Rheology of Low- to Intermediate-Moisture Whole Wheat Flour Doughs

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ABSTRACT

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A generalized model for predicting the viscosity of starch-based products was evaluated for whole wheat flour doughs at low to intermediate moisture content. The model incorporates the effects of shear rate, temperature, moisture content, time-temperature history, and strain history. An Instron capillary rheometer and a Baker Perkins MPF-50 D/25 co-rotating twin-screw food extruder were used to collect viscosity data. Die lengths of 6.35×10^{-3} m and 2.54×10^{-2} m, with diameters of 3.18×10^{-3} m and 1.59×10^{-3} m were used in the capillary rheometer. Extruder dies of 6.35×10^{-3} m diameter, and 2.54×10^{-2} m and 3.18 10^{-3} m lengths were used. Whole wheat flour dough viscosities were evaluated at moisture contents of 0.333, 0.337, 0.385, and 0.436 g water per gram of starch, dry basis, in capillary rheometer tests. Barrel temperatures in the capillary rheometer were maintained at 50, 55, 60, 75, 85, 95, and 110°C over cook times of 1, 2, 3, 6, 12, and 24 min. Overall, the accuracy of rheological modeling for whole wheat flour was not nearly as good as had been observed in previous studies on corn starch and potato flour. This lack of accuracy may be attributed to the presence of flour components such as bran, protein, and lipids, which the model does not account for and which most probably altered the starch gelatinization kinetics.

The food industry is constrained from effective use of extruders, in part, because of lack of adequate models for describing the effects of extrusion process variables on extrudate viscosity. Many rheological models incorporating the effects of shear rate, moisture, and temperature on material viscosity during extrusion cooking of cereal products have been proposed (Harper et al 1971, Remsen and Clark 1978, Cervone and Harper 1978, Bhattacharya and Hanna 1986, Morgan et al 1989). In addition to shear rate, moisture, and temperature, Remsen and Clark (1978) also incorporated the effects of time-temperature history. Morgan et al (1989) developed a model for extrusion of protein doughs that includes the effects of strain history in addition to the four variables above.

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Recently, Mackey et al (1989) modified the model of Morgan et al (1989) for use in determining the viscosity of starch-based products. Symbols used in this equation and the remainder of the text are defined in Table I. The resulting model is:

$$\eta = \left[\left(\frac{\sigma_o}{\dot{\gamma}} \right)^{n_1} + \mu_\infty \dot{\gamma}^{\,n_2 - n_1} \right]^{1/n_1}$$
$$\left\{ \exp \left[(\Delta E_v / R)(T^{-1} - T_r^{-1}) + b(\mathrm{MC} - \mathrm{MC}_r) \right] \right\}$$
$$\{1 + A'[1 - \exp(-k_a \psi)]^\alpha\} \{1 - \beta[1 - \exp(-d\,\phi)]\} \quad (1)$$

This model does not account for the effects of nonstarch components on the gelatinization kinetics of starch. It is well known, however, that various components of cereal doughs (protein, lipids, and pentosans) or ingredients added to the product (sugar, salt, etc.) can greatly affect the extent of gelatinization (Eliasson 1983, Lund 1984, Olkku 1978, Ghiasi et al 1982). Because gelatinization significantly affects viscosity, it is reasonable to expect that the presence of dough components such as protein, salts, or lipids would also affect the viscosity of materials during extrusion cooking.

Although the model above has been shown to be quite accurate for calculating the viscosity of potato flour (Mackey et al 1989) and corn starch (Mackey and Ofoli 1989), both of these materials are relatively pure starches. It should be possible, therefore, to evaluate the effect of the presence of gluten, lipids, and other nonstarch components on model performance by using the model to predict the viscosity of a product containing significant levels of one or more of these components. Native whole wheat flour provides an ideal material for this exercise.

The objective of this study was to assess the validity of using the model of Mackey et al (1989) for predicting the viscosity of native whole wheat flour extrudates.

TABLE I
Symbols and Definitions

Nomenclature	
A'	relative increase in viscosity due to gelatinization, dimensionless
b	index of moisture content effects on viscosity, dimensionless
C_s	starch concentration, wet basis decimal
d	index of strain history effects on viscosity, sec
D	capillary diameter, m
ΔE_v	free energy of activation, cal/g mol
ΔE_g	energy of gelatinization, cal/g mol
k_a	reaction transmisson coefficient, $°K^{-1}$ sec^{-1}
L	capillary length, m
MC	moisture content, dry basis, decimal
MC_r	reference moisture content, dry basis, decimal
n_i,	power indices, dimensionless
ΔP	pressure drop, Pa
Q	volumetric flow rate, m^3 sec^{-1}
R_o	capillary radius, m
R	universal gas constant, 1.987 cal/g mol-K
t	time, sec
T	temperature, °K
T_r	reference temperature, °K
Greek symbols	
α	index of molecular weight effects on viscosity, dimensionless
β_o, β_r	index of effects of temperature and moisture on gelatinization, dimensionless
β	index of effect of strain history on viscosity at infinite ϕ, dimensionless
η	apparent viscosity, Pa·sec
$\eta_{\dot\gamma}$	apparent viscosity corrected for shear rate, Pa·sec
$\eta_{\dot\gamma, T}$	apparent viscosity corrected for shear rate and temperature, Pa·sec
$\eta_{\dot\gamma, T,MC}$	apparent viscosity corrected for shear rate, temperature, and moisture content, Pa·sec
$\eta_{\dot\gamma, T,MC,\psi}$	apparent viscosity corrected for shear rate, temperature, moisture content, and time-temperature history, Pa·sec
$\eta_{\dot\gamma, T,MC,\psi,\phi}$	apparent viscosity corrected for shear rate, temperature, moisture content, time-temperature history, and strain history, Pa·sec
$\dot\gamma$	shear rate, sec^{-1}
ϕ	strain history, dimensionless
ψ	time-temperature history, K·sec
σ_o	yield stress, Pa
σ_w	shear stress at the wall, Pa
μ_∞	high shear limiting viscosity, Pa·sec^{n2-n1}

MATERIALS AND METHODS

Whole wheat flour (International Multifoods, Minneapolis, MN) containing 14–14.5% protein (wb) was used as test material for the extrusion runs and capillary rheometer tests. Moisture contents used were 0.333, 0.337, 0.385, and 0.436 g of water per gram of flour (25.0, 25.2, 27.8, and 30.4%, wb, respectively). These moisture contents were used because at higher moisture the stickiness of the material made rapid loading of the capillary rheometer difficult, and at lower moisture damage to the Instron load cell or capillary rheometer plunger would occur.

For capillary rheometer tests, flour and water were mixed for 5 min in an institutional mixer and allowed to equilibrate at room temperature (24°C) for 18 hr. Samples were then placed in plastic freezer bags and stored at 0°C. Prior to capillary rheometer tests, the samples were allowed to thaw for a minimum of 12 hr. Moisture content was determined by drying for 12 hr at 100°C. No change in moisture content was observed during frozen storage.

An Instron capillary rheometer and a model 4202 universal testing machine (Instron Corp., Canton, MA) were used to measure apparent viscosity of the doughs. Dies of 2.54 cm (1 in.) and 0.635 cm (¼ in.) length, and 4.90×10^{-2} cm (1/8 in.) and 1.59×10^{-2} cm (1/16 in.) diameter were used, giving L/D ratios ranging from 2 to 16. Three replicates for each plunger velocity, temperature, moisture content, cook time, and L/D ratio were performed.

Force versus plunger displacement data were collected, and force at the die entrance was calculated by extrapolation of the force versus displacement curves to the die as described by Einhorn and Turetzky (1964). Barrel drag was then subtracted from the corrected force. A correction for entrance effects was made using the technique described by Bagley (1957). Pressure drop through the capillary die was calculated from the force at the die entrance. Shear rate and shear stress were calculated using the Rabinowitsch equation:

$$\dot\gamma_w = \frac{3Q}{\pi R_o^3} + \sigma_w \left[\frac{d(Q/\pi^3 R_o^3)}{d\sigma_w}\right] \quad (2)$$

TABLE II
Determination of Equation 1 Parameters

Correction	Equation Used[a]	Constant Variables[b]	Parameters Determined
Shear rate	$\eta_{\dot\gamma} = \left[\left(\frac{\sigma_o}{\dot\gamma}\right)^{n_1} + \mu_\infty \dot\gamma^{n_2-n_1}\right]^{(1/n_1)}$	$T = 323.15°K$ (50°C) MC = 0.333, db $\psi = 0.0$ $\phi = 0.0$	n_1, n_2 μ_∞, σ_o
Temperature	$\eta_T = \eta_{\dot\gamma} \exp\left(\frac{\Delta E_v}{R}[T^{-1} - T_r^{-1}]\right)$	MC = 0.333, db $\psi = 0.0$ $\phi = 0.0$	ΔE_v
Moisture content	$\eta_{MC} = \eta_T \exp(b[MC - MC_r])$	$\psi = 0.0$ $\phi = 0.0$	b
Temperature-time history	$\eta_\psi = \eta_{MC}[1 + A(1 - e^{-k_a\psi})^\alpha]$	$\phi = 0.0$	A_1, k_a, α
Strain history	$\eta_\phi = \eta_\psi[1 - \beta(1 - e^{-d\phi})]$	None	d, β

[a] Reference variables: $T_r = 323.15°K$ (50°C); $MC_r = 0.333$ dry basis.
[b] The experimental protocol was designed to ensure that each set of data used to correct for a particular variable was collected under conditions such that the variables listed as constant were held constant.

and the standard expression for shear stress at the wall of a capillary:

$$\sigma_w = \frac{\Delta P R_o}{2L} \tag{3}$$

Slip analysis was performed according to the method described by Darby (1976).

Temperatures of 50, 75, 85, 95, and 110°C were used in this study. At 50°C the materials were compressed in the capillary barrel and allowed to equilibrate for 6 min. Cook times of 1, 2, 3, 6, 12, and 24 min at 75, 85, 95, and 110°C were performed at each moisture content after compression.

Corrections for shear rate, temperature, moisture, time-temperature history, and strain history were made in that order, according to the procedure summarized in Table II. Since the parameter estimation procedure used in this study required that any variable on which no corrections have been made yet must be held constant, the experimental protocol was implemented to provide enough data under each appropriate set of constant conditions to enable each correction to be made. Therefore, the shear rate correction was performed by using only data collected at constant temperature (50°C), moisture (0.333% db) and time-temperature history ($\psi = 0$), and fitting viscosity versus shear rate to the model of Ofoli et al (1987), using the Marquardt compromise method in the nonlinear regression program of SAS (SAS Institute, Cary, NC). Strain history was assumed to be negligible in the capillary rheometer.

All of the above data were then pooled with additional data collected at varying temperatures but at constant moisture content (0.333% db) and time-temperature history ($\psi = 0$). Temperature correction was performed with this new set of data by linearly regressing ln η versus $(T^{-1} - T_r^{-1})$ to obtain $\Delta E_v / R$. The moisture correction parameter was determined by pooling this last set of data with data collected at varying moisture contents and constant time-temperature history ($\psi = 0$) and linearly regressing ln η versus $(MC - MC_r)$. Finally, the entire set of available data was combined to provide a correction for time-temperature history as described by Dolan et al (1989).

TABLE III
Screw Configuration Used for Extrusion Tests

Section Length (cm)	Type of Screw	Location
17.8	Feed screw	Feed inlet
8.9	30° forwarding paddles	
7.6	Feed screw	
6.4	45° forwarding paddles	
10.2	Single lead	
8.9	30° forwarding paddles	
7.6	Feed screw	
7.6	Single lead screws	Extruder die

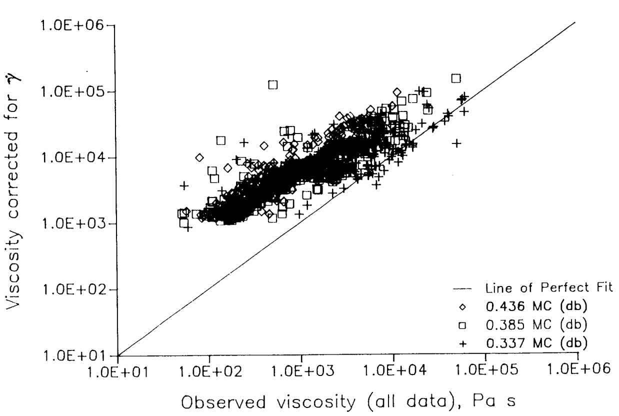

Fig. 1. Viscosity corrected for shear rate versus all data.

Experimental extrusion tests were conducted on a Baker Perkins MPF-50D corotating twin screw extruder (APV Baker, Inc., Grand Rapids, MI) with the screw configuration shown in Table III. Feed rates of 1.01×10^{-2} and 1.64×10^{-2} kg/sec were used at 200 and 400 rpm. Two dies 3.17×10^{-3} m in diameter and 6.40×10^{-3} and 2.54×10^{-2} m in length were used. Pressure drop and extrudate temperature at the die were recorded 2 min after extruder operating conditions had been changed to allow equilibrium conditions to be attained.

RESULTS AND DISCUSSION

Final equilibrium moisture contents of the wheat flour samples were determined to be 0.333, 0.335, 0.385, and 0.436 g of water per gram of wheat flour (25.0, 25.2, 27.8, and 30.4%, wb, respectively). No loss in water was observed during storage.

The extrudates exhibited the appearance of slip, with a "shark skin" texture on the surface. However, analysis for slip was inconclusive at all moisture contents and temperatures. The inability to correct for slip may be due to the "slip-stick" phenomenon, where there is a combination of friction and slip occurring at the capillary wall. Because slip-stick rather than pure slip occurred, Darby's (1976) method for slip correction did not yield meaningful results.

The reference moisture content was set at 0.333 g water per gram of (25.0%, wb) flour. A yield stress of 93.8 kPa was estimated by extrapolating shear stress versus shear rate curves to zero shear rate. After determining the yield stress, the other parameters in the shear rate model were determined via nonlinear regression.

Modeling of Capillary Rheometer Data

A plot of predicted viscosities corrected for shear rate only versus viscosity data at all shear rates, temperatures, moisture

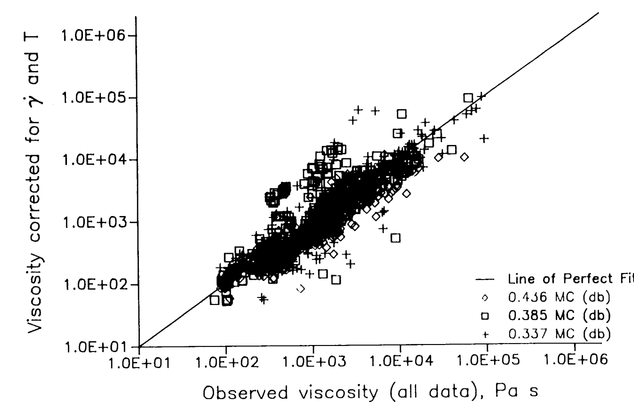

Fig. 2. Viscosity corrected for shear rate and temperature versus all data.

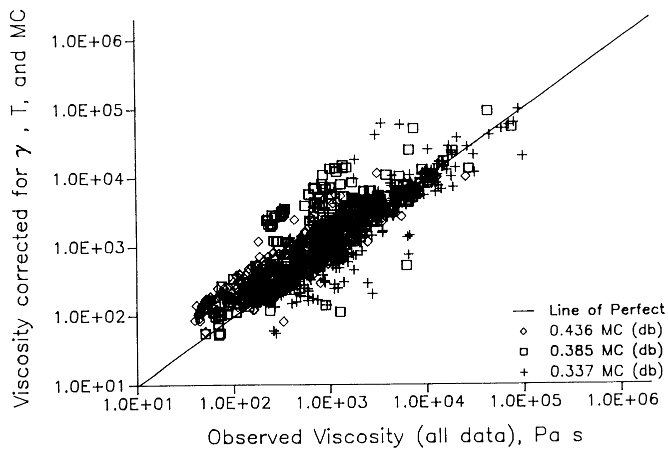

Fig. 3. Viscosity corrected for shear rate, temperature, and moisture content versus all data.

contents, and time-temperature histories is shown in Figure 1. The line of perfect fit is the line $y = x$ and represents where all the data should fall if the analysis had resulted in a perfect simulation. Almost all the data points lie above the line of perfect fit, indicating that shear rate by itself is insufficient to accurately model the viscosity of whole wheat flour. This is further supported by the low correlation coefficient ($R^2 = 0.462$) obtained in the regression analysis.

Consequent correction for temperature and moisture yielded ΔE_v and b values of 5,354.4 cal/g-mol and −7.91, respectively. These values are both within the range reported by other researchers who have studied similar materials. Figure 2 shows predicted viscosities corrected for shear rate and temperature versus viscosities observed under all conditions. The location of the data with respect to the line of perfect fit shows clearly that the correction for temperature improved the accuracy of the prediction ($R^2 = 0.579$), even though there is still a scattering of data.

After correction for moisture content, there was only a slight improvement in the accuracy of the fit ($R^2 = 0.595$). This apparent lack of moisture effects on the viscosity is not unusual, however, since corrections for both shear rate and temperature had been made before the correction for moisture content. It may also be evidence of the fact that many of these variables are interrelated. The plot of predicted viscosities corrected for shear rate, temperature, and moisture content versus all observed viscosities is given in Figure 3.

A comparison of experimental data on viscosities of cooked and uncooked doughs showed little difference at low moisture contents and moderate temperatures. For example, there was no noticeable difference between the viscosities of cooked and uncooked doughs at 75 and 85°C at a moisture content of 0.337 g of water per gram of starch, and at 75°C at 0.355 g of water per gram of starch. This is an indication that starch gelatinization and protein denaturation did not contribute to the viscosity of the dough at these moisture contents. A more plausible explanation, however, is that because of competition for water at these low moisture contents, neither of the two phenomena actually took place.

Lund (1984) observed that the transition temperatures on a differential scanning thermogram increase as the moisture content decreases. This transition temperature is indicative of the onset of gelatinization. Since gluten, lipids, and solutes can also compete with starch for water, they can affect the threshold temperature of gelatinization. The combination of low moisture content and the presence of gluten and other nonstarch components most likely caused the gelatinization temperature to shift upwards. Since neither gelatinization nor denaturation took place, there was no resulting change in the viscosity of the doughs.

At higher moisture contents, the viscosity was found to be a function of the cook temperature (Fig. 4), similar to the results of Mackey and Ofoli (1989). Using observed and predicted viscosities at a 24-min cook time, ln A' versus ln C_s data were regressed for each cook temperature. Relationships between α and β_0 and temperature were then developed. An Arrhenius relationship between inverse temperature and β_0 was observed. This is similar to what was observed for corn starch (Mackey and Ofoli 1989). However, in contrast to corn starch, the correlation between α and the reciprocal temperature did not produce an Arrhenius relationship.

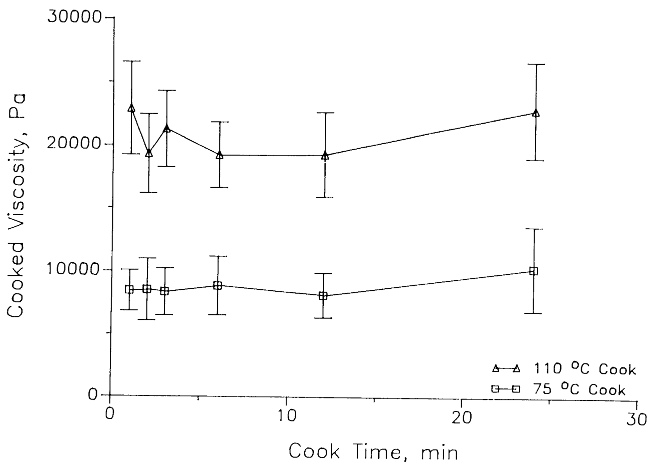

Fig. 4. Cooked whole wheat flour viscosity versus cook time at 100 sec^{-1} and 0.443 g of water per gram of flour moisture content.

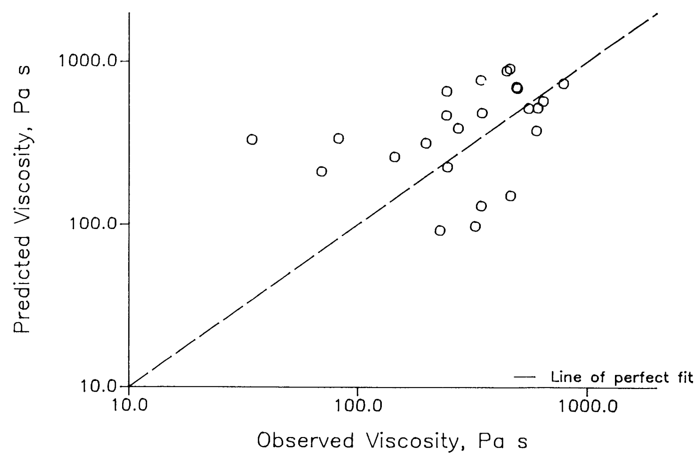

Fig. 6. Predicted versus observed viscosities corrected for shear rate, temperature, moisture, and time-temperature history in extruder dies for whole wheat flour doughs.

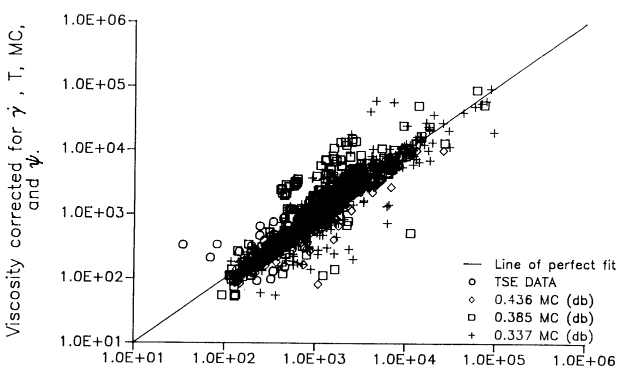

Fig. 5. Viscosity corrected for shear rate, temperature, moisture content, and time-temperature history for all data.

TABLE IV
List of Model Parameters Used in Equation 1

Parameter	Value or Relationship
σ_0	93.8 kPa
μ_∞	1.05×10^5 Pa·sec
n_1	1.0
n_2	0.40
ΔE_v	5,354.4 cal/g-mol
b	−7.91
A'	$\exp\left(-59.99 + \frac{2.08 \times 10^4}{T}\right)(C_s)^{\left[-143.6 + \frac{5.07 \times 10^4}{T}\right]}$
ΔE_g	2.51×10^4cal/g-mol
k_a	2.5 sec^{-1} °K at 0.346 g/g, db
	1.1 sec^{-1} °K at 0.337 g/g, db
	1.1 sec^{-1} °K at 0.385 g/g, db
$[1 - \beta(1 - e^{-d\phi})]$	1.0

The equations for α and β_o are:

$$\alpha = -143.5 + \frac{5.07 \times 10^4}{T} \quad (4)$$

and

$$\beta_o = \exp\left(-60.0 + \frac{2.08 \times 10^4}{T}\right) \quad (5)$$

Viscosity was also observed to be a function of cook time, enabling time-temperature history parameters to be calculated according to the procedure described by Morgan et al (1989) and Dolan et al (1989). The parameter ΔE_g was found to be unaffected by moisture content. This is in agreement with the finding of Dolan et al (1989) that there is little difference between ΔE_g values for high-moisture corn starch solutions of varying starch concentrations. Burros et al (1987) estimated activation energies for corn starch over moisture contents similar to those in this study and observed no significant differences between activation energies.

The activation energy (ΔE_g) in this study was found to be 25,100 cal/g-mol. The constant k_a was found to be slightly affected by moisture content. However, this may be due to normal sample variations and not because of any moisture content dependency. Values for k_a were 2.5 for 0.436 g of water per gram of solids (30.4%, wb) and 1.1 for 0.385 and 0.337 g of water per gram of solids (27.8 and 25.2%, wb, respectively).

Figure 5 is a plot of predicted viscosity corrected for shear rate, temperature, moisture content, and time-temperature history versus all observed viscosities. The fit of the data has improved slightly in comparison to the fit after correcting for shear rate, temperature, and moisture content only. This improvement is, however, not as dramatic as that observed for native corn starch (Mackey and Ofoli 1989). However, as indicated by the narrow heavily shaded band, most of the data falls along the line of perfect fit. The important point, however, is that the fit is relatively poor ($R^2 = 0.644$). In comparison, the R^2 for potato starch was 0.95 (Mackey et al 1989) and 0.98 for corn starch (Mackey and Ofoli 1989); the difference is that these last two materials are relatively pure starches.

Several factors may be responsible for the relative inaccuracy of modeling the rheological behavior of whole wheat flour: 1) the presence of protein, bran, and other flour components is not accounted for in the model, and most probably had an effect on the time-temperature parameters; 2) "slip-stick" phenomena or friction effects may have had a greater effect on the viscosity of whole wheat flour than on native corn starch and potato starch; 3) the low moisture content used in this study probably amplified either or both of the effects already mentioned; 4) elastic effects due to the presence of gluten. The model does not account for energy losses due to the elasticity of the dough, which may be why the overall fit is worse for whole wheat flour than for corn starch.

Modeling of Extrusion Data

Predicted viscosities, after correction for shear rate, temperature, moisture content, and time-temperature history are also plotted in Figure 5. Extrudate properties varied from unpuffed and unswelled material to a highly puffed breadlike material that did not hold the puffed appearance upon cooling. Unlike the case for corn starch (Mackey and Ofoli 1989), strain history did not appear to affect wheat flour during extrusion. No clear relationship could be developed between observed viscosity and work input.

With no information available to correct for strain history, the strain history term was set to unity. Regression of predicted viscosities corrected for shear rate, temperature, moisture content, and time-temperature history versus observed viscosity indicates that the fit of the extrusion data is slightly worse than was obtained for the data from the capillary rheometer (Fig. 6).

Inability to correct for strain history for whole wheat flour compared with corn starch may be attributed to the differences in the general makeup of the two materials. Corn starch is nearly 100% starch, whereas the whole wheat flour consists of starch, bran, wheat protein and some lipids. The inclusion of water and the mixing in the extruder most likely caused development of the gluten, which may have contributed to increase in viscosity that was not counteracted by a lowering of the viscosity by mechanical breakdown of starch.

A list of the parameters in Equation 1 and/or their relationships are given in Table IV. It is important to note that the order in which corrections are made may affect the final results, even though the same degree of accuracy should be obtained. Also, for whole wheat flour, some of the parameters may depend on temperature, moisture content, or both.

CONCLUSIONS

Capillary rheometer tests were performed on whole wheat flour at moisture contents of 0.333–0.436 g of water per gram of solids (25.0–30.4%, wb), cook times of 1–24 min, temperatures of 50, 55, 60, 75, 85, and 110°C, and shear rates of 1–1,000 sec^{-1}. Overall fit of the predicted versus observed capillary rheometer viscosities for whole wheat flour was less than were observed for potato flour and native corn starch. This lack of accuracy may be attributed to the effects of flour components (bran, protein, fat) that are not accounted for by the general model.

No differences between the viscosities of cooked and uncooked doughs at 75 and 85°C at a moisture content of 0.337 g water per gram of starch, and at 75°C at 0.355 g of water per gram of starch were observed. At high moisture contents, viscosity did change with cook time. The most plausible explanation for this is that competition for water by flour components such as gluten probably shifted the threshold temperature upwards, making complete gelatinization impossible. In the absence of gelatinization, there was no measurable change in viscosity at low moisture content for different time-temperature histories.

Different strain histories did not appear to affect the twin-screw extrusion data, and therefore the strain history term was set to unity for analysis of all extrusion data. Again, the presence of nonstarch components may have had an effect on the ability to detect changes due to strain history.

This study indicates that mechanisms to account for the effects of whole wheat flour constituents (bran, protein, fat, etc.) and additives (salt, sugar, etc.) are necessary. It is also necessary to incorporate the effect of gluten on material viscosity. In particular, the effects of flour constituents on the correction for time-temperature history and strain history should be studied. Further research into quantification of strain history within the extruder is also desirable. The question of whether an accurate measurement of shear rate in each extruder zone is necessary to quantify strain history or if measurement of work input alone is adequate must also be addressed.

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