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The rheology of bread dough made from four commercial flours

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Pr. Gras · F. Bekes CSIRO Plant Industry Grain Quality Research Laboratory North Ryde, NSW 1670, Australia Abstract A selection of four commercial flours has been subjected to extensive rheological measurements as part of a comprehensive program of wheat improvement. The results have been used to determine which of the many types of rheological measurements provide significant discrimination between various types of modern baker's flours (including biscuit flours) and to procure data suitable for use in mathematical models describing the dough rheology. The rheological measurements undertaken include oscillatory shear at low amplitude, steady shear at a low shear rate, stress relaxation and extensional viscosity testing. Although oscillatory shear data show minor differences between these flours, the other tests show significant variations and these provide very good discrimination between the different flour types in comparison with conventional dough testing (e.g. by the extensograph). The current dough rheological measurements provide further insight into molecular structure. In the future, mathematical (constitutive) models are expected to provide a means of predicting processing and baking behaviour of bread dough.

Key words Flour dough rheology Oscillatory shear · Steady shear Stress relaxation · Extensional viscosity · Trouton ratio · Error analysis – extensional viscosity Relaxation time spectra · Extensograph · Flour dough testing

Introduction

A selection of four commercial flours has been subjected to extensive rheological measurements as part of a comprehensive program of wheat improvement (under the auspices of the Quality Wheat CRC in North Ryde, Sydney). The intent is to determine which of the many types of rheological measurements provide significant discrimination between various types of modern bakers' flours (including biscuit flours). The rheological measurements undertaken include oscillatory shear at low amplitude, steady shear at a low shear rate, stress relaxation, creep and extensional viscosity testing. Ultimately mathematical models will need to be developed to describe dough

rheology as a means of predicting processing and baking behaviour of bread dough.

The relaxation time spectrum of bread dough is important and this information is required if one wishes to develop a multi mode Maxwell model. The spectrum is also important as it can be used to calculate any viscoelastic material function (e.g. Mead 1994; Weese and Friedrich 1994) and may also be useful in calculating molecular weight distributions (Thimm et al. 2000). Standard procedures (Honerkamp and Weese 1993) are used here to reduce the data.

An analysis is made of the errors in tensile flow. All tensile testers, including empirical instruments such as the extensograph used in the bread industry, are

Table 1 Flour information and mixing times

Flour	Type (description)	Protein content (%)	Water absorption (%)	Mixing time (s)	% HMW/total protein	UPP (%)	FFF MW (kD)
A98	Strong	14.3	63.5	440	31.6	55.8	9940
B98	Strong	12.3	63.0	420	28.8	53.8	6891
C98	Weak (biscuit)	10.3	60.7	330	26.3	45.1	5000
D98	Weak (biscuit)	8.6	55.0	270	28.3	40.5	4584

Table 2 Viscometry

Flour	Peak stress (kPa)	Peak strain	Average slope
A98	2.8	19	~0.5
B98	~ 2.8	19	~ 0.5
C98	1.8	22	~ 0.3
D98	1.0	25	~ 0.2

Table 3 Extensograph

Flour	Rmax	Extensibility (cm)
A98	500	22.0
B98	475	21.1
C98	160	17.4
D98	195	18.8

subject to these experimental errors. As will be shown, these errors can be significant.

Materials and methods

The flours

Four commercial flours, A98, B98, C98, D98, were supplied by Weston Food Laboratories (details as shown in Tables 1, 2 and 3). Protein content was measured by the Kjeldahl method. Water absorption was determined from extensograph measurements supplied by Weston Food Laboratories. All doughs contained 0.2 g of salt and water (no yeast). Mixing times were determined from mixing curves obtained on a 10-g mixograph (Table 1). The doughs were mixed to their respective peak dough development times using procedures developed by Gras et al. (1990).

Flour characterisation

The flours were characterised by Size Exclusion (SE HPLC), Reverse Phase (RP HPLC), Gel Electrophoresis and Field-Flow Fractionation (FFF). Table 1 contains RP HPLC in column 6 and Gel Electrophoresis as UPP score in column 7. FFF data has been incorporated in Table 1. Extensograph measurements are provided in Table 3.

Experimental techniques

The oscillatory shear measurements were made on a Reologica Stresstech rheometer (a stress controlled rheometer) with a 25-mm parallel plate configuration (gap 2 mm) in the frequency range 0.01–90 Hz at high and low strains. Viscometry (steady shear) was

undertaken on a Bohlin VOR rheometer (a strain rate controlled rheometer) with a 30-mm parallel plate configuration (gap 2 mm) at a shear rate of 0.1 s⁻¹. Elongation was performed on a United Testing Machine (tensile tester) also using 30-mm plates at an elongation rate of 0.1 s⁻¹. In all cases, the plates were coated with sandpaper. There is good evidence to suggest that use of sandpaper, as plate roughening, eliminates slip between the dough and the plate (Safari and Phan-Thien 1998; Phan-Thien et al. 1997). In the case of many systems, it has been demonstrated that plate roughening does reduce, if not eliminate, slip between the sample and the plate (e.g. Keentok 1982 and references cited therein). Samples were coated with food-grade grease to reduce drying and then rested for 20 min before testing (to allow relaxation of the stresses imparted during sample handling); it has been shown that resting for 45 min is unnecessarily long.

Results

Oscillatory shear testing

The dynamic moduli G' and G" measured at low strain (strain = 5×10^{-4} , within the linear viscoelastic region) are plotted as a function of frequency for the four flour doughs (refer to Fig. 1). Data are shown up to 60 Hz and we believe these to be the reliable range. Each curve is the average of two sets of measurements. The curves have similar shapes and form parallel curves. There is a perceptible trend as one goes from a strong to a weak flour. The strongest flour is the lowest curve

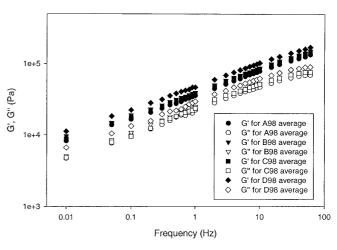


Fig. 1 Dynamic data for four flours at low strain

and the weakest flour is the highest, in the order A98, B98/C98, D98. The degree of dough strength based on UPP% from gel electrophoresis (Table 3) shows the same order as found from rheology. Calculated Payne scores show the same order as UPP score. Such a rheological ordering has also been found by Navickis et al. (1982) who noted that this differentiation was more pronounced at higher water absorption. This may be the reason such a differentiation has not been commonly reported. Safari-Ardi and Phan-Thien (1998) have also shown a differentiation of a strong from a weak dough at low strain, also with this 'reverse' order. Measurements were also taken at high strain (strain = 0.01, outside the viscoelastic limit); however these curves showed less differences between the four flours.

At all frequencies, G' > G'' indicating elasticoviscous behaviour (viscosity is not insignificant). The lack of a plateau in G', and the strong frequency dependence of G' and G", indicate that bread dough is not an elastic gel (Ross-Murphy 1984). Cross-linking is likely to be weak giving rise to a weak structure which is easily destroyed on application of shear ('nonlinked entanglement' of Vinogradov and Malkin 1980). This liquid-like behaviour of the network is consistent with the noncovalent cross-link model of gluten described in the review of Bloksma and Bushuk (1988). In this model, 'elasticity is ascribed to deformation within glutenin subunits and viscosity to slip between the interacting molecules of the gluten complex'. Bloksma and Bushuk (1988) suggest that the noncovalent crosslink model has superseded the earlier continuous protein network model.

Plots of the moduli G' and G" (and the dynamic viscosity) against strain reveal the strain dependence of these properties. All the flours showed approximately the same limit of linear viscoelasticity, namely $1-2\times 10^{-3}$, and this is in agreement with other published data (e.g. Phan-Thien et al. 1997, Safari-Ardi and Phan-Thein 1998). Thus these commercial flours cannot readily be distinguished on the basis of the strain limit of linear viscoelasticity. The magnitude of this strain limit is very small and is indicative of particulate suspensions having a 'weak' structure (Ross-Murphy 1984).

Relaxation time spectra

Relaxation time spectra may be generated from dynamic (oscillatory shear) data using non-linear regularisation by Tikhonov regularisation (Honerkamp and Weese 1993; Friedrich et al. 1995). The program NLREG of Honerkamp and Weese (1993) was used to invert dynamic data to produce a relaxation time spectrum (Phan-Thien and Safari-Ardi 1998).

Dynamic data for the four bread doughs were inverted to obtain relaxation time spectra shown in Fig. 2 (data for C98 is not included because of ripple causing poorer accuracy; C98 is comparable to D98). The plotting symbols are larger than the size of the computed error (typically 1–3%). However it is likely that the error in log H is larger than the computed error (which is calculated by the program).

The spectra for flour doughs (Fig. 2) do not follow the traditional box-and-wedge distribution seen in polymer melts with a narrow molecular weight distribution (White 1980; van den Tempel 1980). There are several possible reasons for this. Bread dough has a very broad molecular weight distribution, the highest fraction of which has a high molecular weight in excess of 1 million. Also, bread dough has a complex structure and contains starch particles (i.e. is a suspension). The broadness of the molecular weight distribution and the high molecular weight is confirmed by FFF for the current flours (see for example the molecular weight in Table 1). Other flours also display a broad molecular weight distribution, e.g. Wahlund et al. (1996) demonstrated many fractions beginning at 23,000 and extending to 11,000,000. These spectra have a strong similarity to the relaxation time spectra of filled polymer melts (e.g. Ferry 1961) and plastic dispersions (van den Tempel 1980). The existence of a solid phase in bread dough (consisting of starch granules of two different size distributions) is well known in cereal chemistry (Bloksma and Bushuk 1988).

Comparison of the relaxation spectra (Fig. 2) reveals that there are measurable differences showing a consistent trend of a preponderance of long relaxation times associated with the strong flours and short relaxation times with the weak flours. The strongest flour has more long relaxation times and less short relaxation times

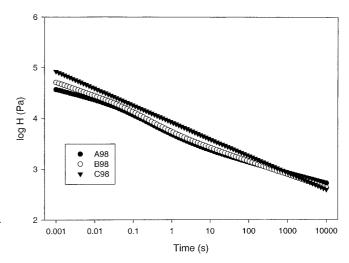


Fig. 2 Relaxation time spectra of flours

than the next strongest flour while the weakest has less of the long relaxation times than any of the others. It should be emphasised that there is a perceptible difference between C98 and D98: C98 has less of the long (and short) relaxation times than D98. This is in accord with RP-HPLC data which shows % HMW for C98 and D98 as 26.3% and 28.3% respectively (Table 1). On the basis of the relaxation time spectra, the ordering of the flours (strongest to weakest) is A98, B98, D98 and C98. On the basis of % HMW/total protein (refer to Table 1), the ordering is also A98, B98, D98 and C98. The agreement between rheology (as determined by relaxation spectrum) and molecular weight characterisation (by RP-HPLC) is gratifying.

Phan-Thien and Safari-Ardi (1998) show the effect of water concentration on the relaxation spectrum of one type of bread dough. Phan-Thien and Safari-Ardi (1998) also show an inconsistent ordering of the curves for the different water absorptions which cannot be explained easily. The relaxation spectra for a bread dough, computed from oscillatory data and from relaxation data by Phan-Thien and Safari-Ardi (1998), show a significant difference in the high relaxation time end of the spectrum (t > 100 s). This is likely to be due to the difference in the duration of the test to obtain the oscillatory data (<9 min) and the relaxation data (~80 min). Cunningham and Hlynka (1954) found that the dough rest period affects the relaxation spectrum: the spectrum with the short rest period (<5 min) shows a spectrum extending to 1000 s whereas the spectrum with the long rest period (>45 min) shows a loss of relaxation times > 10 s. Thus the spectrum obtained from the relaxation data of Phan-Thien and Safari-Ardi (1998) has almost certainly lost the high relaxation time tail from the spectrum due to over-resting of the sample. Long-duration tests (>60 min) are not recommended for dough rheology testing as the dough degrades with time.

Extensional testing

Extensional or tensile testing was undertaken on the United Tensile Testing Machine at two strain rates 0.1 s⁻¹ and 1.0 s⁻¹. Tensile stress at an extension rate of 0.1 s⁻¹ is plotted as a function of time for the four flours from an average run (refer to Fig. 3) and in all cases it has been assumed that the shape of the sample is roughly cylindrical. The diameter of the dough filament was calculated assuming it is cylindrical in shape and that the volume of the sample remains constant with time. The measurements were repeated (and in some instances triplicated).

The curves can be characterised by the peak stress and peak strain at which the sample ruptures (refer to Table 4). The error quoted is the experimental error as

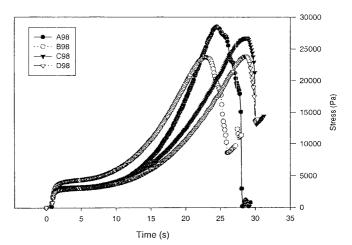


Fig. 3 Extensional data for four flours at a strain rate of 0.1 s⁻¹

Table 4 Elongational flow at strain rate of 0.1 s⁻¹: peak stress, peak time and peak strain for four flours

Flour	Peak stress (kPa)	Peak time (s)	Peak strain
A98 B98 C98 D98	$\begin{array}{c} 28.5 \pm 0.1 \\ 23.6 \pm 0.1 \\ 26.7 \pm 0.1 \\ 23.9 \pm 0.1 \end{array}$	$\begin{array}{c} 24.5 \pm 0.1 \\ 23.0 \pm 0.2 \\ 28.7 \pm 0.1 \\ 28.7 \pm 0.1 \end{array}$	2.5 2.3 2.9 2.9

the statistical error is considerably less. There was little variation in this data (<1%, refer to Table 4), which is very satisfactory. On the basis of peak strain, the weak flours have the highest rupture strain (2.9) and the strong the lowest (2.4). On the basis of peak stress, the strongest flour has the highest stress (and hence the highest extensional viscosity) while the weakest has the lowest stress. Using both peak stress and peak strain, the four flours can be readily distinguished.

The discrimination of C98 from D98 will now be calculated. The discrimination of a property here is defined as the ratio of the property divided by the magnitude of the experimental error of the property. On the basis of peak stress, the ratio of C98 to D98 is 26.7/23.9 with an error of 0.1. Thus the discrimination is 11. The comparative figure for the extensograph data for C98 and D98 (assuming an error of 1 cm) is 1.4.

It is helpful to compute the Trouton ratio, T, given by $T = \eta_E/\eta_0$, where η_E is the extensional viscosity and η_0 is the limiting zero-shear-rate viscosity. Since bread dough is elasticoviscous and does not exhibit pure elasticity (except at very small strains; Phan-Thien et al. 1997), it is possible to talk of η_0 . η_0 is difficult to measure accurately for bread dough since the viscosity is non-Newtonian at all accessible frequencies. However, in this instance η_0 has been estimated from G''/ω at 0.01 Hz. At a strain rate of 0.1 s⁻¹ we have T = 4, 3, 3 and 2 for A98, B98, C98 and D98 respectively. Since T = 3.0 for a Newtonian

liquid, we have pseudo-Newtonian behaviour for all but the strongest flour (A98), an unexpected result which requires further investigation. The T values obtained at a higher extension rate (1.0 s^{-1}) were generally an order of magnitude smaller and thus occur on the extensionthinning part of the η_E curve. At a slower extension rate of 0.01 s⁻¹, data for an older Baker's flour (B97) showed that the T values were higher by a factor of 4 relative to T at 0.1 s⁻¹. So it is suggested that the 0.01 s⁻¹ extension rate corresponds to a peak in the η_E curve. Measurements of tensile stress at a strain rate of 0.01 s⁻¹ (rather than 0.1 s⁻¹) may provide a more sensitive measure of tensile properties. Thus it may be profitable to make future measurements at the 0.01 s⁻¹ extension rate, which is comparable to extension rates found in baking (Bloksma 1990).

Error analysis for extensional testing

An international collaboration on extensional testing of a standard polymer solution has revealed significant experimental errors in measuring tensile stress and extensional viscosity (James and Walters 1993; Tanner and Walters 1998). Part of the reason for the discrepancies lies in the lack of reaching a steady state in the extensional flow. Care has been taken in this respect in the current measurements. Measurement of the actual strain rate as a function of time has shown that the United Testing Machine can take 1-10 s to reach its target strain rate. Allowance must also be made for the tensile stress relaxation time of the dough sample in assessing whether a steady state is reached in a typical run. Tensile data for polymer melts show that this relaxation time falls mostly in the range 10-30 s, depending on the strain rate, the slowest relaxation time for the slowest strain rate (Prud'homme 1991). On this basis, for the current experiments at least 20 s must be allowed for equilibration of the sample and thus the duration of a 'pull' has to be greater than this to ensure equilibration. This effectively eliminates the strain rate of 1.0 s⁻¹ (duration ~3 s); however 0.1 s⁻¹ or slower is likely to reach some sort of 'steady state'. On the other hand, a strain rate as low as 0.001 s⁻¹ may be problematic for other reasons. It should be said that the extensograph is also subject to the same problem and typical run times for the extensograph indicate a likelihood of unsteadiness.

Another consideration is that the estimated experimental errors are in fact much larger than expected. A simple analysis was made using typical values of the experimental error in force and radius. These equations give rise to the experimental errors listed in Table 5. Near the end of the pull, the errors can be substantial, of the order of 20% in both the tensile stress and the strain rate. Since this analysis only includes errors in the radius

Table 5 Estimated tensile stress and strain rate errors derived from errors in force and radius

	δF/F (%)	2δr/r (%)	δσ/σ (%)	δε/ε (%)
Start of pull	2	1	4	2
Near end of pull	2	10	22	20

and force, and excludes all other effects, it is likely that the actual total error is larger. The same type of error is present in extensograph measurements; however it is likely that the magnitude of the errors present in extensograph measurements are considerably higher due to rheological imperfections such as unsteadiness of the strain rate and the difficulty of calculation of the tensile stress and the strain rate.

Viscometry

Since bread dough is a time-dependent elasticoviscous material which apparently does not show a steady state viscosity, it is not valid to do the true steady state shear stress against shear rate curve. It is more usual to plot shear stress against time (or strain) at a fixed shear rate (refer to Fig. 4) and measurements may then be taken at a number of shear rates. The curves show three regions (Phan-Thien et al. 1997): region 1 is the region of linear viscoelasticity (Fig. 4, time ~0.1 s), region 2 is the region of strain-softening (time between 0.1 s and 10 s) and region 3 is the region of fracture (time > 10 s). Figure 4 shows that the four flours are readily distinguished by viscometry at a shear rate of 0.1 s⁻¹. Viscometry ranks the flours in the order A98 = B98, C98 and D98. This order is similar to UPP scores shown in Table 1 (order: A98, B98, C98 and D98).

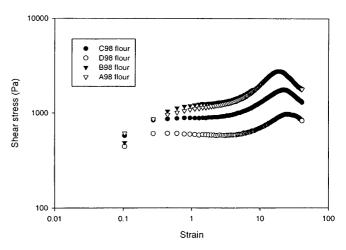


Fig. 4 Viscometry for four flours at shear rate of 0.1 s⁻¹

The viscometry curves can be analysed also in terms of the peak stress, peak strain and the average slope of the curve in the strain-softening region and this information is given in Table 2. The only comparable literature is that of Phan-Thien et al. (1997) and their data for an uncharacterised strong flour agrees with that for A98 in terms of all three parameters. In comparison, extensograph extensibility cannot differentiate C98 from D98 nor A98 from B98 (refer to Table 3) and a similar picture exists for extensograph Rmax.

Viscometry provides the most accurate discrimination on the basis of peak stress: from Table 2, C98 and D98 are 1.8 and 1.0 respectively with an error less than 0.1. Thus the ratio of the peak stresses is 1.8 and, relative to the error, this is 18. The comparative figure for the extensograph data for C98 and D98 (assuming an error of 1 cm) is 1.4.

Stress relaxation

The stress relaxation test provides the shear modulus G as a function of time at a given strain. High-strain data (100%, strain 0.15) are plotted for the four flours in Fig. 5. The two strong flours, having the highest G, are easily differentiated from the two weak flours. The two weak flours can be distinguished from each other, D98 having a longer relaxation time tail which extends past 1000 s. From polymer rheology, this indicates that D98 has more of a high MW tail than C98. This observation is supported by the relaxation time spectra of C98 and D98 (see below for further discussion on relaxation time spectra). Molecular weight characterisation by RP-HPLC shows that D98 has more HMW glutenin than C98 (refer to Table 1). By stress relaxation, the ranking is A98 = B98, D98 and C98, which is comparable to ranking by % HMW/total protein when allowance is

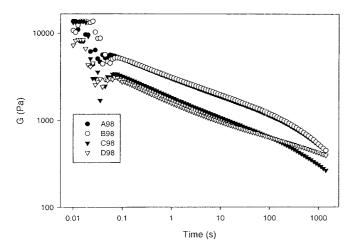


Fig. 5 Stress relaxation at high strain for four flours

made for experimental error (error on $HMW \pm 1\%$) (refer to Table 1; ranking order is A98, B98, D98 and C98). The same ordering (i.e. extra strong, strong, medium and weak) from stress relaxation was also seen by Safari-Ardi and Phan Thien (1998). In comparison, extensograph extensibility cannot differentiate C98 from D98, nor A98 from B98 (refer to Table 3) and a similar picture exists for extensograph Rmax.

It is important to note that none of the stress relaxation curves in Fig. 5 reaches a plateau at large t, a finding consistent with Phan-Thien and Safari-Ardi (1998). It is not possible to extend the measurements to much longer times as the bread dough degrades significantly after this duration. From this experimental data it is not possible to definitively rule on the existence of an elastic modulus (Phan-Thien and Safari-Ardi 1998) and hence on solid-like behaviour. However, within practical experimental limits these stress relaxation curves appear to show a viscous response. MacRitchie (1980) has come to a similar conclusion on the basis of creep data for bread dough (furthermore he concludes that there is no cross-linking in bread dough).

Conclusions

All the rheological properties of bread dough are sensitively dependent on the dough composition, i.e. water and salt content, and the mixing time. Care has been taken in the selection of water absorption and mixing time. It is possible to distinguish the four commercial flours on the basis of viscometry, stress relaxation and oscillatory shear at low strain (when care is taken). Other rheological tests, including tests under large strain conditions, may also prove to be useful for discrimination between flours. It has also been shown that basic rheological measurements offer discrimination in cases where the extensograph cannot discriminate. Calculations have been made of the degree of discrimination between the bread doughs using basic rheological measurements. In the case of C98 and D98, for viscometry, the degree of discrimination is 18, while for elongational flow it is 11. These figures compare with 1.4 for the extensograph. Thus basic rheological measurements are considerably more precise in their discrimination between flours than conventional dough testing with the extensograph.

Different rheological tests are sensitive to different molecular properties. In terms of bread dough rankings, viscometry approximates UPP score whereas stress relaxation approximates data obtained from RP HPLC.

Oscillatory shear data indicate there is a 'weak' network in bread dough (i.e. approximating a non-covalent crosslink model). Similarly the stress relaxation data approximate a viscous response. Gras et al. (2001) discuss how a weak entanglement/crosslink model may

apply to dough rheology. The relaxation time spectra cover a broad range of relaxation times (0.001–10,000 s). Several inferences can be drawn from this: there is a broad molecular weight distribution, which is borne out by molecular weight characterisation using FFF, the molecular weight is high (again supported by FFF) and there is a suspended phase (well known in the cereal chemistry literature). Characteristics of the relaxation time spectrum can be related to the strength of the bread dough.

In future experimental work, it is recommended that extensional viscosity measurements are undertaken also

at 0.01 s⁻¹, as stronger viscoelastic effects may occur at that elongation rate and thus would provide better discrimination. Also it is recommended that the dough diameter is measured during tensile testing so that the diameter correction can be applied.

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