Relationship of Creep-Recovery and Dynamic Oscillatory Measurements to Durum Wheat Physical Dough Properties[1]

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ABSTRACT

Durum wheat gluten strength is important in determining extrusion properties and pasta cooking quality. Durum wheats varying in strength were tested using an alveograph and a 2-g micro-mixograph, both widely accepted techniques for determination of physical dough properties. Doughs from the 2-g micro-mixograph were characterized by dynamic oscillatory and large deformation creep tests using a controlled stress rheometer. Mechanical properties obtained from both testing regimes were strongly correlated with many of the parameters provided by the alveograph and micro-mixograph. Maximum strain attained after 5 min creep ranged from <5% for the strongest least extensible cultivar to >25% for the weakest cultivar, with a coefficient of variation among replicates of <10%. Storage modulus (G') at 2 Hz ranged from ≈7,000 Pa for the weakest cultivar to >16,000 Pa for the strongest, least extensible cultivars, with a coefficient of variation of <6%. Tan δ (G''/G') values were ≈0.4 for the strongest versus >0.5 for the weakest cultivars, indicating the larger contribution of the elastic component in the strong cultivars. The rheometer allows discrimination of durum wheat cultivars of varying gluten strength while requiring less sample than traditional physical dough testing techniques.

Extrusion properties of durum wheat semolina doughs and pasta cooking quality are largely determined by gluten strength of the raw material. Several chemical and instrumental methods have been developed to assess gluten strength, with attempts to relate data from these methods to processing characteristics and end-product quality (Cole 1991). Traditional empirical rheological methods used in bread dough testing such as the farinograph, mixograph, and alveograph have been adapted for monitoring of durum wheat semolina gluten strength to provide practical information to processors (Irvine et al 1961, Quick and Donnelly 1980, Walle and Trentesaux 1980). An alveograph, which subjects doughs to biaxial extension, has become the instrument most widely accepted by commercial durum wheat processors and it is used in international trade specifications because it provides information on both dough strength and extensibility (Dexter et al 1994). The alveograph requires relatively large amounts of semolina (250 g), which can be a hindrance where sample size may be a limiting factor such as in screening of plant breeders' lines or in reconstitution experiments.

Development of a 2-g direct drive mixograph or micro-mixograph interfaced with a personal computer for data collection and interpretation has provided a means of establishing traditional empirical mixing characteristics where sample size is limited (Rath et al 1990). Khatkar and coworkers (1996) used the micro-mixograph and a small-scale baking procedure to evaluate the baking potential of flours from wheats of diverse genetic origin. They concluded that the micro-mixograph may be a valuable alternative to baking trials in assessing flour baking potential. A limitation of the micro-mixograph is its inability to provide information on dough extensibility.

Kieffer et al (1998) developed a micro-scale extension test for dough and gluten that was similar to an extensigraph test using doughs prepared with 2% salt and mixed to a constant consistency in a 10-g farinograph. They found that extension results in combination with flour protein or wet gluten content could reliably predict loaf volumes of micro-scale baked bread.

Several techniques have been employed for investigating the fundamental mechanical properties of wheat flour doughs including stress relaxation (Launay and Buré 1974, Fu et al 1997), creep and creep recovery (Hibberd and Parker 1979, Campos et al 1997), and dynamic oscillatory measurements (Abdelrahman and Spies 1986, Faubion and Hoseney 1990, Amemiya and Menjivar 1992). Dynamic oscillatory measurements have been most commonly used to assess fundamental mechanical characteristics of wheat flour dough, however, there is no consensus as to their practical advantage over established empirical methods. Using dynamic oscillatory measurements, Janssen and coworkers (1996) concluded that there was no direct relationship for either dynamic moduli or tan δ to loaf volume but that the information obtained was complementary to biaxial extension and other rheological measurements. Kokelaar et al (1996) suggested that biaxial extension tests were more relevant to breadmaking quality and that small strain dynamic measurements were not related to biaxial extension measurements. Huang and Kokini (1994) measured biaxial extensional viscosities and related these measurements to loaf volume development during proofing. Subsequently, Huang and Kokini (1999) linked biaxial extensional measurements to loaf volume expansion during proofing. Campos and coworkers (1997) used dynamic oscillatory and creep measurements on undeveloped doughs and doughs prepared by mixing in a farinograph bowl in an attempt to decouple the effects of hydration and energy input. They reported that undeveloped doughs exhibited lower creep compliance and lower complex modulus (G^*) values than developed doughs. They also found that hard wheat dough was more viscous and much more resistant to deformation than soft wheat dough, which was consistent with farinograph data showing hard wheat dough to be stronger with much longer stability and a lower mixing tolerance index.

Our objective was to establish a small-scale method that would provide information on durum semolina dough strength and extensibility and relate to traditional empirical methods. Dynamic rheometers require relatively small samples and are capable of providing information under conditions of small and large deformations, but to date, the limiting factor determining sample size has been the mixers used to prepare the dough. The advent of the micro-mixograph has provided a means of working with small (2-g) dough samples. Factors considered in the study included the effect of semolina protein content, wheat cultivar, and environment.

MATERIALS AND METHODS

Wheat

Composites of commercially grown durum wheat collected from a variety of locations in western Canada were prepared at four protein levels for each of three years (n = 12). All samples graded

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No. 2 Canada western amber durum (CWAD) or better. Varietal distribution within the composites was assessed by electrophoresis (Tkachuk and Mellish 1980) to confirm relatively consistent distribution of cultivars among samples. All samples contained on average 65% of the cultivar Kyle, which is the proportion of Kyle found in the CWAD crop during these years (Canadian Grain Commission, *unpublished data*).

Preparation of composites over three crop years with differing annual growing conditions allowed us to consider the effect of environment as well as protein content, while recognizing that within each crop year there were micro-climates that had their own unique effects. Areas producing extremes of either high or low protein wheat tended to be very localized and may have had different climatic and soil fertility conditions from the rest of the growing area.

Wascana, Kyle, Durex, and AC Melita wheat cultivars were grown at the Agriculture and Agri-Food Canada (AAFC) Semiarid Prairie Agricultural Center in Swift Current, Saskatchewan, in 1996, and at AAFC field plots in Regina, Saskatchewan, in 1997. Additional plots of Stewart 63 were grown in 1997 at AAFC Swift Current.

The cultivars Kyle and Wascana were representative of moderate strength and relatively extensible cultivars grown in western Canada for the past 30 years. AC Melita is a new CWAD cultivar with stronger gluten registered in response to market demand for greater gluten strength in durum wheat. Durex is a so-called desert durum cultivar with very strong gluten developed for production in the southwestern United States. The additional strength of AC Melita and Durex is evident from higher alveograph values (W = work of deformation until rupture; L = length of the curve; P = maximum peak height; P/L = a ratio of peak height to extensibility), and longer farinograph development times and stabilities as compared with moderate strength CWAD cultivars.

Stewart 63 was added to extend the range in dough strength. It is a γ-gliadin 42 durum wheat that is no longer registered to be grown in western Canada because of its inferior pasta-making characteristics and very weak dough mixing properties. Wascana, also a γ-gliadin 42 type, represents the extreme high end of dough strength observed in γ-gliadin 42 types. All other cultivars were γ-gliadin 45 types. The presence of γ-gliadin band 42 has been associated with poor pasta quality while band 45 has been associated with superior quality (Damidaux et al 1978). Italian cultivars Creso, Grazia, and Simeto were supplied by Instituto Nazionale della Nutrizione, Rome, Italy, from the 1996 Italian harvest.

Milling

All wheats were cleaned and tempered overnight to 16.5% moisture content. Commercial composites were milled using a four-stand laboratory mill (Allis-Chalmers, Milwaukee, WI) in conjunction with a laboratory purifier (Black 1966) using the procedure of Dexter et al (1990). The milling area was controlled for temperature (21°C) and relative humidity (60%). Semolina yield range was 65.0–70.8% of clean wheat on a constant moisture basis, with a typical particle size distribution of 6% held on a 420-µm sieve, 65% held on a 250-µm sieve, 19% held on a 177-µm sieve, 6% held on a 149-µm sieve, and 4% through a 149-µm sieve.

Semolina Tests

Analytical results were expressed on a 14% mb for semolina. Moisture content was measured by single-stage air-oven using Approved Method 44-15A (AACC 1995). Protein content was determined by a combustion nitrogen analyzer (model FP-428, Leco Corp. St. Joseph, MI) calibrated against ethylenediaminetetraacetic acid (EDTA). Analysis for starch damage was made using the Megazyme kit according to Approved Method 76-31 (AACC 1995).

Physical Dough Tests

Alveograph curves were obtained using constant pressure (model MA82, Chopin SA, Villeneuve-la-Garenne, France) following Standard No. 121 (ICC 1980). Values for P, L, and W were automatically calculated by the instrument. Mixograph data were obtained using a 2-g direct-drive mixograph (National Manufacturing Division, TMCO, Lincoln, NE) at fixed water absorption of 50% (14% mb), resulting in a final dough moisture content of 42.7%. Samples and water were weighed to 0.001 g. The temperature of the mixing bowl was maintained at 25°C. Tests were performed in triplicate. Micro-mixograph parameters were determined using Mixsmart computer software provided with the instrument using mid-line analysis. Farinograph (C.W. Brabender Instruments, South Hackensack, NJ) mixing characteristics were obtained using Approved Method 54-21 (AACC 1995).

Rheological Testing

A dynamic stress rheometer (model SR500, Rheometric Scientific, Piscataway, NJ) was used in both oscillatory tests (strain-controlled) and creep (constant stress) in shear mode. The rheometer was equipped with 25-mm diameter serrated upper and lower parallel plates that were maintained at 25°C. Initially samples were prepared by mixing at 50% absorption on 14% mb in the 2-g mixograph to establish peak mixing time. Preliminary rheological testing of mixograph doughs using samples mixed to peak gave unsatisfactory precision in the resulting rheological data (data not shown), probably because peak mixing times varied by up to 0.5 min among replicates. Subsequently, when doughs were mixed to 1 min past peak, precision improved. Mixing to 1 min past peak was therefore adopted for preparation of dough for rheological testing.

A sample of dough was removed from the bowl and a 2-g portion was rounded by hand and placed between the plates of the rheometer. The sample was gently flattened to fit the plate geometry using a Teflon-coated spatula. The upper plate was lowered to a fixed gap of 2.75 mm. Exposed edges of dough were liberally coated with mineral oil to prevent drying.

Strain sweeps conducted at 1 Hz indicated linear response at strains between ≈0.25 and 0.6%, which is in agreement with previously published data (Dus and Kokini 1990, Amemiya and Menjivar 1992, Lindahl and Eliasson 1992). Subsequent oscillatory measurements were taken at 0.5% strain over a frequency range of 0.5–10 Hz.

Kyle and AC Melita semolina samples were used to determine rest times required before frequency sweeps, as they represent the typical range of dough strength found in commercially grown durum wheat in western Canada. Oscillatory measurements were taken immediately after loading on the rheometer and every 5 min thereafter to a maximum of 30 min on three separately prepared doughs of each cultivar. Data obtained were storage modulus (G'), shear loss modulus (G''), complex modulus (G^*), and tan δ (G''/G'), although only G' and tan δ are discussed. Statistical comparisons were made at 2 Hz, which is approximately the midpoint in frequency sweeps. Our results indicated that a rest time of 15 min after loading in the rheometer and coating with oil was sufficient to achieve reproducible results, and this was adopted as the standard rest time for subsequent analyses.

Creep-recovery tests were conducted immediately after oscillatory measurements on the same dough sample. Dynamic oscillatory testing at low strain is nondestructive and would not have caused changes to the sample structure. A stress of 100 Pa, which exceeded the region of linear viscoelasticity, was applied for 300 sec, sufficient for the sample to reach steady-state flow as determined by the instrument software (which allows for 10% slope variation). Creep tests performed at large deformation were essentially micro-scale shear-extension tests. Results reported are the average of three replicates, where each replicate represents a separately mixed dough. Tests including rest time, frequency sweeps, and creep to maximum % strain were completed in <30 min.

Water Absorption

Effect of water absorption on rheological data was determined using a set of pure cultivars. They were prepared in the micro-mixograph at two additional water absorptions (48 and 52%) for oscillatory and creep testing as described above. Semolina and water amounts were controlled so as to maintain constant dough weight of 3 g in the micro-mixograph bowl.

Experimental Design and Statistical Analysis

Analytical data were the average of duplicate tests. Micro-mixograph testing was conducted in randomized complete block design, in triplicate, and also expressed as averages. The commercial semolina samples and pure cultivars were tested as separate series. Oscillatory and creep-recovery testing were conducted in triplicate and the averages reported. Coefficients of variation were calculated for oscillatory and creep data. Alveograph data were reported as the average of five tests on a single batch of dough, while farinograph data were from single tests. All statistical analyses were performed using SAS software (SAS Institute, Cary NC).

RESULTS AND DISCUSSION

Dough Resting Time

There is a great deal of variation in the literature concerning the amount of time required for dough relaxation after sample mixing before oscillatory or steady-shear testing, ranging from 1 min (Lindahl and Eliasson 1992) to 1 hr (Baltsavias et al 1997). In some studies, dough was rested after mixing and before loading, and then rested again after loading. Amemiya and Menjivar (1992) rested samples for 2 hr before loading and then for 5 min after loading. Other workers rested samples after loading, and before testing. With so much diversity of sample handling in the literature, we decided to determine the effect of resting time after loading on the results of dynamic oscillatory tests without having previously rested the dough in the mixer.

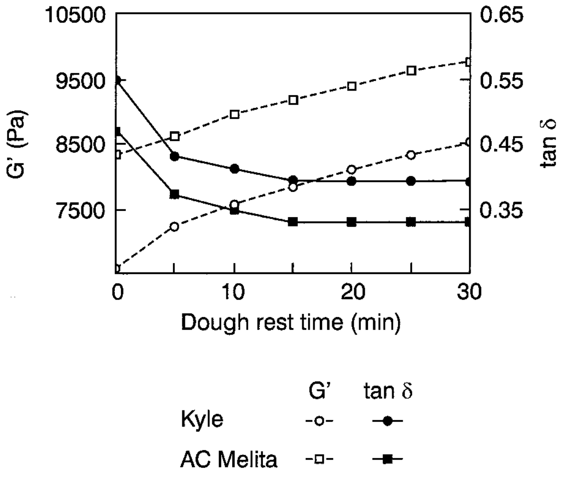

Fig. 1. Relationship between dough rest time after sample loading between dynamic rheometer parallel plates and dynamic oscillatory parameters of storage modulus (G') and tan δ (G''/G') for durum wheats Kyle and AC Melita.

Ideally, rest times would be derived from the longest relaxation time obtained during stress relaxation testing. However, not having equipment capable of performing stress relaxation tests, we determined the amount of rest time required beyond which significant changes in G' and tan δ no longer occur (Fig. 1).

Tan δ decreased and G' increased with resting time for both Kyle (moderate strength) and AC Melita (very strong). The two cultivars were clearly differentiated throughout the complete range of rest times examined, and the pattern of change with rest time was similar for both. There was no statistically significant ($P > 0.05$) change in either G' or tan δ beyond 10 min of resting when compared at a fixed frequency of 2 Hz, although G' showed a continual increasing trend up to 30 min. Drying of the exposed surface of the dough would account for an increase in G', but we observed no evidence of drying up to 30 min of rest time. Lindahl and Eliasson (1992) also noted an increase in G' over time for durum wheat doughs, however, they reached a plateau region at rest times >20 min. Differences in rest time required to reach levels where there were no longer any significant changes may be due to differences in moisture content. Lindahl and Eliasson (1992) were working with dough at 37% moisture content, whereas our dough was at 42.7% moisture content. Also, Lindahl and Eliasson used a farinograph for sample preparation, developing dough through kneading, which is quite different from the elongation and rupture action of the mixograph. To satisfy a minimum requirement of 10 min, we chose 15 min as our rest time to ensure we were within the region where there would no longer be any significant changes in G' and tan δ. We found that exposed surfaces of dough, even though coated with oil, tended to dry and form a skin if left for periods >30 min.

Dough Moisture Content

Moisture content of dough has an effect on dynamic mechanical results (Navickis et al 1982, Berland and Launay 1995, Edwards et al 1996). The alveograph procedure (ICC 1980) recommends a fixed absorption level of 50%. Using a fixed absorption level is often a concern when working with flour samples because milling technique can have a profound effect on degree of starch damage, which, in turn, affects water absorption. As starch damage increases, dough becomes stiffer, resulting in reduced alveograph extensibility, increased peak height, and increased area under the curve (Dexter et al 1994). Milling procedures used to produce semolina generally do not induce high levels of starch damage. Analyses for starch damage indicated that levels in our semolina samples were maintained at ≤4.7%. Variations in semolina-water absorbing capacity are largely attributed to protein content (Quaglia 1988).

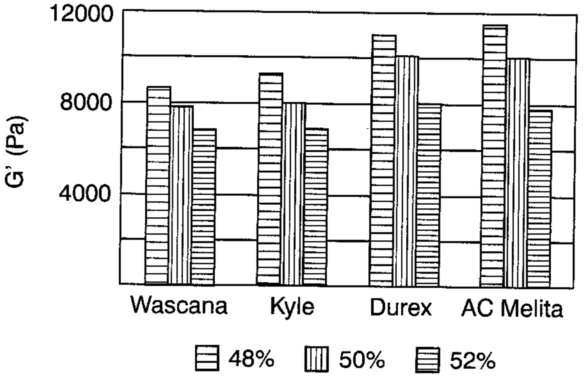

Fig. 2. Effect of semolina water absorption on storage modulus (G') at 2 Hz for four durum wheats.

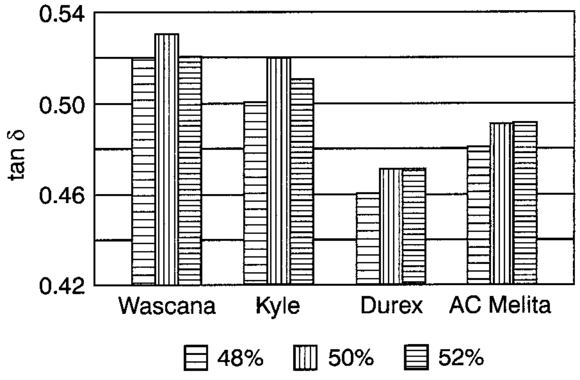

Fig. 3. Effect of semolina water absorption on tan δ (G''/G') at 2 Hz for four durum wheats.

Samples of Kyle, AC Melita, Durex, and Wascana were prepared at two additional absorption levels (48 and 52%) to assess whether relative values for the different cultivars obtained by oscillatory and creep tests were affected by absorption. Farinograph absorptions of these four samples were within a relatively narrow range of 56.8–60.4%. Kyle and Wascana were representative of the moderate strength, relatively extensible model for durum wheat grown in western Canada over the past several decades. In contrast, AC Melita and Durex, a cultivar from the southwestern United States, are typical of stronger durum that has become more popular in recent years.

Variations in moduli due to mixing and loading of samples resulted in coefficients of variation of <5.5% between replicates. Under oscillatory test conditions, there was a shift to lower moduli (G' and G″) with increasing moisture content. This phenomenon has been well documented (Navickis et al 1982, Dreese et al 1988, Edwards et al 1996). G' values ranked the samples in consistent order at each moisture content (Fig. 2). Differences in G' values for a given cultivar due to absorption were statistically significant ($P < 0.05$). Tan δ values were not significantly ($P > 0.05$) affected by moisture content (Fig. 3), which is in agreement with reports by Berland and Launay (1995) and Dreese et al (1988). Although tan δ values were not significantly different at different absorptions, we observed significant ($P < 0.05$) differences between cultivars.

Creep data at 48 and 52% absorption bracketed those of 50% for all four samples, ranking them in the same order at all absorption levels (Fig. 4). Increasing water absorption increased maximum strain attained, which one would expect, as adding water would facilitate flow. Coefficients of variation were ≤9.7% among replicates. Differences in creep parameters were significant ($P < 0.05$) at different absorption levels and among cultivars at a given absorption level. Wascana was consistently most extensible, and Durex and AC Melita, which were indistinguishable from each other, were least extensible at all absorption levels.

Confirmation that differences in G' and creep among cultivars could not be explained based on water absorption made it reasonable to test samples at a fixed absorption. From a practical standpoint, we found that absorption levels >50% resulted in doughs that were sticky and difficult to transfer to the rheometer, particularly for weaker cultivars. Because 50% is the recommended water absorption level for alveograph testing, and the dough was relatively easy to handle, all further testing was conducted at a fixed absorption of 50%.

Creep Times

We used several creep times before deciding on 300 sec. Under the default criteria of our rheometer, which allowed for 10% slope variability and required a minimum number of 25 data points to fit a straight line, steady-state flow was attained at 300 sec. Using the cultivars Kyle, AC Melita, Durex, and Wascana, we allowed

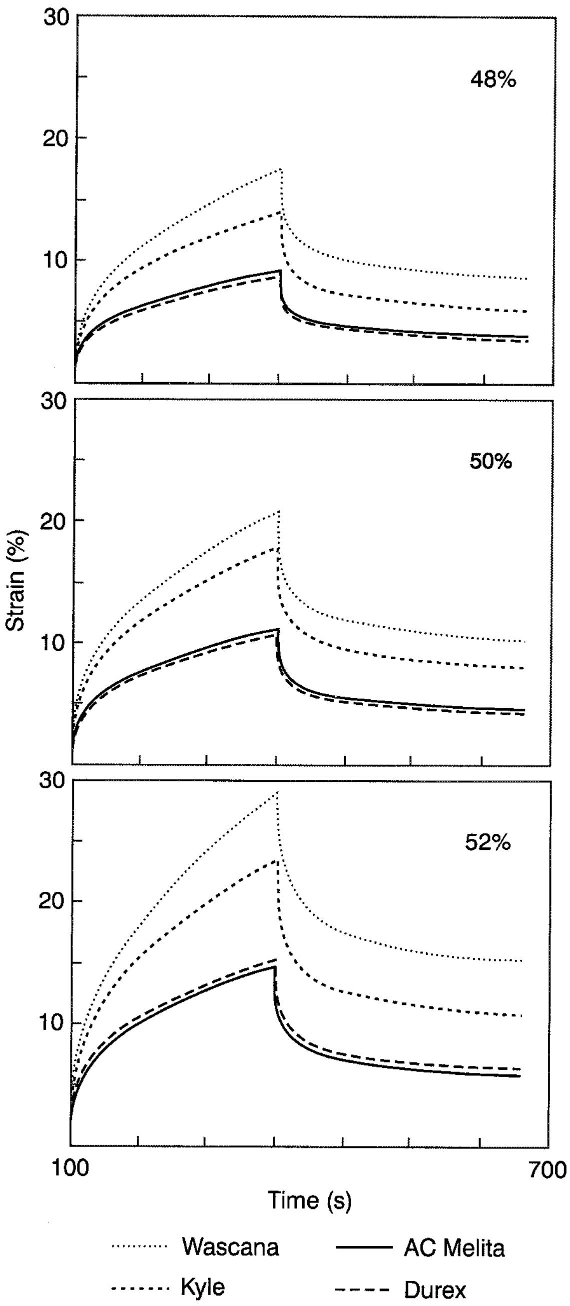

Fig. 4. Creep-recovery in shear of four durum wheats at three different semolina-water absorption levels. Applied stress = 100 Pa.

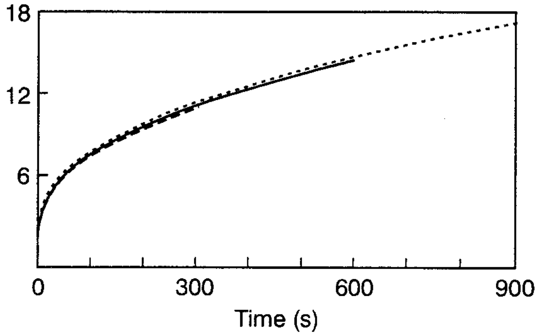

Fig. 5. Creep tests conducted for 300, 600, and 900 sec on separately prepared doughs to establish creep time required to attain steady state.

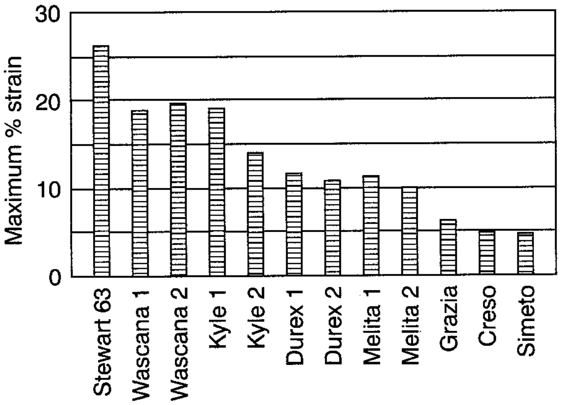

Fig. 6. Maximum % strain of durum wheats as measured using a creep-recovery test in shear mode using 100 Pa stress applied for 300 sec.

doughs to creep for 300, 600, 900, and 10,000 sec. In all cases, the strength of the samples ranked in the same order (data not shown), and up to 900 sec, the curves indicated steady-state flow (Fig. 5). At 10,000 sec, however, the curves were no longer indicative of steady-state. This may have been more a function of drying of the sample than a characteristic of the dough, as we observed formation of a skin at the exposed surface of the dough even though it was well coated with oil.

We chose 300 sec as the standard creep time for succeeding experiments because not only was steady-state attained but the shorter time reduced the possibility of variability resulting from sample drying. Our stated objective was to develop relatively rapid small-scale tests that related to traditional rheological tests used for screening of durum wheat. Consistent with that objective, we found that maximum % strain reached after 300 sec was sufficient for discrimination of samples varying in strength.

Effect of Protein Content

Several studies have found that dynamic moduli of doughs are affected by flour protein content (Navickis et al 1982, Amemiya and Menjivar 1992, Lindahl and Eliasson 1992), with higher protein content resulting in higher moduli. Hibberd (1970), using starch and gluten doughs, saw an increase in tan δ with increasing proportions of gluten. In contrast, Abdelrahman and Spies (1986) found a decline in tan δ with addition of gluten to flour. Generally, studies that compared doughs of variable protein content used wheats of diverse genetic origin that varied also in gluten strength, making it difficult to isolate effects solely due to protein content. Alternatively, studies that varied gluten-to-starch ratios did result in the ability to artificially isolate the effect of protein content but at a fixed gliadin-to-glutenin ratio. Under field production, as wheat protein content increases the relative proportion of gliadin increases (Dexter et al 1989, Wieser and Seilmeier 1998). Gliadin contributes to the viscous nature of wheat doughs and to its extensibility (Orth and Bushuk 1972, Scanlon et al 1990, Fido et al 1997). To establish the effect of durum wheat protein content in its native form on dynamic moduli and large deformation creep, we used composites of commercially grown Canadian durum wheat prepared over three years at four protein levels (high, medium-high, medium-low, low).

Farinograph absorptions increased with increasing protein content (Table I), a well-documented phenomenon (Tipples et al 1978). There were no significant ($P > 0.05$) differences in dough development time or stability resulting from differences in protein content. Micro-mixograph curves were affected by protein content. Lower protein samples generally had somewhat longer mixing times, lower peak dough resistance, narrower bandwidth at peak, more rapid breakdown, and lower work input requirements (Table I), which is in agreement with work done by Khatkar et al (1996). Alveograph data indicated reduced extensibility at low protein content, with the higher protein sample series having longer L values and lower P/L ratios (Table I). High protein also resulted in increased strength indicated by higher W values. Dexter et al (1994) demonstrated that increasing protein content in durum wheat resulted in increased alveograph extensibility (L), but had little effect on resistance to deformation (P), leading to lower P/L values and larger W values.

We were unable to detect a significant effect ($P > 0.05$) of protein content on G' by least significant difference. Environment had a very strong effect on G' ($P < 0.001$). Samples collected during the 1995 harvest had the highest G' values and those collected in 1997 had the lowest.

Tan δ was strongly affected by both protein content and environment ($P < 0.001$), and there was a significant interaction between the two factors ($P < 0.001$). Tan δ increase was strongly correlated with increasing protein content ($r = 0.93$), with each protein level being significantly different from the others (Table I). Gliadin content accumulates as durum protein content increases (Dexter et al 1989), concomitant with increased alveograph extensibility (Dexter et al 1994). Therefore, the increase in tan δ with increasing protein content was predictable.

Maximum creep (% strain) was significantly affected by both protein content and environment ($P < 0.001$). Least significant difference separated the two higher protein samples, which were more extensible, from the two lower protein samples (Table I). Alveograph P/L value, a ratio of peak height to extensibility, increased from low and medium-low to high and medium-high protein content (Table I), indicating increased extensibility as protein content increased. Composites prepared from the 1997 crop exhibited the highest creep values and those from 1995 exhibited the lowest.

TABLE I
Rheological Characteristics of Protein Composites Shown as Three-Year Averages

					F Values		
	High	**Medium-High**	**Medium-Low**	**Low**	**Protein**	**Environment**	**P × E**
Protein, %	14.7	12.6	10.8	8.9			
Farinograph							
Water absorption,%	61.43a[a]	59.47ab	57.77bc	56.23c	15.15*[b]	2.03	. . .[c]
DDT[d], min	3.53a	3.37a	3.17a	3.23a	1.04	9.66*	. . .
Stability, min	4.07ab	3.73b	4.43ab	4.70a	2.49	4.78	. . .
Micro-mixograph[e]							
MT, min	2.74b	2.51c	2.84b	3.03a	16.58**	1.04	4.40*
PDR, units	69.1a	57.2b	48.8c	42.8d	542.21**	71.18**	35.03**
MS, units	17.5a	14.4b	13.7bc	13.6c	59.20**	26.25**	3.82*
WI, % torque min	131.4a	99.3b	98.6b	97.6b	38.28**	9.44**	3.64*
Alveograph[f]							
L, mm	91.85a	81.08ab	65.03bc	54.73c	4.91*	0.95	. . .
P/L	0.658b	0.668b	0.867ab	1.031a	3.66	2.71	. . .
$W \times 10^{-4}$ J	129.8a	95.7b	88.0b	87.3b	11.63*	5.24*	. . .
Creep							
Maximum % strain	16.06a	15.74a	12.42b	11.93b	25.32**	92.88**	5.39*
Dynamic oscillatory[g]							
G', Pa	9,386b	9,388b	10,206a	9,860ab	4.84*	58.49**	1.59
Tan δ	0.514a	0.495b	0.470c	0.460d	280.01**	32.50**	14.50**

[a] Values followed by the same letter are not significantly different.

[b] *,** = significant at $P < 0.05$ and 0.001, respectively.

[c] Insufficient sample prevented replication of farinograph and alveograph tests, therefore interaction terms could not be calculated.

[d] Dough development time.

[e] MT = mixing time; PDR = peak dough resistance; MS = mixing stability; WI = work input.

[f] L = length of curve; P/L = peak height/curve length; W = work of deformation until rupture.

[g] G' = storage modulus; tan δ = shear loss modulus/storage modulus.

Because there was a pattern of declining creep and increasing G' with age of the samples, the possibility of oxidation modifying dough properties during semolina storage cannot be ruled out.

North American Durum Cultivars

Typical of very strong gluten types, Durex and AC Melita micro-mixograph curves had longer mixing times, higher mixing stabilities, and higher work input requirements than the other three North American cultivars (Table II). Stewart 63 had the weakest mixing characteristics with very short mixing time, rapid curve breakdown, and low work input.

Peak dough resistance (peak height) was strongly affected by semolina protein content. Nevertheless, even at low protein content, Durex 2 (11.6%) and AC Melita 2 (10.1%) had stronger mixing curve characteristics (longer mixing time, higher mixing stability, and work input) than did Kyle 1 and Wascana 1, which were both significantly higher in protein (14.1 and 14.9% respectively). This is indicative of the superior strength of the Durex and AC Melita.

North American durum wheat cultivars were generally extensible, with low, long alveograph curves (Table II). Most had P/L ratios of <1. Extensibility was, to some degree, related to protein content, with the lower protein replicates having lower L values and higher P/L ratios than the higher protein counterparts. Differences in optimum water absorption requirements indicated by farinograph (Table II) were not sufficient to account for alveograph dough extensibility differences. Durex and AC Melita had the strongest curves according to W values (range 187×10^{-4} to 263×10^{-4} J). Stewart 63 was weakest of the cultivars ($W = 45 \times 10^{-4}$ J).

Although there were significant differences in G' among samples ($P < 0.001$), the only cultivar to show a significant difference at different locations was Wascana (Table III). Durex and AC Melita samples were not significantly different from one another and exhibited the highest G' values. Stewart 63 had the lowest G', as expected, given its poor performance in alveograph, farinograph, and micro-mixograph testing. Wascana and Kyle had intermediate G' values. There was strong correlation between G' and micro-mixograph mixing time ($r = 0.86$), mixing stability ($r = -0.80$), and work input ($r = 0.84$). Alveograph P/L and W were also strongly related to G' ($r = 0.93$ and 0.78, respectively).

The lack of difference in G' between cultivar replicates was of particular interest because there were large differences in protein content between growing locations. For example, there was a 4% difference in protein content between samples of AC Melita, yet G' values were not significantly different from each other. This tends to indicate that values obtained were indicative of intrinsic gluten quality characteristics of a particular cultivar and were not a function of protein content.

Cultivar had a significant effect ($P < 0.001$) on tan δ values (Table III). However, when tested by least significant difference, cultivars segregated by protein content rather than by protein strength as measured by farinograph, alveograph, or micro-mixograph. Tan δ tended to decrease as dough protein content decreased. The same trend was demonstrated for the commercial CWAD composites varying in protein content (Table I). It appeared that tan δ was not an effective parameter for evaluation of durum semolina dough strength when assessing samples of varied environmental origin due to the strong influence of protein content.

The large deformation creep tests were successful at ranking cultivars according to the expected level of dough strength as measured by alveograph (Fig. 6). Differences between cultivars in maximum % strain attained during creep were significantly different ($P < 0.001$). Stewart 63 was the weakest, most extensible cultivar, reaching 26.1% strain during creep. Durex and AC Melita were the most resistant to deformation and were not significantly different from each other, nor were there differences between environments. The difference in protein content between locations was as much as 4%, and Stewart 63 had a protein content of 12.2%, which was lower than all of the cultivars grown at AAFC in Swift Current. One would expect, based solely on protein content as seen with the CWAD protein composites, that the higher protein samples of Wascana, Kyle, Durex, and AC Melita from AAFC Swift Current should have been more extensible than Stewart 63. However, this was not the case. Therefore, the greater maximum creep of Stewart 63 could not be accounted for by protein content, so logically it must reflect reduced resistance to deformation due to weaker gluten.

The two samples of Wascana were not significantly different from each other and were intermediate in extensibility values between Stewart 63 and Durex and AC Melita. The samples of Kyle were different from each other. Kyle 1 (14.1% protein) was ranked similarly to Wascana, whereas Kyle 2 (9.8% protein) was less extensible than Kyle 1, but more extensible than either Durex or AC Melita. Kyle 2 reduced extensibility was consistent with its low protein content. As noted earlier, commercial CWAD samples of high and medium-high protein exhibited greater maximum creep than low and medium-low protein samples. The commercial samples were made up predominantly of the cultivar Kyle, therefore the large reduction in extensibility with very low protein may be characteristic of the cultivar.

TABLE II
Physical Dough Properties of North American Durum Wheat Cultivars

		Wascana		Kyle		Durex		AC Melita	
Location	**Stewart 63**	**1**	**2**	**1**	**2**	**1**	**2**	**1**	**2**
Protein, %	12.2	14.9	10.0	14.1	9.8	12.9	11.6	14.1	10.1
Farinograph									
Water absorption,%	56.9	57.1	53.3	56.8	53.7	57.3	57.7	60.4	55.9
DDT[a], min	3.2	4.3	3.2	3.3	3.7	4.8	5.0	4.2	4.8
Stability, min	2.4	4.2	2.9	5.5	7.2	8.7	12.4	5.3	12.6
Micro-mixograph[b]									
MT, min	1.69e[c]	2.04d	2.42c	2.51c	2.99b	3.18b	3.71a	3.16b	3.60a
PDR, units	42.8f	54.9d	39.3g	64.5b	43.7f	72.2a	59.1c	71.1a	49.1e
MS, units	10.9ef	12.7de	9.5f	16.3c	14.6cd	28.2a	27.3a	23.2b	22.3b
WI, % torque min	53.2d	75.9c	67.8cd	109.1b	82.2c	147.7a	147.9a	151.3a	123.8b
Alveograph[d]									
L, mm	60	93	63	108	86	93	63	113	67
P/L	0.6	0.4	0.5	0.4	0.7	0.9	1.7	0.6	1.2
$W \times 10^{-4}$ J	45	72	45	108	98	251	263	214	187

[a] Dough development time.

[b] MT = mixing time; PDR = peak dough resistance; MS = mixing stability; WI = work input.

[c] Values followed by the same letter are not significantly different.

[d] L = length of curve; P/L = peak height/curve length; W = work of deformation until rupture.

TABLE III
Dynamic Oscillatory Measurements of North American Durum Wheat Cultivars Using 0.5% Strain[a]

Wheat	Protein (%)	G' (Pa)	Tan δ
Stewart 63	12.2	7,175d[b]	0.515a
Wascana 1	14.9	8,813b	0.527a
Wascana 2	10.0	7,844cd	0.484bc
Kyle 1	14.1	7,965b–d	0.518a
Kyle 2	9.8	8,544bc	0.476cd
Durex 1	12.9	10,087a	0.471cd
Durex 2	11.6	10,573a	0.483bc
AC Melita 1	14.1	10,178a	0.492b
AC Melita 2	10.1	10,188a	0.462d

[a] Data taken at 2 Hz.
[b] Values followed by the same letter are not significantly different ($P > 0.05$).

The creep maximum % strain was strongly negatively correlated with micro-mixograph mixing time ($r = –0.97$), mixing stability ($r = –0.83$), and work input ($r = –0.96$), as well as with alveograph P/L ($r = –0.77$) and W ($r = 0.90$).

Italian Durum Cultivars

Italian durum wheat lines Creso, Grazia, and Simeto were characterized by strong, inextensible gluten properties that resulted in very high, short alveograph curves with P/L ratios ranging from 1.6 to 3.5 (Table IV). Micro-mixograph data (Table IV) also indicated strong gluten characteristics with longer mixing time and higher work input than the North American cultivars even though all three samples were relatively low in protein (9.7–11.1%). They made an ideal series to determine whether our testing protocols were reflective of intrinsic gluten quality characteristics because they exhibited such different rheological characteristics, but they had protein content similar to several of the North American lines.

All three Italian cultivars had much higher G' values and lower tan δ than any of the North American cultivars analyzed, consistent with their alveograph and micro-mixograph results (Table IV). Maximum % strain also was very low: 4.7–6.1% compared with 9.8–26.1% for the North American cultivars (Fig. 6). Grazia exhibited the greatest extensibility of the three Italian cultivars by alveograph, dynamic oscillatory, and creep measurements. Reduced extensibility of the Italian lines, relative to North American lines, resulting in low creep, high G', and low tan δ for the Italian lines, could not explained exclusively by low protein content. The effect of environment can not be ruled out as a contributor to the qualitative differences between the Italian and North American lines. However, the three Italian samples came from different regions of Italy, and their similarity in rheological properties to each other suggests that they are intrinsically different from North American cultivars.

CONCLUSIONS

Tandem use of the 2-g micro-mixograph and a dynamic rheometer provided an excellent means of assessing dough strength of durum semolina and should prove useful, particularly where sample size is limiting, such as in reconstitution experiments. Dynamic mechanical measurements were able to segregate samples according to dough strength as measured by the alveograph while using far less sample. Alveograph P/L ratio and W value were strongly correlated with G' ($r = 0.93, 0.78$) and with large deformation creep maximum % strain ($r = –0.77, –0.90$).

Intrinsic dough strength could be established either by measurement of G' or by maximum creep. Tan δ was strongly influenced by protein content. Therefore, ideally, comparisons among samples of durum wheat semolina should be made on samples grown under similar environmental conditions. The large deformation creep method had the advantage that one could discern very quickly from the maximum height of the curve (% strain), without any further calculations, the relative strength and extensibility of dough samples.

TABLE IV
Italian Durum Wheat Physical Dough and Dynamic Rheological Properties

	Creso	Grazia	Simeto
Protein, %	9.7	11.1	10.0
Farinograph			
Water absorption,%	57.4	58.0	57.4
DDT[a], min	3.3	4.0	2.8
Stability,min	7.3	6.6	8.5
Micro-mixograph[b]			
MT, min	4.43a[c]	3.86b	4.07ab
PDR, units	50.6b	62.0a	58.5a
MS, units	21.9b	29.3a	27.2ab
WI, % torque min	174a	163a	169a
Alveograph[d]			
P/L	3.5	1.6	2.6
$W \times 10^{-4}$ J	245	275	231
Dynamic oscillatory[e]			
G', Pa	16,204a	13,695b	16,300a
tan δ	0.427a	0.427a	0.416b

[a] Dough development time.
[b] MT = mixing time; PDR = peak dough resistance; MS = mixing stability; WI = work input.
[c] Values followed by the same letter are not significantly different.
[d] P/L = peak height/curve length; W = work of deformation until rupture.
[e] G' = storage modulus; tan δ = shear loss modulus/storage modulus.

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