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Elastic properties of gluten representing different wheat classes

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ABSTRACT

Traditional instruments used to evaluate dough and/or gluten rheological properties do not provide unambiguous separation of elastic and viscous behaviors. Recovery after shear creep and cyclic large deformation cyclic tensile testing were used here to decouple elastic and viscous effects. A large variation in the recoverable shear strain (∼7.2% to ∼28%) was seen for glutens from 15 U.S. popular common wheat cultivars with varying HMW subunits. Sedimentation values ranged from 29 to 57 ml for 12 hard wheat cultivars and 15 to 22 ml for three soft wheat cultivars. The tensile force at 500% extension ranged from 0.12 to 0.67 N for hard wheat glutens and from 0.10 to 0.20 for soft wheat glutens. However, the recoverable work after large extension was less than 40% of the total work of extension. In addition, recoverable work in tensile testing was highly correlated with the total work of extension ($r^2 = 0.97$) and mixograph mix times ($r^2 = 0.81$). Good to excellent bread volume was obtained for several cultivars from this sample set. This suggests that optimizing water absorption for mixing doughs to achieve maximal bread volume compensates for the wide range of viscoelastic behaviors of gluten.

1. Introduction

It is generally accepted that what makes wheat unique among the cereals is the viscoelasticity of the hydrated gluten proteins. Schofield and Scott Blair (1937) were the first to obtain stress–strain curves for wheat dough and gluten washed from the same dough, and based on the resemblance between the two sets of data deduced that the elasticity of wheat dough was due to its gluten. It is worth noting that those original rheological tests on dough and gluten involved extension to large strains (5 or 6 times the original length), but to lengths less than the breaking point, followed by immediate retraction at the same rate. Thus, elastic and plastic (viscous) effects could be unambiguously separated.

The key point is that separation of reversible elastic recovery from plastic flow requires a cyclic test, for example, some kind of deformation followed by a free recovery. Small amplitude oscillatory testing is also a form of cyclic testing, but has been used mostly in combination with time–temperature superposition to characterize the linear viscoelasticity of bulk linear, amorphous polymers through the glass transition to the rubbery plateau and then to the polymer melt state. The fact that gluten shows a simultaneous elastic and plastic deformation at ambient temperatures indicates that gluten lies in the rubbery flow region of viscoelasticity and shows similarities to both uncrosslinked (raw) natural rubber and thermoplastic polymer melts. It was reasoned here that the "dual nature" of gluten, it being more extensible than raw latex rubber, but more elastic than a polymer melt, means that more than one type of rheological test would be needed to characterize its unusual type of viscoelasticity.

However, there is surprisingly little viscoelastic property data in the literature available for large sample sets of gluten obtained from well-characterized wheat cultivars that would help to answer that question. Many of the published studies on rheological properties of gluten that have appeared since the early work in the 1930s involved only a single (or a few) gluten sample(s), or were done before the time that the HMW-GS and/or polymeric gluten proteins became a main focus of biochemical wheat quality. Dobraszczyk and Morgenstern (2003) have reviewed that early literature. Shewry et al. (2000) noted that the "visco-elastic potential" of

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individual subunits is different, and that those differences are expressed through the properties of glutenin polymers and aggregates. However, the actual viscoelasticity of gluten itself will also involve interplay between gliadin and glutenin structures, perhaps down to the subunit level. Wet gluten can be obtained easily and rapidly directly from flour using a gluten washing machine such as the Glutomatic (Perten AB, Huddinge, Sweden); so a rapid test of gluten quality based on direct measurement of its viscoelasticity may be useful in breeding programs, import/export programs, and maintaining consistency of flour during milling, or as a guide to blending flours to achieve consistent gluten quality.

Although both creep-recovery and stress relaxation experiments have been used to characterize the viscoelasticity of dough and gluten since the 1930s, more recent work has emphasized creep-recovery. Schober et al. (2002) used creep-recovery (5 min creep followed by 5 min recovery) as one index of gluten quality for 25 spelt cultivars. They made the point that fundamental rheological tests are not used routinely to characterize large wheat cultivar sample sets. This may have to do with the apparently very long experimental times needed to achieve steady-state flow conditions for creep of doughs in the linear viscoelastic regime ($\sim$5000 s at 10–50 Pa applied stress; Edwards et al., 2001), and/or equilibrium recovery conditions for gluten after reaching steady flow in creep ($\sim 10^5$ s; Lefebvre et al., 2003) at around room temperature. Wang and Sun (2002) compared the recoverable strain of 23 wheat doughs prepared from flours representing wheat with a wide range of protein content, physical dough properties, and baked loaf volume. They found a high correlation between recoverable strain of dough prepared at fixed water absorption of 54% and loaf volume ($r = 0.939$), which was interesting since the high stress used resulted in predominately viscous deformation during creep. There was no correlation between maximum total creep strain and baking volume. However, Janssen et al. (1996) clearly showed that the total creep strain was much greater for gluten obtained from a poor breadmaking cultivar than for one obtained from a good breadmaking cultivar. Bockstaele et al. (2008) also looked at the relationship between creep-recovery and bread volume for seventeen European wheat cultivars. One of their results was that a few HMW 5 + 10 cultivars gave a high Glu-1 score, but low bread loaf volume. This indicates that rheological measurements on dough or gluten are needed to fully characterize a wheat cultivar.

The main point of this experimental work then was to determine whether wheat class was a good indicator of relative gluten strength. A second objective was to determine the range of gluten strength for glutens representing U.S. wheat classes with known differences in their biochemical properties. Such viscoelastic property data would complement the extensive literature on biochemical aspects of wheat quality.

2. Materials and methods

2.1. Materials

Fifteen wheat cultivars were obtained from certified seed representing five U.S. wheat classes harvested in 2005: Hard Red Winter (HRW), Hard Red Spring (HRS), Soft Red Winter (SRW), Hard White (HDWH), and Soft White (SWH). They were milled into flours using A Buhler Mill model MLU-202 following Approved Method 26-21A (AACCI, 2000) for experimental test milling. Different sieves were used for hard and soft wheats. The cultivars were characterized in terms of their flour protein, high molecular weight glutenin subunit (HMW-GS) composition, sedimentation volume, wet gluten content, gluten index (GI) and bread loaf volume (Table 1). A main interest was how well the rheological properties determined here correlated with bread loaf volume.

2.2. Protein content and composition

During the extraction of protein fractions, all the steps of protein precipitation with acetone (40 and 80%) at −20 °C were extended from 24 to 48 h. The total protein on each fraction was determined using approved methods 46-30 (AACCI, 2000) based on Dumas's nitrogen combustion in a LECO FP-528 nitrogen analyzer (LECO Corporation, St Joseph, MI). EDTA was used as standard and the protein to N ratio was 5.7.

Table 1
Physicochemical properties, high molecular weight glutenin subunit (HMW-GS) composition and bread loaf volume (BLV) of 15 U.S. cultivars.

Cultivar	Flour protein (%)	HMW-GS	Wet gluten (%)	Gluten index	Gluten MC[a] (%)	SED[b] (ml)	BLV[c] (cc)
HRW							
TAM 110	13.8	2*, 7 + 8, 2 + 12	39.7 ± 1.1	81.9 ± 3.8	66.4 ± 0.3	55	919
Jagger	11.0	1, 17 + 18, 5 + 10	27.6 ± 1.6	99.6 ± 0.0	63.9 ± 0.7	36	831
Jagalene	10.0	1/2*, 17 + 18, 5 + 10	24.3 ± 1.5	98.5 ± 1.5	65.6 ± 0.7	36	769
HRS							
Alsen	15.7	2*, 7 + 9, 5 + 10	39.9 ± 0.8	95.6 ± 1.5	64.5 ± 0.6	57	919
Briggs	13.4	1/2*, 7 + 9/17 + 18/, 5 + 10	34.5 ± 1.3	93.1 ± 1.4	64.7 ± 0.1	43	825
McNeal	14.1	1, 17 + 18/7, 5 + 10	33.8 ± 1.3	99.4 ± 0.0	63.9 ± 0.1	56	956
Reeder	12.9	2*, 7 + 9, 5 + 10	36.7 ± 0.9	88.4 ± 2.6	65.2 ± 1.1	52	856
Hollis	12.9	2*, 17 + 18, 5 + 10	32.6 ± 1.1	96.3 ± 0.7	65.3 ± 0.1	50	881
Norpro	11.8	2*, 7 + 9, 5 + 10	31.5 ± 1.0	88.6 ± 1.0	66.0 ± 0.7	36	788
HDWH							
Blanca Grande	12.9	1, 17 + 18, 5 + 10	36.3 ± 0.8	93.4 ± 0.6	65.5 ± 0.8	49	913
Trego	10.4	2*, 7 + 9/20x + 20y, 5 + 10	26.8 ± 1.1	97.6 ± 1.2	64.5 ± 0.2	38	744
SRW							
Patterson	8.5	1, 7,5 + 10	24.8 ± 1.0	65.1 ± 7.2	68.8 ± 0.4	15	738
Roane	7.7	Null, 7 + 8, 2 + 12	20.7 ± 1.2	92.8 ± 3.7	68.3 ± 1.2	17	688
SWH							
Stephens	11.4	Null, 7 + 9, 2 + 12	35.5 ± 1.1	42.7 ± 0.3	66.0 ± 0.0	22	675
Eltan	11.1	1, 7, 5 + 10	31.5 ± 1.1	81.5 ± 2.3	67.7 ± 0.4	29	863

[a] Gluten moisture content.
[b] SED, sedimentation.
[c] BLV, bread loaf volume.

2.3. HMW-GS composition

The allelic variation of HMW-GS was determined in one dimensional sodium dodecyl polyacrylamide gel according to the method described by Pfluger et al. (2001) with the following modifications: gliadins were not extracted and a resolving gel of 12% acrylamide was used. HMW-GS alleles were identified using cultivars 2174, Briggs and Intrada as standard cultivars and the method of Payne and Lawrence (1983) and Shan et al. (2007).

2.4. Sedimentation test

Using approved method 56-61A hand mixing procedure (AACCI, 2000), the volume (in ml) of sediment in a cylinder after mixing lactic acid into a flour suspension for 5 min was read as the sedimentation value. From a bread baking standpoint, quality and quantity (strength) of gluten in wheat flour can be reflected by this test. Analysis was done in duplicate.

2.5. Bread loaf volume

Pup loaves were baked using Approved Method 10-10B (AACCI, 2000). Fermentation time was 180 min; proof time 55 min and loaves were baked at 425 °F for 15 min. Mixing times varied from short (<4 min) to normal (4.5–6 min) to slightly long (>6.5 min). Absorptions were normal (62.5–65%) except for McNeal, which had an absorption of 67%. Loaf volume (see Table 1) ranged from poor (650 cc) to good (700–800 cc) to excellent (>800 cc) (Table 1).

2.6. Gluten recovery and preparation of samples for rheometry

Flour (10 g) was loaded into each of two washing chambers on a Glutomatic 2202 gluten washer (Perten Instruments AB, Huddinge, Sweden) and washed with 2% salt solution according to Approved Method 38-12 (AACCI, 2000). Each gluten sample at the end of washing was weighed to obtain the wet gluten content. Samples were centrifuged and weighed again to get the gluten index. The gluten remaining on the centrifuge sieves was combined into a single sample for rheometry. The two samples were kneaded together by hand for 50 s until the color was a uniform light tan. The combined sample was formed into a ball and then pressed between two steel plates, which were lubricated with a small amount of spray vegetable oil and had a gap of 2.5 mm. The sample was allowed to rest between these plates for 1 h. After resting, a 25 mm diameter disc was cut from the pressed sample for creep-recovery experiments. The lubricating oil aided in the transfer of the cut gluten sample from the pressing plates onto the rheometer. Gluten is soft and tacky/sticky and cannot be easily handled by hand without destroying its shape. The gluten sample was carefully scraped off the bottom pressing plate with a sharp paint scraper, and then gently transferred to the rheometer base plate with the help of a small metal scalpel. The same procedure was used to obtain samples for the tensile testing, except the sample was cut into a different shape. Enough gluten was recovered from the hard wheat flours using the above procedure to obtain samples both for rheometry and large deformation tests from a single Glutomatic washing. However, two Glutomatic washings were needed for each replicate for the soft wheat flours, due to the large amount of gluten that passed through the sieves during centrifugation, and hard wheat flours with low protein contents to obtain samples for creep-recovery and tensile testing. Thus, rheological testing was performed only on gluten retained on the Glutomatic sieve after centrifugation. It is not known how this fraction may differ as compared to the pass through gluten fraction.

A "window-pane" technique was used to load the sample for tensile testing into a vertical tensile grip attachment. Again, the goal was not to touch the delicate gluten sample directly with one's fingers when transferring to the vertically oriented tensile grips. The sample tabs at each end were 175 mm × 62 mm. The sample was tapered to a width of 10 mm for a length of 12.7 mm. The thickness was 2.5 mm. Velcro dots were attached to both ends of a piece of square construction paperboard with a window cut from the center of the piece of paperboard. The piece of gluten was picked up, then simply contacted by both pieces of Velcro and thus became attached to the stiff paperboard. The whole assembly was then easily slid into the tensile grips, the edges of the cardboard were cut with scissors, and then the test was run.

2.7. Creep-recovery tests

A controlled stress rheometer (TA Instruments AR1000, New Castle, DE) equipped with parallel plate configuration (25 mm diameter crosshatched upper plate and 2.5 mm gap) was used. Before loading the gluten sample, a thin layer of super glue (QuickTite, Loctite North America, Rocky Hill, CT) was applied to prevent slippage between the sample and the bottom plate. The top plate was then lowered to just contact the sample. Any small excess sample was trimmed from the edges. In addition, a plastic housing surrounded the sample, and humidified air was continually pumped through this sample chamber. The sample edge was coated with mineral oil (Mineral Oil U.S.P., Rite Aid Corporation, Harrisburg, PA) to help prevent sample drying during the test. Temperature was controlled at 25 °C by the lower plate and the applied stress was 40 Pa for all glutens.

Based on preliminary results obtained for a rehydrated commercial gluten powder, the creep time was chosen as the time needed to obtain values of the recoverable deformation that did not change appreciably with longer creep times up to 10,000 s (Liang, 2006). The most noticeable effect of longer creep times was a lack of superposition between the short time creep and short time recovery curves, not an increase in the value of the recoverable compliance. Near equilibrium values of the recoverable strain of gluten were obtained for creep times much shorter than those needed to achieve steady-state viscous flow if the recovery time was chosen to be substantially longer than the preceding creep time. Thus, the creep time in these experiments was set to 100 s and the recovery time was 1000 s. These creep-recovery test conditions were used here to determine primarily the elastic recovery of gluten in the absence of substantial plastic flow as an objective measure of its elasticity. Each sample was re-tested a second time without removing from the rheometer to check for irreversible changes in the sample due to time in the rheometer and/or shear strain. No significant variation of results was seen for any of the samples at 25 °C. Duplicate samples were run for each creep-recovery, which consisted of separate Glutomatic washings for each replicate.

2.8. Extension testing

2.8.1. Cyclic large deformation testing – determination of maximum force, Degree of Elasticity (DE) and set

A texture analyzer (TA.XTPlus, Texture Technologies Corp., Scarsdale, NY/Stable Micro Systems, Godalming, Surrey, UK) with a 5 kg load cell and tensile grips was used. The rate of extension was 1 mm/s to a total strain (L/L_o) of 500% based on the crosshead movement. The rate of the return cycle was also 1 mm/s. In no case did any gluten samples break into two pieces in this testing mode. Tensile testing was at least duplicated, which consisted of separate Glutomatic washings for each replicate. Degree of Elasticity (DE) was determined by the ratio of recoverable work and total extension work. Set (permanent deformation) was defined as the percentage increase in sample length immediately measured after the first retraction cycle. In practice, the increase in sample length was taken as the elongation on retraction where the force first fell to zero. Visually, this point correlated with the first appearance of slack in the sample as it was retracted further to the initial point.

2.9. Statistical analysis

All cultivars were tested in at least duplicates using independent samples. One-way analysis of variance (ANOVA) with a significance level of $\alpha = 0.05$ was performed to compare mean values for the 15 cultivars to determine statistically significant differences for the parameters obtained from cyclic large deformation tests. All analyses were conducted by statistical software SPSS® Release 13 (SPSS, Chicago, IL, USA).

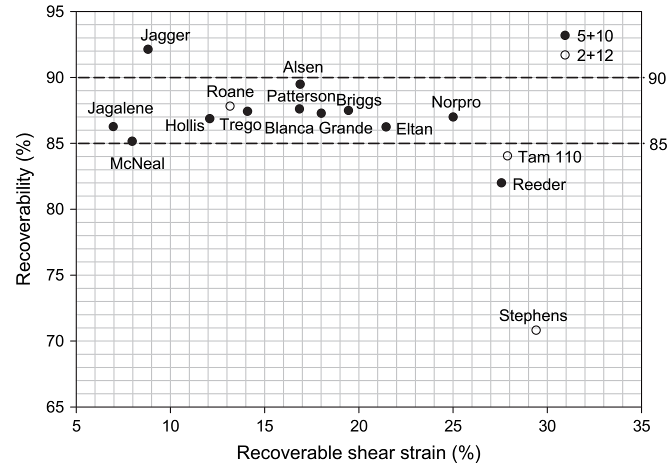

Fig. 2. Recoverability (recoverable shear strain/total shear strain × 100) versus recoverable shear strain (%) for 15 U.S. wheat cultivars.

3. Results and discussion

Recovery curves are shown in Fig. 1 for the 11 hard wheat glutens. Although all glutens showed a similar form of delayed elasticity in recovery, it was interesting to note the broad range of final recoverable shear strains shown by these hard wheat glutens. It is unlikely that differences in water content for these Glutomatic glutens alone, which varied only from ~63 to ~66%, could account for such a wide variation in the recoverable strain. Jagalene gluten showed the lowest recoverable strain (~7.0%), while Reeder and TAM 110 glutens showed the highest (~28% recoverable strain). TAM 110 is a 2 + 12 hard wheat cultivar, while Reeder is a 5 + 10 hard wheat cultivar. Thus, for this set of hard wheat cultivars, equally high recoverable strain was seen for both a 2 + 12 and a 5 + 10 cultivar, and thus Glu-D1 subunits alone cannot explain differences in these creep-recovery results. There was a clear separation point for the glutens between 1 and 10 s, where the recovery process became slower than the preceding creep process to reach the same value of the % strain. Prior to the separation point, the recovery and creep curves superposed, which is an indication of the width of the rubbery plateau in seconds. In this case, the rubbery plateau was very short and the glutens were mainly in the rubbery flow region of viscoelasticity. In this region, viscous flow is superposed on the recoverable orientation process.

Delayed elasticity is essentially a relaxation process, i.e., elastic structural domains "relax" into their equilibrium strain condition for the applied stress probably at least partly via a dynamic bond-breaking and re-forming process for gluten. Gluten is known to show essentially complete stress relaxation when held at small constant deformation for a long enough time, presumably due to the same bond-breaking and re-forming processes that are active in creep. However, the fact that all hard wheat glutens tested here showed as high as 80% recoverability after cessation of creep implies that some fraction of the initial bonds in the unstressed gluten remained intact during creep, and thus formed the basis of the restoring force. It has been widely accepted that the restoring force in gluten is at least partly entropic in nature with a return to lower free energy state (Belton, 1999).

Recovery curves for the four soft wheat glutens were surprisingly similar to those of the hard wheat glutens (data not shown), except the lowest value of the recoverable shear strain was about 13%, as compared to about 7.0% for the lowest hard wheat gluten. The lowest and the highest recoverable shear strain were shown by 2 + 12 soft wheat glutens, Roane and Stephens, respectively. The two intermediate recoverable shear strain soft wheat glutens (Eltan and Patterson) were 5 + 10 cultivars. Thus, no trend from low to high recoverable shear strain was seen for the Glu-D1 subunits for these soft wheat glutens. Moisture content of the soft wheat glutens ranged from 66 to 68.6%.

The sedimentation volumes for the soft wheat flours were 17 ml for Roane (although its GI was high, 92.8), 15 for Patterson, 22 for Stephens and 29 for Eltan. The GI also varied widely for the soft wheat glutens from 42.7 to 92.8 (Table 1). The sedimentation volumes for the hard wheats ranged from 38 to 57 (Table 1). The bread loaf volume ranged from 675 to 863 cc for the soft wheats and from 744 to 956 cc for the hard wheats. Eltan, a soft white

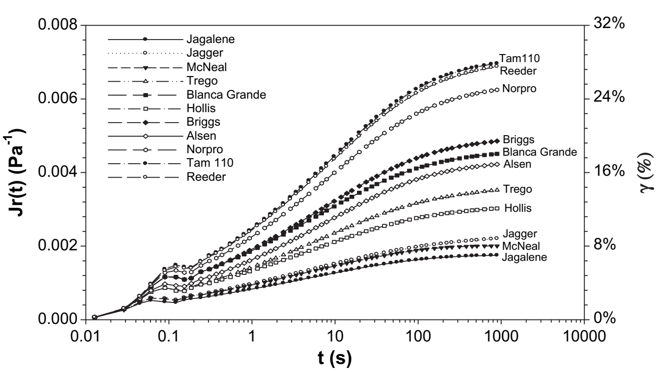

Fig. 1. Recovery curves for 11 hard wheat glutens.

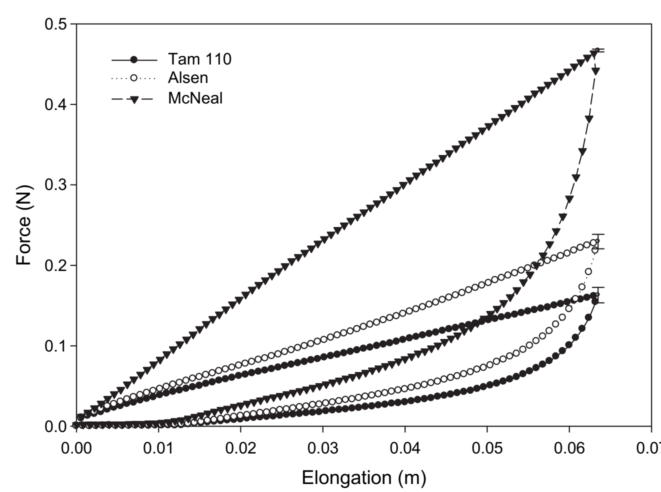

Fig. 3. Representative tensile extension and recovery curves for three glutens.

Table 2
Rheological properties of the cyclic tensile test: work of extension to 500% strain, recoverable work, DE (Degree of Elasticity; the ratio of recoverable work to work of extension), set (the percentage increase in sample length after recovery), force at 500% strain, which was also the maximum force for all 15 cultivars, and recoverable shear strain after creep. Values with the same letter are not significantly different.

Cultivar	Work of extension (N m) × 100	Recoverable work (N m)	DE (%)	Set (%)	Maximum force (N)	Recoverable shear strain (%)
TAM 110	0.56 ± 0.03 a	0.19 ± 0.01 a	34.45 ± 0.67 a	96.1 ± 2.2 a	0.16 ± 0.01 a	27.92 ± 0.74 a,i
Jagger	1.63 ± 0.40 b	0.59 ± 0.15 b	35.90 ± 0.91 a	91.4 ± 4.5 a,i	0.51 ± 0.13 b,h	8.84 ± 0.03 b,c,d,e
Jagalene	2.5 ± 0.20 c	0.72 ± 0.04 c	30.51 ± 0.74 a,d	85.9 ± 2.2 b,i,j	0.67 ± 0.07 c	7.00 ± 0.44 b
Alsen	0.73 ± 0.02 a,i	0.28 ± 0.01 a,j	38.89 ± 2.17 a	99.7 ± 0.6 a	0.23 ± 0.01 a,i	16.92 ± 0.35 c,f,j,m
Briggs	0.90 ± 0.28d i,j	0.31 ± 0.06 d,j,k	35.46 ± 4.28 a	98.5 ± 3.1 a	0.27 ± 0.079 a,j	19.46 ± 0.78 a,c,h
McNeal	1.52 ± 0.06 b,e	0.54 ± 0.02 b,f,l	35.79 ± 2.81 a	86.7 ± 3.3 c,i,k	0.47 ± 0.00 d,h	8.00 ± 0.41 b,j,k
Reeder	0.41 ± 0.12 a	0.16 ± 0.04 a	40.50 ± 2.14 b,e	102.7 ± 8.1 a	0.12 ± 0.04 a	27.60 ± 1.56 a,l
Hollis	1.06 ± 0.25 f,i	0.40 ± 0.08 g,m	37.76 ± 2.29 a,e	81.6 ± 4.6 d,j,k	0.33 ± 0.08 e,i,j	12.12 ± 0.35 g
Norpro	0.62 ± 0.14 a	0.23 ± 0.04 a	37.59 ± 3.16 a,e	99.9 ± 3.7 a	0.19 ± 0.04 a,i	25.04 ± 2.99 a,m,n,i,o
Blanca Grande	1.09 ± 0.12 g,i	0.37 ± 0.01 h,j,m	34.34 ± 3.00 a,d	93.3 ± 1.7 a,i	0.33 ± 0.03 f,i,j,k	18.06 ± 0.37 d,f,h,o
Trego	1.46 ± 0.20 b,h	0.46 ± 0.03 i,l,m	31.53 ± 2.31 a,d	94.8 ± 0.9 a,i	0.43 ± 0.04 g,h,k	14.12 ± 1.56 b,f,h
Patterson	0.68 ± 0.06 a,j	0.23 ± 0.02 a,k	33.37 ± 0.30 a,d	119.0 ± 3.3 e,l	0.20 ± 0.02 a,i	16.86 ± 1.54 e,f,h,k,n
Roane	0.71 ± 0.07 a,j	0.23 ± 0.02 a,k	32.90 ± 1.43 a,d	110.6 ± 1.2 f	0.20 ± 0.02 a,i	13.20 ± 1.31 b,f,h
Stephens	0.36 ± 0.07 a	0.10 ± 0.01 a	28.24 ± 1.66 c,d	148.1 ± 3.6 g	0.10 ± 0.02 a	29.43 ± 0.05 i,l
Eltan	0.70 ± 0.19 a,j	0.24 ± 0.05 a,k	35.38 ± 3.38 a	113.7 ± 1.6 h,l	0.21 ± 0.06 a,i	21.48 ± 2.65 a,f,i

cultivar gave a bread volume of 863 cc, which was similar to several hard wheat cultivars. Thus, wheat class is not a perfect indicator of bread loaf volume.

In order to visualize better the results of creep-recovery, the recoverability (%) was plotted versus the recoverable shear strain (%) in Fig. 2. Eleven of the fifteen glutens showed recoverability between 85 and 90% for these test conditions. Jagger was a little higher at 92% recoverability, while TAM 110 and Reeder were a little lower at 84 and 82%, respectively. Stephens's gluten was the lowest at only 71% recoverability. The fact that the soft wheat glutens (except for Stephens) were interspersed with the hard wheat glutens in terms of both recoverability and recoverable strain was surprising. A reasonable assumption would be that the soft wheat glutens, especially the 2 + 12 glutens, will be more extensible, i.e., show higher values of recoverable shear strain for a given stress, than the hard wheat glutens, but this was not the case. One plausible explanation is that creep-recovery testing at "small stresses" for at least some of these glutens reflects primarily contributions from the short-range cohesive forces that are similar in both hard and soft wheat glutens.

3.1. Loading–unloading curves (cyclic large deformation)

The presence of a primary elastic network, without specifying its exact molecular nature, at 25 °C is inferred from the obvious force of retraction shown by gluten when stretched to several times of its original length. This general concept has been said about gluten before, but we are not aware of work that objectively decouples elastic and viscous contributions to the stretching force for a gluten sample set this large and diverse. As described in Materials and methods, the procedure developed involved stretching a gluten sample to 500% extension and returning at the same rate. This is essentially the same test used initially by Schofield and Scott Blair (1937) to separate elastic and viscous effects in dough and gluten. Representative results are shown in Fig. 3 for the same three representative glutens (McNeal, Alsen, and TAM 110). Error bars represent the standard deviation for the maximum loading force for duplicate Glutomatic glutens.

As with the creep-recovery results for these three glutens, the overall shapes of the loading–unloading curves were qualitatively similar (data not shown), but were shifted vertically along the force axis. The shapes of the curves suggested parameterization in terms of the maximum force on loading (F_{max}), the ratio of the work recovered to that expended in stretching the gluten (Degree of Elasticity; DE) and residual strain after recovery (set). Set was defined as the percentage increase in sample length immediately after a cycle of loading and unloading. Gluten in general showed a complex "in-between" material behavior that is characterized by elasticity (snap-back), hysteresis between the loading and unloading curves and set. Differences between glutens can only be described by an array of parameters related to all of these material behaviors. The work of extension, recoverable work, and DE were chosen as objective measures of elasticity and set was chosen as an objective measure of plastic flow, i.e., irrecoverable strain. Maximum force could be taken as an indication of gluten strength. Data for all fifteen cultivars is reported in Table 2. No significant differences of these parameters were found among the wheat classes, or between soft and hard wheat flours. Therefore, wheat class or soft/hard classifications could not reflect the rheological behaviors of glutens and there are always exceptions in each category.

It was reasoned that recoverable work calculated from the unloading curve might be related to the recoverable shear strain in creep-recovery, since both are measures of elasticity of gluten albeit obtained under very different stress–strain conditions. The r^2 value between the two was 0.75. The recoverable work in turn was highly correlated to the mixograph mix time as shown in Fig. 4. This suggests that elastic effects are important in dough mixing. In addition, given the relatively low value of a set it was reasoned that hysteresis was due, mainly to delayed elastic effects seen in creep-recovery for all glutens.

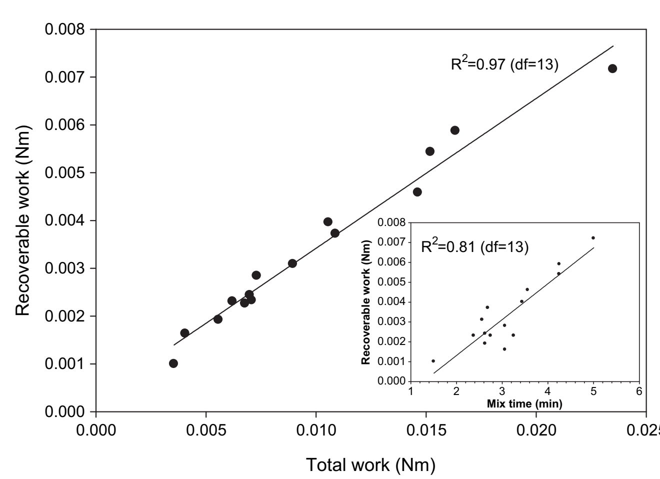

Fig. 4. Correlation of the total work of extension and the recoverable work of the cyclic extension test and mixograph mixing time and recoverable work.

The better developed the network, the lower was the recoverable shear strain, and the higher was the total work of extension in tensile tests. Total work of extension is plotted versus the recoverable work in Fig. 4. The correlation was very high ($r^2 = 0.97$), which indicates a cause and effect relationship between pure elastic properties and total resistance to extension Thus, creep under small stresses in the linear viscoelastic regime is related to large deformation tensile testing.

4. Conclusions

All glutens tested in this study in both small and large deformation experiments prominently expressed delayed elasticity. A wide range of recoverable strain from 7% to 28% was found for these wheat classes, but the wheat classes did not group together. This indicates that wheat class alone is not sufficient to predict gluten strength. Glu-D1 glutenin subunits did not play a systematic role in differences in the rheological properties of these glutens, which was indicated by the anomalously low recoverable shear strain of Roane ("2 + 12") compared with the anomalously high recoverable shear strain given by Reeder ("5 + 10"). Thus, a combination of biochemical and rheological measurements is needed to fully characterize and compare wheat cultivars.

A good correlation was found between the recoverable work of the extension test and the recoverable strain in the small deformation test and the mixograph mix time. A striking result was the high correlation between recoverable work and total work of extension for the large deformation tensile tests even though the recoverable work itself was always less than about 40% of the total work. The viscous dissipation during stretching may be due to more frictional losses in the elastic elements sliding by each other than a viscous phase in gluten itself, e.g. gliadin, which may offer little resistance to extension on its own. It can be concluded then that elastic properties play a key role in the extensional properties of gluten.

However, gluten elasticity by itself could not be used to rank the flours according to their baking quality. Large deformation stress relaxation and/or compression-recovery experiments may be more effective at producing correlations between gluten viscoelasticity and baking volume data. Further work is needed to better understand how to influence the viscoelastic properties of gluten in breeding programs.

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