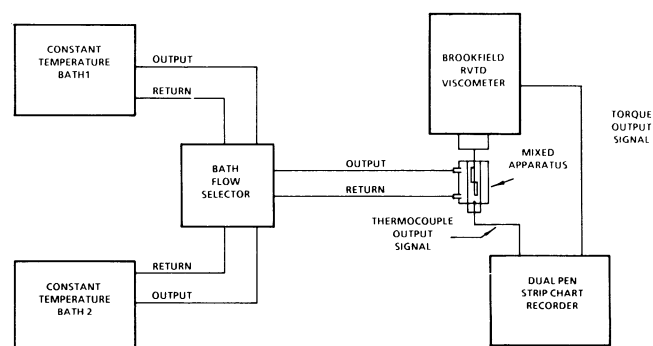


Fig. 1. Overall mixer viscometer system.

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TABLE I
Statistical Summary of Experimental Data

Variable	Mean (n)	Standard Deviation	Standard Deviation With Replication Effect Removed	Coefficient of Variation, %
Torque (g·cm)				
at first peak	0.4 (31)	0.13	0.09	22.5
at second peak	14.7 (48)	1.54	1.24	8.4
between peaks	4.7 (48)	0.23	0.21	4.5
at onset of cooling	10.8 (48)	1.05	0.93	8.6
at end of cooling	14.5 (48)	1.77	1.33	9.2
Time (min)				
at first torque peak	3.44 (31)	0.41	0.42	12.2
at second torque peak	6.05 (48)	0.93	0.86	14.2
between torque peaks	2.76 (31)	0.80	0.69	25.0
Temperature (°C)				
at first torque peak	63.5 (31)	2.36	2.17	3.4
at second torque peak	84.1 (48)	2.78	2.55	3.0
between torque peaks	69.0 (48)	1.92	1.77	2.6
pasting temperature	61.4 (48)	2.33	2.00	3.3

- 1 (45°C) to flow through the jacket of the mixing apparatus.
2. The mixing apparatus was assembled with the flag impeller, cup, and thermocouple in the appropriate locations.
3. The viscometer was started, with the speed of rotation set at a constant value of 100 revolutions per minute.
4. Using a pipette, a 13-ml sample of test material was taken from the 500-ml beaker and placed in the small sample adapter.
5. The sample was allowed to reach an equilibrium temperature of 45°C. This took approximately 1 min and was monitored by observing the temperature curve being continuously produced on the strip chart recorder.
6. The solenoid switching system, which changed fluid flow through the mixing apparatus jacket from bath 1 (45°C) to bath 2 (95°C), was activated. This initiated the heating cycle, completed in 10 min, after which cooling (flow from bath 1) fluid was automatically returned to the jacket.
7. The cooling cycle (about 2 min) was completed, and the viscometer was turned off.
8. The mixer apparatus (cup and flag impeller) was cleaned, and the next test was initiated from step 2.

The importance of loading the sample into the mixer while the impeller was rotating should be noted. This technique was adopted to minimize the problem of starch particles settling to the bottom of the mixer cup during the early stages of testing. If the practice is not observed, particle distribution may be nonuniform in the sample, resulting in a high starch concentration in the bottom of the cup. This, if present, may be identified by carefully investigating (visually) the contents of the cup at the completion of testing.

Experimental data from each test were collected on a dual pen, strip chart recorder (Fig. 1). Torque required to maintain the constant speed of the flag impeller and the temperature of the test sample were recorded simultaneously. Curves (Fig. 4) illustrate the type of information that may be collected using the mixer viscometer instrument. Different types of starch may exhibit significantly different types of curves, and alternative identifying characteristics may be required.

Experimental Design

Data were collected to assess the variability in torque-time-temperature curves introduced by impeller and viscometer changes. Four flag impellers and three RVTD Brookfield viscometers were used for a total of 12 treatment combinations. Tests (four replications of a four impeller times three viscometer factorial) were run on four separate days. The order of the 12 runs per day was determined by complete randomization. Each of the 48 (12 × 4) tests was conducted with a 6% (dry basis) raw corn starch slurry.

RESULTS AND DISCUSSION

Typical curves (Fig. 4) yielded results that were generally similar to those that may be obtained with a Visco/Amylo/Graph (Shuey and Tipples 1980). There are, however, two notable differences: testing time is significantly shorter, and the first torque peak (number 1, Figure 4) is not observed on Visco/Amylo/Graph curves. The first torque response may be due to an initial swelling and rupturing of granules that may correlate to mechanical damage of the granules incurred during drying and handling of the material. Double torque peaks have also been observed by other researchers (Osorio and Steffe 1988, Eliasson 1986).

A series of analyses of variance was run on the 12 variables identified in Figure 4. Results indicate statistically significant (but small) variation in measurements associated with replication and no statistically significant variation associated with impellers or viscometers. Hence, comparisons made using data generated on the same day can be made more accurately than those made from data collected across days. Day-to-day variation was strongly influenced by the data produced on one particular day that may have been adversely influenced by partial plugging of the constant temperature bath fluid line. Considering the standard deviation, with the replication effect removed, Table I gives a measure of the variation expected from experimental and measurement error when comparing impellers and viscometers on a given day.

The first torque peak was observed in 31 of the 48 tests (data not presented) performed. Chi-square tests were run that indicated the existence of the first torque peak was associated with viscometer, not impeller or replication. This result suggests that one viscometer tested may have less torque sensitivity at low torque values than the other two units used in experimentation. Reduced sensitivity may miss or smooth out the first torque peak.

A correlation matrix, involving all 12 variables, was calculated. The investigation showed a high correlation within the separate torque time and temperature data sets, meaning (not surprisingly) that some of the information collected was redundant. Hence, in characterizing starch for a particular industrial application it may not be necessary to quantify all the points identified in Figure 4. In addition, the shape curves for many modified corn starches may not have all the characteristics found for raw corn starch. As the instrument is utilized for other starches, alternative characteristics points may be required.

CONCLUSIONS

A rapid, low-cost rheological instrument and methodology for characterizing the gelatinization behavior of raw corn starch slurries was developed in this study. Instrument sensitivity to changes in fluid flow behavior and reproducibility of experimental

data were excellent. The new instrument will provide a valuable tool to evaluate the rheological behavior of gelatinizing corn starch slurries. Short testing times make the procedure particularly applicable for quality assurance. The methodology is well suited for starch gelatinization evaluation at multiple locations. Curve characteristics are similar to those of the Visco/Amylo/Graph currently used in the industry, thus facilitating interpretation by technical personnel.

Future Research

Important areas of research will involve new methods of utilizing the instrument developed in this study. Characteristics of the first torque peak may provide a means of evaluating mechanical damage of starch granules that occurs during drying and handling. In addition, the value of the instrument may be increased by exploring the use of established mixer viscometry techniques for evaluating time-dependent behavior (Steffe and Ford 1985), power law fluid properties (Castell-Perez et al 1987), and yield stresses (Dzuy and Boger 1985). The instrument may also be valuable in developing rheological models to explain the behavior of gelatinizing starch solutions (Dolan et al 1988). Instrument design improvements (bath switching system and thermocouple mounting unit) are being implemented, and a commercial version of the instrument described in this paper may be obtained from Brookfield Engineering Laboratories Inc., Stoughton, MA.

ACKNOWLEDGMENTS

We thank Dennis Gilliland (Michigan State University) for his statistical consultation and the Corn Refiners Association for providing financial

support and industrial project advisors: P. Method and W. Yackel, A.E. Staley Manufacturing; S. Abell, Pennick and Ford Limited; B. Hovden, Cargill, Inc.; Brookfield Engineering Laboratories is acknowledged for providing instruments used in data collection and continued efforts to develop a commercial version of the system developed in this study.

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[Received July 12, 1988. Revision received October 14, 1988. Accepted October 17, 1988.]