Capillary Rheometry of Corn Endosperm: Glass Transition, Flow Properties, and Melting of Starch[1]

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ABSTRACT

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A capillary rheometer was manufactured to study the properties of corn endosperm. Samples were tested at or near the pressures and temperatures encountered in high-temperature, short-time extrusion. The rheometer was designed to prevent moisture loss during testing. At a set pressure, raising the temperature caused corn endosperm particles to soften and change shape, resulting in a pressure drop as the voids in the sample were reduced. The temperature at which the pressure drop occurred was considered the glass transition temperature. Continued heating caused the pressure to rise and drop a second time as the sample softened and flowed through the capillary. Thermal analysis by differential scanning calorimetry showed that complete melting of starch crystals was not necessary to permit capillary flow. Pressure and temperature conditions sufficient to initiate flow were measured for opaque and vitreous corn flours and expressed as a boundary curve defining the flow region. The position of the curve shifted as a function of sample moisture content. The vitreous corn sample had a rough (unstable) flow that could be eliminated by addition of a small amount (2% w/w) of vegetable oil. When isolated corn starch was studied in the capillary rheometer, results showed that, under certain conditions, starch crystal melting was affected by pressure and time. A model was developed to account for the effects of pressure, temperature, time, and sample moisture on starch crystal melting.

Extrusion technology has been used in the cereal grain industry for several decades. Extrusion is efficient because it uses high pressure, high temperature, and shear force to effectively process or cook cereal grains at relatively low moisture contents. Because a variety of products (breakfast cereals, snack foods, pasta, etc.) can be made using this technology, the applications of extrusion in food production are diverse (Harper 1989).

Rheology is defined as the science of the study of deformation and flow of materials. Under stress, cereal materials (biopolymers) inside an extruder soften, deform, and flow through the die to form a product—a true rheological phenomenon (Kokini et al 1992). However, the fundamental understanding of food extrusion is much less advanced than that of polymeric plastics. Unlike its use in the plastics industry, a rheological approach to food extrusion was developed only on an experimental basis (Baianu 1992).

Most polymer melts are non-Newtonian fluids (Cheremisinoff 1993). Some of them are pseudoplastic and obey the power law, $\tau = k\gamma^n$ with $n < 1$, where τ is shear stress, γ is shear rate, and k is consistency index. Apparent viscosity for a pseudoplastic fluid is $k\gamma^{n-1}$ and decreases with increasing shear rate (shear thinning). Other polymer melts are thixotropic, displaying a reversible decrease in shear stress with time at constant shear rate and temperature (Cheremisinoff 1993).

Two types of rheometers have been used in food extrusion research: extruder-fed rheometers and laboratory capillary rheometers. An extruder-fed rheometer uses a capillary (or slit) connected to the extruder barrel. Padmanabhan and Bhattacharya (1989) used a Brabender laboratory food extruder mounted with capillary dies to investigate the pressure drop at the exit die for corn meal melts. Pressure transducers mounted along the axial direction of the capillary measured pressure. Apparent melt viscosities were calculated based on wall shear stress (a function of pressure gradient, which is the slope of the axial pressure profile) and wall shear rate (a function of flow rate and die geometry). As shear rate increased, melt viscosity decreased. Thus, corn meal melt showed pseudoplastic flow, with a power law exponent of $n = 0.23$. An increase in either moisture content (15–30%) or temperature caused a decrease in melt viscosity.

Fujio et al (1991) developed a piston-type capillary rheometer that differs from capillary rheometers commonly used in the plastics industry, in that a piston pushes the melt from one chamber, through the capillary, and into another chamber, rather than pushing it into the atmosphere. This arrangement has two advantages: it prevents moisture loss during testing and maintains extrudate temperature. Operating temperature and pressure can be varied to mimic conditions encountered in commercial extrusion operations. The rheometer can measure initial-flow conditions (temperature and pressure) and melt viscosity. The former is measured either by increasing pressure at constant temperature or by increasing temperature at constant pressure. The latter is obtained by moving the plunger at a given speed to produce a specific flow rate and measuring the corresponding pressure drop across the capillary die.

A number of studies have dealt with the rheological properties of cereal extrudates. Brent et al (1997a,b) studied both the thermomechanical and viscoelastic properties of cereal melts. Kaletunc and Breslauer (1993) investigated the glass transitions of extrudates and how they affect end-product properties. Fan et al (1996a,b) looked at the effect of sugars on extrusion of maize grits. However, little has been reported on differences in feed materials used for extrusion or how to measure differences in the starting material.

Rheology has a logical relationship to extrusion, and capillary rheometry could be a potential test to characterize raw materials and provide information useful for extrusion. The objective of this study was to use a capillary rheometer based on the design of Fujio et al (1991) to determine the flow and melting properties of corn endosperm and attempt to relate that behavior to high-temperature, short-time (HTST) extrusion.

MATERIALS AND METHODS

Samples

Two corn flours, one primarily from opaque and one primarily from vitreous endosperm, were obtained from Crete Mills (Crete, NE). The opaque corn flour had a protein content (N × 6.25) of 6.00% and a lipid content of 2.35% (both at 14% moisture basis [mb]). The vitreous corn flour had a protein content (N × 6.25) of 7.20% and a lipid content of 0.36% (both at 14% mb). Isolated corn starch (CPC International, Englewood Cliffs, NJ) and vegetable oil (Food Club, TOPCO Associates, Skokie IL) were purchased from a local market.

Moisture Adjustment

Sample moisture levels of 15 and 19% were used throughout the study. To adjust sample moisture, distilled water was added to a

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sample of 10 or 15 g in a 60-mL glass jar. Immediately after adding water, the sample was mixed thoroughly with a glass stirring rod. The jar was sealed, and the sample was tempered at ≈23°C for ≈48 hr. In the middle of the tempering period, the sample was stirred with a glass rod to ensure uniform moisture distribution.

Capillary Rheometer

A capillary rheometer, the design of which was a modification of that reported by Fujio et al (1991), was manufactured in the Physics Department instrument shop at Kansas State University, Manhatten. Fig. 1 is a schematic diagram of the rheometer. The capillary rheometer had two chambers, top and bottom, separated by an interchangeable capillary die. The standard capillary was 1.5 mm in diameter and 25.4 mm long, although diameters of 0 (blank die), 0.5, 1, 2, and 2.5 mm also were available. Each chamber contained a piston. A sample pellet was placed between the end of the top piston and the die. The two pistons were mounted together through side bars, such that they moved together. This arrangement caused the volume of the chambers to remain constant and, more importantly, prevented moisture loss to the atmosphere. Because moisture content affects many properties of cereal materials, this feature was an advantage over capillary rheometers commonly used in the plastics industry, with which material is extruded into the atmosphere and moisture is lost.

Two-band heaters (120 V, 550 W, model NB0400, Omega Engineering, Stamford, CT) were mounted around the outside of the chambers to heat the sample during testing. To facilitate cooling, both the top and bottom chambers were grooved around the outside to accept 0.635-cm copper tubing (not shown in Fig. 1). Copper tubing was wrapped around each chamber and underneath the heating coil. At temperatures >100°C, air was passed through the tubing and used as the cooling medium. At temperatures <100°C, water was used as the cooling medium.

Tranducers in the rheometer measured the change in pressure applied to a sample as it was heated. The pressure in each chamber was measured by a pressure transducer (model PX602, sensitivity ±0.4%, Omega Engineering) whose signal was transmitted to a personal computer and recorded by a data acquisition board (eight-channel strain-gauge board, model OMD-5508BG, version 3.0, Omega Engineering). When temperature was a process variable, the end point of the scan was controlled by a temperature controller (model CN382, Omega Engineering). End-point temperature, but not the rate of heating (°C/min), was controlled. Temperature and pressure were recorded at a rate of one data point per second, and the data were used to plot pressure as a function of temperature.

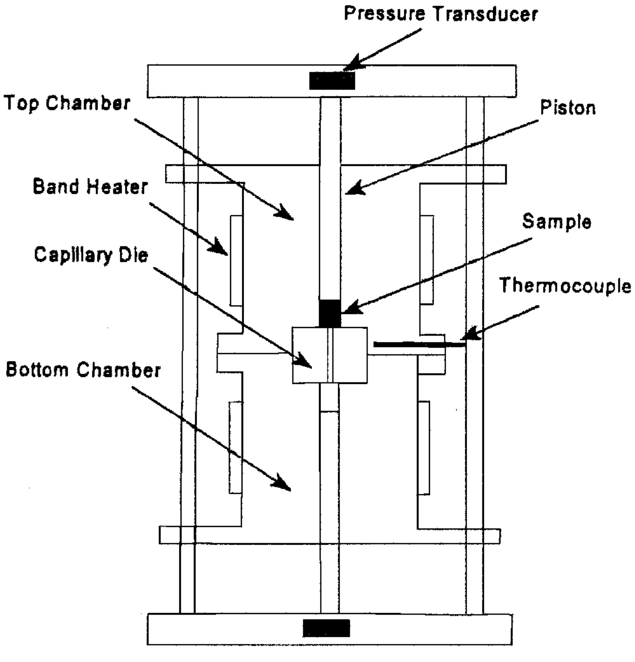

Fig. 1. Schematic diagram of capillary rheometer.

Sample Pellet Preparation

A 1.35-g sample with known moisture was placed in a 10.9-mm i.d. sample preparation die made by the physics department's shop at Kansas State University. A laboratory hydraulic press (model C, Fred S. Carver, Menomonee Falls, WI) was used to form a sample pellet 10 mm high. The pellet was placed in a glass jar sealed to prevent moisture loss. Forming the pellet from material with a known weight and forming it into a known volume was more reproducable than attempting to apply a standard pressure.

Measurement of Initial-Flow Conditions

To measure the conditions of initial flow, a sample pellet was loaded into the rheometer, the pressure was preset at 20–65 MPa with the laboratory hydraulic press, and the sample was heated from 23 to 170°C. These ranges covered the pressures and temperatures that commonly occur in HTST extrusion operations. Both temperature and pressure were continuously recorded by the data acquistion board. As the sample was heated, at some point, it started to flow through the capillary, resulting in a pressure drop. The pressure and temperature at this point were recorded as the conditions necessary for initial flow.

Testing Protocols to Facilitate Sample Flow

Preliminary studies determined that certain samples did not flow when tested in the capillary rheometer, especially at low sample moisture and low preset pressure. Visual examination of the material after testing revealed that a solid bridge had developed at the capillary inlet. Probably because of vapor evaporation from the sample into the chamber, the glassy bridge remained throughout the temperature scan.

A two-step ("flip-over") method was developed to overcome this phenomenon. First, the sample was heated to 90°C at a preset pressure of 30 or 40 MPa to complete particle adaptation. After which, the rheometer was cooled and disassembled, and the sample was inverted, so the bridge side was in contact with the piston, and the glassy side was in contact with the inlet of the capillary. Because water evaporation from the glassy side was slow, the lack of flow was overcome.

Defatting of Corn Flour

Corn flour was defatted in a Soxhlet extractor using petroleum ether. The sample was extracted for 20 hr and air-dried in a hood for 2 hr. The air-dried sample was extracted again with fresh petroleum ether for 5 hr and air-dried for two days.

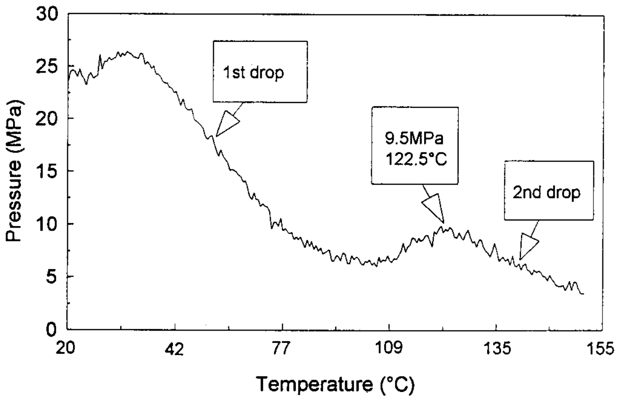

Fig. 2. Test profile from capillary rheometer for opaque corn flour at 15% moisture.

Treatment of Corn Starch with the Capillary Rheometer

Isolated corn starch was used to study the effects of moisture, temperature, time, and pressure on starch crystal melting in the capillary rheometer. A blank die (no capillary) was used to prevent flow. The moisture content of the corn starch was adjusted to 15 or 19% before pelleting. The starch pellet was heated in the capillary rheometer to 50, 60, 70, 80, or 90°C and cooled at once or held at that temperature for 5 min and cooled. The samples were subjected to DSC analysis. The holding pressure (pressure during holding) ranged from 7.5 to 20 MPa and covered the pressures commonly applied in HTST extrusion operations.

Scanning Electron Microscopy

Scanning electron microscopy (SEM) was used to examine the microstructure of sample pellets tested in the capillary rheometer. An electron microscope (ETEC U-1 Auto Scan, Perkin-Elmer Electron Beam Technology, Hayward, CA) was used. Samples were mounted on aluminum studs, vacuum-coated with gold-palladium, and examined at an accelerating voltage of 10 kV.

DSC

A differential scanning calorimeter (model DSC-2, Perkin-Elmer, Norwalk, CT) was used to measure the enthalpy of starch crystal melting in the sample after testing in the capillary rheometer. Zeleznak and Hoseney's (1987) DSC method was followed. The enthalpy of the unheated sample was considered 0% melting. To calculate percent melting, the enthalpy of the sample was divided by the enthalpy of the unheated sample, and the result was multiplied by 100.

Data Analysis and Statistics

SigmaPlot (Jandel Scientific, San Rafael, CA) scientific graphing software was used to perform nonlinear curve fitting of the initial-flow condition data for opaque and vitreous corn flours. SAS STAT procedures (SAS Institute, Cary, NC) were used to conduct analysis of variance to analyze the enthalpy data for starch crystal melting as affected by pressure or time. Multivariable regression was conducted to develop a model to account for the effects of several factors (pressure, temperature, time, and sample moisture) on the extent of starch crystal melting during testing.

RESULTS AND DISCUSSION

Rheometer Calibration

The pressure data acquisition system was calibrated with a universal testing machine (model 4502, Instron, Canton, MA). The calibration curve for either chamber (top or bottom) was linear (R^2 = 0.999) over the range tested (7.5–80 MPa).

To test the ability of the hydraulic press to maintain a set pressure, pressure was set at 25.5 MPa, and changes in pressure were monitored over a 15-min period (the time required to test a sample) at room temperature. A linear pressure drop of 2.5 MPa was found during the holding time.

Selection of Capillary Diameter

Capillaries with different diameters were tested for their suitability to determine conditions for initial flow. When a smaller diameter was used, sample cleaning after testing became very difficult (material stuck very tightly in the capillary). A diameter of 1.5 mm appeared best suited to the task, minimizing the apparent lack of pressure drops experienced with larger diameters. In most cases, sample cleaning was accomplished easily using an electric drill to remove the sample from the capillary. Therefore, a 1.5-mm-i.d. capillary was used throughout the study.

Pressure vs. Temperature Profiles

A typical test protocol was: set pressure at a predetermined value with the Craver press, turn heater on, and constantly record both temperature and pressure. Plotting pressure as a function of temperature produced the profile shown in Fig. 2. In this case, the sample tested was opaque corn flour at 15% moisture. Preset pressure was 25 MPa.

As temperature increased, a pressure drop (the first pressure drop) began at ≈30°C and ended at ≈100°C. We assumed that sample flow through the capillary caused this pressure drop. However, when the capillary rheometer was opened after the temperature scan (to 100°C), we found that no flow had occurred.

The following hypothesis was proposed to explain the pressure drop. As temperature increased, the endosperm particles softened and deformed under pressure. Thus, the voids between particles decreased, resulting in the pressure drop. To test this hypothesis, SEM photographs were taken of corn starch pellets before and after testing in the capillary rheometer. The photographs revealed that at high temperatures the starch granules changed shape and became more closely packed. We hypothesized that deformation occurs when, as the sample is heated above glass transition (T_g), the amorphous polymers comprising the starch granules become mobile, and the granules become plastic and change their shape under pressure.

During the temperature scan, after particle deformation was completed pressure again increased until a second pressure drop occurred (Fig. 2). The pressure buildup was caused by thermal expansion of the sample. The second pressure drop occurred because the material flowed through the capillary. Flow was verified by the appearance of extrudate that had flowed through the capillary.

The melting enthalpies for starch crystals in samples heated to various points along the temperature scan are shown in Table I. As

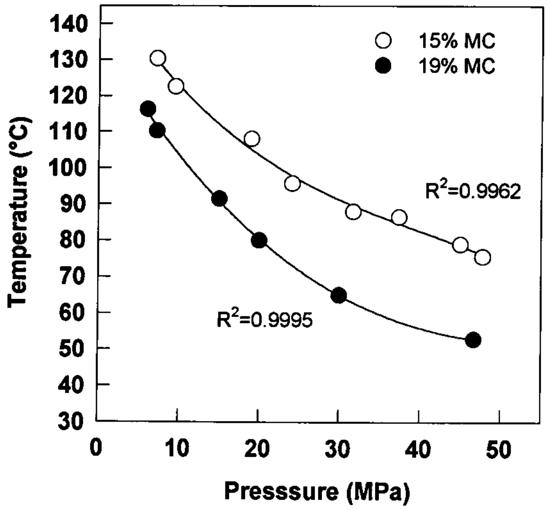

Fig. 3. Initial-flow conditions for opaque corn flour.

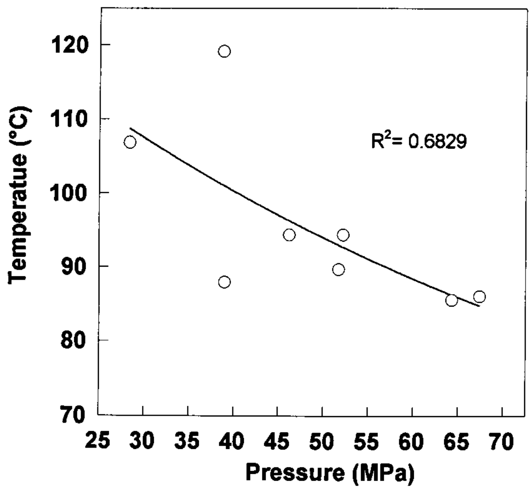

Fig. 4. Initial-flow conditions for vitreous corn flour at 15% moisture.

estimated from the data, only ≈15% of the total starch crystals had melted at the pressure peak related to capillary flow (9.5 MPa, 122.5°C, Fig. 2). Therefore, complete starch crystal melting apparantly was not required for sample flow to occur. These findings led to a reconsideration of the term melt, which is commonly used to describe material in the extrusion process. Strictly speaking, the melt refers to the material that becomes plastic and flowable, not necessarily to the melting of crystals.

A combination of pressure and temperature at the peak was the required condition for initial flow. In extrusion processing, material under the initial-flow condition would be expected to be under an enhanced shear (internal shear within the material), in addition to the shear caused by the mechanical action of the screw or piston. The above discussion may explain why the pressure (7.6–9.7 MPa) and temperature (≈130°C) measured in the cooking zone of a pilot-scale extruder during the production of corn curls (Zhang and Hoseney 1998) were near or above the initial-flow conditions, as shown in Fig. 2. These conditions may be requirements for effective extrusion cooking of expanded snacks.

Effect of Corn Flour Characteristics on Initial-Flow Conditions

The conditions for initial flow (i.e., pressure and temperature at the peak) could be changed by altering the preset pressure. Higher preset pressure caused the peak to occur at a lower temperature, whereas lower preset pressure caused the peak to occur at a higher temperature. In this way, a series of flow conditions was obtained for opaque and vitreous corn flours, which when plotted resulted in the curves shown in Figs. 3 and 4. Any condition above these curves would result in sample flow; therefore, the region above the curve is a flow region, and the curve itself is the boundary of the flow region.

Opaque corn flour produced a smooth curve and, with a nonlinear curve fitting, produced a R^2 value close to 1 (Fig. 3). An inverse relationship was observed between the temperature and pressure required to produce flow: higher temperature required lower pressure. Increasing sample moisture (from 15 to 19%) moved the flow boundary to lower temperatures and pressures.

Initiating flow of vitreous corn flour was difficult. When the flip-over method was used to facilitate flow, the curve shown in Fig. 4 was obtained. In contrast to the behavior of opaque corn flour, the vitreous corn flour curve showed poor regression ($R^2 = 0.683$). Variation was large, especially in the low-pressure region.

TABLE I
Percent Starch Crystals Melted During Capillary Rheometry[a]

Temp. (°C)	Enthalpy (J/g)[b]	Percent Melted[c]
20	9.07 ± 0.25	0
52	9.00 ± 0.17	1.4
82	8.71 ± 0.08	4.6
110	8.20 ± 0.17	10.1
130	7.58 ± 0.13	17.0
150	5.40 ± 0.04	40.8
175	0.00	100

[a] Opaque corn flour at 15% moisture and 25 MPa.
[b] Mean ± standard deviation for two measurements (dry basis).
[c] Assuming 9.07 J/g (at 20°C) represents 0% starch crystal melting.

TABLE II
Effect of Time and Temperature on Melting Enthalpy[a]
of Extruded Corn Starch[b]

	Temperature (°C)[c]		
Time (min)	50	60	70
0	2.61a	2.46a	2.31a
5	2.61a	2.31b	2.05b

[a] Means (dry basis) in a column followed by different letters are significantly different ($P < 0.05$).
[b] 15% moisture and 15–20 MPa.
[c] Heating time: 4.7 min to 50°C, 6.0 min to 60°C, 7.4 min to 70°C.

Extrudate Flow and Appearance

Extrudates of opaque corn flour exiting the capillary had a smooth surface, whereas vitreous corn flour produced extrudate with a rough surface. The saw-toothed pattern (Fig. 5) suggested that part of the flowing sample adhered to the surface of the capillary with sufficient force to impede its flow. With flow stopped, the pressure at the wall-extrudate interface increased, until it overcame the adhesive force, and flow resumed. As the edge of the sample flowed, the pressure dropped, and the sample again adhered to the capillary surface. This gave a slip-stick pattern of flow through the capillary and produced saw-toothed extrudate.

To determine whether aspects of the composition of the vitreous corn flour caused the rough-flow phenomenon, the chemical components of the flour samples were compared. Because opaque corn flour had a significantly higher lipid content, commercial vegetable oil was added to vitreous corn flour to bring its lipid content to the same level as that of opaque corn flour (2.35%), and the sample was tested in the capillary rheometer. The flow characteristics changed, as did the extrudate appearance (Fig. 5). The extrudate showed a smooth surface similar to that of the opaque flour extrudate. When lipid was extracted from the opaque corn flour and the lipid-free sample was tested in the capillary rheometer, the extrudate exhibited rough flow and surface similar to those found for the unsupplemented vitreous corn flour. We concluded that low lipid content was the factor responsible for stick-slip flow and rough extrudate surface.

Our study of stick-slip flow may have practical significance. In industrial extrusion operations, surging also is an unstable flow phenomenon that, in some cases, may be caused by cereal materials sticking to the metal surfaces of the extruder. The addition of low levels of vegetable oil may be useful in overcoming this problem.

Effects of Rheometer Conditions on Starch Crystal Melting

The effects of time and temperature, under various processing conditions, on starch crystal melting are shown in Table II. The holding pressure for all tests was 15–20 MPa. At 50°C, time (5 min) had no effect on starch crystal melting. However, as holding temperature increased to 60°C, longer time resulted in more crystal melting (as indicated by lower enthalpy [J/g]). At 70°C, time had an even greater positive influence on crystal melting. Clearly, if the temperature is high enough (>50°C), increased residence time can result in more starch crystal melting.

Pressure also affected starch crystal melting. At a constant temperature (60°C), increasing the preset pressure from 0 to 40 MPa decreased enthalpy from 10.97 to 10.30 J/g, i.e., more starch crystal melting occurred, probably because at temperatures above the T_g of starch, mobility of starch molecules in amorphous regions of the granule under pressure facilitated crystal melting (Slade and Levine 1988).

The data showed that four factors—time, pressure, temperature, and sample moisture—contributed positively to starch crystal melting

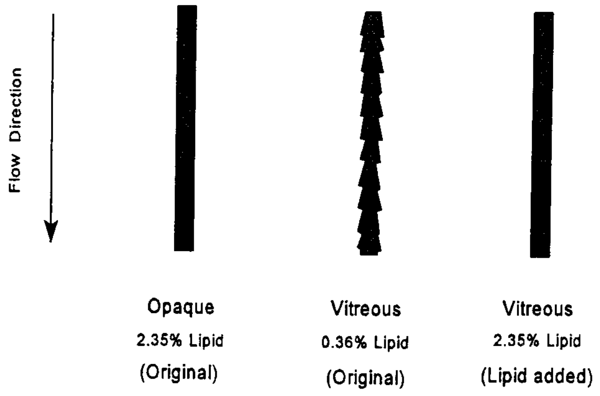

Fig. 5. Extrudate characteristics for vitreous corn flour with lipids added, compared with original opaque and vitreous corn flours at 15% moisture.

in this system. Table III summarizes the extent of starch crystal melting (determined by DSC) under different experimental conditions. Because the capillary rheometer was unable to maintain a constant preset pressure during temperature scanning, two additional factors were included in the data analysis: preset pressure related to holding pressure and time needed to reach holding temperature (Table III). Multivariable regression was performed, and the following equation was obtained:

$$Y = -131.6 + 0.928X_1 + 0.699X_2 + 1.543X_3 + 5.44X_4,$$

where Y is percent starch crystal melting, X_1 is the holding pressure (MPa), X_2 is the holding temperature (°C), X_3 is the holding time (min), and X_4 is percent moisture of corn starch. The apparent valid ranges for variables X_1, X_2, X_3, and X_4 were 7.5–20 MPa, 50–90°C, 0–5 min, and 15–19%, respectively. The R^2 for the regression was 0.938. This model was simple, lacking both higher order and interaction terms, probably because the lower limits of the independent variables in the ranges already (or nearly) allowed each individual variable to function linearly. This model also was reasonable, because the coefficient for each independent variable was a positive number, which was consistent with our observations of the positive effects of these factors on starch crystal melting.

Based on this equation, if each coefficient is multiplied by the range values of its corresponding variable, the relative contribution to starch crystal melting by each variable is 7–19 for X_1, 35–63 for X_2, 0–8 for X_3, and 82–103 for X_4. X_2 (holding temperature) and X_4 (percent moisture) had a much greater effect on Y (percent starch crystal melting) than did X_1 (holding pressure) and X_3 (holding time).

These results suggest that if the mechanical shear is constant, starch melting can be achieved by adjusting time, pressure, temperature, and sample moisture. Pressure alone may not contribute much to starch melting (cooking). However, in industrial extrusion processes, pressure can enhance starch melting (cooking) when combined with increased temperature and enhanced shear. The short time duration of the extrusion process is reflected in our model, in which time had less effect on starch crystal melting than did other factors.

TABLE III
Percent Crystal Melting of Corn Starch Under Different Temperature and Pressure Conditions

Moisture (%)	Preset Pressure (MPa)	Holding Temp. (°C)	Holding Pressure (MPa)[a]	Time (min)[b]	Holding Time (min)[c]	Percent Melting[d]
15	30	50	20	4.6	0	2.7
15	40	60	20	6.4	0	8.2
15	40	70	15	7.4	0	13.8
15	47	80	13	8.8	0	20.2
15	46	90	10	10.3	0	27.4
15	28	50	16	4.6	5	2.6
15	41	60	17	5.6	5	13.8
15	39	70	15	7.5	5	22.5
15	45	80	12	8.5	5	25.8
15	45	90	12	10.1	5	31.1
19	30	50	15.5	4.8	0	22.1
19	30	60	12	6.0	0	22.5
19	37	70	13	7.4	0	28.6
19	31	80	7.5	8.5	0	35.1
19	34	90	9	9.8	0	43.8
19	31	50	19	4.9	5	44.2
19	30	60	12	6.1	5	30.7
19	35	70	16	7.1	5	42.3
19	36	80	16	8.7	5	49.1
19	38	90	17	9.5	5	55.1

[a] At a given holding temperature, nearly constant holding pressure resulted.
[b] Time needed to heat the capillary rheometer to the holding temperature.
[c] Time during which holding temperature and pressure were maintained.
[d] Assuming enthalpy of untreated sample pellet = 0% crystal melting.

Predictions of percent crystal melting of corn starch by this model are applicable to the conditions in the above ranges and to our capillary rheometer. Meeting these requirements would automatically account for the effect of the preset pressure and the time to reach the holding temperature. However, the regression used to develop the model did not have lower limit data points, so deviations in predictions would occur if all independent variables were to simultaneously take on their lower limit values.

CONCLUSIONS

A specialized capillary rheometer was used to analyze the melt (flow) behavior of corn endosperm at low moisture. Under elevated pressure, an increase in temperature caused corn endosperm particles to become plastic, deform in shape, and compact. Complete starch crystal melting was not required to initiate plastic flow. The pressure and temperature required to cause flow could be expressed as a boundary curve defining a flow region. The boundary curve was sample- and moisture-dependent.

Opaque corn flour had smooth flow, whereas vitreous corn flour had rough (unstable) flow. The lower lipid content in vitreous corn flour was responsible for the rough flow.

In addition to being affected by temperature and sample moisture, starch crystal melting was affected by pressure and time under certain conditions. A linear model was established to account for the effects of all four factors (pressure, temperature, time, and sample moisture) on crystal melting of corn starch.

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