Influence of Mixing Conditions and Rest Time on Capillary Flow Behavior of Wheat Flour Dough

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ABSTRACT

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The capillary flow properties of wheat flour dough were evaluated as a function of mixing time (3–60 min) and rest time (0 or 120 min) after mixing. The description of experimental flow properties was conducted using the classical Bagley approach. Long mixing times were more efficient in decreasing the resistance to elongational flow than in decreasing the resistance to shear flow. The effects of rest time on the rheological properties were estimated by calculating the percent changes in values during resting and appeared to depend on the mechanical history of dough (mixing conditions). The observed changes in rheological properties were related to possible changes in dough structure. Exponential models were used to describe the changes in farinograph consistency (R^2 = 0.981) and extensional viscosity (R^2 = 0.744 to 0.949) as a function of the mechanical energy input during mixing.

The fundamental understanding of the flow behavior of wheat flour dough is a huge field of research due to the complex structure of dough, anomalies in flow behavior, and wide range of testing modes. The dependence of dough properties on factors such as time, work input, and the rate of work input coupled with the extreme sensitivity of dough properties to water level and biological activities complicate the task of obtaining reliable and meaningful data.

The effect of mixing conditions on the rheological properties of wheat flour dough has been widely published (Kilborn and Tipples 1972; Mani Lindborg et al 1997; Letang et al 1999; Wooding et al 1999; Cuq and Kokini, unpublished). An increase in mixing time or in mechanical energy input (MEI) during mixing (above the optimum consistency) induces great changes in mechanical properties of wheat flour dough. The softening effect of long mixing time was indicated by the decrease in dough consistency and tenacity (measured with the farinograph) and also in viscoelastic properties of dough when measured immediately after mixing (Dempster and Hlynka 1958; Dreese et al 1988; D'Appolonia and Kurneth 1990; Mani et al 1992). The mechanical properties of wheat flour dough also greatly depend on time after the mixing. Changes in extensional properties and a softening effect on dough properties are reported as a function of rest time (Bogdan and Moor 1971; Rasper and Preston 1991; Edwards et al 1999; Cuq and Kokini, unpublished). Determination of rheological properties of wheat flour dough is usually made after resting for a time sufficient to reach stabilization (time-independent properties). Resting times of 30–120 min are generally considered sufficient to reach stabilized conditions (Edwards et al 1999).

The importance of fundamental investigations of flow behavior for wheat flour dough is recognized (Faridi and Faubion 1990; Menjivar 1990). Measurement in simple shear is the most commonly used rheological testing mode, but characterization of extensional flows has attracted a great deal of interest because extensional and elongational flows are involved in dough processing. Characterizing the shear and extensional viscosity properties of wheat flour dough is considered a critical step to better understanding flow behavior (Bagley et al 1998).

Capillary rheometry provides reliable information on shear and extensional flow properties for dough systems (Steffe 1992). Two types of capillary rheometer have been used for cereal-based dough. Extruder-fed rheometers (a capillary connected to the end of an extruder) can be adapted for the characterization of time-dependent materials because the thermomechanical history in the extruder is constant during measurement. Extruder-fed rheometers have been used to characterize cereal-based dough, particularly in regard to extrusion-cooking applications (Senouci and Smith 1988; Padmanabhan and Bhattacharya 1989; Wang et al 1990; Vergnes et al 1993; Singh and Smith 1999). Piston-driven capillary rheometers can be adapted to study the behavior of time-independent fluids. These rheometers are filled before measuring, and residence time of fluid in the rheometer depends on measurement time. These rheometers have been used to characterize cereal-based dough (Remsen and Clark 1978; Kieffer et al 1982; Dus and Kokini 1990; Mackey and Ofoli 1990; Madeka and Kokini 1992; Zhang et al 1998; Singh and Smith 1999). Because shear flows are relatively weak along the barrel (before the capillary), piston-driven capillary rheometers are more particularly adapted to the characterization of shear sensitive fluids such as wheat flour dough. Piston-driven capillary rheometers have been used to describe the rheological properties of wheat flour dough and more particularly to characterize the effect of process conditions on flow behavior. The effect of capillary geometry, flour type, dough water content, dough protein content, mixing time, and rest time were described previously (Sharma and Hanna 1992; Sharma et al 1993ab; Huang 1998; Breuillet et al, unpublished).

The objective of the present study was to investigate the effect of mixing time (3–60 min) and rest time after mixing (0 or 120 min) on the flow properties of wheat flour dough evaluated by using a piston-driven capillary rheometer. The description of experimental flow properties was conducted using the classical Bagley approach considering linear functions between pressure drop and L/D ratio. Shear and extensional flow properties were considered with regard to specific changes in dough structure during mixing and resting. Apparent nonlinear behavior of Bagley plots was related to possible changes in dough structure during flow along the capillary rheometer.

Theory

A pressure drop P over a fixed capillary length L causes flow in a capillary of diameter D. Determining rheological properties using a capillary rheometer makes numerous assumptions: the flow is laminar and steady; the material is incompressible; the material properties are independent of pressure or time; the temperature is constant; there is no slip at the capillary wall; the exit-effects are negligible. Under these conditions, the derivation of the Poiseuille law yields to the definition of the apparent wall shear stress ($\sigma_{w,c}$) (Equation 1) (Philippoff and Gaskins 1958). Assuming a Newtonian flow, the apparent wall shear rate ($\dot{\gamma}_{w,c}$) is proportional to the volumetric flow rate Q (Equation 2).

$$\sigma_{w,a} = \frac{PD}{4L} \tag{1}$$

$$\dot{\gamma}_{w,a} = \frac{4Q}{\pi R^3} \tag{2}$$

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The use of Poiseuille equations to describe the capillary flow properties requires a correction that takes into consideration the transition from the barrel to the capillary. The Bagley correction method accounts for excess pressure loss at the entrance of the capillary (Bagley 1957; Han and Charles 1971; Steffe 1992). The entrance pressure drop is experimentally evaluated using a set of capillaries with the same diameter but different L/D ratios. The total pressure drops P are collected for each capillary and plotted against L/D. The extrapolation of linear regression lines to $P = 0$ results in the Bagley end correction factor (Bagley e). The Bagley e gives the imaginary extension to the capillary length ($L+eR$) related to the entry losses in excess of the fully developed flow losses (Boger and Binnington 1977). The extrapolation of Bagley plots at L/D = 0 results in the entrance pressure drop P_0 (pressure drop through a capillary of 0 length). The Bagley e calculates the pressure drop along the capillary from the measured total pressure drops ($P_{cap} = P - P_0$). The corrected wall shear stress ($\sigma_{w,c}$) is then calculated by correcting the capillary length with the Bagley e (Equation 3).

$$\sigma_{w,c} = \frac{P}{4\left(\frac{L}{D} + e\right)} \tag{3}$$

To account for non-Newtonian behavior, correction of the wall shear rate is made using the Rabinowitsch-Mooney equation (Equation 4) (Steffe 1992). The pseudo-plasticity index (n) of the power law equation (Equation 5) is defined by the slope of the straight line of the plot Log($\sigma_{w,c}$) versus Log($4Q/\pi R^3$). The corrected shear viscosity ($\eta_{w,c}$) is obtained by the ratio of the corrected shear stress over the corrected shear rate (Equation 6).

$$\dot{\gamma}_{w,c} = \left(\frac{3n+1}{4n}\right)\dot{\gamma}_{w,a} = \left(\frac{3n+1}{4n}\right)\frac{4Q}{\pi R^3} \tag{4}$$

$$\sigma_{w,c} = K\,\dot{\gamma}_{w,a}{}^{n} \tag{5}$$

$$\eta_{w,c} = \frac{\sigma_{w,c}}{\dot{\gamma}_{w,c}} \tag{6}$$

As the flow converges from the barrel into the capillary, uniaxial extensional flow occurs at the entrance of the capillary (Cogswell 1972). According to the Cogswell method, extensional stress (σ_E) extensional rate ($\dot{\varepsilon}_E$) and extensional viscosity (η_E) can be calculated from the measurements of the capillary entrance pressure drop (P_0) (Equations 7–9) (Binding 1988; Bagley et al 1998).

$$\sigma_E = \frac{3\,(n+1)}{8}\,P_0 \tag{7}$$

$$\dot{\varepsilon}_E = \frac{4\,\sigma_{w,c}}{3\,(n+1)\,P_0}\,\dot{\gamma}_{w,a} \tag{8}$$

$$\eta_E = \frac{\sigma_E}{\dot{\varepsilon}_E} = \frac{9\,(n+1)^2}{32}\,\frac{P_0{}^2}{\sigma_{w,c}\,\dot{\gamma}_{w,a}} \tag{9}$$

MATERIALS AND METHODS

Wheat Flour

Commercial unbleached bread wheat flour (Heckers, 5-lb bags) was purchased in a local market. Wheat flour was stored at room temperature until used. The initial water content of flour was determined by weight measurements after drying at 135°C for 2 hr. Protein and ash contents were 11.65 and 0.515% (w/w), respectively, according to producer specifications. Water contents (12.3–12.8% wb) were determined in triplicate for each bag.

Dough Mixing

Wheat flour and water were mixed with a farinograph (Brabender, Duisburg, Germany) using a large bowl at constant temperature (30°C). The mixing temperature was controlled by using a cryostat (Lauda Brinkmann RC6, Germany) and water circulating in the jacket surrounding the bowl. The damping adjustment was set to ≈0.6 sec for the pointer to go from 1,000 to 100 BU on the scale head. Dough mixing was done at 63 rpm (speed 2) and constant weight of dough (480 g). Flour was first mixed alone for 3 min (to reach the mixing temperature) before the addition of the calculated amount of water (at <20 sec) to attain the final dough moisture content (45% wb). The dough consistency (middle of the curve bandwidth) was recorded from the farinograph plots. The torque applied on the motor housing during mixing was calculated using Equation 10:

$$\text{Torque (Nm)} = b \times \text{deflection (BU)} \tag{10}$$

where b is a constant related to the change in the torque per unit of deflection ($b = 9.8\ 10^{-3}$ Nm/BU) (Bloksma 1990). Coefficients of variation (12–18 measurements) for dough consistency (Nm) were ≈4.5% irrespective of mixing time. The mixing action of the farinograph was characterized (Equation 11):

$$\text{MEI} = \int_{t=0}^{t=\text{mixing time}} \frac{\left(\frac{\text{Torque}(t+\Delta t)+\text{Torque}(t)}{2}\right)\Omega\,\Delta t}{1000\,M} \tag{11}$$

where MEI is mechanical energy input (kJ/kg), M is weight of dough (0.48 kg), t is mixing time (sec), Δt is increment time (sec), and Ω is rate of rotation (6.6 rad/sec). Two paddles turn in opposite directions and run at a differential speed of 3:2. The slow paddle operates at the same speed as the shaft from the dynamometer (63 rpm) and the fast paddle operates at ≈95 rpm (Shuey 1990).

Immediately after mixing, dough samples (50–80 g) were placed in two glass beakers and covered with aluminum foil. One dough sample was immediately tested with the capillary rheometer and the other was stored in the fermentation cabinet of the extensigraph at constant temperature (30°C) for 120 min before testing.

Capillary Extrusion Tests

A capillary rheometer (model 3211 series 290, Instron, Canton, MA) with a synchronous motor and drive system that drive a plunger at a constant rate into a barrel (9.52 mm diameter, ≈25 cm length) was used for the tests. Temperature was kept constant at 30°C during the experiments. A set a six capillaries with the same inside diameter (1.32 mm) and same entrance angle (45°) but different lengths (6.6, 13.2, 19.8, 26.4, 39.6 and 52.8 mm) was used. The L/D values of the selected capillaries were 5, 10, 15, 20, 30 and 40. The capillaries were screwed at the bottom of the barrel. The barrel was fitted with small pieces of dough that were introduced and pushed down into the barrel using the plunger. Completely filling the barrel required ≈3–5 min. The plunger was then run down on the top of the sample and stopped at the first appearance of dough at the bottom of the capillary. The dough was allowed to rest for 3–5 min. The experiments were performed at increasing shear rates (13, 40, 133, 400 and 1,333/sec) corresponding to increasing plunger speeds (0.254, 0.762, 2.54, 7.62, and 25.4 mm/min). During each run, the five plunger speeds were tested. The pressure needed to extrude the dough through the capillary was calculated from the measured values of forces acting on the plunger (recorded when constant reading values were reached). The volumetric flow rate (Q) was directly calculated as the product of the set plunger speed and cross-sectional area of the barrel. Measurements were conducted in duplicate (for dough mixed 3, 15, or 60 min) or triplicate (for dough mixed 6 or 30 min) for each condition (five plunger speeds and six L/D ratios) using fresh mixed dough for each experiment. The coefficients of variation for total pressure drop were determined in triplicate (≈11%). These relatively high values could be associated with great sensitivity of dough structure during the filling of the barrel.

RESULTS

Dough Mixing

The mixing behavior of wheat flour and water is characterized by farinograph curves (Fig. 1). Consistency curves during dough mixing show a classical shape with a short peak time (at 5.5 Nm after 2 min of mixing) and long stability after the maximum (≈30 min). Similar farinograph curves are observed for strong bakery flours (D'Appolonia and Kurneth 1990; Boyacioglu and D'Appolonia 1994).

The MEI during mixing is nearly proportional to the mixing time after the dough development (2 min or 6.3 kJ/kg) and until stabilization (30 min or 125 kJ/kg) (Fig. 1). MEI reached 235 kJ/kg at 60 min of mixing (Table I). Similar calculations of MEI during mixing were found in literature (Kilborn and Tipples 1972; Frazier et al 1975; Oliver and Allen 1992; Cuq and Kokini, unpublished).

An increase in mixing time from 3 to 60 min (or in MEI from 10 to 235 kJ/kg) produced great changes in mechanical properties of wheat flour dough. Long mixing had a classical softening effect, illustrated by the decrease in dough consistency (from 5.5 Nm at 3 min to 4.2 Nm at 60 min) (Fig. 1). The softening effect of long mixing has been described for farinograph consistency (Dempster and Hlynka 1958; D'Appolonia and Kurneth 1990) and also for viscoelastic properties when measured immediately after the mixing (Dreese et al 1988; Mani et al 1992; Cuq and Kokini, unpublished).

Capillary Flow Behavior

The capillary flow properties of wheat flour dough were measured after a period of rest to stabilize rheological properties. According to Cuq and Kokini (unpublished), resting the dough for 120 min at 30°C was sufficient to obtain dough with time-independent properties. To evaluate the rheological properties of fresh dough, we also conducted measurements with the capillary rheometer immediately after the mixing (without resting). Under these conditions, the properties of wheat flour dough are not time-independent and the use of a piston-driven capillary rheometer is thus, theoretically, not recommended (Steffe 1992). However, we proposed that the changes in dough properties as a function of time are negligible during measurements. Only 15 min from the end of mixing to the end of measurement are required to conduct experiments with a capillary rheometer. The apparent capillary flow properties measured under these conditions represented properties of fresh dough.

For each mixing condition, the total pressure drops were measured during capillary extrusion of wheat flour dough as a function of apparent shear rate (13–1,333/sec) and capillary dimension (L/D = 5–40). The Bagley plots (total pressure drop vs. L/D ratio) were constructed for wheat flour dough as a function of mixing time (6–60 min), immediately after mixing, and after 120 min of rest at 30°C. Bagley plots for wheat flour dough mixed for 6 min and rested for 120 min are presented in Fig. 2. According to the Bagley method (1957), we have considered the Bagley plots for wheat flour dough as linear functions of L/D ratio (at a given shear rate). The experimental points are well described by linear regressions immediately after mixing or after 120 min of rest (R^2 = 0.856 to 0.994). Similar linear descriptions of Bagley plots for wheat flour dough have already been presented (Kieffer et al 1982; Sharma et al 1993a; Wang 1995; Bagley et al 1998; Huang 1998; Breuillet et al, unpublished).

The capillary behavior of wheat flour dough was first characterized by the values of entrance pressure drop (P_0), that were determined by extrapolating the linear functions to L/D = 0 (Table I). The measured values of P_0 for wheat flour dough is relatively high (90–1,820 kPa) and indicate a large contribution of elongational flow at the capillary entrance. The organized gluten network in the dough structure could explain these high values of P_0. Branched

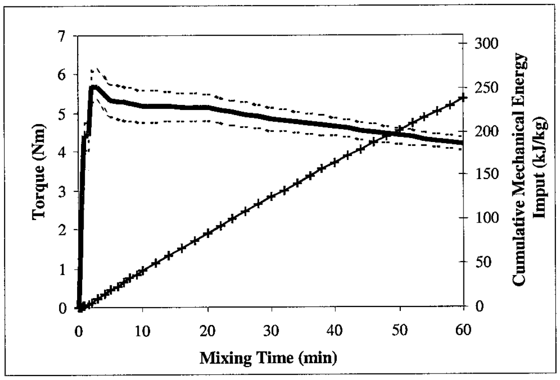

Fig. 1. Farinograph plots for torque (————) and cumulative mechanical energy input (+++++) vs. mixing time at 30°C for wheat flour dough. Dashed lines are limits of farinograph curve bandwidth.

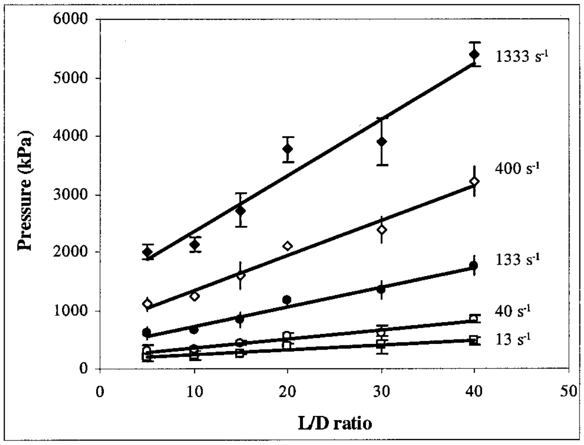

Fig. 2. Bagley plots for wheat flour dough mixed for 6 min and measured after resting 120 min at shear rates from 13 to 1,333/sec.

TABLE I
Entrance Pressure Drops (P_0) for Wheat Flour Dough Measured Immediately After Mixing or After Resting 120 min as a Function of Mixing Time (3–60 min)

Rest Time (min)	Mixing (min)	Mechanical Energy Input kJ/kg	Mechanical Energy Input Standard Deviation	P_0 (kPa) at Different Apparent Shear Rates (sec^{-1}) 13	40	133	400	1,333
0	3	11.0	(0.5)	170	300	560	980	1,820
	6	24.3	(0.9)	280	300	560	1,000	1,480
	15	62.3	(2.3)	160	230	410	680	1,120
	30	124	(4.0)	110	150	390	820	1,330
	60	233	(9.0)	120	80	200	610	1,110
120	3	11.0	(0.5)	110	180	360	720	1,390
	6	24.3	(0.9)	160	210	380	730	1,390
	15	62.3	(2.3)	140	160	310	580	1,190
	30	124	(4.0)	120	140	360	730	1,300
	60	233	(9.0)	90	110	220	580	1,080

polymers and polymers with a high density of entanglements generate high resistance to flow at the capillary entrance (White 1973; Di et al 1997; Liang and Ness 1997; Koizumi and Usui 1999). An increase in apparent shear rate (from 13 to 1,333/sec) produced a large increase in P_0 values, irrespective of mixing and rest time (Table I). For instance, for dough mixed 3 min and measured immediately after mixing, P_0 increased from 170 to 1,820 kPa. Similar increases in P_0 with shear rate were reported for wheat flour dough (Wang 1995; Huang 1998; Breuillet et al, unpublished) and also for synthetic fluids (Bagley 1957; Philippoff and Gaskins 1958; Langelaan et al 1994; Koizumi and Usui 1999).

Comparing elongational flow (at the entrance of the capillary) to shear flow (along the capillary) was estimated by the ratio $P_0/(P - P_0)$ (Breuillet et al, unpublished). It was not possible to determine a significant effect of apparent shear rate on ratio $P_0/(P - P_0)$ (data not presented). The calculated values of ratio $P_0/(P - P_0)$ are reported in Table II. High values of $P_0/(P - P_0)$ were noticed for the shorter capillaries. This implies a high contribution of entrance flow properties in the total flow properties of wheat flour dough. As expected, increasing the capillary length (L/D 5–40) produced a large decrease in the $P_0/(P - P_0)$ ratio. The contribution of shear flow along the capillary increased with the capillary length.

TABLE II
Elongational Flow at Capillary Entrance (P_0) and Shear Flow Along Capillary ($P_0 - P$) for Dough Measured Immediately After Mixing or After Resting 120 min as a Function of Mixing Time (3–60 min) and Capillary Length[a]

Rest (min)	Mixing (min)	$P_0/(P - P_0)$ at Different Capillary Lengths (L/D ratios)					
		5	10	15	20	30	40
0	3	3.48	1.21	1.11	0.62	0.52	0.34
	6	3.63	1.49	1.03	0.59	0.54	0.35
	15	1.65	1.65	0.82	0.50	0.41	0.26
	30	1.72	1.05	0.68	0.41	0.31	0.23
	60	2.70	0.71	0.86	0.37	0.30	0.21
120	3	3.07	1.51	1.02	0.66	0.57	0.35
	6	2.43	1.77	1.06	0.59	0.52	0.35
	15	2.07	1.40	0.86	0.86	0.54	0.32
	30	2.24	1.16	0.76	0.50	0.39	0.27
	60	1.94	0.75	0.69	0.46	0.28	0.22

[a] Values are averages at different shear rates.

TABLE III
Bagley End Correction Factor (*e*) for Wheat Flour Dough as a Function of Mixing Time (3–60 min) Measured Immediately After Mixing or After Resting 120 min

Rest (min)	Mixing (min)	*e* at Different Apparent Shear Rates (sec^{-1})				
		13	40	133	400	1,333
0	3	12.6	13.7	12.5	13.5	18.4
	6	24.0	11.9	11.1	12.2	11.8
	15	14.7	11.4	9.6	8.7	10.2
	30	9.9	6.7	8.4	10.7	10.6
	60	17.5	4.2	4.5	8.0	8.5
120	3	14.7	13.5	13.2	14.5	18.2
	6	19.2	14.2	11.4	12.1	14.5
	15	23.8	11.8	10.5	10.5	13.6
	30	13.8	7.9	10.2	11.5	11.8
	60	11.5	7.6	6.1	9.2	9.5

TABLE IV
Power Law Parameters for Wheat Flour Dough Measured Immediately After Mixing or After Resting 120 min as a Function of Mixing Time (3–60 min)

Rest (min)	Mixing (min)	Power Law Parameters		
		n	*K* (Pa.sec^n)	R^2
0	3	0.45	1,090	0.991
	6	0.52	871	0.991
	15	0.52	754	0.992
	30	0.53	788	0.994
	60	0.63	405	0.988
120	3	0.52	491	0.996
	6	0.55	518	0.996
	15	0.59	360	0.993
	30	0.56	534	0.998
	60	0.58	470	0.988

The Bagley *e* were determined for wheat flour dough from the Bagley plots by extrapolating the linear functions to $P = 0$ (Table III). The calculated values of Bagley *e* for wheat flour dough are relatively high (4–24) when compared with the tested capillary length (L/D 5–40). This might indicate that some capillaries used in our experiments were not long enough (Bagley et al 1998). High values of the Bagley *e* were also reported in literature for wheat flour dough (Huang 1998; Breuillet et al, unpublished) and need to be carefully considered. The effect of apparent shear rate on the Bagley *e* is not very clear (Table III), even if a slight increase in Bagley *e* seemed to be observed with increasing shear rates from 40 to 1,333/sec. Similar ambiguous trends for the effect of shear rates on Bagley *e* have been described in literature for wheat flour dough (Wang 1995; Bagley et al 1998; Huang 1998; Breuillet et al, unpublished).

The shear flow curves for wheat flour dough were constructed by plotting the corrected wall shear stress against the apparent wall shear (Fig. 3). Shear flow curves were well described (R^2 = 0.988–0.998) by power law models (Equation 5). The parameters of power law models are presented in Table IV. The values of power law index (n = 0.45–0.63) characterize the classical shear thinning behavior of wheat flour dough. The values of *n* were used to calculate the corrected wall shear rate using the Rabinowitsch-Mooney equation (Equation 4).

Shear viscosities were calculated for wheat flour dough (Table V) and plotted as a function of the corrected shear rates (Fig. 4). The calculated values of shear viscosity for wheat flour dough were 12–192 Pa.sec. These values are in same range as those previously determined by Huang (1998) and Breuillet et al (unpublished) for wheat flour dough with a capillary rheometer. The decreases in shear viscosities with increasing shear rates were described with power law models (R^2 = 0.940–0.979) (Fig. 4). The power law parameters are presented in Table V. As expected, the power law index measured from the shear viscosity curves (n = 0.23–0.52) were similar to those found for the shear stress curves (Table IV). The present values of power law index are consistent with the published data for wheat flour dough (Sharma et al 1993a; Berland and Launay 1995; Huang 1998).

Extensional viscosities for wheat flour dough were calculated from values of corrected shear stress and entrance pressure drop (Table VI). Extensional viscosities were plotted as a function of the corrected extensional rate (Fig. 4). The calculated values of extensional viscosity for wheat flour dough were 21–1,300 kPa.sec. These values are in same range as the biaxial extensional viscosity (100–10,000 kPa.sec) determined for wheat flour dough under low deformation rates (Huang and Kokini 1993; Baltsavias et al 1999). The measured extensional viscosities for wheat flour dough seem to display power-law dependency on extensional rate (Fig. 4). Acceptable linear descriptions of extensional viscosity curves (in double Log scales) were obtained for wheat flour dough mixed for 3 or 6 min (Fig. 4). The power law parameters are presented in Table VI. The power law indexes measured from extensional viscosity curves (n_E = 0.39–0.63) also indicate a shear thinning behavior for extensional properties. These values are slightly higher than those found for the shear viscosity curves. Similar strain rate and thinning behaviors were observed for wheat flour dough during biaxial extension (Huang and Kokini 1993; Baltsavias et al 1999).

Extensional viscosities for wheat flour dough are significantly higher than the shear viscosities measured in the same range of strain rate. Viscosities were compared by calculating the Trouton ratio (η_E/η). The calculated values of Trouton ratio (135–443) are relatively high. High values of Trouton ratio for wheat flour dough are due to high values of extensional viscosity that could result from the possible occurrence of strain hardening at the entrance of the capillary (Huang and Kokini 1993; Barakos and Mitsoulis 1995). Similar values of Trouton ratios for wheat flour dough were described in literature (Bagley et al 1998; Huang 1998; Breuillet et al, unpublished). Schluentz and Steffe (1992) indicated that the convergence patterns for wheat flour dough did not follow straight lines and, thus, calculating extensional viscosity from converging capillary data should be viewed with caution.

Effect of Mixing Time

The effect of mixing time (or MEI during mixing) was first considered on the Bagley plot parameters: entrance pressure drop P_0 (Table I), $P_0/(P - P_0)$ ratio (Table II), and Bagley *e* (Table III). An increase in mixing time from 3 to 6 min did not significantly change the values of P_0 and $P_0/(P - P_0)$ ratio (even if slight differences are observed). An increase in mixing time from 3 to 6 min produced a decrease in Bagley *e* (except values measured at 13/sec). For long mixing, an increase in mixing time from 6 to 60 min produced a decrease in P_0, in $P_0/(P - P_0)$, and in Bagley *e* (Table II). For instance, for wheat flour dough measured at 133/sec without resting, an increase in mixing time (from 3 to 60 min) produced a decrease in P_0 (from 560 to 200 kPa) and Bagley *e* (from 12.5 to 4.5).

The effect of mixing time was also considered on shear viscosity, on extensional viscosity, and on the parameters of the related power law models (Tables V and VI). An increase in mixing time did not seem to significantly affect the values of shear viscosity, irrespective of the shear rate (Table V). For the shear viscosity curves, an increase in mixing time (from 3 to 60 min) seemed to produce a slight increase in power law index *n* (from 0.45 to 0.63) and a decrease in consistency *K* (from 964 to 371 Pa.sec^n) when measured immediately after the mixing. After 120 min of rest, the effect of mixing time was not very clear, even if a slight increase in power law index *n* (from 0.52 to 0.58) is observed. On the other hand, an increase in mixing time produced a large decrease in extensional viscosity irrespective of strain rates and rest time (Table VI). Some discrepancies in linearity in power law models were observed for wheat flour dough when mixed 30 or 60 min (Fig. 4). An increase in mixing time did not significantly affect the values of extensional power law index (n_E) but produced a large decrease in extensional consistency (K_E). No significant change in Trouton ratio for wheat flour dough was noticed as a function of mixing time (data not presented).

Long mixing times (or high MEI) seemed to be more efficient in decreasing the resistance to elongational flow (large decrease in extensional viscosity) than in decreasing the resistance to shear flow (slight changes in shear viscosity). Shear flow and elongational flow properties are thus not affected at the same level by the changes in mixing time. These changes in capillary flow properties as a function of mixing time could imply significant changes in dough structure.

Effects of Rest Time

The effects of rest time on rheological parameters (*y*) determined with the capillary rheometer were estimated by calculating the %

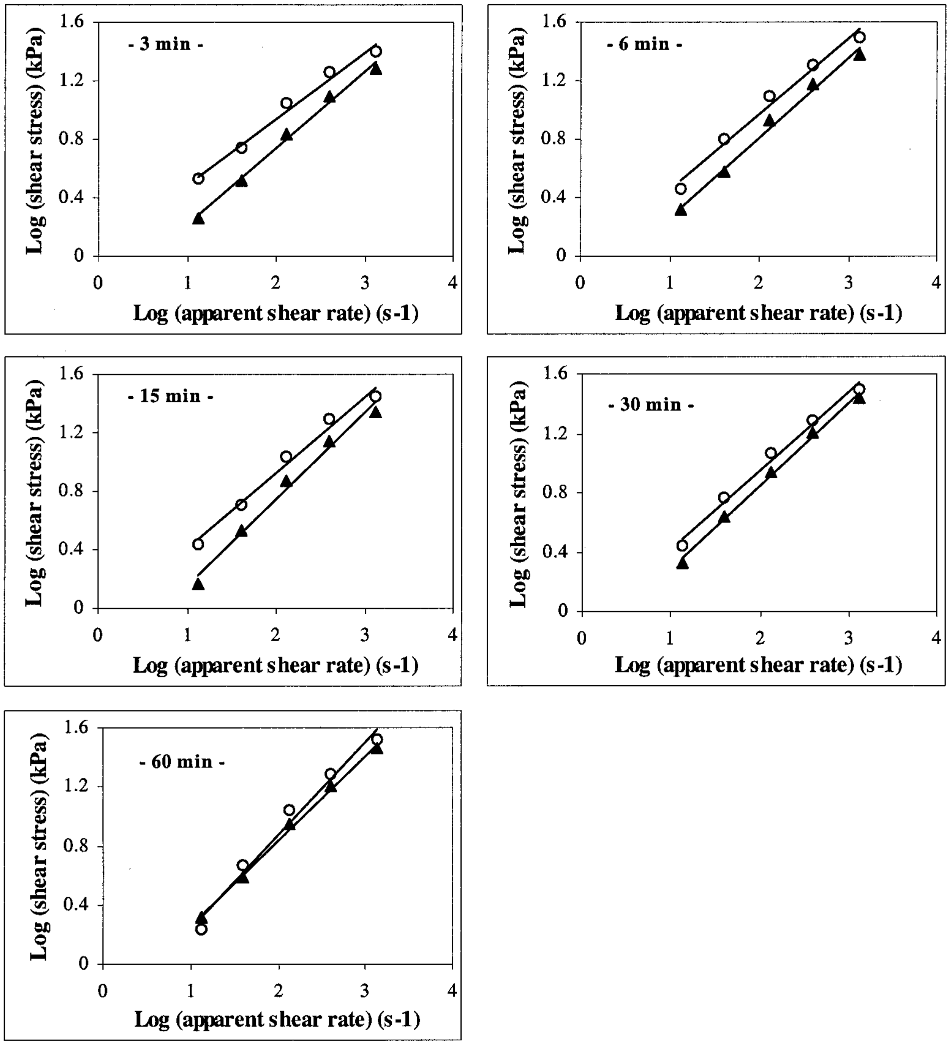

Fig. 3. Flow curves for wheat flour dough as a function of mixing time and after resting 0 min (○) or 120 min (▲).

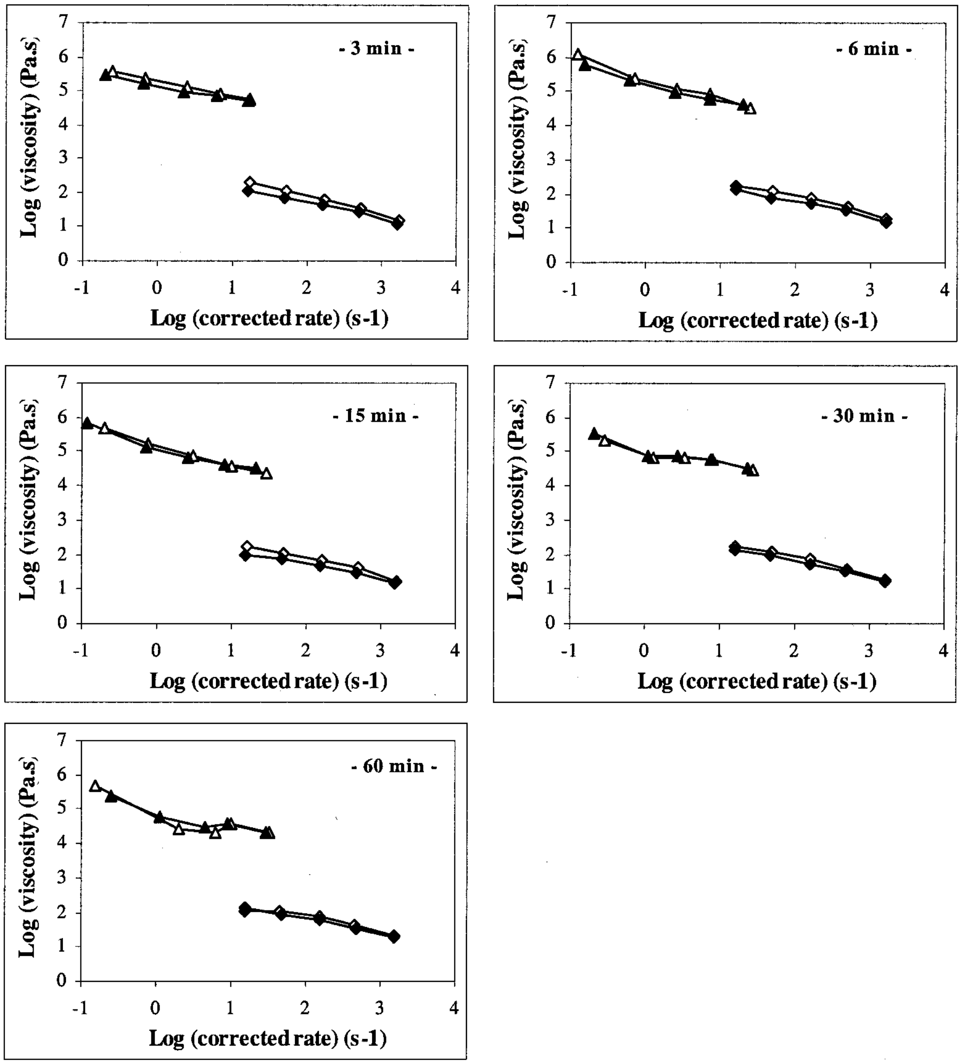

Fig. 4. Shear viscosity curves (◇) and extensional viscosity curves (Δ) for wheat flour dough as a function of mixing time and after resting 0 min (open symbols) or 120 min (solid symbols).

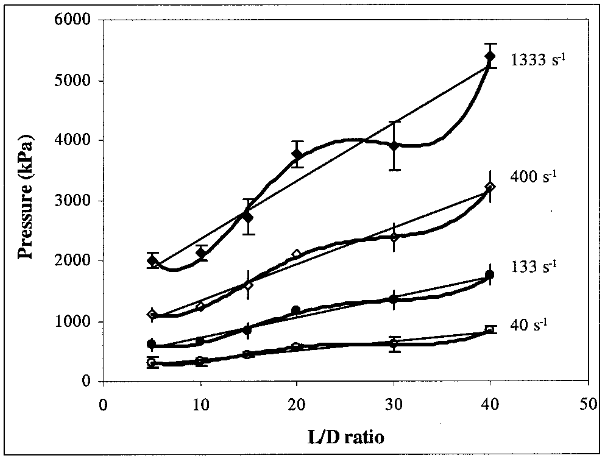

Fig. 5. Nonlinear description of Bagley plots for wheat flour dough mixed for 6 min and resting for 120 min at shear rates from 13 to 1,333/sec.

changes in parameter (Δy) between the values measured immediately after mixing (y_0) and those measured after 120 min of rest (y_{120}) (Equation 12).

$$\Delta y = [(y_{120} - y_0)/y_0] \times 100 \qquad (12)$$

The effect of rest time was estimated on Bagley plot parameters and flow properties (shear viscosity, extensional viscosity, and the parameters of related power law models) of wheat flour dough. Average values of percent changes during resting Δy were calculated from measurements made at different shear rates for each mixing time (Table VII). For wheat flour dough mixed 3 min, we can observe large negative values for percent changes during resting of entrance pressure drop (ΔP_0 = –33%), shear viscosity ($\Delta\eta$ = –32%), shear consistency (ΔK = –54%), extensional viscosity ($\Delta\eta_E$ = –22%), and extensional consistency (ΔK_E = –26%). We also observed slight positive values for percent changes during resting of Bagley *e* (Δe = +3%), power law index for shear (Δn = +15%) and extensional viscosity ($\Delta\eta_E$ = +6%) curves. These changes in dough properties during resting imply that dough is softened during resting if it was initially mixed for a short time.

Increasing the mixing time (from 3 to 60 min) reduced the intensity of changes in flow properties during resting. The Δy values came closer to zero (Table VII), except for Bagley *e*. Thus, the softening effect of dough properties during resting is less pronounced for long mixing times. In addition, we observed changes for ΔP_0 and $\Delta\eta_E$ (ΔP_0 = +6% and $\Delta\eta_E$ = +25%) when dough was mixed for 60 min (Table VII). The changes in capillary flow properties imply that wheat flour dough tends to strengthen during resting if it was initially mixed for very long times.

The shear and extensional viscosity are affected differently by resting, and the effect of rest time on the capillary flow properties

TABLE V

Shear Viscosity and Power Law Parameters for Shear Viscosity Curves for Wheat Flour Dough Measured Immediately After Mixing or After Resting 120 min as a Function of Mixing Time (3–60 min)[a]

Rest (min)	Mixing (min)	Shear Viscosity (Pa.sec) at Different Apparent Shear Rates (sec^{-1})					Power Law Parameters ($Pa.sec^n$)		
		13	40	133	400	1,333	n	K	R^2
0	3	192	105	65	35	14	0.45	964	0.988
	6	175	127	76	41	19	0.52	781	0.980
	15	165	104	66	40	17	0.52	677	0.982
	30	169	118	72	39	19	0.53	708	0.986
	60	112	102	72	42	21	0.63	371	0.934
120	3	111	67	42	25	12	0.52	441	0.991
	6	128	78	52	31	15	0.55	468	0.988
	15	94	72	48	30	14	0.59	327	0.970
	30	133	90	55	33	17	0.56	483	0.993
	60	131	81	56	33	18	0.58	427	0.992

[a] Wheat flour dough mixed 3 min shows large negative values for % changes during resting of entrance pressure drop (ΔP_0 = –33%), shear viscosity ($\Delta\eta$ = –32%), shear consistency (ΔK = –54%), extensional viscosity ($\Delta\eta_E$ = –22%), and extensional consistency (ΔK_E = –26%). Also slight positive values were observed for % changes during resting of Bagley e (Δe = +3%), power law index for shear (Δn = +15%), and extensional viscosity (Δn_E = +6%) curves.

TABLE VI

Extensional Viscosity and Power Law Parameters for the Extensional Viscosity Curves for Wheat Flour Dough Measured Immediately After Mixing or After Resting 120 min as a Function of Mixing Time (3–60 min)

Rest (min)	Mixing (min)	Extensional Viscosity (kPa.sec) at Different Extensional Rates[a] (sec^{-1})					Power Law Parameters ($kPa.sec^n$)		
		0.2	1	4	8	20	n_E	K_E	R^2
0	3	380	243	124	79	59	0.60	171	0.864
	6	1293	231	120	79	34	0.39	212	0.878
	15	457	175	77	38	22	0.45	130	0.894
	30	216	68	64	58	28	0.62	62	0.838
	60	470	24	20	37	21	0.48	48	0.603
120	3	311	156	93	68	49	0.63	126	0.847
	6	614	205	89	60	41	0.51	149	0.818
	15	712	130	69	43	35	0.47	126	0.817
	30	331	73	75	58	32	0.57	110	0.832
	60	229	61	28	37	21	0.61	71	0.642

[a] Average extensional rates.

depends on the initial mixing conditions. A softening effect during resting was observed when dough was mixed for short times (≤15 min); a strengthening effect was observed when dough was mixed for longer times (≥30 min). Similar opposite effects of rest time on rheological properties as a function of mixing conditions of wheat flour dough were reported in our previous work (Cuq and Kokini, unpublished). The changes in capillary flow properties of wheat flour dough during resting depend on mechanical history. Additionally, the experimental farinograph consistency is not correlated (R^2 = 0.131–0.676) to the shear viscosities, but it is well correlated (R^2 = 0.905–0.940) to the extensional viscosities.

The structure of wheat flour dough is not heterogeneous. As a consequence, this parameter cannot be considered responsible for our results. Additionally, it is difficult to visually estimate the structure of the extrudate dough. In future studies, it could be very interesting to develop a visual description of dough extrudate using image analysis techniques.

DISCUSSION

Dough Structure and Capillary Flow Properties

Using a capillary rheometer to characterize the rheological properties of wheat flour dough permitted estimation of the effect of mixing time and rest time after mixing on extensional and shear flow properties. The observed changes in rheological properties could be associated with possible changes in dough structure.

For short mixing times (low MEI), the structural changes in dough during mixing were essentially from the formation of the gluten network through SS bonds and chain entanglements (Bloksma 1972). The gluten network is sufficiently formed when it is stretched during mixing. In these conditions, the MEI is not high enough to induce molecular scission in the gluten network. These changes in dough structure explain the increase in dough consistency measured during the first part of the mixing (Fig. 1).

Increasing the mixing time (over the development time) produces large changes in dough structure. Entanglement points between chains diminish and relaxation by SS bond interchanges between protein chains cannot take place quickly enough to prevent local extension of the chains and molecular SS scission (Danno and Hoseney 1982). These changes in structure of the protein network could explain the softening effect of long mixing times on farinograph consistency (Fig. 1).

During resting, physicochemical processes occur in dough that affect the rheological properties (Bloksma 1972). The softening effect can be related to the mechanical relaxation of the gluten network that was stressed during mixing. The strengthening effect can be related to chemical events and formation of new cross-links in the gluten network due to SS-SH interchange reactions in the presence of oxygen (Mani Lindborg et al 1997). The respective contribution of physical and chemical events during resting seems to depend on the mechanical history of dough (mixing time or level of MEI during mixing).

Changes in capillary flow properties of wheat flour dough during resting were not affected in the same way by changes in mixing time, which could be related to possible changes in dough structure.

The shear viscosities (flow properties along the capillary) are not really affected by an increase in mixing time (Table V). In addition, the changes in shear viscosity during resting are less important when the dough was mixed for longer times (Table VII). Thus, the shear properties seem not to be significantly affected by a decrease (during long mixing) or by an increase (during subsequent resting) in the density of chain entanglements and SS bonds that are involved in dough structure.

TABLE VII
Influence of Resting Time (0–120 min) on Capillary Flow Properties of Wheat Flour Dough as a Function of Mixing Time (3–60 min)

Mixing (min)	Changes in Capillary Parameters During Resting (% of initial value)							
	Bagley Plots		Shear Viscosity			Extensional Viscosity		
	$\Delta(P_0)$	$\Delta(e)$	$\Delta(\eta)$	$\Delta(n)$	$\Delta(K)$	$\Delta(\eta_E)$	$\Delta(n_E)$	$\Delta(K_E)$
3	–33	3	–32	15	–54	–22	6	–26
6	–27	11	–28	6	–40	–19	32	–30
15	–16	16	–29	13	–52	17	5	–3
30	–6	14	–19	6	–32	18	–7	12
60	6	35	–12	–8	15	28	25	–8

TABLE VIII
Exponential Model Describing the Effect of Mechanical Energy Input on Rheological Properties of Wheat Flour Dough[a]

	Rest (min)	Model Coefficients		
		β_1	y_i	R^2
Consistency (farinograph)	…	–0.0012	5.49	0.981
Shear viscosity	0	–0.0001	7.96	0.403
Extensional viscosity	0	–0.0027	5.01	0.744
Shear viscosity	120	0.0004	0.27	0.462
Extensional viscosity	120	–0.0014	5.29	0.949

[a] Average values calculated from measurements at different deformation rates.

The extensional viscosities (flow properties at the entrance of the capillary) are greatly reduced by increased mixing time when measured immediately after mixing (Table VI). In addition, the extensional viscosities increased during resting when the dough was mixed for long times (Table VII). Extensional properties are greatly affected by a decrease (during long mixing) or by an increase (during subsequent resting) in the density of chain entanglements and SS bonds that are involved in dough structure. The effect of mixing time on extensional viscosity is similar to the effect on dough consistency when measured from farinograph torque (Fig. 1). Extensional deformations greatly contribute to the mixing effect of farinograph.

On the other hand, shear and extensional viscosities decreased during resting when the dough was mixed for shorter times (Table VII). The mechanical relaxation of the stretched gluten network seems to contribute to the shear and extensional properties of wheat flour dough.

Previously, Breuillet et al (unpublished) demonstrated that the capillary flow properties of wheat flour dough are not affected in the same way by changes in dough structure due to changes in moisture content. The shear viscosities were greatly affected by changes in moisture content and were related to the properties of the flowing dough structure and, more particularly, to the contribution of low energy interactions (essentially hydrogen bonds). On the other hand, the extensional viscosities were not really affected by the changes in moisture content and were related to the properties of the extended gluten network and, more particularly, the contribution of chain entanglements and covalent bonds.

Modeling

The effects of MEI during mixing on the capillary flow properties of wheat flour dough were described using exponential functions (Equation 13):

$$y = y_i \exp(\beta_1 \text{ MEI}) \qquad (13)$$

where y is the considered rheological property, and β_1 and y_i are the coefficients of the model. Linear regressions in Log scale were used to estimate ability of the model to describe changes in farinograph consistency (Table VIII). The exponential model fitted well (R^2 = 0.981) with the changes in farinograph consistency as a function of MEI during mixing. The negative values of the energy factor (β_1 = –0.0012) means that wheat flour dough is softened by increased MEI. Similar negative energy factors for the farinograph consistency of wheat flour dough were described in a previous work (Cuq and Kokini, unpublished). The exponential model was also used to describe the capillary flow properties (shear and extensional viscosities). As expected, and according to the description of experimental data, unacceptable regression coefficients (R^2 = 0.403–0.462) were found between MEI during mixing and the shear viscosities measured with the capillary rheometer. The shear viscosity is thus not significantly dependent on MEI during mixing. On the other hand, the model fitted well the experimental data for extensional viscosities (R^2 = 0.744–0.949). The negative values of the energy factor for extensional viscosity (β_1 = –0.0027) measured immediately after the mixing indicate that wheat flour dough is softened by increased MEI. After 120 min of resting, the value of energy factor closer to zero (β_1 = –0.0016) implies that the effect of MEI during mixing is less pronounced. Negative energy factors were also reported describing extrusion of starchy products (Vergnes and Villemaire 1987; Senouci and Smith 1988).

These models provide valuable information that could be useful for engineering process simulation, design calculations, control purposes, or optimization studies (Senouci and Smith 1988). However, the use of the measured capillary flow properties for engineering process simulation is limited to the range of variables studied, using equipment with similar characteristics, and should be considered with caution due to the relative sensitivity of the dough structure. Indeed, measuring capillary flow properties for fresh wheat flour dough has several constraints: 1) high mechanical stress applied to dough during filling the barrel (before capillary measurements) could affect the measured capillary flow properties; 2) characterization of capillary flow properties of fresh dough immediately after mixing is theoretically not recommended due to the time-dependence of rheological properties; 3) characterization of capillary flow properties of dough after a resting period (to reach time-independent properties) should be considered with caution because the dough properties change during resting and because the changes in dough properties during resting depend on its mechanical history.

Linearity of Bagley Plots

The Bagley plots constructed for wheat flour dough were considered as nearly linear. However, a close-up examination of experimental data show that the initial part of the Bagley plot (L/D 5–20) could be approximated by a concave upward curve (Fig. 5). Indeed, the measured values of the pressure drop at L/D 20 are observed above the regression lines, while values at L/D 30 are slightly under the regression lines. Similar disturbances in linearity are observed irrespective of mixing and rest time. The Bagley plots might be described as slightly nonlinear, with an initial concave upward curve and S-shape curve (data not presented). Several hypotheses were discussed by Breuillet et al (unpublished) to explain the nonlinear behavior of wheat flour dough.

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