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Viscoelastic properties of durum wheat and common wheat dough of different strengths

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Abstract Linear viscoelastic properties (LVP) were determined for five durum wheat doughs and five common wheat doughs (representing four different classes of Canadian common wheat) of different strength using creep testing. A creep time of 10,000 s was sufficient to reach a state of steady state flow for all of the doughs. Creep compliances were analyzed in terms of a Burgers model. For the durum doughs, the entire elastic compliance curve was shifted to higher values as the strength of the dough (as measured by extensigraph) decreased, while the steady state viscosity increased with strength. For common wheat doughs, the elastic compliance curves were steeper and the steady state viscosities were lower than for durum doughs of comparable extensigraph strength. The retardation strengths associated with a maximum in the retardation spectra were lower for the stronger durum doughs than for common wheat doughs of comparable strength. Differences in the LVP between durum and common wheat doughs of similar extensigraph strength were interpreted in the context of physical gels with crosslinks and entanglements, whose contributions to material properties are difficult to distinguish in short-time creep or dynamic measurements. The increased extensibility of common wheat doughs relative to durum doughs of comparable extensigraph strength was attributed to a higher molecular weight fraction in the polypeptide chains, similar in some respects to end-linked bimodal polymer networks. The idea of considering these doughs as physical gels was supported by their stress relaxation behavior.

Key words Dough rheology · Durum wheat · Common wheat · Creep

Introduction

Baked goods and pasta products are generally produced from different classes and cultivars of wheat flours. Common wheats (*Triticum aestivum*) are preferred for white pan bread, Asian noodles, and cakes and pastries. Breeding targets for common wheat intended for high volume pan bread include a balance between elasticity and extensibility to ensure good sheeting properties, and ability to expand and hold gas during the baking process (Dexter 1993). For Asian noodles intermediate strength is preferred, with an emphasis on extensibility for optimum sheeting properties. For confectionery products, a weak gluten is best to impart the desired textural attributes.

Durum wheats (*Triticum durum*) are used around the world primarily for the manufacture of pasta, which is extruded, and for couscous, which is made by agglomerating semolina (a coarse flour). Extensibility is not required for either process, although strong gluten is recognized as an asset for good pasta cooking quality (Feillet and Dexter 1996). Efforts to breed strong gluten durum wheat lines for improved pasta texture have resulted in tenacious inextensible gluten (Quaglia 1988; Rao et al. 2000b). Durum wheat is increasingly being used for flat breads and specialty breads, primarily in Mediterranean countries. There is interest in developing strong cultivars with a better balance between resistance to extension and extensibility for more general end-use application (Redaelli et al. 1997; Liu et al. 1996).

Genetically, durum wheats are tetraploids (AABB), and are lacking the D genome found in hexaploid common wheats (AABBDD). The D genome is believed to code for the very high molecular weight glutenins that give bread wheats their superior bread making qualities. Redaelli et al. (1997), working with common wheat near-isogenic lines, demonstrated that chromosome 1D strongly influenced both dough elasticity and extensibility. Removal of the D genome from hexaploid bread wheat greatly reduces its baking potential (Kerber and Tipples 1969) and the absence of the D genome is considered at least partly responsible for the relatively poor bread making quality of durum wheat.

There is an abundance of experimental evidence indicating that both wheat flour doughs and their respective glutens (hydrated wheat flour proteins) are viscoelastic. This literature has been summarized in a number of reviews from different perspectives (e.g., Baird and Labropoulos 1982; Faubion and Hoseney 1990; Eliasson 1990). Baird and Labropoulos (1982) focused on those studies leading to well-defined material functions and application of viscoelastic models to food doughs in general. They noted that food doughs exhibit both storage and loss moduli in small amplitude oscillatory testing, stress overshoot at the start up of shear flow, stress relaxation at the cessation of flow, viscosity which depends on shear rate, and normal stress differences. However, they also noted that the chemistry of food systems was much more complicated than for polymeric systems such as polymer melts in which the viscoelastic properties are attributed to an entanglement network. Even so, attempts have been made to apply constitutive models to doughs and glutens. Dus and Kokini (1990) have modeled the nonlinear viscoelastic properties of a hard wheat dough and Wang and Kokini (1995) the same for a commercially available hydrated gluten using the Bird-Carreau model. Although agreement between the model and experimental data was good for the wheat dough and gluten, it must be pointed out that the Bird-Carreau model was developed and applied to dilute polymer solutions and polymer melts. Thus, the empirical constants of the Bird-Carreau model obtained for wheat doughs and glutens, which do show evidence of entanglement plateaus in stress relaxation data (e.g., Bohlin and Carlson 1981; Rao et al. 2000a), probably cannot be related to the same molecular theories of linear viscoelasticity as was originally done. As will be discussed below, the fact that doughs and gluten are gel-like materials at rest, but show extensive creep flow and stress relaxation at long times, suggests that wheat doughs and glutens should be considered as physical gels containing both reversible crosslinks and entanglements.

Unlike synthetic polymers, which are generally characterized by their molecular properties (e.g., molecular weight, polydispersity index, zero shear viscosity), individual wheat cultivars are classified by cereal scientists according to their strength. Strength is related to the mixing properties of a particular cultivar and the resistance to extension of the dough after mixing. Generally, as the strength of a cultivar increases the dough development time increases, and its subsequent resistance to extension also increases. However, strength in this context does not have the same precise meaning that it does in materials science, e.g., stress at rupture in a tensile test. Strength in the cereal science sense is probably an attempt to capture in a single concept the blend of strength, extensibility, and toughness that a dough exhibits. Although the strength of mixed doughs clearly originates from its gluten, the actual molecular structure of the hydrated gluten polymer is still a matter of debate.

Gluten has been characterized as having a bimodal distribution between monomeric gliadin proteins and polymeric glutenin proteins (Wrigley and Bekes 1999). However, the glutenin fraction itself is also polydisperse. Hydrated gliadin alone is viscous, while hydrated glutenin is a tough, rubbery material. These phenomena have sometimes led to the erroneous assumption in the cereal science literature that gliadin provides the viscous component of gluten, while glutenin alone is responsible for its elastic properties. In part this may be due to the extensive use of large strain, empirical instrumental methods to determine the physical properties of doughs (i.e., their strength) rather than characterization of their strength in terms of their fundamental linear viscoelastic properties (LVP). For polymer systems of very high molecular weight, determination of the LVP is increasingly being viewed as a means of molecular characterization due to the difficulties in solubilizing and separating very large polymers using traditional gel permeation chromatography or SE-HPLC (Marin and Montfort 1996). Applying some of these same rheological approaches to wheat doughs of different strength may help to understand better the underlying macromolecular structures in gluten that are responsible for its unique physical properties.

The actual macromolecular structure of the glutenin polymer is still not known for certain. The varied protein-protein interactions in gluten and their possible relationships to dough rheology have been summarized very well by Wrigley et al. (1998). However, it is still worth interpreting some of the earlier hypotheses for its structure in a modern polymer science context. Two hypotheses, one proposed by Greenwood and Ewart (1975) and the other by Bloksma (1975), together seem to capture the essence of what the structure of glutenin polymer might be. Greenwood and Ewart proposed that the entangled long, linear (unbranched) molecules thought to be needed for rubber-like elasticity were formed in glutenin during dough mixing by the end-to-end linking ("concatenation" in the author's words) of individual polypeptides via disulfide (S-S) bonds. The rubber-like elasticity of glutenin was supposedly due to entanglements and other secondary interactions, but polymerization of polypeptide units by SS bonds was a prerequisite to entanglements.

Bloksma (1975) proposed that the elastic deformation of dough at a particular stress was related to the number of rheologically effective disulfide cross-links, and that viscous flow was the result of breaking and reforming of these disulfide bonds via a thiol-disulfide interchange reaction. This latter hypothesis is very similar to that for a cross-linked rubber with breaking and reforming of cross-links during deformation as described by Alfrey (1945). Taken together, these two hypotheses actually describe a physical gel consisting of entangled polymer chains and physical crosslinks together. Networks with reversible junctions exhibit highly enhanced viscoelastic properties which are quite different from polymer melts with entanglements (Tanaka and Edwards 1992a).

Furthermore, it is generally assumed that polypeptide chains in the glutenin protein fraction have the potential for forming one or more disulfide bonds only at their ends. Thus, the number of physical crosslinks would actually increase as the average molecular weight of this effective fraction decreased for the same protein content. This suggests that strength in bread wheat and durum doughs may be strongly influenced by lower molecular weight glutenin polypeptides. This is contrary to the conventional wisdom that strength in glutenin is due primarily to entanglements and/or the molecular weight distribution in the highest molecular weight fraction only (Weegels at al. 1996), which assumes gluten is similar to an uncrosslinked polymer melt.

Relatively few studies have determined the linear viscoelastic behavior of doughs or glutens in creep or stress relaxation as it relates to differences in strength, especially differences between durum and common wheat doughs. None appear to include interpretation of the experimental results in the context of physical gels. For a physical gel, the LVP will clearly depend upon both the number of crosslinks and entanglements, and the time scale of measurement relative to the average lifetime of the crosslinks and the terminal relaxation time of the physical gel (Leibler et al. 1991). Previous experimental creep results have indicated that gluten gel can be considered as ideal elastic at shorter times, and viscous at longer times, with an intervening period of retarded elasticity (Hibberd and Parker 1979; Funt Bar-David and Lerchenthal 1975). Gluten gel has also been found to relax stress completely in about 10,000 s (Funt Bar-David and Lerchenthal 1975; Bohlin and Carlson 1981). All of these results are consistent with a physical gel containing physical crosslinks and entanglements. Clearly, both crosslinks and entanglements could contribute to short time or high frequency small amplitude oscillatory measurements of dough strength, or high rate of deformation large-strain rheological assessments of dough strength. Physical crosslinks would also be expected to increase the longest relaxation times of the physical gel and increase its viscosity (Leibler et al. 1991).

The objectives of this work were then to determine the LVP of doughs from common wheat and durum wheat cultivars representing a broad range of strength and of known end product quality, using creep testing. Creep testing was chosen because it is assumed that the blend of elasticity and viscosity of doughs is somehow critical to their functional performance in processing. However, there is very little published data comparing the dynamic and/or steady state elastic compliances or zero shear viscosities of doughs of different strength. In addition, this work will be supported by size exclusion high performance liquid chromatography (SE-HPLC) analysis of the relative amounts and distributions of the glutenin and gliadin protein fractions, large strain (nonlinear) descriptive rheological properties, and baking performance of each cultivar. Recent work characterizing some of these same doughs in stress relaxation showed that stronger doughs were characterized by longer relaxation times and more extensive entanglement plateaus (Rao et al. 2000a, b). It was of interest here to determine whether differences in dough strength among and between bread wheat and durum doughs would also be reflected in their retardation times, compliances, and viscosities. It is expected that this integrated approach involving the chemistry, descriptive rheology, and LVP (both stress relaxation and creep) of a broad range of wheat cultivars will result in new insights as to how differences in macromolecular structures in doughs are related to differences in their strength and their bread-making performance.

Materials and methods

Wheat cultivars

Five cultivars of Canada Western Amber Durum (CWAD) wheat with a wide range in gluten strength were chosen for this study. Stewart 63 is an old variety no longer grown commercially, with very weak gluten. Wascana, AC Avonlea and AC Morse are currently registered varieties ranging in gluten strength from relatively weak (Wascana) to moderately strong (AC Morse and AC Avonlea). AC Pathfinder was registered in 1998 for test marketing outside of the CWAD class as a blending wheat, due to its extraordinarily strong gluten.

The five common wheat cultivars chosen represent four different classes of Canadian common wheat, all with different targeted end uses and, therefore, different gluten quality characteristics. All were bred and selected for a high degree of dough extensibility, but for different degrees of elasticity. Canada Western Extra Strong (CWES) wheat (breeding line ES 12) is primarily intended as a blending wheat to improve dough strength, so CWES varieties have extraordinarily strong gluten and long mixing requirements. Canada Western Red Spring (CWRS) is a premium bread wheat class for production of high quality bread, with moderate to strong gluten and balanced dough properties. The varieties Neepawa and Laura represent the minimum and maximum gluten strength for the class, respectively. The Canada Prairie Spring White (CPSW) class (breeding line HY 443) is intended primarily for production of Asian noodles and has more moderate gluten strength and lower protein content than CWRS. Canada Western Soft White Spring (CWSWS) wheat (breeding line SWS 238) is primarily used for production of cakes and cookies, so has intrinsically low protein and weak gluten.

Wheat processing properties

Unlike synthetic polymers or elastomers, it is nearly impossible to characterize the molecular level properties of the gluten (e.g., molecular weight distribution, molecular weight between cross-links, zero shear viscosity, number of effective disulfide bonds, etc.) in a mixed dough. Therefore, Tables 1 and 2 and Figs. 1 and 2 represent the analytical composition of the ten wheat cultivars and doughs used in this study, along with their mixing properties and baking characteristics.

Wheats were milled as previously described (Rao et al. 2000a, b). Flour and semolina protein contents (%N X 5.7) were determined by Combustion Nitrogen Analysis (LECO Model FP-428 Dumas CNA Analyzer, St. Joseph, MD). Optimum water absorption (Tables 1 and 2) was determined by farinograph (CW Brabender, South Hackensack, NJ) by AACC (1995) Method 54–21. A 2-g direct drive mixograph (National Manufacturing Division, TMCO, Lincoln, NE) was used to rank further samples according to mixing strength and to determine time required to mix to peak consistency (Figs. 1 and 2).

Doughs from different wheat cultivars are necessarily prepared at different moisture contents (absorption) due to their inherent differences in water absorption. Extensigraph (CW Brabender, South Hackensack, NJ) tests were performed by the American Association of Cereal Chemists (AACC) (1995) Method 54-10 at farinograph water absorption to establish dough strength. The strength of a wheat cultivar can be quantitatively evaluated from its maximum resistance to extension in Brabender units (BU), extensibility (curve length in mm), and area under the extensigraph curve. Extensigraph curves are shown in Figs. 1 and 2 for each cultivar.

Table 1 Durum wheat semolina quality, dough mixing properties, and baking quality

	AC Pathfinder	AC Morse	AC Avonlea	Wascana	Stewart 63
Semolina protein, %	11.9	12.0	12.8	12.2	12.0
Farinograph					
Absorption, %	57.8	55.7	55.4	55.0	55.0
2 g Mixograph					
Time to peak, min	3.6	3.4	2.7	2.6	2.0
Absorption, %	50	50	50	50	50
Dough moisture content, %	42.7	42.7	42.7	42.7	42.7
Dough protein, %	7.9	8.0	8.5	8.1	8.0
SE-HPLC					
Gliadin/glutenin	0.60	0.73	0.71	0.76	0.89
Unextractable protein, %	17	16	15	15	16
Remix-to-peak bread					
Remix-to-peak energy, whr/kg	3.3	2.3	2.0	1.3	0.5
Loaf volume, cc	715	610	550	470	390
Baking Strength Index, %	92	78	66	59	50

Table 2 Common wheat flour quality, mixing properties, and baking quality

	ES 12	Laura	Neepawa	HY 443	SWS 238
Flour protein, %	12.0	12.3	13.6	11.7	9.8
Farinograph					
Absorption, %	62.9	64.5	65.9	65.1	56.1
2 g Mixograph					
Time to peak, min	6.1	3.9	3.2	2.8	1.5
Absorption, %	60	60	60	60	50
Dough moisture content, %	46.3	46.3	46.3	46.3	42.7
Dough protein, %	7.5	7.7	8.5	7.3	6.5
SE-HPLC					
Gliadin/glutenin	0.53	0.67	0.72	0.76	0.60
Unextractable protein, %	25	13	16	12	11
Remix-to-peak bread					
Remix-to-peak					
Energy, whr/kg	3.6	2.6	2.9	1.9	0.6
Loaf volume, cc	920	925	860	740	390
Baking Strength Index, %	118	115	96	97	54

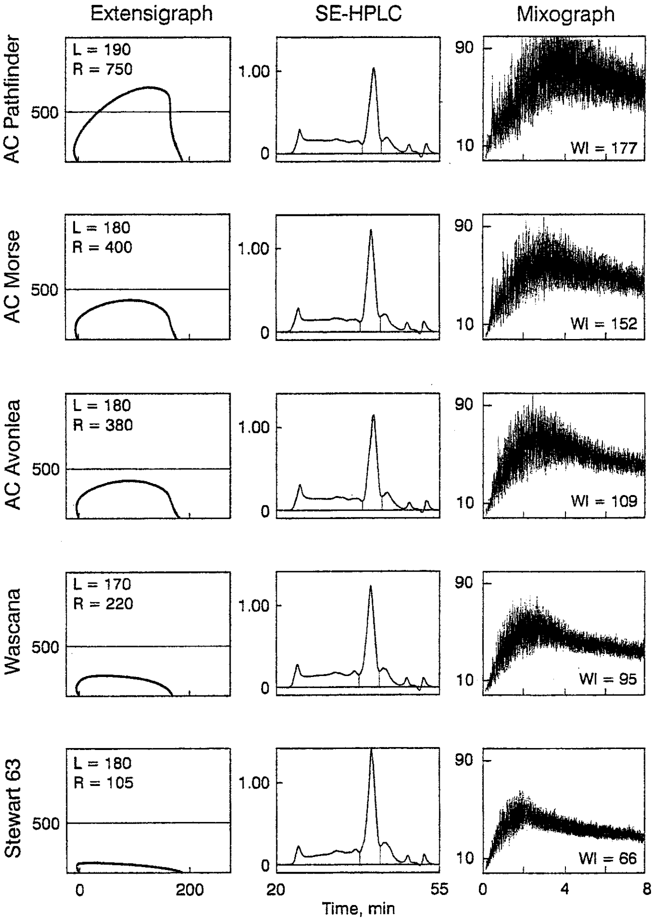

Fig. 1 Durum wheat semolina extensigrams, SE-HPLC profiles, and 2g-mixograms. L = extensibility, mm; R = maximum curve height, Brabender Units (BU); WI = work input to peak, arbitrary units

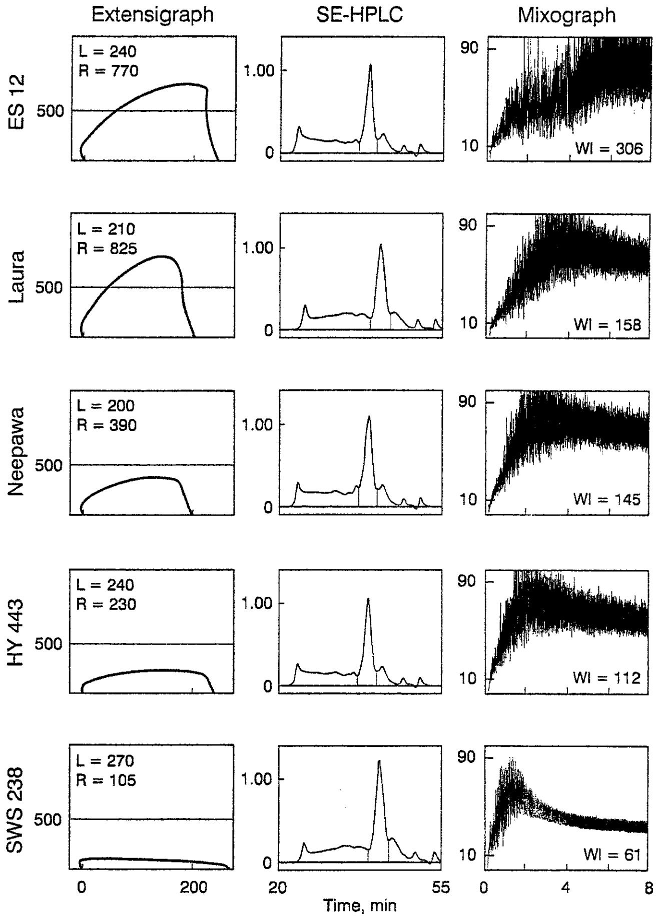

Fig. 2 Common wheat flour extensigrams, SE-HPLC profiles and 2g-mixograms. L = extensibility, mm; R = maximum curve height, BU; WI = work input to peak, arbitrary units

Using these criteria, the order of strength of the five durum cultivars (high to low) was (AC Pathfinder > AC Morse > AC Avonlea > Wascana > Stewart 63. These extensigraph rankings related directly to mixograph peak resistance and work input (Fig. 1). Similarly, for the common wheat cultivars, the order of strength was ES12 > Laura > Neepawa > HY 443 > SWS 238. Although empirical in nature, the extensigraphs do show large differences in the dough extensional properties for the same experimental conditions (sample geometry and timeframe of deformation).

A long fermentation straight dough (remix-to-peak) baking procedure was used to evaluate baking potential (Kilborn and Tipples 1981). After initial mixing, dough is subjected to a 160-min initial fermentation, followed by remixing in a recording dough mixer to 10% past peak consistency, sheeting molding, and panning, prior to final proof and baking. Loaf volumes are determined by rapeseed displacement. Baking strength index, a measure of loaf volume potential at a given protein level, is determined as described by Tipples and Kilborn (1974).

The breadmaking performance of the durum wheat cultivars, as given by bread loaf volume (Table 1), corresponded to the order of strength, with higher strength giving greater loaf volumes, although even the strongest durum had only 80% of the baking potential of a good bread wheat. Among the common wheat cultivars, ES 12, Laura, Neepawa, and HY 443 all gave good bread loaf volumes, and BSI values higher than for any durum, although BSI rankings were related to order of strength. The very weak soft wheat cultivar SWS 238 gave a low value for loaf volume (Table 1).

Size exclusion high performance liquid chromatography (SE-HPLC)

Wheat protein extraction and separation were performed as described by Nightingale et al. (1999). Relative proportions of the extracted major gluten protein fractions were measured by SE-HPLC. Chromatograms are shown in Figs. 1 and 2. The relative proportions of gliadins and glutenins in protein extracts were derived from areas under the SE-HPLC profiles. The protein content of the pellet remaining after centrifugation following protein extraction was determined by Combustion Nitrogen Analysis. The unextractable protein was considered to represent very high molecular weight glutenin polymeric protein (Weegels et al. 1996), and was included in the glutenin portion for calculation of the ratios of gliadin to glutenin (Tables 1 and 2). However, it may be that this glutenin fraction represents a tight protein network of some sort as opposed to HMW linear protein molecules that can be removed by extraction. The presence of specific HMW glutenin subunits in bread wheats affects the relative proportion of unextractable protein, with those considered to confer superior physical dough properties and baking characteristics making the polymer more insoluble (Hargreaves et al. 1996).

Creep experiments

Doughs were prepared for creep testing by mixing semolina or flour and water to 1 min past peak in the 2 g mixograph (Table 1). Dough water absorption was adjusted to approximately 5% below farinograph water absorption. Accordingly, durum wheat semolina and SWS flour were prepared at 50% water absorption (on a 14% flour moisture basis) resulting in dough with 42.7% moisture content. The other common wheat flours were prepared at 60% absorption, giving a final dough moisture content of 46.3%. Final protein content of each prepared dough is presented in Table 1.

Each dough was subject to creep for 10,000 s using a Rheometrics SR500 controlled stress rheometer (Rheometrics Scientific, Piscataway, NJ) in shear. The rheometer was fitted with 25-mm serrated parallel plates and was gapped to 2.75 mm. Excess dough was carefully trimmed and the exposed edges coated with silicon grease to prevent surface drying of the sample. In addition, the rheometer was equipped with a humidity chamber to prevent further dough drying. Samples were rested for an additional 15 min after loading prior to testing. All experiments were conducted at 25 °C at or below the critical stress for each cultivar, which ranged from 10 Pa to 50 Pa and was based on the agreement of creep compliance curves obtained at different stresses.

Creep tests were obtained on three separately mixed doughs for each cultivar. The time of testing combined with the number of replicates made recovery experiments impractical. However, this may not be a serious concern for this study as results are primarily comparative in nature. We are interested in determining trends in the LVP with the known strengths of the doughs rather than attempting to predict their recovery behavior from creep data.

Analysis of creep curves

This length of creep time was sufficient to reach a state of constant shear rate for the last several thousand seconds of the test. All of the creep curves showed a similar general form, an apparent instantaneous strain followed by a retarded elastic strain and finally steady state creep. Since all of the creep results were obtained in the linear regime, results are presented as the creep compliance J(t). The compliances were modeled (or curve fitted) using a Maxwell body in series with two Kelvin-Voigt bodies (Burgers model) giving a total of six parameters as shown in Eq. (1) below:

$$J(t) = 1/G_1 + 1/G_2[1 - \exp(-tG_2/\eta_2)] + 1/G_3[1 - \exp(-tG_3/\eta_3)] + t/\eta_1 \quad (1)$$

For convenience, $1/G_1$ actually represents a "short time" compliance (J_1) which lumps together all retardation processes up to 10 s as instantaneous relative to the 10,000 s creep time, $1/G_2[1 - \exp(-tG_2/\eta_2)] + 1/G_3[1 - \exp(-tG_3/\eta_3)]$ will be referred to as retarded elastic compliance (J_2), and t/η_1 will be referred to as viscous flow (J_3), all relative to the creep time of 10,000 s. Dus and Kokini (1990) indicated that the viscosity of a 40% moisture wheat flour dough tended to a zero shear viscosity for shear rates less than 10^{-5} s^{-1}. Such low steady state shear rates were achieved here.

Model parameters (mean values and S.D.) are shown in Table 3 for the durum doughs and Table 4 for the common wheat doughs. In addition to the rheological model parameters, the retardation spectrum was also obtained for each dough using a linear least squares analysis software package provided with the rheometer (RSI Orchestrator). Although empirical in nature, interpretation of the Burgers model parameters and the retardation spectra, in the context of physical gels, provides a powerful heuristic framework for understanding the relationships between macromolecular structures, dough strength and end product quality.

Results

Figure 3a, b shows representative results for the total compliance and the elastic component of the total compliance for the weakest and strongest durum and

Table 3 Six parameter Burgers model representing creep compliance data (10,000 s) for durum wheat semolina. Evaluation performed on average curve from triplicate measurements in the linear viscoelastic range. Mean values ± standard deviation

Parameters	Stewart 63	Wascana	AC Avonlea	AC Morse	AC Pathfinder
J_1 ($1/G_1$; 1/Pa)	$(5.66 \pm 0.10) \times 10^{-4}$	$(4.28 \pm 0.15) \times 10^{-4}$	$(3.20 \pm 0.24) \times 10^{-4}$	$(2.74 \pm 0.09) \times 10^{-4}$	$(2.05 \pm 0.15) \times 10^{-4}$
η_1 (Pa s)	$(0.51 \pm 0.01) \times 10^{7}$	$(0.86 \pm 0.01) \times 10^{7}$	$(0.90 \pm 0.05) \times 10^{7}$	$(1.18 \pm 0.04) \times 10^{7}$	$(1.98 \pm 0.11) \times 10^{7}$
G_2 (Pa)	$(1.12 \pm 0.06) \times 10^{3}$	$(1.77 \pm 0.17) \times 10^{3}$	$(2.70 \pm 0.34) \times 10^{3}$	$(3.05 \pm 0.32) \times 10^{3}$	$(5.40 \pm 1.06) \times 10^{3}$
η_2 (Pa s)	$(0.32 \pm 0.02) \times 10^{6}$	$(0.47 \pm 0.04) \times 10^{6}$	$(0.72 \pm 0.08) \times 10^{6}$	$(0.83 \pm 0.11) \times 10^{6}$	$(1.35 \pm 0.29) \times 10^{6}$
G_3 (Pa)	$(0.37 \pm 0.01) \times 10^{3}$	$(0.66 \pm 0.05) \times 10^{3}$	$(1.01 \pm 0.07) \times 10^{3}$	$(1.08 \pm 0.06) \times 10^{3}$	$(1.89 \pm 0.12) \times 10^{3}$
η_3 (Pa s)	$(0.82 \pm 0.01) \times 10^{6}$	$(1.42 \pm 0.11) \times 10^{6}$	$(2.21 \pm 0.17) \times 10^{6}$	$(2.44 \pm 0.19) \times 10^{6}$	$(4.19 \pm 0.40) \times 10^{6}$
τ_2 (η_2/G_2; s)	284 ± 4	265 ± 5	266 ± 10	273 ± 13	250 ± 5
τ_3 (η_3/G_3; s)	2207 ± 46	2175 ± 91	2179 ± 64	2267 ± 143	2213 ± 73

Table 4 Six parameter Burgers model representing creep compliance data (10,000 s) for common wheat flour. Evaluation performed on average curve from triplicate measurements in the linear viscoelastic range. Mean values ± standard deviation

Parameters	SWS 238	HY 443	Neepawa	Laura	ES 12
J_1 ($1/G_1$; 1/Pa)	$(3.50 \pm 0.37) \times 10^{-4}$	$(4.50 \pm 0.14) \times 10^{-4}$	$(4.57 \pm 0.21) \times 10^{-4}$	$(4.51 \pm 0.57) \times 10^{-4}$	$(2.83 \pm 0.14) \times 10^{-4}$
η_1 (Pa s)	$(6.61 \pm 0.68) \times 10^{6}$	$(3.96 \pm 0.19) \times 10^{6}$	$(3.54 \pm 0.08) \times 10^{6}$	$(3.62 \pm 0.49) \times 10^{6}$	$(7.58 \pm 0.20) \times 10^{6}$
G_2 (Pa)	$(2.07 \pm 0.37) \times 10^{3}$	$(1.56 \pm 0.07) \times 10^{3}$	$(1.52 \pm 0.09) \times 10^{3}$	$(1.54 \pm 0.26) \times 10^{3}$	$(3.09 \pm 0.30) \times 10^{3}$
η_2 (Pa s)	$(6.17 \pm 1.44) \times 10^{5}$	$(4.97 \pm 0.22) \times 10^{5}$	$(4.59 \pm 0.14) \times 10^{5}$	$(4.34 \pm 0.79) \times 10^{5}$	$(8.63 \pm 0.76) \times 10^{5}$
G_3 (Pa)	$(5.91 \pm 0.77) \times 10^{2}$	$(4.06 \pm 0.18) \times 10^{2}$	$(3.26 \pm 0.02) \times 10^{2}$	$(4.21 \pm 0.44) \times 10^{2}$	$(7.75 \pm 0.29) \times 10^{2}$
η_3 (Pa s)	$(1.48 \pm 0.25) \times 10^{6}$	$(1.11 \pm 0.05) \times 10^{6}$	$(0.97 \pm 0.02) \times 10^{6}$	$(1.16 \pm 0.16) \times 10^{6}$	$(2.09 \pm 0.14) \times 10^{6}$
τ_2 (η_2/G_2; s)	298 ± 18	319 ± 1	303 ± 8	282 ± 6	280 ± 8
τ_3 (η_3/G_3; s)	2496 ± 153	2735 ± 68	2975 ± 81	2746 ± 130	2702 ± 94

common wheat doughs, respectively. The elastic compliance was obtained by subtracting the viscous flow component (J_3 of the model) from the experimental creep curve. The model total compliance curve is also plotted with the experimental curves for the durum wheats, and shows very good agreement except at the shortest times. The ratio of J_1 between AC Pathfinder and Stewart 63 was 0.36. Both cultivars then appeared to reach a steady state elastic compliance within the 10,000 s creep time with the ratio between AC Pathfinder's and Stewart's retarded elastic compliances being reduced to 0.20. The steady flow viscosity was nearly four times greater for the AC Pathfinder dough, even though the moisture contents and total protein contents of these two doughs were essentially identical (Table 1). However, the ratio of gliadin/glutenin was 0.6 for the AC Pathfinder, while it was 0.89 for Stewart 63. However, it is also clear from the SE-HPLC traces in Fig. 1 that the distribution of the glutenin fraction is different for the two cultivars. Both show a prominent peak in the HMW region, indicating that both cultivars contain at least some amount of the highest molecular weight component, but the stronger AC Pathfinder also shows higher amounts of the intermediate higher molecular weight material, while the Stewart 63 molecular weight distribution is skewed more towards the lower molecular weight intermediate materials. The significantly lower value of J_1 for the AC Pathfinder (2.05e−4) vs Stewart 63 (5.66e−4) can be reasonably interpreted as indicative of an increase in the number of initial elastically effective network chains terminated by either physical crosslinks or entanglements. This is consistent with the higher dynamic moduli reported for AC Pathfinder dough relative to Stewart 63 (Rao et al. 2000b). The other three durum cultivars of intermediate strength showed qualitatively similar creep behavior as the AC Pathfinder and Stewart 63 (data not shown). The retarded and total elastic compliances for all five durums are shown in Figs. 4a and 5a and will be discussed later together with the common wheat doughs.

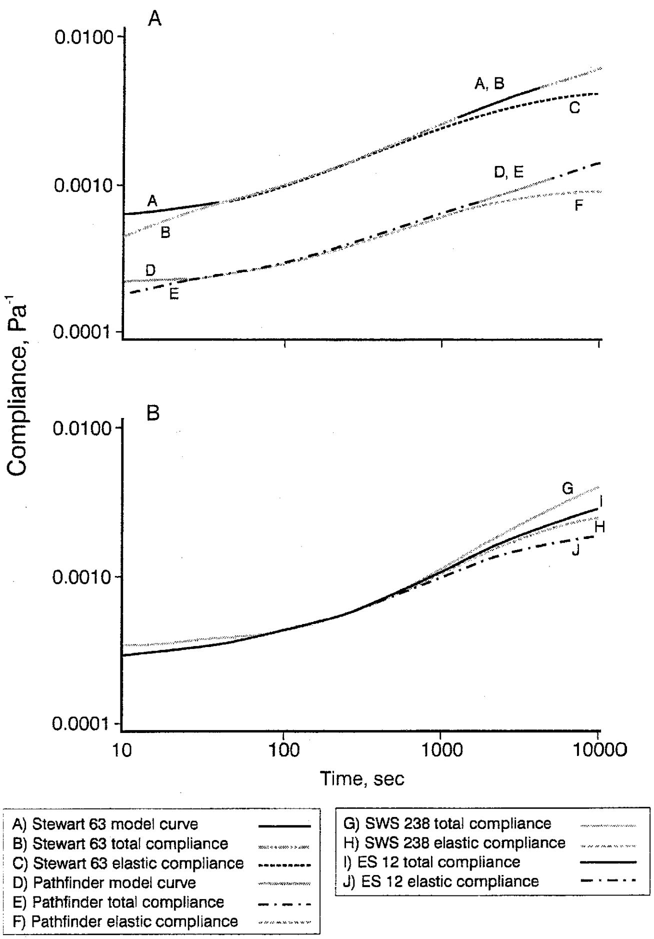

Fig. 3 **a** Total compliance and elastic compliance ($J(t)–t/\eta_1$) plotted for average of triplicate measurements, and model total compliance and elastic compliance from six parameter Burgers model, AC Pathfinder and Stewart 63. **b** Total compliance and elastic compliance ($J(t)–t/\eta_1$) plotted for average of triplicate measurements for ES 12 and SWS 238

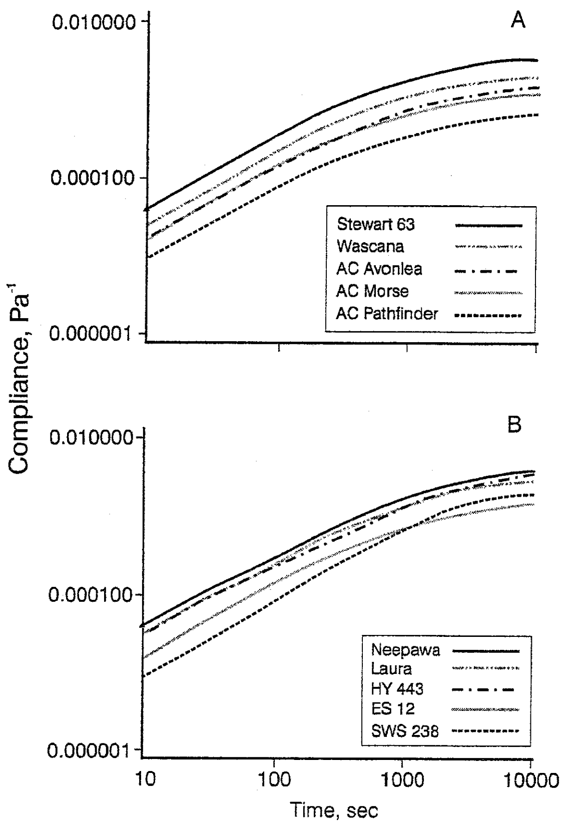

Fig. 4a, b Retarded elastic compliance $[J(t) - (J_1 + t/\eta_1)]$ calculated from model curves derived from six parameter Burgers model compliance curves (Tables 3 and 4): **a** durum wheat cultivars; **b** common wheat cultivars

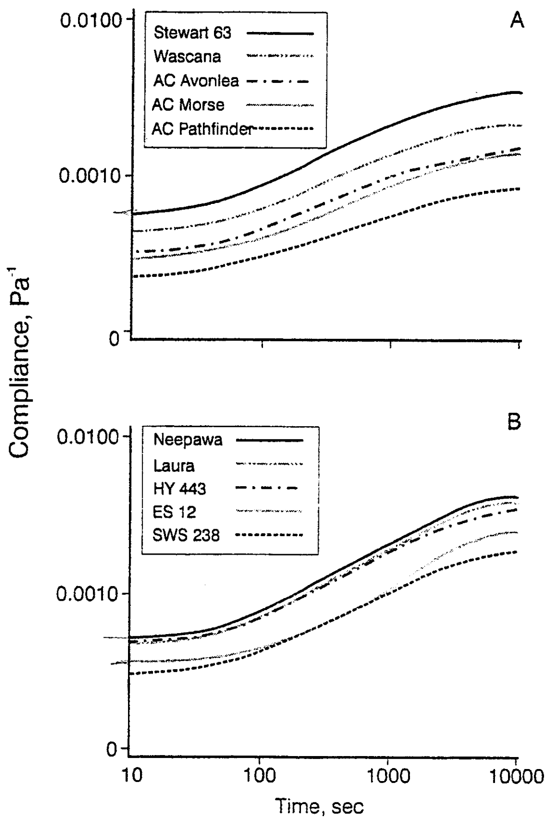

Fig. 5a, b Elastic compliance ($J(t) - t/\eta_1$) of individual wheat lines calculated from model compliance curves: **a** durum wheat semolina doughs; **b** common wheat flour doughs

There are both similarities and differences in the creep behavior of the doughs for the two common wheat cultivars of very different strength as compared to the durum doughs as shown in Fig. 3b. Like the durum doughs, both common wheat doughs also show elastic behavior at short times, but differ at longer times. This result is qualitatively similar to the work of Muller (1969), who found that the elastic compliances of glutens from soft and hard wheats were surprisingly similar, given their large difference in strength. The steady flow viscosities for the common wheat doughs of different strength were much more similar than was found for the durum wheats, their ratio being only 1.15 vs the factor of 3.38 found for AC Pathfinder and Stewart 63. The absolute values of viscosities were also lower for the common wheats than for the durum wheats. Also, the ES 12 dough showed a higher steady state compliance than the AC Pathfinder dough indicating a higher degree of configurational changes of the original network for the ES 12. Unlike the intermediate strength durums, the three intermediate strength common wheat doughs all showed quite similar total compliance curves (data not shown) as indicated by the model parameters in Table 4.

As compared to the two durums of different strength, the large difference in strength for the two common wheat doughs was only evident in their longer-time retardation behavior. This was reflected in their model τ_3 values; 2702 s for ES 12 vs 2496 s for SWS 238, while τ_3 was essentially identical for the two durums. Also, in general τ_3 was larger for all of the common wheat doughs relative to the durum doughs, regardless of strength. Overall, it is worth noting that short-time creep testing and/or high frequency dynamic testing of doughs, will apparently not reveal important differences in the long-range configurational and viscous deformations of doughs of different strength.

Elastic compliances

Given the supposed importance of elasticity in doughs, the elastic compliances were also examined separately. Figure 4a, b shows the retarded elastic compliances for the five durum and common wheat doughs, respectively. For the durums, it is apparent that all of the curves have a similar shape, consistent with their similar model retardation times. The trend was for higher elastic compliances throughout the 10,000 s creep time as the ratio of gliadin to glutenin increased at essentially constant dough protein content. This result combined with the decrease in viscosity as strength decreased suggests a lower degree of crosslinking as strength decreased. Also, the lower the compliance curve was, the higher was the extensigraph strength. This is particularly apparent for the AC Avonlea and AC Morse cultivars, which show only small differences in retarded elasticity over time and differ by only 20 BU in their extensigraph values. This suggests that not only do these two doughs have similar glutenin contents, but that their distribution of retardation times are also similar, which is consistent with their similar SE-HPLC traces (Fig. 1). Overall, these results suggest that under a small applied stress stronger durum doughs show less configurational rearrangement of the original network and less viscous flow, which is consistent with the idea that they are more crosslinked.

For the common wheat doughs, the SWS 238 sample has an obviously different sort of character (distribution of retardation times) than the other common wheats. This could be due to its lower moisture and protein contents relative to the other common wheat doughs, combined with its relatively high proportion of glutenin (Table 2). The lower moisture content could explain its stiffness at short times. Looking at the other four common wheat doughs, they show a qualitatively similar behavior, although the compliance curve for the ES cultivar is clearly shifted to lower values than the other three cultivars. This could be explained by its lower gliadin to glutenin ratio (0.53), relative to Laura, Neepawa and HY 443, which were higher and very similar to each other. In fact, their J_1 values are identical (Table 1), and the other model parameters are also close to each other. Unlike for the durum doughs of different strength, the three MS common wheat doughs show very different extensigraph strengths for similar retarded elastic compliance curves and similar ratios of gliadin to glutenin.

Figure 5a, b shows the time course of the total elastic compliances (J_1 and J_2) for the durum and common wheat doughs, respectively. It is apparent that the strong and moderately strong durum doughs all show flatter trajectories to their steady state elastic compliance values than the extra strong and moderately strong common wheats. For example, after 1000 s of creep the Neepawa, Laura, and HY 443 common wheat doughs all have compliances well above 0.001 Pa^{-1}, while the extra strong ES12 is just about at 0.001 Pa^{-1}. For the durum doughs, the moderately strong AC Avonlea and AC Morse are just approaching 0.001 Pa^{-1}, while the strong AC Pathfinder dough is well below this value. The weak Wascana and very weak Stewart 63 durum doughs are somewhat similar to the moderately strong bread wheat doughs, although their extensigraph strengths and bread loaf volumes are significantly lower than for the bread wheat doughs.

Retardation spectra

Subtle differences in the distribution of retardation times are easier to see in the retardation spectra than in the parameters of the mechanical model. The retardation spectra represent a continuous distribution of retardation times and their intensity in a particular timeframe, while the mechanical model gives only average retardation times over a larger time frame. Retardation spectra for all of the doughs are shown in Fig. 6a, b on the same scale for the durums and common wheats, respectively. Although the curves are not very smooth, they do illustrate that the retardation spectra are composed of two distinct processes widely separated in time. This is consistent with the well-known two-stage stress relaxation behavior exhibited by both doughs and gluten (Bohlin and Carlson 1981). Differences between the durum doughs and common wheat doughs are clearly most evident for the longer retardation times. For the common wheat doughs (excepting SWS 238), higher extensigraph strengths are associated with higher retardation strengths for the maximum in the retardation spectrum, which also leads to higher viscosities as indicated in Table 4. Surprisingly, just the opposite trend is found for the durum doughs. Stronger durum doughs show lower retardation strengths for the longest retardation times, but also higher viscosities. This anomalous effect for the durum doughs could be due

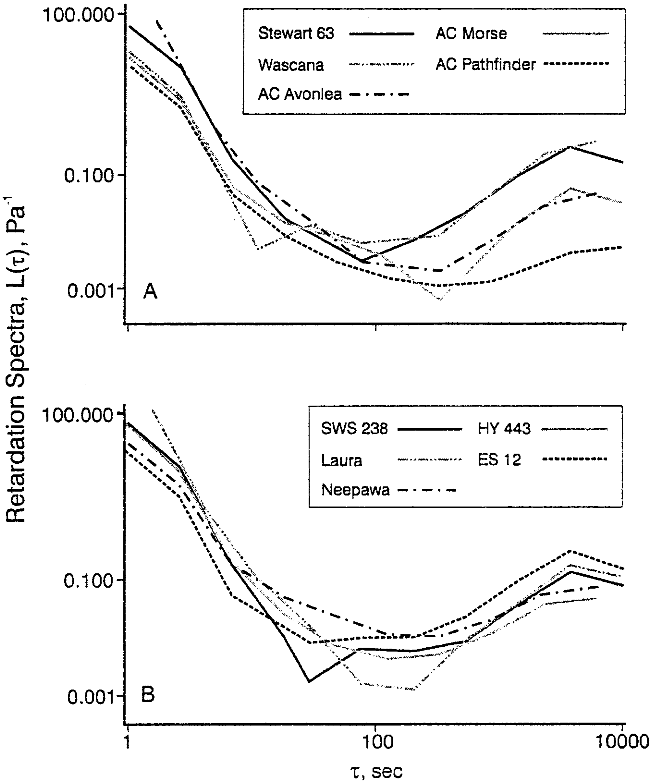

Fig. 6a, b Retardation spectra, L(τ), for individual wheat lines calculated by least squares analysis of compliance data collected over 10,000 s: **a** durum wheat semolina doughs; **b** common wheat flour dough

to the stronger influence of physical crosslinks on their LVP (higher steady state viscosities for lower molecular weights) as discussed by Tanaka and Edwards (1992b) for physical gels in the unentangled regime. The long time viscoelastic properties of common wheat doughs appear to be more similar to physical gels containing both entanglements and crosslinks, but where the overall dynamics are determined more by the entanglements.

Discussion

It should be appreciated that an unequivocal connection between rheological measurements and underlying molecular structures cannot be made for these gluten doughs. The chemistry of the gluten complex within a dough is simply too complex. Also, these doughs vary in concentration of protein and polymeric protein (glutenin), as well as molecular weight distribution within the polymeric protein fraction. However, these results have shown that the LVP are sensitive to apparent differences in these underlying macromolecular structures, which in turn appear to be related to the concept of dough strength. The higher overall viscosities of the durum doughs, the greater sensitivity of the viscosity to the extent of plasticization by gliadin and their inextensibility relative to the common wheat doughs suggests strength in the durum doughs is related to the number of physical crosslinks in the glutenin fraction. This is consistent with the concepts of physical gels where a high concentration of lower molecular weight chains with functional crosslinking groups can show unusually high viscosities. However, those concepts are limited to the LVP. The main purpose of this work was to integrate these LVP with the large strain results (Figs. 1 and 2) and the chemical and composition data (Tables 1 and 2) to understand better the relationships between the macromolecular structures in doughs, their large strain strength, and end product quality.

Gluten doughs as multimodal networks

If one were to assume that the numerous physical crosslinks in gluten were permanent rather than physical in nature, then there would be an obvious analogy between gluten and polydimethylsiloxane (PDMS) bimodal networks. In short, bimodal PDMS networks containing very short chains end linked with relatively long chains have unusually large ultimate strengths considering their large extensibilities (Wang and Mark 1992). As described in the introduction and in many cereal science textbooks, the strength of various wheat cultivars has been linked to their glutens, particularly their HMW glutenin fraction, but not necessarily to their distribution of chain lengths within this fraction. Also, focusing only on the highest molecular weight glutenin fraction neglects the contributions shorter polypeptides can make to strength via their crosslinks, without necessarily decreasing extensibility.

There has also been discussion of the molecular nature of this phenomenon in the literature. Andrady et al. (1980) refer to the fact that fewer short chains in a bimodal network permits a more extensive reapportioning of the strain within the network, with a corresponding increase in extensibility. Smith et al. (1990) showed that the tensile strength of bimodal PDMS as determined at an extension rate of about 0.3 min^{-1} showed a maximum at about 30–40 wt% short chains. These authors explain the relatively high elongations of these crosslinked networks as being due to the nonaffine nature of the deformation, that is the longer chains in the connected network deform proportionately more than the short chains. However, these long chains must also move through the matrix of shorter chains as they are extended, thus resulting in substantial viscous dissipation (and higher strength) than for the long chains alone. Thus, the higher extensibilities of strong bread wheat doughs generally could be due to a lower proportion of shorter, crosslinked polypeptides.

For the common wheat doughs, there were large variations in extensigraph strength even though the retardation spectra for the doughs were fairly similar, relative to the durum doughs. Differences in strength for these doughs could be due to fundamentally different distributions of polypeptide chains (and their interactions) than for the durums. It is possible that strength in bread wheat doughs is more related to differences in entanglement networks as suggested by the stress relaxation results of Rao et al. (2000a), and that these differences become more evident in the extensigraph which presumably has a much higher effective rate of strain than the creep tests. In the shorter experimental time frame of the extensigraph it may not be possible to distinguish the contributions of entanglements and physical crosslinks to dough strength, in the same way as both may contribute to the high frequency dynamic elastic moduli. However, differences in macromolecular structures between the bread wheats and durum doughs do become evident at longer creep times.

The basic idea of the bimodal networks as expressed by Smith et al. (1990) is that the high tensile strengths are probably due to the low mobility of the chains resulting in high dissipation of energy, which delays rupture. This general concept can be tested by looking at some results for the rate of stress relaxation for some of these same doughs.

Stress relaxation

The stress relaxation behavior of some of these same doughs has previously been determined (Rao et al. 2000a, 2000b). The relaxation times should be an indication of their relative rates of molecular motions in dissipating stress. For convenience the initial modulus (Pa) in stress relaxation (G_o) and the time in seconds to relax 75% of the initial stress (t_{75}) will be used for comparison. The values for G_o and t_{75} (G_o; t_{75}) for the durums were (12,750; 50.1), (6753; 20.0), (6507; 20.0), (6587; 11.2), and (6087; 8.4) for the AC Pathfinder, AC Morse, AC Avonlea, Wascana, and Stewart 63, respectively. There is a clear trend for the extensigraph strength to be related more to the relaxation times than for the initial modulus in stress relaxation. This would tend to support the idea expressed here that strength of the doughs is somehow related to their relative molecular mobilities. It is also interesting to note that the AC Avonlea and AC Morse showed essentially identical stress relaxation responses, just as they did here in creep and the extensigraph.

Similar stress relaxation data is available for only three of the common wheat doughs. Values of G_o and t_{75} were (13,040; 50.1), (5658; 13.4), and (5213; 5.0), for ES 12, Neepawa, and HY 443, respectively. It is quite clear that the extensigraph strength of the common wheat doughs also appears to be related to their relative rates of relaxation. In particular, the strength of the Neepawa (390 BU) was significantly higher than for the HY 443 (230 BU), and this may be reflected in the significantly greater value of t_{75} for the Neepawa dough. In addition, the relaxation times were identical for the AC Pathfinder durum dough as for the ES 12 bread wheat dough and their extensigraph strengths were also very close, 750 BU and 770 BU, respectively. However, the ES 12 dough was more extensible and showed a higher loaf volume. Neepawa and HY 443 doughs showed lower relaxation times (13.4 s and 5.0 s, respectively) than durum doughs of comparable strength and ratio of gliadin to glutenin (20 s for AC Avonlea and 8.4 s for Wascana, respectively), and also had higher loaf volumes. As discussed above, the three bread wheat doughs also showed higher creep compliances than for durum doughs of similar strength. The trend for higher relaxation times for the durum doughs of comparable strength coupled with their known lack of the D-genome coding for high molecular weight polypeptides further supports the view that the material properties of durum doughs are influenced more by physical crosslinks than the common wheat doughs.

Conclusions

A unique aspect of this work is the interpretation of the creep behavior of doughs of different strength in the context of physical gels containing physical crosslinks and/or entanglements. The overall higher steady state viscosities of the durum doughs and their relative inextensibility suggests that strength in the durum doughs is primarily a function of the density of physical crosslinks present. This is consistent with their lower creep compliances relative to common wheat doughs of similar strength, but higher steady state viscosities. The increase in retardation strength at long times and steady state viscosity for stronger common wheat doughs suggests a greater role of the larger polypeptides in determining strength of bread wheat doughs. It is pointed out that it may be difficult to distinguish between physical crosslinks and entanglements at short time in creep or dynamic tests, but distinct differences between the durum and bread wheat doughs were observed at longer times in creep and stress relaxation. This could help explain the observation of Ammar et al. (2000) that part of the large strain deformation energy for durum doughs did not contribute to improving baking performance. A similar result was found here where durum doughs gave lower bread loaf volumes than for bread wheat doughs with similar extensigraph strength.

By analogy to published literature regarding bimodal PDMS networks, the results presented and discussed here for durum and common wheat doughs suggest that, in either case, the strength of these doughs was fundamentally related to their underlying molecular mobilities. This is particularly evident in their respective rates of stress relaxation, where slower relaxation processes were associated with higher strength for both durum and common wheat doughs. However, the experimental relaxation times most likely represent different molecular processes in durum and bread wheat doughs.

The idea presented here that the relative balance between strength and extensibility in wheat flour doughs depends upon the distribution of short chains to long chains in their crosslinked and/or entanglement network structures needs to be tested further. Along the lines of the PDMS networks, closer examination of the molecular weight distribution and extent of crosslinking in the glutenin fraction is needed. It would also be helpful if an estimate of the average lifetimes of the various physical crosslinks in doughs could be determined.

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