Rheological Characteristics of Intermediate Moisture Blends of Pregelatinized and Raw Wheat Starch

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Rheological properties of intermediate moisture (35–45% wet basis) doughs from pregelatinized and raw wheat starch blends of various ratios were characterized using off-line capillary rheometry and online slit-die extrusion. In the case of capillary rheometer, viscosity of blends decreased by up to 50% as pregel starch concentration increased from 5 to 45%, whereas tests could not be conducted beyond 45% pregel starch concentration. For slit-die extrusion, viscosity was at a minimum at 60% pregel concentration, and it decreased by as much as 65% as pregel concentration increased from 0 to 60%. As pregel concentration increased (from 5 to 45% for the rheometer and from 0 to 60% for the extruder), the amount of water available in the system for gelatinization of existing raw starch granules decreased due to the stronger water-binding capacity of pregelatinized starch. This led to decreased additional conversion in the rheometer and extruder, which in turn caused a decrease in the volume fraction of starch and a reduction in viscosity.

KEYWORDS: Wheat starch; raw and pregelatinized starch blends; rheology; degree of gelatinization; water binding; volume fraction

INTRODUCTION

Extrusion is a thermomechanical processing operation widely used for converting raw materials such as cereal grains into a variety of finished products such as snack foods, breakfast cereals, pet foods, and baked goods. In conventional extrusion cooking processes, starch-containing materials are heated under pressure at moisture contents usually <20% [wet basis (wb)] and at temperatures ≤200 °C (*1*). At such moisture contents and temperatures, starch granules undergo gelatinization, which is a physiochemical phenomenon involving swelling and loss of crystallinity of the granules. Most conventional extrusion processes have residence times of <1 min, which is enough to obtain complete gelatinization or conversion of starch under the extreme shear and thermal conditions. However, for commercial reasons, control of gelatinization is important in food systems as it has a direct impact on final product texture. For example, in some products such as shortbread biscuits complete gelatinization might not be desirable for acceptable texture, whereas in others such as wafers or puffs, full gelatinization of starch is required (*2*).

Supercritical fluid extrusion (SCFX) is an innovative technology for the manufacture of cereal-based puffed products that utilizes supercritical CO_2 (SC-CO_2) for product expansion, replacing steam in conventional extrusion (*3, 4*). Typically, SCFX processing has a high-moisture, low-shear, and low-temperature (60–80 °C) environment in which various degrees of starch gelatinization can be used to achieve expanded products of different textural characteristics. Apart from directly affecting the mechanical properties of the solid matrix, the degree of gelatinization also impacts the processing rheology of the starch melt/dough. This in turn affects the bubble growth kinetics and expansion of the product.

A number of studies have reported the rheological behavior of dilute starch solutions (5–15% starch) as gelatinization proceeds due to time–temperature treatments (*5, 6*). These studies observed an increase in viscosity as gelatinization increased and modeled it by first-order reaction kinetics. The increase in viscosity was attributed to swelling of the starch granules and the consequent increase in the volume fraction of starch. Similar results were obtained for starch doughs with limited water content (25–36% wb) (*7, 8*). However, all of these studies were conducted with raw starch. It is expected that the rheology of blends of pregelatinized and raw starches with limited water would be affected in a different manner by the final degree of gelatinization of these blends because of competition between the two components for available water (*9*). The rheological properties of two-component blends have also been studied, particularly for synthetic polymers. Viscosity–composition curves for various polymer blends have been reported to be of four types (*10*): (i) viscosity decreases (or increases) monotonically with blend composition; (ii) viscosity decreases (or increases), exhibiting an S-shape behavior; (iii) viscosity goes through a minimum; and (iv) viscosity goes through a maximum. The diversity of the viscosity–composition curves was attributed to the morphological state of any blend. It was the objective of this study to establish the effect of

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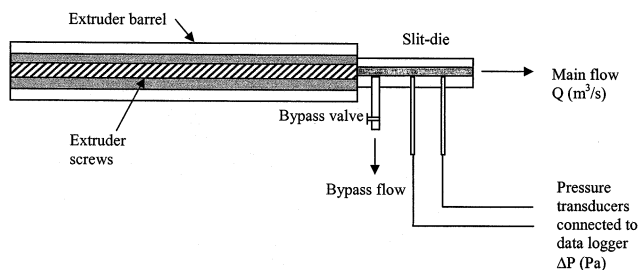
Figure 1. Schematic for on-line measurement of flow properties of wheat starch pregel–raw blends using slit-die extrusion.

composition on the rheology of blends having raw and pregelatinized starch as the two components and to understand the underlying role of degree of gelatinization and starch volume fraction.

MATERIALS AND METHODS

Materials. Raw wheat starch Midsol 50 (20% amylose, 70% amylopectin, 9% moisture, and <1% protein and ash) and pregelatinized wheat starch Pregel 10 (21% amylose, 73% amylopectin, 5% moisture, and <1% protein and ash) of the same hard red winter wheat cultivar were obtained from Midwest Grain Products (Atchison, KS). The same batch was used for all experiments.

Capillary Rheometer. A capillary rheometer (Instron model 3211, Canton, MA) was used for measurement of off-line rheological properties. For each test run, ~30 g of sample was loaded in the rheometer barrel maintained at a temperature of 70 °C and equilibrated for 15 min. Capillaries of 1.22 mm diameter (*D*) and 25.5, 50.9, and 76.8 mm lengths (*L*) (*L/D* ratios of 20.9, 41.7, and 62.9, respectively) were used at crosshead speeds of 0.06, 0.2, 0.6, and 2 cm/min. Raw data of force versus plunger speed were used to calculate shear stress and shear rate at the capillary wall after various corrections described later had been applied. All tests were done in triplicate.

For preparing the test samples, four wheat starch blends containing ratios of pregelatinized to raw starch of 5:95, 15:85, 30:70, and 45:55 (hereafter referred to as 5, 15, 30, and 45% pregel blends, respectively) were premixed. Deionized water was added to 20 g of blend to bring the moisture content to 35% wb. Samples were hand-kneaded for 10 min at room temperature and then divided into small pellets to be loaded into the temperature-controlled rheometer barrel. The actual moisture contents of samples, before and immediately after the rheometer tests, were determined using the oven-drying method (*11*). Above 45% pregel concentration, the sample became too sticky and difficult to load into the rheometer barrel and, thus, rheometer data for a full range of pregel/raw ratios could not be obtained.

Slit-Die Extrusion. A slit-die attached to a corotating twin-screw Wenger TX-57 Magnum extruder (Wenger Manufacturing, Sabetha, KS) with barrel diameter of 52 mm and an *L/D* ratio of 28.5 was used to measure on-line viscosity of starch blends. The slit-die used in this study was 325 mm long and had a slit width (*W*) and height (*H*) of 50.8 and 3.175 mm, respectively. A schematic of the experimental setup is shown in **Figure 1**. Specific mechanical energy (SME) input of the extruder was 560 kJ/kg. The temperature of the melt in the extruder barrel and slit-die was maintained in the range of 65–70 °C. The slit-die had three ports along its length at distances of 38, 170, and 300 mm from the die entrance. Pressure transducers, which were calibrated periodically prior to use, were inserted at the second and third ports of the slit. Pressure data from the transducers were recorded in a data logger every 10 s.

A bypass valve was attached to the first slit-die port to get different shear rates in the slit-die and yet ensure that the extruder barrel conditions and extrudate treatment history were constant. Flow rates of approximately 15, 30, and 45 kg/h were obtained by changing the opening of the bypass valve. Actual melt flow rates were measured periodically by weighing extrudate collected in a container during a fixed time interval (30 s). The raw data of pressure and melt flow rate were used to calculate shear stress and shear rate as discussed later.

For preparing the test samples, six wheat starch blends containing ratios of pregelatinized to raw starch of 0:100, 20:80, 40:60, 60:40, 80:20, and 100:0 (hereafter referred to as 0, 20, 40, 60, 80, and 100% pregel blends, respectively) were premixed using a rotary mixer. The starch blend ratios chosen for slit-die extrusion studies were different from those for capillary rheometer studies in order to cover the whole range from 0 to 100% pregel, which was not possible for the latter. To limit the shear stress below the rating for the slit-die, the moisture content used in the extrusion studies was adjusted to 45% wb, which was higher than that for the capillary rheometer. The actual moisture content of samples exiting from the die was determined using the oven-drying method (*11*).

Degree of Gelatinization Measurements. The degree of gelatinization (or extent of starch conversion) of the starch-based dough/melt before and after the rheometer and extruder was measured using differential scanning calorimetry (DSC) (TA Instruments DSC 2920, New Castle, DE) following the method described by Wulansari et al. (*12*). Heat flow and temperature were calibrated, using mercury, distilled water, and indium standard (T_0 = 156.78 °C and heat of fusion of 28.45 J/g). About 2–4 mg of each dough sample was placed in the aluminum pan, and distilled water added to bring the total weight to 15 mg. This dilution ensured sample moisture content >61%, which is required for complete gelatinization of starch granules. The pan was then hermetically sealed and equilibrated in the DSC cell for 30 min before scanning from 20 to 110 °C at 10 °C/min. Onset temperature (T_o), peak temperature (T_p), and gelatinization enthalpy ΔH (J/g of sample) were recorded for each run. Degree of gelatinization (DG) was calculated using the average gelatinization enthalpy of the native wheat starch sample as zero conversion (*12*)

$$\%\mathrm{DG} = \frac{\Delta H_0 - \Delta H_1}{\Delta H_0} \times 100 \tag{1}$$

where ΔH_0 was the gelatinization enthalpy of the native wheat starch and ΔH_1 was the gelatinization enthalpy of the dough sample. It follows from eq 1 that the DG of raw or native wheat starch was 0.0%. Samples were collected before and after each test run for calculation of the DG.

Specific Mechanical Energy. SME input in the extruder was calculated according to the equation (*13*)

$$\mathrm{SME} = \frac{n_{\mathrm{actual}}}{n_{\mathrm{rated}}} \frac{P}{\dot{m}} \frac{\%\tau}{100} \tag{2}$$

where n was screw speed, P was rated power, $\dot{m}$ was mass flow rate, and $\%\tau$ was load factor. SME input in the rheometer was calculated as

$$\mathrm{SME} = \Delta P A \nu / \dot{m} \tag{3}$$

where ΔP was the pressure drop across the the capillary, A was the area of cross section of the capillary, and ν was the speed of the extrudate through the capillary.

RESULTS AND DISCCUSION

Viscosity Determination Using the Capillary Rheometer. The calculations for apparent viscosity from the capillary rheometer raw data were as reported in the literature (*14*). A plot of pressure drop (ΔP) across the capillary length versus *L/D* ratio was used to calculate the entrance pressure loss (ΔP_{en}) at each plunger speed by extrapolating to *L/D* = 0 (**Figure 2**). The true pressure drop ΔP_t and the shear stress at the wall (τ_w) were calculated using eqs 4 and 5

$$\Delta P_t = \Delta P - \Delta P_{\mathrm{en}} \tag{4}$$

$$\tau_w = \frac{\Delta P_t}{2(L/R)} \tag{5}$$

where *L* and *R* were capillary length and radius, respectively.

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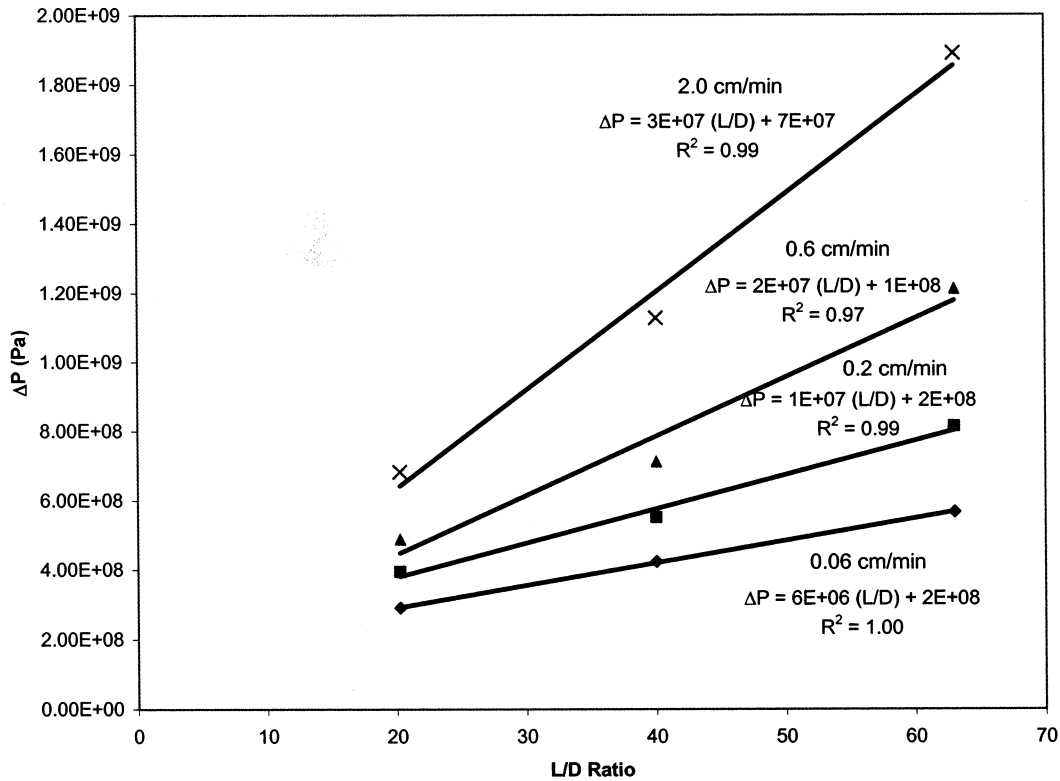
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Figure 2. Plot of ΔP versus L/D at different plunger speeds for 15% pregel wheat starch dough. The intercept of the fitted straight line at the Y-axis gives the entrance pressure drop (ΔP_{en}).
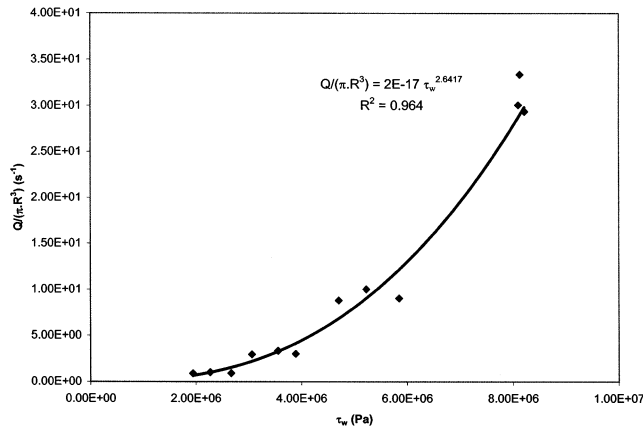

Figure 3. Plot of $Q/(\pi R^3)$ versus τ_w for the capillary rheometer (15% pregel wheat starch dough). The fitted equation was used in the Rabinowitsch–Mooney equation for calculating the shear rate.
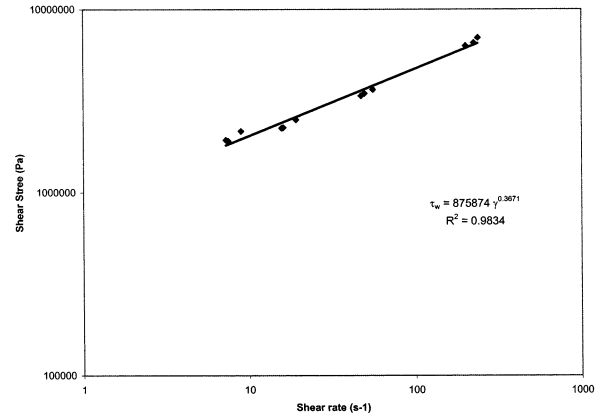

Figure 4. Logarithmic plot of shear stress (τ_w) versus shear rate ($\dot{\gamma}_w$) for the capillary rheometer (15% pregel wheat starch dough). The fitted equation was used to obtain values for K and n.

The wall shear rate ($\dot{\gamma}_w$) was calculated using the Rabinowitsch–Mooney equation

$$\dot{\gamma}_w = \left(\frac{3Q}{\pi R^3}\right) + \tau_w \left[\frac{d(Q/\pi R^3)}{d\tau_w}\right] \qquad (6)$$

where Q was the volumetric flow rate (m³/s).

For evaluating the second term in eq 6, $Q/\pi R^3$ and τ_w were plotted and a relationship between the two was determined by curve fitting. An example of this is shown in **Figure 3**. The relationship between $Q/\pi R^3$ and τ_w obtained in this case was

$$Q/\pi R^3 = (2.0 \times 10^{-17})\tau_w^{2.6417} \qquad (7)$$

Taking a derivative of eq 7 with respect to τ_w and substituting in eq 6, the wall shear rate $\dot{\gamma}_w$ was

$$\dot{\gamma}_w = 3Q/\pi R^3 + (5.2834 \times 10^{-17})\tau_w^{2.6417} \qquad (8)$$

As shown in **Figure 4**, the data for 5, 15, 30, and 45% pregel wheat starch doughs were fitted to eq 9

$$\tau_w = K\dot{\gamma}_w^n \qquad (9)$$

which is the constitutive equation for power law liquids, to determine the values of consistency coefficient (K) and flow behavior index (n). These parameters are shown in **Table 1**.

Fitting the same τ_w versus $\dot{\gamma}_w$ data to a constitutive equation that took into account yield stress did not improve the R^2 value significantly. Thus, absence of yield was assumed. The values

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Table 1. Consistency Coefficient (K) and Flow Behavior Index (n) Values for Wheat Starch Pregel–Raw Blends

pregel concn (%)	consistency coefficient, K (kPa·s^n)	flow behavior index, n	R^2
5	1363.3	0.34	1.00
15	875.8	0.37	0.99
30	865.6	0.34	0.99
45	666.2	0.34	1.00
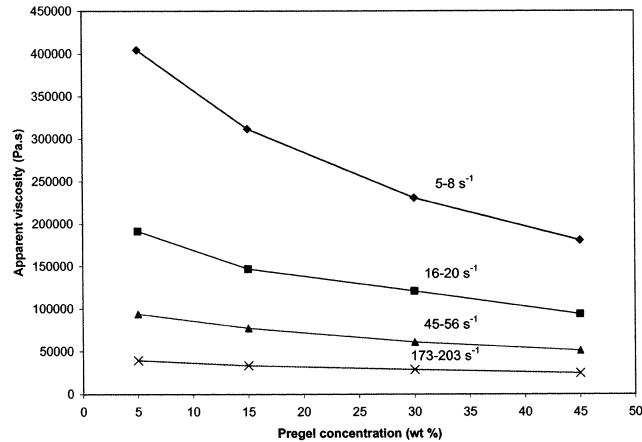
Figure 5. Apparent viscosity of wheat starch raw–pregel blends versus pregel concentration at different shear rates, obtained from the capillary rheometry (35% wb moisture, 70 °C).

of K determined in this study (**Table 1**) were 5−10 higher than those previously determined for wheat starch dough using capillary rheometry (*15*), although the temperature (100 °C) and moisture content (21−28% wb) of the dough in the latter case were different. The flow behavior index (n) for wheat starch doughs reported by the same authors (0.20−0.39) (*15*) compared well with n of 0.34−0.37 determined in this study (**Table 1**). Less than unity values of n, indicating shear thinning behavior, have been reported for various other intermediate-moisture cereal (corn grits, corn flour, oat flour, and wheat flour) doughs with flow behavior indices in the range of 0.20−0.75 (*15*, *16*).

The apparent viscosity of the dough (η) was calculated by dividing the wall shear stress (τ_w) by the wall shear rate ($\dot{\gamma}_w$). The apparent viscosity versus weight percent pregel curves at various shear rates are shown in **Figure 5**. The results suggest that the apparent viscosity of wheat starch dough decreased as the amount of pregelatinized starch increased. This trend was more clearly observed at lower shear rate measurements, where the differences in dough apparent viscosity for different blends were more pronounced. For the shear rate range of 5−8 s^{-1}, viscosity decreased by as much as 50% as pregel concentration increased from 5 to 45%. These results are explained in terms of volume fraction and additional conversion of starch in the following sections.

Viscosity Determination Using the Slit-Die. The shear stress at the walls of the slit-die was calculated from the pressure drop (ΔP) across the two transducers separated by a distance of ΔL, using the following relationship (*14*):

$$\tau_w = \Delta PH/2\Delta L \qquad (10)$$

The shear rate at the walls was calculated as (*14*)

$$\dot{\gamma}_w = \frac{2n+1}{3n}\frac{6\dot{m}}{\rho WH^2} \qquad (11)$$
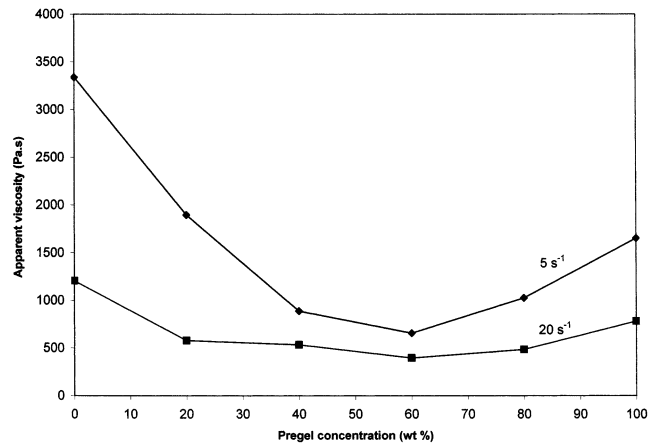
Figure 6. Apparent viscosity of wheat starch raw–pregel blends versus pregel concentration at different shear rates, obtained from slit-die extrusion (45% wb moisture, 65–70 °C).

where W and H are the slit width and height (m), $\dot{m}$ is the mass flow rate (kg/s), and ρ is the melt density (kg/m^3).

The apparent viscosity was calculated by dividing the shear stress (τ_w) by the shear rate ($\dot{\gamma}_w$). **Figure 6** shows the apparent viscosity as measured by slit-die extrusion versus pregel concentration at two different shear rates. Up to 60% pregel concentration, similar to capillary rheometry results, the viscosity of the wheat starch dough decreased as the amount of prege-latinized starch increased. For the shear rate of 5 s^{-1}, viscosity decreased by as much as 65% as the pregel concentration increased from 0 to 60%. Interestingly, as pregel concentration increased beyond 60%, the above trend was reversed and viscosity increased. Thus, a minimum viscosity is obtained at 60% pregel concentration. These results, like the capillary rheometer results, are explained in terms of additional conversion and volume fraction of starch in the following sections.

The viscosity measured by slit-die extrusion (**Figure 6**) was up to 2 orders of magnitude lower than that obtained from the capillary rheometer (**Figure 5**), and this can be attributed to the higher moisture content of the extruder melt (45% wb as compared to 35% wb for rheometer samples) and also greater SME input (560 kJ/kg as compared to 35 kJ/kg for the rheometer) and shear history in the extruder as compared to the capillary rheometer. Similar differences (up to 2 orders of magnitude) in the viscosities of wheat flour (*7*) and cornstarch (*8*) doughs have been reported, as obtained by capillary rheometry and slit-die extrusion.

Effect of Volume Fraction of Starch on Viscosity. The trends in viscosity of the raw and pregelatinized wheat starch blends can be better understood from the point of view of volume fraction of starch and the difference in water-binding capacities of raw and pregelatinized starches. It is a well-reported fact that due to heat treatment (above gelatinization temperatures) and subsequent increase in degree of gelatinization, dilute solutions of starch thicken and increase in consistency (*5*, *6*, *17*). In the case of dilute cornstarch solutions (13% starch), viscosity increased by as much as 400% as heat treatment proceeded and degree of gelatinization increased from 0 to 100% (*5*). The rise in viscosity with increasing gelatinization was attributed to the swelling of starch granules and the concomitant increase in volume fraction of starch. Similar observations have been made for intermediate-moisture starch doughs, where the water available for gelatinization was limited (*7*, *8*). In the case of intermediate-moisture (25−36% moisture content wb) corn-

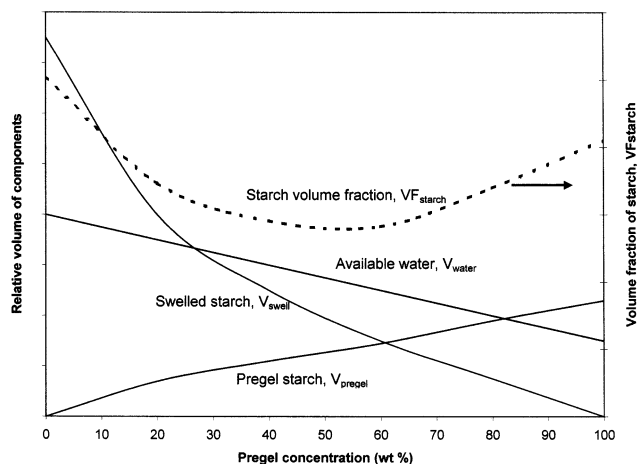**6744** *J. Agric. Food Chem.,* Vol. 50, No. 23, 2002

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Figure 7. Relative volume of swelled starch, pregel starch, and available water, and overall starch volume fraction (dotted line) as a function of pregel concentration (an interpretive chart).

starch doughs, viscosity increased by as much as 60% on cooking (*8*).

In the system studied here, the volume fraction of starch depended on the volumes contributed by three components—pregelatinized starch, swelled starch granules, and available water. The starch volume fraction (VF_{starch}) can be written as

$$VF_{starch} = \frac{V_{swell} + V_{pregel}}{V_{swell} + V_{pregel} + V_{water}} \qquad (13)$$

where V_{swell}, V_{pregel}, and V_{water} are volumes contributed by swelled granules, pregelatinized starch, and available water, respectively.

Figure 7 shows the expected variation in V_{swell}, V_{pregel}, V_{water}, and VF_{starch} with pregel starch concentration. At lower pregel concentrations, the volumes contributed by swelled starch granules (V_{swell}) would contribute predominantly to the overall starch volume fraction as compared to that by pregelatinized starch (V_{pregel}), although V_{swell} would decrease as the proportion of raw starch in the original formulation decreased. Also, the water-binding capacity of pregelatinized wheat starch (0.42 g/g of starch) is at least 10% higher than that of raw wheat starch (0.38 g/g of starch) (*9*). This implied that as pregel concentration in the blend increased, the water available for gelatinization of raw starch decreased, leading to reduced conversion of raw starch granules in the rheometer or extruder, although all blends underwent the same heat treatment. This further decreased V_{swell}, and thus, as pregelatinized starch concentration increased, overall starch volume fraction (VF_{starch}) decreased and, consequently, the viscosity of the starch blend decreased, as was observed (**Figures 5** and **6**).

On the other hand, as pregelatinized starch concentration increased beyond 60%, the decrease in available water volume (V_{water}) tended to have an opposite effect (according to eq 13) and increased the overall starch volume fraction. This effect was probably predominant when the pregel concentration was >60% and led to an increase in viscosity with further increase in pregel concentration. Thus, a minimum viscosity was observed at 60% pregel concentration for slit-die extrusion rheometry.

The reason for the decrease in V_{swell} was further confirmed from the additional conversion of samples in the rheometer or extruder (which is an indirect measure of V_{swell}) calculated from the degree of gelatinization of samples (measured by DSC) before and after the rheometer, as discussed below.
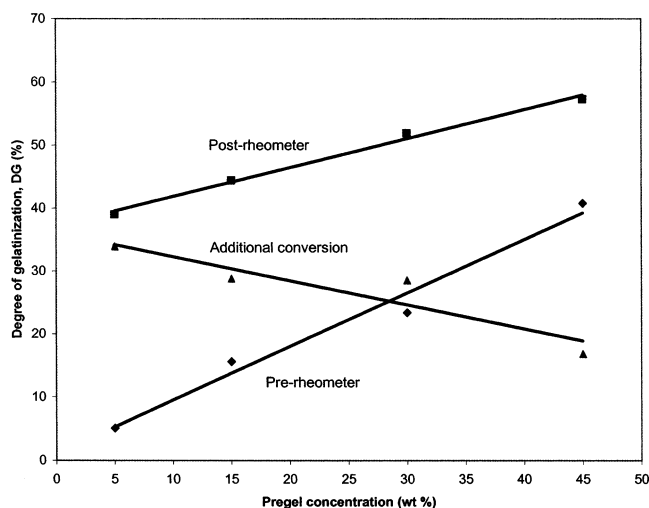

Figure 8. Degree of gelatinization (DG) of wheat starch raw–pregel blends used in the capillary rheometer (35% wb moisture, 70 °C).
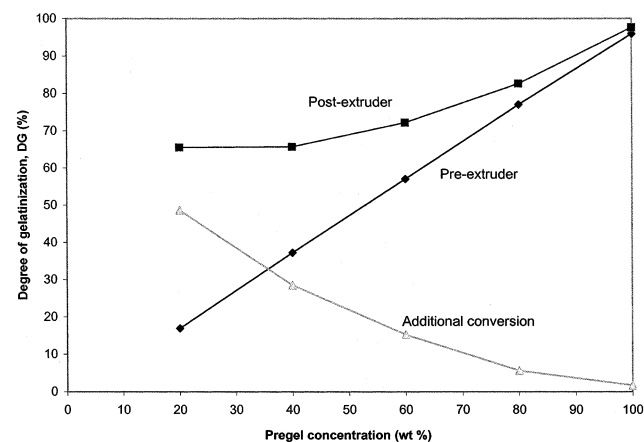

Figure 9. Degree of gelatinization (DG) of wheat starch raw–pregel blends used in slit-die extrusion (45% db moisture, 65–70 °C).

Degree of Gelatinization and Additional Conversion of Starch. The average complete gelatinization enthalpy for native wheat starch sample (ΔH_0) was found to be 11.6 J/g of dry starch. As mentioned earlier, the DG of raw starch was 0.0%. Prior to the capillary rheometer tests, the DG values for the four levels of pregel/raw starch blends were calculated to be 5.1, 15.6, 23.4, and 40.8% (**Figure 8**). These values were close to their corresponding pregel concentrations (5, 15, 30, and 45%). Similarly, prior to slit-die extrusion DG values for the different levels of pregel/raw starch blends were calculated to be 16.9, 37.2, 57.0, 77.0, and 95.9% (**Figure 9**) and were close to their corresponding pregel concentrations (20, 40, 60, 80, and 100%). DSC results for samples collected after the rheometer and slit-die extrusion runs showed that the starch-based dough experienced additional conversion or gelatinization inside the rheometer and the extruder (**Figures 8** and **9**). For both rheometer and extruder samples, although the final DG of wheat starch blends increased with increasing pregel concentration, the additional conversion during the rheometer or extrusion runs decreased because of the reduction in the amount of raw starch and water available for starch conversion. For rheometer samples, additional conversion decreased from 44 to 17% as pregel concentration increased from 5 to 45% (**Figure 8**), whereas additional conversion for extruder samples decreased from 48 to <1% as pregel concentration increased from 20 to 100% (**Figure 9**). Similar results were observed for cooking of

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cornstarch blended with guar gum in the presence of limited water (*18*), where the conversion of starch decreased as the amount of added guar gum increased because of the higher water-binding capacity of the latter. Because additional conversion is a measure of the volume contributed by swelling of starch granules, the DSC results supported the reasoning discussed earlier for viscosity trends observed for blends of raw and pregelatinized wheat starch.

Implications. The apparent viscosity of blends of raw and pregelatinized wheat starch varied depending on the blend composition. In the case of the capillary rheometer, viscosity decreased up to 50% as pregel starch concentration increased from 5 to 45%, whereas tests could not be conducted beyond 45% pregel concentration. For slit-die extrusion, viscosity reached a minumum at 60% pregel concentration and decreased by as much as 65% as pregel concentration increased from 0 to 60%. By using different blends of raw and pregel starch, not only can end products of different degrees of gelatinization be produced, but also the difference in rheology would lead to different expansion characteristics during extrusion and, thus, greater flexibility in final product texture can be achieved. Further research is needed to determine the gas-holding capacity and expansion characteristics of such raw and pregelatinized blends.

ABBREVIATIONS USED

ΔH_0, gelatinization enthalpy of native wheat starch; ΔH_1, gelatinization enthalpy of dough sample; %DG, percent degree of gelatinization; SME, specific mechanical energy input; n_{actual}, actual screw speed; n_{rated}, rated screw speed; P, rated motor power; $\dot{m}$, mass flow rate; %τ, load factor; A, area of cross section of capillary; v, extrudate velocity through capillary; ΔP, pressure drop across capillary; ΔP_t, true pressure drop; ΔP_{en}, entrance pressure drop; τ_w, wall shear stress; L, capillary length; R, capillary radius; $\dot{\gamma}_w$, wall shear rate; Q, volumetric flow rate; K, consistency coefficient; n, flow behavior index; H, slit height; W, slit width; ρ, density of melt; VF_{starch}, volume fraction of starch; V_{swell}, relative volume of swelled starch; V_{pregel}, relative volume of pregel starch; V_{water}, relative volume of water.

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